

ELASTIC RECOIL DETECTION ANALYSIS ON THE ANSTO HEAVY ION MICROPROBE

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Abstract

The ANSTO heavy ion microprobe is capable of focussing heavy ion with an ME/q^2 of up to 100 amu MeV. This makes the microprobe ideally suited for heavy ion Elastic Recoil Detection Analysis (ERDA). However, beam currents on a microprobe are usually very small, which requires a detection system with a large solid angle.

We apply microbeam heavy ion ERDA using a large solid angle ΔE -E telescope to layered structures. We demonstrate the capability to measure oxygen and carbon with a lateral resolution of 20 μ m, together with determination of the depth of the contamination in thin deposited layers.

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INTRODUCTION

The High Energy Heavy Ion Microprobe[1] at the Australian Nuclear Science and Technology Organisation (ANSTO), was designed to use microbeams for high energy and heavy ion IBA techniques. It can focus ions with a mass energy product of up to $ME/q^2 = 100$ MeV amu. We have demonstrated the use of Heavy Ion Induced X-ray Emission with 9MeV He and 36 MeV C, as well as the use of the high energy $^{16}\text{O}(\alpha,\alpha)^{16}\text{O}$ resonance at 7.62 MeV for the detection of oxygen [2] on the microprobe. All these techniques utilise the high energy, heavy ion capabilities of the ANSTO microprobe.

PIXE and RBS are the most commonly used elemental analysis techniques on ion microprobes. However, PIXE is generally limited to elements heavier than Mg while RBS requires to de-convolute the backscattering spectra originating from different target elements. This makes the detection of light elements such as oxygen or carbon difficult with both techniques.

Heavy Ion Elastic Recoil Detection Analysis, which is complementary to RBS, provides a way to

profile these light elements. This is the reason, why the use of heavy ion ERDA has dramatically increased over the past 10 years and has become a standard IBA technique in many laboratories with Tandem accelerators. Using very heavy projectiles ERDA, is almost a universal technique, suitable to measure the whole range of elements from H up to As and Ge or heavier[3,4].

EXPERIMENTAL

In ERDA the detected particle is the scattered target atom. This allows the separation of the energy spectra from different target elements. However, this requires the measurement of two parameters to distinguish different recoil species at the same energy. There are two ways to achieve this separation, one is through the velocity of the recoils, the other one is through their initial energy loss. The first employs a Time of Flight setup, while the latter uses a ΔE -E gas detector. Time of Flight detection systems generally have a small detection angle, which makes them unsuitable for the use with microbeams. Microbeams require detection systems with a large solid angle to compensate for small

target currents, which are the result of the small spot sizes. To use heavy ion ERDA on the ANSTO microprobe a ΔE -E detector has been designed, that provides a large solid angle.

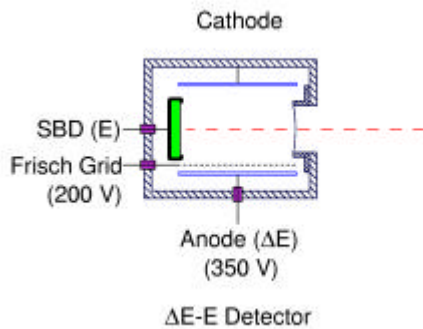


Fig. 1: Schematic of the ΔE -E detector used for the experiments.

The ΔE -E detector has a surface barrier E detector and a ΔE gas detector. This provides a very compact design, which is a requirement to place it as close as possible to the target. Fig. 1 shows a schematic of the ΔE -E detector. The ΔE gas detector part is 40 mm long and was operated at 20 Torr with P10. Under these conditions the ΔE signal is large enough to allow the separation of the light elements. The window of the detector consists of a 185 $\mu\text{g}/\text{cm}^2$ mylar foil, which is supported by a wire mesh.

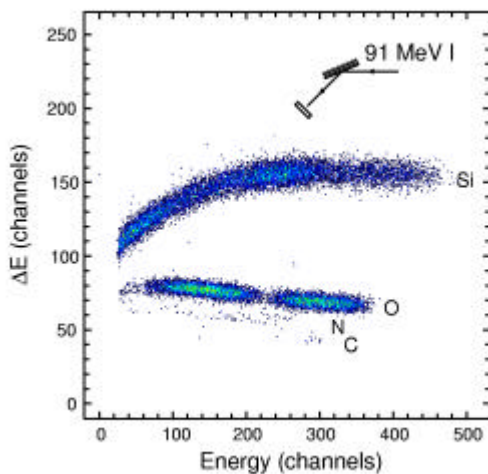


Fig. 2: ΔE -E coincidence spectrum taken of a SIMOX sample with 91 MeV I.

The detector has a solid angle of 0.8 msr and is 215 mm from the target. The scattering angle of the detected recoils is 45° . At this angle iodine, which was used as a projectile, can only scatter directly into the detector of target elements with a mass greater than 90 amu. The detector can be separated from the target chamber through a gate valve, in order to change samples without venting the detector.

However, this limits the minimum distance of the detector from the target.

RESULTS AND DISCUSSION

Various samples have been measured in order to verify the species and depth resolution of the detector. Fig 2 shows the ΔE -E coincidence spectrum from a silicon sample, with a surface and buried oxide layer. The buried oxide was created by high energy oxygen implantation and subsequent annealing. The thickness of surface and buried oxide layers are 330nm and 290nm, respectively. The oxides are separated by a 200nm Si layer. Si, O, N and C can clearly be identified in the spectrum. The coincidence spectrum shows the two oxide layers both in the oxygen and silicon signal. Traces of nitrogen and carbon are also visible in the spectrum. The ΔE -E spectrum shows that carbon is a surface contamination, while the N is buried inside the sample and was probably introduced during the annealing process.

The spectrum shows that the two oxide layers separated by 200nm of Si can be resolved. Due to the large solid angle, the depth resolution is in the order of 50nm. The detector accepting recoils from different scattering angles degrades the depth information. The depth resolution can be increased, with a position sensitive detector that can be used to correct for the change in scattering angle[4]. However, in this initial test it was our aim to demonstrate the use of microbeam ERDA.

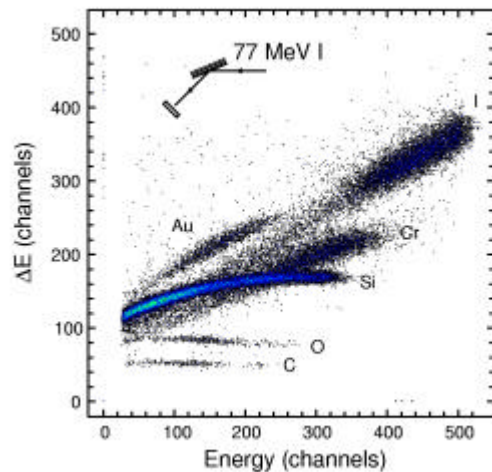


Fig. 3: ΔE -E coincidence spectrum taken of the test structure analysed with 77 MeV I.

A sample was prepared by depositing a 40nm layer of Cr on a $\langle 100 \rangle$ silicon wafer followed by a layer of 100nm Au. Subsequently a chessboard pattern, with $100 \times 100 \mu\text{m}$ squares was etched onto the sample for the use with Heavy Ion Microbeam ERDA. Fig 3. shows the ΔE -E coincidence

spectrum of the sample taken with a 77 MeV I beam. The spectrum shows the different signals from Si, Cr and Au, the elements present in the sample. The Cr layer, which is buried under the Au, almost merges into the Si signal. This is due to the relatively low energy of the primary beam. ERDA using a gas detector generally works best with a primary beam energies of 1-2 MeV/amu. This is especially true for detection angles greater than 30° with less efficient energy transfer to the recoil. Lower energies cause a very shallow analysis range, particularly for heavy elements. However, in this application the low energy was chosen, because of the higher recoil cross section.

Beside the major elements, the spectrum also shows traces of carbon and oxygen. Fig. 4 shows the energy profiles of the two elements. The surface energies for C and O are approximately at channels 220 and 270, respectively. The profiles show, that both impurities are predominantly underneath the surface, but also show a small surface peak. The maximum of the oxygen and carbon concentration corresponds to a depth of approximately 120nm, which is in the centre of the Cr layer. The width of the contamination estimated from the profiles, is much wider than the Cr layer. This is probably due to the acceptance angle of the detector, increasing the depth resolution.

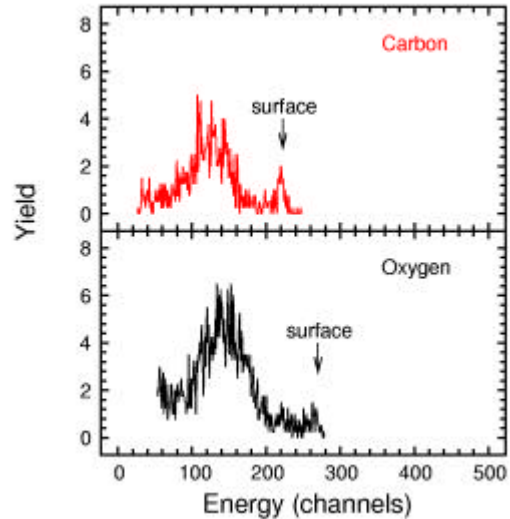


Fig. 4: Carbon and oxygen depth profiles extracted from the coincidence spectrum in Fig. 3.

Apart from the elements present in the sample the coincidence spectrum also shows a signal for the scattered iodine. At a scattering angle of 45° iodine can only scatter of elements heavier than yttrium directly into the detector. Therefore the iodine signal is due to the gold pattern on the sample.

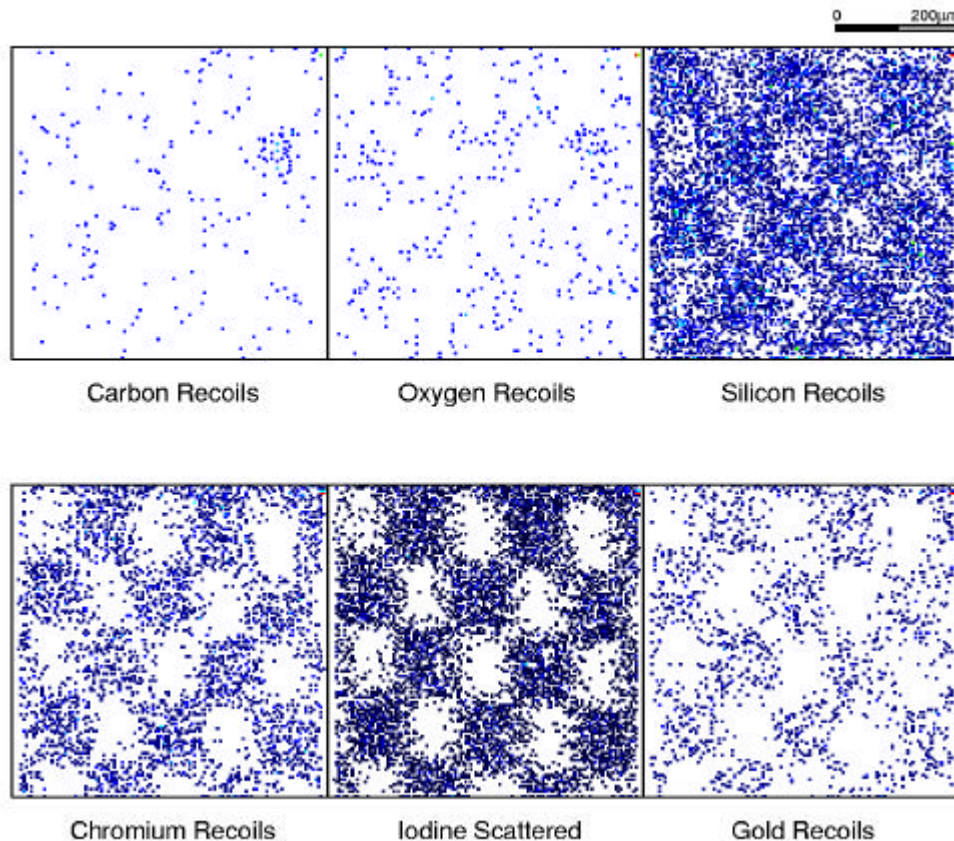


Fig. 5: Elemental maps for carbon, oxygen, silicon, chromium, iodine and gold.

Using the trace for each element in the ΔE -E spectrum, elemental maps have been constructed. The maps are shown in Fig. 5. The silicon map was constructed only from the surface part of the Si trace, while all the other maps were constructed using the full trace for the element. The 22.5° tilt of the sample relative to the beam, will distort the elemental maps from the sample. To compensate for this distortion the scan size in x was adjusted accordingly, which explains the square pattern shown in the maps. The total scan area of the maps is about $450 \times 450 \mu\text{m}$. The spot size in these measurements was approximately $20 \mu\text{m}$. At this spot size, the target current was a few tens of pA, resulting in a count rate of about 100 counts/sec. At the moment this limits the minimum spot sizes achievable, since smaller object aperture setting, will reduce the count rate even further and thus making the measurement times for the maps unreasonably long. However, this can be overcome, by making the solid angle of the detector even larger. Solid angles of 5-10msr are easily obtainable with ΔE -E detectors[5].

The Cr, I and Au maps clearly show the chessboard pattern of the sample. All three agree very well, with the I scattering map showing the best statistics, because I scattering has a higher cross sections at this angle compared to Cr and Au recoils. The Si map shows a pattern which is inverted to the other patterns, with higher Si concentrations in areas where the Si is not covered by the Au and Cr layers.

Both the light impurities, carbon and oxygen are concentrated in areas where the silicon is covered by metals. An area of increased contamination is visible in one of the squares at the top right corner. This area shows increased contamination in both carbon and oxygen. Further analysis shows that this increased contamination is due to oxygen and carbon deeper in the sample and does not correlate with the surface contamination of carbon and oxygen. The maximum of both the carbon and oxygen peak is at about $120 \mu\text{m}$, which means that the C and O are in the Cr layer. Both are probably introduced during the sputter deposition of the Cr.

This demonstrates that Heavy Ion Microbeam ERDA is a powerful tool to locate light element impurities in specimens. Microbeam ERDA allows to locate them not only laterally but also allows to determine where they are located relative to the surface.

CONCLUSIONS

We have demonstrated the use of Heavy Ion Microbeam ERDA. Microbeam ERDA can be used to map elements both laterally as well as vertically in the sample. A cluster of high carbon and oxygen contamination was measured in the sample, in one of

the metallised squares. Impurities could be attributed to the Cr layer. However, a limitation is the low count rate, due to small target currents. This requires larger solid angles of the detector, which can either be achieved by making the detectors larger or by locating closer to the target.

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