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APPLICATION OF HEAVY-ION BACKSCATTERING SPECTROMETRY TO THE ANALYSIS OF SEMICONDUCTOR MATERIALS

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Abstract

Unique features of Heavy-Ion Rutherford Backscattering Spectrometry (HIRBS) for the analysis of medium to high atomic number materials are described. Comparisons between 20 MeV $^{16}$O ions and 1.5 MeV $^4$He ions are made based on calculations and experimental data. The results are characterized in terms of mass resolution, depth resolution, maximum accessible depth, and the minimum detectable limit for minor impurities. Backscattering spectra obtained on gallium arsenide and germanium samples are shown which clearly illustrate the advantages of heavy ion RBS for the analysis of high atomic number materials.

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Introduction

The potential advantages of Heavy-Ion Rutherford Backscattering Spectrometry (HIRBS) over conventional RBS using hydrogen or helium ions have been pointed out by several authors and a number of comparative studies have been performed. The principal advantage of HIRBS is the improved mass resolution for the analysis of high atomic number samples. Additional advantages which can be exploited in certain applications include increased accessible depth and a smaller minimum detectable limit for impurity concentrations. These advantages have recently become increasingly important for the development of devices using compound semiconductors containing elements of high atomic number. We present the results of a study in which quantitative results for the sensitivity, depth and mass resolution for HIRBS are compared to $^4\text{He}$ RBS in applications involving gallium-arsenide and germanium.

A typical RBS measurement consists of bombarding a sample with a collimated beam of energetic ions. A charged particle spectrometer (typically a silicon surface barrier detector) is placed at a fixed backward angle relative to the sample and an energy spectrum of backscattered ions is recorded. The improved mass resolution of HIRBS relative to conventional RBS arises from the increased dispersion of backscattered particle energy as a function of target mass. If one assumes elastic nuclear scattering between an incident ion of mass $M_1$ and a target atom of mass $M_2$, then the energy of the scattered ions is given by:

$$E_i = E_0 \left[ \frac{(M_2^2 - M_1^2 \sin^2 \theta)^{\frac{1}{2}} + M_1 \cos \theta}{M_2 + M_1} \right]^2$$

where $E_0$ is the energy of the incident projectile, $E_i$ is the energy after scattering and $\theta$ is the scattering angle. For the special case of $180^\circ$...
backscatter, this reduces to the more familiar expression:

$$E_i = E_0 \left( \frac{1 - M_1/M_2}{1 + M_1/M_2} \right)^2$$

(2)

Figure 1(a) shows plots of the backscattering coefficient $K = E_i/E_0$ for the case of $^4\text{He}$ and $^{16}\text{O}$ projectiles and are presented for the case of a 170° scattering angle. The increased slope of the $^{16}\text{O}$ curve for target masses $A > 50$ clearly indicates the improved mass resolution achieved. The curves in Fig. 1(b) show mass resolutions calculated using $aM/aE$ as derived from the relationships of Fig. 1(a) combined with a typical experimental ion detection energy resolution (full width half maximum) of 15 keV for $^4\text{He}$ and 100 keV for $^{16}\text{O}$. Under these assumptions, the ability to resolve adjacent target isotopes extends to $A = 150$ for $^{16}\text{O}$ compared to $A = 50$ for $^4\text{He}$.

In addition to providing mass identification, the RBS method can also measure the depth distribution of elements present in the near surface region. This capability is the result of the continuous energy loss processes which the incident ions undergo while traversing the sample before and after a Rutherford backscattering interaction. Insofar as the energy loss rate $(dE/dx)$ is different for heavy and light ions, it is anticipated that parameters affecting profile measurements such as depth resolution and maximum accessible depth will be different for HIRBS. Finally, the minimum detectable amount of impurity atoms which can be measured is likewise influenced by the type of incident ion. The backscattered signal is proportional to the Rutherford scattering cross section which varies as:

$$\frac{d\sigma}{d\Omega} \propto \left( \frac{Z_1Z_2e^2}{4E_0} \right)^2$$

(3)

where $Z_1$ and $Z_2$ are the projectile and target atomic numbers and $e$ is the electronic charge.
Experimental

Measurements were performed using both the LBL 2 MeV Van de Graaff facility ($^4\text{He}^+$ at 1.5 MeV) and the heavy-ion capability of the 88" cyclotron ($^{16}\text{O}^{2+}$ at 20 MeV). A surface barrier detector depleted to a depth of 200 µm and with 20 keV FWHM energy resolution for 5 MeV alpha particles was used for both series of measurements. The spectrometer was calibrated for energy response and detector resolution using alpha particles from a $^{244}\text{Cm}$, $^{241}\text{Am}$ source. The observed energy resolution for $^{16}\text{O}$ at 20 MeV was 105 keV. The 20 keV resolution observed for the $^4\text{He}$ particles is slightly worse than normally employed for RBS measurements. Since the emphasis of the study was on the heavy-ion studies, no attempt to improve the resolution below this value was made. However, in the calculated comparisons between the result of the two measurements, a more realistic resolution of 15 keV is assumed for the $^4\text{He}$ case.

The incident particle beams were collimated to a 1.5 mm diameter spot size with an angular spread of less than 1°. The detector was placed at a scattering angle of 170° and collimated to a solid angle of 2.7 msr for all measurements. Typical beam currents ranged from 30 – 100 nA and a total charge between 100 and 400 µC was normally accumulated. Pulse height data were accumulated in a 1024 channel analyzer and stored on cassette tape for subsequent plotting and analysis.

Results and Discussion

Figure 2 shows backscattering spectra obtained using both 1.5 MeV $^4\text{He}$ and 20 MeV $^{16}\text{O}$ ions incident on a sample consisting of a 1000 Å gold layer deposited on a single crystal germanium substrate. The data have been adjusted such that full range on the abscissa corresponds to the full energy of the
incident beam for either projectile. The increased separation between the upper edge of the gold distribution and the germanium edge clearly demonstrates the improved mass-resolving power of heavy ions. The width of the scattering distribution associated with the gold layer is proportional to the projectile energy loss and the width of the deposited layer.

The improved mass resolution is illustrated in Fig. 3 which shows spectra obtained with thin (~50 Å) films of elemental copper and silver deposited on carbon substrates. The doublet structure due to the natural stable isotopic composition is clearly evident in each case. The measured mass resolution is consistent with the calculated curves shown in Fig. 1(b). Conventional \(^4\)He backscattering would not produce a doublet structure even in the more favorable case of \(^{63,65}\)Cu. Data obtained from these and other elemental films have been used to determine absolute scattering cross sections for \(^{16}\)O ions. Agreement with the calculated Rutherford scattering cross section is within 10% for most cases which is consistent within the present experimental errors.

The energy loss distributions for the gold layer which are shown in Fig. 2 also illustrate the increased depth range which can be analyzed with HIRBS. The energy loss distribution associated with the gold layer has a smaller energy width in the oxygen backscatter spectrum relative to the helium. This fact, combined with the increased separation due to the improved mass resolution, results in an increase in the maximum thickness of a gold layer which can be measured without interfering with the germanium substrate signal. This same argument applies for implanted layers and represents an inherent advantage of HIRBS for all types of samples. However, it should be pointed out that as the thickness of the layer is increased, the edge at the low energy side of the distribution becomes broadened due to straggling in the energy loss process. This effect is more severe for the case of heavy ions and results in a degradation in depth resolution at the larger thickness.\(^5\)
The application of the method to an ion-implanted gallium-arsenic sample is illustrated in Fig. 4. The sample was prepared by implanting $10^{16}$ atoms/cm$^2$ of isotopically separated $^{130}$Te into a crystalline gallium-arsenic substrate. The implantation energy was 400 keV corresponding to a projected range $R_p$ and straggling width $\Delta R_p$ of approximately 970 Å and 490 Å respectively. Analysis of the HIRBS data in Fig. 4 using semi-empirical energy loss data for $^{16}$O in gallium-arsenic yields experimental values of $R_p = 1097$ Å and $\Delta R_p = 529$ Å$^6$. This agreement is excellent considering the preliminary nature of the measurements and the large uncertainties associated with the literature values for dE/dx. Similar agreement has been obtained for tellurium implants performed at other implantation energies and fluxes.

The use of an isotopically separated implanted species was dictated by the desire to have an unambiguous distribution to deconvolute from the observed HIRBS spectra. Because of the mass resolution achieved with heavy ions, an element with multiple isotopes will be at least partially resolved and the observed distribution must be corrected for the broadened shape of the thin layer response. For example, the two most prominent isotopes of tellurium, $A = 128$ and $A = 130$ are separated by a backscattered energy equivalent to 100 Å of depth in the gallium-arsenic spectrum.

Measurements performed on epitaxially-grown layers of tin-doped gallium-arsenic have likewise been complicated by the large number of tin isotopes which are present in the naturally occurring elemental material. In cases where the use of separated isotopes is impractical, careful interpretation of the HIRBS results will require more elaborate deconvolution techniques than normally employed.

From data obtained with varying implant energies and dopant concentrations it is possible to measure the minimum detectable limit for the detection of
tellurium in gallium-arsenic. Using the present experimental configuration, we estimate a minimum detectable amount of \(3 \times 10^{14}\) atm/cm\(^2\) as compared with \(1 \times 10^{14}\) atm/cm\(^2\) for \(^4\)He using the result of Ref. 1. Since the HIRBS data are not at present count-rate limited, improved sensitivities could be obtained by increasing the geometry efficiency of the detector using an annular detector positioned around the beam axis. A counting efficiency increase of 20 times could easily be achieved with no effective loss in energy resolution for the case of heavy-ion detection.

A summary of the comparison data between \(^{16}\)O and \(^4\)He is given in Table I for the use of several impurity substances in two representative substrates, germanium and silicon. The depth resolution was calculated using the surface approximation for the energy loss factor\(^1\) and assumes an energy resolution of 100 keV for \(^{16}\)O and 15 keV for \(^4\)He. The depth resolution will worsen as a function of depth due to energy loss straggling, particularly for the heavy ions. We conclude that the depth resolution under these conditions are comparable for the two ion beams except for the case of very light or very heavy materials where the two methods exhibit their respective advantages. The accessible depth is significantly greater for heavy ions particularly for the larger mass dopant where the advantage is as great as a factor of five. The sensitivity to small quantities of impurities or minimum detectable limit is comparable for the two methods for the cases studied.

These advantages together with the improved mass resolution discussed in reference to Fig. 1 can be exploited in the analysis of samples in several areas of interest. These include measurements on thin film structures involving medium to high atomic number materials (\(Z > 50\)), depth profiles of implanted or diffused layers particularly in heavier, compound semiconductors, and applications to a wide variety of metallization related problems.
Acknowledgements

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References


Figure Captions

Fig. 1. a) Kinematic factor vs. target mass for $^4$He and $^{16}$O projectiles calculated for 170° scattering angle. b) Mass resolution vs. target mass as calculated from curves in Fig. 1a) assuming $\Delta E = 20$ keV for $^4$He and $\Delta E = 100$ keV for $^{16}$O.

Fig. 2. Comparison of experimental spectra obtained from a 1000 Å gold layer on a single-crystal germanium substrate using $^4$He and $^{16}$O. The full-scale abscissa corresponds to the incident beam energy in each case.

Fig. 3. Spectra obtained from 50 Å layers of natural copper and silver using 20 MeV $^{16}$O as the scattered projectile.

Fig. 4. Backscattered spectrum using 20 MeV $^{16}$O ions scattered from a gallium-arsenic sample which had been implanted with $^{130}$Te at a concentration of $10^{16}$ atoms/cm$^2$ using an implant energy of 400 keV. The projected range and layer width were 1490 Å and 490 Å respectively.
TABLE I. Summary of the Comparison Data Between $^{16}O$ and $^4He$

<table>
<thead>
<tr>
<th></th>
<th>ACCESSIBLE DEPTH (μm)</th>
<th>DEPTH RESOLUTION (Å)</th>
<th>MINIMUM DETECTION AMOUNT (Atoms/cm$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>4He $^*$</td>
<td>16O $^{**}$</td>
<td>4He</td>
</tr>
<tr>
<td>P → Si</td>
<td>0.14</td>
<td>0.3</td>
<td>320</td>
</tr>
<tr>
<td>As → Si</td>
<td>1.0</td>
<td>2.5</td>
<td>300</td>
</tr>
<tr>
<td>Sb → Si</td>
<td>1.16</td>
<td>3.4</td>
<td>290</td>
</tr>
<tr>
<td>Bi → Si</td>
<td>1.35</td>
<td>4.1</td>
<td>290</td>
</tr>
<tr>
<td>As → Ge</td>
<td>0.01</td>
<td>0.04</td>
<td>260</td>
</tr>
<tr>
<td>Sb → Ge</td>
<td>0.24</td>
<td>0.9</td>
<td>250</td>
</tr>
<tr>
<td>Bi → Ge</td>
<td>0.4</td>
<td>1.5</td>
<td>250</td>
</tr>
</tbody>
</table>

$^*$E$_0$ = 2.0 MeV, $\Delta E$ = 15 keV

$^{**}$E$_0$ = 20 MeV, $\Delta E$ = 100 keV
Figure 1

KINEMATIC FACTOR
\( \theta = 170^\circ \)

- \( ^4\text{He} \)
- \( ^{16}\text{O} \)

1.5 MeV \(^4\text{He} \)
\( \Delta E = 20\text{keV} \)

20 MeV \(^{16}\text{O} \)
\( \Delta E = 100\text{keV} \)
Figure 2

Scattered intensity ($\theta = 170^\circ$)

1.5 MeV $^4\text{He}$

20 MeV $^{16}\text{O}$

Relative energy ($E/E_0$)
Figure 4
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