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Quantitative depth profiling of light elements by means of the ERD $E \times B$ technique

F. Schiettekatte [a], A. Chevarier [b], N. Chevarier [b], A. Plantier [b], G.G. Ross [a,*]

[a] INRS-Énergie et Matériaux, 1650 montée Ste-Julie, Varennes, Québec J3X 1S2, Canada
[b] IPNL, Université Claude Bernard, F69622 Villeurbanne, Cedex, France

Abstract

ERDA [J. L'Écuyer et al., J. Appl. Phys. 47 (1976) 381] is a technique of great interest for quantitative depth profiling of light elements in matter. The use of crossed electric and magnetic fields ($E \times B$ filter) [G.G. Ross et al. J. Nucl. Mater. 128/129 (1992) 484; G.G. Ross and L. Leblanc, Nucl. Instr. and Meth. B 62 (1992) 484] in place of the traditional absorber, enhances the resolution by eliminating the straggling induced normally by the absorber and removes the uncertainty on the absorber thickness. The $E \times B$ filter allows the simultaneous detection of different particles such as H, D and He. This work presents the first ERD $E \times B$ depth profiling by means of a heavy ion beam. Compared with the usual ERD $E \times B$ with 350 keV He, the 2.54 MeV ^{15}N beam enhances scattering cross section by a factor of 3, has equivalent depth resolution (1–3 nm at surface) and gives a depth probe twice deeper. However, ^{15}N ions sometimes induce high desorption compared to He. H, D and He were implanted in Be and Si at energies from 800 eV to 10 keV. The experimental depth distributions are compared with those obtained by TRIM95 [J.F. Ziegler and J.P. Biersack, The Stopping and Range of Ions in Solids (Pergamon, New York, 1995)] and by other experimental techniques. Reproducibility is very good between the different results obtained experimentally. Profile modification induced by the ion beam is also shown.

1. Introduction

The Elastic Recoil Detection Analysis (ERDA) is a method of growing interest for quantitative profiling of light atoms. Since its development by L'Écuyer et al. [1] in 1976, the technique has greatly improved. One of those improvements is the replacement of the absorber by an $E \times B$ filter [2,3]. The use of an $E \times B$ filter instead of an absorber allows much more flexibility in recoils selection without any disturbance (additional straggling, energy shift, etc.).

Up to now, ERD $E \times B$ has been used mainly with alpha particle beam although the use of heavier particles has many advantages. For example, it raises the scattering cross section because the Rutherford cross section increases as $\sim (Z_{\text{ion}} M_{\text{ion}})^2$ and its greater stopping power tends to increase the resolution.

This paper presents the first H and He profiles obtained by ERDA with the $E \times B$ filter using an heavier ion beam (^{15}N). The beam energy (2.54 MeV) is close to the stopping power maximum and allows to get a depth resolution as good as possible. Section 2 describes briefly the experimental setup and shows how ERD $E \times B$ can be used to detect several masses simultaneously. In Section 3, the ^{15}N beam is assessed in terms of depth resolution, efficiency factor and profile modification due to ion beam bombardment. Finally, some results of He, D and H profiling are presented in Section 4 and are compared with the values obtained with other experiments and simulations.

2. Experimental setup

2.1. $E \times B$ filter with a ^{15}N beam

The replacement of the absorber by an $E \times B$ filter in front of a silicon detector in ERD [1] experiments has been first proposed by Ross et al. [2,3] and used with a 350 keV He beam. Subsequently, it has been successfully applied by Roux et al. [5] with a 2.5 MeV alpha beam. The $E \times B$ filter consists of crossed electric and magnetic fields. With appropriate settings, a wide energy domain can be found where the particles of the same q/m ratio undergo nearly the same deflection.

Compared with the absorber, the $E \times B$ filter has many advantages. It avoids straggling caused by the absorber and the uncertainty on its thickness regularity. Depth resolution losses are mainly limited by the recoil straggling in the absorber. Thus, the replacement of the absorber by an

* Corresponding author. Tel. +514 929 8108, fax +514 929 8102, e-mail ross@inrs-ener.uquebec.ca.

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$E \times B$ filter considerably enhances the depth resolution. The $E \times B$ filter also avoids an energy shift which, moreover, varies according to the particle types that pass through an absorber. However, a disadvantage of the $E \times B$ filter is that the solid angle is usually reduced to avoid particle deflection on the electrodes in the $E \times B$ filter. A fraction of the recoils is also lost when a charge state is selected. That fraction can be measured precisely by means of the $E \times B$ filter (see Ref. [11]).

The results presented in this paper were obtained by means of a 2.54 MeV ^{15}N beam incident at an angle of 15° with respect to the surface. The detection angle was 30° with respect to the beam with a solid angle of 6.6×10^{-5} sr. The $E \times B$ filter has a length of 0.115 m and a magnetic field of 0.29 T. The electric field was set to 1.5 MV/m. Fig. 1 shows the deflection at the filter exit of H, D, ^4He and ^{15}N as a function of particle energy.

2.2. Simultaneous detection of several masses

It is well known that ERDA allows the simultaneous detection of several elements and isotopes [6]. This is also possible with the ERD $E \times B$ using heavy ions because the selectivity power of the $E \times B$ filter is far better. With an absorber, one can only block the heavier recoils by increasing the thickness or changing the absorber which limits the Z selectivity and the analysis depth. Moreover, the absorber applies a stronger energy shift to the heavier atoms (recoiled at higher energy), squeezing them together with the lighter atoms on the energy spectrum.

With the $E \times B$ filter, the position of the detector and its collimator, as well as the strength of the electric and magnetic fields can be adjusted in such a way that different elements and isotopes can easily be detected simultaneously. In Fig. 1, it is seen that H, D and He can be detected simultaneously as well as separately by means of a mobile detector combined with a variable detector collimator aperture (see Ref. [3]). Particles of the same q/m ratio undergo the same deflection for the same speed and can therefore be separated in energy, as discussed in Ref. [3]. Fig. 2 shows an example of simultaneous analysis of the adsorbed surface H peak on a Be sample implanted with helium. Fig. 2a shows the energy spectrum while the depth profiles are plotted in Fig. 2b.

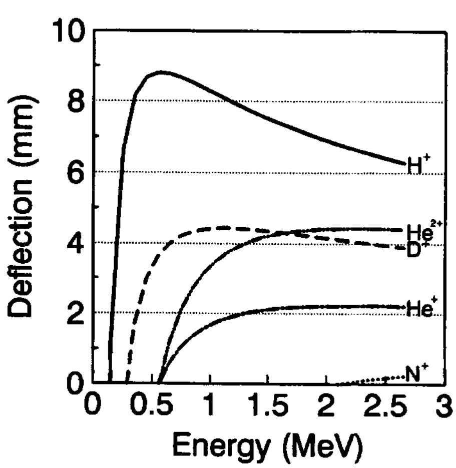

Fig. 1. Deflection of particles vs. energy at the exit of the $E \times B$ filter. $|B| = 0.29$ T and $|E| = 2.8$ MV/m.

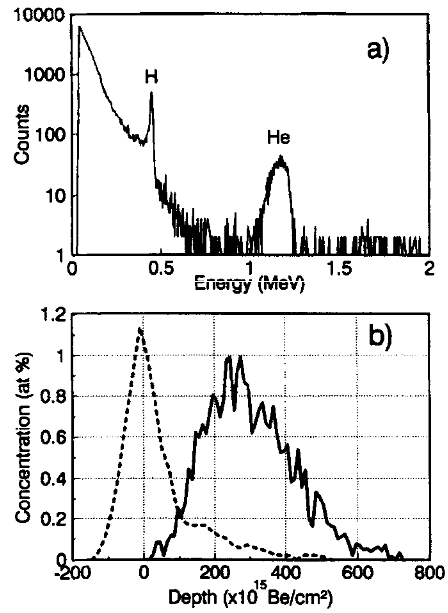

Fig. 2. Example of simultaneous particle detection: 2.33×10^{15} He/cm^2 implanted at 1.5 keV in beryllium with surface hydrogen. (a) Energy spectrum. (b) Corresponding hydrogen (- - - - - -) and helium (———) depth profiles. Beam fluence is 1.5×10^{16} N/cm^2.

3. Assessment of the ERD $E \times B$ profiling by means of ^{15}N beam

This section discusses the opportunity of using the ERD E × B technique with a ^{15}N ion beam. Table 1 shows a comparison between ^{15}N and He beams (in combination with ERD $E \times B$) for the depth profiling of hydrogen, deuterium and helium. The comparison is made in terms of the depth resolution, an efficiency factor, the background noise level and the effects of ion beam induced desorption.

The depth resolution is evaluated for H, D and He in beryllium at surface and at 100 nm. It has been calculated by means of the program DEPTH of Szilágyi [7]. Incident and detection angles were those of the experimental setup described above (15° and 30°, respectively), with a solid angle of 6.6×10^{-5} sr. In depth, the depth resolution is essentially degraded by multiple scattering of the recoiled particle, so it is not much affected by the beam type. N beam slightly improves surface depth resolution and worsens also slightly the 100 nm depth resolution.

Because of its energy, the 2.54 MeV ^{15}N beam has a much more deeper probe. For an equivalent depth probe with a He beam, one should use a 1 MeV beam. Such He energy worsens depth resolution by a factor of 3 compared with 350 keV He beam. A 2.54 MeV ^{15}N beam raises depth probe without affecting significantly the depth resolution.

The ERD $E \times B$ efficiency factor is not only affected by the Rutherford cross section but also by the recoil charge fraction. Thus, the efficiency factor is calculated as follows:

$$\mathrm{d}\sigma/\mathrm{d}\Omega \times \cos^3\theta \times \text{charge fraction},$$

where the $\cos^3\theta$ term eliminates the geometric dependence of the cross section. Table 1 shows that in all cases, ^{15}N enhances efficiency by a factor of approximately 3.

The background noise (when using ERD $E \times B$) is mainly due to the wide angle scattering of the particles on the electrodes of the electric field. For this reason, a precise collimation is required at the filter entrance in order to minimize the angular dispersion of the scattered particles which are the main contributors to the noise level. Otherwise, the background noise depends only on the substrate (increasing with Z), so it can generally be measured precisely and subtracted from the signal. As an example, in spite of a 0.2 at.% noise for the depth profiling of hydrogen in Be, a sensitivity of 0.02 at.% is achieved with the experimental setup presented before and a fluence of 1×10^{16} N/cm^2.

The major problem experienced with N beam is the strong beam induced hydrogen desorption in some materials. N ion has a much higher stopping power (130 eV/[10^{15}at/cm^2] maximum in Be) than the He ion (30 eV/[10^{15}at/cm^2] maximum in Be). Thus, for materials where moderate H desorption is observed when a 350 keV He beam is used (as for H in Be, H in C, etc.), a strong desorption process occurs with a 2.54 MeV N beam. However, H profiling in Si, as well as D and He detection in Be and Si are much less affected. Some examples of beam induced profile modification are introduced in the next section but the subject will be discussed extensively in a subsequent article.

In brief, a 2.54 MeV N beam has a better efficiency factor, equivalent depth resolution, much deeper probe and permits easy simultaneous detection of particles with a reduced (and precise) background noise. But a 350 keV He beam is still the better way to measure hydrogen profiles in materials strongly affected by beam induced desorption.

4. He, H and D profiling in materials

Beryllium and silicon have been implanted with helium, hydrogen and deuterium at various energies and concentrations. The following section presents some profiles obtained with a 2.54 MeV ^{15}N beam in comparison with the profiles measured by other ERD setup and those obtained by TRIM95 [4] simulation. For ^{15}N beam measurements, a spectrum to depth profile conversion is made by means of ALEGRIA [8].

4.1. He profiles in Be

Helium has been implanted in beryllium at 1.5, 5 and 10 keV. The depth profiles obtained, still resolution-broadened, appear in Fig. 3. The 10 keV He profile has been plotted together with a TRIM95 simulation and a profile obtained by ERDA (absorber) with a ^{35}Cl beam [9]. Experimental measurements are very similar, while the TRIM95 simulation is very asymmetric and overestimates the mean range by 25%. The integral of the depth profiles corresponds to the implanted fluence which confirms that the technique is quantitative without the use of any standard.

4.2. Comparison of He and D profiles in beryllium

Helium and deuterium have been implanted at low concentration in beryllium at 1.5 keV and 1.6 keV, respec-

Table 1
Comparison between ^{15}N and He profiling of hydrogen, deuterium and helium in beryllium. Incident angle: 15°, detection angle: 30°, solid angle: 6.6×10^{-5} sr

Recoil:	Hydrogen		Deuterium		^{4}He	
Beam:	^{15}N 2540 keV	^{4}He 350 keV	^{15}N 2540 keV	^{3}He 350 keV	^{15}N 2540 keV	^{3}He 350 keV
Resolution [nm] (surface/100 nm)	2.9/6.6	3.3/5.2	1.8/5.8	2.5/4.7	1.0/4.8	1.5/5.4
Depth probe [nm]	200	100	300	100	150	100
Efficiency factor [b]	10.1	3.51	2.84	0.67	3.02	1.1
Background noise level (experimental) [at.%]	0.2	0.2	0.1	1	0.2	3
Beam induced desorption	high/low	moderate /low	low	low	low	none

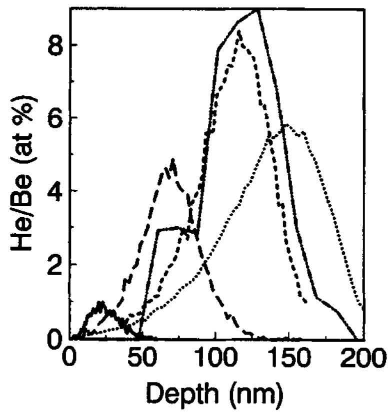

Fig. 3. Depth profiles of helium implanted in beryllium at 1.5 keV (———), 5 keV (— — —) and 10 keV (- - - - - -). 10 keV profile is compared with a TRIM95 [4] simulation (· · · · · ·) and a profile obtained by ERDA with a ^{35}Cl beam (– · · – · · –).

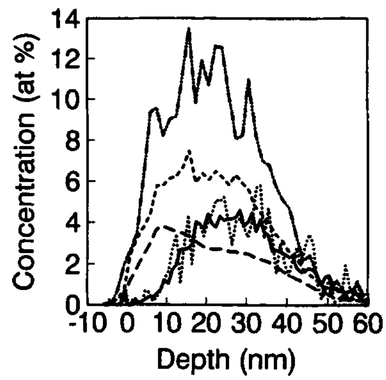

Fig. 5. Depth profile evolution of H implanted in Be and Si at 800 eV under a 2.54 MeV ^{15}N beam bombardment. H in Si profile obtained with fluences of 1×10^{15} N/cm^2 (———) and 2×10^{16} N/cm^2 (· · · · · ·). H in Be profile obtained with fluences of 1×10^{14} N/cm^2 (– · · – · · –), 1×10^{15} N/cm^2 (- - - - - -) and 2×10^{16} N/cm^2 (— — —).

tively. The experimental profiles (still resolution-broadened) and TRIM95 simulation profiles are shown in Fig. 4. It is seen that the helium profiles of experiment and simulation are similar.

The D profile obtained by means of a ^{15}N beam is compared with a TRIM95 simulation and to a profile obtained by means of ERD E × B with a 350 keV He beam [10]. Experimental profiles are quite similar and almost symmetric while simulation gives an asymmetric profile, with a mean range 30% shorter.

4.3. Hydrogen in beryllium and silicon

As mentioned in Section 3, a 2.54 MeV ^{15}N beam induces strong H desorption in some materials such as beryllium. An example is given in Fig. 5. Hydrogen has been implanted in Be and Si. The H depth profiles have been obtained by means of a N beam of different fluences. No significant profile modification induced by the beam is observed for hydrogen in silicon. But strong beam induced desorption is observed during the depth profiling of H implanted in Be. This ion induced desorption is enhanced by the larger energy loss of the incoming N ions, compared with the He beam which induces moderate desorption in this case.

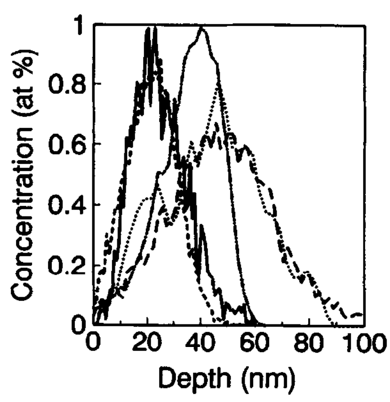

Fig. 4. Depth profile of He (———) and D (— — —) implanted in Be at 1.5 keV and 1.6 keV, respectively. He profile is compared with TRIM95 [4] simulation (- - - - - -). D profile is compared with TRIM95 simulation (– · · – · · –) and to a D profile obtained by ERD E×B with a 350 keV He beam (· · · · · ·).

5. Conclusion

Depth profiling of light elements by means of ERD $E \times B$ is already known to have several advantages compared with classical ERDA with absorber. It avoids straggling and energy shift due to the absorber, and improves the isotope selectivity capabilities.

The use of ^{15}N ion beam enhances the efficiency factor and depth probe without affecting depth resolution. It also improves the filter selectivity and separation, allowing easy simultaneous mass detection. Hydrogen and helium profiling can be achieved with a sensitivity of 0.02%.

Comparison of depth profiles measured by means of ERD $E \times B$ with a ^{15}N beam with previous measurements obtained with other experimental setups (such as ERD $E \times B$ with ^{4}He beam and ERDA with ^{35}Cl beam) shows excellent agreement. The integral of the depth profile corresponds to the implanted fluence which confirms that the technique is quantitative without the use of any standard. However, because of its higher energy loss (compared with ^{4}He beam), the N ion induced desorption is enhanced especially when hydrogen depth profiling is performed.

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