

Identification of electron-irradiation defects in semi-insulating GaAs by normalized thermally stimulated current measurements

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 (Received 19 September 1996)

Primary defects induced by 1 MeV electron irradiation have been quantitatively studied in semi-insulating (SI) GaAs by using normalized thermally stimulated current spectroscopy, a new technique. Defects identical to (or similar to) those known in the thermally stimulated current literature as T_6^* (0.13 eV), T_5 (0.34 eV), and T_4 (0.31 eV) are produced at rates 0.70, 0.08, and 0.23 cm⁻¹, respectively; T_5 is also a strong trap in unirradiated SI GaAs. The defects T_6^* and T_4 correspond closely to the irradiation-induced traps $E2$ (0.14 eV) and $E3$ (0.30 eV), studied extensively by deep-level transient spectroscopy and Hall-effect measurements and assigned to the As vacancy. We thus infer that traps T_6^* and T_4 (and probably also T_5) in SI GaAs have As-vacancy character. [S0163-1829(97)04503-7]

INTRODUCTION

High-energy electron irradiation has been employed to study primary defects (vacancies, interstitials, and, sometimes, antisites) in many metal and semiconductor materials.¹ Typically, the electron energy necessary to displace an atom will be a few hundred keV; thus, the common choice of 1-MeV irradiation will produce only one, or at most a few, displacements and no massive damage, such as is often found with heavy-ion irradiation. Semiconducting GaAs has been investigated in this manner for several decades (for reviews, see Refs. 2 and 3). Many techniques have been employed, but quantitative analysis has mainly relied on temperature-dependent Hall-effect (TDH) (Ref. 4) and deep-level transient spectroscopy (DLTS) measurements, or other methods involving capacitance.³ However, the TDH and DLTS techniques cannot be applied in semi-insulating (SI) GaAs, an important material that forms the basis of the GaAs microwave and integrated-circuit industries. A well-established method for looking at traps in SI materials is thermally stimulated current (TSC) spectroscopy;⁵⁻⁸ however, TSC is not considered to be a quantitative technique because it involves carrier mobility, lifetime, and geometric factors, which are either unknown or poorly known. In this work we first show how to quantify a TSC spectrum, by normalizing with infrared photocurrent, and then apply this quantitative method (called NTSC) to study traps produced by electron irradiation in SI GaAs. The NTSC traps T_6^* (0.13 eV) and T_4 (0.31 eV), which sometimes appear in as-grown (unirradiated) SI GaAs, are shown to be equivalent to the DLTS electron traps $E2$ (0.14 eV) and $E3$ (0.30 eV), respectively, and are assigned to the As vacancy V_{As} or its complex. Another defect, T_5 (0.34 eV), which is always prominent in as-grown SI GaAs, grows with irradiation, but at a smaller rate than that found for either T_6^* or T_4 . The other two most prominent TSC traps in as-grown SI GaAs, T_2 (0.63 eV) and T_3 (0.50 eV), are unaffected by 1-MeV irradiation.

THEORY

The idea behind the normalization procedure is that both TSC and infrared (IR) photocurrent (PC) are linearly propor-

tional to carrier lifetime $\tau(T)$, mobility $\mu(T)$, and certain geometric factors;⁵ thus, their ratio (I_{TSC}/I_{PC}) mostly involves only quantities that are either known or can be fitted. The only remaining unknown quantity, in general, is the absorption coefficient $\alpha(T)$; however, fortunately, for IR excitation of electrons from $EL2$ (the As_{Ga} -related defect that is dominant in SI GaAs), $\alpha(T)$ is well known.^{9,10} The TSC for electron traps can be shown to obey⁵

$$I_{TSC} = e \mu_n \tau_n \frac{wd}{l} V N_T e_n \exp\left(-\int_{T_0}^T \frac{e_n}{\beta} dT'\right), \quad (1)$$

where a rectangular sample is assumed (length ℓ , width w , and thickness d), V is the applied voltage, N_T the trap density, T_0 the starting temperature, β the heating rate, and e_n the emission rate given by

$$e_n = \frac{16\pi m_n^* k^2}{h^3} \left(\frac{g_0}{g_1} \sigma_{n0} e^{\alpha_T/k}\right) T^2 e^{-(E_0+E_\sigma)/kT}. \quad (2)$$

Here, g_0 (g_1) is the degeneracy of the unoccupied (occupied) state, $\sigma_n = \sigma_{n0} \exp(-E_\sigma/kT)$ is the electron capture cross section, α_T is defined by $E = E_0 - \alpha_T T$, and E is the energy of the trap defined with respect to the conduction band. Often the term $(g_0/g_1)\sigma_{n0}\exp(\alpha_T/k)$ is called the *apparent* capture cross section σ_a , and $(E_0 + E_\sigma)$, the *apparent* energy, E_a . For a nonrectangular sample, the factor wd/ℓ will change; however, it will cancel out anyway in the ratio I_{TSC}/I_{PC} as shown below.

We now turn to the derivation of the PC under illumination by IR light (1.1 μm in our case) of intensity I_0 photons/cm² s. In a thick sample ($\alpha d \gg 1$, where α is the absorption coefficient), all of the light will be absorbed except for that reflected by the front surface; in other words, the *effective* light intensity is $I_0(1-R)$. However, in a thin sample, multiple reflections involving the back surface also must be considered. Consider a small region at distance x from the sample surface and of length dx ; then the volume

concentration of electrons n_{1d} produced on the *first downward* pass of the light (before reflection at the back surface) is

$$\begin{aligned} n_{1d} &= \frac{1}{d} \int_0^d I_0(1-R)\alpha_n e^{-\alpha x} \tau_n dx \\ &= I_0(1-R)\alpha_n \tau_n \frac{1-e^{-\alpha d}}{\alpha d}, \end{aligned} \quad (3)$$

where α_n is the portion of the absorption which produces free electrons and α is the total absorption. The intensity of light reaching the backside is $I_0(1-R)\exp(-\alpha d)$ and the intensity reflected back toward the upper surface is $I_0R(1-R)\exp(-\alpha d)$. Thus, the concentration of electrons produced during the first *upward* pass is $n_{1u} = R \exp(-\alpha d)n_{1d}$. On the *second downward* pass the concentration is $n_{2d} = R^2 \exp(-2\alpha d)n_{1d}$, and the final result, after many passes, is

$$n = n_{1d} + n_{1u} + n_{2d} + \dots = I_0 \alpha_n \tau \frac{1-e^{-\alpha d}}{\alpha d} \frac{1-R}{1-R e^{-\alpha d}}. \quad (4)$$

For SI GaAs, $R(1.1 \mu\text{m}) \approx 0.305$, and typically $\alpha \approx \alpha_n \approx 1 \text{ cm}^{-1}$ and $d \approx 0.06 \text{ cm}$. Thus, $\alpha d \ll 1$, and $n \approx I_0 \alpha_n \tau$. This derivation reveals an interesting fact: for a very thin sample, the effectively longer path length due to the multiple reflections in the sample itself exactly makes up for the reflection lost from the front surface.

The PC is then given by $I_{\text{PC}} = en\mu_n(wd/\ell)V$ so that $I_{\text{TSC}}/I_{\text{PC}}$ becomes

$$\frac{I_{\text{TSC}}}{I_{\text{PC}}} = \frac{N_T e_n \exp\left(-\int_{T_0}^T \frac{e_n}{\beta} dT'\right)}{I_0 \alpha_n \frac{1-e^{-\alpha d}}{\alpha d} \frac{1-R}{1-R e^{-\alpha d}}}. \quad (5)$$

Here we are neglecting the PC due to holes since $\alpha_p(1.1 \mu\text{m}) < \alpha_n(1.1 \mu\text{m})$ and $\mu_p \ll \mu_n$. For SI GaAs, $\alpha_n(\lambda) = \sigma_{vn}(\lambda)N_{\text{EL2}}^0$ and $\alpha(\lambda) = \sigma_{vn}(\lambda)N_{\text{EL2}}^0 + \sigma_{vp}(\lambda)N_{\text{EL2}}^+$. The photoionization coefficients, σ_{vn} and σ_{vp} , for $\lambda = 1.1 \mu\text{m}$, are well known.^{9,10} Also, N_{EL2}^0 and N_{EL2}^+ can be determined from transmission measurements at two different wavelengths, say 1.1 and 1.2 μm .¹⁰ Thus, a fit of $I_{\text{TSC}}/I_{\text{PC}}$ as a function of sweep temperature, $T = T_0 + \beta t$, will give N_T , σ_a , and E_a as fitting parameters. However, in general, σ_a cannot be fitted accurately, as is also the case in DLTS analysis (e.g., see Ref. 11, p. 202).

Equation (5) assumes that all of the N_T traps are filled by the illumination, which will not be true if the illumination excites electrons out of the trap even while it is providing conduction-band electrons from other sources (*EL2* or the valence band) which can be captured by the trap. In steady state (long illumination time) the occupied fraction will be

$$\frac{N_T^0}{N_T} = \frac{nv_n\sigma_n + I_0\sigma_{vpT}(\lambda)}{nv_n\sigma_n + pv_p\sigma_p + I_0[\sigma_{vnT}(\lambda) + \sigma_{vpT}(\lambda)]}, \quad (6)$$

where $\sigma_{vnT}(\lambda)$ is the photoionization cross section for electron excitation from the trap T to the conduction band by light of wavelength λ , and $\sigma_{vpT}(\lambda)$ is the analogous term for hole excitation. For trap filling, we used 1.45-eV light, an energy just below the band gap at 83 K, the temperature at which the traps were filled. The reason for filling with 1.45-eV light, rather than the 1.1- μm light used for the PC, is that the latter can also cause *EL2* quenching, which complicates the analysis.⁷ To test the importance of Eq. (6) in the present analysis, we employed a wide range of filling wavelengths λ and intensities I_0 , and measured N_T (or really N_T^0) for each. The conclusion was that only trap T_5 was affected much by λ or I_0 , but that even for T_5 , the chosen conditions [$\lambda = 0.855 \mu\text{m}$ (1.45 eV) and $I_0 = 3.3 \times 10^{14} \text{ photons/cm}^2 \text{ s}$] gave nearly the maximum peak height. Thus, we believe that the ratio N_T^0/N_T in our experiment is within 10% of unity for all of the traps, T_6^* , T_5 , and T_4 .

For trap T_6^* , which has a peak at 91 K, we must also consider emission during the 30-s time interval that the sample sits at 83 K after the light has been turned off but before the sweep has begun. Fortunately, the fitting of Eq. (5) to the NTSC spectrum gives $e_n(T)$ [Eq. (2)] and therefore the loss of neutral (filled) T_6^* traps can be easily calculated:

$$\frac{[T_6^*]^0(t)}{[T_6^*]^0(0)} = e^{-e_n(83 \text{ K})t}, \quad (7)$$

where $t = 30 \text{ s}$ in our case. It turns out that the loss is about 25%, and this factor must be included in the analysis.

Finally, we must analyze the effects of electron energy loss in a thick sample. Let E_d be the absorbed energy necessary to displace an atom ($E_d \approx 10 \text{ eV}$ in GaAs).³ Then the minimum (threshold) electron energy E_t that will transfer at least E_d to an atom of mass M is given by (Eq. 1.46, Ref. 1).

$$E_d = 2 \left(\frac{m_e}{M} \right) \frac{E_t(E_t + 2m_e c^2)}{m_e c^2}. \quad (8)$$

Thus, $E_t \approx 0.27 \text{ MeV}$ for $E_d = 10 \text{ eV}$. A numerical calculation using electron energy-loss theory¹² (most of the loss is due to electronic collisions) gives a range of 970 μm for an initially 1-MeV electron to fall to 0.27 MeV. Thus, appreciable energy loss will occur in a typical 600–700- μm -thick sample, and more displacements will occur near the upper surface than the lower surface. This situation is discussed in Ref. 1, and Eq. 1.57 of that reference can be rewritten as follows:

$$N_T = N_T(E) \left\{ 1 - \frac{d}{2[R(E) - R(E_{\text{th}})]} \right\}, \quad (9)$$

where $E = 1 \text{ MeV}$, and $E_{\text{th}} = 0.27 \text{ MeV}$, in this case. Thus, if samples of different thickness d are available, then a plot of N_T vs d will have an intercept $N_T(1 \text{ MeV})$ at $d = 0$. Such an analysis will allow comparison with DLTS results, which are concerned only with near-surface defects and thus can be analyzed by assuming a constant energy of 1 MeV.

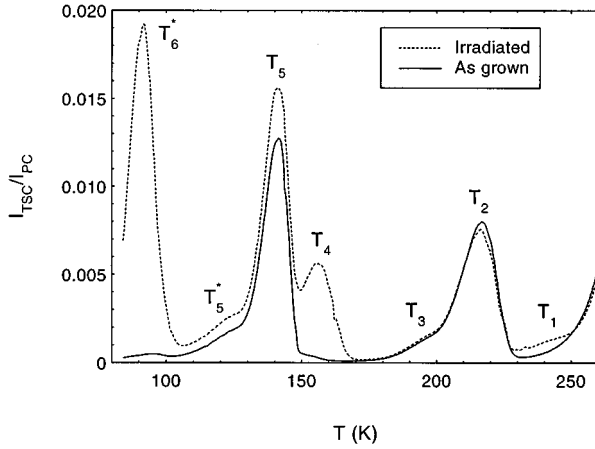


FIG. 1. Normalized thermally stimulated current spectra of as-grown and irradiated (5×10^{14} , 1-MeV electrons/cm²) semi-insulating GaAs.

EXPERIMENTAL RESULTS

Three adjacent 6×6 mm² pieces were cut from a 100-mm-diameter, 615- μ m-thick SI GaAs wafer grown by the low-pressure liquid-encapsulated Czochralski method. Hall-effect analysis determined a resistivity of 3.4×10^7 Ω cm, a mobility of 7200 cm²/V s, and a carrier concentration of 2.6×10^7 cm⁻³, all at 296 K. Transmission measurements [see the discussion following Eq. (5)] gave $N_{EL2}^0 = 1.5 \times 10^{16}$ cm⁻³, and $N_{EL2}^+ = 1 \times 10^{15}$ cm⁻³, typical results for such wafers. Ohmic contacts were formed from In dots on the sample corners, and the In was annealed at 425 °C for 5 min. Current was passed between diagonal contacts. (The geometric factor in such a case is not wd/ℓ , but it cancels out anyway in the ratio I_{TSC}/I_{PC} .) One of the three samples was lapped to 415 μ m, and another to 215 μ m, in order to apply Eq. (9).

Electron irradiation was carried out in a van de Graaff accelerator capable of supplying 40 μ A of 2.2-MeV electrons. However, in this experiment, a 1-MeV beam of electrons was passed through approximately 10 cm of air before hitting the target. This resulted in a target flux of only 250 nA/cm² that was applied for 6 min, giving a total dose of 5×10^{14} 1-MeV electrons/cm². This small dose had only a slight effect on the electrical properties, such as the dark current and photocurrent, but was large enough to produce substantial changes in several trap concentrations. NTSC data for the 215- μ m-thick sample are presented in Fig. 1. Clearly, very large increases of traps T_6^* and T_4 occur, smaller increases of traps T_5 , T_5^* , and T_1 , and no measurable increase of traps T_2 and T_3 . Data for all other samples investigated by us look substantially the same. Quantitative, least-squares fits to Eq. (5), without any approximations, were carried out for traps T_6^* (80–110 K), T_5 , and T_4 (130–170 K). The latter two traps had to be fitted simultaneously, i.e., $I_{TSC}/I_{PC} = (I_{TSC}/I_{PC})_5 + (I_{TSC}/I_{PC})_4$, because of the strong overlap. Excellent fits for all traps were achieved, as shown in Fig. 2 for traps T_5 and T_4 of the 215- μ m-thick sample. The fitting parameters are summarized in Table I. The N_T value in column 3 is determined by subtracting N_T (as-grown) from N_T (irradiated) for each of the traps.

From Table I, it is seen that the apparent trap concentrations vary with thickness. This effect was predicted by Eq.

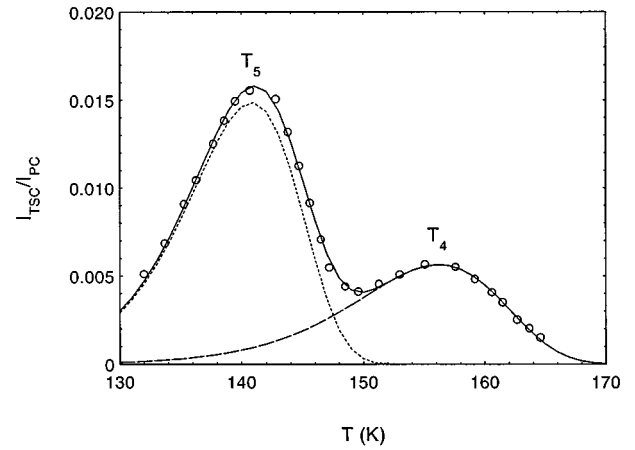


FIG. 2. A fit of the 130–170-K region in Fig. 1, which includes traps T_5 and T_4 .

(9) and is due to the electron energy loss in the thick samples. Plots of N_T vs d for traps T_6^* , T_5 , and T_4 are shown in Fig. 3, and straight lines are clearly found for traps T_6^* and T_5 , although the relationship for T_4 is more dubious. The concentration values extrapolated to $d=0$ [i.e., $N_T(1$ MeV) in Eq. (7)] are given in Table I, and lead to production rates of 0.70, 0.078, and 0.23 cm⁻¹ for traps T_6^* , T_5 , and T_4 , respectively. The corresponding values of $[R(E) - R(E_{th})]$ are 920, 840, and 1530 μ m, respectively, but the last value should be discarded, because the fit to Eq. (9) for trap T_4 is not nearly as good as the fits for the other two traps. (With only three points, if one of them is off, the whole fit is compromised.) As mentioned earlier, a numerical calculation of $[R(1$ MeV) - $R(0.27$ MeV)], from detailed nuclear scattering theory,¹² gives a value 970 μ m, in good agreement with our fits of the T_6^* and T_5 data. Thus, we evidently have included the electron energy-loss effect properly. It is recommended that such an analysis be employed for all investigations which use GaAs samples of normal thickness (600–800 μ m) and which involve the whole bulk. As mentioned earlier, however, DLTS analysis is not affected by energy loss be-

TABLE I. Fitting parameters for traps T_6^* , T_5 , and T_4 in samples of thickness (d) 215, 415, and 615 μ m, subjected to irradiation by 5×10^{14} 1 MeV electrons/cm².

d (μ m)	Trap	N_T (10^{14} cm ⁻³)	E_a (eV)	σ_a (cm ²)
215	T_6^*	3.10	0.13	4×10^{-19}
	T_5	0.34	0.34	3×10^{-14}
	T_4	1.10	0.31	7×10^{-17}
415	T_6^*	2.72	0.12	3×10^{-19}
	T_5	0.29	0.35	4×10^{-14}
	T_4	0.95	0.32	4×10^{-16}
615	T_6^*	2.33	0.12	1×10^{-19}
	T_5	0.25	0.33	1×10^{-14}
	T_4	0.94	0.30	5×10^{-17}
0 ^a	T_6^*	3.51		
	T_5	0.39		
	T_4	1.15		

^ay-axis intercept ($d=0$) of N_T vs d plot.

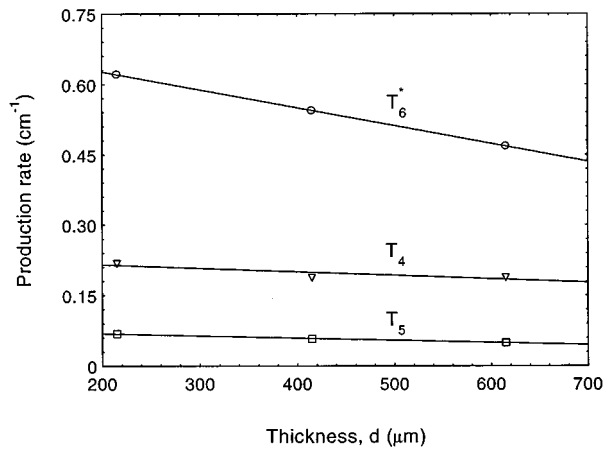


FIG. 3. The 1-MeV-electron production rates of traps T_6^* , T_5 , and T_4 as a function of sample thickness.

cause it involves only a small region (typically 0.1–1 μm) near the surface.

DISCUSSION

The main 1 MeV irradiation traps found by DLTS in *conductive*, *n*-type GaAs samples are $E1$ ($E_a=0.045$ eV, $\sigma_a=2\times 10^{-15}$ cm^2 , $r=1.5$ cm^{-1}), $E2$ (0.14 eV, 1×10^{-13} cm^2 , 1.5 cm^{-1}), and $E3$ (0.30 eV, 6×10^{-15} cm^2 , 0.4 cm^{-1}), where r is the production rate.³ Since the $E1$ peak would occur well below 80 K in the TSC experiment (as it does also in the DLTS experiment), we do not see a TSC peak analogous to $E1$ with our apparatus. However, we see strong similarities between $E2$ and T_6^* (0.13 eV, 4×10^{-19} cm^2 , 0.7 cm^{-1}), and between $E3$ and T_4 (0.31 eV, 1×10^{-16} cm^2 , 0.2 cm^{-1}). That is, (1) in each case (DLTS and TSC), they are the main irradiation traps; (2) their energies ($E2$ with T_6^* , and $E3$ with T_4) are very close; and (3) their respective production rates are within a factor 2. Only the capture cross sections differ greatly, especially between $E2$ and T_6^* .

Regarding the production rates, we notice that the ratios $r(E2)/r(E3)$, and $r(T_6^*)/r(T_4)$, are equal, within error; thus, it seems that a systematic error could be present in the NTSC analysis, the DLTS analysis, or both. In the NTSC case, the light intensity I_0 has some degree of uncertainty, because its measurement is accomplished by replacing the whole sample stage with a calibrated photodetector, and it is difficult to ensure that the sample and detector are in the exact same positions. For the DLTS case, calibration is accomplished through measurement of the background shallow donor concentration by the C - V technique, and this method also has sources of error, such as the determination of the diode area A ($n_{CV}\sim A^{-2}$). Thus, perhaps a factor 2 between the DLTS and NTSC production rates should not be considered unreasonable. It is also possible, of course, that the primary defect (probably V_{As}) production rate in SI material is inherently lower than that in a conductive material, due perhaps to charge-state effects. However, a recent Hall-effect study⁴ of the 0.15-eV defect in conductive GaAs finds a production rate of 0.6 cm^{-1} , very close to our NTSC value. At this point, we do not know why the DLTS production rate

differs from that found by the NTSC and Hall-effect methods.

The differences in apparent capture cross section σ_a are more difficult to resolve; however, several points can be noted in this regard. (1) In the DLTS case, and usually in the TSC case (but not here), cross sections are measured as an intercept of an Arrhenius plot.^{5,11} Because a slight error in slope (or E_a) gives a large error in the intercept (σ_a), the latter can be very inaccurate. As an example, in a DLTS study of the same sample by several different laboratories, the E_a of $EL2$ varied only from 0.72–0.84 eV, but the σ_a varied from 4×10^{-17} to 5×10^{-15} cm^2 , while the “accepted” value is 1×10^{-13} cm^2 (Ref. 11, p. 201). In our comparison, we are determining σ_a by two different methods (DLTS and NTSC), and in two different types of GaAs (conducting and semi-insulating, respectively). Thus, large disagreements in the values of σ_a should perhaps be expected. (2) The DLTS experiment is affected only by the surface region (typically 0.1–1.0 μm), and also involves a very high electric field (typically 10^4 – 10^5 V/cm). Such high fields can greatly affect emission rates. (3) Note that the T_5 and T_4 energies are quite similar, 0.31 and 0.34 eV, respectively, and their respective NTSC peaks are close enough that they might not be resolved in the DLTS experiment. Since σ_a for T_5 , $\sim 3\times 10^{-14}$ / cm^2 , is much higher than that of T_4 , the combination of the two σ_a 's might be similar to that of $E3$. The same situation might apply to the $E2/T_6^*$ combination; i.e., there could be another NTSC trap, with a similar energy to that of T_6^* , but a higher cross section. Its peak could occur below 83 K, in which case we would not see it. The DLTS defect $E2$ might then be a combination of T_6^* and this unseen NTSC peak. However, without lower-temperature measurements we cannot resolve this issue.

SUMMARY

We have developed a form of thermally stimulated current measurements, called normalized TSC, or NTSC, which eliminates uncertainties due to mobility, lifetime, and geometric factors, but which adds a new factor, the absorption coefficient. Fortunately, the infrared absorption coefficient in semi-insulating GaAs is well known and thus allows the NTSC technique to be completely quantitative in this material. Also, our analysis does not invoke the usual approximations (e.g., an Arrhenius plot of peak positions for different temperature sweep rates), but instead the whole NTSC spectrum, for only one sweep rate, is fitted exactly to the derived formula. This methodology was applied to 1-MeV electron irradiation in semi-insulating GaAs. Three irradiation traps were found: T_6^* at 0.13 eV, T_5 at 0.34 eV, and T_4 at 0.31 eV. Of these traps, only T_5 is commonly found at high concentrations in unirradiated SI GaAs; T_6^* and T_4 are sometimes found in low concentrations, although they may involve slightly different configurations (complexes) than those produced by the irradiation. The two main traps observed in *conductive* GaAs, by DLTS, namely, $E2$ and $E3$, are identified with T_6^* and T_4 , respectively. (However, $E3$ may involve both T_4 and T_5 .) From extensive previous analysis of the DLTS defects, we then identify T_6^* with the isolated As vacancy V_{As} , and T_4 and T_5 with $V_{\text{As}}\text{-As}_i$ complexes.

ACKNOWLEDGMENTS

We wish to thank M. Mier for the absorption measurements, T. Cooper for Hall measurements, L. Callahan for sample preparation, and R. Heil for manuscript preparation.

D.C.L. and Z-Q.F. were supported by U.S. Air Force Contract No. F33615-95-C-1619, and part of the work was performed at the Avionics Directorate, Wright Laboratory, Wright-Patterson Air Force Base, Ohio.

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