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Hall-effect measurements in Cd-implanted GaAs[†]

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Cadmium ions were implanted in GaAs crystals at 135 keV to doses ranging from 10^{12} to 10^{16} ion/cm² at room temperature. Sheet-resistivity and Hall-effect measurements were carried out as a function of temperature, 4.2–300°K, after annealings at 700, 800, or 900°C in an Ar ambient. The sample surfaces were protected with pyrolytically deposited Si₃N₄. Significant *p*-type conduction was observed when samples with doses $\geq 10^{13}$ cm⁻² were annealed at $\geq 700^\circ\text{C}$. For doses below 10^{14} cm⁻² nearly complete electrical activity was attained after an 800–900°C anneal. The Cd profiles were determined by differential Hall-effect measurements in conjunction with the acid-etch layer-removal technique. Above about 800°C diffusion becomes important and significantly flattens the implantation profile.

PACS numbers: 72.20.M, 61.80.P, 72.80.E, 81.20.M

I. INTRODUCTION

It is now well established that ion implantation is a promising method for producing both *p*-type^{1–5} and *n*-type^{2–6} layers in GaAs, at lower annealing temperatures ($\leq 900^\circ\text{C}$) than those required for the diffusion process (900–1200°C). The annealing of an ion-implanted sample is necessary in order to reduce the damage caused by the ions and to allow the bulk of the ions to become electrically active by moving to substitutional lattice sites. Unfortunately, at temperatures of about 600°C, or even less, the GaAs surface begins to decompose significantly,⁷ and incongruently, and it is necessary to cap the surface, usually with either SiO₂ or Si₃N₄ layers of 100–1000 Å thickness. Both of these encapsulating agents inhibit As out-diffusion, but it has been found that Ga diffuses through SiO₂⁸ much more readily than through Si₃N₄.^{9,10} Although the Si₃N₄ layers have sometimes in the past failed to adhere to the GaAs surfaces at high temperatures, it has recently been shown that encapsulating in a pyrolytic reactor, if carried out properly, can lead to a protective layer capable of withstanding 950°C.¹¹

Previous studies^{1–3} of electrical measurements in Cd-implanted GaAs have been carried out at energies ≤ 60 keV on samples with surfaces protected by an SiO₂ layer^{1,2} or by another GaAs sample placed on top.³ We have supplemented and confirmed these measurements by implanting 135-keV Cd ions into samples protected by pyrolytically deposited Si₃N₄. Furthermore, we have performed detailed investigations of the temperature dependence and depth dependence (profile) of the electrical properties, two areas which have not been explored extensively by the previous workers. This has led to an increased understanding of the implantation behavior of this important dopant for *p*-type GaAs.

II. EXPERIMENTAL CONSIDERATIONS

Cadmium ions were implanted at 135 keV to doses of 10^{12} , 10^{13} , 10^{14} , 10^{15} , and 10^{16} ion/cm² into undoped

single-crystal *n*-type substrates¹² ($n \approx 10^{16}$ cm⁻³) oriented in the $\langle 100 \rangle$ direction. The implantations were carried out at room temperature, using ion current densities of 0.1–1.0 $\mu\text{A}/\text{cm}^2$. The projected range, from LSS theory,¹³ is about 390 Å. Prior to implantation the samples were etched in freshly prepared 5H₂SO₄:1H₂O₂:1H₂O solution, and then a 100–500-Å Si₃N₄ layer was deposited on each sample at 720°C in

FIG. 1. The dependence of the sheet hole concentration (p_s) and the effective mobility (μ_{eff}) upon the Cd-ion fluence (ϕ). The implantation energy is 135 keV.

FIG. 2. The dependence of the sheet resistivity (ρ_s) on the anneal temperature (T_A). The annealing time was 10 min.

a pyrolytic reactor.¹⁴ The Cd ions were implanted through the Si_3N_4 layer.

After implantation, the samples were annealed at 600, 700, 800, or 900 °C in flowing Ar gas. Following the anneals, the Si_3N_4 layers were removed by a 48% HF solution, and Hall-bar-shaped mesas were etched on the implanted layer. Then Ohmic In contacts were ultrasonically soldered to the implanted p -type layer with electrical isolation provided by the p - n junction formed.

The dc Hall-effect and resistivity measurements were carried out using an apparatus which makes use of electrometers operated in a unity-gain mode to isolate the sample and effectively reduce cable capacitances. A He gas-flow cryogenic system provided temperatures from 4.2 to 300 °K. The measured quantities were the sheet resistivity ρ_s and the sheet Hall coefficient R_s . From these were calculated the effective sheet hole concentration, $p_s \equiv (eR_s)^{-1}$ (assuming a Hall factor of unity), and the effective Hall mobility, $\mu_{\text{eff}} \equiv R_s/\rho_s$. The bulk (volume) resistivity ρ and hole concentration p were calculated as a function of depth by measuring R_s and ρ_s as successive layers of the implanted section were removed by using a diluted (1:1:50) solution of H_2SO_4 :30% H_2O_2 : H_2O at 0 °C. Such an etch produces uniform damage-free GaAs surfaces,^{4,15} removing material at a rate of about 50 Å/min, as determined by a Sloan Dektak microtopographer.

III. RESULTS AND DISCUSSIONS

A. General annealing characteristics

The general annealing characteristics, as shown in Figs. 1 and 2, are quite similar to those reported previously² and thus will not be discussed in great detail here. The significant features are as follows: (i) the effective mobility recovers rapidly at annealing temperatures between 700 and 800 °C, although it never reaches the values ($\sim 450 \text{ cm}^2/\text{V sec}$) expected for lattice scattering alone; (ii) the sheet carrier concentration is close to the concentration of implanted ions for $\phi \leq 10^{14} \text{ cm}^{-2}$ and $T_A \approx 800$ to 900 °C; and (iii) the sheet resistivity can be made $\leq 10^3 \Omega/\square$ for $\phi \geq 10^{15} \text{ cm}^{-2}$ and $T_A \geq 800$ °C.

The mobility is low ($\leq 10 \text{ cm}^2/\text{V sec}$) for $\phi \geq 10^{14} \text{ cm}^{-2}$ and $T_A \leq 700$ °C, because this high a fluence creates an amorphous layer,^{16,17} and 700 °C is insufficient to anneal this gross damage in only 10 min. In fact, it is thought that for $\phi = 10^{16} \text{ cm}^{-2}$, even a 900 °C anneal will not remove all the lattice damage,¹⁶ and this problem may contribute to the decrease in μ_{eff} with increase in ϕ for the 800 and 900 °C anneals. Another reason for this decrease in μ_{eff} is, of course, the increased Cd-impurity scattering with increasing ϕ . The sheet resistivity for $\phi = 10^{15} \text{ cm}^{-2}$ is actually lower than that for $\phi = 10^{16} \text{ cm}^{-2}$, as seen in Fig. 2. This is due to the above-mentioned decrease of μ_{eff} with ϕ , as well as the fact that p_s does not continue to increase for $\phi \geq 10^{15} \text{ cm}^{-2}$ because of the solid solubility limit, to be discussed later.

FIG. 3. The sheet Hall coefficient (R_s) versus inverse temperature ($1/T$) for various anneal temperatures.

FIG. 4. The effective mobility (μ_{eff}) versus temperature (T) for various anneal temperatures.

A seemingly anomalous result is the fact that, for $\phi = 10^{13} \text{ cm}^{-2}$, p_s is lower for $T_A = 900^\circ\text{C}$ than for $T_A = 800^\circ\text{C}$. A possible explanation is that diffusion, which will be discussed later, is causing a greater total number of compensating centers to be "felt" after the higher-temperature anneal. However, experimental error cannot be ruled out since this measurement was not repeated, and the same is true for the inverted values of p_s after 700 and 800°C anneals, with $\phi = 10^{16} \text{ cm}^{-2}$.

B. Temperature dependence of R_s , μ_{eff} , and ρ_s

The temperature dependences of R_s , μ_{eff} , and ρ_s are displayed in Figs. 3, 4, and 5, respectively. The maxima in R_s versus T^{-1} , Fig. 3, immediately suggest impurity conduction^{18,19}; i. e., above the temperature (T_M) of maximum R_s , the hole current is predominantly carried in the valence band, while below T_M , the predominant current is via thermally activated hopping between acceptors. Above a critical concentration of acceptors this impurity conduction becomes "free" and the impurity levels exhibit bandlike properties. This phenomenon occurs at the so-called Mott concentration (N_c). Quantitative discussion will be deferred until Sec. III C, but, qualitatively, we would expect R_s to become almost temperature independent as the hole concentration approaches degeneracy. Even μ_{eff} should flatten out since the predominant mobility-limiting mechanism at very

high impurity concentrations is the Coulombic scattering from ionized impurities, and, at degeneracy, most of the current-carrying holes will have the almost temperature-independent Fermi velocity and thus will see a nearly constant cross section. The R_s and μ_{eff} data bear out this general picture, becoming relatively temperature independent for high ϕ , at least at low temperatures.

We now consider the R_s and μ_{eff} data in more detail. For $\phi = 10^{13} \text{ cm}^{-2}$ it is very doubtful that the Mott concentration has been exceeded, at least for $T_A = 800$ or 900°C , even though some bending over of R_s seems to be occurring at $T_A = 800^\circ\text{C}$. This speculation will be confirmed in Sec. III C in which it will be shown that $p \ll N_c$ (cf. Fig. 6 and Sec. III 3C). In fact, for $\phi = 10^{12} \text{ cm}^{-2}$, it was impossible to even observe a p -type layer, suggesting that in this case $N_A < N_D$, the background donor doping concentration ($N_D \sim 10^{16} \text{ cm}^{-3}$). For $\phi \geq 10^{15} \text{ cm}^{-2}$, on the other hand, the holes appear to be nearly totally degenerate for $T_A = 700^\circ\text{C}$, but not for $T_A = 800$ or 900°C . As will be discussed later, this is probably due to diffusion at the higher temperatures which lowers the bulk concentration. The data for $\phi = 10^{13} \text{ cm}^{-2}$, $T_A = 900^\circ\text{C}$, may indicate two acceptor levels, although the experiment would have to be repeated to confirm this.

FIG. 5. The sheet resistivity (ρ_s) versus inverse temperature ($1/T$) for various anneal temperatures.

FIG. 6. The bulk hole concentration (p) and mobility (μ) as a function of depth in the implanted layer. The LSS and diffusion profiles were calculated for $\phi = 10^{14} \text{ cm}^{-2}$.

The μ_{eff} data (Fig. 4) in general follow the expected behavior, although not totally. The data at $T_A = 800^\circ\text{C}$, for example, show the effects of increasing impurity scattering, as the maxima move toward higher temperatures with increasing ϕ . Then, at low temperatures, impurity conduction leads to a relatively constant μ_{eff} . The data at $T_A = 900^\circ\text{C}$ are somewhat less explicable. The double hump in the $\phi = 10^{13} \text{ cm}^{-2}$ data is very unusual and may not be real. Also, it is not obvious why the curves for $\phi = 10^{14}$, 10^{15} , and 10^{16} cm^{-2} are so similar, although, as will be seen in Fig. 6, the bulk hole concentrations are nearly the same, and thus the ionized-impurity scattering contribution may also be nearly the same for each fluence. For $T_A = 700^\circ\text{C}$, the relative positions of the curves are as expected, although the curves for $\phi = 10^{14}$ and 10^{16} cm^{-2} do not show the impurity-conduction tail at low temperatures. The steep (faster than $T^{3/2}$) temperature dependence of μ_{eff} below the maximum, seen in several of the curves at all three annealing temperatures, has also been observed in bulk samples, and may be due to very strong compensation.²⁰ This leads to a spatial dependence of the acceptor energy, forming peaks and valleys, and results in an "activated" mobility which can be strongly temperature dependent.

Resistivity data, when impurity conduction is important, are often analyzed according to the relationship^{18,21}

$$\rho^{-1} = C_1 \exp(-\epsilon_1/kT) + C_3 \exp(-\epsilon_3/kT),$$

where ϵ_1 is the usual activation energy required for the thermal excitation of holes from the acceptor level to the valence band and ϵ_3 is an averaged activation energy for the phonon-assisted hopping of holes from occupied to unoccupied acceptor sites. A third energy, ϵ_2 is also sometimes observed. At high acceptor concentrations, both ϵ_1 and ϵ_3 approach zero, due to screening and overlap effects, and care must be taken in assigning values to these parameters. From the ρ_s data, Fig. 5, the calculated values of ϵ_1 range from 30 to 45 meV (including the $T^{3/2}$ density-of-states correction) and $\epsilon_3 \leq 1 \text{ meV}$. For Cd-doped GaAs, values of 22–35 meV have been reported²² for ϵ_1 (or E_A), in fair agreement with our values. By using a hole effective mass²³ $m_h^* \approx 0.5m_e$ and a static dielectric constant²⁴ $\kappa \approx 12.8$, the "hydrogenic" acceptor energy is calculated to be about 40 meV.

In the interpretation of implantation data such as these, great care must be taken to exclude "profile" effects,²⁵ which closely resemble impurity-conduction effects. These profile effects have been studied by many workers and arise because the implanted layer is non-uniform, and both the hole concentration (p) and mobility (μ) are functions of depth. Then, as the temperature changes the current paths change and the interpretation of temperature-dependent data is difficult. We believe that our data are not strongly influenced by profile effects for two reasons. In the first place, these effects can be shown, by use of the Schwartz inequality, to give a measured p_s which will always be less than the total number of holes; i. e., $p_s \leq \int p(x) dx$. Since our "efficiencies" are good (60–90% for $\phi \leq 10^{14} \text{ cm}^{-2}$) such effects must not be too important. A second reason is that a detailed study of profile effects in Si showed that the respective maxima in R_s and μ_{eff} always occur at nearly the same temperature.²⁵ This is not the case for our data. The explanation for the lack of profile effects is probably the flattening of $p(x)$ and $\mu(x)$ due to diffusion, to be discussed in Sec. III C.

C. Profiles of p and μ

The profiles of p and μ , alluded to in Sec. III B, are displayed in Fig. 6 for $T_A = 900^\circ\text{C}$. The methods of attaining such profiles, by differential Hall-effect measurements, are well documented in the literature.²⁶ The LSS distribution for $\phi = 10^{14} \text{ cm}^{-2}$ is also shown in Fig. 6 and it is seen that the actual profile for $\phi = 10^{14} \text{ cm}^{-2}$ is much flatter, although, unfortunately, the experimental data could not be carried further than 0.1 μm for this particular sample. Also displayed is the diffusion profile expected²⁷ when a source of 10^{14} cm^{-2} Cd ions is placed on a GaAs surface and then heated for 10 min at 900°C . It is clear that this diffusion profile is much like the actual profile. Such a simplified diffusion model is not too inaccurate since the diffusion range at 900°C is much greater than the implantation (LSS) range for 135-keV Cd ions. (In fact, even at 700°C the diffusion range is somewhat greater than the LSS range.) The fact that the $p(x)$ and $\mu(x)$ curves are relatively flat explains why profile effects do not have a strong influence on the R_s - and μ_{eff} -vs- T curves.

FIG. 7. A comparison of theoretical and experimental hole mobilities as functions of hole concentration. See the text for details.

It is noted that the $p(x)$ curves for $\phi = 10^{14}$, 10^{15} , and 10^{16} cm^{-2} are not too different, and that the $\phi = 10^{16} \text{ cm}^{-2}$ curve actually lies below the $\phi = 10^{15} \text{ cm}^{-2}$ curve. One reason for this behavior is the solid solubility limit of Cd in GaAs, which is reported²⁸ to be about $2 \times 10^{19} \text{ cm}^{-3}$ for a vapor source at 900°C . This value is being approached for $\phi \gtrsim 10^{15} \text{ cm}^{-2}$. Another possible reason is the increase in compensating damage defect centers with increasing ϕ , some of which are probably not annealed out, even at 900°C . This could cause the inversion of the $\phi = 10^{15}$ and 10^{16} cm^{-2} curves, also apparent in the p_s curves of Fig. 1.

The Mott criterion,¹⁸ for the critical acceptor concentration at which the impurity conduction becomes "free" (zero activation energy), is given by $N_c \approx (0.25/a_H)^3 \approx 6 \times 10^{18} \text{ cm}^{-3}$, where a_H is the effective Bohr radius for holes of effective mass $m_p^* = 0.5m_e$, in a medium with a dielectric constant $\kappa \approx 12.8$. This assumes a hydrogenic model, which may not be too inaccurate since the predicted activation energy is about 40 meV, close to the measured Cd level. The Fermi level, for $T \ll T_{\text{Fermi}}$, should cross to the valence band at a concentration^{29,30} $N_{cb} \approx 5N_c \approx 3 \times 10^{19} \text{ cm}^{-3}$. If $p \approx N_A$, then for $N_A > N_{cb}$ the holes will be totally degenerate and R_s should be independent of temperature (the Fermi temperature for $p = 3 \times 10^{19} \text{ cm}^{-3}$ is about 800°K). In Fig. 3, it is seen that the R_s curves have humps for $\phi \gtrsim 10^{14} \text{ cm}^{-2}$ and $T_A \gtrsim 800^\circ\text{C}$, and thus are not totally independent of temperature; however, they appear to have almost a zero activation energy at low temperatures, in the impurity-conduction regime. From this fact we would guess that $N_c < p < N_{cb}$, or $6 \times 10^{18} < p < 3 \times 10^{19} \text{ cm}^{-3}$, and indeed our calculated $p(x)$ curves in Fig. 6 are in that range.

The $\mu(x)$ curves in Fig. 6 show that $\mu \lesssim 100 \text{ cm}^2/\text{V sec}$ for $\phi \gtrsim 10^{14} \text{ cm}^{-2}$, whereas the lattice-limited mobility is about $450 \text{ cm}^2/\text{V sec}$ at room temperature.³¹ This suggests that ionized-impurity scattering is probably an

important factor, and we have used the Brooks-Herring relationship³² (assuming $N_I \approx p$) to calculate a theoretical μ -vs- p curve, which is compared with the experimental data in Fig. 7. Also, some measured bulk mobilities^{19,33} are shown. The agreement, which is within a factor of 2, should probably be considered satisfactory, although some additional defect scattering is undoubtedly present in the sample and could explain the lower experimental mobilities. For $x \lesssim 0.1 \mu\text{m}$ the mobility falls, perhaps due to residual defects which are not annealed out at 900°C , and which do not diffuse into the bulk as fast as the Cd ions.

IV. SUMMARY

As has been shown before, Cd implantation can produce a good p -type layer in GaAs, with $\rho_s \lesssim 10^3 \Omega/\square$. The Si_3N_4 surface protection does not appear to have significant advantages over SiO_2 in this case. The profiles of p and μ indicate considerable diffusion for $T_A \gtrsim 800^\circ\text{C}$, with a layer thickness of about $1 \mu\text{m}$ produced by a 10-min anneal at 900°C . At this annealing temperature the solid solubility limit of Cd in GaAs, about $2 \times 10^{19} \text{ cm}^{-3}$, is reached with $\phi \approx 10^{15} \text{ cm}^{-2}$, and the holes are nearly degenerate. The implantation efficiencies range from 50 to 90% for $\phi \lesssim 10^{14} \text{ cm}^{-2}$. From ρ_s -vs- T data a range of acceptor activation energies from 30 to 45 meV is calculated, and these numbers compare favorably with the calculated hydrogenic acceptor energy, about 40 meV.

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