RUTHERFORD BACK SCATTERING USING HEAVY ION BEAMS

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1. INTRODUCTION

Ion Beam Analysis (IBA) techniques have been increasingly used in the analysis of industrial materials during the last 10 - 15 years. General methods of Nuclear Reaction Analysis (NRA) for determination of specific elements are given elsewhere in these proceedings (see P.Missailides et. al.). In this contribution, we are restricted to the use of Rutherford Scattering in the determination of composition and profile of thin films.

The Rutherford Back Scattering [1] method provides a relatively simple technique for analysis over a wide range of elements. The method is based on the simultaneous measurement of yield and energy loss of particles scattered at large angles (150°-170°) after a Rutherford collision.

Usually, low energy He particles are used for thin samples, although in some cases higher energies are required [2,3]. In the Institute of Nuclear Physics at "Demokritos" we used alternative heavier ion beams of ^{7}Li and ^{12}C for characterization of thin films, either home made or of interest on Materials Science.

2. THE RUTHERFORD BACK SCATTERING METHOD

In the Rutherford Back Scattering method, the sample is illuminated with an ion beam of energy low enough, so that the interaction is Rutherford scattering and the cross section known and easy to calculate and the reaction products are detected at large angles. As fig. 1. shows, if the sample has a thickness ρ and the incident particles have energy E_0, the resulting spectrum line is not a Gaussian

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type peak, but is rather stretched to a plateau of width ΔE. The width of the plateau is related to the combined energy loss of incident beam and scattered particle and leads to the determination of the thickness and profile of the sample, while the yield depends on the number of atoms of a particular element and leads to the elemental composition.
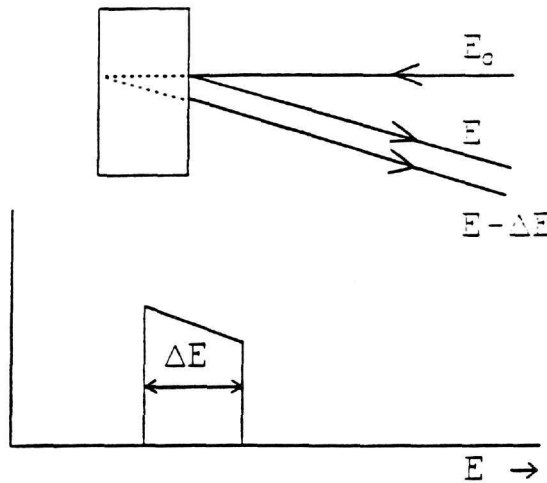
Rutherford Back Scattering

Fig. 1. The Rutherford Back Scattering mechanism.

The choice of the bombarding energy and the type of projectile to be used, depends only on the thickness and stopping power of the material, so that the resulting width ΔE is measurable, compared to the system resolution. The only restriction is that the energy of the particle is low enough and the interaction is still Rutherford Scattering, so that the cross section is easy to calculate accurately.

Usually, ^{4}He beams of energy 1-3 MeV are used for sample of thicknesses ranging from some hundred Å to some thousand Å. In the TANDEM accelerator laboratory at the INP "Demokritos", we utilized alternative beams of ^{7}Li and ^{12}C at energies 7 and 10 MeV respectively for Heavy Ion Rutherford Back Scattering (HIRBS). At these energies, the interaction is fully Rutherford Scattering for all the heavy elements we are interested. A small deviation exists [4] for low Z elements (like Si) which is within the accuracy of the measurement.

There are some advantages as well as some disadvantages in using HIRBS instead of the traditional light ion RBS. The advantages are:

Better depth resolution provided by the increased stopping power of the heavy ion.

Lower minimum measurable thickness

In the side of disadvantages we could note the following:

Poorer knowledge of the stopping power coefficients for heavy ions, compared to light ions (mean accuracy 9%).

Higher detectable mass number threshold, for kinematical reasons. As a practical rule, this threshold is twice the mass number of the projectile. Thus, light elements like O, C, N can not be detected using a projectile like ^{12}C.

Poorer detector resolution for heavy ions than for light ions.

Since the commonly used computer codes for RBS analysis (like RUMP[5]) do not handle projectiles heavier than ^{4}He, a new computer code, named HISCAT was written, which can handle any ion beam and is now running at the computer PRIME 750 of the laboratory. The program uses Ziegler's "Universal Energy Loss Coefficients"[6] and the subroutine ZSTOP (by Ziegler) to calculate the energy loss of any ion through any material and the usual Rutherford scattering cross section formula to produce the normalized yield. Finally, it outputs a simulation spectrum in a format that can be read by the existing analysis codes of the laboratory (ANALYSIS,DAMA,ZEUS).

3. EXPERIMENTAL PROCEDURE:

Beams of ^{7}Li and ^{12}C were generated by the Inverted Sputter source and accelerated by the TANDEM TN11 accelerator of the INP "Demokritos" to reach energies of 7 and 10 MeV respectively. These energies are a compromise between the required low energy for Rutherford Scattering and good beam optics conditions at the 5.5 MV terminal voltage accelerator. The beam was led to an 80 cm diameter scattering chamber which has the ability to carry five different targets. A 50 μm surface barrier detector was placed at a distance 16 cm from the target, collimated by a 5 cm diameter collimator, thus having an angle opening of $\Delta\theta=1.8°$. The beam spot on the target was kept at $2 \times 2mm$ via a set of two beam collimators. The solid angle was found by normalizing on the Rutherford cross section of standard targets of Au and Lu.

4. APPLICATIONS:

We now routinely apply the HIRBS technique in characterization of thin films in a number of problems. Some of the fields of our interest was:

i) Characterization of thin films used as standards for calibration in other nuclear techniques like PIXE, XRF, PIGE etc. These standards are produced by evaporation on Kapton and are characterized by both HIRBS and XRF. The spectrum of a 205 $\mu g/cm^2$ KBr film on Kapton backing is shown in fig. 2 together with the simulation spectrum. The increased background in the spectrum is typical of measurements · using ^{7}Li beam and is due to ^{7}Li breakup to σ and t.
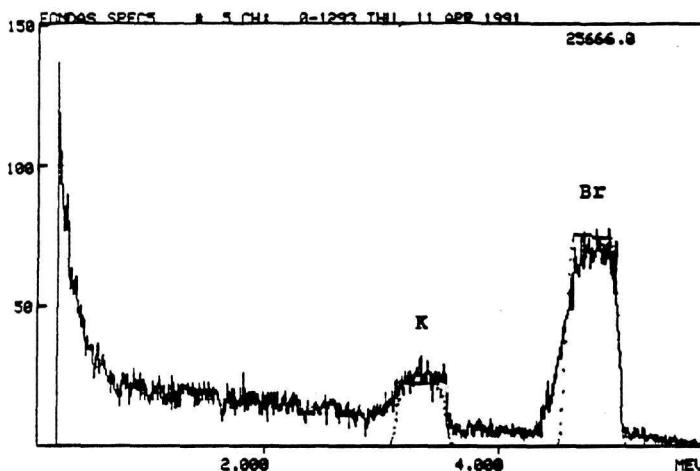

Fig. 2. RBS spectrum of a KBr film, using ^{7}Li beam.

ii) Characterization of films of magnetic and superconducting materials manufactured in the Institute of Materials Science, at "Demokritos". A Spectrum of a magnetic material taken with ^{12}C beam is shown in fig. 3. The plateaus of Fe, Nd and Dy are pronounced.

iii) Characterization of films of metal silicides produced by the Institute of Materials Science, which attracted most of the beam time devoted to RBS.

5. STUDY OF METAL SILICIDES:

Silicides of certain metals are of great importance to Materials Science and research for new materials, because they combine the metallic nature, low resistivity and good thermal and mechanical properties. Such materials are prime candidates to replace Al in the construction of Very Large Scale Integrated circuits (VLSI) because they have all the useful properties of Al, while they lack its diffusion in Si and its poor thermal
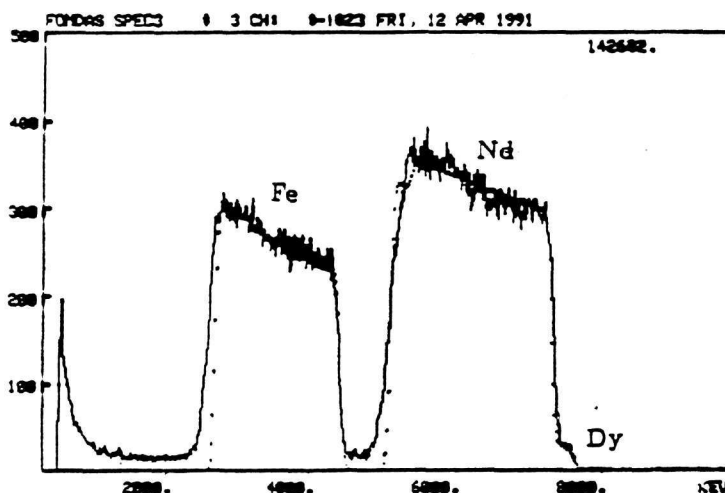
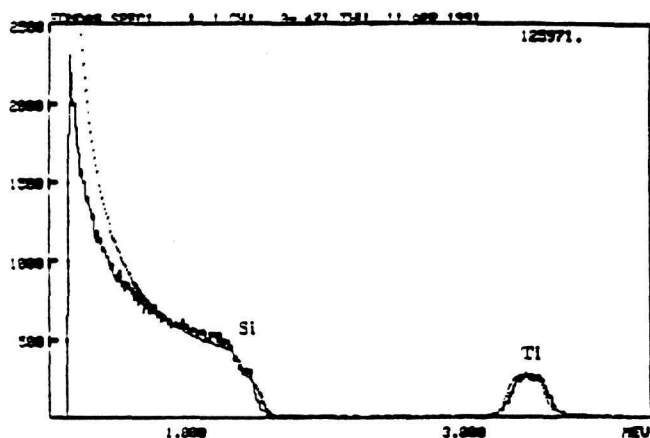Fig. 3. RBS spectrum of a magnetic material, using ^{12}C beam.

Fig.4. Spectrum of TiSi$_2$ on Si substrate, using ^{12}C beam.

and mechanical endurance.

The Institute of Materials Science at "Demokritos", has started a research project on the construction of these materials and their characterization using RBS, X-ray spectroscopy and electron microscopy. The Institute of Nuclear Physics collaborates in the characterization of thickness and composition of those films, using Heavy Ion RBS (HIRBS). These silicides are produced by evaporation of a metal on Si backing and annealing to 500°-1000°. This way phases

of metal silicides are formed, of thickness varying from 500 A to 2000 A. The aim of the project was to find the best conditions of sample preparation to achieve the desired phase of MSi_2.

In the course of the project, we studied silicides of Ti and Lu. We used a ^{12}C beam at energy 10 MeV for RBS measurements to find the exact thickness, profile and composition (M - Si phase) of the specimen. The analysis was done using the computer code HISCAT.

Fig. 4. shows the spectrum of 1332 ±110 Å of $TiSi_2$ on Si backing, together with the computer simulation. We examined a number of 8 samples of different thicknesses of Ti on Si annealed at different temperatures and the results were the following.

> As the RBS profile shows, the transition area between the silicide and the Si substrate is not sharp, indicating problems in the sample preparation.

> In most of the cases, the composition resulted by RBS corresponds to phase of TiSi, or an admixture of TiSi and $TiSi_2$, rather than a pure $TiSi_2$ phase.

> In the cases where the treatment problems are overcome and pure $TiSi_2$ is formed, the product shows the desired low resistivity.

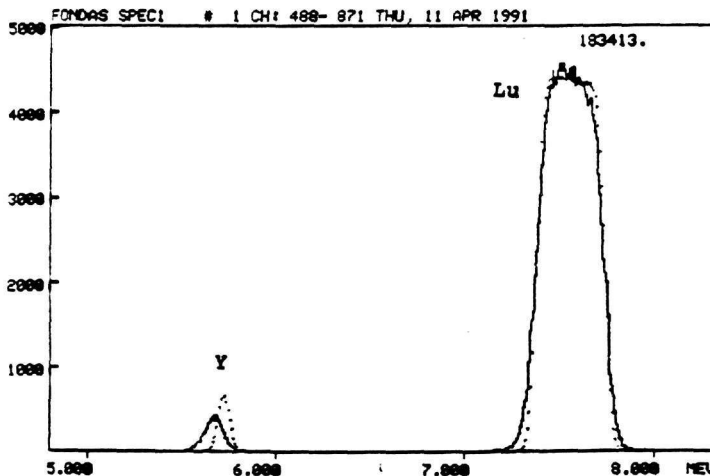FONDAS SPEC1   # 1 CH: 488- 871 THU, 11 APR 1991

Fig. 5. Part of RBS spectrum showing the Lu and Y peaks.

Turning now to the side of Lu silicides, samples were prepared by evaporating a thin layer of Y on a Si wafer and, on the top of that, a thicker Lu layer was evaporated. The RBS spectrum containing the Lu and Y plateaus is shown in fig. 5 together with the computer simulation. Although the simulation of Lu is good, the Y peak is much wider than expected, although it contains the same number of Y atoms as the

simulation. A good fit was obtained when we assumed an admixture between Y and Si. The final product was then annealed to 800°, thus forming phases of Lu - Si. Fig. 6 shows the RBS spectra before and after annealing. The results show that Si atoms really transpass the Y layer and form a phase of Lu-Si, but these phases do not correspond to pure $TiSi_2$.
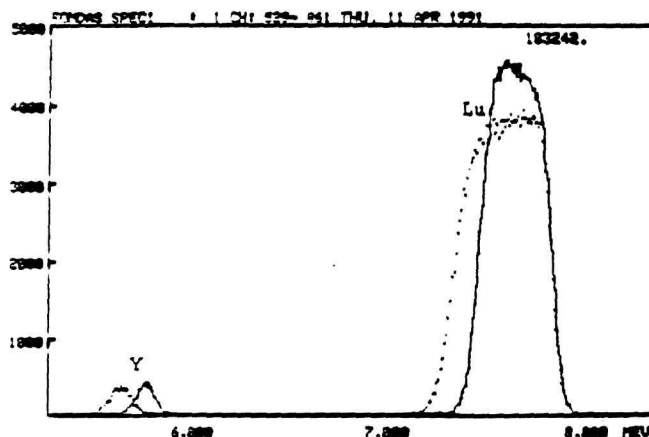

Fig. 6. Part of RBS spectrum using ^{12}C beam of Lu-Y-Si (solid line) and Lu-Y-Si annealed at 800°.

6. CONCLUSIONS:

We utilized heavy ion beams for RBS measurements and a new, reliable computer code for analysis. We now routinely use this method for characterization and profile of thin films. This technique was also found very useful in profiling films of metal silicide in the search for new materials, which is a project under continuation.

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A NEW SOURCE FOR PRODUCTION OF ^{15}N BEAM *

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The resonance of the nuclear reaction H(^{15}N, α, γ)^{12}C at 6.385 MeV has a small resonance width (1.8 keV) and therefore gives a substantially sensitive depth resolution (10 nm from the surface to 2 μm depth) though at a lower cross section [1]. The operating ^{15}N$^+$ ion beam at the 5.5 MV tandem accelerator of the Demokritos National Research Center was generated with a sputter source [2] using a flow of ^{15}N$_2$ gas on a carbon cone at a pressure of 10^{-6}mbar. During these experiments a high gas consuption and low efficiency was indicated, resulting in a low beam-current intensity generated in the sputter source. In the meantime a satisfactory solution has been proposed and developed. In the present letter we describe operation of a solid cone of Ti(C_xN_{1-x}) as a solid sputter source.

Titanium carbonitride Ti(C_xN_{1-x}) was selected as the nitrogen-containing solid beacuse this compound has high thermal stability and can therefore be sintered to a solid pellet under vacuum without and essential loss of nitrogen.

Ti powder obtained by the decomposition of titanium hydride is sufficiently reactive to react with gaseous ^{15}N$_2$. A detailed report of the parameters influencing the reaction kinetics these conditions is given elsewhere [3].

After a reaction period of 3 h at 1000°C between the Ti powder and the ^{15}N$_2$ under a gas pressure of about 0.8 bar, a gold-coloured powder was obtained which consisted of two phases, according to X-ray diffraction analysis. The cubic "TIN" was detected as the main phase and the hexagonal Ti$_2$N as the secondary phase. Graphite
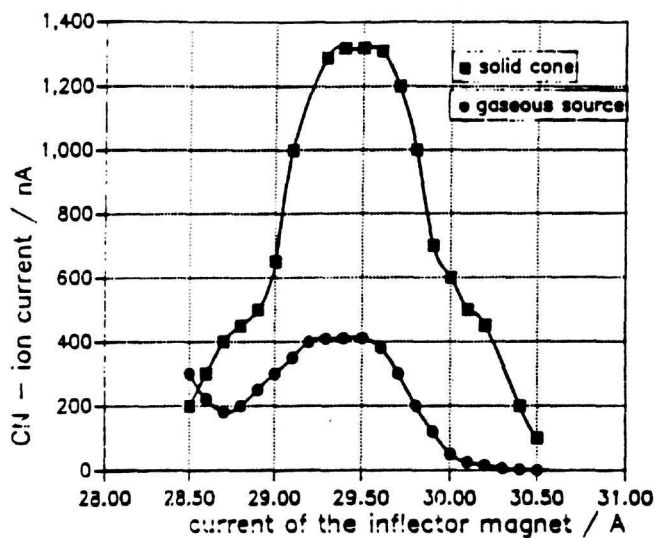
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Fig. 1. After the inflector magnet a three times higher CN⁻ ion current is observed using the solid cone compared to that using the conventional gaseous source.
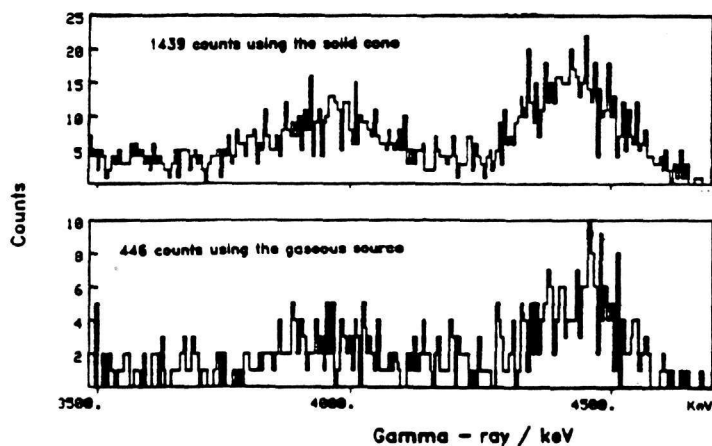

Fig. 2. Partial gamma-ray spectra obtained under similar conditions using solid cone and gas flow of $^{15}N_2$.

was added to this product (C: Ti= 0.4). The mixtures were then pressed and subsequently sintered under a vacuum for 4 h at 1700°C. Two cubic phases were observed after this heat treatment with lattice parameters α_1 = 427.86 ±0.06 for the main phase and α_1 = 431.43 ±0.04 pm for the secondary phase, corresponding to two carbonitrides of composition $TiC_{0.54}N_{0.45}$ and $TiC_{0.93}N_{0.07}$ respectively..

A comparison of the two $^{15}N^+$ ion source was made under similar conditions. It can be seen in fig. 1 that the negative (CN$^-$) current measured in the exit of the sputte source using the solid cone (upper spectrum) is about three times higher than that measured using the gaseous source (lower spectrum). Similar results have been obtained from the analysed $^{15}N^+$ ion current intensity, i.e. 25 or 8 nA at the resonance energy (6.385 MeV), respectively. Fig. 2 shows a comparison between two spectra of the integral gamma-ray emissiion during 236 s from a Ta sample loaded with hydrogen at a concentration of about 20%. The count rate registered under these emissions is also three times higher (1439 and 446, respectively) using the solid cone compared to the gaseous ^{15}N ion source.

The duration of a pellet is also much higher than that of one $^{15}N_2$ bottle. While a one-litre bottle is already consumed after about 200 h of operation, the first pellet runs for 6 months without any change in current indensity. Considering that 4-8 pellet of the above mentioned dimensions can be produced using one litre of nitrogen gas, this is obviously an economic advantage for operation of the beam with the solid ^{15}N ion source.

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