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Micron-scale analysis of SiC/SiC$_f$ composites using the new Lisbon nuclear microprobe

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Abstract

A new nuclear microprobe has now been commissioned at ITN, Lisbon. This paper first describes the layout and modes of operation of the new microprobe, which is located on a 3.1 MV single-ended Van de Graaff accelerator. Within two days of first commissioning the microprobe, a spatial resolution of ~1.5 μm was achieved for backscattering analysis, and a resolution of ~1.0 μm for transmission work. The steps taken to produce this resolution, and the remaining factors, which further limit it are discussed. The first results from this microprobe for the spatially resolved analysis of SiC/SiC$_f$ ceramic composites are presented here. These materials have applications in fusion technology and structural changes were investigated after exposure to lithium orthosilicate and lithium titanate breeder materials in fusion relevant conditions. Ti and Cr rich precipitates could be found in the samples that were exposed to lithium titanate.

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1. Experimental set-up

The layout of the new microprobe, which has recently been commissioned at ITN, Lisbon, is shown in Fig. 1. The locations of the other beam lines on the 3.1 MV single ended Van de Graaff accelerator are also shown. The microprobe is located on an existing nuclear reaction analysis beam line, mainly used for (p, γ) analysis in the past.

Two sets of micrometer driven slits are used to define the size of the object aperture and the collimator aperture which is placed to define the beam divergence into the lenses. Each slit blade is a tungsten carbide cylinder polished to better than 1 μm. There is a steering magnet located close to the object aperture to optimise the amount of

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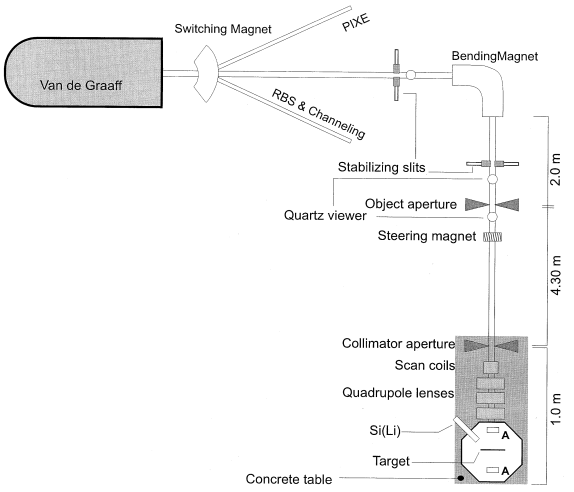

Fig. 1. Location of the ITN microprobe and its associated components on the 3.1 MV single ended Van de Graaff accelerator.

beam that is transmitted through the collimator aperture.

The microprobe lenses, scanning coils, collimator slits and chamber are mounted on a single concrete block, which rests on a layer of polystyrene to minimise the transmission of vibrations from the ground. The microprobe lens system is a high excitation magnetic quadrupole triplet [2], with dimensions as given in [3]. The object distance is 4.3 m and the image distance is 16 cm, giving demagnifications of $D_x = 49$ in the horizontal direction and $D_y = 15$ in the vertical direction as calculated using PRAM computer code. [1] The main limitation on the demagnification is the restricted beamline length, which limits the object distance. These values were also measured by observing the area of the divergent beam on a glass at the end of an extension tube positioned in the back of the chamber. The measurements were made for a fixed beam divergence θ_0 into the lenses in the horizontal and vertical directions. The beam is focused in the chamber such that the angle of the beam in the image plane θ_i can be measured. The demagnification in a particular plane is given by the ratio θ_i/θ_0. In this way a demagnification of $D_x = 47$ and $D_y = 11$ could be measured. The larger difference observed between the measured and calculated demagnification in the vertical plane is being investigated.

While it was possible to get a beam current of 1 μA around the 90° magnet, the beam transmission into the microprobe improved when this was reduced to approximately 0.5 μA. With this beam current value and the microprobe slits fully open the maximum beam transmitted into the microprobe chamber was approximately 50 nA. Work is currently in progress to further improve the matching between the accelerator and the microprobe focusing conditions.

Fig. 2 shows the measured brightness in the microprobe chamber as a function of different object and aperture sizes, for 2.3 MeV protons and 1.6 MeV He^+ ions. The measured brightness of ~1 pA/(μm^2 mrad^2 MeV) is similar to other values quoted for accelerators used for microprobes (for example, see [1, p. 56]). Contrary to what happens in other microprobe set-ups there is no marked increase of brightness when the collimator aperture is reduced, as shown in Fig. 2(a). However, as the size of the object aperture is reduced, then brightness does increase noticeably, as shown in Fig. 2(b). This is probably caused by the accelerator optics, which is not optimised for the microprobe, resulting in the beam focus after the 90° magnet being displaced from the object aperture. This causes the object aperture to influence the divergence of the beam entering the microprobe. This is being further studied with the aim of eliminating this effect and improving the beam brightness.

The focused beam is raster scanned over the sample surface using magnetic dipoles that are located before the lenses. The maximum area that can be scanned using 2 MeV protons is 2.6×2.6 mm^2.

For PIXE analysis, a Link Si(Li) detector with an active area of 80 mm^2, located at a backward angle of 45° is mounted in the microprobe chamber. Distance from detector to sample can be altered and a minimum value of 25 mm can be reached. For backscattering analysis there is a surface barrier detector with an active area of 200

[1] Obtained from D.N. Jamieson, University of Melbourne, Australia.

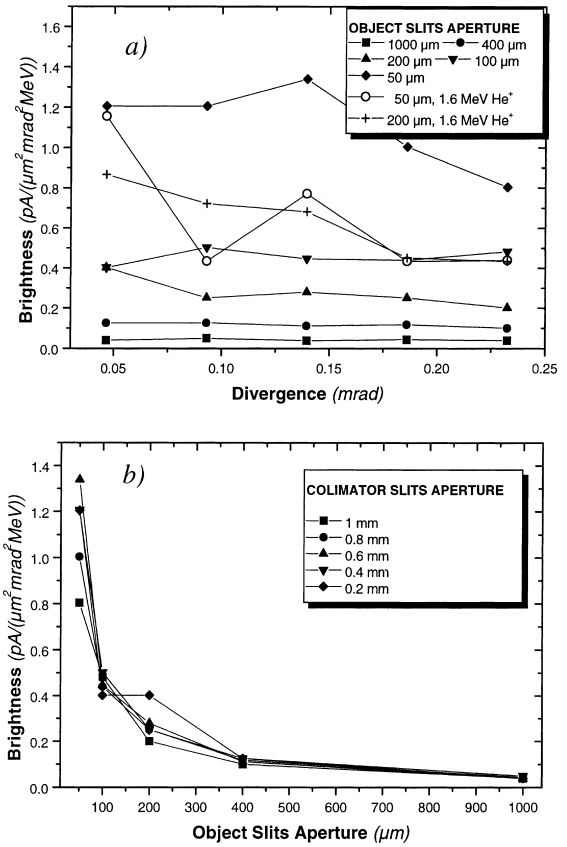

Fig. 2. Brightness curves for 2.3 MeV protons and 1.6 MeV He^+ ions, as measured in the microprobe sample chamber, as a function of (a) beam divergence into the lenses and (b) as a function of object aperture size.

mm^2, located at a backward angle of 40° in the Cornell geometry and at a distance of 30 mm from the sample. For transmission analysis, there is a rotating flange to allow a surface barrier detector to be wound on to the beam axis. There is also provision to mount a cheap silicon photodiode at this same location to prevent beam damage to expensive surface barrier detectors. There are also plans to mount a one-axis goniometer on the *xyz* stage, for channeling analysis in backscattering and transmission modes, similar to that described in [1].

To focus the beam there is a front viewing stereo zoom microscope at a backward angle of 45°. The chamber is vacuum pumped using a diffusion pump, and a pressure of 10^{-6} mbar is attainable. The whole beamline area was checked with a search coil for stray fields, which would degrade the resolution [4]. Several nearby components were found which gave large high frequency fields, particularly vacuum gauges measuring devices, a turbo control unit and a computer monitor. These were either changed or moved further away to eliminate or reduce their effects.

The data acquisition system is a PC-based Oxford Microbeams 1000e unit [5], capable of collecting approximately 5000 events per second from up to eight detectors without a prohibitive dead time.

The beam was first focused optically on a glass and then the transmission detector was used to produce STIM images of a 2000 mesh grid. Small variations in the quadrupole lens currents were made to optimise the beam resolution, with the object and collimator slits made very small. Fig. 3 shows an image of a 2000 mesh copper grid, with a period of 12.7 μm and linescan across it, showing a spatial resolution of ~1 μm. This was at a very low current of ~1 fA needed for STIM analysis. At a beam current of 100 pA, the spatial resolution was approximately 2 μm.

2. Microprobe analysis of SiC/SiC_f materials

One of the first applications of the new microprobe was the study of the behaviour of SiC/SiC_f composite materials when exposed to Li orthosilicate or Li titanate and kept at 800°C for 216 and 1000 h. Preliminary results are shown here and a more detailed description will be presented elsewhere [6].

Apart from the severe structural alterations of the surface that could be observed in all the exposed samples, a 500 × 500 μm^2 scan of the samples in contact with Li titanate during 1000 h (Fig. 4) reveals the formation of Ti and Cr precipitates. Also high amounts of K were found to be spread all over the sample surface. This high content of K was not observed before in any of the other samples and presumably corresponds to a large contamination from an unknown source.

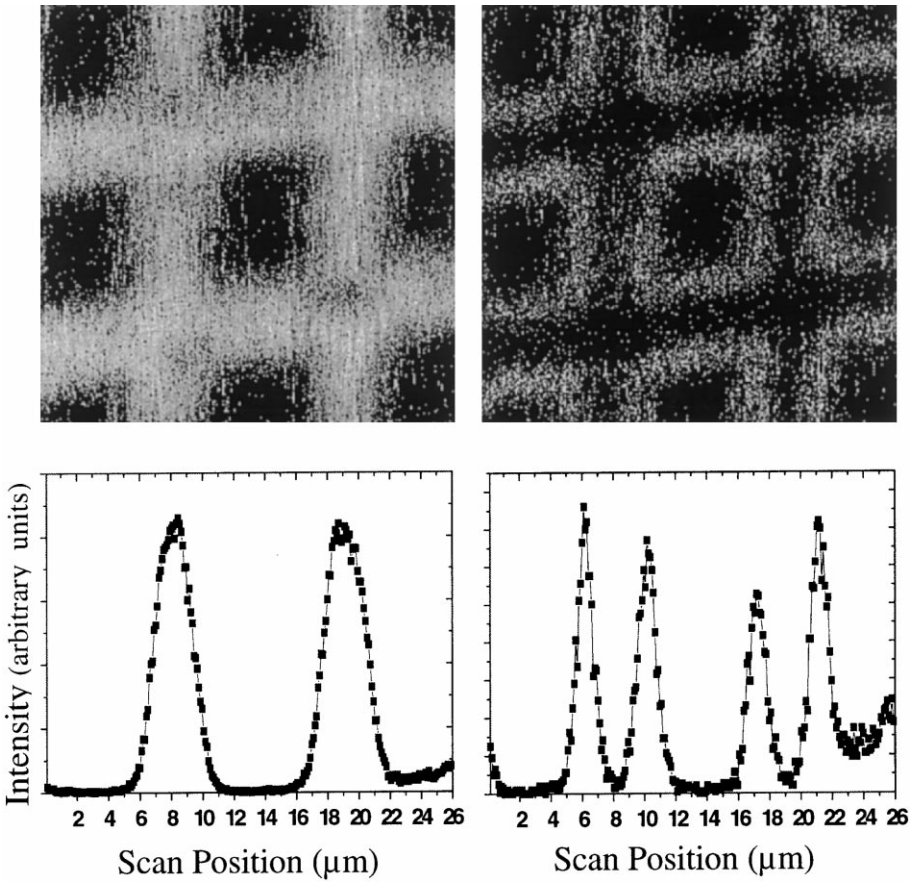

Fig. 3. STIM images of a 2000 mesh copper grid and linescan across it, showing a spatial resolution of ~1 μm.

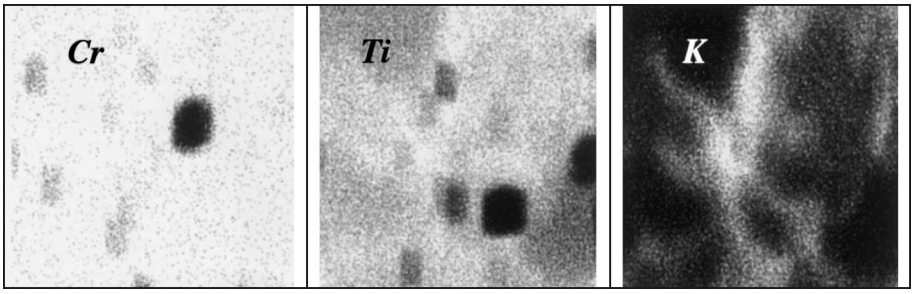

Fig. 4. 500 × 500 μm^2 PIXE maps of the sample exposed during 1000 h to Li titanate. Low to high yield of events is represented by variation from white to black.

3. Conclusions

Although the short distance (4.3 m) existing between the object and the collimator slits of the microprobe directly reflects the relatively low lens demagnification, a good spatial resolution was possible (~1 μm with currents of ~1 fA used for transmission work and ~2 μm for beam currents

~100 pA). The resolution was not constrained by the beam brightness which is comparable to other microprobe set-ups. Nevertheless, the accelerator beam optics are not optimised and are probably responsible for the large brightness variation as a function of object aperture size.

Strong surface alterations are induced in the SiC/SiC_f composites when exposed to lithium titanate or lithium orthosilicate in fusion relevant conditions. Ti and Cr precipitates were found in the composites exposed to lithium titanate as well as a large contamination of K.

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