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Exfoliation process of InP caused by ^{1}H and ^{4}He ion implantation and its correlation with ion range parameters

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Abstract

Exfoliation of crystalline InP by 0.6–2.1 MeV ^{1}H and 1.0–2.6 MeV ^{4}He ion implantation at random orientation has been studied. The correlation between the depth of the produced craters and ion range parameters has been established by measuring the implanted ^{4}He ion depth and defect distributions by elastic ^{1}H backscattering and ^{4}He ion channelling methods, respectively. The measurements indicate that the produced crater depths correlate with maximum ion range values. The modal range values of the implanted ^{4}He ions were determined from the depth profiles obtained by the elastic proton backscattering technique. The crater depths deviate from the modal ranges by 9% to 3% for 1.0 to 2.5 MeV implanted ^{4}He ions. The deduced ranges are in good agreement with the simulated values obtained by the Monte Carlo calculations. © 1997 Elsevier Science B.V.

1. Introduction

New applications of high dose ^{1}H and ^{4}He ion implantation in semiconducting materials have been reported recently. The use of the so called Smart-Cut process in preparing thin silicon on insulator (SOI) wafers has been described by Bruel [1]. In this technique high dose hydrogen ion implantation is followed by sample annealing and the resulting process involves the basic mechanism of exfoliation which is well known. When gaseous elements are used in the implantation, gas filled bubbles are formed near the end of the projected range of the ions. With increasing concentration the density of the bubbles increases and eventually plastic deformation occurs. In another application the gas atoms are allowed to permeate out of the bubbles formed in the material by ^{1}H and ^{4}He ion implantation and empty cavities are left behind [2,3]. The formed cavities have been shown to function as good gettering sites for metallic impurities in the case of silicon [4–6]. This is expected to have significant importance in modern device processing. The use of the cavities for electrical isolation has been also considered [7]. For example, in these novel applications the knowledge of the exfoliation process and accurate range parameters of the implanted ions in semiconductor materials are of essence.

In our recent studies we have investigated the exfoliation process caused by MeV ^{1}H and ^{4}He ion implantation in GaAs [8,9]. The relation of the formed crater depths with the ion range parameters was established. The present work continues these studies applied to the compound semiconducting material of indium phosphide. The main emphasis is on the

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exfoliation process itself but also modal range values for ^{4}He ions in InP are determined. No previous range values have been reported in the literature. Recently the stopping powers of InP for ^{1}H and ^{4}He ions have been presented covering the energy region 20–180 keV [10] and for ^{4}He ions in the energy region 0.5–4.5 MeV [11,12].

2. Experimental

The ^{1}H and ^{4}He ion beams were obtained from the 2.5 MV Van de Graaff accelerator and the 4.5 MeV proton beam from the 5 MV EGP-II-10 tandem accelerator of the laboratory. Energy calibration of the VdG accelerator was achieved by employing the resonance reactions ^{27}Al (p, γ)^{28}Si at 991.9 keV and 1799.8 keV and ^{20}Ne(α, γ)^{24}Mg at 1929 keV. The n-type InP single crystal (100), 2° off-axis samples were doped with sulphur corresponding to a resistivity of 9.6×10^{-4} Ω cm.

The random direction implantations of ^{1}H and ^{4}He ions in InP were performed at room temperature. To avoid channelling effects the samples were tilted at 7.5° off beam direction and rotated around an axis perpendicular to the sample normal during the implantations. A Dektak IIA profilometer was used in the surface profile measurements. For the backscattering and channelling measurements an ion-implanted silicon detector was placed at the angle of 170°. In the channelling measurements a three-axis goniometer system was employed. The angular resolution of the goniometer system was 0.02° and the angular divergence of the beams was less than 0.02°.

3. Measurements and results

Implantation energies of the ^{1}H and ^{4}He ions were between 0.6–2.1 MeV (doses varied from 6×10^{16} to 2×10^{18} cm^{-2}) and 1.0–2.6 MeV (8×10^{15} to 8×10^{17} cm^{-2}), respectively. The implanted areas were about 0.10 cm^{2} and the beam currents were varied between 0.3–2.0 μA. A threshold value of the ion fluence of 2 μA cm^{-2} was observed for the exfoliation process to take place.

To study the correlation of the obtained crater depth with the ^{4}He ion depth distribution, the implanted helium profiles were determined for several implantation energies by 2.7 and 4.5 MeV elastic proton backscattering. The details of the application of the ^{4}He(p, p)^{4}He elastic scattering to helium depth profiling may be found in Ref. [8]. As an example, the helium concentration profile of the 1.4 MeV ^{4}He ion implantation is presented in Fig. 1. The corresponding crater depth value is 4.7 μm. According to the measured helium profiles it may be concluded that the crater depths are well correlated with the ion maximum ranges. The simulated ion and deposited energy distributions from Monte Carlo calculations [13] are presented for comparison in Fig. 1. It may be noted that the calculated profile agrees well with the measured profile.

As the calculation of the experimental depth profiles, such as shown in Fig. 1, rely on the semiempirical proton stopping powers [14] for InP, further measurements were conducted to confirm the crater depth-ion range relation. The crystal structures and defect distributions below the surfaces of craters were studied by the Rutherford backscattering and channelling technique using 2.0 MeV ^{4}He ions. The spectra obtained for random and ⟨100⟩ axial directions are presented in Fig. 2 for the 0.6 MeV ^{1}H and 1.4 MeV ^{4}He ion implanted InP samples. The ex-

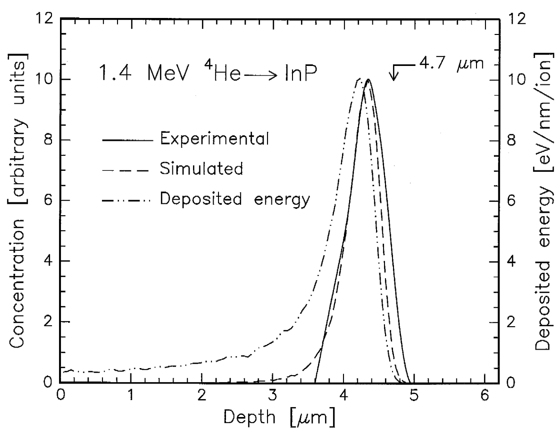

Fig. 1. Depth distribution of 1.4 MeV ^{4}He ions implanted in InP at random orientation. The experimental profile is shown by the solid line. The corresponding measured crater depth is indicated by the arrow. The simulated range (dashed) and deposited energy distributions (dashdotted curve) are from Monte Carlo calculations.

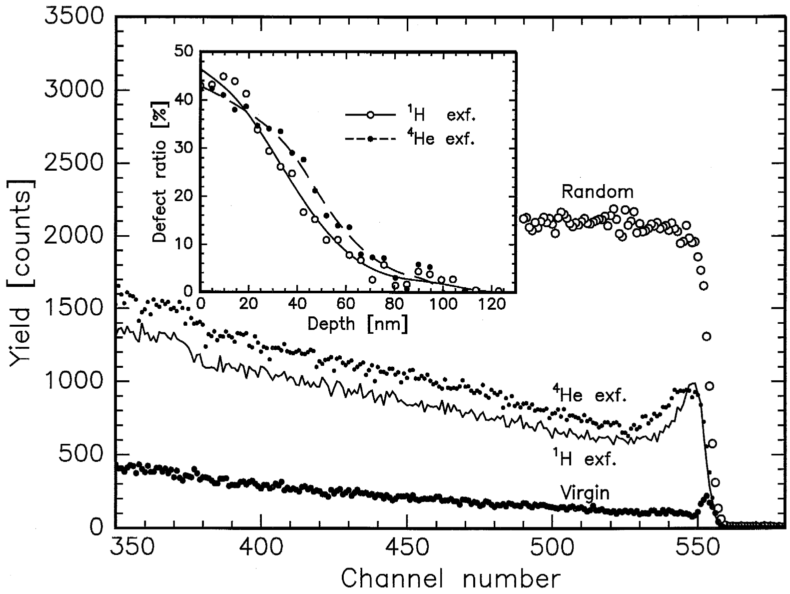

Fig. 2. Random and channelling spectra (⟨100⟩ axial direction) obtained from the bottom of ^{1}H and ^{4}He induced craters by 2.0 MeV ^{4}He Rutherford backscattering. In the insert, the deduced defect distributions are shown for the 0.6 MeV ^{1}H and 1.4 MeV ^{4}He ion implanted InP.

tracted defect depth profiles are shown in the insert. The profile for the ^{4}He implanted sample may be now compared with the depth profile of Fig. 1. The fact that defects are found only about 50 nm below the crater surface shows that the crater extends to almost the end of the ion range.

Further evidence supporting our conclusions was obtained by measuring the helium concentration pro-

Table 1
Measured and simulated modal ranges and crater depth values for ^{4}He and ^{1}H ions in InP

^{4}He ions				^{1}H ions		
Energy (MeV)	Ion range (μm)	Simulation (μm)	Crater depth (μm)	Energy (MeV)	Simulation (μm)	Crater depth (μm)
1.0		3.2	3.4	0.6	6.3	6.6
1.1	3.3	3.5	3.6	0.7	7.8	8.0
1.2		3.8	3.7	0.8	9.5	10.0
1.3		4.1	4.3	0.9	11.2	11.8
1.4	4.3	4.3	4.7	1.0	13.0	13.5
1.5		4.6	4.8	1.1	15.0	15.4
1.6		4.9	5.1	1.2	17.1	17.8
1.7	5.1	5.3	5.5	1.3	19.2	19.0
1.8		5.5	5.9	1.4	21.6	21.2
1.9		5.9	6.2	1.5	24.0	23.4
2.0	6.3	6.2	6.6	1.6	26.4	26.3
2.1		6.5	6.9	1.7	28.8	30.1
2.2		6.9	7.1	1.8	31.7	31.6
2.3		7.2	7.4	1.9	34.4	34.3
2.4		7.6	7.8	2.0	37.5	37.2
2.5	8.0	8.0	8.2	2.1	40.3	39.9
2.6		8.4	8.5			

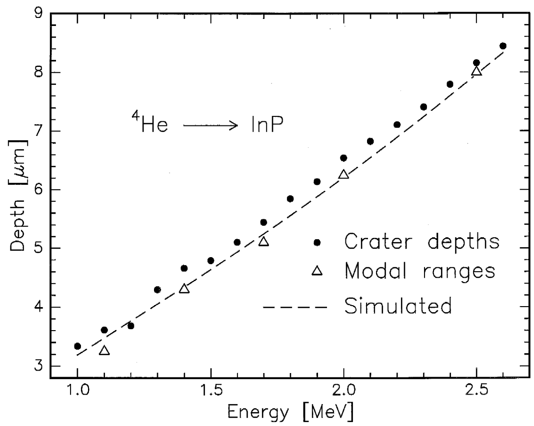

Fig. 3. The measured modal ^{4}He ion ranges and crater depths in InP. Simulated range values were obtained by Monte Carlo calculations.

files by the ^{4}He(p, p)^{4}He reaction from the surfaces of the craters formed during the ^{4}He ion implantation. In the measurements of the 1.1 and 1.7 MeV ion implanted samples about 70 nm thick helium distributions were observed at the crater surfaces immediately after the exfoliation and without breaking the vacuum. This result also supports our crater depth versus ion range calculations.

The modal ^{4}He ion ranges obtained from the measured depth profiles are given in Table 1 together with the simulated range values obtained by Monte Carlo calculations. In the simulations a density value of 4.79 g/cm^3 for InP has been adopted from Ref. [15]. In determining the profile for the 2.5 MeV helium implanted sample, protons with energy of 4.5 MeV were used. The corresponding crater depths are also given in Table 1 and the data for ^{4}He ions is presented in Fig. 3. The present experimental modal range values are in good agreement with the values obtained by Monte Carlo simulations. The crater depths systematically exceed the range values by 9 to 3% for 1.0 to 2.6 MeV implanted ^{4}He ions.

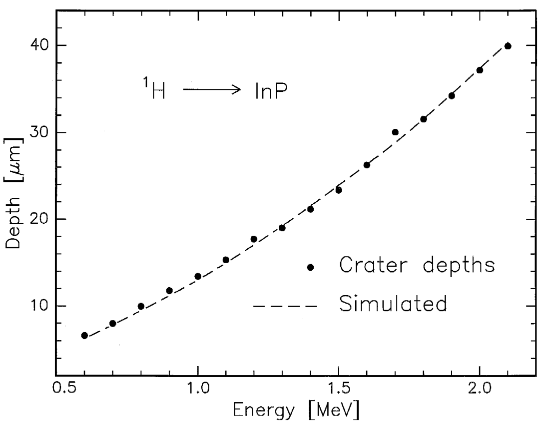

Fig. 4. The crater depths produced in InP by proton bombardment and the corresponding simulated modal range values obtained by Monte Carlo calculations.

In the case of ^{1}H ion implanted InP the obtained crater depth values and the calculated modal range values obtained by Monte Carlo calculations are given in Table 1 and presented in Fig. 4. Taking into consideration the magnitude of the range straggling of the implanted ^{1}H ions (0.5 to 2.15 μm for 0.6 to 2.0 MeV) it may be assumed that the present crater depth values relate to modal range values with an uncertainty below ± 4%.

4. Discussion

In studying the exfoliation process several noteworthy features were observed. In most cases the samples exfoliated directly during the irradiations but sometimes an annealing at a temperature of 400°C in a vacuum tube furnace for a few minutes was necessary. This did not have any effect on the obtained crater depth. In addition, the effects of the beam current and fluence during implantation on the exfoliation process were studied. It was found that if the fluence was kept below about 2 μA cm^{-2} the exfoliation did not occur even for a total dose of 10 mC and after annealing at elevated temperatures. This is could be related to slow permeation of the gas during implantation from the crystal material which thus prevents blistering.

Direct experimental verification of the crater depth versus range parameter relation of the implanted protons is in principle possible by ion beam techniques. For example, the reaction ^{1}H(^{15}N, $\alpha\gamma$)^{12}C or elastic recoil detection analysis (ERDA) can be employed together with etching techniques or taking advantage of the exfoliation phenomenon as in our previous study [16]. Due to the uncertainties associated with these techniques, e.g., the rather inaccurately known stopping powers of InP for ^{15}N ions, possible surface roughness disturbing in ERD analysis, the depth-accuracy achievable by these techniques is not sufficient. However, according to the defect profile measurements as shown in Fig. 2, it must be concluded that the exfoliation of proton bombarded InP occurs in similar way as helium ion bombarded InP.

The good agreement of the present modal range values with Monte Carlo simulations (see Fig. 3) indicates that the stopping powers of ^{4}He ions in InP do not deviate significantly from the semiempirical predictions used in the calculations [14]. At least in the energy region 1.0–2.6 MeV, this is in disagreement with the results of Ref. [12] where a correction factor of 1.10 was obtained for the semiempirical stopping powers. Furthermore, the correction factor was found constant in the studied energy range of 0.5 to 4.5 MeV. Even if the experimental range values for ^{1}H ions were not obtained in the present work, the systematical behavior of the crater depths versus the simulated modal ranges indicates that also in the case of protons the semiempirical stopping powers are in good agreement with experimental values.

The uncertainty in the crater depth values obtained arise from the uncertainties in the profilometer measurements and from the nature of the exfoliation process itself. Uncertainties of about 5% and 4% may be assigned to the depth values given for ^{4}He and ^{1}H ions, respectively. The uncertainty in the measured ^{4}He ion range values consist of the proton stopping power inaccuracy for InP and spectrum data analysis yielding an overall uncertainty of about 4%.

In this work the exfoliation process of InP caused by 0.6 to 2.6 MeV ^{4}He and ^{1}H ions has been studied. From depth profile and defect distribution measurements it could be concluded that the exfoliation takes place at depths close to the maximum ion range rather than at the concentration maximum. The behavior of InP is in this respect different to that of GaAs. In our previous study [9] GaAs was observed to exfoliate under controlled conditions systematically from the concentration maximum, corresponding thus well with modal ion range values. The difference in the behavior may be due to material hardness. GaAs is about 1.5 times harder than InP [17] and thus needs a higher pressure for the exfoliation to take place.

In conclusion, the observed complexity of the exfoliation process should be taken in account when applying it to range determinations and possible technological processes.

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References

[1] M. Bruel, Nucl. Instr. Meth. B 108 (1996) 313.
[2] J. Wong-Leung, C. Ascheron, M. Petravic, R.G. Elliman, J.S. Williams, Appl. Phys. Lett. 66 (1995) 1231.
[3] C.C. Griffioen, J.H. Evans, P.C. de Jong, A. Van Veen, Nucl. Instr. Meth. B 27 (1987) 417.
[4] S.M. Myers, D.M. Follstaedt, J. Appl. Phys. 79 (1996) 1337.
[5] J. Wong-Leung, J.S. Williams, E. Nygren, Nucl. Instr. Meth. B 106 (1995) 424.
[6] J. Wong-Leung, J.S. Williams, Nucl. Instr. Meth. B 118 (1996) 34.
[7] C.H. Seager, S.M. Myers, R.A. Anderson, W.L. Warren, D.M. Follstacdt, Phys. Rev. B 50 (1994) 2458.
[8] J. Räisänen, E. Rauhala, Nucl. Instr. Meth. B 93 (1994) 1.
[9] E. Rauhala, J. Räisänen, Nucl. Instr. Meth. B 94 (1994) 245.
[10] S.R. Lee, R.R. Hart, Nucl. Instr. Meth. B 28 (1987) 470.
[11] D.W. Hetherington, Nucl. Instr. Meth. B 90 (1994) 84.
[12] D.W. Hetherington, Nucl. Instr. Meth. B 115 (1996) 319.
[13] J.F. Ziegler, SRIM-96 computer program, private communication.
[14] J.F. Ziegler, J.P. Biersack, U. Littmark, The Stopping and Range of Ions in All Elements, Pergamon, New York, 1985.
[15] Properties of Indium Phospide, INSPEC, IEE, London, 1991.
[16] T. Ahlgren, K. Väkeväinen, J. Räisänen, E. Rauhala, J. Keinonen, Appl. Surf. Sci. 90 (1995) 419.
[17] Indium Phospide: Crystal Growth and Characterisation, Semiconductors and Semimetals, Vol. 31, Academic Press, New York, 1990, p. 225.