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Integration Of SIMS Into A General Purpose IBA Data Analysis Code

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Abstract. IBA techniques such as RBS, ERDA, NRA, or PIXE are highly complementary, and are often combined to maximize the extracted information. In particular, they have different sensitivities to various elements and probe different depth scales. The same is true for secondary ion mass spectrometry (SIMS), that can have much better detection limits for many species. Quantification of SIMS data normally requires careful calibration of the exact system being studied, and often the results are only semi-quantitative. Nevertheless, when SIMS is used together with other IBA techniques, it would be highly desirable to integrate the data analysis. We developed a routine to analyse SIMS data, and implemented it in NDF, a standard IBA data analysis code, that already supported RBS, ERDA, resonant and non-resonant NRA, and PIXE. Details of this new routine are presented in this work.

Keywords: SIMS; Ion Beam Analysis; Rutherford backscattering; NDF

PACS: 34.50.Bw; 82.80.Yc; 81.05.Ea

INTRODUCTION

Ion beam analysis (IBA) techniques such as Rutherford backscattering spectrometry (RBS), elastic recoil detection analysis (ERDA), nuclear reaction analysis (NRA), or particle-induced X-ray emission (PIXE) are highly complementary, because they have different sensitivities for various elements and probe different depth scales. On the other hand ion mass spectrometry (SIMS), can, for many species, probe a much wider range of concentrations, with much better sensitivity and lower detection limits. SIMS is, however, very different from the other IBA techniques, both experimentally and in the methods used to analyse the data. Nevertheless, in cases where SIMS is used in conjunction with other IBA techniques, it would be highly desirable to integrate the data analysis, ensuring self-consistency between all results. A routine to analyse SIMS data was developed and implemented in NDF [1,2], a standard code for IBA data analysis. Details of this new routine are presented in this work.

The new code was tested by studying deuterium retention in carbon samples subject to plasma irradiation in the JET tokamak [3,4]. SIMS and IBA

techniques were extensively used for the analysis of those first wall components removed from JET. Lateral microbeam PIXE was performed on some samples, that is, cross sections of the samples were measured, obtaining in this way depth profiles with PIXE that probe as deep as SIMS did. RBS, ERDA, and NRA data were also obtained from the same samples. All the data are fitted simultaneously, with the same depth profile.

EXPERIMENTAL DETAILS

A large number of samples from different regions of tiles removed from the JET after the 2005–2007 campaign [5] were analysed with multiple techniques. The carbon-based samples also contain hydrogen and deuterium, beryllium, boron, oxygen, tungsten, nickel contaminants and some other trace impurities such as iron and cobalt, that were not measured with SIMS. Also the carbon isotopic ratio can change near the surface which is due to the $^{13}\text{CH}_4$ puffing experiment done in 2007. Rutherford backscattering was made with a 2 MeV ^4He beam detected at a 140° scattering angle and with different angles of incidence, with a ^1H beam at energies up to 2.4 MeV, detected at a 140°

scattering angle and normal incidence. ERDA was made with a 2 MeV ^4He beam at a 24° recoil detection angle; in this case, RBS spectra were simultaneously collected at an 160° scattering angle. NRA was made with a ^3He beam with energies between 0.8 and 2.3 MeV, taking advantage of the $^2\text{H}(^3\text{He},\text{p})\alpha$ reaction with ^2H , with $Q=18352$ keV. A stopping foil was used to stop the α particles from hitting the detector, so only the p were detected. Lateral microbeam PIXE was done on some samples: a 2 MeV proton beam, focused down to a $3\times 4\ \mu\text{m}^2$ beam spot size, was used to measure a cross section of the samples. The SIMS analysis was made with a double focussing magnetic sector instrument using a 5 keV O_2^+ primary ion beam, at currents typically 500 nA.

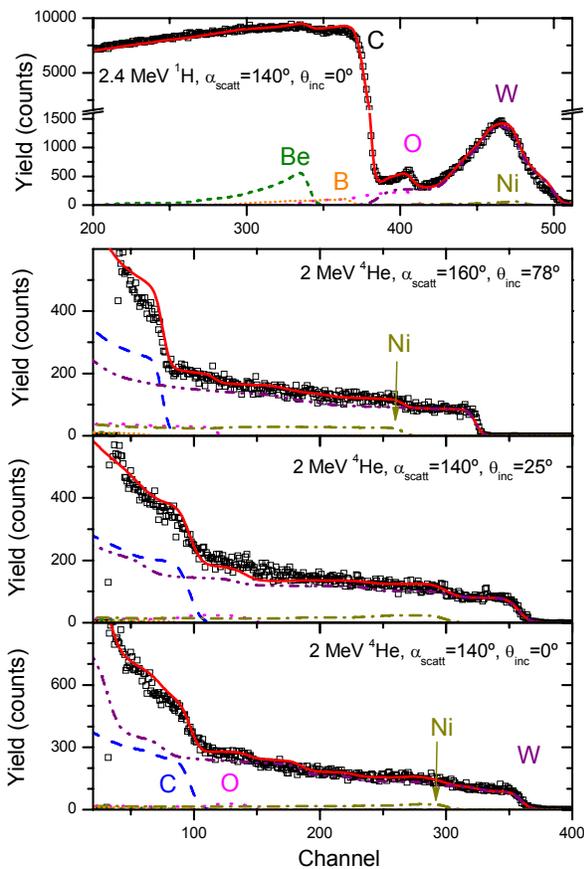


FIGURE 1. All RBS spectra collected for a given sample, including ^4He and ^1H beams.

CALCULATIONS

The NDF code already had implemented the techniques RBS, ERDA, both resonant and non-resonant NRA, and PIXE [6]. In all these techniques,

NDF takes as input a depth profile, calculates the corresponding theoretical spectrum, and compares the calculation to the experimental data. This can be done both interactively, with the user changing manually the depth profile until a satisfactory fit is achieved, or in an automated way with different fitting algorithms. The crucial point is that any number of spectra from any combination of the techniques implemented can be analysed simultaneously: that is, from the assumed depth profile one theoretical spectrum is calculated for each of the experiments made. Optimisation is made globally on all data together. This is the ideal way of extracting the complementary information that each technique provides. For instance, the depth profile of the major heavy elements is easily extracted with ^4He -RBS (particularly if different angles of incidence and/or different beam energies were used), ^1H -RBS can be used for enhanced sensitivity to light elements, ^4He -ERDA for hydrogen isotopes, PIXE for trace elements, and NRA for specific isotopes. This complementarity is not only related to the elements present, given that the techniques probe very different depth scales. ERDA at the usual beam energies probes a few hundreds of nm; ^1H -RBS can probe several μm , and standard PIXE even deeper.

In all these techniques, translating a depth profile into the calculated spectrum involves a long series of complex calculations, particularly if advanced physics, such as plural and multiple scattering, is included [6]. In SIMS, in principle this could also be the case. One could, from the depth profile, and including chemical information, use data bases of experimental sputter rates and secondary ion yields, calculate neutralisation for the set-up used, and finally calculate measured yields [7]. However, these processes are very complex and there is as yet no theory that leads to satisfactory quantitative results: the sputter rate for one given ion depends strongly on the exact overall sample composition, on the incident ion species, angle of incidence and energy, and the relationship between the detected ion yield and the total sputter yield depends on many parameters. In practice, careful calibration of the exact system being studied is necessary (and IBA techniques are often used for that), and SIMS is often a semi-quantitative technique.

In NDF, the depth profile is translated into a yield vs. depth in two steps. In the first step, the concentration of one given element or isotope at a given depth is translated into a yield using a response function that must be input by the user. This response function will normally incorporate the results of the calibration of the system. However, in many cases the same element observed with SIMS is also seen, albeit in a narrower surface layer, in a measurement of the same sample made with RBS, NRA, or ERDA. In this case, there is an intrinsic self-calibration, as the

calibration of the SIMS yield is made by a measurement of the exact same sample, and is obtained during the data analysis process. In fact, the response function in NDF can be fixed as defined by the user, but it can also be a fit parameter. NDF will find the optimal solution that constrains the elemental concentration to the value that it must have in the depth range accessed by the IBA technique, and will adjust the SIMS response function accordingly to obtain good agreement in that depth range. Outside that depth range, the response function thus derived is used as well.

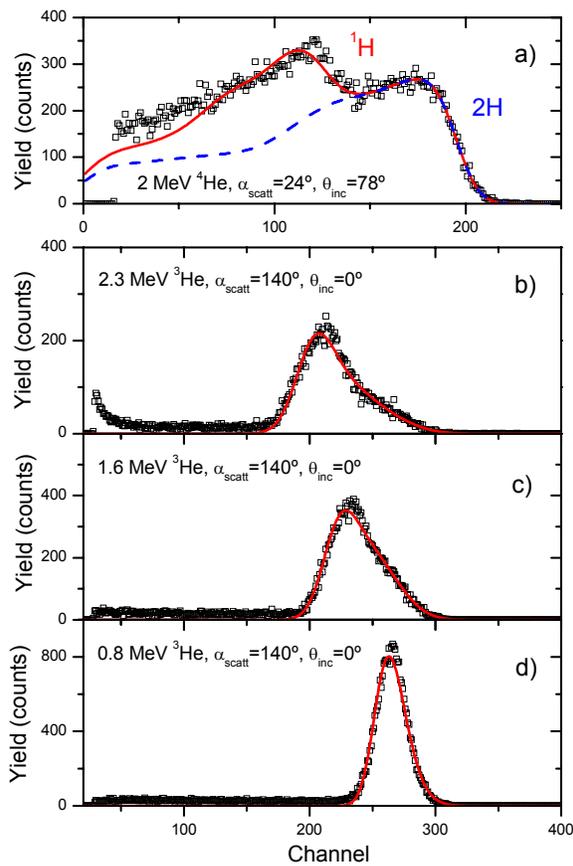


FIGURE 2. Data (squares) and fits (solid lines) for a) ^4He -ERDA spectrum, and b), c) and d) ^3He NRA spectra, that probe D only. Protons, which are a reaction product, are detected.

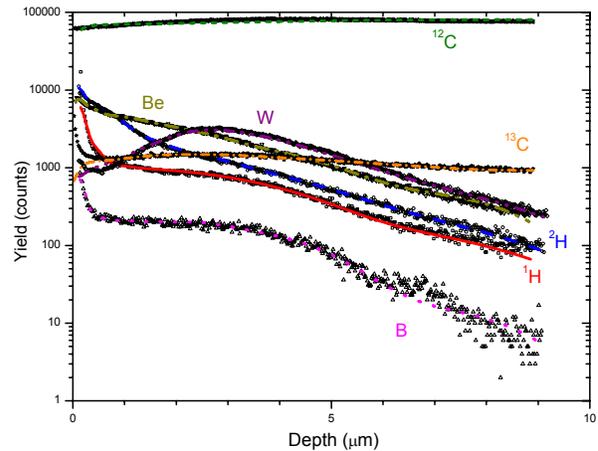


FIGURE 3. SIMS data (symbols) and fits (lines) for all elements but Ni.

In the second step, the depth, which is an areal density in NDF, is translated into a real depth scale. NDF uses areal density (with unit 10^{15} at./ cm^2) in all its internal calculations, given that this is the natural quantity for techniques such as RBS, ERDA, or even PIXE, that are not sensitive to density and only depend on the amount of matter crossed by the beam: SIMS, on the contrary, is a real depth profiling technique (at least if one accepts the approximation usually made that the sputter rate is constant and known). To calculate a thickness corresponding to a given areal density, the density of the layer in question must be known. NDF can use a weighted average of the bulk elemental densities in that layer, but this will often lead to a large error. If, however, the density is known, then it can be input by the user. Finally, if a given element has a well defined depth profile visible in SIMS and in a different IBA technique, then the density is determined by the data being analysed. In this case, NDF can treat the density as a free parameter, which is effectively determined from the self-consistent analysis made.

RESULTS AND DISCUSSION

We will illustrate the possibilities of the new approach using one given sample, from the JET outer poloidal limiter. The four RBS spectra collected from this sample are shown in Fig. 1. The ^4He -RBS show that there is W and Ni at the surface and their concentrations are derived. From the proton spectrum most of the W profile is clearly seen, although it is superimposed to the Ni signal. Also, a peak corresponding to surface O is observed. The Be signal is observed as a small peak on top of the C signal, but

whether its concentration changes with depth cannot be derived. Finally, the depth resolution is not enough to distinguish ^{12}C from ^{13}C (due to the $^{13}\text{CH}_4$ puffing), and the B signal is submerged in the much larger C signal.

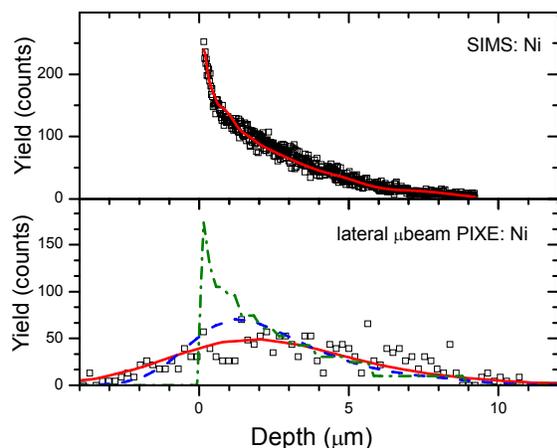


FIGURE 4. SIMS and lateral microbeam PIXE data for Ni. For the lateral microbeam PIXE, simulations for the final profile obtained, but with 10 nm (dash-dotted line) and the nominal 3 μm (dashed line) depth resolution are also shown.

The ^1H and ^2H signals are clearly seen in the ERDA spectrum shown in Fig. 2, and their surface concentrations can be determined, even if the superposition of the two signals leads to some uncertainty. The NRA data are sensitive only to ^2H , and the three different beam energies, when analysed simultaneously, impose strong constraints on the ^2H profile, taking advantage of the fact that the cross section [8] has a broad but well defined peak around 700 keV.

The SIMS data for all the elements except Ni are shown in Fig. 3. First of all, the ^1H and ^2H profiles are quantitatively constrained by the ERDA and NRA data, that is, response functions could be derived by the simultaneous fitting procedure. The same is true for ^{12}C , Ni, W, and Be. The B is not really seen in the RBS, and the quantification achieved with SIMS is a maximum limit only, given that a large B content would be observed in the proton RBS spectrum. For ^{13}C no internal calibration is obtained from the data, and we simply assumed the natural isotopic distribution for the deepest depth probed with SIMS, when the concentration of the other elements is close to zero.

The W SIMS signal can be directly compared to the W signal observed in the proton RBS spectrum. This effectively determines the density and thus the depth scale. The fit indicates that density is about 16%

smaller than bulk graphite, assuming that sputter rate in SIMS was fairly constant.

The data obtained for Ni both with SIMS and lateral microbeam PIXE are shown in Fig. 4. It is clear that, while SIMS shows a Ni content constantly decreasing with depth, only a very broad signal is seen with PIXE. This is due, first of all, to the different depth resolution of the two techniques. SIMS has a nominal depth resolution around 10 nm at the surface. The microbeam PIXE beam spot was 3 μm . Simulations made for PIXE both with 10 nm and 3 μm are also shown in the figure. It is clear that, for 10 nm depth resolution, the calculated signal approaches the SIMS data. For 3 μm , however, one would expect that a good simulation is reached; however, the calculation is still not as broad as the observed signal, and the best fit was achieved for a resolution of 5.5 μm , which is unrealistically high. This is due to surface roughness that is extensive in irradiated samples. Its effect will be totally different in a technique such as SIMS that probes the sample from the surface in the usual way, and in cross-sectional microbeam PIXE, where the actual surface is probed from the side. In the first case, the effect of roughness is initially small (or non-existent in the limit case of normal incidence) and increases gradually. In cross section analysis, the roughness is immediately fully included in the signal observed. This means that the difference (taken in quadrature) between the apparent and the real depth resolution in PIXE can be taken as due to roughness, which we thus estimate to be around 4.6 μm in this sample.

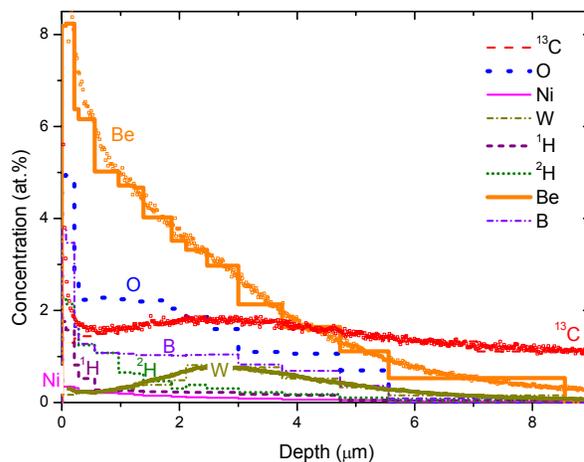


FIGURE 5. Final depth profile reached. The ^{12}C is not shown in this figure. The fitted depth profile is shown as different curves for each element. The point-by-point calculation (symbols) is also shown for selected elements.

The final depth profile determined is shown in Fig. 5. All the information derived from the 5 techniques used is included. We note that all the spectra from all the techniques, shown in Figs 1 to 4, were fitted with this same depth profile; one single fit was made, not separate fits to each spectrum. Note that NDF makes also an inverse calculation, deriving from each data point a concentration point [9]. This is also implemented for SIMS, using the response function input by the user (or derived during the fit). The results of this point-by-point calculation are shown for some of the elements only.

SUMMARY

We developed a simple routine to handle SIMS data, as well as any other real depth profiling techniques where a calibration of the depth and concentration scale can be obtained either from calculation or from comparison with other data, and included it in the code NDF, that already implemented RBS, ERDA, resonant and non-resonant NRA, and PIXE. We demonstrate the power of combining multiple techniques when analysing complex samples, by giving the example of a carbon based sample irradiated in the JET tokamak. The new routine is open source, and is available in the NDF web site http://www.itn.pt/facilities/lfi/ndf/uk_lfi_ndf.htm

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