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Temperature dependence of the $EL2$ metastability in semi-insulating GaAs: Thermal hysteresis between the metastable and reverse transitions

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The temperature dependence of the photoquenching of the $EL2$ level in semi-insulating GaAs is studied by photocurrent and thermally stimulated currents. The observations made in these experimental procedures together with other results reported in the literature reveal that the metastable transformation of $EL2$ cannot be fully achieved when the sample temperature is above 85 K. This observation is compared with the thermal recovery of the $EL2$ ground state from its metastable configuration $EL2^*$, which is known to take place between 120 and 130 K; showing thus the existence of a conspicuous thermal hysteresis between both transitions, $EL2\rightarrow EL2^*$ and $EL2^*\rightarrow EL2$. This is analyzed in terms of the existence of a level that would play the role of an actuator of the metastable transformation of $EL2$. The charge state of this level can be altered by both optical excitation and temperature. Above 85 K it would be thermally emptied, being in such a charge state configuration unable to activate the metastable transformation of $EL2$.

INTRODUCTION

Technological interest in semi-insulating GaAs has been increasing in the last few years due to its application to high speed integrated circuits (IC's) and optoelectronics. The high resistivity of this material is ensured mainly by an omnipresent native deep donor, the so-called $EL2$ level,[1] that pins the Fermi level at the midgap, thus rendering the material electrically compensated. The main property of the $EL2$ level is an optically induced transition to a metastable state $EL2^*$, which is believed to be optically and electrically inactive.[2–4] This defect transformation is achieved by persistent illumination with subband gap light of the 1–1.3-eV spectral range. It is usually assumed that such a transformation is performed at temperatures below 130 K, which is the threshold temperature for the thermally assisted $EL2^*\rightarrow EL2$ restoration.[5–7] A detailed study of the temperature dependence of both transitions, $EL2\rightarrow EL2^*$ and $EL2^*\rightarrow EL2$, reveals a thermal hysteresis between them. One can experimentally observe that the high-temperature limit up to which the $EL2\rightarrow EL2^*$ transformation is achieved is significantly below the temperature threshold for the pure thermally induced $EL2^*\rightarrow EL2$ recovery, that in semi-insulating specimens is known to occur in the 120–130-K thermal interval.[5–7] Different experimental proofs of this astonishing hysteresis can be found in the literature; thus infrared absorption evidenced that most of the samples cannot be photoquenched, or that they show incomplete quenching above 80–85 K.[8,9] Photocurrent measurements reveal clearly the inability of semi-insulating samples to be photoquenched in this temperature range. Furthermore, TSC (thermally stimulated current) experiments demonstrated that the family of $EL2$-related traps cannot be photoquenched at temperatures above 85 K.[10,11] We present herein a photocurrent and TSC study of this thermal hysteresis in semi-insulating GaAs; this is explained in terms of a coupling between $EL2$ and another defect, which is henceforth labeled the actuator of the metastability. Depending on the charge state of this defect, the transition to the metastable state can be triggered or not. The charge of this level can be modified by quenching light excitation, and it can be thermally emptied at 80–85 K. This thermal release of charge will account for the temperature threshold of the $EL2\rightarrow EL2^*$ transformation.

EXPERIMENTAL SETUP AND SAMPLES

The experimental arrangement has been described elsewhere,[10–14] for both photocurrent and TSC experiments. Photocurrent measurements were carried out with the sample mounted in a closed-circuit helium cryostat (10–300 K). Electric contacts were made by alloying indium, and annealing at 400 °C in forming gas for 15 min. All the measurements were done at low bias in the linear part of the I_{ph} vs V characteristic plot in order to avoid nonlinear effects at the contact region; the linearity was tested by means of a lateral electrode. Photocurrent excitation was made with light from a 250-w halogen lamp filtered through either a monochromator and a band pass filter or an interference filter set. Electric current was recorded by either a logarithmic picoammeter or a digital electrometer.

TSC (thermally stimulated current) was carried out in a cryogenic system specially prepared to have a reliable temperature scan control between 4 and 300 K. The sample was mounted in a specially designed holder and

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then submerged in a liquid-helium dewar. The temperature was varied by displacing the sample holder with a stepping motor. The temperature scans were therefore reliably controlled. On the other hand, spurious infrared radiation was not present, thus avoiding unattended optical emptying of the shallowest carrier traps. The excitation was made through an optical guide mounted in the sample holder and emerging directly onto the sample. Photoquenching excitations were achieved with an yttrium aluminum garnet (YAG) laser (1.06 μm), while trap filling was done with monochromatic light from a halogen lamp and interference filters. Details of these measurements will be added further in the text.

The samples used for measurements were semi-insulating undoped LEC (liquid encapsulated Czochralski) grown. The room-temperature resistivity was $\approx 10^8-10^9$ Ω cm. The typical concentration of *EL*2 measured by infrared absorption was about $1-2\times10^{16}$ cm^{-3}. A few HB (horizontal Bridgman) samples were also studied, basically showing the same experimental behavior.

RESULTS

The signature of the *EL*2 level is the photoquenching of the extrinsic photoresponse. This photoquenching is observed by different experimental means; i.e., optical absorption,[1,2,8,9] photocapacitance,[3] photoluminescence,[4] photoelectron paramagnetic resonances,[15] photocurrent, etc.[12,16,17] It results that the way the photoquenching is seen depends on the physical magnitude that is being measured, since each one probes different aspects of the electrical and optical features of *EL*2 during the metastable transformation. This is consistent with a rather complex defect structure, as we will show below.

Photocurrent response is a function of the free-carrier photogeneration rate, the carrier mobility, and the mean photocarrier lifetime. All these parameters are dependent on the defect concentration and on the microscopic nature of the defects. Due to these different contributions it is complicated to assess the concentration of the different defects involved in the photocurrent response. In spite of this, the high sensitivity of the technique, as well as its complex response, provides valuable information about the physical mechanisms involved in the metastable transformation of *EL*2.

A systematic study of the photocurrent response of a great number of semi-insulating GaAs samples coming from different suppliers was carried out. In this study special attention was paid to the optical and thermal history of the samples in relation to the metastable transformation of *EL*2.

The I_{ph} (1.1 eV) vs t transients as obtained at different temperatures are shown in Fig. 1. Photoquenching cannot be achieved above 80–85 K in most of the many samples studied. A similar observation was reported by Mitchel, Rea, and Wu,[18] who associated such an observation with the anomalous quenching of the oxygen-related defect denoted *EL*0. One exception to this general behavior was an In-doped sample, which kept its quenching ability up to 100 K. In relation to this, it is worth noting that the samples that did not exhibit photocurrent quenching in this temperature range did not show photoquenching of the optical absorption either, which is a conclusive proof of the nonachievement of the metastable transformation at that temperature. Below this temperature both the photocurrent and optical transmission bleaching are performed in the usually reported terms.

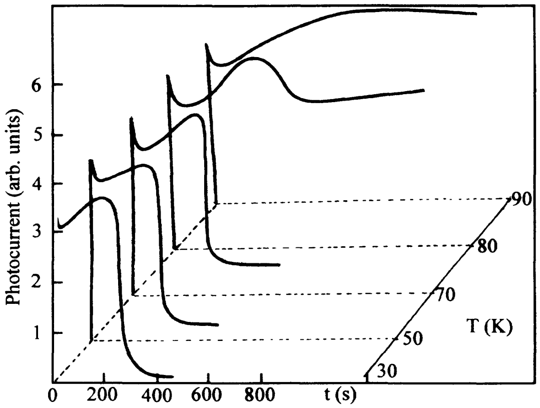

FIG. 1. Photocurrent (1.1 eV) quenching transients at different temperatures, showing that photoquenching cannot be fully achieved above 80 K.

In a similar way the family of *EL*2-related traps that are seen to photoquench under persistent excitation with quenching light cannot be quenched at temperatures exceeding the above-mentioned threshold, as reported in TSC measurements,[10,11] while below this temperature threshold these traps are photoquenched.[10,11,19] The family of *EL*2-related traps consists of all the traps detected by TSC that undergo metastable changes following excitation with quenching light. In this frame the traps that we are analyzing are B_1, B_2, and C_5. These changes affect both occupancy and trap parameters (capture cross section and activation energy).[10,11,29,30]

The typical photocurrent transient for quenching light excitation consists of two well-defined parts, henceforth labeled *A* and *B*, respectively. Part *A* does not correspond to the photoquenching of the photocurrent (no carrier removing is seen at this stage) but to a situation where the photoionization of *EL*2 is the main source of free carriers, which are majority electrons, as deduced from the *n* type of the photocurrent.[20,21] Part *B* is dominated mainly by the loss of optical activity of the *EL*2 levels as a consequence of the $EL2\rightarrow EL2^*$ transformation and the corresponding free-electron removal. Following this, weak *p*-type current remains.[20,21]

It is worth noting that the photocurrent transients necessarily obey physical mechanisms more complicated than those controlling optical absorption. The photocurrent transient will be determined by both photoionization and recombination and capture processes that limit the mean lifetime of photocarriers. We emphasize these points in relation to the importance that these mechanisms could have on the photocurrent transient. In this context we need to check the relation between photocurrent quenching and the metastability of *EL*2. This may be inferred from both the thermal recovery and

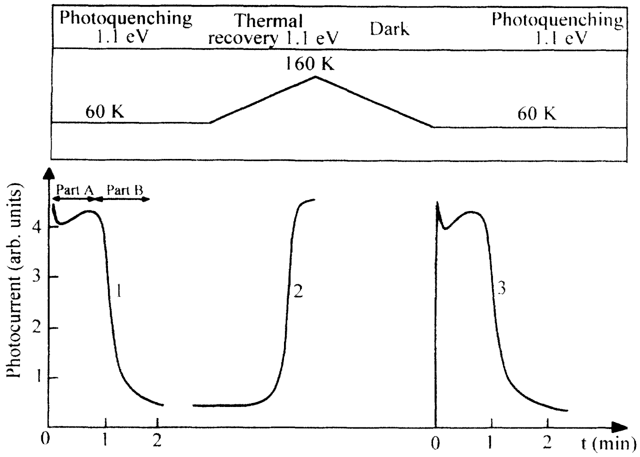

FIG. 2. Sequential photoquenching transient, thermal recovery, and photoquenching transient, relating both effects to the metastability of $EL2$.

spectral efficiency of photocurrent quenching. Figure 2 shows a typical result obtained in a thermal recovery experiment; first the photocurrent is completely bleached by 1.1-eV persistent light excitation at low temperature (60 K), then the sample is slowly warmed up under illumination (1.1 eV), and the photocurrent is recorded showing a steplike rising at around 120 K; this is similar to that obtained in optical-absorption restoration, since an activation energy of 0.3 eV is measured when the p to n conversion of the photocurrent is considered.[22] After cooling down in darkness and then exciting with 1.1-eV light, the original photocurrent transient is reliably reproduced. Similar measurements were performed in TSC in order to identify the traps related to $EL2$. The thermal recovery of the TSC signal is also accomplished at 130 K with an activation energy close to 0.3 eV. On the other hand, it was shown elsewhere[17,23,24] that the optical efficiency spectrum for the photocurrent quenching agrees with that deduced from other experimental techniques, Fig. 3. These experimental results evidence a close relation between the photocurrent quenching and the metastable transformation of $EL2$.

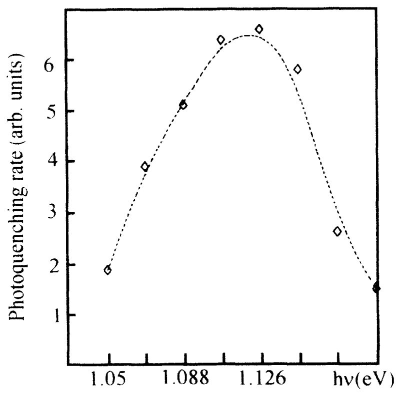

FIG. 3. Spectrum of photoquenching as obtained from photocurrent quenching transients.

Different interpretations can be deduced from the absence of photocurrent quenching above 80–85 K.

(i) Quenching is produced but another photocurrent effect, the so-called EPC (enhanced photocurrent),[25,26] prevents its observation. This possibility can be rejected because, after illumination with quenching light at 80 K, photocurrent quenching is observed when the sample is cooled down in darkness to a temperature low enough to achieve the photoquenching effect.

(ii) There is competition between photoquenching and a restoration effect known as the photoassisted thermal recovery (PATR),[27] balance being dominated by PATR above 80 K and PCQ below this temperature. This point requires some discussion in order to avoid confusion about the origin of the thermal hysteresis. The PATR effect was discussed in Ref. 27; this effect was reported to occur only in a limited number of samples, being only partial in the semi-insulating samples that exhibited it, with no more than 20% of the total quenching produced at lower temperature. Thermal hysteresis is different, since it was a general observation over the many samples we have studied. On the other hand, it is not a partial effect. In order to clarify this we performed the following sequential measurements: Bleaching was thoroughly achieved at 30 K, then the sample was warmed up between 85 and 95 K under quenching light excitation and then cooled down to 30 K in darkness; the subsequent illumination with quenching light did not show any vestige of photoquenching, thus ruling out any PATR effect. Another proof was obtained since photoquenching is observed at 80 K after a previous excitation in the low-temperature range with quenching light without achieving the photocurrent quenching; this is described with more detail in Ref. 28.

These results make clear that the observation of photocurrent bleaching requires a previous optical-assisted process that most probably takes place during the initial stages of the photocurrent transient, which we have generally labeled part A. This optical process is not possible above 80 K. Below this threshold it can be activated and it remains stable as the temperature is kept below 90–100 K; above this temperature the activation produced at low temperature no longer works and the photoquenching cannot be done. If photocurrent quenching is completely achieved, no significant changes are seen after annealing at such a temperature. The $EL2$ levels that are activated are called quenchable levels, $EL2_q$, in contrast to the levels that are not activated and that are labeled nonquenchable, $EL2_{\mathrm{nq}}$.

It is not impossible for the metastable transformation of $EL2$ to start before photocurrent quenching is observed; such a transition to metastability occurs only when suitable conditions are previously built up by optical excitation. In other words, a triggering of the metastable transition is needed. This stage will consist of the formation of the quenchable levels, $EL2_q$.

In Fig. 4 some TSC (thermally stimulated current) experiments are presented that strongly reinforce our hypothesis about the need for a permanent change of the defect charge state prior to the metastable transformation of $EL2$. It is known that the same light that quenches

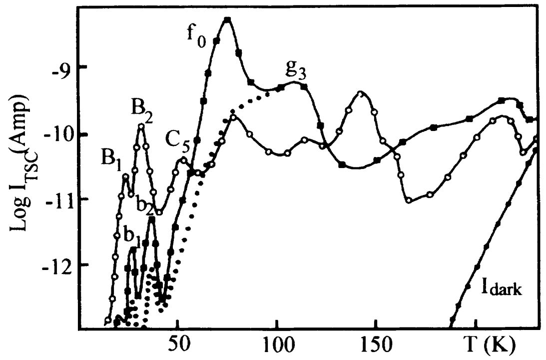

FIG. 4. TSC (thermally stimulated current) spectra obtained under different bleaching conditions at 4 K, showing photoquenching of traps labeled B_1, B_2, and C_5. (○) before photoquenching, (●) after photoquenching, and (■) after photoquenching and optical recovery with near-band-gap light. b_1 and b_2 do not correspond to the same defect as B_1 and B_2, respectively, as can be deduced from the calculated trap parameters (Ref. 10): B_1 ($E_t=11.2$ meV, $\sigma=2\times10^{-22}$ cm^2), b_1 ($E_t=24$ meV, $\sigma=1.6\times10^{-20}$ cm^2), B_2 ($E_t=42$ meV, $\sigma=5\times10^{-18}$ cm^2), and b_2 ($E_t=49$ meV, $\sigma=2\times10^{-18}$ cm^2).

the $EL2$ photoresponse is capable of producing some deep changes in the TSC spectrum,[10,11,29,30] affecting both the occupancy of traps and their physical parameters. These changes in the TSC spectrum do not merely consist of the different trapping rate due to the quenching of the center providing the photocarriers, they are also the result of a close relation between $EL2^*$ and several of those traps.[10,11,29,30] In Fig. 5 the temperature dependences of both the photoquenching and thermal recovery of three of the most significant of the $EL2^*$-related traps are shown, labeled B_1, B_2, and C_5; see Fig. 4. These measurements were carried out as follows: (i) The sample is excited with quenching light at a temperature T_q. (ii) The sample is cooled down in darkness to 4 K. (iii) At this temperature the sample is illuminated with band-gap light in order to fill the traps. (iv) The TSC is recorded. (v) This procedure is carried out for different T_q's and the integrated intensity of the TSC peaks is plotted as a function of T_q.

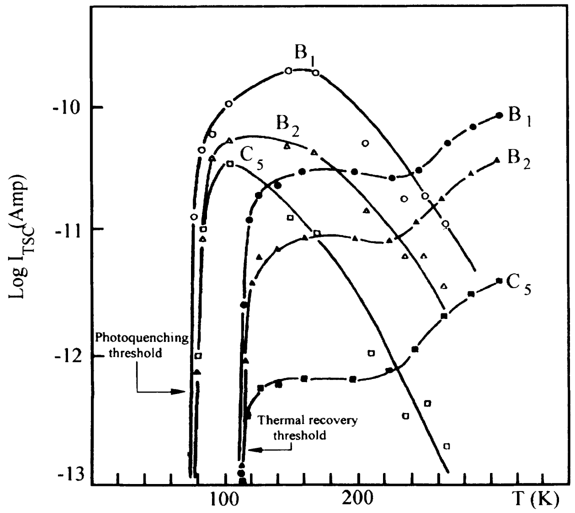

FIG. 5. Evolution of the current intensity of TSC peaks B_1, B_2, and C_5 (see text) showing two well-defined temperature thresholds for photoquenching and thermal recovery.

The plots corresponding to the thermal recovery of the $EL2$-related TSC peaks in Fig. 5 were obtained according to the following sequence: (i) The sample is excited with quenching light at 4 K. (ii) The sample is then warmed up in darkness to a temperature of restoration, T_r. (iii) The sample is then cooled down in darkness to 4 K. (iv) Band-gap light illumination fills the traps. (v) The TSC is recorded and the intensity plotted as a function of the restoration temperature T_r.

The difference between both plots is clearly seen; on the one hand, photoquenching is limited to temperatures below 85 K, while once it has been produced the threshold for the reverse transformation is 110–115 K. It should be noted that the activation energy is 0.3 eV, which again is consistent with the fact that the $EL2$ metastability is involved.

In Fig. 6 the thermal recovery of the photocurrent is shown as compared to the temperature dependence of the photocurrent quenching; the existence of a thermal hysteresis between both of these transitions [($EL2\rightarrow EL2^*$) and ($EL2^*\rightarrow EL2$)], is evident. This is in full agreement with TSC results, Fig. 5. It should be noted that the temperature margin separating the quenchability from the nonquenchability is very narrow, as can be observed in the sharp slope of the photoquenching in Figs. 5 and 6. The difference in the temperature thresholds between both kinds of experiments can arise from better precision in the temperature control of the TSC experiments, as was pointed out in the description of the experimental setup.

The observed thermal hysteresis enables us to claim that the metastable transformation of $EL2$ cannot occur as long as the temperature is above 85 K, while once the metastable state has been built up at lower temperature it remains stable up to 130 K, without significant changes

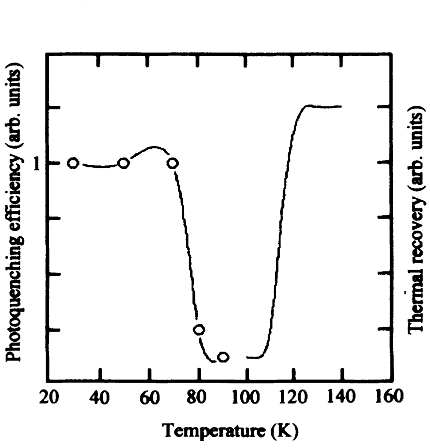

FIG. 6. Thermal hysteresis between photocurrent quenching and the thermal recovery of the photocurrent, showing the existence of different temperature thresholds for either of these processes.

between 85 and 110 K. The existence of a level activating the quenchability of $EL2$ was recently argued by the authors[28] in order to account for several experimental results tightly related to those presented herein; in this framework we will try to present a relationship between such an actuator level and the thermal hysteresis herein investigated.

DISCUSSION

The experimental results concerning photocurrent and TSC presented above show in an unambiguous way that the $EL2$ metastable transition requires a previous transformation to be accomplished, which converts $EL2$ into its quenchable state, $EL2_q$. Such a transformation cannot be fully achieved as the temperature is above 80–85 K. The existence of an optically induced charge transfer involving defects other than $EL2$ could constitute a reliable explanation for the activation as a triggering mechanism of the metastability of $EL2$. In the literature there are numerous references to such processes; a family of levels has been reported to be related to $EL2$ as demonstrated in TSC (Refs. 10, 19, 29, and 30) and electron paramagnetic resonance (EPR) (Refs. 31–33) experiments by several authors. Charge photoexcited from $EL2$, either neutral or ionized, is trapped by other levels, either donors or acceptors. A great deal of attention must be paid to these acceptor levels, since the sample after the metastable transformation remains semi-insulating, which means that the holes released during the sequential transformation $EL2^+ \rightarrow EL2^0 \rightarrow EL2^{*0}$ have to be trapped elsewhere if the usually accepted hypothesis of the neutrality of metastable $EL2^*$ is true;[3] in other words, this last assertion necessarily implies the existence of additional hole traps deep enough to warrant the electric compensation once the $EL2$ level is in the metastable state and hence electrically inactive.

An important point in relation to this is to decide if the holes are bound as a consequence of metastable transformation or if they are already trapped before the transition to the metastability; this last hypothesis seems to agree better with the existence of an activation of the metastability of $EL2$. It should be noted that prior to photoquenching, an excitation with light other than quenching light, i.e., near band-gap light, can modify the photoquenching transient, thus accounting for the activation of the metastability. It was shown by Kaufmann, Wilkening, and Baeumler[34] that the $FR3$ EPR signal, corresponding to the paramagnetic charge state of an unknown acceptor level, can be permanently photoexcited with 0.9-eV photons, even though a complete excitation is achieved only with quenching light. All that converges to the idea of charge-transfer processes during and prior to the metastable transformation.

It follows that the existence of a hole trap level that under suitable charge state conditions could trigger the transient to the metastable state of $EL2$ is a reliable hypothesis that could account for the complex mechanisms leading to the metastability. This hole trap level will henceforth be labeled the actuator of the metastability of $EL2$, and the quenchable $EL2_q$ levels are those $EL2$ levels associated with an actuator level that has trapped a hole. When the temperature is raised above 80–85 K the hole is thermally related and the metastability is not triggered. The experimental results we have presented suggest that the thermal emission of a hole from the actuator level takes place at around 80 K. The thermal hysteresis can thus be related to the trapping and detrapping of the charge on the actuator level. The occupancy of such a level can be modified substantially by the illumination with quenching light below 80 K. Above this temperature the charge can be thermally released, thus preventing the quenchability of $EL2$. These features are closely related to the f_0 trap of the TSC spectrum.[10] For instance, we can tentatively propose a relation between $EL2$ and the f_0 trap that could be the actuator level under specific charge conditions reached by suitable optical excitation. Nowadays we are not sure about this relation because we cannot elucidate whether the charge can be trapped at f_0 before the $EL2 \rightarrow EL2^*$ transformation, which could be a consistent test for the importance that f_0 has on the activation of the metastability. In this frame, the behavior of f_0 in the experimental conditions of Fig. 5 does not help to elucidate its role. It presents a strong enhancement without changing its trapping parameters ($E_t = 78$ meV and $\sigma = 7 \times 10^{-21}$ cm^2), which suggests that it becomes a dominant trapping level under quenching conditions.

The existence of acceptor levels deeper than C and Zn shallow acceptor impurities is now well established in semi-insulating GaAs in concentrations lying in the 10^{15}–10^{16}-cm^{-3} range.[35–40] The nature of these acceptors is not well determined today, but they are thought to be related to native defects, either alone or complexed. The concentration of the actuator level should be practically the same as that of $EL2$ in order to account for the full quenching of $EL2$, as well as for the electric compensation after the $EL2^+ \rightarrow EL2^{*0}$ transformation.

The observation of very close experimental features of the activation in different samples might imply that the relation between $EL2$ and the actuator level does not constitute a random association, but it must obey preferential defect association typical of the As-rich GaAs growth. A relation between $EL2$ and acceptor levels has been claimed in order to account for the electric compensation in one of the most generally approved $EL2$ defect models, the $\mathrm{As_{Ga}}$-As_i complex.[37] The scattering of the photocurrent transients measured could indicate that the activation mechanisms can be influenced by competition with other trapping defects; this could account for changes in the charge-transfer mechanisms leading to the activation of the metastability; the existence of such transfers seems to be of capital importance for understanding both metastability and compensation in the metastable configuration. This agrees with the idea of a family of $EL2$ defects. It should be noted that photoquenching is more easily achieved in irradiated[38] (with either protons, electrons or γ rays) and in In-doped[39] samples, which is consistent with a stronger coupling between the defects conforming the metastable transformation in these specimens that are characterized by the presence of native defects and lattice hardening, respectively. It should be noted that photoquenching in the 80-K temperature range is better accomplished in HB samples than in undoped LEC samples.[18]

The thermally activated charge release from the actuator level satisfactorily explains the existence of an upper temperature limit for the $EL2 \rightarrow EL2^*$ transition. But it raises a new question as to the problem of the thermal recovery of $EL2$, or the reverse $EL2^* \rightarrow EL2$ transition; in other terms this is the problem of the thermal stability of $EL2^*$ in the 85–110-K temperature margin.

It has been well established that there is not a significant thermal recovery of $EL2$ below 110 K;[5–7] in fact, after photoquenching, thermal annealings below 110 K do not reproduce any vestige of the quenching transient, which is, however, reproduced by annealing above this temperature. This leads to different issues for this problem.

(i) The charge transferred to the actuator level does not necessarily remain there once the metastable state has been raised.

(ii) The actuator level retains the charge, but the thermal release of this charge after the metastability was done does not mean any change for the metastable state.

(iii) The metastable transformation affects the electronic levels of the actuator, therein locking the trapped charge as the temperature is kept below the thermal recovery threshold.

Hypotheses (i) and (ii) would assign the role of a catalyst of metastable transformation to the actuator level, since the stability of the metastable state once it has been raised does not depend on the charge state of the actuator; simultaneously these hypotheses implicitly assume that the holes initially in $EL2^+$ after photoneutralization have to be bound in a center other than the actuator. A possible candidate is $EL2^{++}$, that is deeper in the band gap than $EL2$.[41]

The third hypothesis would imply that the actuator is integrated in the metastable complex and that the holes released by $EL2^+$ are trapped in the actuator itself. Thus it could be argued that the thermal release of a hole from the actuator would induce the thermal recovery of the ground state of $EL2$. This model excludes the identification of the f_0 traps as the actuator level, as deduced from the observation of the f_0 trap in the TSC spectrum after photoquenching. In this case the actuator will be thermally emptied at 80–85 K before photoquenching, and at 120–130 K after photoquenching. This behavior could be related to some of the traps that undergo photoquenching, whose thermal behavior is well described in Fig. 5. Thus the role of the B_1, B_2, and C_5 traps might be the formation of a complex with $EL2^*$. At the present time this role cannot be established unambiguously; however, these traps can be ruled out as the actuator levels, since their thermal emission in the absence of quenching is well below the temperature threshold of the metastable transformation (≈ 85 K).

CONCLUSION

The observation of a thermal hysteresis between $EL2 \rightarrow EL2^*$ and $EL2^* \rightarrow EL2$ transformation leads us to assume the existence of an actuator level, the charge state of which controls the transit to the metastable state. This is only possible under a specific charge state of this level, which can be charged by quenching light excitation. Before photoquenching this level is thermally ionized at 80–85 K, thus preventing the transition to the metastable state. The thermal stability of the metastable state between 85 and 110 K suggests several possible relations between $EL2$ and the actuator level. The first one is that the actuator level is only a catalyst for the metastable transformation. The second is that it is a part of the metastable complex. This last hypothesis supports the idea that electrical compensation under bleaching is ensured by the actuator level itself.

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