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Infrared absorption properties of the *EL*2 and the isolated As_{Ga} defects in neutron-transmutation-doped GaAs: Generation of an *EL*2-like defect

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The *EL*2 and the isolated As_{Ga} antisite defects in neutron-transmutation-doped (NTD) GaAs were studied by using the infrared (ir) absorption technique concurrent with thermal annealing. The results show that irradiation with low thermal-neutron doses partially decomposes the *EL*2 complex in semi-insulating (si) GaAs grown by the liquid-encapsulated Czochralski (LEC) growth technique. On the other hand, a small amount of *EL*2 is generated in as-grown Ga-rich undoped *p*-type LEC GaAs. The *EL*2 defect in low-dose thermal-neutron-irradiated samples (both si and *p*-type) was found to be stable up to 850 °C. High neutron-irradiation doses, however, completely annihilate *EL*2 but generate a different *EL*2-like defect (*DL*2). The *DL*2 defect is observed after annealing the high-dose NTD samples for 6 min at 600 °C. The *DL*2 concentration is observed to be larger than that of *EL*2 in as-grown LEC si GaAs by a factor of 2.3 or higher. The photoquenching and thermal recovery properties of *DL*2 and *EL*2 defects are identical. However, the *DL*2 defect does not exhibit the same thermal stability or the zero-phonon line of the *EL*2 defect. Thermal annealing kinetics shows that *DL*2 is composed of three point defects. The residual absorption (unquenchable component) after photoquenching the *EL*2 (*DL*2) defect is interpreted as the photoionization of the isolated As_{Ga} antisite. The present results support the identification of *EL*2 as a complex defect and cast further doubt on the validity of the model which identifies *EL*2 as the isolated As_{Ga} antisite defect.

I. INTRODUCTION

Several advances have been made in the last few years toward understanding and identifying the native defect in GaAs known as *EL*2. There is a general consensus linking *EL*2 to the As_{Ga} antisite defect.[1] This correlation is mainly based on stoichiometry studies[2,3] and on electron-paramagnetic-resonance (EPR) measurements.[4,5] Indeed, the *EL*2 EPR spectrum is identical to that of the isolated As_{Ga} antisite introduced by neutron irradiation,[6–8] electron irradiation,[9] and plastic deformation.[10] There are problems associated with the identification of *EL*2 as being the isolated As_{Ga} antisite defect, however, two of them being related to (a) the question of whether the artificially introduced isolated As_{Ga} really exhibits metastability, and (b) whether or not *EL*2 is actually a double donor. In regards to point (a), neutron irradiation has been reported to introduce an isolated As_{Ga} antisite which does not show the metastability,[6,11] whereas plastic deformation seems to introduce isolated As_{Ga}, which does.[10] The isolated As_{Ga} antisite introduced by electron irradiation has been reported as both having metastability[9,12] and not having it.[13] Theoretically, there is also controversy over whether or not a point defect such as As_{Ga} can exhibit metastability, about which more will be said later. As far as point (b) is concerned, there appears to be strong experimental evidence indicating that the isolated As_{Ga}, whether grown in or artificially introduced, is a double donor with energy levels at about 0.75 and 1.0 eV below the conduction band.[4] It is commonly stated that *EL*2 is also a double donor but the fact is that there is a lack of direct experimental evidence to support this view. The *EL*2 ir absorption spectrum shows a threshold near 0.82 eV which is commonly identified with the first energy level of the double donor, but the spectrum shows no evidence of a second energy level. Recently, Omling *et al.*[14] have used a junction space-charge technique which seems to indicate that a second energy level exists near 0.54 eV above the valence band. The question of why the ir absorption technique is unable to detect this second level in semi-insulating si-GaAs (si denoting semi-insulating), if it exists, is still open.

In addition, the EPR technique does not allow one to distinguish isolated As_{Ga} antisite defects from complexes of As_{Ga} with vacancies and interstitials in the second neighbor (or higher) shell. Therefore, EPR alone can be misleading as to which defect one is observing. An alternative technique known as optically detected electron-nuclear double resonance (ODENDOR) was also employed[13] to study the *EL*2 defect. The results obtained with this technique supported the identification of *EL*2 with an arsenic antisite (As_{Ga})-arsenic interstitial (As_i) model proposed by von Bardeleben *et al.*[9] There are, however, some difficulties associated with the As_{Ga}-As_i model. First, an as yet unobserved acceptor defect should exist with a concentration comparable with the *EL*2 concentration.[15] Second, a shallow electronic energy level lying within the effective-mass range is obtained when As_{Ga} and As_i were brought together.[16] This shallow level has not yet been observed. Third, the condi-

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tions of keeping As_i near As_{Ga} are not known.

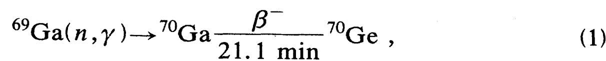
Besides the experimental advances, a few theoretical attempts[17–21] have been made to identify the *EL*2 defect. The most exciting calculations came from two independent groups[20,21] who have shown the existence of the metastability for the isolated As_{Ga} antisite when it undergoes a symmetry-breaking distortion. Unfortunately, major problems exist for the latter calculations. These problems can be summarized as follows: (1) the presence of the As_i defect near the As_{Ga} antisite which is detected by the ODENDOR measurements;[13] (2) the site symmetry of *EL*2 is lower than the T_d point-group symmetry;[22,23] (3) neutron-irradiation effects demonstrate the complexity of the *EL*2 defect;[24] (4) photoquenching and recovery effects are very complex;[25–27] and, finally, (5) the zero-phonon line (1.039 eV) and the broad transition (1.18 eV) in the ir absorption spectrum may not be related.[28]

In this article we extend the previous studies of the thermal neutron-irradiation effects on the *EL*2 defect.[24] The results support the complex models proposed for *EL*2 and cast doubt on the validity of the *EL*2 isolated As_{Ga} antisite model. The present interpretation and speculations will focus on two important aspects of neutron-irradiation effects. First, the low and high irradiation doses have distinctive and drastic effects in GaAs. Second, the photoionization of the isolated As_{Ga} antisite is believed to be responsible for the residual ir absorption which is observed after *EL*2 is photoquenched (unquenchable component). Thermal annealing of the isolated As_{Ga} antisite, *EL*2, and an *EL*2-like defect, will be presented.

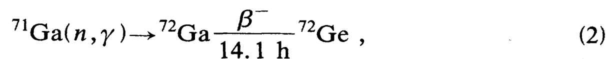
II. THERMAL NEUTRON-IRRADIATION EFFECTS IN GaAs

Useful methods of studying the *EL*2 defect are to artificially introduce it, alter its atomic configuration, or introduce other defects (which can be thermally annealed and which can cause structural modifications) by electron irradiation, fast or thermal neutron irradiation, plastic deformation, or ion implantation (for a review on irradiated and implanted GaAs, see Refs. 29 and 30). The present study focuses on the thermal neutron-irradiation effects in GaAs.

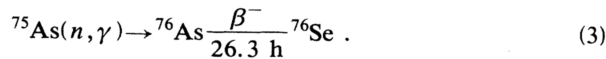
Several processes occur during thermal neutron irradiation. First, neutron transmutation doping (NTD) occurs due to the following reactions:[31–33]



$$^{69}\mathrm{Ga}(n,\gamma)\rightarrow{}^{70}\mathrm{Ga}\xrightarrow[21.1\ \mathrm{min}]{\beta^-}{}^{70}\mathrm{Ge}\ , \quad (1)$$



$$^{71}\mathrm{Ga}(n,\gamma)\rightarrow{}^{72}\mathrm{Ga}\xrightarrow[14.1\ \mathrm{h}]{\beta^-}{}^{72}\mathrm{Ge}\ , \quad (2)$$



$$^{75}\mathrm{As}(n,\gamma)\rightarrow{}^{76}\mathrm{As}\xrightarrow[26.3\ \mathrm{h}]{\beta^-}{}^{76}\mathrm{Se}\ . \quad (3)$$

The abundance of the isotopes involved and the cross sections for these reactions are such that the ratio of Se and Ge concentration produced is

$$[\mathrm{Se}]/[\mathrm{Ge}]=1.538\ . \quad (4)$$

The recoils of the high-energy β and γ particles emitted during NTD cause Se and Ge atoms to be displaced from lattice sites to interstitial positions.

Second, collisions of high-energy electron and gamma rays and fast neutrons (which are present with less than 1% of the total thermal neutron flux used in the present study) with the lattice atoms stimulate the displacement of the host atoms to form interstitials, vacancies, and antisite defects (for a detailed discussion of the formation of the antisite, see Ref. 4). Third, a disorder of the crystal structure takes place when a beam of highly collimated charged particles (for example, the β ray in the NTD process) bombards a sample at an intersecting angle smaller than the critical angle between the incident beam and index axis causing a channeling effect.[34] Fourth, cascade collisions occur (especially in high doses of irradiation) that produced defects that the can overlap and interact to form clusters and complex defects.

The overall situation of neutron irradiation may then complicate the *EL*2 studies where there is already enough controversy. Fortunately, much of the irradiation-induced damage and defects (such as vacancy, interstitial, and antisite-related defects) can be thermally annealed out at relatively low temperatures (< 550°C). Hence, neutron irradiation is a powerful method that can be utilized to obtain useful information about the atomic structure of the *EL*2 defect.

III. EXPERIMENTAL TECHNIQUE

Several samples were cut from liquid-encapsulated Czochralski (LEC) GaAs boules obtained from three different suppliers. The samples were either si-GaAs or undoped Ga-rich *p*-type LEC GaAs. The *p*-type samples were conductive with the Fermi level pinned at the 78-meV energy level prior to irradiation. Infrared-absorption measurements were obtained using a CARY 2300 spectrophotometer. A closed-cycle refrigerator was used to cool the sample in the dark to 9 K. The monochromatic spectrophotometer light was weak enough ($\sim 5\times10^{-5}$ W/cm^2) so that a noticeable photoquenching effect is not induced. Quenching of the *EL*2 defect was achieved with an external 100 W quartz-halogen lamp or 1.12-eV monochromatic light with an intensity of 4 mW/cm^2. The thermal neutron irradiation was performed at the Texas A&M University Research Reactor using different fluxes for certain periods of time as shown in Table I. Impurities introduced by the NTD are also shown in Table I. Thermal annealing was performed in an inert-gas atmosphere. The upper temperature limit of the furnace was 850 °C. The temperature was controlled within ±10 °C. No encapsulation was made and the loss of As from the GaAs surface at the upper temperature limit and short periods of time (< 30 min) did not introduce any noticeable effect on the *EL*2 defect in the control samples.

TABLE I. Characteristics of the GaAs samples used in the present study. The *p*-type samples are undoped Ga-rich as-grown LEC GaAs. The fast neutron flux was less than 1.0% of the thermal neutron flux. si denotes semi-insulating and p denotes *p* type. Ge and Se are created by the NTD process.

Sample no.	Type before irradiation	Thermal neutron flux (10^{12} n cm^{-2} s^{-1})	Irradiation time (10^3 s)	[Ge] (10^{15} cm^{-3})	[Se] (10^{15} cm^{-3})
1	si				
2	si	4.3	0.3679	0.0500	0.0769
3	si	4.3	3.679	0.5000	0.7690
4	si	4.3	367.9	50.000	76.900
5	si				
6	si	4.3	3.679	0.5000	0.7690
7	si				
8	si	4.0	0.240	0.0303	0.0466
9	si	2.7	0.960	0.0819	0.1260
10	si	2.7	3.120	0.2662	0.4094
11	*p*				
12	*p*	4.0	0.240	0.0303	0.0466
13	*p*	2.7	0.960	0.0819	0.1260
14	*p*	9.0	2.700	0.7679	1.1810

IV. RESULTS AND DISCUSSIONS

A. Reduction of the *EL*2 concentration

The ir absorption spectra of *EL*2 before and after photoquenching are shown in Fig. 1 for three samples. These spectra are superimposed on a background and residual absorption. The background absorption is increased as the neutron-irradiation doses are increased and it is considered as being due to the induced radiation damage. The residual absorption or the unquenchable component (UQC), which remains after a complete photoquenching of *EL*2, is observed before neutron irradiation and found to be sample dependent. Its magnitude depends on the postgrowth annealing. The UQC was not observed in the *p*-type control sample (no. 11). The quenchable component (QC) is obtained by subtracting the UQC from the ir absorption spectra before photoquenching and it is attributed to the *EL*2 defect. The QC obtained from Fig. 1 is plotted in Fig. 2(a) for three samples. The residual absorption or UQC is plotted in Fig. 2(b) for four samples exposed to different NTD doses. The UQC is mainly attributed to the photoionization of the isolated As_{Ga} antisite (this will be discussed in more detail in a following section). It is clear from Figs. 1 and 2 that neutron irradiation decreases the *EL*2 concentration and increases the isolated As_{Ga} antisite concentration.

Besides the absorption band between 1.04 and 1.27 eV, it is found that a broad peak around 1.4 eV can be observed when a baseline correction is made for the *EL*2 ir absorption spectrum. The 1.4-eV broad peak has received little attention in the literature, even though it can provide useful information about the *EL*2 defect. We tested the neutron-irradiation effects on the absorption bands observed between 1.04–1.27 eV and 1.3–1.5 eV and found that these bands are affected dramatically by neutron irradiation. To clarify this effect, we subtracted the sample no. 3 spectrum from the sample no. 1 (control sample) spectrum and plotted the results on the bottom of Fig. 2(a) (see the dotted curve). The results show two peaks at ~ 1.24 and ~ 1.4 eV. A similar behavior is observed in sample no. 6 (see Fig. 3). The photon irradiation has a different effect, i.e., the difference between two

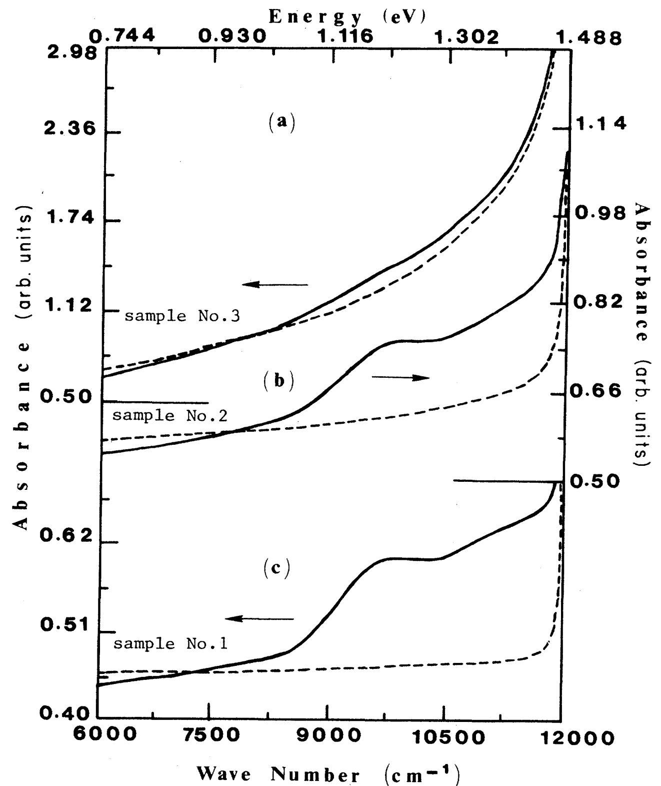


FIG. 1. The ir absorption spectra of *EL*2 before (solid lines) and after (dashed lines) photoquenching at 9 K. The samples were neutron irradiated in a flux of 4.3×10^{12} n cm^{-2} s^{-1} for (a) 3.679×10^3 s, sample no. 3; (b) 3.679×10^2 s, sample no. 2; and (c) no irradiation, control sample no. 1.

spectra taken before and after photoquenching for a short period of time, as illustrated in Fig. 3. The observation of the two peaks at 1.24 and 1.4 eV indicates the complexity of the *EL*2 ir absorption spectrum.

It should be pointed out that two transitions were observed at 1.05 and 1.29 eV in the integrated magnetic circular dichroism (MCD) of as-grown si-GaAs (see Ref. 35). The two peaks in the MCD were interpreted as two intracenter transitions within the As_{Ga} antisite. The presence of two peaks in Fig. 3 (present work) and in the MCD measurements does not by any means suggest that the two sets of peaks are identical or represent the same

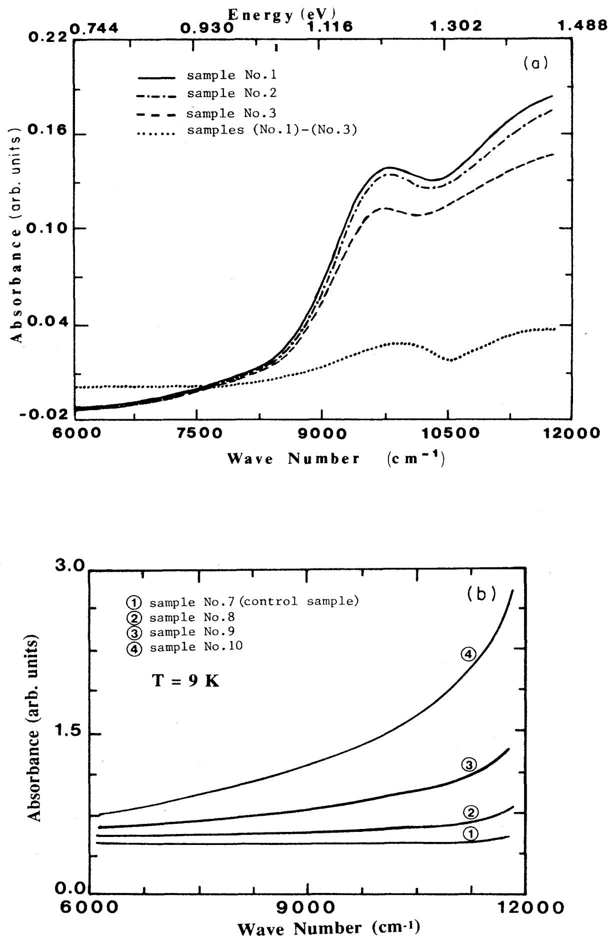


FIG. 2. (a) The *EL*2 ir absorption difference spectra for the three samples in Fig. 1. These curves were obtained by subtracting the spectrum after photoquenching from the spectrum before photoquenching. The dotted curve is the result of subtracting the sample no. 3 spectrum from the sample no. 1 (control sample) spectrum. The *EL*2 concentration is decreased by increasing the neutron-irradiation dose. (b) Spectra of the unquenchable component of the ir absorption after *EL*2 is completely photoquenched at 9 K for four samples with different NTD doses (see Table I). The control sample was sample no. 7. The unquenchable component is increased by increasing the NTD dose and it is interpreted as being due primarily to the isolated As_{Ga} antisite photoionization.

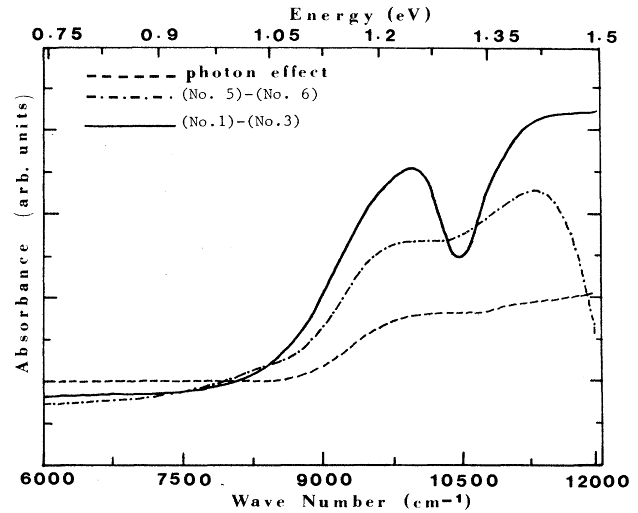


FIG. 3. The difference between spectra of neutron-irradiated and unirradiated samples as compared to photon-irradiation effect (the difference between spectra taken before and after photoquenching for a short period of time). Two peaks were observed at 1.14 and 1.4 eV in the resultant spectra. These peaks were interpreted as charge-transfer transitions between the constituent components of the *EL*2 defect. Spectra are displaced vertically with respect to each other for clarity.

processes. This is because the MCD technique looks at the positive charge state of *EL*2 while the ir absorption technique may only probe the neutral state.

The reduction of the *EL*2 concentration (the concentration was estimated from Martin's curve[36]) due to low thermal-neutron-irradiation doses was observed in all irradiated samples. Low-dose fast-neutron-irradiated samples were found to exhibit the same behavior. The reduction of the *EL*2 concentration is mainly due to the partial decomposition of the *EL*2 complex. Thermal annealing seems to support this conclusion, as we will see in the following sections.

B. Generation of the *EL*2 defect in undoped *p*-type GaAs

Vacancies, interstitials, and Frenkel-pair defects are expected to be introduced in GaAs by particle (such as electron, neutron, or ion) bombardment. These primary defects can interact to form more complex defects. Hence, creation of *EL*2 in GaAs becomes possible. It is found in the present study that the *EL*2 defect can become observable in undoped Ga-rich *p*-type LEC GaAs by irradiating the sample with low thermal-neutron doses. This defect is found to be thermally stable up to the maximum temperature limit reached in the present work (850 °C).

Several samples were irradiated and tested. The results are shown in Table II. The control sample (no. 11) contains the double acceptor which is attributed to the Ga_{As} antisite[37] and *EL*2 has not been observed in this sample. From Table II one can notice that the *EL*2 concentration is independent of the irradiation dose within experimental error. This may be due to the balance between the

TABLE II. The *EL*2 concentration in thermal neutron-irradiated *p*-type GaAs before thermal annealing. The irradiation doses are listed in Table I.

Sample no.	[*EL*2] (10^{16} cm^{-3})
11	
12	0.46±0.05
13	0.49±0.05
14	0.54±0.10

generation and destruction rates of the *EL*2 complex. The question of why *EL*2 might be generated in Ga-rich *p*-type GaAs and decomposed in si-GaAs can be answered as follows. First, the two materials are different. The *p*-type GaAs does not contain measurable *EL*2, while si-GaAs contains $\sim 1.5\times10^{16}$ cm^{-3} of *EL*2 prior to irradiation. Second, the generation rate of *EL*2 in *p*-type GaAs does not increase by increasing the irradiation dose and [*EL*2] is very small as compared to [*EL*2] in si-GaAs. This behavior, as mentioned above, may indicate the presence of a balance between the generation and destruction rates of the *EL*2 complex. Third, *EL*2 may be generated in si-GaAs, but the destruction rate is larger than the generation rate. Fourth, it is possible that *EL*2 exists in the *p*-type samples, but it was compensated by the double acceptor and other impurities such as carbon. Neutron irradiation may introduce donors shallower than *EL*2 that can compensate the acceptors leaving *EL*2 uncompensated. The latter point may be supported by the fact that [*EL*2] does not increase by increasing the NTD dose.

C. Generation of the isolated As_{Ga} antisite and an *EL*2-like (*DL*2) defect in GaAs

So far we have shown the effects of the low NTD doses in GaAs. The high doses, on the other hand, cause more dramatic effects on the *EL*2 defect. In addition, the GaAs samples exposed to high irradiation doses become opaque. As an example we studied sample no. 4, which was irradiated for over 100 h in a flux of 4.3×10^{12} $n\,cm^{-2}\,s^{-1}$ (see Table I). Thermal annealing was then necessary to perform the ir absorption measurements.

An unidentified defect was observed at 0.65 eV in sample no. 4 after it was annealed for 30 min at 400 °C. This broad peak is presented in Fig. 4 (dashed spectrum). It is noted that the 0.65-eV peak has disappeared after annealing the sample at 500 °C for 30 min. The thermal-annealing behavior of the 0.65-eV peak is similar to that of the *U* band observed by the deep-level transient spectroscopy (DLTS) measurements in fast-neutron-irradiated GaAs samples.[38-41] In addition, the energy level of the *U* band (0.55 eV below the conduction band) is in excellent agreement with the broad peak observed at 0.65 eV (see Fig. 4). Hence, we identify the 0.65-eV peak with the *U* band observed in the DLTS measurements.

It was shown that the annealing of the *U* band is accompanied by an increase in the *EL*2 signal.[38] This behavior is not observed in the present ir absorption measurements. The quenchable component of the ir absorption spectrum was not observed after annealing the sample for 30 min at 500 °C. There is a possible explanation for this discrepancy. The DLTS measurements are sensitive to both charged and neutral states of a defect such as *EL*2, while the ir absorption apparently records only the neutral state. Therefore, *EL*2 (or the *EL*2-like defect) is still compensated by an unknown acceptor(s) even after annealing the sample at 500 °C.

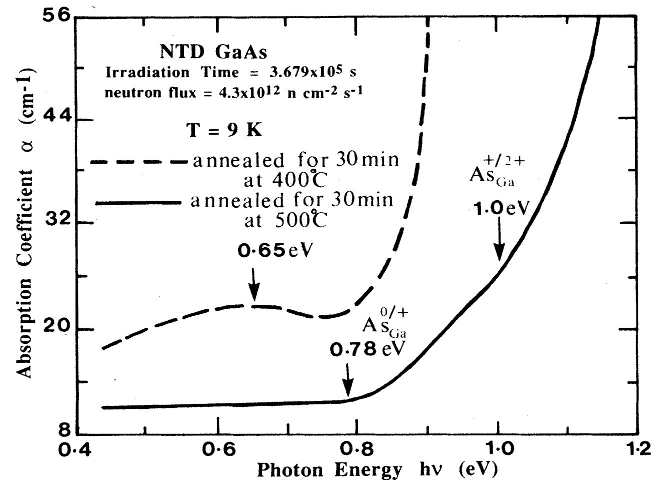


FIG. 4. Thermal annealing of sample no. 4 at 400 °C (dashed spectrum) and 500 °C (solid spectrum). The broad peak observed at 0.65 eV in the dashed spectrum is identified as the *U* band (see Ref. 38). The threshold at 0.78 eV and the shoulder at 1.0 eV in the solid spectrum were identified with the two charge states of the isolated As_{Ga} antisite (see Ref. 4).

Many vacancy and interstitial-related defects in addition to radiation damage are annealed at 500 °C. A threshold at $\sim$0.78 eV and a shoulder at $\sim$1.0 eV were observed after annealing the sample for 30 min at 500 °C (see the spectrum presented by the solid line in Fig. 4). The threshold and the shoulder in Fig. 4 are in excellent agreement with the EPR measurements of the isolated As_{Ga} antisite observed in fast-neutron-irradiated GaAs samples.[4] This observation is in support of the present proposal that the unquenchable component of the ir absorption spectra is primarily due to the photoionization of the isolated As_{Ga} antisite. The correlation between the unquenchable component and the photoionization of the isolated As_{Ga} antisite is also supported by the increase of the unquenchable component as the NTD dose is increased [see Fig. 2(b)] which is in agreement with the EPR measurements.[4,6-8] As will be discussed below, the annealing kinetics of the unquenchable component at 600 °C also support the above correlation.

Annealing the sample (no. 4) for 6 min at 600 °C reveals more interesting information. A quenchable component is now observed in the ir absorption spectrum. The result is plotted in Fig. 5. The defect responsible for the quenchable component in this figure is identical to the *EL*2 defect in two respects. First, it can be quenched with white light or 1.12-eV monochromatic light at low temperatures. Second, it is thermally recovered at temperatures ≥ 150 K. It is found, however, that the

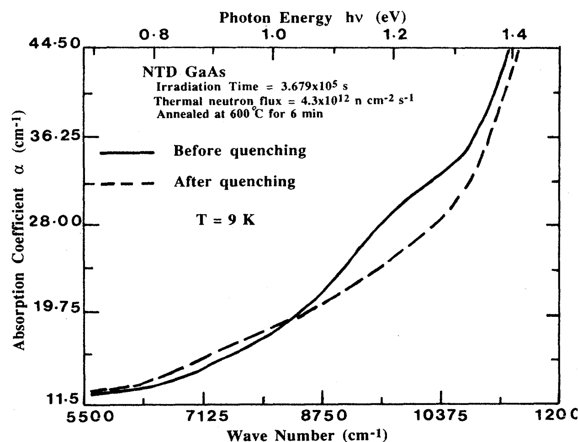


FIG. 5. An $EL2$-like defect labeled $DL2$ was observed in sample no. 4 after thermal annealing for 6 min at 600 °C. It is not clear whether this defect is generated during the neutron irradiation or during the thermal annealing.

quenchable component of this defect (we will refer to this defect as $DL2$ for simplicity) as shown in Fig. 5 is different from the $EL2$ defect in LEC si-GaAs in several respects. First, the $DL2$ concentration is much higher than the $EL2$ concentration. For a comparison, we plotted the $DL2$ and $EL2$ spectra in Fig. 6. Martin *et al.*[38] observed that the $EL2$ concentration is increased by increasing the neutron-irradiation dose after annealing the samples for 15 min at 600 °C. We argue that the $EL2$ defect observed by Martin *et al.* is not the $EL2$ defect, but the $DL2$ defect. Second, the $DL2$ ir absorption spectrum is different from the $EL2$ spectrum at lower energies (see Fig. 6). Third, the $DL2$ defect does not possess a ZPL at 1.039 eV. We found that samples irradiated with low doses do not show the ZPL before annealing at 600 °C. However, the ZPL is observed in these samples after annealing them for 15 min at 600 °C. While the $EL2$ concentration is decreased by increasing the irradiation dose, the ZPL intensity is found to increase by increasing the dose[42] (within the low-dose limits). This behavior is rather surprising and it indicates that the ZPL and the broad peak at 1.18 eV are two different and distinctive transitions in agreement with Mochizuki and Ikoma.[28] Fourth, the $DL2$ defect is thermally unstable at 600 °C while $EL2$ is thermally stable up to 1000 °C (see Refs. 43 and 44).

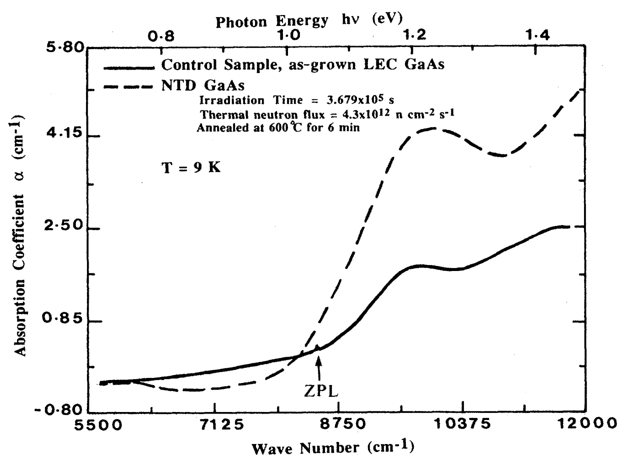


FIG. 6. Comparison between the ir absorption coefficient of the $DL2$ defect in irradiated sample no. 4 and the $EL2$ defect in control sample no. 1. The ZPL was not observed in the $DL2$ defect. The $DL2$ concentration is larger than the $EL2$ concentration by a factor of 2.3.

The annealing behavior and the thermal stability of the $DL2$ defect are identical to that of the EPR defect observed in high-dose irradiated GaAs samples.[45–47] The photoquenching and thermal recovery of the defect observed by the EPR technique[45–47] have not been tested and therefore a comparison between the $DL2$ defect and the EPR defect is difficult. In addition, the EPR signal of the isolated As_{Ga} antisite in the NTD GaAs samples was found to be unquenchable.[6]

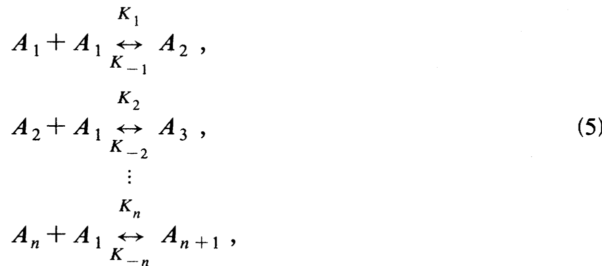
The $DL2$ defect still can be related to the $EL2$ defect. One possibility is that the atomic structure of the $EL2$ defect is formed by high neutron-irradiation dose, but this atomic structure interacts with other defect complexes and clusters that were formed during irradiation. This interaction may modify or alter the behavior (such as thermal stability and ir absorption spectrum) of $EL2$ to give the $DL2$ defect. Therefore, understanding the $DL2$ defect may reveal important information about the $EL2$ defect.

D. Thermal annealing

Compensation mechanisms, interaction between defects, destruction of complex defects, and generation of new defects are processes one may encounter when dealing with the neutron-irradiation effects on the $EL2$ defect. Before thermal annealing, the DLTS spectra[38] in heavily irradiated GaAs are dominated by the $EL6$ defect and by a broad peak known as the U band. The U band was observed to vanish and gradually shift toward $EL2$ (again, we emphasize that the notation $DL2$ is used for the $EL2$-like defect in heavily irradiated GaAs samples to distinguish it from $EL2$ in as-grown LEC and low-dose irradiated GaAs samples) by thermal annealing between 400 and 500 °C. The broad peak observed at 0.65 eV after 30-min annealing at 400 °C in Fig. 4 resembles the U-band behavior. The thermal-annealing behavior of the U band may reflect the interaction between the $DL2$ defect and other defects. This conclusion can be understood in terms of the increased separation between defects as the annealing temperature is increased, which results in isolated defect behavior.

Many defects (both acceptors and donors) introduced by the neutron irradiation seem to anneal out at temperatures less than 600 °C. This may result in uncompensating the $DL2$ defect, although it is not clear to us whether $DL2$ is formed during irradiation or during the thermal annealing between 500 and 600 °C. It should be pointed out that impurities (Ge and Se) introduced by the NTD (see Table I) form deep complexes as observed in photoluminescence measurements[48,49] and may play an important role in the compensation mechanisms especially after annealing the samples above 500 °C. The results of iso-

thermal annealing of *DL*2 and the unquenchable component (the isolated As_{Ga} antisite) in sample no. 4 is shown in Fig. 7. The isothermal annealing of *EL*2 in the control sample (no. 1) is also shown for comparison. The annealing temperature was $T_a = 600\,^{\circ}\mathrm{C}$. In order to analyze the isothermal-annealing results of Fig. 7, we adopted the chemical reactions of generation (annihilation) of complex defects as described by Suezawa and Sumino,[50,51] who extended the work of Kaiser *et al.*[52] These reactions can be written as



$$
\begin{aligned}
A_1 + A_1 &\underset{K_{-1}}{\overset{K_1}{\leftrightarrow}} A_2 \,, \\
A_2 + A_1 &\underset{K_{-2}}{\overset{K_2}{\leftrightarrow}} A_3 \,, \\
&\vdots \\
A_n + A_1 &\underset{K_{-n}}{\overset{K_n}{\leftrightarrow}} A_{n+1} \,,
\end{aligned}
\tag{5}
$$

where A_n is the concentration of a defect consisting of n atoms and K_n and K_{-n} are the reaction constants for generation and annihilation of defects, respectively. For a point defect or a defect with one atom, the annealing kinetics equation can be written as

$$A_1(t) = A \exp[-\lambda_1(T)t] \,, \tag{6}$$

where A is the initial concentration, $\lambda_1(T)$ is the annealing rate which depends on the temperature T and reaction constant, t is the annealing time, and $A_1(t)$ is the defect concentration at a given annealing time. The situation becomes difficult when the defect consists of two or more point defects. Using Eq. (5) one can find analytical solutions for a defect consisting of two or three point defects. For a complex of three point defects, the analytical solution can be written as

$$A_3(t) = \pm B(1 - \{[1 + \lambda_3(T)t]\exp[-\lambda_3(T)t]\}) \,, \tag{7}$$

where + (−) is for the defect generation (annihilation), B is a constant depending on the initial concentration, $\lambda_3(T)$ is the same as $\lambda_1(T)$, t is the time, and $A_3(t)$ is the defect concentration at a given time. For a defect composed of two point defects, see Refs. 9, 50, and 51.

The isothermal-annealing data of *DL*2 in Fig. 7 were found to be fitted by Eq. (7) with the negative sign (see solid line in Fig. 7). Thermal kinetic equations of a defect with one or two point defects were tried and it was found that these equations cannot be used to fit the *DL*2 data. On the other hand, the unquenchable component in sample no. 4 was fitted by Eq. (6) (see the dashed-dotted curve in Fig. 7). The latter observation supports the proposal that the unquenchable component is due to a point defect which is identified by us as the isolated As_{Ga} antisite. It is also observed that the unquenchable component is reduced by thermal annealing at 600 °C and it approaches the values found in as-grown LEC GaAs.

The isothermal-annealing data of the unquenchable component in Fig. 7 deviate from linear behavior when annealed at 600 °C for more than 35 min. The reason is as follows. It is observed that the *DL*2 defect can be photoquenched completely during the isothermal annealing at 600 °C for the first 35 min. After this annealing time, *DL*2 was found not to photoquench completely. The residual absorption is then due to more than one species of defect. After reaching the annealing time of 50 min, we were unable to quench *DL*2 at all. The result is shown in Fig. 8, in which the unquenchable component spectrum is plotted as a function of annealing time. The unquench-

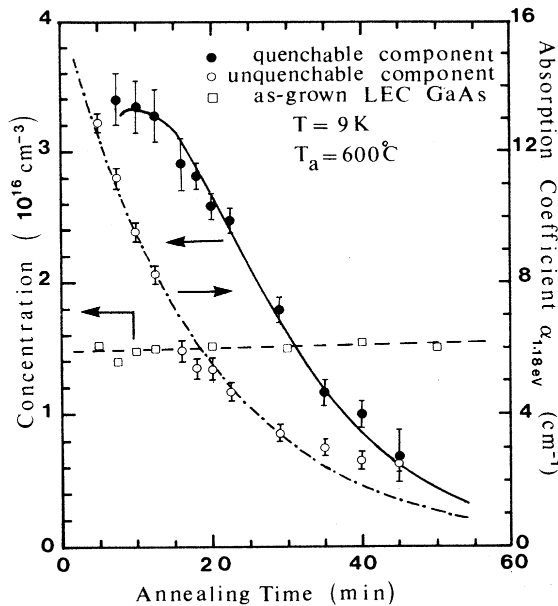


FIG. 7. Isothermal annealing at 600 °C for the *DL*2 defect in sample no. 4 (●), the unquenchable component (photoionization of the isolated As_{Ga} antisite) in sample no. 4 (○), and the *EL*2 defect in control sample no. 1 (□). The *DL*2 data were fitted by Eq. (7) (solid line) and the data for photoionization of the isolated As_{Ga} antisite were fitted by Eq. (6) (dotted-dashed line). There is no noticeable change of the *EL*2 concentration in control sample no. 1.

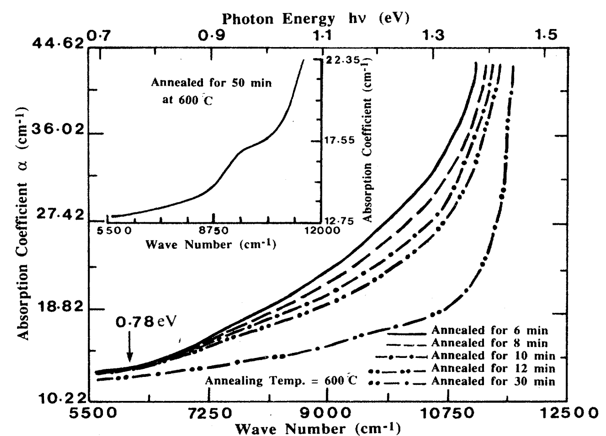


FIG. 8. Spectra of the photoionization of the isolated As_{Ga} antisite (unquenchable component) after annealing sample no. 4 at 600 °C for different periods of time. It is found that *DL*2 cannot be quenched after annealing the sample for 50 min at 600 °C (the inset). See the text for possible explanation.

able $DL2$ defect is plotted in the inset of Fig. 8. The sample was then left for several weeks at room temperature. The photoquenching behavior of $DL2$ was checked every two weeks and it was found that $DL2$ did not photoquench during the first six weeks. After this period of time, the $DL2$ defect was observed to be quenchable. This behavior was also observed in a second sample with high irradiation dose.

There are two possible explanations for the above anomalous behavior. First, the $DL2$ defect is apparently composed of three point defects as shown from the fitting of the isothermal-annealing data by using Eq. (7) (see Fig. 7). These point defects can be antisites (As_{Ga}) and interstitials (vacancies). It is possible that one of these three point defects, which may be responsible for the transformation of the $DL2$ from the normal state to the metastable configuration, may anneal out before the other two, preventing $DL2$ from photoquenching. This point defect could be generated during long-time annealing at room temperature from nearby clusters (dislocations) formed during the neutron irradiation. The second speculation is based on the results of Samuelson and Omling.[53] They found that $EL2$ does not quench in $GaAs_{1-x}P_x$ when $x \geq 0.3$. Samuelson and Omling speculate that at this value of x the excited state of $EL2$ emerges from the conduction band and prevents the transformation of $EL2$ to the metastable configuration. We offer an alternative explanation. It is well known that shallow impurities such as Si, Te, etc. exist in GaAs and its alloys. These shallow impurities, especially Si, become deep centers when the band gap is increased by alloying or applying hydrostatic pressure. The deep centers (known as DX centers) are very sensitive to light and can be ionized to give n-type persistent photoconductivity. The electrons that are released from the DX centers can be captured by the metastable $EL2$ defect causing an Auger-type recovery.[54] The above explanation can be applied to explain the unquenchable $DL2$ defect in Fig. 8. Internal stress caused by dislocations and band bending due to clusters formed during the neutron irradiation may play an important role in the formation of defects (such as the DX centers) that emit electrons[55,56] when the sample is irradiated by photons. The emitted electrons will be captured by the metastable $DL2$ ($EL2$) defect causing the recovery. Long-term annealing at room temperature may affect the electron emitting defects in a way that they are either annealed out or no longer capable of emitting electrons. The thermal stability of $EL2$ in samples no. 10 (low NTD dose), no. 14 (p-type GaAs after low NTD dose), no. 4 after 50-min annealing at 600 °C and six-weeks annealing at room-temperature (heavily irradiated sample), and as-grown LEC si-GaAs was checked up to 850 °C. The results are shown in Fig. 9. The $EL2$ concentration in sample no. 10 is always smaller than the $EL2$ concentration in the as-grown sample (no. 7). This is strong evidence that low doses of neutron irradiation partially decompose the $EL2$ complex and the reduction of $[EL2]$ prior to annealing is not due to the compensation of $EL2$ by acceptors introduced by NTD. The $EL2$ defect in NTD p-type GaAs (sample no. 14) was also found to be thermally stable up to 850 °C. The $DL2$ defect in sample no. 4 was found to anneal out completely at 750 °C. For another sample with the same NTD dose as no. 4, it was found that 95% of $DL2$ was annealed out during the first 90 min at 600 °C.

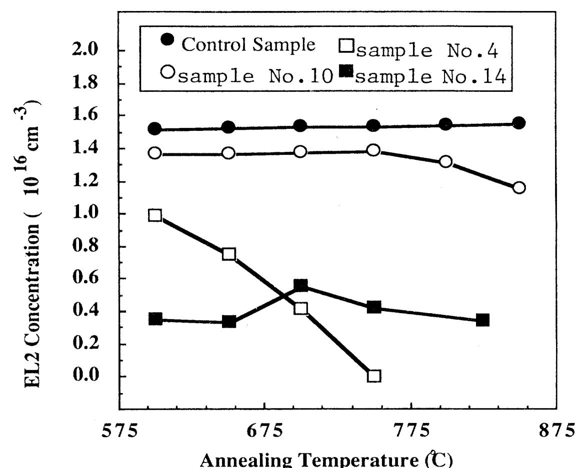


FIG. 9. Sequential isochronal annealing of $EL2$ in as-grown control sample (●), low-dose-irradiated si-GaAs, sample no. 10 (○), low-dose-irradiated p-type GaAs, sample no. 10 (■), and $DL2$ in heavily irradiated si-GaAs after annealing for 50 min at 600 °C and for six weeks at room temperature, sample no. 4 (□). The $EL2$ defect is thermally stable up to the maximum temperature limit reached in the present study (850 °)C while $DL2$ is annealed out completely at 750 °C. The samples were annealed for 15 min at each temperature.

E. Modeling

Controversy still exists over the identification of the atomic nature of $EL2$. The identification of $EL2$ with the isolated As_{Ga} antisite is supported by recent theoretical calculations[20,21] which show the existence of a metastable state for the isolated As_{Ga} antisite. Many experimental measurements[13,22–28] disagree with such identification. The present measurements support the identification of $EL2$ with the $As_{Ga}+X$ complex defect. The atomic nature of X is still a matter of controversy. The intention of the present study is not to identify X, but rather to expose the complex nature of the $EL2$ defect.

The unquenchable component of the ir absorption spectra seems to correlate with the generation [see Fig. 2(b)] and the thermal annealing (see Figs. 7 and 8) of the isolated As_{Ga} antisite. In addition, the threshold energy (0.78 eV) of the unquenchable component agrees with the EPR results for the isolated As_{Ga} antisite.[4] These pieces of evidence led us to propose that the isolated As_{Ga} antisite undergoes photoionization which can be distinguished from $EL2$ in the ir absorption measurements.

The presence of an $EL2$-like defect ($DL2$) with high concentration in heavily irradiated GaAs samples (see Figs. 5 and 6) could support the identification of $EL2$ with the isolated As_{Ga} antisite model if not for the fact that $EL2$ is not increased by electron irradiation.[57] The isolated As_{Ga} antisite concentration does increase as the electron irradiation dose is increased.[58,59] One possible

explanation[29] for the absence of $EL2$ generation in electron-irradiated samples is that two As_i atoms are required to create $EL2$, assuming that $EL2 = As_{Ga} + As_i$ complex,[9,13] which gives a very small introduction rate for $EL2$.

If the hypothesis of relating the isolated As_{Ga} antisite to the unquenchable component in the ir absorption spectra (the isolated As_{Ga} antisite undergoes only photoionization) is to be considered valid, then the ZPL observed at 1.039 eV and the broad peak observed at 1.18 eV in the $EL2$ ir absorption spectrum cannot be related to the As_{Ga} antisite involved in the $EL2$ complex. Hence, the absence of the ZPL from the unquenchable component casts doubt on the interpretation of the ZPL as being the A_1 to T_2 transition within the As_{Ga} antisite of a T_d symmetry[60] and lends support for the ZPL as being a transition within an orthorhombic complex.[61]

The absorption band between 1.03 and 1.27 eV (we will refer to this band as AB for simplicity) has been the subject of numerous studies.[4,22,53,60–65] It contains a broad peak at 1.18 eV, a ZPL at 1.039 eV, and phonon replicas separated by ~ 11 meV. The AB was interpreted as a transition from $EL2$ to the L minimum of the conduction band,[66–68] an intracenter transition within the As_{Ga} antisite,[69] and an intracenter transition within the gallium vacancy.[70] It has been shown[71] that the AB cannot be related to the gallium vacancy and the ZPL may not be associated[28] with the broad peak at 1.18 eV.

It is clear from the present results that neutron irradiation increases the isolated As_{Ga} antisite and decreases the $EL2$ concentration. High doses of neutron irradiation seem to destroy the $EL2$ complex completely in as-grown LEC si-GaAs and generate an $EL2$-like defect ($DL2$) that is thermally unstable. At this point, it is not clear whether $DL2$ is formed during irradiation or during the thermal annealing between 500 and 600 °C. The generation of the isolated As_{Ga} antisite and consumption of interstitials and vacancies by neutron irradiation were discussed previously in more detail.[4] Based on the previous[24] and present results, the rate of the isolated As_{Ga} antisite generation is much higher than the rate of X-component destruction (reduction of $EL2$ concentration). If $EL2$ is a complex involving As_{Ga} and an X component(s), and if X is to be identified[9,13] as As_i, then the reduction of $EL2$ concentration is the result of a decrease in the As_i concentration. This process is accompanied by the reduction of the AB signal as well as the absorption band between 1.3 and 1.5 eV [see Figs. 2(a) and 3]. This behavior leads us to propose that the absorption bands between 1.03–1.3 eV and 1.3–1.5 eV are charge-transfer transitions between the As_{Ga} and As_i point defects. The present results do not rule out any charge-transfer transitions between As_{Ga} and vacancies. Recent theoretical calculations[16,72,73] predict different possibilities of charge-transfer transitions between As_{Ga} and As_i in support of the present interpretations. It should be emphasized that As_i was taken as an example to illustrate the charge-transfer transitions between the constituent components of the $EL2$ complex. Hence, the present results do not rule out other $EL2$ complex models.

According to the isothermal-annealing data (Fig. 7) and the annealing kinetics equation [Eq. (7)], $DL2$ seems to be composed of three point defects. If $DL2$ is to be considered as $EL2$ which is modified by the presence of clusters and dislocations introduced by NTD, then the As_{Ga}-As_i model,[9,13] which is modified to a split interstitial[74,75] for the metastable state, is no longer valid. The annealing behavior of $DL2$ is similar to the generation of $EL2$ at 668.4 °C in LEC rapidly cooled GaAs (see Ref. 51), i.e., the generation of $EL2$ is fitted by Eq. (7) with the positive sign. Two models were proposed for the $EL2$ atomic structure in which three point defects exist. The first model[19,61] indicates that the $EL2$ defect is composed of the As_{Ga} dimer (i.e., two As_{Ga} atoms) plus an As_i in the position symmetric with respect to both As_{Ga} atoms. The second model[70,76] proposes that $EL2$ is an As_{Ga} plus a divacancy (gallium vacancy–arsenic vacancy). The present results and those of Suezawa and Sumino[51] seem to support these models. It is, however, possible that a vacancy that cannot be detected by the ODENDOR measurements[13] may exist as part of $EL2$ (see Ref. 27) near the As_{Ga}-As_i pair. The latter speculation requires additional investigations by using other techniques such as a positron annihilation experiment. The point is that the present results do not support the identification of $EL2$ with the isolated As_{Ga} antisite. In addition, the atomic structure of the $EL2$ defect may be more complex than a simple pair defect.

V. SUMMARY AND CONCLUSIONS

In the present study, we have shown the thermal neutron-irradiation effects on the $EL2$ defect in LEC GaAs. The atomic structure of the $EL2$ complex is partially decomposed by a low thermal-neutron-irradiation dose. The amount of destruction of this complex is increased by increasing the dose. The $EL2$ complex is completely eliminated and an $EL2$-like defect ($DL2$) is generated in heavily irradiated samples. On the other hand, a small amount of $EL2$ is generated in as-grown Ga-rich p-type LEC GaAs samples by low NTD doses. Once $EL2$ is generated in p-type GaAs samples, it becomes independent of dose within experimental error. This indicates that a balance between generation and destruction rates is reached. The $EL2$ defect in low-dose irradiated si-GaAs samples was found to be stable up to 850 °C, which supports the hypothesis that the reduction of the $EL2$ concentration is not due to compensation mechanisms but rather to destruction of the $EL2$ defect. The $EL2$ defect in p-type GaAs is also found to be stable up to 850 °C.

The isothermal annealing of the $EL2$-like defect ($DL2$) was performed at 600 °C. The data fitting indicates that $DL2$ is a complex composed of three point defects. If the $DL2$ defect is to be identified as $EL2$ modified by the presence of dislocations and clusters introduced by NTD, then the current results (including the reduction of the $EL2$ concentration by increasing the NTD dose) support $EL2$ as being a complex defect and cast doubt on the identification of $EL2$ with the isolated As_{Ga} defect.

The unquenchable component of the ir absorption spectra was interpreted as being due primarily to the photoionization of the isolated As_{Ga} antisite. This interpretation is supported by the increase of the unquenchable component as the NTD dose is increased and the thermal-annealing kinetics at 600 °C. The ZPL does not seem to be an internal transition within the As_{Ga} antisite. In addition, the absorption bands between 1.03–1.3 eV and 1.3–1.5 eV were interpreted as charge-transfer transitions between the constituent point defects of the *EL*2 complex.

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