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Electron paramagnetic resonance of electron-irradiated GaP

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An electron paramagnetic resonance (EPR) study of electron-irradiated GaP is presented with emphasis on the properties of the isolated gallium vacancy which can be deduced from its EPR. Further confirmation of the assignment of this spectrum to the vacancy is presented with additional discussion of defect structure. Details of the optical excitation of the EPR-active charge state are given. The vacancy is introduced at a high rate (~ 1 cm^{-1}) in p-type material for electron irradiation at energies of 1–2 MeV. An electron fluence study is reported. High concentrations of isolated vacancies are not found in heavily irradiated n-type samples. The vacancy EPR has been observed from 1.4 to 150 K. The vacancy disppears in isochronal annealing studies at temperatures from 300 to 350 °C. Reverse annealing was observed following intense room-temperature laser excitation. A brief discussion of other EPR spectra observed in e-irradiated GaP is given.

I. INTRODUCTION

Point defects in III-V semiconductors are undergoing intense study for three reasons: (1) general improvement of the material quality aimed at better electronic devices, (2) the solution of specific problems such as the low efficiency of light-emitting diodes, and (3) lessening of the degradation in quality under adverse conditions such as radiation or high temperature. In recent years a great deal has been learned about the electronic properties of point defects, such as energy levels and capture cross sections, through the use of photoluminescence (PL) and deep-level transient spectroscopy (DLTS). For example, DLTS studies[1-3] have shown that defects which occur in bulk material are different from those which occur in epitaxial material, and that the defect concentrations of vapor-phase and liquid-phase epitaxial materials also differ.

Where controlled doping can be carried out, the identity of the defect seen by PL or DLTS can be inferred from the relation between the signal amplitude and the doping level. This procedure breaks down somewhat with intrinsic defects: lattice vacancies, interstitials, and antisites (cation-anion replacement defects). These defects are particularly important to material growth, where lack of perfect stoichiometry produces them, and in radiation damage. Many experiments involving changes in stoichiometry have been performed and analyzed in terms of vacancy concentrations. However, cation deficiency, for example, in a III-V semiconductor can be made up by III-ion vacancies (V_{III}), V-ion interstitials (V_I), and V-ion antisites (V_{III}). On the basis of thermodynamic arguments, antisites were predicted to be dominant in as-grown material.[4] Thus there has been a great need for positive identification of the intrinsic defects in III-V materials.

Electron paramagnetic resonance (EPR) has been successful in studying intrinsic defects in Si,[5] II-VI and more-ionic compounds.[6] The technique is hampered in the III-V's by large linewidths which arise from the presence in all III-V nuclei of magnetic moments and the covalency of the material which guarantees that the wave function of any defect will be spread over at least a couple of neighboring shells of host atoms. Despite this difficulty, progress has been made recently on understanding transition-metal impurities in III-V's.[7,8] On intrinsic defects the P_{Ga} antisite,[9] the V_{Ga} vacancy,[10] and an antisite-impurity pair $P_{Ga}-X$[11] have recently been identified using EPR. The present work is a continuation of, and an enlargement on, the EPR study of V_{Ga}.[10]

After a description of the experimental techniques in Sec. II, the further results and discussion of defect identification and structure, results on defect introduction, temperature effects, and annealing are given in Sec. III. Other spectra are given in Sec. IV and concluding remarks in Sec. V.

II. EXPERIMENTAL ASPECTS

The EPR experiments were performed using two spectrometers. The first is a Varian E-3 operating at X band (9 GHz). Temperatures from 4.5 K to above room temperature were obtained with an insert Dewar or gas-flow systems. The sample is optically excited through slits in the cavity wall with a Bausch and Lomb high-intensity monochromator. The second is a home-built spectrometer operating at K band (24 GHz). The microwave bridge utilizes a Gunn oscillator and a Schottky-barrier detector. The cavity, a TE_{011} cylindrical mode with central ac-

cess tube,[12] is immersed in liquid helium in a S.H.E. Corporation fiberglass Dewar with a hold time of 46 h (4.5 l capacity). The sample is optically excited by two 700-nm-light-emitting diodes (Hewlett-Packard HEMT-6000) mounted in the cavity wall. At room temperature the two diodes emit 30 μW of light for 25 mA of current.

The samples were cut from wafers of liquid-encapsulation Czochraliski- (LEC) grown GaP to dimensions of $3\times16\times0.5$ mm^3. Specimens of this size can be used in either EPR spectrometer. The samples were oriented with the static magnetic field in the $(0\bar{1}1)$ plane. The irradiations were performed at the NRL 2-MeV Van de Graaff accelerator in air with the sample on a water-cooled block. The current density was 4.6×10^{12} $e/$cm^2 sec.

In order to determine defect concentrations, integrations of the EPR data were performed using a Nicolet 1070 signal averager (see Fig. 1). The spin density was obtained by comparing the value of the second integral, which is proportional to the spin susceptibility, to the second integral of a standard sample. The signal from Te donors in Te-doped GaP was used as the standard. The donor concentration was taken to be twice the room-temperature electron concentration because of incomplete ionization at room temperature.[13] The standard has the advantage of identical shape, dielectric constant, and EPR linewidth to the v_{Ga} signal in an irradiated sample. Thus only the spectrometer gain needs to be changed to make a comparison. The absolute error in determining the defect intensity is conservatively estimated to be a factor of 2, largely arising from the uncertainty in the Te concentration of the standard.

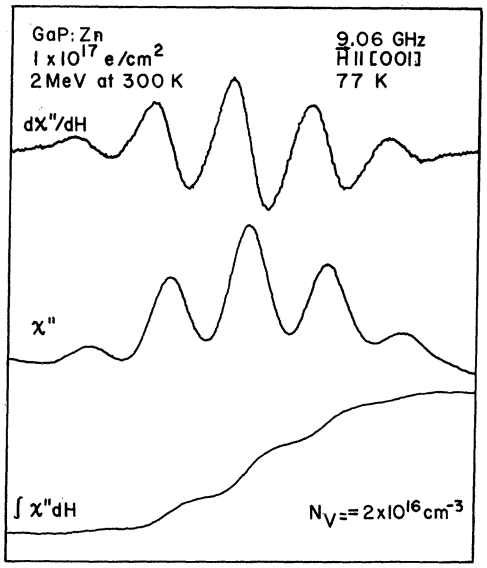

FIG. 1. Digitized EPR data used to determine the vacancy concentration. Signal-averager output of the spectrum, dx''/dH; first integral, x''; and second integral, $\int x''dH$, are shown. Comparison of the second integral value to that for a standard sample provides the concentration.

III. RESULTS

A. EPR spectrum and defect identification

Following electron irradiation, an EPR spectrum, designated NRL-1, is dominant in GaP:Zn. This spectrum has been studied at low temperatures and requires near-band-gap optical excitation in order to be observed.[10] The essential features are a partially resolved anisotropic hyperfine (hf) structure and an isotropic g-value. The five-line spectrum with the magnetic field H along the [001] axis has intensity ratios 1:4:6:4:1 which indicates the interaction of a spin-$\frac{1}{2}$ electron with four equivalent spin-$\frac{1}{2}$ nuclei. This spectrum can be described by the spin Hamiltonian

$$\mathcal{H} = g\mu_B \vec{H}\cdot\vec{S} + \sum_{I=1}^{4} \vec{S}\cdot\underline{T}^I\cdot\vec{I}^I, \tag{1}$$

where the first term represents the Zeeman interaction and the second term describes a ligand hf interaction with the four equivalent neighbors. The spin Hamiltonian parameters at K band at 4.2 K are

$$g = 2.0130 \pm 0.0015,$$

$$T_{\parallel} = (104\pm3)\times10^{-4}\ \text{cm}^{-1},$$

$$T_{\perp} = (41.5\pm1.5)\times10^{-4}\ \text{cm}^{-1},$$

$$\Delta H = 40\ \text{G},$$

where ΔH is the full width of the EPR lines, and $T_{\parallel}$ and $T_{\perp}$ are the diagonal elements of $\underline{T}$ along the principal axes.

The tetrahedral coordination of the defect with $I=\frac{1}{2}$ nuclei, which is necessary to explain the spectrum, unambiguously indicates that the defect is centered at one of the two lattice positions which are surrounded by a tetrahedron of the (100% abundant $I=\frac{1}{2}$) ^{31}P atoms. These are the Ga site and an interstitial site. Analysis of the hf parameters using a molecular-orbital model shows that a symmetric relaxation of the nearest neighbors takes place.[10] The wave function on each neighbor was found to be 8% s like and 92% p like with 88% of the total wave function residing on the four nearest neighbors.[14] Following some simple orthogonality considerations,[15] it is estimated that the nearest neighbors relax 14% of a bond length away from the defect center.

Arguments were given in Ref. 10 which assigned

the spectrum to the Ga vacancy. These arguments are expanded upon in this paper. First, the spectrum is observed in irradiated material only and the signal intensity increases with electron fluence. The strong localization of the defect wave function on the nearest neighbors along with the sign and magnitude of the shift from the free-electron g value are consistent with a center involving broken bonds.[16] The g shift is also consistent with the values for V_{Zn} in ZnSe (Ref. 17) and V^+ and V^- in Si.[18,19] The absence of a central hf splitting suggests the V_{Ga}.

Some additional consideration of an alternate defect assignment can be made. As will be shown in a later section, defect concentrations of up to $7\times10^{16}\,\mathrm{cm}^{-3}$ have been achieved by electron irradiation. Mass spectroscopy of the starting material by Metals Research indicates that there are six impurities, Zn, O, S, As, Si, C with concentrations greater than $2.5\times10^{16}\ \mathrm{cm}^{-3}$, allowing for some error in concentration measurements. Zn, the intentional dopant, can be ruled out as the source of the present spectrum since its EPR is known.[20] Oxygen and sulfur, from group VI of the Periodic Table, are unlikely to appear on the Ga sublattice. Arsenic on the Ga site would be similar to P_{Ga}, which has been observed through EPR.[9] Because there is no central hf splitting (As has $I=\frac{3}{2}$), As is ruled out. The EPR of Si on a Ga site has been studied.[21] Carbon is not known to occupy the Ga site in GaP.[20,22,23] Infrared absorption studies indicate that C on the P site changes with electron irradiation to a trigonal center involving C.[24] The EPR of the other group-IV elements Si, Ge, Sn on the Ga site has been studied[21,25,26] and the results are similar. All three elements exhibit small, *negative* g shifts with no resolved hf structure. Since the EPR of C on the Ga site, if it occurs, would be similar to that of Si, Ge, and Sn, and since the ir study[24] finds a trigonal center, C is ruled out. Therefore, the assignment of the new spectrum to the Ga vacancy is well founded.

B. Electronic structure

Features of the vacancy electronic structure can be inferred from the EPR results. In T_d (tetrahedral) symmetry the four electronic states are split into a triplet t_2 and a singlet a_1. Any partial occupation of the degenerate triplet level is unstable to a distortion to a lower symmetry according to the Jahn-Teller theorem. Such symmetry-lowering distortions take place for the different charge states of the vacancy in Si.[5] However, in the present case the isotropy of g implies a cubic symmetry and the hf structure denotes tetrahedral cubic symmetry even to 4.2 K (Ref. 10) and lower temperature (see below). In the simplest analysis,[10] the cubic symmetry implies that the charge state which gives rise to the EPR contains a single electron (an unpaired spin) in an a_1 state. If the singlet level is below the triplet, the charge state of the vacancy would be V^{4+}, which is considered unlikely. If the singlet state is above the triplet, the charge state is V^{2-}. Note that this is not the equilibrium charge state in irradiated p-type GaP.

Correlations have been observed between these EPR results and the behavior of a long-lifetime component in positron annihilation experiments on irradiated GaP:Zn.[27] The longer positron lifetime implies that the defect responsible is either neutral or negatively charged. Thus there is independent support for a negatively charged state.

In the ionic, or crystal-field, limit the proper ordering of the singlet and triplet is obvious—the singlet state above the triplet is the ordering for surrounding negative charge. In a covalent semiconductor, the proper ordering is not clear. For the d electrons of transition elements in Si, the successful model has the ordering of the triplet (t_2) and doublet (e) different depending on whether the impurity occupies the interstitial or substitutional site.[28] A physical picture of this reversal has been proposed.[29] Theoretical studies of group-III lattice vacancies in III-V semiconductors predict that the singlet is well below the triplet.[30-32] Since there is disagreement between the simplest analysis and the recent theories, it is appropriate to consider other models for the electronic structure.

It has been suggested[33] that the isotropy of the vacancy spectrum is due to dynamic Jahn-Teller effect; that is, the vacancy is rapidly reorienting between different distortion modes. The electronic structure would have a singly occupied t state uppermost in energy. This possibility is difficult to confirm. Additional experiments were performed to pumped He temperatures to see whether a static distortion would occur. The identical spectrum was observed at 1.4 K. Spin-lattice relaxation was fast enough that absorption spectra could be obtained with about 10 μW incident on the cavity. The fast spin-lattice relaxation may in itself be taken as some evidence for a dynamic Jahn-Teller effect. However, it appears that the spin-lattice relaxation is generally faster in the wide-band-gap III-V's than in Si. The antisite spectrum was observed with the vacancy spectrum at 1.4 K although, for the same power, it was somewhat saturated. For Cr in GaAs, a known Jahn-Teller system, a full 20 mW produces only slight saturation at 1.8 K.[34] Thus the moderate spin-lattice rate for the vacancy does not offer

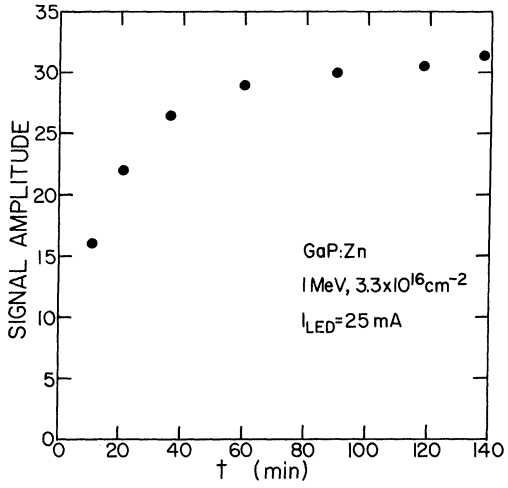

FIG. 2. Time dependence of the photoexcitation of the vacancy signal.

strong support for a dynamic Jahn-Teller description.

Further experiments and/or better theoretical understanding are required to understand the electronic structure.

C. Optical excitation in *p*-type samples

Following irradiation of *p*-type samples, optical excitation is required before the vacancy EPR spectrum is observed. This indicates that the charge state which exhibits EPR is not the equilibrium state. The photoexcitation process involves charge exchange between the vacancy and an unknown trap. Detailed descriptions of the possible processes have been given.[35,36] Generally, one does not know whether the parameters of the process are characteristic of the trap which exhibits EPR or the unknown trap.

The photoexcitation of the vacancy EPR takes place very slowly, over a period of two hours (see Fig. 2). The excitation is slow both at 77 and 4.2 K. It does not matter whether the sample is lightly irradiated, such that the Fermi level is still at the acceptor level, or heavily irradiated, such that the sample is compensated. The P_{Ga} antisite[9] can be photoexcited in two *p*-type samples before or after low-fluence electron irradiation. Its excitation is rapid—taking a few minutes or less. Since both the antisite and the vacancy acquire additional negative charge in the photoexcitation, they must be exchanging charge with the same unknown and omnipresent trap. Thus the slow photoexcitation is attributed to the vacancy—indicating either a small electron-capture cross section or a small photoionization cross section.

Two other aspects of the photoexcitation are important. From a 77-K study only light with wavelength shorter than 750 nm is effective in producing the spectrum. There is also an increase in the optical absorption in the near-band-gap range which accompanies the electron irradiation[37] (see Fig. 3). The change in optical density of even a 0.5-mm-thick sample is significant.

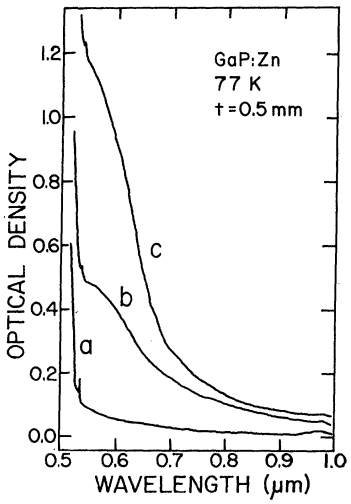

FIG. 3. Optical density of an EPR sample in the near-band-gap region. Data are shown for the sample as grown (a) and following 2-MeV irradiations of 1.7×10^{16} cm^{-2} (b), and 3.3×10^{16} cm^{-2} (c).

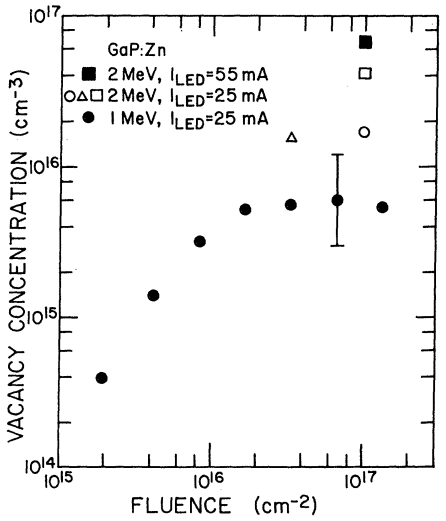

FIG. 4. Vacancy concentration versus electron fluence for *p*-type samples. The absolute error is ±100%. Comparisons between points can be made to greater precision, ±5%. Data for two electron energies and two photoexcitation intensities are shown. The optical darkening affects the photoexcitation for fluences greater than 10^{16} cm^{-2}. Different starting materials are indicated by different symbols.

This must be recognized in trying to study quantitatively heavily irradiated samples.

D. Defect introduction in *p*-type samples

The vacancy is introduced at a high rate (~1 cm^{-1}) for room-temperature electron irradiation of *p*-type GaP (see Fig. 4). The filled circles depict a systematic 1-MeV study of a Zn-doped sample with room-temperature hole concentration of 2×10^{17} cm^{-3}. The sample was irradiated on a (111) face. The same face was irradiated in each subsequent step. EPR data were taken at 4.2 K in the *K*-band system. For fluences below 2×10^{16} cm^{-2} the sample remains conducting at high temperatures and the optical darkening is negligible. In this range the introduction rate is linear with fluence. At higher fluences, the sample is no longer conducting and the vacancy concentration appears to saturate. However, this can be accounted for by the darkening of the sample.

Selected results from 2-MeV irradiations on other Zn-doped samples are also shown. Room-temperature carrier concentrations for these samples are also in the low to mid 10^{17} cm^{-3} range. The higher-energy electrons produce greater vacancy concentrations as one would expect. Note that the EPR signal increases when LED current, and therefore light intensity, is increased. Thus the condition of optical saturation has not been achieved. Thus the introduction rates indicated by the 25-mA data are smaller than the actual rates.

E. Dependence on temperature and on sample type

Following optical excitation the vacancy spectrum has been observed in irradiated *p*-type samples from 1.4 to about 150 K. At 4.2 K and below, the signal persists after the light excitation is removed. At 77 K and above, a complicated decay takes place (see Fig. 5). At 130 K it consists of a fast and a slow component. The decay becomes more rapid for $T > 130$ K until no signal can be seen at 150 K. Further work is underway to see whether the energy level might be found from such experiments, as was the case for V^+ in Si.[5]

Since the *p*-type samples are compensated by electron irradiation,[38] the Fermi level changes from the Zn level position to the center of the gap with electron fluence. Optical excitation is required throughout in order to observe the EPR. Thus the occupancy level for the EPR active charge state is either below the Zn level or in the upper half of the energy gap.

The vacancy spectrum has been observed at 77 K in the dark in irradiated Te-doped ($n = 1.5$ $\times10^{17}$ cm^{-3} at 300 K) and undoped ($n = 1.7\times10^{16}$ cm^{-3}) samples. 2-MeV irradiations of (1–2) $\times17^{17}$ cm^{-2} produced only very weak signals. Optical excitations produced little or no change in signal strength. Smaller fluences produced no detectable vacancy signal. Thus it appears that the *isolated* vacancy is not an important defect in *n*-type GaP.

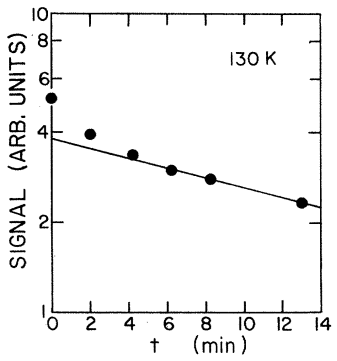

FIG. 5. Signal decay in the dark versus time at 130 K. Two time constants are apparent from the data.

F. Defect annealing

Isochronal annealing studies were performed on *p*-type samples. For the data shown in Fig. 6, a *p*-type sample was irradiated with 1-MeV electrons to a fluence of 3×10^{16} cm^{-2}, thus avoiding the serious optical absorption present from higher fluences. This fluence is sufficient to compensate the sample and the change in Fermi level is evident from the presence of the antisite signal in the dark. The EPR amplitude following one hour of optical excitation was measured following 15-min isochronal anneals. After the 300 °C anneal, optical excitation is again required in order to see the

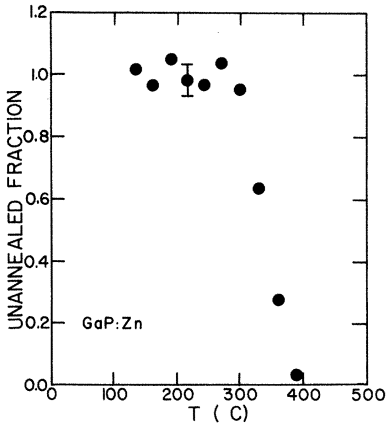

FIG. 6. Isochronal annealing study. Fifteen-minute anneals were made. The concentration is reduced to less than 5% of its original value by 390 °C.

antisite. The amplitude of the vacancy EPR decreases to 5% in the anneals from 300 to 390 °C. A small isotropic signal near $g=2$ which is present throughout the earlier annealing grows larger following the 360 and 390 °C anneals. Because of the presence of the optically excited antisite signal after the vacancy has annealed, the disappearance of the EPR is indicative of the disappearance of the vacancy (see subsection C).

A similar study using different p-type starting material showed annealing at 300 °C, somewhat lower than the data of Fig. 6. Thus the annealing process may involve long-range diffusion of an impurity or depend on impurities.

Radiation-induced defects in GaP were found to exhibit enhanced annealing under conditions of strong recombination—as in a forward-biased diode.[39] Thus change in the vacancy signal was studied following strong laser excitation at room temperature. An irradiated p-type sample was illuminated for 25 minutes with the 0.3-W, 647.1-nm line and for 50 minutes with the 0.15-W, 568.2-nm line of a krypton laser. The signal following the laser irradiation was 1.4 times the original signal—a "reverse annealing" effect. Apparently the vacancy does not undergo an enhanced annealing. The increased signal could arise from the breakup of vacancy complexes either due to a direct interaction with the light or in a recombination-enhanced process.

IV. OTHER SPECTRA

The preceding sections have described the properties of the Ga vacancy as studied through its EPR spectrum. Other spectra have been observed during these investigations and a brief discussion of them is presented here.

A spectrum which has been attributed[11] to a P on a Ga-site defect paired with an impurity in the nearest-neighbor shell is observed in irradiated n-type samples. Parameters obtained from simulating the data, but neglecting the small anisotropy in neighbor hf interaction, are given in Table I. This spectrum has been observed in derivative of absorption mode for temperatures from 20 to 300 K.

At 4.2 K in heavily irradiated n-type material, a new spectrum is observed designiated as GaP-NRL-3 in Table I. Excellent signal to noise has not been achieved for this spectrum and its parameters are only approximate. All parameters are isotropic. No defect model is proposed at the present time.

Samples from four Zn-doped crystals have been studied. Two exhibit the P antisite[9] in as-grown condition and two do not. Following irradiation the P_{Ga} antisite is observed in all samples from all p-type crystals. Although it is possible that the appearance takes place due to a Fermi-level change or photoexcitation trap change, the antisite is most likely a radiation-induced defect in p-type GaP. Since there are P interstitials and Ga vacansies produced in the irradiation process, high P_{Ga} production is quite plausible.

In an irradiated p-type sample a narrow line designated GaP-NRL-4 is observed near $g=2$. This line becomes stronger after the 360 and 390 °C anneals as described in Sec. III F. The narrow linewidth and annealing behavior suggest that this line may arise from a precipitate.

V. SUMMARY

A description of the conditions under which the EPR spectrum attributed to the Ga vacancy in GaP is observed has been given. The identification of the defect as the vacancy stands following an analysis of impurity defect models. Further

TABLE I. Defects in electron-irradiated GaP.

Spectrum	Model	G value	Hyperfine interactions ($\times 10^{-4}$ cm^{-1}) Central	Neighbor
GaP-NRL 1	Ga vacancy	2.0130	None	Four $I=\frac{1}{2}$, $T_{\parallel}=104$ $T_{\perp}=41.5$ ([111] axis)
GaP-NRL 2	P_{Ga}-X pair	2.006	$I=\frac{1}{2}$ 704	Three $I=\frac{1}{2}$ $T=109$ (Slight anisotropy)
GaP-NRL 3	?	2.01	$I=\frac{1}{2}$ 700	One $I=\frac{1}{2}$ $T=225$
GaP-NRL 4	?	2.003	None	None

discussion of the question of lack of a distortion and of the defect charge state was presented. The isolated vacancy is introduced at a high rate when p-type GaP is electron irradiated. The vacancy thermally anneals between 300 and 400 °C. In contrast to many defects seen in DLTS,[39] the isolated vacancy exhibits reverse annealing after laser excitation. The energy level for the EPR-active charge state is in the upper half of the band gap. Parameters of other spectra observed during the study of the vacancy have been presented.

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