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On compensation and conductivity models for molecular-beam-epitaxial GaAs grown at low temperature

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Molecular-beam-epitaxial GaAs grown at 200 °C has an extremely high ($> 10^{19} \text{ cm}^{-3}$) concentration of As_{Ga} defects and, after an anneal at 550–600 °C, a high concentration of As precipitates. The relative roles of the As_{Ga} defects and As precipitates in compensation and conductivity is controversial. Here criteria are developed to distinguish between two existing models.

I. INTRODUCTION

Molecular-beam-epitaxial (MBE) GaAs is normally grown at temperatures of 580–600 °C and, at these temperatures, it is relatively routine to attain shallow donor N_D and acceptor N_A concentrations in the $10^{14}\text{--}10^{15} \text{ cm}^{-3}$ range, and even smaller deep donor (N_{DD}) and acceptor (N_{AA}) concentrations. Recently, however, Smith *et al.*¹ showed that MBE GaAs grown at 200 °C had much different properties, and that when used as a buffer layer could remarkably improve some critical characteristics of GaAs metal-semiconductor field-effect-transistor (MESFET) devices; since then many groups have studied the application of this material to a variety of other devices.^{2–6} The outstanding characteristic of low-temperature-grown MBE (LTMBE) GaAs is a large excess of As (1%–2%), which leads to a deep donor (As_{Ga} related) concentration $N_{\text{DD}} > 10^{19} \text{ cm}^{-3}$ and, after a 600 °C anneal, large ($\sim 60 \text{ \AA}$), dense ($\sim 10^{17} \text{ cm}^{-3}$) precipitates of As.^{7–13} The point of this paper is to discuss some controversial aspects of how these two entities affect the compensation and conductivity of LTMBE GaAs.^{10,12}

II. THE POINT DEFECT (“STANDARD”) MODEL

Look *et al.*¹⁰ have proposed a model in which the compensation is accomplished in exactly the same way as that in semi-insulating (SI) GaAs, but in which the conductivity involves an additional component, i.e., carrier hopping between the EL2-like centers. Others have proposed similar models, although less extensive and differing in some details.^{14,15} The compensation of SI GaAs is well explained by a simple charge-balance equation:¹⁶

$$\begin{aligned} n + (N_A + N_{\text{AA}} - N_D) \\ &= \frac{N_{\text{DD}}}{1 + (g_1/g_0)(n/N_C)e^{E_{\text{DD}}/kT}} \\ &= \frac{N_{\text{DD}}}{1 + (g_1/g_0)(n/N_C)e^{-\alpha/k_e E_{\text{DD}}/kT}}, \end{aligned} \quad (1)$$

where $N_C = 2(2\pi m^*k)^{3/2} T^{3/2}/h^3$, the effective conduction-band density of states. Also, $E_{\text{DD}} = E_{\text{DD}0} - \alpha T$ with $E_{\text{DD}0} = 0.748 \text{ eV}$, $\alpha \approx 3.3 \times 10^{-4} \text{ K}^{-1}$, and $g_1/g_0 = 2$ for

the deep donor EL2 ($N_{\text{DD}} = [\text{EL2}]$). Because $E_{\text{DD}0}$ is so large, the “low-temperature” form of Eq. (1) holds up to about 600 K:

$$n = [(N_{\text{DD}}/N_A^{\text{net}}) - 1] C_1 T^{3/2} e^{-E_{\text{DD}}/kT}, \quad (2)$$

where $N_A^{\text{net}} = N_A + N_{\text{AA}} - N_D$ and $C_1 \approx 1.85 \times 10^{15} \text{ cm}^{-3} \text{ K}^{-3/2}$.^{10,17} Typical values of N_{DD} and N_A for SI GaAs grown by the liquid-encapsulated Czochralski (LEC) method are 1×10^{16} and $1 \times 10^{15} \text{ cm}^{-3}$, respectively, giving $n \approx 1.5 \times 10^7 \text{ cm}^{-3}$. Usually $N_A^{\text{net}} \approx [C]$, and since n , $[C]$, and $[\text{EL2}]$ can all easily be measured by independent techniques (n by Hall effect, $[C]$ and $[\text{EL2}]$ by absorption spectroscopy), the validity of Eq. (2) for undoped, SI GaAs is well documented and is fully accepted by workers in the field.^{18,19} [Minor questions, such as whether native-defect acceptors are comparable to carbon in importance, do not detract from the overall applicability of Eq. (2).] For LTMBE GaAs, the model of Ref. 10 again assumes the validity of Eq. (2), but with a much higher value of N_{DD} , namely, $3 \times 10^{19} \text{ cm}^{-3}$ for material grown at 200 °C and not annealed. This number is confirmed by 1.1- μm absorption measurements, the same technique used for $[\text{EL2}]$ determination.⁹ After annealing at 550 °C, the number decreases to about $3 \times 10^{18} \text{ cm}^{-3}$.

The conductivity σ and Hall coefficient R in LTMBE GaAs are intimately connected with the compensation mechanism, because the conduction-band contribution to σ is just $\sigma_1 = en\mu_1$, where the mobility μ_1 is mainly due to scattering from neutral deep donors, and the Hall coefficient contribution is $R_1 = 1/en$. However, as mentioned earlier, there is another contribution to the conductivity, because the close spacing between the $3 \times 10^{19} \text{ cm}^{-3}$ deep donors promotes carrier hopping. As discussed in Ref. 10, the hopping conductivity at room temperature and above is given by

$$\sigma_2 = C_2 e^{-\gamma/aN_{\text{DD}}^{1/3}} e^{-\epsilon_3/kT}, \quad (3)$$

where C_2 and γ are constants, a is the extent of the deep-donor (As_{Ga}) wave function, and ϵ_3 is the difference between the isolated donor energy and the Fermi energy. However, hopping conductivity leads to a vanishing Hall coefficient, so that $R_2 = 0$. Then the combined conductivity and Hall coefficient equations are¹⁶

$$\sigma = \sigma_1 + \sigma_2, \quad (4)$$

$$R = \frac{R_1\sigma_1^2 + R_2\sigma_2^2}{(\sigma_1 + \sigma_2)^2} = \frac{R_1\sigma_1^2}{(\sigma_1 + \sigma_2)^2}. \quad (5)$$

By changing only the parameter N_{DD} , this model fits both the σ vs T_m and R vs T_m (T_m is measurement T , not annealing T) data very well for $T_m = 300$ – 400 K (the only range measured), as a function of sample annealing temperature, $T_a = 250$ – 550 °C. Note that the value of R at $T_m = 300$ K covers nearly eight orders of magnitude over this range of T_a .¹⁰

Thus, there can be little doubt concerning the basic validity of the conductivity/compensation model given in Ref. 10. However, that is not to say that all of the fitted parameters are firmly established, or are in agreement with the results of other experiments. In particular, consider N_A^{net} . The data of Ref. 10 are well fitted, over the entire range $T_a = 200$ – 550 °C, by the value $N_A^{\text{net}} \approx 10^{15} \text{ cm}^{-3}$. This value makes some sense because it is about the expected concentration of C, and C is also observed in photoluminescence data. Furthermore, passivation experiments with 1000-Å, 200 °C cap layers are consistent only with $0 < N_A^{\text{net}} < 1 \times 10^{16} \text{ cm}^{-3}$ or else $N_A^{\text{net}} < 0$.²⁰ However, such a low N_A^{net} is in apparent disagreement with EPR experiments carried out in similar unannealed samples^{7,8} which give $[\text{As}_{\text{Ga}}^+] \approx N_A^{\text{net}} \approx 5 \times 10^{18} \text{ cm}^{-3}$. (In annealed samples, $[\text{As}_{\text{Ga}}^+]$ is below the EPR sensitivity limit, about $1 \times 10^{18} \text{ cm}^{-3}$ in such thin layers, and thus could possibly be in agreement with the Hall value of N_A^{net} .) Although this discrepancy is serious and is not resolved yet, we offer one possible solution here. Because of sensitivity problems, the EPR experiments have been performed in relatively thick samples, $d \gtrsim 2 \mu\text{m}$. On the other hand, it is also known that the 200 °C samples become polycrystalline, and form large pyramidal defects for $d \gtrsim 2 \mu\text{m}$.^{21,22} It is possible that such massive defects, or also perhaps grain boundaries, surfaces, and interfaces, are decorated with rather shallow acceptor impurities or defects, such as V_{Ga} , which would then attract electrons from the As_{Ga}^0 , forming As_{Ga}^+ . This picture is, of course, entirely equivalent to the “depletion” process, operative with metal Schottky barriers or with surface states on conductive layers [see the discussion following Eq. (8), below]. If about 15% of the As_{Ga} volume were thus affected, then the EPR experiment could be explained by the As_{Ga}^+ near the large defects or grain boundaries, while the conductivity would take place in the other 85% of the sample, which would presumably have a small N_A^{net} and thus contain mostly As_{Ga}^0 . An obvious experiment that would shed light on this issue would be to carefully measure both the Hall and EPR concentrations on the same samples as a function of sample thickness to see if the heavily defected regions produce more As_{Ga}^+ .

III. THE AS-PRECIPITATE MODEL

We now consider another point of contention, i.e., the role of the As precipitates in compensation and conductivity. Warren *et al.*¹² have offered the interesting suggestion that the high resistivity of the samples grown at 200 °C and annealed at 600 °C is due to overlapping of the depletion regions formed between the metallic As clusters and the

“conductive” bulk. They have even suggested that the SI nature of common LEC GaAs substrates could be explained by the same mechanism. This latter assertion can be strongly refuted, as explained below, but the former merits careful consideration. We will consider, in order, the effect of the precipitates on SI GaAs, undoped LTMBE GaAs, and doped LTMBE GaAs.

The problem with applying the As-precipitate model to standard SI GaAs substrate material, grown by the LEC method, is twofold: (i) There simply aren't enough precipitates to deplete a significant fractional volume; and (ii) the standard compensation model [Eqs. (1) and (2)] works very well and can be verified by independent experiments. In regard to the first point, Martin *et al.*²³ have summarized the annealing data of several groups and found that, for common annealing temperatures between 800 and 950 °C, As precipitates range in size $2r_0$ from 1000 to 2000 Å, and concentration N_p , 10^7 – 10^9 cm^{-3} . By using the maximum r_0 (1000 Å), maximum potential difference ΔV (0.7 V), maximum N_p (10^9 cm^{-3}), and minimum N_{DD} , or N_D , or N_A ($1 \times 10^{15} \text{ cm}^{-3}$), Eqs. (6)–(8) in the following section give a depleted fraction $f < 0.001$. Thus, the As-precipitate model cannot explain the semi-insulating nature of the most common type of SI GaAs. In regard to the second point, above, the standard compensation model can be independently checked for validity. For example, N_{DD} (i.e., [EL2]) is well calibrated with the 1.1-μm electronic absorption, N_A^{net} (mostly C) with a 582-cm⁻¹ local-vibrational mode (LVM) absorption or with secondary-ion mass spectroscopy SIMS, and n with Hall-effect measurements. Such measurements have been carried out in our own laboratory and in those of others, and have confirmed the validity of Eq. (2).^{18,19} In simpler terms, if $N_A^{\text{net}} > 0$, and [EL2] (i.e., N_{DD}) $> N_A^{\text{net}}$, then the Fermi level E_F will be near midgap, whether or not there are As precipitates. Since the Schottky barrier energy due to As is also about one-half the gap energy, there will be little or no potential difference between the metal and bulk, and thus almost no depletion in the region of the precipitates. Therefore, the model presented by Warren *et al.* cannot apply to standard SI GaAs.

We will next apply the As cluster model to LTMBE GaAs layers. Warren *et al.*¹² have found that a layer grown at 200 °C and annealed for 10 min. at 600 °C contains As precipitates of concentration N_p about 10^{17} cm^{-3} , and average radius r_0 of about 30 Å. We will use these same numbers for purposes of illustration, in spite of the fact that different growth conditions will likely lead to different concentrations and sizes. The idea proposed by Warren *et al.* is that a metallic As cluster will form a Schottky barrier and pin E_F at about $E_C - 0.8 \text{ eV}$ in n -type material, and $E_V + 0.6 \text{ eV}$ in p -type material. A charge Q will deposit on the metallic sphere in order to balance this barrier. Gauss' law for a sphere of radius r_0 gives

$$Q = 4\pi\epsilon_0\Delta V, \quad (6)$$

where ΔV is the difference in potential between the metal surface and the semiconductor bulk. For an undoped LTMBE layer, this charge will have to come from the

EL2-like deep donors (there are very few shallow donors), and leave a positively charged sphere of radius r_s concentric with each metallic sphere. The radius r_s can be found from the condition of charge balance around each sphere:

$$(4\pi/3)(r_s^3 - r_0^3)eN_{DD} = Q. \quad (7)$$

The fraction of sample volume filled by these depleted spheres of radius r_s is then

$$f \simeq (4\pi/3)r_s^3 N_p. \quad (8)$$

From the data of Ref. 10, for an anneal temperature of 550 °C, the fitted parameters are $N_{DD} \simeq 3 \times 10^{18} \text{ cm}^{-3}$ and $N_A \simeq 7 \times 10^{14} \text{ cm}^{-3}$ so that from Eq. (2), $n \simeq 7.4 \times 10^9 \text{ cm}^{-3}$, or $E_F \simeq E_C - 0.45 \text{ eV}$. (For SI GaAs, $E_F \simeq E_C - 0.6\text{--}0.7 \text{ eV}$, because N_{DD} is much lower.) Therefore, $\Delta V \simeq 0.35 \text{ V}$, giving $Q \simeq 9e$ [from Eq. (6)], $r_s \simeq 90 \text{ \AA}$ [from Eq. (7)], and $f \simeq 0.3$ [from Eq. (8)]. Thus, about 30% of the sample volume would be filled with these insulating spheres. However, it should be remembered that the annealed material would be highly resistive to begin with ($\sigma = ne\mu \simeq 10^{-6}\text{--}10^{-5} \Omega^{-1} \text{ cm}^{-1}$), without any precipitates, so that the effect of the precipitates in undoped LTMBE material, with $N_A^{\text{net}} > 0$ and $N_{DD} > N_A^{\text{net}}$, is minimal. If N_p were higher, so that the depleted spheres did overlap and pinned E_F between $E_C - 0.8 \text{ eV}$ and $E_V + 0.6 \text{ eV}$, then the sample would be p type, contrary to experiment. Furthermore, it can easily be shown that the maximum σ in this case would be about $4 \times 10^{-8} \Omega^{-1} \text{ cm}^{-1}$, two orders of magnitude lower than observed. (Note that hopping conduction is negligible for samples that have experienced an annealing temperature $T_a > 500 \text{ }^\circ\text{C}$.)

It is also of interest to look at the effect of the As precipitates on transport properties. Because the depleted spheres have very low carrier concentration (since E_F is near midgap), and because they have an electric field that opposes carrier penetration, they can be modeled as spheres with vanishing conductivity imbedded in a host having conductivity σ and mobility μ . In this limit, Voronkov *et al.*^{24,25} find that

$$\sigma_{\text{meas}} = \sigma(1 - \frac{3}{2}f), \quad (9)$$

$$\mu_{\text{meas}} = \mu[(1 - \frac{9}{4}f)/(1 - \frac{3}{2}f)]. \quad (10)$$

For $f \simeq 0.3$, $\sigma_{\text{meas}} \simeq 0.55\sigma$ and $\mu_{\text{meas}} \simeq 0.6\mu$. However, a problem immediately arises here. Suppose we measure μ at 400 K to minimize hopping conduction with respect to band conduction; then the maximum possible $\mu_{\text{meas}} = 0.6(5600) \simeq 3400 \text{ cm}^2/\text{V s}$, since the lattice-limited mobility at 400 K is about $5600 \text{ cm}^2/\text{V s}$.¹⁶ However, Look *et al.*¹⁰ found that μ (400 K) $\simeq 3800 \text{ cm}^2/\text{V s}$, and this includes strong scattering from the neutral N_{DD} . It follows therefore that $f < 0.3$.

A final observation on mobility concerns the fact that, if the As precipitates are responsible for the high resistivity of annealed material, then the mobility should go through a strong minimum as the depleted spheres just begin to overlap, requiring percolated conductivity between the remaining conductive pockets. Warren *et al.*¹² noted this fact

also. This mobility decrease is in addition to the one described by Eq. (10). However, a strong decrease in mobility as a function of annealing temperature is not found in the samples of Ref. 10. In fact, the exact opposite occurs.

IV. CONCLUSIONS

The following criteria can then be used to determine if As precipitates are affecting the compensation or conductivity of undoped LTMBE GaAs.

(i) If $N_A^{\text{net}} > 0$ and $N_{DD} > N_A^{\text{net}}$, then the sample will already be highly resistive, with $\sigma \simeq 10^{-6}\text{--}10^{-5} \Omega^{-1} \text{ cm}^{-1}$, if $N_{DD} \simeq 10^{19} \text{ cm}^{-3}$.

(ii) If As precipitates are overlapping and pinning E_F between $E_V + 0.6 \text{ eV}$ and $E_C - 0.8 \text{ eV}$, then σ should be lower than $10^{-7} \Omega^{-1} \text{ cm}^{-1}$, and the sample will be weakly p type.

(iii) As a function of annealing temperature σ will decrease monotonically due to the loss of hopping conduction, the decrease of N_{DD} , and the increase of volume occupied by the As precipitates. However, μ should drop sharply as the depleted spheres begin to overlap, because then percolated conductivity will be necessary, but μ should rise again when the whole sample is uniformly semi-insulating. Thus, μ should go through a strong minimum as a function of T_a .

By comparing these criteria with the data of Ref. 10, it is clear that As precipitates are not greatly influencing the compensation or conductivity. Other sets of data will have to be examined individually to determine if the precipitates are important.

Finally, we want to examine the effect of As precipitates on doped LTMBE layers. It is known that the doping of MBE GaAs grown at low temperature is very difficult. We have attempted to dope 200 °C-grown material with $2 \times 10^{17} \text{ cm}^{-3}$ Si, but the conductivity is about the same as that of undoped material, dominated by hopping in the deep-donor band.²⁶ Since the Si is definitely present in these samples, as shown by SIMS, it is either not activated (i.e., not present in the form of Si_{Ga}), or it is fully compensated by acceptors ($N_A^{\text{net}} > 0$), or it is compensated by As precipitates. However, it is doubtful that As precipitates exist at all in unannealed material, because they are not seen by TEM.¹¹ We next note that a 600 °C anneal, which will produce the precipitates, does not produce a conductive sample.²⁶ To explain this, it is theoretically possible that the anneal activates the Si, and that the precipitates then compensate the Si; however, no annealing experiments that we have carried out give any indication that the Si activates. A final point is that there is evidence (from EPR) that acceptors in the mid- 10^{18} cm^{-3} concentration range can exist in 200 °C unannealed material, which would give $N_A^{\text{net}} > 0$ even in the presence of $2 \times 10^{17} \text{ cm}^{-3}$ Si. However, it is also argued by the same researchers that these acceptors anneal out at 600 °C (Ref. 7); therefore, they could not compensate the Si in annealed material. From these considerations, we believe that our doping experiments are explained by the nonactivation of Si, rather than by compensation due to As precipitates or

by other acceptors. However, other doping experiments that do not produce conductivity may be consistent with one of these other cases.

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