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Beam Interactions with Materials & Atoms

# The nuclear microprobe: a unique instrument

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**Abstract**

There are several features characterising high-energy ion interactions with matter which enables techniques which utilise the high-energy ion beam to have unique or advantageous characteristics. (i) The lack of primary particle bremsstrahlung enables Particle Induced X-ray Emission (PIXE) to be carried out with very little X-ray background, and therefore correspondingly high analytical sensitivity. (ii) The ion beam can penetrate many microns beneath the surface of the specimen with relatively little scattering, and therefore information on structures beneath the surface can be investigated at relatively high spatial resolution. (iii) The ion beam has a well defined depth in the sample, the depth depending on its energy, and this allows more complex three-dimensional structures of better depth definition to be micromachined compared with its X-ray counterpart LIGA. Illustrating these three important features are descriptions of recent work carried out on the elemental analysis of single cells, determination of the annealing efficiency of phosphorous implanted diamond, multi-layer integrated circuit fault finding, and proton micromachining of high aspect ratio structures.

## 1. Introduction

The Nuclear Microprobe (or Nuclear Microscope) is a relatively complex instrument based around a small particle accelerator and a sophisticated high-energy (MeV) ion focusing system (Fig. 1). Over the last two decades nuclear microprobe technology has improved steadily, such that spatial resolutions at the 100 nm level can now be achieved. Simultaneously, there has been a large increase in the development of available techniques in which the interaction between the high-energy ion probe and the sample can be utilised (see Table 1). These parallel developments have resulted in a versatile analytical instrument of great potential, which is providing characterisation and analyses of many samples over a wide range of scientific disciplines. There are however many other types of analytical instruments commercially available which utilise a variety of other probes (e.g. low-energy heavy ions, electrons, X-rays, UV, laser light) and which detect an equally diverse range of interaction products. Currently, many of these instruments are easier to use, cheaper to buy and maintain, occupy less space, and can perform specific analyses faster and more sensitively than the nuclear microprobe. It is important therefore to identify the areas in which the nuclear microprobe offers either unique information or highly advantageous features.

The features which allow the nuclear microprobe to provide unique or novel information from a specimen are linked to the physical properties of the high-energy ions and their interaction with matter. This paper describes three of these properties, and illustrates how these properties can be utilised in a unique or advantageous manner by referring to recent examples utilising the nuclear microprobe.


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## 2. Some unique characteristics of the nuclear microprobe

In general the path of the high-energy ion in matter is determined by electronic collisions, since the probability of a nuclear collision is relatively small. Since the average energy transfer per electron collision is relatively low (due to the large mass difference between the ion and the electron), then many thousands of collisions occur and the path can be statistically predicted (see for example [1] and the computer code TRIM [2]). The ion path can be characterised by three important features:

(i) The degree of scattering per collision is small [3]. The ion does not suffer large angle collisions and travels in an approximate straight line as it passes into the sample. One consequence of the lack of large angle scattering coupled with the small transfer of energy per collision, is that a focused ion beam will not directly produce bremsstrahlung radiation, and therefore the corresponding background X-ray radiation is extremely low.

(ii) The ion penetrates the surface of the specimen and travels through many atomic layers before it comes to rest. The range is therefore relatively high, particularly for high-energy light ions such as protons. A consequence of the relatively high range, e.g. 2 MeV protons will penetrate 47 microns in silicon, is that information can be extracted from below the surface of the sample. An added advantage is that due to the lack of large angle scattering, a focused proton beam will effectively maintain its spatial resolution as it penetrates the specimen. This is particularly true for thin samples, e.g. a 2 MeV proton beam travelling through a carbon film of 5 microns, will experience less than a 20 nm spread in the beam profile.

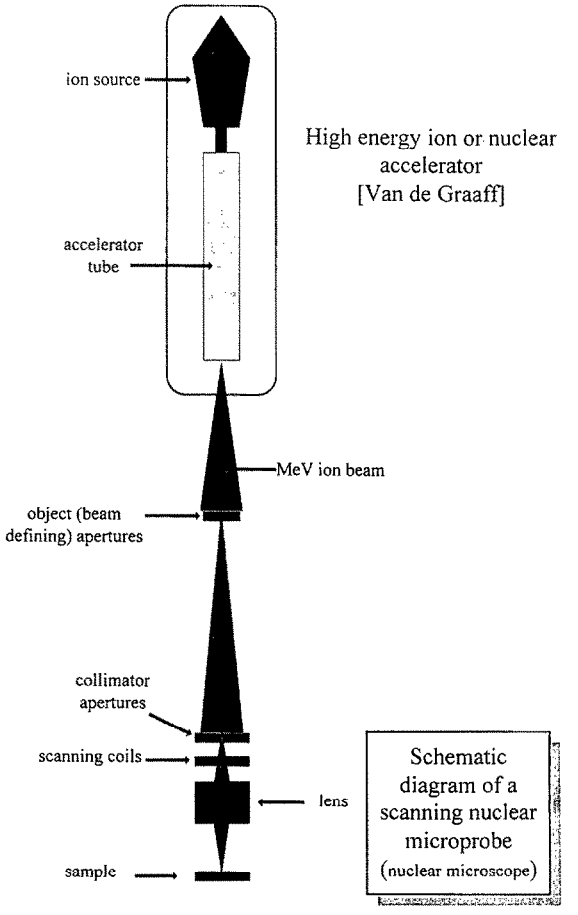


Fig. 1. Schematic diagram of a scanning nuclear microprobe (or nuclear microscope): A beam of high-energy ions pass through an object aperture and are demagnified by a strong focusing lens system to form a probe at the sample.

Table 1
High-energy ion beam techniques

| |
|---|
| Particle Induced X-ray Emission (PIXE) |
| Rutherford Backscattering Spectrometry (RBS) |
| Scanning Transmission Ion Microscopy (STIM) |
| Ion beam Induced Charge (IBIC) |
| Ion beam Induced Luminescence (IBIL) |
| Secondary Electron Imaging (SEI) |
| Single ion machining |
| Single event upsets |
| Single ion irradiation |
| Deep ion beam lithography |
| Ion beam tomography |
| Microchanneling using RBS, PIXE or STIM |

(iii) Due to the statistical nature of the collision process, the degree of straggling is small and can be as little as a few percent e.g. the range straggling of 2 MeV protons in silicon is approximately 1.6 microns. The ions therefore travel through the material and stop at approximately the same depth in the sample. Utilising these three characteristics can greatly enhance the potential of the nuclear microprobe as described below.

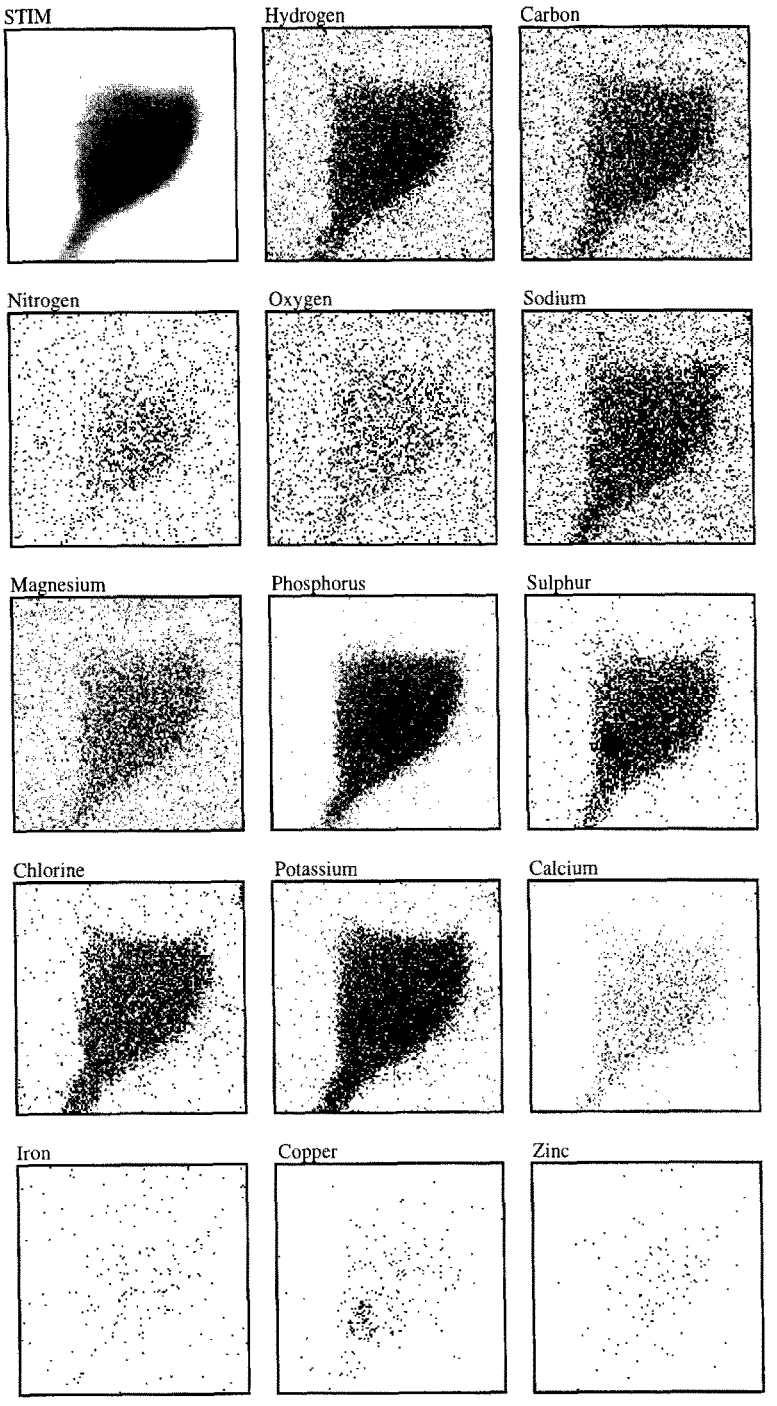


Fig. 2. Off-axis STIM, RBS and PIXE maps from a single cancer cell which has been grown in culture, rinsed in sucrose solution and freeze dried. Scan size 50 microns; 2 MeV protons.

## 3. Quantitative microanalysis at micron spatial resolutions

Due the almost complete lack of background X-ray radiation from primary particle bremsstrahlung, Particle Induced X-ray Emission (PIXE) exhibits a greatly superior analytical sensitivity compared with the analogous electron probe microanalysis. Minimum detection limits for PIXE of 1 part per million are common [4], and when used in conjunction with Rutherford Backscattering Spectrometry (RBS) [5], and Scanning Transmission Ion Microscopy (STIM) [6], a powerful and highly quantitative package of ion beam analysis techniques results (see for example [7]).

One of the many research fields in which quantitative microanalysis at high spatial resolutions can be utilised to good effect is in the analysis of single biological cells [8]. Cells grown in culture can be prepared in a monolayer for nuclear microprobe analysis such that their ionic integrity is preserved [9], and subsequent mapping with a proton beam can yield whole cell elemental concentrations at the parts per million level. Fig. 2 shows the type of information that can be extracted from a single cell; off-axis STIM provides information on the shape, density and hydrogen concentrations, RBS provides C, N and O concentrations, and PIXE the concentrations of Na, Mg, P, S, Cl, K, Ca, Fe, Cu and Zn and other trace elements present down to the ppm level. This type of analysis is important because it can provide quantitative information on the uptake of elements by the cell, or its elemental response to a wide variety of external stimuli (see for example [10]).

The analysis of cells in situ is also possible (see for example [11]) by identifying cells in thin tissue sections using STIM and then analysing using PIXE and RBS. This approach has the added advantage that the elemental levels of the cell can be assessed while in its natural surroundings, thereby providing valuable information on various disease states. This has been particularly useful in the study of degenerative diseases such as Alzheimer's disease, Atherosclerosis, Parkinson's disease etc. (see for example [12,13]).

It is worth noting that the extraction of elemental information at the ppm level and submicron elemental mapping of biological cells can also be achieved by Secondary Ion Mass Spectrometry (SIMS) [14]. Although SIMS has the added advantage that isotopes can also be mapped, it is not a quantitative technique. The unique feature of the nuclear microprobe is that quantitative trace elemental analysis at high spatial resolutions can be readily achieved.

## 4. Sub-surface analysis

The penetration of the ion beam into the sample allows analysis not only at the surface of the specimen, but also from the underlying atomic layers. A well known example of this is the use of broad MeV alpha beams for the non-destructive analysis of layered samples using RBS [5]. The use of a highly focused scanning ion beam can provide three-dimensional information from a sample. One of the interesting areas which is being vigorously pursued in many countries, particularly Japan, Australia, the USA and Singapore, is the use of the nuclear microprobe in the micro-electronics industry. The example chosen to illustrate the potential of the nuclear probe

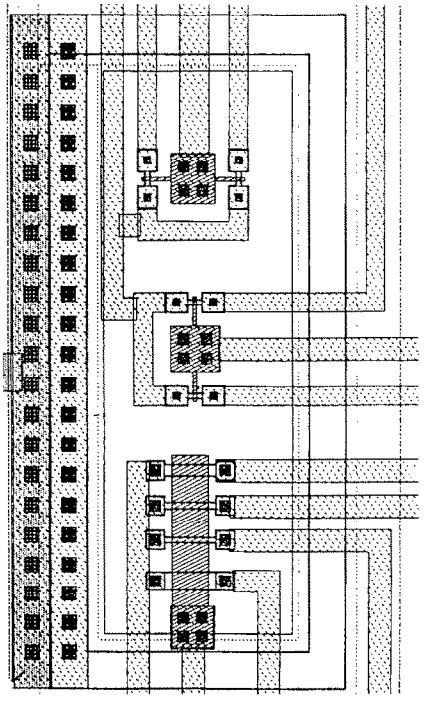

Fig. 3. Metallisation layout of a multilayer IC with submicron features.

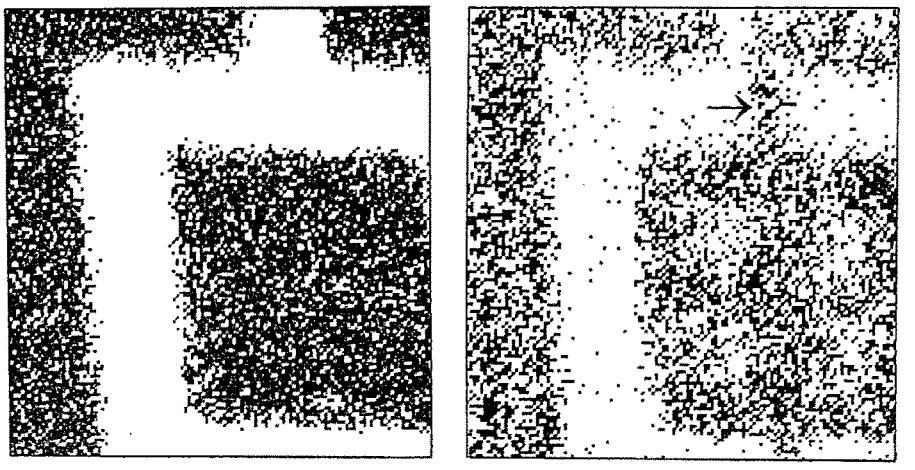


Fig. 4. High magnification PIXE images (W and Ti) from the central region of the IC depicted in Fig. 3. Scan size 20 × 20 microns. 3 MeV protons.

for non-destructive three-dimensional analysis is the characterisation of a faulty multi layer IC with sub-micron features [15]. Fig. 3 shows the layout of the IC, which has three metallisation layers, and Fig. 4 shows titanium and tungsten PIXE images (corresponding to one of the structures just above the centre of the layout diagram). Comparing Figs. 3 and 4, it is clear that a Ti connection is misplaced by about 1 micron (see arrow in Fig. 4), which is the probable reason why this IC was faulty. STIM images of the sample after thinning [15] confirmed the diagnosis. Further information extracted using RBS yielded the thickness of the metallisation layers, and also that the IC was covered with a 4.1 micron passivation layer of silicon dioxide. It perhaps can be emphasised that although many other techniques had been previously applied used on this IC, only the subsurface high-resolution analysing capability of the nuclear microprobe was able to identify this fault. In addition, the extraction of information at depth in the IC is not limited to the use of PIXE, RBS and STIM. Recent advances in the technique Ion Beam Induced Charge (IBIC) microscopy (see for example [16]) enables information on the active regions of multilayer ICs to be mapped by detecting the electron/hole pairs produced by the ion beam.

A second example describes the unique role of the nuclear microprobe in the subsurface analysis of laser annealed phosphorus implanted diamond [17], the successful development of which would have important consequences in the micro-electronics in-

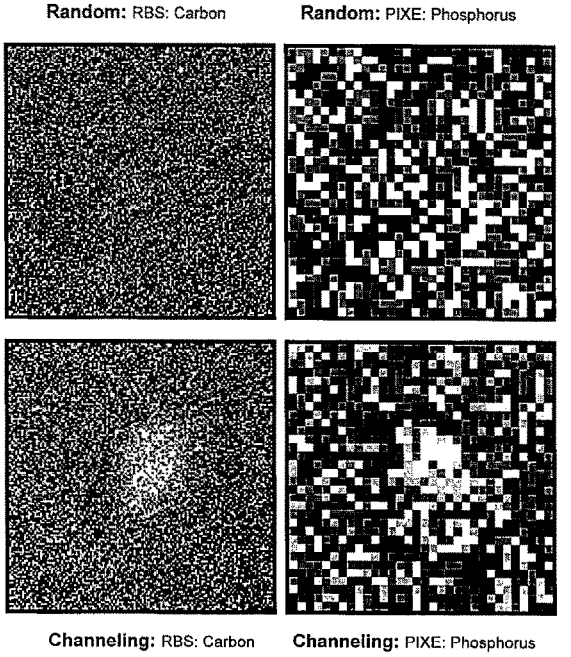


Fig. 5. Random and channeling RBS and PIXE maps of laser spot annealed phosphorous implanted diamond. Scan size 40 microns; 1 MeV protons.

dustry. Potentially the best technique for producing n-type diamond is the implantation of high-energy P ions into the pure material, provided the P ions can be incorporated into the diamond lattice sites and the damage to the diamond lattice incurred during the implantation process can be repaired. One way of annealing the sample is to use fast laser pulses, which appears to minimise the graphitisation of the matrix [17]. In order however to find optimum conditions for this procedure, a way must be found to investigate the annealing process and in particular whether the implanted P atoms have moved to the diamond lattice sites. Such a task can be performed by the nuclear microprobe using the technique of channeling [5], a technique where the ions are channelled down a crystal axis in order to determine the degree of lattice damage through interaction primarily with interstitial atoms. The task however is not easy: The implantation dose is of the order $10^{15}$ P ions/cm$^2$ (equivalent to about 1–2 monolayers), the implantation range of the 4 MeV P ions in the diamond lattice is about 1.34 microns on average (equivalent to a depth of about 3000 layers of C atoms), and the annealed region is of the order of the laser pulse size (about 10 microns). PIXE and RBS random and channeling maps over the annealed region are shown in Fig. 5, and the RBS and PIXE micro-channelling spectra extracted from the annealed spot, and from both virgin and implanted diamond are shown in Figs. 6 and 7. These results

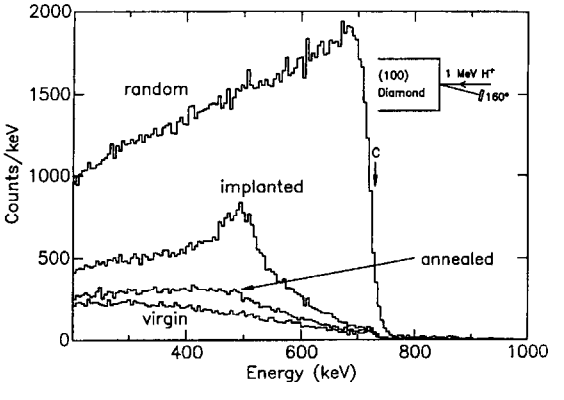


Fig. 6. RBS spectra extracted from the P implanted region (random), and channeled spectra from the implanted region, from the virgin diamond and from the regrown laser annealed spot. 1 MeV protons.

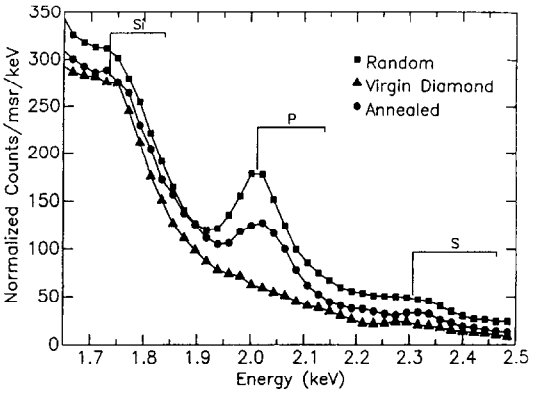


Fig. 7. PIXE spectra from the P implanted region (random), from the virgin diamond, and from the annealed spot (channeled). 1 MeV protons.

show that 40% of the P atoms in this particular implantation and laser treatment have migrated to the diamond lattice sites, and that the damage incurred to the diamond lattice during implantation is almost completely repaired.

## 5. Micromachining using high-energy light ions

One of the exciting new areas of research in recent years is the production of three-dimensional micromechanical devices with high aspect ratios e.g. activators, motors, sensors etc. Ionising radiation passing into a resist material such as polymethylmethacrylate (PMMA) will break molecular bonds and reduce greatly the length of the polymer chain. The application of a selective chemical developer can then remove the exposed resist leaving behind a specific structure in PMMA, and this structure can be further processed by electroplating, etching etc to produce a metallic micro-component. In order to machine high aspect ratio devices, the use of a penetrating beam of X-rays, usually from a synchrotron source, can being used [18]. This process, (known by its acronym LIGA) is used in conjunction with a mask, which enables a specifically designed pattern of X-rays to be transmitted through to the resist material. One consequence of using a mask is that the structures produced in PMMA are prismatic, ie the walls of the structures are invariably vertical,

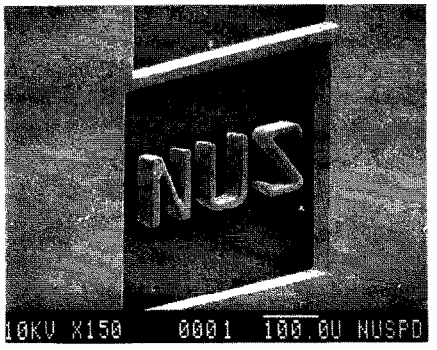


Fig. 8. Letters (60 microns deep with 10–20 micron wide walls) micro-machined into thick PMMA using 2 MeV protons.

and this feature limits the complexity of shapes which can be produced.

A high-energy ion beam also penetrates deeply into PMMA and produces exposed resist which can be selectively developed [19]. As a result, high aspect ratio structures can also be produced using a focused scanning ion beam [20] (Fig. 8). There are advantages in using high-energy ions: (i) The beam can be focused to submicron dimensions and directly scanned across the PMMA, thereby removing the requirements of using a mask. (ii) The ion beam has a well defined range in PMMA (unlike X-rays), and therefore the construction of slots, channels, holes etc will have a well defined depth. (iii) The use of ions beam of different energies enables slots, channels and holes to be manufactured with different specific depths. (iv) By changing the angle of the PMMA with respect to the beam, complex non-prismatic shapes can be machined.

The use of the focused ion beam has great potential in the new field of micro-component and micro-electro-mechanical systems (MEMS) development.

## 6. Conclusion

There are several special features when high-energy ions interact with matter which enable techniques which utilise the high-energy ion beam to have unique or advantageous characteristics: (i) The lack of primary particle bremsstrahlung enables the measurement of ion beam induced characteristic X-ray radiation (PIXE) to be carried out with very little background, and therefore much higher analytical sensitivities can be achieved compared with the analogous technique using electrons. (ii) The ion beam can penetrate may microns beneath the surface of the specimen, and therefore information on structures beneath the surface can be investigated. This information is not accessible to the many and varied techniques which rely on probing the sample surface (e.g. Secondary Ion Mass Spectrometry (SIMS), Auger Electron Spectroscopy (AES), Energy Dispersive X-ray analysis (EDX) etc.). (iii) The ion beam has a well defined depth in the sample, depending on its energy, and this allows more complex and better defined structures to be micromachined compared with the X-ray counterpart LIGA.

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