THE USE OF MeV PROTON NON—RUTHERFORD ELASTIC BACKSCATTERING FOR THE ANALYSIS OF LOW Z ELEMENTS

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ABSTRACT

The MeV proton non- Rutherford elastic backscattering (PEBS) has been used to measure a variety of low Z element- containing samples including thick SiC film, N implanted stainless steel, thin films interface (Ag on Cu) and very deep (4μ m) SOI structure by high energy oxygen implantation. It is demonstrated that by using the significant enhancement of cross sections for low Z elements and selecting the proper energy region of the excitation curves both the sensitivities for detecting low Z elements and the accessible depth of the PEBS technique are remarkably improved over the ordinary RBS method. The disadvantages of the PEBS as compared with RBS as well as high energy He elastic backscattering (HeEBS) are also discussed.

Keywords: RBS   MeV proton elastic backscattering for materials analysis

I . INTRODUCTION

It has long been recognized that the conventional He backscattering is not a reliable technique for detecting low Z elements in heavier substrates[1], because of the Z square dependence of the Rutherford scattering cross sections. Consequently, a variety of ion beam analytical methods has been developed to overcome the weakness. Among those well known are the use of specific nuclear reactions (NRA)[2] and forward elastic recoil detection (FERD) methods[3]. However, these two methods in general need more complicated procedures both in geometry and data analysis. On the other hand, the non- Rutherford proton elastic backscattering (PEBS) at MeV region seems to be a promising phenomenon that can be utilized for quantifying and profiling low Z elements while maintaining the simplicity of the data reduction and the equipments. At a few MeV energy region there are significant elastic nuclear potential and resonant scattering processes for common light elements such as C, N, and O. The corresponding cross sections can be enhanced many times relative to Rutherford values and the excitation curves usualy appear themselves as a general increase/decrease or resonant features with several keV in width. Although as early as 70s PEBS had been used to measure implanted He and other low Z elements in metal films or polymers[4,5], because of the lack of a comprehensive tabular for the cross sections of interest limited applications of this technique have been carried

out[6]. Recently, however, Rauhala et al.[7,8] have published the differential elastic scattering cross sections of 0.7– 2.5 MeV protons (in the laboratory system) for ^{16}O, ^{12}C, ^{14}N and ^{28}Si at scattering angles of interest to the material analysis rather than to the studies of nuclear excited states. By using these data available in Refs.[2,7,8] (see Fig.1 a, b, c, and d) it should be possible to employ protons as probe for material analysis dealing with low Z element– containing samples, and to handle the proton backscattering spectrum with the same equipments and same methods used for conventional He backscattering. In this paper we present some results on the applications of PEBS to various material analysis including thick films containing C and N, thin film interfaces, N implanted steel and high energy implanted O in Si etc. the advantages and disadvantages of PEBS as compared with ordinary He RBS as well as high energy He elastic backscattering are also discussed.
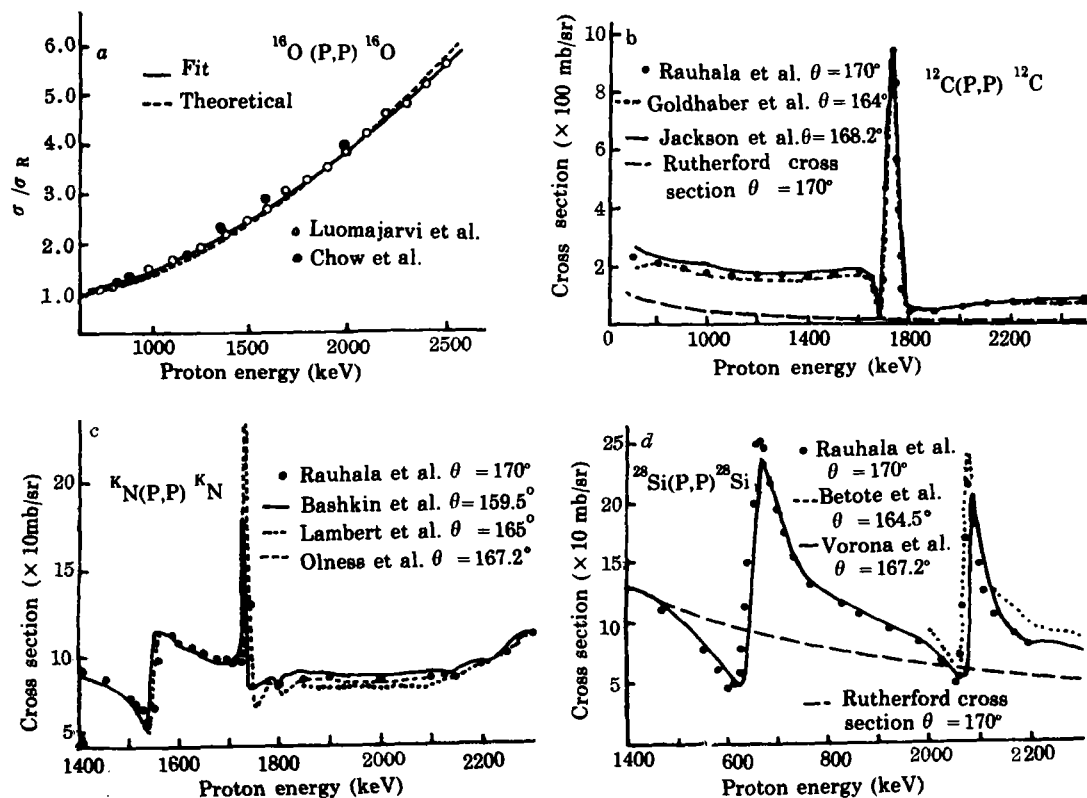

Fig.1 (a) Correction factor σ /σ_R for He Rutherford differential scattering cross section of oxygen as a function of ion energy, θ =170°. (b), (c), (d) Scattering cross sections for elastic backscattering of protons by carbon, nitrogen and silicon, respectively. (in the laboratory system) the curves are taken from published Refs. [2,7,8]

II. EXPERIMENTS

Proton beams with the energies in the range of below 2.5 MeV were introduced by

the 2×3MV tandem (NEC 9 SDH- 2) at Fudan university for PEBS experiments. The scattering angle of 165° was chosen by the Si(Au) surface barrier detector with 100μ m in sensitive depth and with a 3 mm collimater in diameter to collect the backscattered protons from targets. The energy resolution for the whole measuring system (standard RBS set up) is about 18 keV. The samples analysed in experiments were prepared either by deposition with plasma- asisted CVD and vacuum evaporation or by ion implantation in order to explore the unique charactetistic of PEBS technique. The 2 MeV He RBS measurement were also performed on those samples in the same geometry and even same beam spots (1mm) or target area for straightforward comparison with the PEBS measurement.

III. RESULTS AND DISCUSSION

Fig.2 (a) shows the 2.0 MeV He RBS spectrum of a thick silicon carbide film on silicon. It is obvious that due to the kinematics and Z square dependence of cross
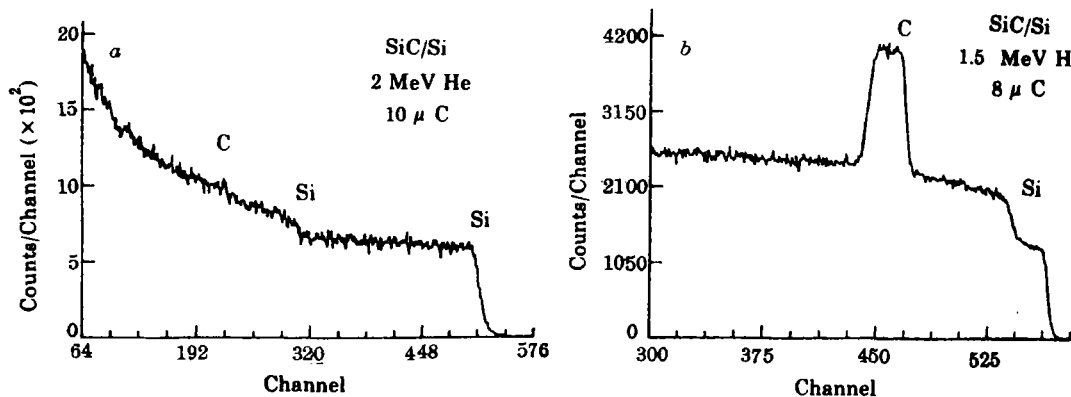

Fig.2 Backscattering spectra of 2.0 MeV He RBS (a) and 1.5 MeV PEBS

(b) of a sample of 1.7μ m SiC film on Si wafer

sections the C signals are superimposed upon that from the heavier Si substrate and the C signal height Hc is only about 10% of the Si signal height H_{si}. Therefore, it is difficult to have accurate values of both stoichiometry and thickness of the film from the RBS spectrum. The spection shown in Fig.2 (b) shown is the 1.5 MeV proton back scattering spectrum of the same sample. The C signals are remarkably enhanced (the cross section is about 8 times larger relative to the
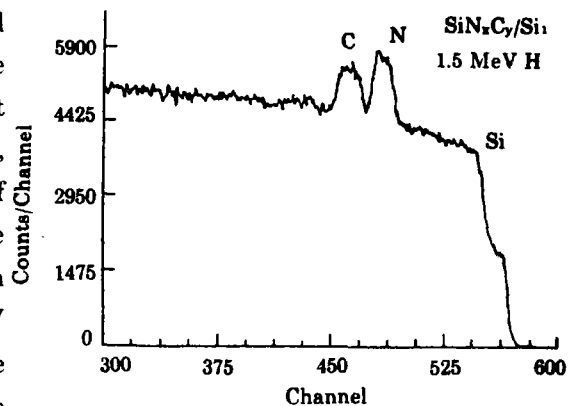

Fig.3 Backscattering spectra with 1.5 MeV protons from a thick silicon−nitride−carbride film on Si substrate

Rutherford one) in PEBS spectrum and because of much less energy loss of proton the spectrum illustrates the whole thickness of the SiC film. The thickness calculated is 1.72μ m and the composition of the film is estimated to be [Si]:[C] = 1:1.1 with the statistic uncertainty of 4%.

The spectrum of a SiN_xC_y film on Si shown in Fig.3 is another example to demonstrate the ability of PEBS to profile C and N simultaneously. However, an additional PEBS measurement with different beam energy other than 1.5 MeV is necessary to obtain the accurate value of the composition for the film, because it is not easy to find proper energy region at which C, N and Si simultaneously have a smoothly varying enhanced cross sections.
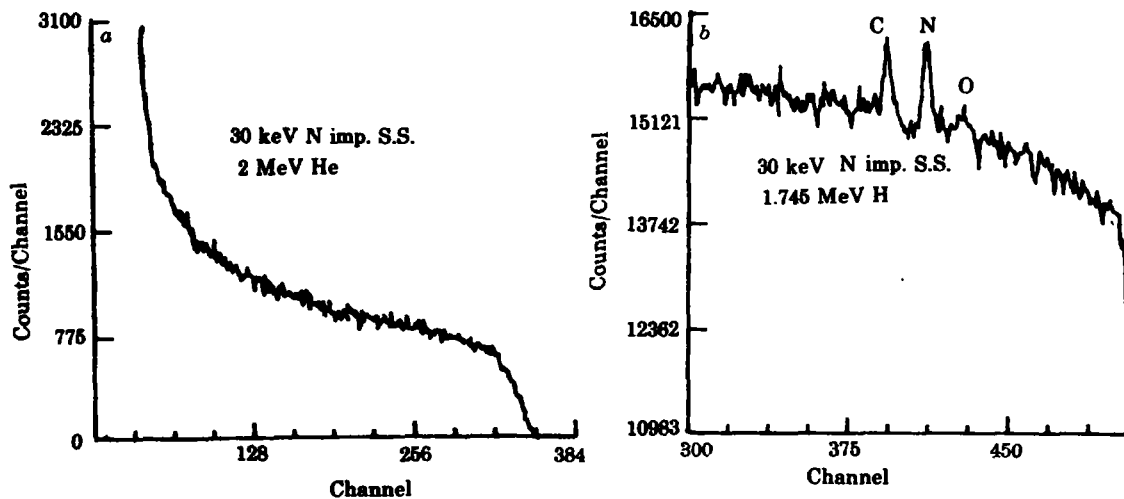

Fig.4   Backscattering spectra with 2.0 MeV He particles (a) and 1.745 MeV protons (b) of a 30 keV ^{14}N implanted (1×10^{17} at./cm^2) stainless steel sample

Note that the N signal in the RBS spectrum is invisible while that in the PEBS spectrum is clearly shown

In Fig.4 shown are results of the backscattering measurements on a 30 keV ^{14}N implanted stainless steel sample (1Cr18Ni9Ti). While in RBS spectrum (a) the N signals is almost invisible, the PEBS spectrum (b) gives the clear presence of implanted nitrogen layer. However, in this case the proton energy selected is crucial, not only to compensate the energy loss of the incoming ions but also to find a energy near the resonance with reasonable width of resonant which can cover the whole depth to be profiled. In the experiment the proton energy chosen was 1.745 MeV, near the 1.735 MeV strong resonance with 15 keV resonant width which was greater than the estimated energy spread (about 5.5 keV) corresponding to the N distribution. The calculated implanted N areal density 1×10^{17} at./cm^2 consists well with the value obtained by other methods.

This method by using the resonant feature rather than of the flat region of the

excitation curve can also be utilized to probe the interface of mutilayer films. Fig. 5
(a) shows the PEBS spectrum of a Ag film on Cu substrate sample for research on
MeV ion- enhanced adhesion. The residual carbon as well as oxygen signals both on
the surface and at interface, which were not able to see in RBS, clearly stand out from
heavy Cu background in PEBS spectrum. The concentrations of surface C, interface C
and interface O calculated are 7×10^{16}at./cm^2, 8×10^{15}at./cm^2 and $2\times 1\,0\,^{17}$at./cm^2,
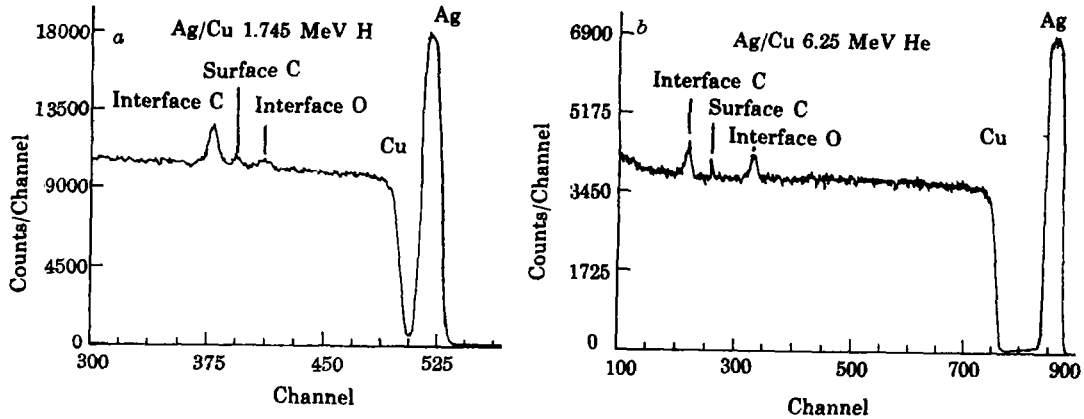respectively.

Fig.5 Backscattering spectra of 1.745 MeV PEBS(a) and 6.25 MeV He EBS (b) from a sample of
silver film on copper substrate

Note that residural C can be seen both on surface and at interface while O only shown at interface

Fig.6 is a PEBS spectrum taken from
a SOI structure, a very promising
technique being used in VLSI. A buried
silicon- oxide layer with several
microns beneath the surface of a Si
wafer was prepared by very high
energy oxygen implantation at 5 MeV
with 1.4×10^{18} at./cm^2 dose, but it was
too deep to be profiled by 2 MeV He
ions in RBS. On the other hand, the
SIMS or AES methods commonly
adopted to obtain the information
about the implanted oxygen have some
problems related to depth scaling as

Fig.6 PEBS spectrum with 2.057 MeV protons
from a SOI sample prepared by 5 MeV O ions
implantation (1.4×10^{18}at./cm^2) of a Si wafer. The
buried silicon—oxide layer is about 4μ m beneath
the surface

well as the destrutiveness during measurement. The PEBS spectrum shown in Fig.6
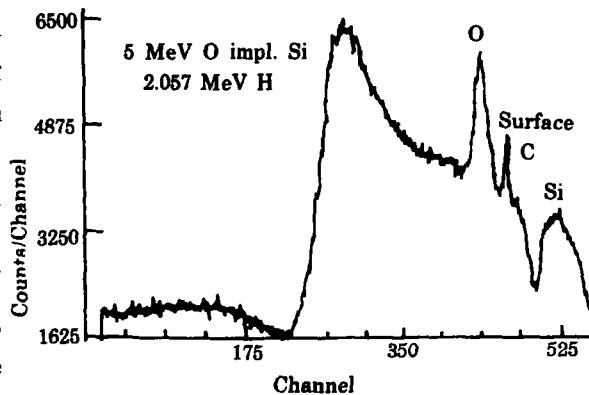was our first try to use proton instead of helium ions for SOI profile. As one can see

in the figure, it illustrates a clear picture of the SOI structure. Due to the rather deep penetrability of MeV proton. The calculated implanted depth of 3.9μ m from the spectrum is in good agreement with SIMS result of 3.9μ m and the expected value 3.70μ m by TRIM 88 program. It is possible to get other parameters of implanted layer from the spectrum and the detailed analysis of the sample will be given in a separated paper elsewhere.

In conclusion we have demonstrated that the enhanced backscattering cross sections of C, N, O and other low Z elements in PEBS enable us to determine the composition of light elements – containing samples more accurately than ordinary RBS does. The another feature of PEBS is to have such a greater penetrability that as thick (or deep) as $5-6$ μ m samples, which are far beyond the RBS probing depth, are easy to profile. Although the less energy loss of proton also leads to poor depth resolution the reduced mass resolution due to the kinematics is not always a serious problem provided very different masses of components in sample are involved. In Fig. 5 (b) shown are the high energy He elastic backscattering (HeEBS) at 6.25 MeV taken from the identical sample in spectrum (a) for probing the residural carbon and oxygen at interface region. The HeEBS is another novel method being used for profiling oxygen in high Tc superconductor[9]. By making a comparison between two spectra in Fig.(5) it is evident that in spite of the worse mass and depth resolution the PEBS shows comparable sensitities for low Z elements to that of the HeEBS. Furthermore, while the higher than $3-5$MeV beam energy needed for HeEBS are usually not available for small accelerators, the $1-2.5$ MeV proton beam for PEBS are easy to offer for most materials analysis lab.. Finally, it is worth to point out that care must be taken as concerning the interferences from possible nuclear inelastic reactions and also in order to obtain accurate results from the PEBS it is better to measure the cross sections of interest under the condition of our own lab., since there must be some variations in energy calibration and scattering geometry from one lab. to another lab.

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