

NEW GENERATION NUCLEAR MICROPROBE SYSTEMS

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ABSTRACT

Over the past 20 years, the minimum probe size for nuclear microscopy has stayed around 1 micrometre. No breakthroughs in nuclear microprobe design have been forthcoming to produce dramatic improvements in spatial resolution. The difficulties of breaking the constraints that are preventing reduction of the probe size have been well recognised in the past.

Over the past 5 years it has become clear that some of these constraints may not be as limiting as first thought. For example, chromatic aberration clearly is not as significant as implied from first order ion optics calculations. This paper reviews the constraints in view of the increased understanding of the past 5 years and looks at several new approaches, presently being evaluated in Melbourne and elsewhere, on how to make progress. These approaches include modified RF ion sources for improved beam brightness and exploitation of relaxed constraints on some lens aberrations allowing the use of high demagnification probe forming lens systems.

INTRODUCTION AND MOTIVATION

The quest for improved spatial resolution in nuclear microprobes has a long history and is the common aspiration of many workers in the field [1]. The first nuclear microprobe system was constructed at Harwell around 1970. It attained a spatial resolution of 2.2 microns with a beam current of 250 pA as recorded in a list of nuclear microprobes compiled by Cookson in 1987 [2]. The beam current is given because, traditionally, spatial resolution measurements are divided into two regimes. These are the high current regime, for a nominal 100 pA beam current, usually regarded as the minimum possible for measurements involving Rutherford Backscattering Spectrometry (RBS) or Particle Induced X-ray Emission (PIXE) and the low current regime for 1 fA or lower beam currents, usually regarded as being appropriate for the techniques of Scanning Transmission Ion Microscopy (STIM) or Ion Beam Induced Charge (IBIC). In the discussion to follow, it is the high beam current regime that applies unless otherwise stated.

As recently pointed out by Doyle in 1999 [3], there has been little progress in making significant improvements in spatial resolution for many years. In fact, in view of the slow progress over the past 30 years, the early result from the Harwell system was very impressive.

The state-of-the-art has now attained sub-micron resolution of around 0.3 micron. The path to this result is shown in the selected and representative record of the quest to improve the spatial resolution of a nuclear microprobe in figure 1 where it is contrasted with the progress in integrated circuit technology. If nuclear microprobe resolution had followed integrated circuit technology, we would have routine sub-100 nm resolution today. Instead spatial resolution been stalled around the 1 micron level regardless of the nature of the probe forming lens system or the particle accelerator that provides the beam. Also, it is clear that progress in the low beam current regime is similarly stalled at around the 100 nm level. The system independence of these results are an indication that some of the limits are external to the nuclear microprobe system itself.

The reasons why the progress has been stalled around the 1 micron barrier for so long has been discussed by many authors [3], [15]. In summary, the reasons are generally attributed to:

- geometrical constraints in the specimen chamber requiring a long working distance due to the requirements of the detectors of the induced radiation or particles,
- limits to the strength of the focusing fields,
- limits due to lens focusing aberrations in particular chromatic and spherical aberration, the influence of stray (parasitic) fields or vibrations
- limitations in the brightness of the ion source.

It is the purpose of this paper to examine the constraints with the view to reviewing new generation probe forming systems that promise to evade these limits in the quest for improved spatial resolution. The discussion is mainly confined to conventional systems of magnetic quadrupole lenses for two reasons. The first is that the cost of purchase, installation and operation of these types of lenses are generally lower than

alternatives such as superconducting solenoids. The second is that the alternatives do not yet appear to offer significant performance advantages and some of the general conclusions presented here may also apply to the alternative systems.

The motivation to improve spatial resolution is clear. A review of the previous conference on nuclear microprobe technology and applications, held in Capetown, South Africa [16], revealed over 100 applications for the nuclear microprobe. To gain an overview of the demand for higher resolution probes, the resolution required for each of the 100 applications was assessed by a cursory inspection of the published images and maps. The histogram of required resolution for each application is shown in figure 2. This figure shows very clearly that the applications were crowded against the “left wall” of 1 micron resolution. It is therefore obvious that many of these published applications, as well as many new applications, would spill over into the sub-micron regime if it became routinely available.

A NEW LOOK AT OLD PROBLEMS

What follows now is a discussion of each of the problem areas where the nuclear microprobe system has been limited and ways are sought to overcome them. The usual starting point of any such discussion is the theory of ion optics [17] where the position of an ion in a focused probe is given by:

$$x_i = (x/x)x_o + (x/q)q_o + (x/qd)q_o d_o + (x/q^3)q_o^3 + (x/qf^2)q_o f_o^2$$

$$y_i = (y/y)y_o + (y/f)f_o + (y/fd)f_o d_o + (y/j^3)f_o^3 + (y/q^2 f)q_o^2 f_o$$

where, with reference to figure 3, (x_o, q_o, y_o, f_o) represents the position and divergence of a beam particle traveling in the z-direction in the xoz and yoz planes from the object, d_o is the particle momentum relative to the mean momentum, the demagnification of the system is $(x/x)^{-1}$ and $(y/y)^{-1}$ in the xoz and yoz planes, (x/q) , (y/f) are the astigmatism coefficients, (x/qd) , (y/fd) are the chromatic aberration coefficients and (x/q^3) , (x/qf^2) , (y/f^3) and $(y/q^2 f)$ are the spherical aberration coefficients. In a well-focused system, the astigmatism coefficients are zero. Additional terms may arise in this expansion owing to parasitic aberration and fifth order aberration that arises substantially from the lens fringing fields. With modern manufacturing methods, parasitic aberrations can essentially be eliminated. Fifth order aberration will be neglected in the following discussion, but some of the

comments below for spherical aberration also apply to fifth order aberration. In what follows, each term is examined to determine at how it may be minimised.

Chromatic aberration

It is tempting to measure the actual momentum spread of the beam from the accelerator and use the formula above to calculate the best probe resolution. Contributions to the final probe size from each of the terms in the formula can be added in quadrature, or a similar method [15]. Invariably these methods overestimate the best probe resolution. The famous case of the Oxford system is a good example. Addition in quadrature gives a figure of 0.9 micron [15], but the experimental result was about 0.3 micron [6], since also equalled by the Oxford system employed in Singapore [9]. Both experimental results are considerably better than theory. The case of the Singapore result is particularly interesting. It must be noted that the Singapore system employs a bright beam accelerator connected to a 15 degree switching magnet to provide beam for the microprobe. This system would not be expected to provide a beam with high chromatic purity. This should be particularly evident with an Oxford high excitation triplet probe forming lens system that has a large chromatic aberration coefficient and so should be particularly sensitive to chromatic aberration. Skilled operation and tuning is no doubt a factor in the excellent performance of the system, but the chromatic aberrations should be severe.

The reason why the theory and the experiment were in such disagreement is not because the theory is wrong. The chromatic aberration coefficients can easily be determined by deliberate changes in the beam energy and measurement of the resulting increase in probe size. The result agrees with theory. Instead, the reason for the disagreement must be because the beam intensity distribution in phase space is highly non-uniform. It is clear that only a small proportion of the beam particles passing through the object collimators have both a large divergence and a large energy error.

Measurement of this effect is difficult, but the ray tracing program MULE [18] can be used as a guide. Figure 4 shows a plot of the cross section of the beam phase space from the HIAF accelerator in Sydney. The phase space cross section was obtained by tracing 14,000 rays from the ion source, through the tandem accelerator (including gas stripper) and the energy analysing magnet to the object collimator. The cross section

shown in figure 4 is taken in the q_o, d_o plane. The cross section shows the high degree of non-uniformity of the beam in this plane. Clearly the intensity of the beam which has both large divergence and large energy spread is very low. The non-uniform phase space of the beam from a typical accelerator produces a far lower beam momentum spread that could be expected if the phase space was uniformly filled. So the consequences of chromatic aberration are not as severe as for a uniform phase space.

Spherical Aberration

The spherical aberration coefficients can be calculated by a variety of models. One widely used model, the rectangular model, considers the field of the quadrupole lenses to have a rectangular profile as a function of the beam direction, z . In this model the spherical aberration coefficients have been shown to be in reasonably good agreement with the coefficients calculated by more sophisticated models. It is a characteristic of many quadrupole lens systems that the coefficients are large. But how good is the rectangular model in reality? Measurement of the spherical aberration coefficients is difficult and there are few reports in the literature. So design of probe forming lens systems is strongly influenced by theory often trading off larger demagnification in favour of smaller spherical aberration.

This conservative approach may lead to a less than optimum design. In fact work by Moloney [19] has now shown that the rectangular model may be inadequate for real quadrupole lenses used on nuclear microprobe lens systems. In his work, detailed three dimensional magnetic field profiles were measured from actual lenses from the Melbourne system. From these detailed field profiles, the spherical aberration coefficients were extracted using numerical ray tracing techniques. The numerical techniques were shown to be self-consistent and independent of the integration algorithm. Large discrepancies were found in the spherical aberration coefficients calculated from the true field profile compared to the rectangular model. In particular, the spherical aberration cross terms were found to be considerably smaller than those given by the rectangular model.

It is not surprising that a discrepancy arises from the fringe fields because they are not accurately modeled by the rectangular model. The fringe field profile needs careful measurement. Conventional measurements are done by fixing the radial position of

the magnetic field sensor as it is moved along the z-axis. Moloney has shown that this cannot detect some important harmonics in the fringe field region and several profiles are required at differing radii for accurate measurements.

The results from the accurate fringe field measurements have implications for lens system design. This is because the spherical aberration cross terms, typically very large in the rectangular model, have a strong influence on the ultimate probe size. This implies that a real lens system is much less sensitive to spherical aberration than implied by most of the theoretical models employed for lens system design that either employ the rectangular model or trace rays through a field profile measured at a single radius from the axis. Furthermore, the flux peaking effect, discussed below, where the beam entering the nuclear microprobe system is concentrated in the paraxial region, also means that the spherical aberration does not grow as fast as expected when the aperture collimator is made larger. These two conclusions mean that spherical aberration may not be such a severe problem for smaller probes.

The Magnification

Having dealt with the aberrations it is now possible to investigate the contribution from the demagnified image of the object. Clearly the magnitude of the demagnification terms $(x/x)^{-1}$ and $(y/y)^{-1}$ should be as large as possible. Indeed this is the route taken by many designers. But the quest for greater demagnification is moderated by the fears of large aberrations because these typically increase as the magnification decreases. This is because for systems with large demagnification, the convergence angle at the image plane must be large and the working distance, from the last lens to the image plane, must be small. But the fear of large aberrations may be unjustified as has been shown. Therefore every effort should be made to increase the demagnification. This is usually done by decreasing the distance from the last lens to the specimen, or by introducing one or more internal beam cross-overs within the probe forming lens system as is done in several of the next generation systems.

To date, lens design for nuclear microprobes has been rather conservative compared to the complex shapes often seen in high resolution electron microscopy. As the first step towards more versatile design, the Oxford compact system [20] and the new CSIRO/MARC quintuplet [21] have employed new types of lenses that have shapes that depart from traditional designs. Both designs include pole overhangs that allow a

stronger lens to occupy the same width as a conventional lens and the working distance to be reduced. In addition the lenses for the CSIRO/MARC quintuplet include yoke cut-outs that allow access for detectors to the front of the specimen. No penalty in the lens field quality has been incurred for these innovations [21]. More radical designs are possible.

Ion Source Brightness

It has been pointed out in a previous paper [15] that an order of magnitude improvement in the spatial resolution requires 5 orders of magnitude improvement in the beam brightness from the accelerator. It must be noted that this conclusion is based on the assumption that the phase space from the accelerator is completely and uniformly filled. However this is not the case. A large collection of measurements from accelerators worldwide showed that most accelerators provide a beam in which the beam flux is strongly intensity peaked in the paraxial region [22]. This effect is seen clearly in the results from the Melbourne 5U Pelletron accelerator which has recently been subject to a major upgrade consisting of replacement of column corona needles with resistors and a complete upgrade of the ion source with stabilised power supplies. Strong flux peaking in the paraxial regime is observed as seen in figure 5. As discussed previously, this flux peaking means that angle dependent aberrations, like spherical aberration, are far less important than for a uniform phase space. In fact if the angle dependent aberrations can be assumed to be always negligible (possibly a rather unrealistic assumption) then the probe diameter in the xoz plane in microns is given by:

$$d_i = 2M \sqrt{\frac{i}{\pi B E A_a / d^2}}$$

where the magnification $M=(x/x)$, i is the beam current (pA), B is the brightness (pA/($\mu\text{m}^2\text{mrad}^2\text{MeV}$), E is the beam energy (MeV), A_a is the area of the aperture collimator (mm^2) and d is the distance from the object to the aperture collimators (m). This expression is for circular object collimators. For square, replace π by 4. So one order of magnitude improvement in spatial resolution requires just two orders of magnitude improvement in brightness. This is considerably less than the 5 orders of magnitude for a uniform phase space. The true figure will be somewhere between 2 and 5, but this reasoning indicates that relatively modest improvements in beam

brightness can be beneficial. In fact the recent upgrade to the Melbourne system resulted in a factor of about 10 improvement which has the potential to lead to an improvement in spatial resolution by a factor of about 3.

For the future, further modest improvement in beam brightness from the Melbourne RF ion source are possible by reducing the gas scattering that occurs when the ion beam emerges from the ion source itself. This can be accomplished by the installation of a recirculating turbopump that pumps the gas away from the exit of the ion source and recirculates it back into the ion source itself. Thus the low energy ion beam emerging from the source does not have to travel through a relatively dense gas cloud at the entrance of the accelerator. Tests of this system are scheduled for late 2000.

Stray Fields

There can be no doubt that the influence of external magnetic fields can be the most significant source of resolution degradation in many laboratories [23]. A very simple demonstration of the effect of a stray DC field is shown in figure 6. A small bar magnet, field strength less than 0.5 T was brought near to the beam pipe just upstream of a lens being tested by the grid shadow method [17]. The huge sextupole aberrations introduced are clearly seen in the shadow pattern. Other non-uniform DC fields in the laboratory, particularly those that extend over a large part of the beam line, can be expected to have a similar effect.

A particularly troublesome source of stray DC fields is the beam pipe itself. Figure 7 shows the effect of small displacements (less than 1 mm) of a stainless steel (type 316) beam pipe (external diameter 10 mm) towards the beam axis at a point 0.25 m upstream of a lens being tested by the grid shadow method. Dramatic parasitic aberrations clearly arise from the beam tube. Such aberrations of the Melbourne system have been measured by the beam rocking technique [24] which is very sensitive to the multipole contamination of the entire beam line. The beam tube responsible for figure 7 was shortly thereafter replaced by an aluminium tube.

Stray AC fields are also very serious. In this case the field bends the beam axis causing a virtual displacement of the object that is translated onto the image plane (specimen) by the lens system. As the field strength varies with time, the position of the virtual object varies leading to a blurred probe. The shift of the focused probe on

the specimen, Δx_i , as a function of the strength of the external field (assumed to bend the beam with a radius less than the length of the beam line) is:

$$\Delta x_i = \frac{MqB_{stray}L^2}{2\sqrt{2Em}}$$

where q is the beam particle charge, B_{stray} is the amplitude of the stray field which is assumed to fill the entire laboratory, L is the length of the beam line upstream of the probe forming lens system, E is the beam energy and m is the beam particle mass. This formula shows that if Δx_i is to be kept below 0.1 μm , B_{stray} must be less than 20 nT. This is a very stringent requirement. In Melbourne, most of the beam line is shielded with 2 layers of mu-metal wrapped on the beam tube together with an outer shield of an iron pipe with a wall thickness of 1 cm. The effect of this shield is as follows: with no shield in place the stray magnetic field typically varies with an amplitude of about 450 nT and a period of about 1 s. With the iron shield alone, the amplitude is reduced to 130 nT and with both the iron and the mu-metal the amplitude is 40 nT. So the shield attenuates by a factor of about 10 but, under these conditions, the spot size will be limited to no smaller than 0.2 μm owing to the effects of the stray field.

Despite the use of heavy shielding, the stray AC fields still have a dismal effect on the Melbourne system as shown by the image in figure 8. This image was obtained with the vertical scan coils disconnected and the data acquisition system set to produce a fast horizontal raster with a slow vertical scan. The specimen was a 2000 mesh grid. The horizontally scanning beam stays fixed at the constant vertical position, but the data acquisition system continued to update the vertical coordinates. The resulting map is therefore an image of the relative position of the vertical grid bars as a function of time. Simultaneously the vertical component of the magnetic field, which will cause a horizontal shift in the position of the grid bars, was measured in the laboratory with a FM300 fluxgate magnetometer sensitive to 1 nT [25]. Figure 8 shows the resulting map with the magnitude of the magnetic field superimposed. It can be seen that the position of the grid bar edges faithfully follow the fluctuations in the external magnetic field which had an amplitude on this day of 2000 nT. The presence of heavy mu-metal and iron magnetic shielding on most of the beam pipe was inadequate under these conditions.

Shielding these stray fields remains a matter of pressing concern for the Melbourne group. The fields were not always so bad. However five years ago traffic on an electric tram route that runs within 50 m of the laboratory was increased by an order of magnitude and this is the most likely the source of the stray fields.

One solution to the stray field problem is shown in figure 9. Between 2 am and 4 am the stray fields reduce to negligible amplitude. Such hours are not very popular with the operators. Heavier shielding will clearly be required, but care must be taken to avoid repeating the experience of the stainless steel beam tube: residual fields in the shielding material must not introduce parasitic aberration into the beam.

CONCLUSION

There are many additional factors that can be taken into consideration in the quest for improved spatial resolution in addition to the factors discussed above. One obvious example is to increase the solid angle and efficiency of the detectors allowing useful data to be obtained with lower beam current. This will also have the very beneficial effect of reducing specimen damage. But it seems that x-ray detectors are limited to 80 mm² and particle detectors have similar limitations.

Breaking the 100 nm barrier will not be easy but it is clear that there are several new directions that are possible. Some of these directions are already well known in the field. In summary, the directions discussed here were:

- Construct the beamline with heavy magnetic shielding to prevent the influence of stray fields but ensure that residual DC fields in the shielding do not introduce parasitic aberrations.
- Increase the demagnification over existing systems by reducing the working distance as much as possible. The final lens could actually be part of the specimen chamber to allow very close proximity of the lens to the specimen. Aggressive cut-outs in the yoke would be provided to allow detectors access to the front of the specimen.
- Flux peaking reduces the impact of aberrations and favours systems with a large demagnification. This also means that relatively modest increases in the beam brightness, can have a significant benefit.

Some of these ideas are already close to fruition. These include the new compact Oxford system [26], for which performance data was not available at the time of writing, but the design looks extremely promising. The CSIRO/MARC quintuplet system [21] has produced excellent results with extremely intense probes being produced despite the relatively low brightness of the associated accelerator. The Leipzig separated doublet [27] has also given good performance and is an example where the demagnification has been maximised. In each case the price paid for high demagnification in a quadrupole system is increased spherical aberration as can be seen in figure 10 which compares the average demagnification and average spherical aberration of these new systems. The Bochum superconducting solenoid offers very low spherical aberration at the cost of running a liquid He plant and may represent the practical limit in the demagnification obtainable from such systems. As discussed, the degradation to the probe resolution due to spherical aberration in the quadrupole systems is abated by the flux peaking effect. Peak performance of these new systems will require stringent elimination of stray fields and specimen drift. But it is the view of the present author that significant further performance is possible in the near future.

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FIGURE CAPTIONS

Figure 1: Progress in the improvement in the spatial resolution of the nuclear microprobe compared to progress in the reduction of feature size in integrated circuit technology (“Moore’s Law). The solid line is the trend in the minimum feature length in integrated circuits and has been taken from Sze [4]. The squares represent the technology used in Intel central processor units. From left to right these are the 8086, 80386, Pentium P5, Pentium Pro (P6) and Merced (P7) [5]. The triangles represent selected nuclear microprobe spatial resolution measurements taken from the literature for high beam current techniques. The circles similarly for low beam current techniques. References for these measurements are in Table 1.

Figure 2: An overview of the spatial resolution employed in the 100 applications presented at the previous conference on nuclear microprobe technology and applications [16]. The nominal probe size required to resolve structures in the specimens was sorted into bins $>100\ \mu\text{m}$, $50\text{-}60\ \mu\text{m}$, $30\text{-}40\ \mu\text{m}$, $10\text{-}20\ \mu\text{m}$, $15\text{-}10\ \mu\text{m}$, $4\text{-}6\ \mu\text{m}$, $2\text{-}3\ \mu\text{m}$, around $1\ \mu\text{m}$ and sub-micron to produce this histogram.

Figure 3: Designation of object ray vector and its corresponding coordinates in the image plane. Note that not all possible coordinates in phase space may be occupied by an actual ray.

Figure 4: A slice of the object plane phase space calculated from the ion optics program MULE [18] for the HIAF system in North Ryde, Sydney [21] by tracing 14,000 rays from the ion source to the object collimator (object plane) of the nuclear microprobe system. The cross indicates the axis of the system. The phase space is very sparse in the high divergence, high momentum spread corner (top right).

Figure 5: Brightness of the Melbourne 5U pelletron accelerator immediately following a major upgrade in mid-2000. Data are for a 2 MeV He^+ beam for object diaphragms diameters of $200\ \mu\text{m}$ (squares), $100\ \mu\text{m}$ (triangles) and $75\ \mu\text{m}$ (circles).

Figure 6: The effect of the inhomogeneous DC magnetic field from a small bar magnet placed near the beam line upstream of a magnetic quadrupole lens being tested by the grid shadow method. Huge parasitic aberrations, in the form of sextupole field components, are introduced.

Figure 7: The effect of a stainless steel beam pipe passing through the bore of a magnetic quadrupole lens being tested by the grid shadow method. Bending the beam tube to bring it into closer proximity to the beam at a point just upstream of the quadrupole lens introduces severe sextupole (and higher order) aberrations.

Figure 8: Horizontal line scans of a fixed grid were collected as a function of time to make a map of the grid bar positions as a function of time. The y-component of the stray magnetic fields in the laboratory was measured simultaneously and is shown superimposed on the map. The position of the grid bars faithfully follows the slowly varying stray field.

Figure 9: The magnitude of the stray magnetic field in the MARC laboratory measured on April 18 2000 commencing at 6pm. The field was measured by a FM300 fluxgate magnetometer.

Figure 10: A comparison between several new generation systems on the basis of their average demagnification and average spherical aberration coefficients.

Table 1:

| Place | Spot size (micron) | Current | Year | Comments (magnetic quadrupole lenses unless stated) Where no reference is given the data was taken from Cookson [2] |
|----------------------------|--------------------|---------|------|---|
| High current regime | | | | |
| Harwell | 2.2x2.2 | 250 pA | 1970 | Quadruplet or triplet on vdG |
| Heidelberg | 2x2 | 150 pA | 1977 | Quadruplet or doublet on tandem |
| Zurich | 11x26 | 400 pA | 1978 | 2 separated doublets on tandem |
| Karlsruhe | 2x2 | 600pA | 1979 | Doublet on vdG |
| Oxford | 1x1 | 80 pA | 1981 | Triplet on tandem |
| Sandia | 2 | 40 pA | 1982 | Doublet on tandem |
| Amsterdam | 3x8 | 50 pA | 1982 | Doublet on cyclotron |
| Lower Hutt | 10x10 | 500 pA | 1982 | Quadruplet on tandem |
| Melbourne | 1 | 100 pA | 1982 | Quadruplet on vdG |
| Lund | 15x15 | 450 pA | 1983 | Quadruplet on vdG |
| Albany | 2x2 | 200 nA | 1985 | Doublet on tandem |
| North Ryde | 5 | 100 pA | 1986 | Electrostatic quadruplet on tandem |
| Oxford | 0.3 | 100 pA | 1991 | Triplet on tandem [6] |
| JAERI | 0.4 | 77 pA | 1995 | Doublet on tandem [7] |
| Bochum | 0.6x0.7 | 100 pA | 1996 | Superconducting Solenoid [8] |
| Singapore | 0.3 | 100 pA | 1998 | Triplet on vdG [9] |
| Low current regime | | | | |
| Melbourne | 0.1 | 0.06 fA | 1987 | Quadruplet on vdG [10] |
| Melbourne | 0.05 | <0.1 fA | 1989 | Quadruplet on vdG [11] |
| Singapore | 0.1 | n.s. | 1995 | Triplet on vdG [12] |
| Schonland | 0.59 | n.s. | 1997 | Triplet on tandem [13] |
| Singapore | 0.1 | 1pA | 1998 | Triplet on vdG [9] |
| Singapore | 0.15 | n.s. | 1999 | Triplet on vdG – micromaching PMMA [14] |