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CALIBRATION PROCEDURE FOR PARTICLE ELASTIC SCATTERING ANALYSIS OF THIN SAMPLES

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A calibration procedure for particle elastic scattering analysis using simultaneous detection of X rays and scattered particles from spiked polystyrene foils of varying thickness is described. Experimental results of proton scattering yield dependence on sample thickness are given for carbon in a polystyrene matrix.

1. Introduction

The Rutherford backscattering method (RBS) is a well established technique [1] for the analysis of thin films, determinations of surface compositions and studies of elemental depth profiles in surface layers of bulk samples. The samples are usually bombarded with low energy protons (≤ 1 MeV) or *He*²⁺ ions (≤ 2 MeV). The simultaneous detection of scattered particles with a surface barrier detector constitutes an attractive complement to particle induced X-ray emission (PIXE) analysis for the determination of light elements. However, even for thin PIXE samples (e.g. aerosol samples) the projectile energies used for traditional RBS analyses are not sufficiently high for elemental resolution of the light elements which cannot be analysed with PIXE. With increasing projectile energy the classical Rutherford scattering formula ceases to be valid, making predictions of the differential cross sections difficult. In this paper a calibration technique for high precision analysis of thin samples is described.

2. Calibration procedure

2.1. Simultaneous detection of X rays and scattered particles

Thin calibration standards are required for high precision analysis. To study matrix effects it is necessary to use standards of varying thickness. Using a well calibrated PIXE setup the calibration procedure is facilitated by the simultaneous detection of X rays and scattered particles. The standard foils can be spiked with a metal to a known concentration and the thickness of the part of the standard foil irradiated can be determined with high precision by PIXE analysis.

2.2. Preparation of thin standards

Preparation of spiked foils for calibration of X-ray analysis has been described by Dzubay and Lamothe [2] and Billet et al. [3]. In selecting material for the thin standards used for calibration several factors have to be considered. The composition must be suitable for both PIXE and elastic scattering analysis, and the levels of impurities must be low. Polystyrene is a polymer of carbon–hydrogen composition which fulfills these requirements. It also has good mechanical strength, and can easily be made into thin foils of varying thickness.

The thin polystyrene foils are prepared from a bulk solution consisting of polystyrene dissolved in toluene [4]. The concentration used was 100 g polystyrene in 1 l of toluene. A clean glass plate was partially immersed in a vertical position into the solution, and then slowly raised out of the solution, whereby the glass plate was coated with a thin film of polystyrene solution. The thickness of the film was varied by changing the speed with which the glass plate was removed. This was achieved by connecting the glass plate, by means of a cord coupling, to a syringe pump motor with variable speed. After allowing the film on the glass plate to dry, the plate was lowered at an angle into a shallow beaker of water. The water wets the surface of the glass plate thus separating the polystyrene film from the plate. The bulk polystyrene matrix is spiked with a known amount of a metal that is easily measured by PIXE, in this case cobalt. Since the ratio of cobalt to carbon is given by the composition of the solution, the values for cobalt can be used as calibration for the elastic scattering analysis. In order to give good pulse counting statistics, it is desirable that the cobalt concentration be rather high. A cobalt concentration of 1800 ppm in the resulting polystyrene foils was chosen.

The polystyrene solution was spiked in the following way. A standard solution containing 1 g Co in 1000 ml...
of water was prepared. To 25 ml of this solution 4 g DDDC (diethylammonium diethylthiocarbamate), a complexing agent, were added causing the formation of a metal–carbamate complex. After evaporation of the water, the dry metal complex, which is soluble in toluene, was added to the polystyrene solution. Thin foils may be spiked with light elements in the same way by spiking a polystyrene solution with known amounts of the element in the form of a toluene-soluble compound.

Foils have been prepared in thicknesses varying from 20 to 1000 μg/cm². The limiting factor is the mechanical strength of the foils. When the thickness is less than about 20 μg/cm², the foils are very fragile and almost impossible to handle with our techniques.

2.3. Calibration

Calibrations for carbon analysis were carried out by irradiating the spiked polystyrene foils with protons of 2.55 and 5.00 MeV. The protons were accelerated with an electrostatic tandem accelerator (NEC 3 UDH). The PIXE setup for routine analysis at the Lund Institute of Technology described by Malmqvist et al. [5] was used. The angle between the beam and sample normal was 22.5° and a partially depleted surface barrier detector (ORTEC TA-014-25-300) collimated to 3.1 mm² was placed under the beam for the detection of protons scattered 155° in a solid angle of 1.8 ± 0.1 msr. The number of Co Kα pulses in the X-ray spectrum, multiplied by a thick target correction factor [6], is a measure of the foil thickness. Fig. 1 shows the number of pulses in the carbon peak of the particle spectrum as a function of the corrected number of pulses in the Co Kα peak. For increasing target thickness there are obvious departures from linearity. The intensity of scattered

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**Fig. 1.** Relation between carbon pulses/μC (A) and cobalt Kα pulses/μC (N) from the simultaneous detection of scattered particles and X rays according to the calibration procedure described in the text. For 2.55 MeV and 5 MeV protons 33 ± 2 and 62 ± 3 pulses/μC, respectively, correspond to 1 μg/cm² polystyrene. The cobalt concentration in the polystyrene was 1755 ± 60 ppm. The particles were scattered 155° into a solid angle of 1.8 msr. The solid lines represent the functions $A = 1.35N + 6.9 \times 10^{-8} \times N^{2.55} \text{ (2.55 MeV)}$ and $0.75N + 8.7 \times 10^{-8} \times N^{2.55} \text{ (5.00 MeV)}$. The uncertainties due to counting statistics are below 1%.

**Fig. 2.** Particle spectrum from a spiked polystyrene foil of thickness 30 μg/cm². The foil was irradiated with 10 μC protons and the scattered particles were detected at a scattering angle of 155° and into a solid angle of 1.8 msr. Sulphur and nitrogen are constituents of the DDDC-compound (C,H₂,N,S,).

**IID. COMBINED TECHNIQUES**
particles from carbon reaching the detector was calculated by summing the number of pulses in the characteristic carbon peak without any correction for the background on the low energy side of the peak. Fig. 2 shows the particle spectrum for a 30 μg/cm² spiked polystyrene foil.

3. Discussion

A peak in an elastic scattering spectrum does not have a gaussian shape and cannot easily be fitted by theoretical models in the case of non-Rutherford
scattering. The low energy tail and the background continuum behind the peak have been of unknown origin [1]. However, recently Weber et al. [7,8] have successfully performed computer simulations of RBS spectra assuming that the low-energy continuum is due to multiple scattering (mainly double scattering) in the sample. Usually in particle elastic scattering analysis, the elements are quantified by simply summing the pulses in the characteristic peak. However, the lack of knowledge about the magnitude of doubly scattered particles in relation to singly scattered particles may cause a significant analytical error. Weber and Mommsen [8] give a quantitative measure of the number of doubly scattered particles in an RBS spectrum:

\[ \text{Bkgnd} = \text{const.} f(E, \theta, Z)d^{2.55}. \]

Bkgnd is the number of particles doubly scattered into a certain solid angle and into an energy interval of the spectrum. \( f(E, \theta, Z) \) is a function, of energy \( E \) of the incident protons, scattering angle \( \theta \) and atomic number \( Z \) of the target nucleus, valid for classical RBS and \( d \) is the target thickness. The proton energies used in the carbon calibration procedure are beyond the valid region of their formula. The \( d^{2.55} \) dependence, however, is a geometrical factor and can be assumed to be independent of energy. In fig. 1 our experimental data are fitted to a function \( A = aN + bN^{2.55} \) (\( A \) = number of pulses in the carbon peak, \( N \) = number of pulses in the Co K\( \alpha \) peak and a measure of the target thickness; \( a \) and \( b \) are constants) which seems to describe the experimental results. The additional \( d^{2.55} \) dependence indicates that a significant number of doubly scattered particles were included when the total number of counts in the characteristic peak was calculated.

4. Carbon analysis

In the analysis of aerosol samples carbon is an element of considerable interest. The foils used for calibration can be used as aerosol collection substrates. Since they have a well determined ratio between the metal and the carbon content, the substrate content of irradiated carbon may be subtracted from, for example, an impactor sample with a fine aerosol fraction. Andreae and Barnard [9] have described a similar technique using the constant ratio between carbon and fluorine in a teflon filter. Fig. 3 shows an example of carbon analysis using scattered \( ^4\text{He}^{2+} \) ions on a silver membrane filter for aerosol collection. Fig. 4 shows an example of proton elastic scattering analysis of carbon on a gold foil. The two substrates were coated with 20 \( \mu \text{g/cm}^2 \) carbon.

5. Conclusions

For elastic scattering analysis of high precision careful calibration with standards of varying thickness has to be performed. By using thin plastic foils spiked with a metal together with simultaneous X-ray and particle detection the calibration procedure is facilitated.

References