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Terwagne, Guy; Deconninck, Gaston Moss

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Concentration profile of light elements near an interface

G. Terwagne and G. Deconninck

LARN, Facultés Universitaires Notre-Dame de la Paix, 22 Rue Muzet, B-5000 Namur, Belgium

The knowledge of depth concentration of atoms introduced by diffusion or implantation in thin layers is essential, but it is difficult to measure. Nuclear resonant reaction analysis is a powerful technique to determine the depth profile of light elements in a matrix. These reactions occur often in the low energy range by accelerating protons or alpha particles with a Van de Graaff accelerator producing resonant reactions in the diffused atom. The depth resolution at surface depends on the resonance width used and the beam energy resolution. The exact localization of the interface remains a problem. The reason is that in the NRA technique, the depth is calibrated using the energy loss of the particle as measured by the magnetic analyser. This scale, however, should be converted into atoms/cm². Moreover, in the case of depth profiling measurement, the density is not uniform. The different origins of errors are: accuracy of the stopping power parameter, validity of Bragg's rule, exact composition of the layer and inhomogeneity. We describe a method that we have used since several years, which reduces considerably these uncertainties. It consists of measuring the energy loss in the thin layer by RBS near resonance energy. The depth profile at interface is then calibrated from the direct observation (RBS) without using stopping power parameters. The method is also insensitive to concentration variations in the surface layer. We used this method for Na, F, Al, in the study of glass coatings. It can be used for all the elements that can be analysed by nuclear resonant reactions.

1. Introduction

Depth profiling of light elements is generally performed by means of nuclear resonant reaction analysis (NRA) induced by protons or sometimes by alpha particles. This technique is quantitative and if there is no beam induced migration, the energy excitation curve is the exact image of the concentration profile.

The particle energy scale (eV) has to be converted into depth scale (atoms/cm²) by using stopping power tables [1]. Unfortunately, the accuracy of the stopping power parameter depends on three factors:

- a) the precision of the elemental atomic stopping power parameter (5–10%),
- b) validity of Bragg's rule for compounds,
- c) the knowledge of the elemental composition and the nature of the chemical bonding [2].

In this paper we propose a technique which rules out these uncertainties, thus improving the accuracy of the nuclear resonant method.

When a charged particles beam penetrates through matter, there is an increasing energy spreading which becomes rapidly a near-Gaussian distribution. The total energy spreading, Γ , can be obtained from the following expression:

$$\Gamma^2 = \Gamma_b^2 + \Gamma_R^2 + \Gamma_{st}^2 \quad (1)$$

Γ_b and Γ_R are, respectively, the beam and the resonance (Breit–Wigner) contributions, which are relatively small at large depth. Γ_{st} is the straggling contri-

bution, which increases with depth and can be calculated from an analytical expression [3] or by Monte Carlo calculations. This curve is used for the deconvolution profile [4]. The exact localization of an interface can be calculated from the particle energy loss which is the average of the Gaussian distribution of width Γ . The energy depth conversion makes use of the stopping power. Using the Rutherford backscattering technique, it is possible in certain cases to measure directly the exact energy loss of the beam at the interface. This value is then used to calibrate on the nuclear reaction excitation curve. By using this procedure, the concentration profiles near interface are obtained without using stopping power so ruling out important errors.

2. Experimental setup

The depth profiles were obtained with an automatic energy scan installed on a 2.5 MV Van de Graaff accelerator. To obtain an excitation curve, the energy of the beam was changed automatically by small energy steps over the whole energy range. Fig. 1 shows the experimental setup of the detection system. The incident proton beam is impinging on the sample which is placed in the NaI detector. The backscattered protons are detected at 177° from the incident beam direction, with a passivated implanted planar silicon detector (PIPS). The detector is collimated with a gold collimator of 4 mm diameter and allows the detection of the

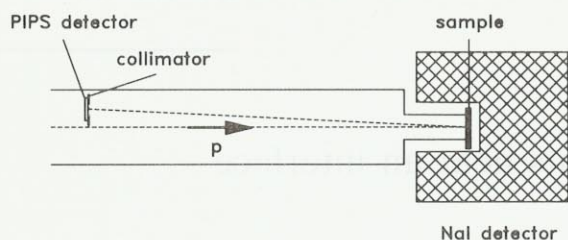


Fig. 1. Schematic view of the experimental setup showing the NaI well detector which is placed in the beam axis and the PIPS detector which is placed at 177° from the incident beam direction. The sample is localized in the hole of the NaI detector.

backscattered protons in the same conditions as for the profiling. Typical current used during measurement was of about 100 nA. The beam was also rastered on the sample in order to avoid overheating during irradiation.

3. Typical application

Typical applications of the method to the study of fluorine and sodium migration in SnO_2 layers on glasses are given. The depth profiles are measured by proton induced resonant reactions. The depth calibration which is essential to localize the interface is done by proton backscattering on Sn.

3.1. Proton energy loss in the layer

The SnO_2 layer (about 2000 Å) contains the element Sn, which is heavier than all the other elements in the glass. For this element the elastic cross section is pure Rutherford at the energy used (maximum 1 MeV) and the kinematical factor is 0.97.

The FWHM of the Sn signal is then twice the energy lost (ΔE_p) by the protons in the SnO_2 layer. ΔE_p then gives the exact position of the interface in terms of energy without using stopping power parameters. In what follows we shall use this value of ΔE_p to calibrate the resonant reactions excitation curves. We shall begin with sodium ($p, \alpha\gamma$) resonance in the $^{23}\text{Na}(p, \alpha\gamma)^{20}\text{Ne}$ reaction at $E_p = 1010.5$ keV, then we proceed with fluorine ($p, \alpha\gamma$) resonance in the $^{19}\text{F}(p, \alpha\gamma)^{16}\text{O}$ reaction at $E_p = 340$ keV.

3.2. Sodium profiling

Profiling of sodium is made by using the $^{23}\text{Na}(p, \alpha\gamma)^{20}\text{Ne}$ at resonance energy $E_R = 1010.5$ keV ($\Gamma_R < 0.5$ keV). 1634 keV γ -rays are detected in a $4 \text{ in.} \times 4 \text{ in.}$ well NaI detector for maximum efficiency. It is essential indeed to reduce the irradiation duration to pre-

vent sodium migration induced by the beam, the beam is rastered on the sample in order to avoid overheating of the glass. The experimental excitation curve is displayed in fig. 2b showing a small surface concentration followed by a steep step and a constant concentration of 16 at.% (glass bulk concentration).

The energy loss ΔE_p is measured by proton backscattering. Fig. 2a shows the Rutherford backscattering spectrum for the SnO_2 layer on glass in the same experimental conditions as used for the profiling, that is $E_p = 1.0$ MeV. The width $2\Delta E_p$ of the Sn signal is 29.2 keV, and can be related directly to the Na depth profile; if we use this value on the excitation curve we obtain the exact position of the interface. The advantage of this procedure is that we do not need to deconvolute the experimental data. Of course, it is possible to obtain a depth scale (in nm) using the stopping power of the SnO_2 layer and the stopping power of glass beyond the interface, but this is not essential for the analysis. What is essential for the

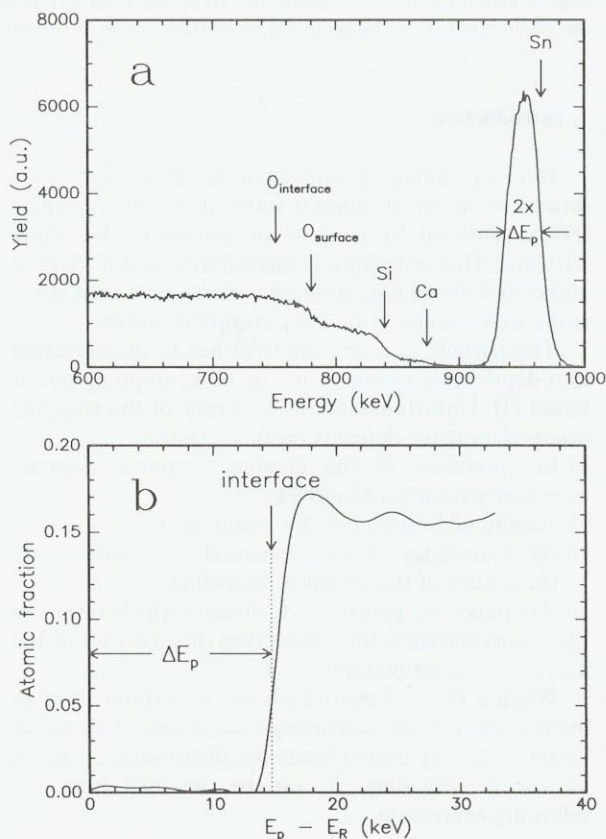


Fig. 2. Determination of Na profile by nuclear reactions; (a) interface depth measurement by 1.0 MeV proton backscattering on Sn, (b) sodium profile after deconvolution of the excitation curve by using $^{23}\text{Na}(p, \alpha\gamma)^{20}\text{Ne}$ at 1010.5 keV. The interface position is obtained from ΔE_p as measured in (a).

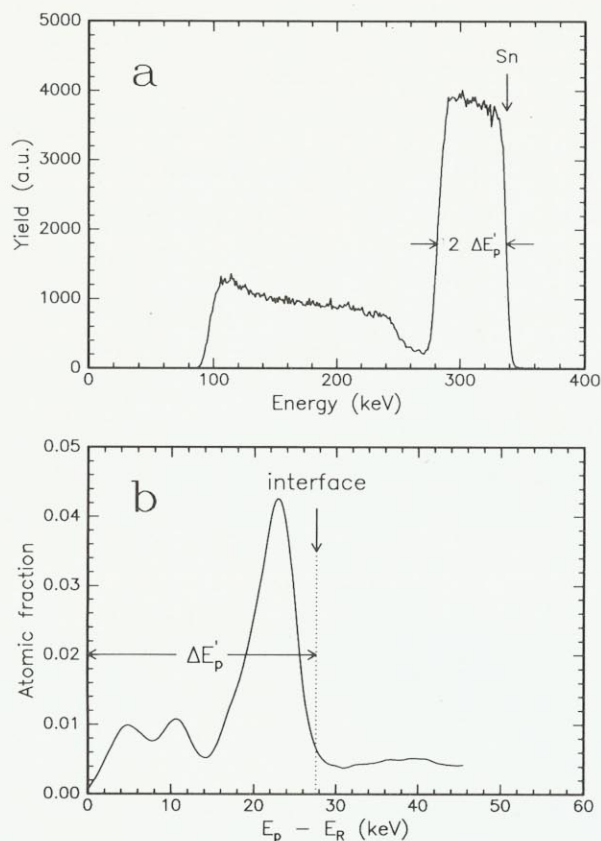


Fig. 3. Determination of fluorine profile by nuclear reactions; (a) interface depth measurements by 340 keV proton backscattering on Sn, (b) fluorine profile after deconvolution of the excitation curve by using nuclear resonant reaction $^{19}\text{F}(p, \alpha\gamma)^{16}\text{O}$ at 340 keV. The interface position is obtained from $\Delta E'_p$ as measured in (a).

customer is the exact localization of the concentration step with respect to the interface.

3.3. Fluorine profiling

Profiling of fluorine is made by using the $^{19}\text{F}(p, \alpha\gamma)^{16}\text{O}$ at resonance energy $E_R = 340$ keV ($\Gamma_R = 2.4$ keV). The interest of this resonance is that it is a lower energy and intense resonance, the next one is far beyond at 487 keV. A small resonance appears also at $E_R = 226.9$ keV [5] but this does not interfere with the 340 keV resonance because the energy loss in the $\text{SnO}_2(\text{F})$ layer is lower than 100 keV and there is no fluorine in the glass. For these reasons, the use of the 340 keV resonance produces very low background and larger depth can be analysed (1.5 μm in glass) without interference. High energy γ -rays (6 to 7 MeV) are detected in a 4 in. \times 4 in. well NaI detector.

The excitation curve is displayed in fig. 3b showing a shallow concentration followed by a huge peak that we have to localize exactly with respect to the interface and to the sodium step (fig. 2b). The energy loss $\Delta E'_p$ corresponding to the interface is measured by proton backscattering, as shown in fig. 3a. The Sn signal is near rectangular with a FWHM, $2\Delta E'_p$, which is found to be equal to 55.2 keV. In fact, the spectra were analysed by using the RUMP code [6]. The value found is very near to the FWHM that we used for sake of simplicity. Let us notice that in the case of fluorine the peak is localized in the SnO_2 layer.

4. Conclusions

By this practical example of a SnO_2 layer doped with fluorine we have shown that it is possible to localize the relative position of the fluorine peak, the interface and the sodium step without using stopping power parameters. In the present simple case, a calculation made using Ziegler's stopping power has given the same results.

We have analysed more complicated layers of unknown composition specially on light elements. In these cases the calculation is impossible. By the present technique, the analysis could be performed exactly. The only condition is that the layer contains an element heavier than all the other atoms present in the matrix.

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