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Stopping powers of GaAs for 0.3–2.5 MeV ^{1}H and ^{4}He ions

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Abstract

Ion backscattering and foil transmission methods have been used to determine the energy loss of 0.3–2.5 MeV ^{1}H and ^{4}He ions in crystalline bulk GaAs and thin foil GaAs grown by molecular beam epitaxy (MBE). The self-supporting GaAs sample foil was produced by floating the MBE-grown GaAs film from an AlAs/GaAs backing by a lift-off process. The stopping powers, corresponding to energy loss of the ions in a nonchanneling direction in the crystal were extracted from the measurements. Ion backscattering and channeling were employed to study the effect of the crystal structure of the thin foil sample on the stopping powers deduced. Deviations from semi-empirical SRIM calculations (version 96.04) were observed in the case of ^{4}He ions for which the stopping powers fall clearly below the calculated values. An average deviation of about 5% is found for the energies studied, from below the stopping power maximum at 0.8 to 2.2 MeV. The results obtained by the two independent experimental methods have been compared and discussed.

1. Introduction

Accurate stopping power values are required in the processing of semiconductors by ion beam implantation as well as in the structural characterization of semiconductor materials by ion beam methods. For example, the depth scales of the samples implanted or characterized by ion beam techniques are based exclusively on the slowing down of ions in the sample material.

Ranges of ^{1}He and ^{4}He ions in GaAs have been investigated in our previous publications [1,2]. No stopping powers for the ions and energies used in the present study can be found in the literature.

In this work, deviations as large as 5–8% were observed in the slowing down of ^{4}He ions in GaAs in the typical energy range of Rutherford backscattering spectrometry (RBS) in comparison to commonly used semi-empirical stopping power calculations. Corresponding errors in the depth scale result, e.g., from RBS analysis of GaAs samples when these calculations are applied.

To verify the systematic deviations of our data from the calculations, two independent methods, the transmission and backscattering methods for determining the stopping powers were used. In the former technique, the stopping is extracted from the energy loss of ions in a sample foil of known thickness, while the latter relies on backscattering yield from a bulk sample and the known stopping power of a reference material.

2. Experimental

2.1. Sample preparation

The GaAs film samples for the transmission measurements were grown by the molecular beam epitaxy (MBE) technique at the Department of Physics of the Tampere University of Technology. A 0.1 μm thick AlAs buffer layer was grown on a 600 μm bulk (100) GaAs sample. GaAs films, typically of 1–2 μm thickness, were then grown on the buffer layer. To prepare thin self-supporting GaAs foils from these samples, a lift-off technique [3,4], was applied. The technique has been modified in our laboratory by experimenting with different etching liquids, omitting the wax support and by testing several etching times. Hydrofluoric acid was chosen as the etching liquid. An etching selectivity of the order of 10^{7} between the AlAs release layer and $Al_{0.4}Ga_{0.6}As$ has been observed, with the onset of etching occurring very suddenly between 40% and 50% Al composition [4].

Sample foils of two different thicknesses were mounted on glass frames with an aperture of 4 mm in diameter. The thicknesses of the GaAs foils on the glass frames were determined by an absolutely calibrated Dektak IIA profilometer from the beam spot area used in the transmission measurements. Thicknesses of 1.00 ± 0.01 and 1.96 ± 0.01 μm for the samples used in the energy loss measurements

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were obtained. The stability of the foils in the ion beam exposure was verified by performing repeated energy loss measurements.

The mass density of the sample material is required in unit conversion from energy loss per μm to energy loss per mg/cm^2. Calculations comparing the signal widths of the GaAs foil from ^{1}H ion backscattering spectra and the thickness measurements by the profilometer yielded a mass density of 5.22 ± 0.03 g cm^{-3} for the MBE-grown etched GaAs foils, a value close to that found in the literature, viz. 5.318 g cm^{-3}. The experimental mass density value was used in the calculations for transmission measurements using the self-supporting GaAs foils.

The pure single crystal GaAs samples for the backscattering measurements were undoped (100), 2° off-axis wafers, supplied by Outokumpu Semitronic Ab.

2.2. Measurements

The ^{1}H and ^{4}He beams were obtained from the 2.5 MV Van de Graaff accelerator of the University of Helsinki. The energy calibration of the beam analyzing magnet was based on the well known ^{15}N(p,αγ)^{12}C, ^{27}Al(p,γ)^{28}Si and ^{13}C(p,γ)^{14}N resonance reactions at $E_p = 429$, 992, and 1747 keV, respectively.

The experimental arrangement used in the transmission measurements is described in Ref. [5]. In brief, the sample foil was interposed into the ion beam scattered from a gold target in front of the detector. The most probable energy loss of the ions transmitted through the foil was obtained by subtracting the energy of the gold scattering signal from that measured without the foil. A silicon surface barrier detector (50 mm^2, 100 μm), with a collimating aperture of 2 mm in diameter and a detection solid angle of 0.07 mSr was positioned at a scattering angle of 135° for the transmission measurements. The GaAs foil was tilted 6° from the normal of the foil surface.

In the backscattering and channeling measurements the standard scattering equipment of the laboratory was used [6]. Collimating slits and apertures were used to limit the beam spot size on the target to 0.5 mm in diameter and the beam angular divergence to 0.04°.

In the backscattering technique for determining the stopping powers, the relative heights of the high energy edges of the GaAs signal and of the Cu signal from a copper reference sample were measured. The use of a reference material eliminates the need of an absolutely calibrated experimental setup and related errors. Copper was chosen partly because of having almost similar mass as GaAs and because of the availability of recent experimental Cu stopping power values that were considered reliable. A beam chopper with a separate pulse analysis system, with an accuracy of better than 2%, was used to measure the relative ion doses for the backscattering spectra of GaAs and Cu. The backscattering experiments were performed by tilting the sample surface normal (⟨100⟩ axis) 5° off the beam direction and rotating the sample continuously around the axis perpendicular to the sample surface during measurement to obtain a nonchanneling orientation of the beam in the sample. The channeling setup was equipped with a three-axis goniometer with an angular resolution of 0.04°. The backscattering data were taken at an angle of 170° and using a detection solid angle of 10.7 mSr. The energy resolution of the detecting system was 14 keV for ^{4}He ions at 2.0 MeV.

The detection system featured a linear energy dependence for ^{1}H ions and for ^{4}He ions above 600 keV. Below $E_{He} = 600$ keV, the nonconstant detector response was determined by measuring the backscattering edge positions from thick Au and C targets as a function of energy.

3. Results

From the transmission energy loss data the stopping powers at the mean ion energies E_{av} in the foil were calculated by dividing the energy losses ΔE in the foil by the areal density $\rho\Delta x$ (ρ stands for mass density, Δx is the foil thickness, and $E_{av} = E - \Delta E/2$, where E = incident energies). To account for the nonlinear dependence on ion energy of stopping powers, small corrections to the mean energies E_{av} were applied.

The corrections were calculated by comparing the measured foil thickness Δx_{exp} to one obtained from the integral

$$\Delta x_{calc} = \int_{E_{in}}^{E_{out}} dE/S(E_{av}),$$

where $S(E_{av})$ is a fit to the experimental data, E_{in} and E_{out} are the ion energies before and after the foil and Δx_{calc} is the calculated thickness. The following formula [7,8] for calculating the stopping powers $S(E)$ was adopted:

$$\begin{aligned} S(E) &= (S_{low} S_{hi})/(S_{low} + S_{hi}), \\ S_{low} &= C_1 E^{C_2} + C_3 E^{C_4}, \\ S_{hi} &= \left(C_5/E^{C_6}\right)\ln\left((C_7/E) + C_8 E\right), \end{aligned} \tag{1}$$

where E is the ion energy and C_i ($i = 1,2,\ldots,8$) are fitting parameters. The task is to vary E_{av} and $S(E_{av})$ until corrected energies E_{eff} are found for which $S(E_{eff}) = (E_{in} - E_{out})/\Delta x_{exp}$. This correction procedure is repeated until the E_{av} values converge to constant values E_{eff}. As a result, the stopping powers, $S = -(1/\rho)(dE/dx)$ (differential energy loss per unit path length), are taken as $\Delta E/\rho\Delta x$ at effective ion energies, E_{eff}.

In the backscattering method, the stopping cross section factors $[\varepsilon]_{GaAs}$ of GaAs were then calculated relative to those of copper from the leading edge heights of the GaAs and Cu backscattering signals. A method introduced by Warters [9,10] was used for deducing the stopping powers from the cross section factors.

The reference stopping powers for copper have been calculated from earlier experimental data in detail in Ref. [11]. In brief, a function of the form [12] $\varepsilon^{-1} = a_0 +$ $a_1 E^{-0.4} + a_2 E^{0.25} + a_3 E^{0.8}$ was used to fit the data and to calculate the copper stopping powers. For hydrogen ion energies below 1 MeV the fitting coefficients a_i from Paul et al. [13] (1% accuracy of the fit) were adopted. Above 1 MeV hydrogen energy, fitting the data points from Refs. [14,15] yielded the coefficients $a_0 = -2.5899 \times 10^{-1}$, $a_1 = 1.3907$, $a_2 = 4.0561 \times 10^{-2}$ and $a_3 = 9.2971 \times 10^{-5}$ (accuracy 1%, ε in units eV/10^{15} at/cm^2). For helium ions, the coefficients $a_0 = -1.1197 \times 10^{-2}$, $a_1 = 2.2068 \times 10^{-1}$, $a_2 = 9.4920 \times 10^{-4}$ and $a_3 = 2.0893 \times 10^{-5}$ (accuracy 2.5%) were derived from the data of Refs. [16,17].

Table 1
Measured values of the stopping powers of GaAs for ^{1}H and ^{4}He ions by the transmission method

Energy [keV]	^{1}H [MeVcm^2/mg]	Energy [keV]	^{4}He [MeVcm^2/mg]
348	0.172 [a]	264	0.546 [a]
552	0.143 [a]	283	0.558 [a]
753	0.125 [a]	356	0.597 [a]
920	0.122 [b]	359	0.616 [a]
954	0.107 [a]	422	0.636 [b]
1021	0.115 [b]	444	0.630 [a]
1121	0.112 [b]	487	0.639 [a]
1154	0.096 [a]	516	0.651 [b]
1224	0.103 [b]	521	0.673 [a]
1326	0.097 [b]	529	0.649 [a]
1351	0.096 [a]	572	0.649 [a]
1426	0.094 [b]	594	0.654 [b]
1526	0.090 [b]	607	0.661 [a]
1550	0.084 [a]	635	0.671 [a]
1624	0.090 [b]	826	0.676 [a]
1724	0.087 [b]	865	0.670 [a]
1747	0.084 [a]	866	0.671 [a]
1825	0.081 [b]	886	0.669 [a]
1924	0.081 [b]	920	0.665 [a]
1946	0.072 [a]	954	0.674 [a]
2024	0.078 [b]	958	0.668 [a]
2122	0.078 [b]	1002	0.657 [a]
2143	0.072 [a]	1045	0.661 [a]
2222	0.075 [b]	1064	0.656 [b]
2322	0.072 [b]	1089	0.659 [b]
2341	0.066 [a]	1103	0.653 [b]
2421	0.069 [b]	1137	0.650 [a]
		1138	0.650 [a]
		1173	0.645 [b]
		1256	0.639 [b]
		1324	0.638 [a]
		1327	0.634 [a]
		1346	0.630 [b]
		1439	0.622 [b]
		1515	0.617 [a]
		1517	0.616 [a]
		1533	0.616 [b]
		1628	0.610 [b]
		1705	0.604 [a]
		1706	0.605 [a]
		1724	0.602 [b]
		1823	0.591 [b]
		1896	0.585 [a]
		1899	0.581 [a]
		1920	0.582 [b]
		2016	0.576 [b]
		2087	0.576 [a]
		2088	0.567 [a]

[a] Data derived from 1 μm thick foils.
[b] Data derived from 2 μm thick foils.

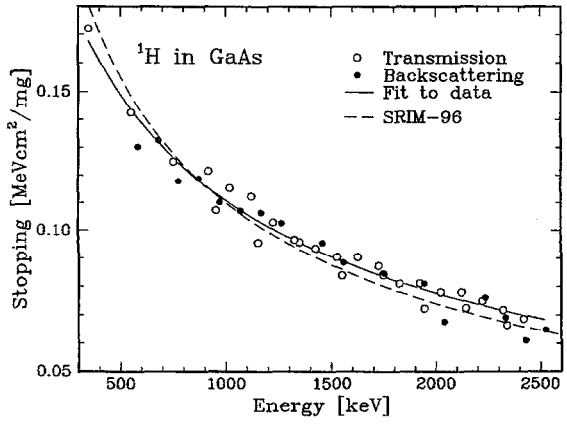

Fig. 1. Stopping powers of GaAs for ^{1}H ions as measured by the foil transmission and ion backscattering methods. Fits to the measured transmission and backscattering stopping data (see text) are shown by the solid curve. The dashed curve represents the semi-empirical calculations by the SRIM-96 code.

Table 2
Measured values of the stopping powers of GaAs for ^{1}H and ^{4}He ions by the backscattering method

Energy [keV]	^{1}H [MeVcm^2/mg]	Energy [keV]	^{4}He [MeVcm^2/mg]
583	0.130	490	0.632
681	0.133	534	0.646
778	0.118	579	0.654
875	0.119	623	0.648
972	0.110	668	0.654
1070	0.107	757	0.670
1167	0.106	1068	0.657
1264	0.103	1336	0.629
1459	0.096	1603	0.600
1556	0.089	1870	0.585
1750	0.085	2137	0.549
1945	0.081		
2042	0.068		
2236	0.076		
2334	0.069		
2431	0.061		
2528	0.065		

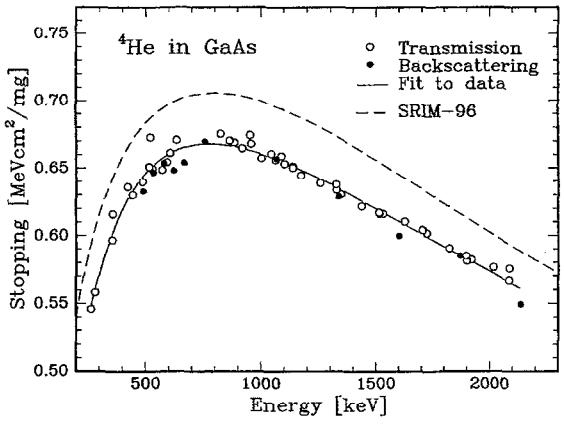

Fig. 2. Stopping powers of GaAs for ^{4}He ions as measured by the foil transmission and ion backscattering methods. The solid and dashed curves as in Fig. 1.

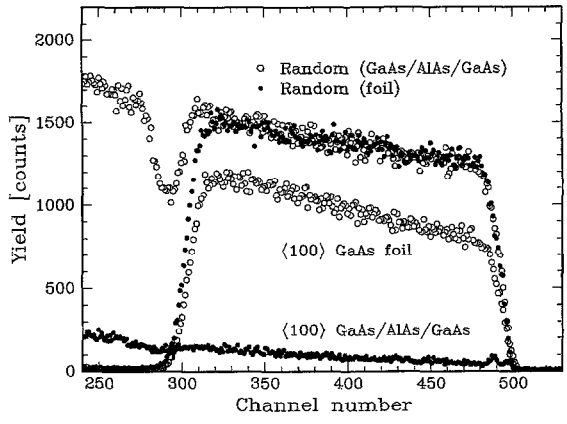

Fig. 3. Ion backscattering channeling spectra for ^{4}He ions incident along the ⟨100⟩ crystal axis on the MBE-grown GaAs film on the backing (⟨100⟩ GaAs/AlAs/GaAs) and the self-supporting foil used in the transmission measurements (⟨100⟩ GaAs foil). Also shown are the spectra taken by rotating the samples (random (GaAs/AlAs/GaAs) and random (foil)).

The GaAs stopping powers obtained by the transmission and backscattering methods for ^{1}H and ^{4}He ions are given in Tables 1 and 2, and illustrated in Figs. 1 and 2. For protons the uncertainties of the stopping powers fall below 3.0% and 3.5% for the transmission and backscattering data, and below 2.0% and 4.0% for helium ions, respectively. In the transmission experiments the major sources of possible errors include the determination of the energy losses, signal edge positions and foil thicknesses. In the backscattering experiments, the possible errors are due to the uncertainties in the stopping powers of the reference material and in obtaining the signal heights for the sample and reference targets. The better accuracy obtained by the transmission technique follows because no reference samples are needed, helium ions show better results than hydrogen because of the larger relative energy loss in the foil.

The ^{1}H and ^{4}He data, taken with the two independent methods are found consistent within experimental accuracy. Best fits to the data by using Eq. (1) are shown in Figs. 1 and 2, the fitting parameters C_i are given in Table 3.

Table 3
The parameters C_i of Eq. (1) fitted to the experimental data. Both backscattering and transmission data points were used

Parameters	^{1}H 400–2500 keV	^{4}He 300–2100 keV
C_1	−4.96687	−19.33131
C_2	90.38460	−0.57604
C_3	−406.36035	506.11796
C_4	−50.09812	−324.30524
C_5	12.08019	32.66547
C_6	0.83178	0.83488
C_7	11.31814	61.44826
C_8	0.01749	0.00978

4. Discussion

The crystal quality of the etched MBE-grown samples was investigated by backscattering channeling measurements. In Fig. 3 two spectra are compared, taken with the samples aligned with ⟨100⟩ crystal axis: a spectrum for 2.0 MeV ^{4}He ions incident on the MBE-grown GaAs film on the backing before the lift-off process and a spectrum obtained from the etched self-supporting foil used in the transmission measurements. Also shown are the spectra taken by tilting and rotating the samples during the measurement to ensure a nonchanneling orientation. It may be concluded that the crystal structure is significantly affected by the lift-off process used for producing the self-supporting foil. The minimum yields, defined as the ratio of the aligned yield to the yield obtained in random orientation in the near-surface region, are found as $\chi_{min} = 0.64$ for the 1 μm and $\chi_{min} = 0.66$ for the 2 μm self-supporting foils used in the measurements, respectively. The high minimum yields indicate large amounts of residual damage in the crystal structure.

When comparing the spectra in Fig. 3, taken from the self-supporting 1 μm foil in the aligned orientation to the spectrum measured by tilting and rotating the sample (nonchanneling orientation), no difference in the signal widths can be observed. By detailed analysis of the spectra for the 1 and 2 μm foils, the widths are found identical to an accuracy better than 0.5%. This is a definite evidence of the same energy loss along the aligned and nonchanneling orientations in our self-supporting GaAs foils. This is

clearly due to the high amount of crystal disorder in the foils, our earlier findings [1,2] show, that for a perfect crystal, ^{1}H ion energy loss along the ⟨100⟩ crystal axis is almost similar to that in random direction, while the energy loss of ^{4}He ions is significantly smaller along the ⟨100⟩ axial direction than in the nonchanneling direction.

We thus conclude, that for both transmission and backscattering techniques used, the stopping powers obtained result from the slowing down of ions along a nonchanneled rather than channeled direction in the crystal lattice.

The present GaAs stopping powers may be compared with the ^{1}H and ^{4}H ion range data in Refs. [1,2]. Good consistency between the stopping data estimated from the earlier range values and the present results may be found. The ranges for ^{1}H ions between 1.0 and 2.4 MeV were found to agree well with the TRIM-92 [7] predictions. For ^{4}H ions between 1.0 and 2.7 MeV, the range data were found to clearly exceed the semi-empirical predictions in the low energy region, by 15% at maximum while at higher energies the difference becomes less pronounced, about 2–3%.

A comparison of the present stopping powers to semi-empirical stopping calculations by SRIM-96 [7], based on the ZBL-85 model by Ziegler, Biersack and Littmark [18] and Bragg's additivity rule [19], is also shown in Figs. 1 and 2. In the case of ^{1}H ions, there is a slight tendency of the data to exceed the semi-empirical predictions above about 1 MeV, while a converse behavior is observed below 1 MeV. For ^{4}H ions, the data systematically fall below the predictions, from about 8% at the lower end of our energy interval to about 3% at the highest energies studied. At the Bragg peak (about 0.8 MeV), the difference is 5%.

The deviations of the data from calculations are due to either different elemental stopping powers of Ga or As, or to the nonvalidity of Bragg's rule. Further experiments are required to assign the cause of deviations to any of the factors above.

The evaluation of stopping powers from solid samples by different ion beam techniques has been treated in general, e.g., in Refs. [20,21]. When the sample material under study has crystalline structure, rendering the fabrication of thin self-supporting foils troublesome, as in the case of, e.g., compound semiconductor materials, some special features must be taken into consideration. The choice of the experimental method is a compromise between the difficulties in sample preparation and the stopping power measurements and the accuracy of the results obtained. The inherently simple and accurate direct method of foil transmission, may be problematic due to crystal structure and film thickness. The channeling effects must be adequately avoided, this may involve a detailed analysis of the sample foil. The thicker the foil, the more the average ion energy in the foil deviates from the effective energy, especially near the Bragg peak. The backscattering method, on the other hand, where the stopping powers are derived from the height of the high energy edge of the signal, requires a minimal amount of sample preparation. This is, however, a more indirect method, requiring either an absolutely calibrated experimental setup or the use of a reference sample. Additional inaccuracies may result from the determination of the energy to which the stopping powers are to be assigned. Also here care must be taken to avoid channeling effects. Tilting and rotating the sample continuously during measurement is possibly the only reliable technique to simulate random orientation for crystalline samples.

The present measurements show that consistent stopping power data may be obtained by the independent methods of foil transmission and backscattering. In the present case the most accurate results were obtained by the transmission method, especially in the case of helium projectiles.

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