Optically detected magnetic resonance studies of neutron-transmutation-doped GaP

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(Received 20 April 1990; accepted for publication 17 September 1990)

A direct proof of neutron transmutation doping (NTD) of GaP is presented on the basis of optically detected magnetic resonance (ODMR). GaP:S samples grown by the liquid-encapsulated Czochralski method were irradiated with thermal neutrons and subsequently annealed at 800 °C. In the ODMR experiments the transmuted Ge substitutional on Ga sites was detected. The NTD process was also found to create deep acceptors; these are tentatively identified as associates of gallium vacancies (V_{Ga}) and germanium donors on gallium sites (Ge_{Ga}). Such identification requires that some of the structural defects (vacancies) created by β and γ recoil during transmutation are stabilized by forming V_{Ga}- Ge_{Ga} complexes.

I. INTRODUCTION

Neutron transmutation doping (NTD) is a well-established method for *n*-type doping of Si. The quality of high-power electronic devices was highly improved due to doping homogeneity and low defect concentration of the NTD Si. Much less attention was paid to transmutation doping of III-V compounds. For GaP, to our knowledge, only one investigation has been reported on NTD GaP (Ref. 1) and two on radiation defects related to fast neutron irradiation of GaP.[2,3] Until very recently no definite proof existed of successful NTD of GaP in which Ge donors are expected to be formed. In the work of Huber and co-workers[1] a rather small neutron fluence was applied and consequently the conductivity of the transmuted and annealed samples was dominated by the electron emission processes from shallower Te donors. Therefore, the authors could only indirectly conclude on NTD of GaP from the fact that their Hall measurements could not be fully fitted when the existence of only one type of donor species (Te) was assumed.

The photoluminescence (PL) and optically detected magnetic resonance (ODMR) studies presented in this paper are performed on GaP material irradiated with a relatively large fluence of thermal neutrons (1.4×10^{19} cm^{-2}). After annealing at 800 °C the irradiated samples showed electrical conductivity related to thermal electron emission from a deep donor with an ionization energy of 200 ± 10 meV, as measured by Barczynska and Goldys.[4] This ionization energy value is in good agreement with the value of 204 meV reported for GaP conventionally doped with Ge, indicating successful NTD.

The present study had a twofold aim. First, we wanted to show that after irradiation and annealing the transmuted Ge enters the Ga site, forming a Ge_{Ga} donor state. Second, we intended to confirm the observation of Huber *et al.*[1] that NTD introduces also some acceptor states which are not annealed out even at 800 °C . The latter fact makes NTD for GaP less advantageous (as compared to NTD of Si).

To achieve these objects the ODMR technique was used. ODMR is a very powerful technique for studying deep donor-acceptor pair (DAP) recombination processes by observing the relevant donor and, in some cases, acceptor resonance signals. It will be shown that due to a larger ionization energy and a more localized character the germanium donor (Ge_{Ga}) resonance signal can easily be distinguished from the signal of the shallower sulfur donor (S_P) state, in spite of their nearly identical *g* factors.

Following this brief introduction, basic information on the NTD process is given in Sec. II and the experimental details are described. Results are presented in Sec. III. In the discussion (Sec. IV) the ODMR data are analyzed. The model for the recombination process is presented and followed by a closer examination of the nature of the deep acceptor centers introduced by NTD.

II. EXPERIMENT

A. NTD process

The basic nuclear reactions for NTD of GaP were given by Huber *et al.*[1] Capture of thermal neutrons by Ga and P (with capture cross sections σ_{th}) transmutes both these nuclei according to the following reactions (the half-life times are given in parentheses):

$$^{69}\mathrm{Ga}(n,\gamma)\rightarrow{}^{70}\mathrm{Ga}(\beta^-)\rightarrow{}^{70}\mathrm{Ge}\ (21\ \mathrm{min}),$$

$$\sigma_{th}=1.68\pm0.07\ \mathrm{b},$$

$$^{71}\mathrm{Ga}(n,\gamma)\rightarrow{}^{72}\mathrm{Ga}(\beta^-)\rightarrow{}^{72}\mathrm{Ge}\ (14\ \mathrm{h}),$$

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$\sigma_{th}=4.86\pm0.25$ b,

$^{31}P(n)\rightarrow{}^{32}P(\beta^-)\rightarrow{}^{32}S$ (14.3 d),

$\sigma_{th}=0.180\pm0.007$ b.

If the thus formed Ge atoms are substitutional on Ga sites and the S atoms on P sites, transmutation results in creating donor states in GaP.

Capture cross sections of the above reactions are negligibly small for fast neutrons. This means that irradiation with fast neutrons will produce mainly lattice defects (antisites, vacancies, interstitials, and complexes of these) without observable transmutation effects.[2] For this reason the ratio of fast to slow neutrons in our experiment was as small as possible. Due to the low concentration of fast neutrons, radiation defects created by NTD are due to β and γ recoil during transmutation. As a result of the recoil after irradiation the majority of the transmuted Ge and S dopants are in interstitial positions. Subsequent annealing is then necessary to render them substitutional. In our experiments the annealing procedure of Huber *et al.*[1] was followed (800 °C, 1 h).

Knowing the thermal neutron capture cross sections the density of transmuted lattice atoms can be calculated:[1]

$$N_{Ge}(cm^{-3})=7.7\times10^{-2}\times\Phi_{th}(cm^{-2}),$$

$$N_S(cm^{-3})=4.7\times10^{-3}\times\Phi_{th}(cm^{-2}),$$

where Φ_{th} is the thermal neutron fluence. In our experiment a 1.4×10^{19} cm^{-2} fluence was used, yielding the following Ge and S concentrations:

$$N_{Ge}=1.1\times10^{18}\ cm^{-3},$$

$$N_S=7\times10^{16}\ cm^{-3}.$$

These numbers are accurate within 5% due to the uncertainties in the capture cross section values. Because the concentration of transmuted S atoms is more than one order of magnitude smaller than the concentration of transmuted Ge, GaP is expected to be essentially doped with Ge. It should be noted that the relatively long decay time of the $^{32}P(\beta^-)\rightarrow{}^{32}S$ reaction poses a practical limitation on the wider application of NTD for *n*-type doping of GaP, since the transmuted samples cannot be radioactive.

B. Experimental details

The starting material was commercially available *n*-type GaP:S grown by the liquid-encapsulated Czochralski (LEC) method. The doping level prior to irradiation was 7×10^{17} cm^{-3}. Neutron irradiation was performed at the SWIERK Nuclear Research Center in Poland with the ratio of thermal to fast neutrons being 1000:1. The thermal neutron fluence was 1.4×10^{19} cm^{-2}. After irradiation the samples were vacuum annealed for 1 h at 800 °C. Hall-effect measurements performed by Barczynska and Goldys[4] showed *n*-type sample conductivity with the thermal activation energy of 200 ± 10 meV, which agrees well with the value of 204 meV characteristic for the Ge donor. The S donor level was not occupied.

Measurements were performed at 4.2 K (PL) or 2.1 K (ODMR). Luminescence was excited with a cw Ar^+-ion laser operating at 514.5 nm. To avoid spurious plasma lines, a 514.5-nm interference filter was used. The emerging luminescence was collected from the laser-irradiated side. It was dispersed by a high-resolution 1.5-m *F*/12 monochromator (Jobin-Yvon THR-1500) with a 600-grooves mm^{-1} grating blazed at 1.5 μm and detected by a nitrogen-cooled Ge detector (North Coast EO-817). The detector output was amplified using conventional lock-in techniques. The lock-in output was digitized and fed into a computer for further data acquisition. The computer also regulated the monochromator and the magnet power supply. Optical filters were placed in front of the entrance slit of the monochromator to select the emission bands of interest.

The ODMR experiments were carried out at 35 GHz, using a split-coil superconducting magnet (Oxford Instruments SM 4) in the Faraday configuration. Changes of emission intensity were detected with the monochromator set to zeroth order. A Gunn oscillator provided microwave power (maximum 230 mW) to a cylindrical TE_{011} cavity with slits for optical access. Microwaves were on-off modulated, typically at 730 Hz. The sample could be rotated to measure the angular dependence of ODMR signals.

III. RESULTS

Four different samples, labeled (a)–(d), were used for the PL and ODMR experiments. They are (a) the starting material LEC GaP:S, (b) the starting material annealed in vacuum for 1 h at 800 °C, (c) the as-irradiated sample, and (d) the irradiated sample which was further vacuum annealed for 1 h at 800 °C. A surface layer of about 50 μm was removed from the irradiated samples before measurements.

In Fig. 1 the PL spectra for the four crystals specified above are presented. The spectra are not corrected for the instrumental response. The PL spectra of samples (a) and (b) differ slightly in their relative ratios of edge and deep emission. In addition, the low-energy part of the PL spectrum of the annealed sample (b) is dominated by the 1.7-eV emission, whereas the 1.5-eV PL is stronger for the starting material (a). The latter observation is in agreement with previous data.[5,6] The as-irradiated material (c) was nontransparent and only a very weak emission was observed peaking at 1.25 eV. After annealing [sample (d)] this 1.25-eV band contributed to the low-energy wing of the PL otherwise dominated by the 1.52-eV emission. The relative intensity of the 1.25- and 1.52-eV PL bands depended on the excitation spot. This effect could be explained by a small inhomogeneity of the irradiated and annealed material which relates to a larger inhomogeneity of the starting LEC material.

The ODMR spectra, measured via monitoring the change of the total PL intensity in resonance, are shown in Fig. 2. The characteristic sulfur donor resonance signal with an isotropic value of $g=1.99\pm0.01$ is observed for

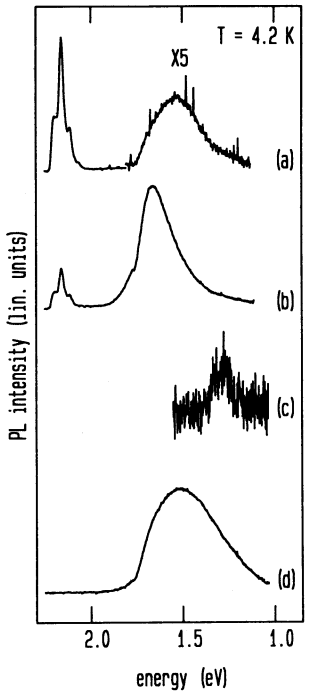

FIG. 1. Photoluminescence (PL) spectra of (a) the starting material LEC GaP:S, (b) the starting material annealed in vacuum (800 °C, 1 h), (c) the as-irradiated sample, and (d) the irradiated sample which was subsequently vacuum annealed (800 °C, 1 h). Spectra were recorded at 4.2 K through a 1.5-m monochromator within the range of the Ge detector using the 514.5-nm Ar^+ line to excite the samples.

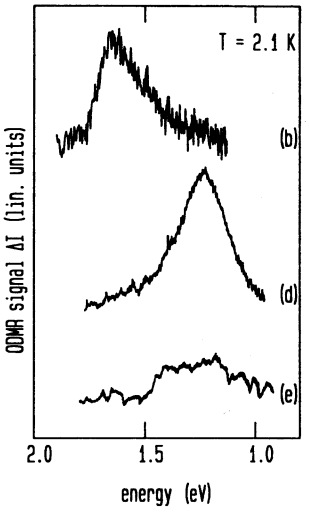

FIG. 3. Spectral dependence of the donor ODMR signals for (b) the starting material LEC GaP:S annealed in vacuum (800 °C, 1 h), (d) the irradiated sample which was subsequently vacuum annealed (800 °C, 1 h), and (e) GaP:Ge. Spectra (b) and (d) are measured at 2.1 K through a 1.5-m monochromator; spectrum (e) is shown for easy comparison and has previously been measured by one of the authors (see Ref. 6) on GaP conventionally doped with Ge.

samples (a) and (b). At slightly lower field an overlapping acceptor-related signal is observed. It is too weak to allow for determination of the acceptor-state symmetry. A similar spectrum has previously been observed for S-doped GaP by one of the authors.[6] As a consequence of the very weak PL, no ODMR spectrum could be observed for the as-irradiated material [sample (c)]. For sample (d) a new isotropic resonance signal was found. This signal has $g = 2.00 \pm 0.01$ and is much broader (53 mT) than the sulfur donor resonance. The identical resonance spectrum

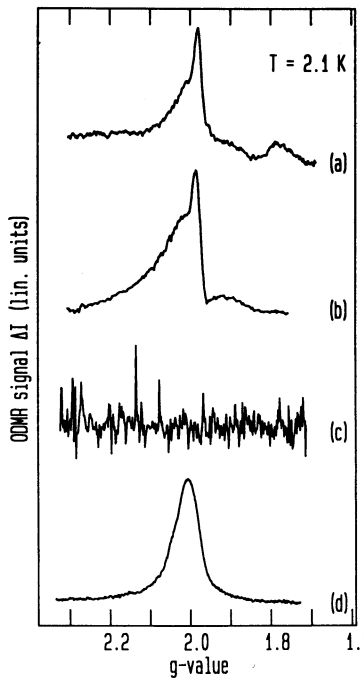

FIG. 2. ODMR spectra measured at 2.1 K of (a) the starting material LEC GaP:S, (b) the starting material annealed in vacuum (800 °C, 1 h), (c) the as-irradiated sample, and (d) the irradiated sample which was subsequently vacuum annealed (800 °C, 1 h). Changes of the total infrared emission (ΔI) under 514.5-nm Ar^+ excitation are plotted against the g value.

was observed for conventionally doped LEC GaP:Ge.[6] Consequently, we identify this signal with the substitutional Ge donor center. No acceptor-related resonance was observed by ODMR for sample (d).

The spectral dependence of the ODMR signals was investigated by setting the magnetic field to resonance and scanning through the luminescence. In this way the emission change is recorded as a function of wavelength. The relevant experimental data are plotted in Fig. 3. For samples (a) and (b) the donor signal is observed on the 1.5–1.6-eV PL, in agreement with previous experimental observations.[6–8] For NTD material [sample (d)] the observed donor ODMR signal is an enhancement of the 1.25-eV emission. This observation agrees with recent ODMR studies of LEC GaP:Ge,[6] as shown in Fig. 3 for easy reference. The 1.52-eV PL dominant for the NTD sample shows only a very weak ODMR signal identical to the sulfur donor resonance observed for samples (a) and (b). This implicates that the S-related 1.5-eV DAP transition contributes to the 1.52-eV band in NTD material. At the same time the low intensity of the ODMR signal suggests that the 1.52-eV band arises from another recombination process which is not "ODMR active." It is probable that the 1.52-eV PL in sample (d) is of the same origin as the 1.7-eV emission observed for the starting material (a).

IV. DISCUSSION

A. ODMR spectrum

The observation of the isotropic $g = 2.00 \pm 0.01$ resonance for NTD GaP after 800 °C isochronal vacuum annealing directly confirms the successful transmutation of Ga into Ge. The signal is identical to the one observed for GaP conventionally doped with Ge, both in ESR[9] and ODMR.[6]

The group-IV shallow donors substituting for Ga (Si, Sn) differ in their properties from group-VI donors substituting for P(S,Te). The explanation for this has been given by Morgan[10] and is shown in Fig. 4. With respect to the Ga site the GaP conduction-band minimum transforms as X_3.

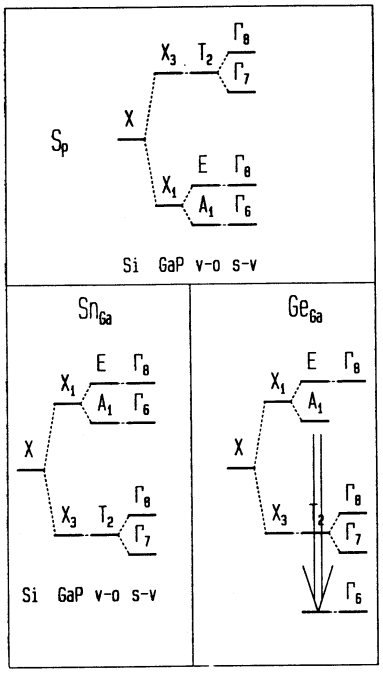

FIG. 4. Energy-level scheme after Morgan (see Ref. 10) of group-IV shallow donors substituting for Ga, e.g. Sn_{Ga}, and group-VI donors substituting for P, e.g. S_P, including valley-orbit (v-o) and spin-valley (s-v) interaction. The strikingly different behavior of the Ge_{Ga} donor as compared to other group-IV donors is clearly indicated. A plausible explanation has been given by Mehran *et al.* (see Ref. 9) and is described in Sec. IV A.

The degeneracy is not lifted by valley-orbit but by spin-valley interaction which splits the orbital triplet T_2 into Γ_7 and Γ_8. The sign of this splitting depends on the difference between the atomic spin-orbit coupling of the donor and the host atom it replaces. The splitting shown in Fig. 4 corresponds to the case of Sn_{Ga} ($\Delta E = +2.1$ meV). The level ordering is reversed for Si_{Ga} ($\Delta E = -0.49$ meV).[11]

The ground states of the Sn_{Ga} and Si_{Ga} donors thus have T_2 symmetry and could be detected in ESR only under large uniaxial stress.[12,13] The situation is remarkably different in the case of the Ge_{Ga} donor. The center behaves like the chalcogenide donors and its isotropic resonance with $g = 2.000 \pm 0.003$ was detected in ESR without uniaxial stress.[9] A plausible explanation is shown after Mehran *et al.*[9] in Fig. 4. The local impurity potential draws down the orbital singlet A_1 below the T_2 state resulting in Γ_6 symmetry of the Ge_{Ga} ground state. The situation is then similar to, e.g., the shallower group-VI donor S_P substituting for P [$E_D = 107$ meV for S_P, $E_D = 204$ meV for Ge_{Ga} (Ref. 11)].

The larger ionization energy of the Ge_{Ga} donor, compared to S_P, results in a more localized electron wave function, which may account for the broadening of the ODMR signal observed for NTD GaP (see Fig. 2). The larger half-width of the Ge_{Ga} donor resonance relates then to the unresolved hyperfine interaction with the four nearest P ($I = 1/2$) neighbors. This is indeed supported by ENDOR measurements of Hausmann and co-workers.[14,15] From their ENDOR data the wave-function localization on the nearest-neighbor shell is estimated to be a factor of 2.8 larger for the Ge_{Ga} donor than for the S_p donor. The hyperfine interaction with P ligands was resolved only for deep Ga site centers such as the Ga vacancy[16] and the P_{Ga} antisite.[17]

B. Recombination transitions

The observation of the Ge_{Ga} donor signal as an enhancement of the 1.25-eV PL band proves the donor-acceptor pair (DAP) nature of this emission. The corresponding acceptor signal was not observed but such a situation is not uncommon in ODMR studies. Since the Ge_{Ga} donor ionization energy is known ($E_D = 204$ meV) the PL and ODMR data indicate that following neutron irradiation and 800 °C annealing of GaP a new deep acceptor state is introduced. The nature of this deep acceptor will be discussed in the next section.

The dominating 1.52-eV PL for NTD GaP is not "ODMR active." Therefore, it cannot be due to, e.g., Ge_{Ga} donor-to-shallower acceptor DAP process and must be of a different origin. We propose that this emission is due to bound-exciton recombination at a neutral complex. This proposition should, however, be considered as a tentative one since it cannot be supported by sufficient evidence. Some underlying S_P-related DAP emission contributes to the 1.52-eV band, as will be discussed later.

Germanium in III-V compounds is an amphoteric dopant, i.e., it can enter the cation site forming a donor state or the anion site creating an acceptor level. Such behavior of germanium was reported previously for NTD GaAs from PL studies.[18–20] Ge on a P site in GaP is a relatively shallow acceptor state with the energy level at $E_V + 258$ meV.[11] Near-edge emission should thus be detected if some of the transmuted Ge atoms enter P sites and become active in a Ge_{Ga}-Ge_P DAP process. This is not confirmed by our PL and ODMR studies, supporting the conclusions of Huber *et al.*[1] for NTD GaP and Satoh *et al.*[20] for NTD GaAs. The latter authors concluded that for GaAs more than 98% of the transmuted Ge enters Ga sites, gaining donor activity.

A possible explanation for the PL observed would be the following. In all spectra of Fig. 1 traces of a zero-phonon line at about 1.75 eV may be seen. If we relate this line to a conduction band-to-acceptor transition, this would place the acceptor at $E_V + 0.61$ eV ($E_G = 2.36$ eV for GaP at 4.2 K). After Ge_{Ga} donors ($E_D = 0.204$ eV) are introduced into the sample during transmutation and annealing, a DAP transition is expected to be seen at 1.55 eV, which is indeed the case [see Fig. 1 (d)]. To account for the 1.25-eV PL observed in the ODMR spectral dependence [Fig. 3 (d)], another state at $E_V + 0.91$ eV would then be required.

However, this model appears oversimplified since it cannot explain the dependence of the DAP PL with respect to the donor ionization energy, as has been observed in GaP doped with S, Se, Te, and Ge by Dishman *et al.*[5] and by one of the authors.[6,8] Hence, a more complex model will be given in the following section.

C. Deep acceptors active in DAP recombination

The nature of the relevant deep emissions in GaP was studied first by Dishman and co-workers.[5] Two broad featureless PL bands at 1.5 and 1.7 eV were attributed by

them to a DAP transition and bound exciton recombination at a neutral complex, respectively. To explain the nonmonotonic dependence of the 1.5-eV PL on the donor (S, Se, Te) ionization energy it was proposed that the donor impurity also participated in the formation of an acceptor. The deep acceptor (at $\approx E_V + 0.7$ eV) active in the 1.5-eV DAP transition was tentatively identified as a complex incorporating a gallium vacancy (V_{Ga}) and two donors (e.g., V_{Ga}-$2S_P$). The V_{Ga}-3 donor complex was tentatively proposed as the neutral complex binding an exciton.[5]

Another model for the 1.5- and 1.7-eV emissions has recently been put forward by Ishikawa *et al.*[21] on the basis of their PL and ODMR data and recent studies of Shibata *et al.*[7] The latter authors observed that photoexcitation of the 1.5-eV PL initial state quenches it and simultaneously enhances the 1.7-eV PL. This observation suggests that both PL bands are related and may be due to DAP processes: the 1.5-eV PL to a transition between a shallow donor and a deep double acceptor in its singly negative charge state, and the 1.7-eV PL to a similar transition but with the acceptor in its neutral charge state.[7,21] The first process should be observed in ODMR, whereas in the second case the spins of the two holes are antiparallel and therefore prior to recombination the acceptor is diamagnetic. The lack of ODMR donor signal for the 1.7-eV PL[6,8,21] is consistent with such a model. The identity of the double acceptor is unknown, although it was proposed by Ishikawa *et al.*[21] to be the anion antisite (Ga_P).

However, the model of Ishikawa *et al.* is not consistent with the work of Dishman *et al.*[5] and recent ODMR experiments of one of the authors.[6,8] From these studies it is evident that the acceptor energy depends on the actual donor impurity which is introduced into the sample. This favors the V_{Ga}-donor complex model, but no direct proof exists to support such an assignment.

The distinct shift of the two PL bands for Ge-doped material (as observed for both conventional[6] and transmutation doping) from 1.5 and 1.7 eV to 1.25 and 1.52 eV, respectively, can also be accounted for by the model of Dishman *et al.*[5] The shift of both emission bands of approximately 200–250 meV is by a factor of 2 larger than the one expected to result from the change of donor ionization energy only. This confirms previous observations that the position of the acceptor level depends on the donor impurity. The 1.52-eV band is most probably of the same origin as the 1.7-eV PL observed for GaP doped with chalcogenides. Even though its energy coincides with the 1.5-eV PL observed for S-, Se-, and Te-doped samples, our ODMR results clearly prove that it is of another origin. The resonance signal due to sulfur-related 1.5-eV DAP emission is observed only very weakly, giving clear evidence that the 1.52-eV band of the NTD sample is not due to the 1.5-eV DAP transition as in S-, Se-, or Te-doped GaP.

Finally, we would like to point out that the $\approx E_V + 0.7$ eV (GaP:S) and $\approx E_V + 0.9$ eV (NTD GaP) acceptors active in the DAP process studied with ODMR are not related to the two $E_V + 0.75$ eV and $E_V + 0.9$ eV dominant hole traps in *n*-type LPE and VPE GaP layers. The $E_V + 0.75$ eV center, which is the more prominent one of the two, displays no optical activity.[22,23] Moreover, its highest concentration in LEC bulk GaP was shown to be in the range of 5×10^{14} cm^{-3} only, which is considerably less than in our experiment. Our data indicate that both 0.7- and 0.9-eV acceptors depend on the actual donor species present in the crystal. This observation favors the Dishman model,[5] while being in clear disagreement with the properties of the $E_V + 0.75$ eV and $E_V + 0.9$ eV hole trap centers.

The fact that the dominant acceptor center includes Ge_{Ga} donors and probably also a Ga vacancy is clearly disadvantageous. First, the self-compensation of the Ge_{Ga} donors in NTD material by Ge_{Ga}-related complex acceptor centers lowers the efficiency of transmutation doping. Second, it stabilizes part of the vacancies created in the as-irradiated material by β and γ recoil processes. Previous ESR (Ref. 2) and positron annihilation[3] studies showed that gallium vacancies (V_{Ga}) and their complexes were the dominant structural defects formed by neutron irradiation. Subsequently, it was shown that annealing around 600 °C reduces the concentration of simple radiation defects like vacancies, antisites, interstitials, and their agglomerates.[3] The ESR measurements of Kawakubo and Okada[2] were first to demonstrate directly that some vacancy-related complexes were not annealed out at temperatures up to 800 °C. Therefore, it seems reasonable to assume that deep acceptor states formed in the neutron-irradiated material during vacuum annealing may be due to V_{Ga}-donor associates. In this way the V_{Ga}-S_P complex acceptors present in the starting material are replaced by V_{Ga}-Ge_{Ga} defects. This explains the low intensity of the sulfur-related 1.5-eV DAP emission in NTD material and a large discrepancy between the concentration of electrically active Ge_{Ga} donors and the Ge_{Ga} concentration in the sample estimated from the thermal neutron fluence.[4] A considerable or even a major part of the transmuted Ge_{Ga} impurities cannot be active as donors due to their participation in the formation of acceptor-type (V_{Ga}-$2Ge_{Ga}$) or neutral (V_{Ga}-$3Ge_{Ga}$) complex centers.

The model presented above accounts for all the experimental evidence gathered in this study and also comprises some of the elements from the literature on GaP. Nevertheless, since at present there is no direct information on the postulated acceptor centers, the model can only be considered as a tentative one.

V. CONCLUSION

The ODMR data confirm directly that NTD of GaP is possible and introduces Ge on Ga sites. The material irradiated with thermal neutrons and subsequently vacuum annealed at 800 °C shows an isotropic Ge_{Ga} donor resonance signal with $g = 2.00 \pm 0.01$. This signal was detected by ODMR on the 1.25-eV PL. The observation proves the DAP character of the 1.25-eV emission. The small inhomogeneity of the NTD material is probably a consequence of the inhomogeneity of the starting LEC GaP:S material.

The ODMR investigations indicate that some of the

structural defects (vacancies) are stabilized by the formation of complexes of acceptor and/or neutral character. Part of the transmuted Ge_{Ga} impurities would then be involved in such complexes. Our data show directly that during annealing following irradiation, deep acceptor states are created and that these states are not annealed out at 800 °C. We have tentatively identified these acceptors involved in the 1.25-eV DAP emission as V_{Ga}-$2Ge_{Ga}$ complexes.

ACKNOWLEDGMENT

This work received financial support from the Foundation for Fundamental Research on Matter (FOM).

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