Electrical properties of nitrogen-doped GaP

G. B. Stringfellow, H. T. Hall Jr., and R. A. Burmeister

Hewlett-Packard Company, 1501 Page Mill Road, Palo Alto, California 94304
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The electrical properties, namely, electron concentration and mobility, have been investigated in the temperature range from 53 to 400 K for undoped and nitrogen-doped VPE GaP. The undoped GaP has a background doping level of $N_D \sim 10^{17}$ cm^{-3} and $N_A \sim 10^{16}$ cm^{-3}. The donor ionization energies vary from 49 to 74 meV and are proportional to $N_A^{1/3}$. These data identify the background donor to be Si. The mobility of the nondoped material is approximately 150 cm^2/V s at 300 K and is dependent on $N_D + N_A$ at 77 K, varying between 377 and 1263 cm^2/V s. NH_3 is added to the system to incorporate nitrogen into the GaP. This is found to decrease N_D to $\sim 2\times 10^{16}$ cm^{-3} and increase the donor ionization energy. This is attributed to reaction of the volatile Si species acting as dopant with NH_3 to form Si_3N_4, thus reducing the Si concentration in the solid. The electron mobility at 77 K is also reduced by the addition of N to the solid. This is attributed to neutral impurity scattering by the nitrogen.

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I. INTRODUCTION

Nitrogen-doped GaP is widely used for yellow-green LED's. Much of this material is grown by LPE[1-3] and more recently work on VPE growth of GaP has been reported.[4-7] To achieve high-efficiency green LED's, nitrogen doping is required. The nitrogen is isoelectronic with phosphorus but because of the large difference in electronegativity between the two it is able to bind an exciton, the recombination of which results in efficient (typically 0.1%) electroluminescence.

The maximum amount of nitrogen which can be incorporated in GaP during LPE growth is approximately 2×10^{18} cm^{-3} [8] at 1000 C, which is believed to be the solid solubility limit.[9] Higher concentrations can be achieved by VPE because of nonequilibrium effects.[6]

Because N and P are isoelectronic, N should not effect the majority-carrier concentration, but calculations of the scattering cross section indicate[10,11] that at high concentrations nitrogen may lower the mobility.

The purpose of this paper is to characterize the electrical properties of VPE GaP over the range of nitrogen concentration from less than 10^{17} to 10^{19} cm^{-3} with particular emphasis on material with large nitrogen concentrations and low values of $N_D + N_A$. In this material at low temperatures, it should be possible to distinguish between scattering due to ionized impurities and that due to nitrogen and thus characterize experimentally the effect of nitrogen on the electron mobility.

II. EXPERIMENTAL

Two types of VPE systems were used to grow the material used in this study. One is the standard process[4-7] used for VPE GaP growth using PH_3 to transport the phosphorus, cylinder HCl to transport the Ga, and H_2 as the carrier gas. The particular system used to grow the layers reported in this study is rf heated so that the hot gases see only graphite and is described in more detail, including a list of typical growth conditions used, in Ref. 6. Another system used to grow several samples is made up entirely of quartz and uses PCl_3 to transport both P and Ga. It is similar to that described by Oldham.[12]

The GaP samples for electrical measurements were grown on (100)-oriented Cr-doped semi-insulating substrates. Typical epitaxial layer thicknesses were 10–50 μm. Measurements of carrier concentration and mobility were made on either rectangular shaped Hall bars or clover-leaf shaped van der Pauw specimens. Either In or Sn-In contacts alloyed at 550 C were found to be Ohmic under most conditions. For some samples, the carrier concentration fell below 10^{12} cm^{-3} during cooling. In this range of n ($<10^{12}$ cm^{-3}), no Ohmic contacts could be found and reliable measurements could not be made. The carrier concentration was calculated as $n = 1/Re$, assuming the Hall coefficient to be unity, and the mobility was calculated as σR.

III. RESULTS AND DISCUSSION

A. Electron concentration vs T

For non-nitrogen-doped GaP, the room-temperature carrier concentration was typically 10^{17} cm^{-3}. In Fig. 1, the carrier concentration is plotted vs $1/T$ for one such sample (371) down to 53 K (see inset). From these data, one can determine the values of the donor and acceptor concentrations, N_D and N_A, and the donor ionization energy, E_D, by fitting the following expression[13] to the data:

$$\frac{n(n+N_A)}{N_D - N_A - n} = \frac{N_c'}{gT^{3/2}} \exp\left(-\frac{E_D}{kT}\right) , \quad (1)$$

where N_c' is the conduction-band density of states divided by $T^{3/2}$ and g is the degeneracy of the donor level. In practice, this is done in several steps. At high temperatures in the exhaustion region, $n = N_D - N_A$. Thus, $N_D - N_A$ was taken to be equal to the value of n measured at 400 K. At low temperatures, the condition $n \ll N_D$, N_A is satisfied and thus from a semilog plot of $nT^{-3/2}$ vs $1/T$, one can obtain E_D. In this region, N_A is given simply by the expression

$$N_A = \frac{N_D - N_A}{n} \frac{N_c'}{gT^{3/2}} \exp\left(-\frac{E_D}{kT}\right) . \quad (2)$$

In direct-band-gap materials, g has a value of 2 reflecting the spin degeneracy. The band structure of GaP has not been completely determined. The conduction-band minima lie along [100] but whether they lie

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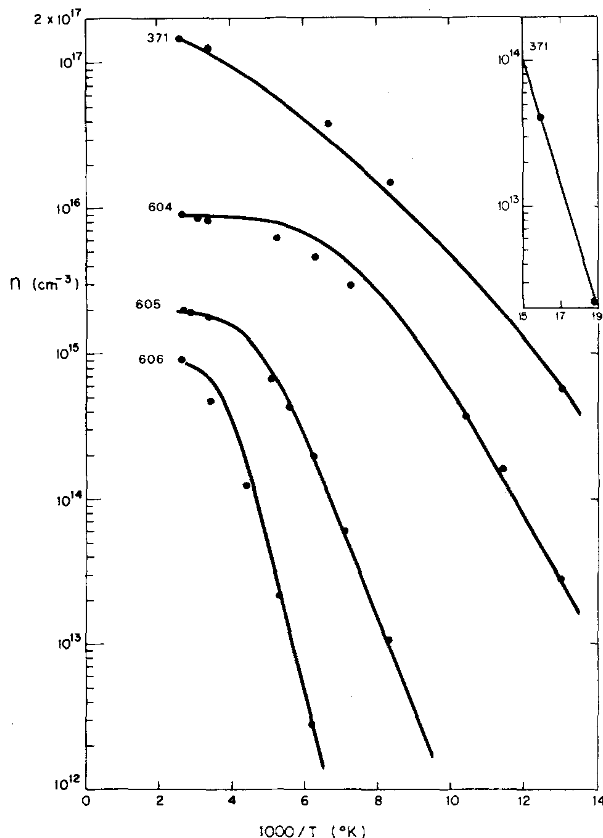

FIG. 1. Free-electron concentration vs $1000/T$ for VPE GaP samples grown with all conditions, excepting NH_3 flow rate, held constant. The nitrogen concentrations, in units of cm^{-3}, for the various samples are $<10^{17}$ (371), 8.8×10^{17} (604), 4.9×10^{18} (605), and 8×10^{18} (606). The solid curves are values of n obtained from Eq. (1) using the parameters N_D, N_A, and E_D listed in Table I.

at the Brillouin zone boundary (giving three equivalent minima) or not (giving six minima) has not been established, although the former is more likely.[14] For the three-valley conduction-band model, the donors occupying P sites have the twofold spin degeneracy; however, the p-like Ga site donors are sixfold degenerate. Independent of the model, the quantity N_c'/g has been experimentally determined to be $\sim4.0\times10^{15}$ $cm^{-3}K^{-3/2}$ for the group-VI donors and $\sim1.3\times10^{15}$ $cm^{-3}K^{-3/2}$ for Si.[15]

In the non-nitrogen-doped GaP obtained in both reactors used in this study, Si is believed to be the major donor impurity. Emission spectrographic analysis reveals Si concentrations of $>10^{17}$ cm^{-3} which is confirmed by 4.2 K PL results which show the Si emission lines[6] normally observed only at concentrations greater than 10^{17} cm^{-3}.[15] On the other hand, S, the other major donor, has been quantitatively determined by 4.2 K optical absorption measurements using the calibration factor from Ref. 15 to have concentrations of less than 10^{16} cm^{-3} in the non-nitrogen-doped GaP samples. Thus, for non-nitrogen-doped GaP layers, the value of N_c'/g $=1.3\times10^{15}$ $cm^{-3}K^{-3/2}$ was used in the calculations.

In Table I, the values of N_D, N_A, and E_D obtained from this analysis are listed for five non-nitrogen-doped samples. The Si concentration is seen to be 1.45×10^{17} cm^{-3} for the sample grown in the PH_3 system (371) and between 6.12×10^{17} and 2.19×10^{17} cm^{-3} for the samples grown in the PCl_3 system (with prefix M) with N_{Si} decreasing with substrate temperature.

In Fig. 1, n is plotted vs $1/T$ for several samples with nitrogen concentrations ranging up to $\sim10^{19}$ cm^{-3}. The carrier concentrations at room temperature are seen to decrease from $\sim10^{17}$ to $\sim10^{15}$ cm^{-3} as the nitrogen concentration increases. At lower temperatures, the falloff in n with increasing nitrogen concentration is even more pronounced. The analysis of these data yield values of N_D, N_A, and E_D listed in Table I, along with data obtained on similar samples not included in Fig. 1. The values of N_D are seen to fall from greater than 10^{17} to $\sim5\times10^{15}$ cm^{-3} as nitrogen is added to the system with the exception of sample 607 for which both

TABLE I. Data obtained from analysis of carrier concentration vs temperature data for VPE GaP samples.

Sample No.	Growth system/ sub. temp.	N_N (cm^{-3})[a]	n_{400} (cm^{-3})	n_{77} (cm^{-3})	N_D (cm^{-3})	N_A (cm^{-3})	E_D (meV)
371	PH_3-HCl/840C	0	1.47×10^{17}	7.10×10^{14}	1.50×10^{17}	2.50×10^{15}	74
M-11	PCl_3/840C	0	5.75×10^{17}	8.05×10^{14}	7.51×10^{17}	1.76×10^{17}	53
M-14	PCl_3/820C	0	1.81×10^{17}	5.25×10^{14}	2.26×10^{17}	4.60×10^{16}	54
M-17	PCl_3/820C	0	2.64×10^{17}	4.42×10^{14}	3.58×10^{17}	9.37×10^{16}	56
M-20	PCl_3/800C	0	1.08×10^{17}	1.30×10^{15}	2.19×10^{17}	1.11×10^{17}	49
604	PH_3/HCl/840C	8.8×10^{17}	9.00×10^{15}	2.40×10^{13}	1.28×10^{16}	3.70×10^{15}	82
315	...	2.2×10^{18}	7.00×10^{15}	5.00×10^{13}	1.70×10^{16}	1.00×10^{16}	70
321	...	2.6×10^{18}	8.50×10^{15}	1.60×10^{13}	8.63×10^{16}	8.07×10^{16}	63
605	...	4.9×10^{18}	2.00×10^{15}	3.00×10^{10}	2.85×10^{16}	2.65×10^{16}	110
606	...	8×10^{18}	9.00×10^{14}	1.10×10^{11} [b]	5.40×10^{15}	4.50×10^{15}	184
607	...	8.8×10^{18}	1.70×10^{16}	1.90×10^{11}	4.44×10^{17}	4.32×10^{17}	87
M-40	PCl_3/800C [c]	0	1.46×10^{15}	7.79×10^{11}	1.11×10^{16}	9.64×10^{15}	91
M-43	...	0	7.70×10^{14}	9.70×10^{12} [d]	7.62×10^{16}	7.55×10^{16}	81
M-45	...	0	2.62×10^{15}	5.46×10^{12}	2.81×10^{15}	1.85×10^{14}	84

[a] In this column, 0 signifies no intentionally added N. The actual concentration is less than 10^{17}.
[b] $T=135$ K.
[c] Oxygen doped.
[d] $T=135$ K.

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TABLE II. Measured and calculated mobilities, expressed in units of $cm^2/V\,s$, in VPE GaP.

Sample No.	Experimental μ_{300}	Experimental μ_{77}	Calculated-77 K μ_{77} [a]	Calculated-77 K μ_I [b]	Calculated-77 K μ_N [c]
371	154	1263	1131	1815	∞
M-11	135	377	702	916	∞
M-14	155	1126	1093	1718	∞
M-17	148	901	891	1267	∞
M-20	161	1262	1047	1607	∞
604	179	1527	1312	15 735	2738
315	176	903	745	10 396	1095
321	160	703	505	1755	927
605	143	478	386	4447	492
606	116	172 [d]	261 [d]	1292 [d]	329 [d]
607	111	93	153	395	274
M-40	176	1424	2365	11 173	∞
M-43	187	845 [e]	970 [e]	1757 [e]	∞
M-45	196	1958	2990	9.4×10^5	∞

[a] Calculated as $(\frac{1}{3000} - 1/\mu_I + 1/\mu_N)^{-1}$.
[b] Calculated using Eq. (3) of the text.
[c] Calculated using Eq. (10) of the text.
[d] $T = 135$ K.
[e] $T = 110$ K.

N_D and N_A are very high for an unknown reason. The value of N_A does not vary systematically with nitrogen concentration.

A likely explanation for this behavior is that for the nitrogen-doped layers the NH_3 present in the gas stream reacts with the volatile Si species transporting the dopant (most likely a compound of the type $SiCl_xH_{4-x}$), to form the very stable compound Si_3N_4 thus lowering the partial pressure of Si in the system and lowering N_{Si} and, hence, N_D. Since S is present in the system at concentrations of $10^{15}-10^{16}$ cm^{-3}, it becomes the dominant donor for the nitrogen-doped samples. Thus, for the analysis of the n-vs-$1/T$ curves for the nitrogen-doped samples, a value of 4.0×10^{15} $cm^{-3}\,K^{-3/2}$ for N_c'/g was used. As will be discussed below, an ammonia flow sufficient to produce a nitrogen doping level of 5×10^{16} cm^{-3} reduces the silicon concentration in the solid to $<10^{16}$ cm^{-3}.

The effect of oxygen in the gas stream should be similar to that of NH_3, that is, it should react with the Si to form SiO_2. To test this, we introduced 40 ppm of H_2O into the gas stream of the PCl_3 reactor. The result was that n at room temperature was reduced to $(7-15)\times10^{14}$ cm^{-3} as expected. The resultant values of doping level and donor ionization energy are also tabulated in Table I.

In principle, the donor ionization energy (E_D) should be different in the undoped GaP ($E_{Si} = 82$ meV) and the nitrogen-doped material ($E_S = 104$ meV). The well-known effect of N_A on the ionization energy would make the measured values somewhat smaller. The values of ionization energy in some samples with high nitrogen concentrations are ~20 meV higher than expected for S. The cause of this is undetermined. For the non-nitrogen-doped samples, a plot of E_D vs $N_A^{1/3}$ is presented in Fig. 2. The data agree with those of Vink *et al.*[15] for intentionally Si-doped GaP and extrapolate to ~82 meV for $N_A = 0$.

B. Mobility in non-nitrogen-doped GaP

The mobility of high-purity ($N_D + N_A < 10^{16}$) GaP has been reported by Craford *et al.*[16] to be ~3000 $cm^2/V\,s$ at 77 K and to vary as T^{-2}. Rode[14] has calculated the lattice mobility for GaP. He finds the dominant scattering mechanism to be intervalley and acoustic mode scattering. The calculated mobilities agree fairly well with Craford's data, but at 77 K, they yield a value of 4500 $cm^2/V\,s$ for the electron mobility. Rode suggests that the calculation probably overestimates the mobility and thus Craford's value is taken to be the best estimate of the 77 K lattice mobility.

The mobilities at 77 K of all samples listed in Table II are somewhat lower than 3000 $cm^2/V\,s$, especially those with large values of $N_D + N_A$, indicating that ionized impurity scattering limits the mobility. The Brooks-Herring model[17] is usually used to describe ionized impurity scattering in III-V semiconductors; however, it is based on the assumption of spherical equal energy surfaces which is not justified for GaP. Nevertheless, it was originally applied to Ge and Si for which the conduction-band minima are not spherical and was found to work fairly well, probably underestimating the mobility by approximately a factor of 2.[17]

We have calculated the Brooks-Herring[17] mobility of GaP using the density-of-states electron effective mass, m_e^*, equal to $0.365m$ [18] and a value of 11.10 [19] for the static dielectric constant, which yields

$$\mu_I = \frac{6.72\times10^{17}T^{3/2}}{N_D + N_A}\left(\ln(1+b) - \frac{b}{1+b}\right)^{-1}, \qquad (3)$$

where

$$b = 4.38\times10^{14}T^2/n'$$

and

$$n' = n + (n + N_A)\left(1 - \frac{n + N_A}{N_D}\right).$$

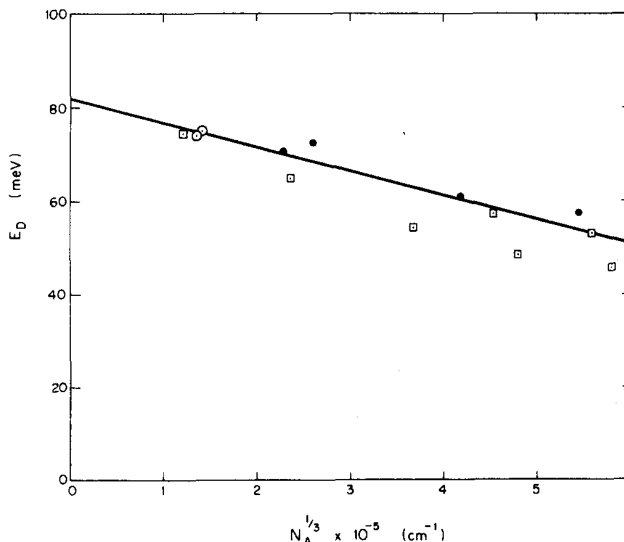

FIG. 2. Donor ionization energy vs $N_A^{1/3}$ for n-type nominally undoped VPE GaP samples. ○: PH_3 + HCl system; ☐: PCl_3 system. The solid circles (●) are for intentionally Si-doped samples from Ref. 15.

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This result calculated at 77 K combined with the value of 3000 $cm^2/V\,s$ for lattice scattering yields calculated mobilities smaller than experimental values by ~3×, similar to the results in Si.[17] By using the value of 3000 $cm^2/V\,s$ for the lattice mobility, the ionized impurity mobility could be calculated for each experimental point. The ratio of the experimental to calculated ionized impurity mobility was then determined to be 3.37 ($\sigma = 1.02$) for the six non-nitrogen-doped samples in Table II judged to have the most reliable mobility values. The best fit to the experimental data is thus obtained by multiplying the constant in Eq. (3) by 3.37 which yields a value of 2.26×10^{18} ($\pm 0.69\times10^{18}$). The resulting expression for combined lattice and ionized impurity scattering at 77 K is then, assuming that the mobilities combine as the reciprocals of the individual mobilities,

$$\mu_{77} = \left(\frac{1}{3000} + \frac{(N_D + N_A)[\ln(1+b) - b/(1+b)]}{1.53\times10^{21}}\right)^{-1}. \quad (4)$$

This agrees with experiment very well for the non-nitrogen-doped samples listed in Table II. Having an accurate calculation of lattice plus ionized impurity scattering is important because it allows the effects of nitrogen on the mobility to be clearly distinguished from the ionized impurity and lattice scattering. In Fig. 3, the 77 K mobility is plotted vs $N_D - N_A$ including not only the present work but other data reported in the literature. Our data cover the same range of $N_D - N_A$ and are in excellent agreement with that of Vink *et al.*[15] and generally agree with the more scattered data of Craford *et al.*[16] which lie at lower values of $N_D - N_A$, and of Taylor *et al.*[20] All of the samples used in these studies were grown by VPE. The data of Montgomery[21] and Toyama[22] are considerably lower. These two authors prepared their samples by slow cooling from a Ga melt as compared to epitaxial techniques for the other work. This may result in inhomogeneities (Ga inclusions) or other defects which cause lower apparent mobilities.

The curves in Fig. 3 were calculated using Eq. (4) for various values of the compensation ratio, $N_D + N_A / N_D - N_A$. The values of n at 77 K were calculated by solving Eq. (1) using $N_c'/g = 1.3\times10^{15}$ cm^{-3} and obtaining E_D from the straight line in Fig. 2, i.e.,

$$E_D = 0.082 - 4.4\times10^{-8} N_A^{1/3}. \quad (5)$$

These values are typical of Si, but the calculations carried out for S gave very nearly identical results. The experimental compensation ratio was less than 1.8 for all samples included in the plot. The calculation is seen to describe well the dependence of 77 K mobility on impurity concentration.

One interesting feature not commonly observed on such plots is the increase of mobility as $N_D - N_A$ increases at high values of $N_D - N_A$ for compensated samples, resulting in the mobility of a compensated sample being larger than that of an uncompensated sample. The reason for this is that, for a compensation ratio of unity, $N_A = 0$ and $n' = n(2 - n/N_D) \simeq 2n$, whereas for a compensation ratio of 2, $N_A = \frac{1}{3}N_D$ and $n' = 2N_D/g$. Since $N_D \gg n$, this results in more screening of the scattering centers and, hence, a larger mobility for the compensated case, in spite of the fact that $N_D + N_A$ is three times as large.

C. Mobility in nitrogen-doped material

For nitrogen-doped samples, the 77 K mobilities are lower than for the non-nitrogen-doped samples described above even though $N_D + N_A$ is also generally lower. In Fig. 4, the 77 K mobility is plotted vs nitrogen concentration in the solid. This plot includes only those samples where $N_D + N_A < 2\times10^{17}$ cm^{-3} to avoid large changes in mobility due to fluctuations in $N_D + N_A$. These results indicate that nitrogen itself may act as a scattering center and that the resultant neutral impurity scattering may dominate the mobility, particularly at higher doping levels. The scattering cross section for nitrogen treated as a deep narrow potential

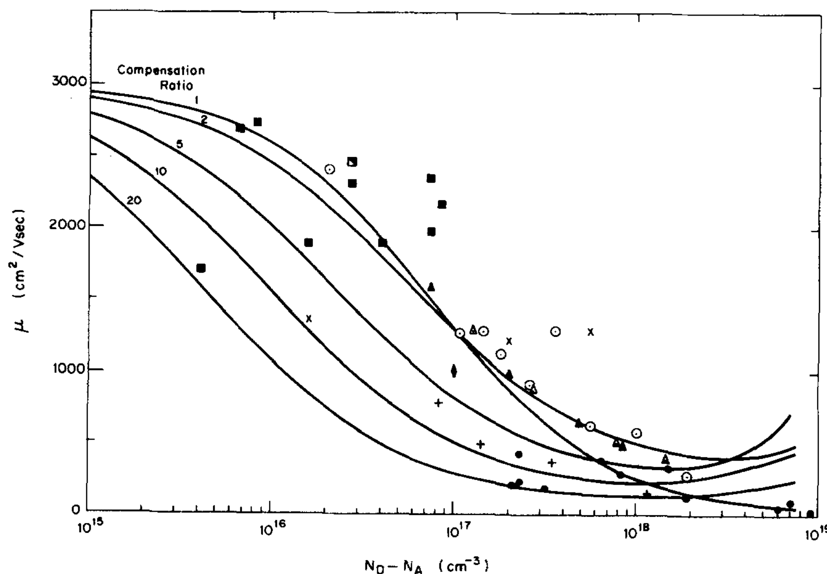

FIG. 3. Electron mobility at 77 K vs $N_D - N_A$. The curves were calculated as described in the text for combined lattice and ionized impurity scattering for values of $(N_D + N_A)/(N_D - N_A)$ of 1, 2, 5, 10, and 20. The experimental data are from Refs. 15 (▲), 16 (◻), 20 (●), 21 (+), 22 (×), 23 (◇), and the present work (⊙). The open, solid, and half-filled data points are for Si, S, and Te doping, respectively.

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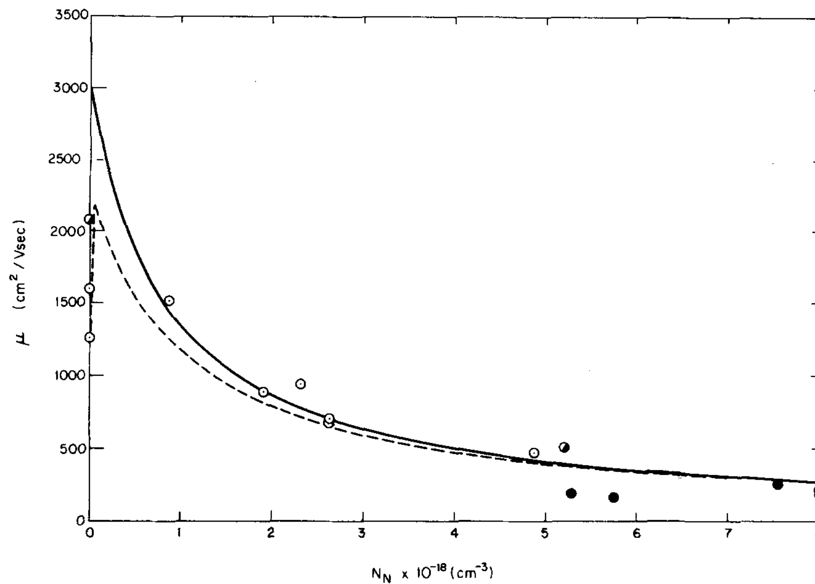

FIG. 4. Electron mobility at 77 K vs nitrogen concentration. The solid line is calculated for combined lattice and nitrogen impurity scattering. The broken curve also includes ionized impurity scattering with N_D+N_A and $n(77\ K)$ changing from 2×10^{17} cm^{-3} and 7×10^{14} cm^{-3} to 5×10^{16} cm^{-3} and 1×10^{13} cm^{-3}, respectively, as nitrogen is added to the system. The data points are VPE GaP from the present work (⊙, PH_3 system; ●, PCl_3 system) and from Ref. 24 (◑).

well has been calculated by Faulkner[10] and is given by Dean[11] to be

$$\sigma=\frac{4\pi}{3}\frac{\hbar^2}{2m\epsilon_k}\frac{\epsilon_k}{E_I+\epsilon_k}\,, \tag{6}$$

where E_I is the binding energy of the isoelectronic trap N and ϵ_k is the electron energy as a function of momentum k. The scattering lifetime is simply

$$\tau=1/N_N\sigma\nu, \tag{7}$$

where the velocity ν may be written $(2\epsilon_k/m^*)^{1/2}$. Thus, the lifetime is given by the expression

$$\tau=a\left(\frac{E_I}{\epsilon_k^{1/2}}+\epsilon_k^{1/2}\right) \tag{8}$$

with

$$a=\frac{3m_e^{*3/2}}{2^{3/2}\pi\hbar^2N_I}\,. \tag{9}$$

Hence, the average lifetime is[25]

$$\langle\tau\rangle=\frac{4a}{3\sqrt{\pi}}\left(\frac{E_I}{(kT)^{1/2}}+2(kT)^{1/2}\right)\,. \tag{10}$$

The mobility is, of course, $e\langle\tau\rangle/m_e^*$. Using a value of $m_e^*=0.365$ [18] and $E_I=8$ meV,[10] we arrive at an expression for the mobility due to scattering from N centers,

$$\mu_N=\frac{7.83\times10^{19}}{N_N}\left(\frac{116}{T^{1/2}}+2T^{1/2}\right)\,. \tag{11}$$

Again, assuming the mobilities can be combined as the sum of reciprocals, this can be combined with the lattice mobility at 77 K, 3000 cm^2/V s, to yield the solid curve in Fig. 4. The calculation describes very well the reduction of mobility due to nitrogen doping, although it is somewhat larger than experimental values for $N_N=0$ because of the neglect of ionized impurity scattering. When this is included, calculated as described in Sec. IV and, for the sake of producing a smooth curve, the value of N_D is decreased from 2×10^{17} cm^{-3} for $N_N=0$, to 2×10^{16} cm^{-3} with the addition of NH_3 to the system, N_A is held constant at 1×10^{16} cm^{-3}, and n at 77 K is calculated from Eq. (2) using the appropriate values of E_D and N_c'/g for Si$(N_N=0)$ and S$(N_N>0)$, the broken curve in Fig. 4 is obtained. To get an idea of how much NH_3 is really necessary to reduce the background Si concentration to the point where it does not affect N_D, we can do a thermodynamic calculation similar to those described in Ref. 6. Such a calculation predicts that enough NH_3 to produce a nitrogen concentration in the solid of 5×10^{16} cm^{-3} is sufficient to reduce the Si concentration in the solid at 840 °C to less than 10^{16} cm^{-3}.

Figure 5 is a plot of electron mobility vs temperature for samples chosen to have $N_D+N_A\leq5\times10^{16}$ cm^{-3} to avoid as far as possible effects of ionized impurity scattering. The calculated curves are for combined lattice and nitrogen scattering, using the equation

$$\mu=\left(\frac{1}{3000(77/T)^2}+\frac{N_N}{7.83\times10^{19}(116/T^{1/2}+2T^{1/2})}\right)^{-1}. \tag{12}$$

The neutral impurity scattering does not drop off rapidly with decreasing temperature and thus at very low temperatures ionized impurity scattering should dominate in even these samples.

IV. CONCLUSIONS

We have investigated the effects of nitrogen doping on the carrier concentration and mobility of n-type samples of VPE grown GaP. We observe that two major effects of nitrogen doping are a reduction of the free-electron concentration at all temperatures and a reduction of the mobility at low temperatures. At 77 K, the mobility is reduced 10× by the addition of 10^{19} cm^{-3} of nitrogen to solid.

The first effect is attributed to reaction of the volatile Si species, which give the background doping levels,

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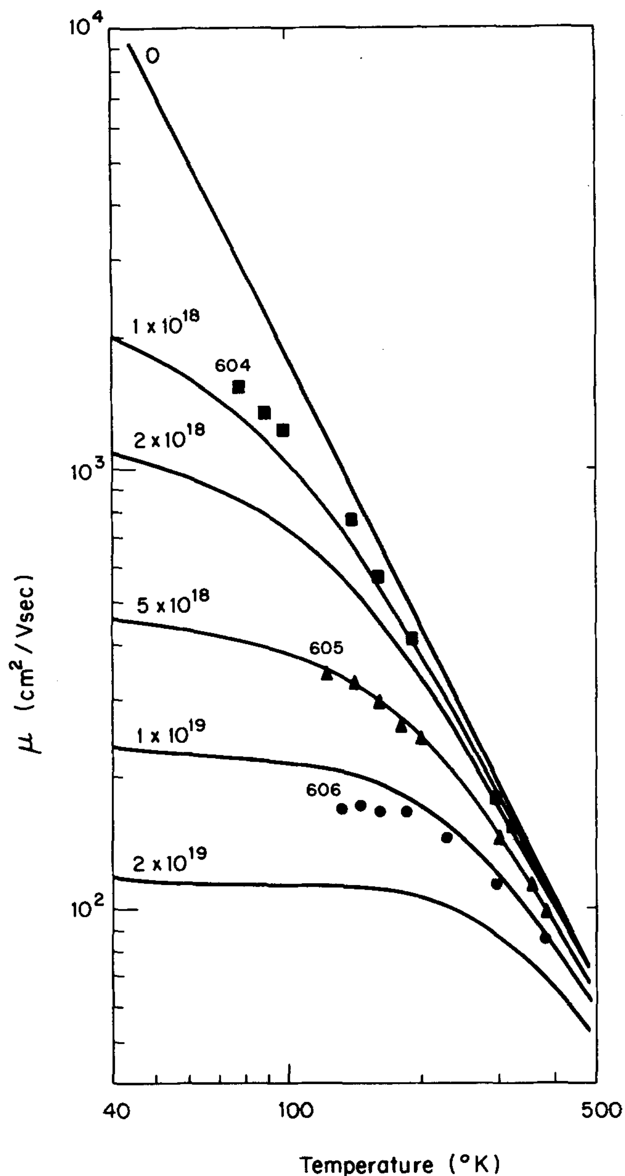

FIG. 5. Electron mobility vs temperature for samples with the following nitrogen concentrations: 8.8×10^{17} cm^{-3} (■), 4.9×10^{18} cm^{-3} (▲), and 8×10^{18} cm^{-3} (●). The solid curves are calculated for combined lattice and neutral impurity (nitrogen) scattering using Eq. (12) and the nitrogen concentrations indicated.

with the NH_3 to produce the stable compound Si_3N_4. This lowers N_D in the GaP from $\sim 2\times10^{17}$ cm^{-3} in non-nitrogen-doped samples to $\sim 5\times10^{15}$ cm^{-3} in nitrogen-doped samples. The same effect can be produced by adding H_2O to the gas stream which reacts with the Si to form SiO_2.

The reduction of mobility by nitrogen is shown to be a result of neutral impurity scattering by the nitrogen. The mobility calculated for this form of scattering using the scattering cross section of Faulkner[10] agrees with the experimental results very well.

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