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DEFECTS IN SEMI-INSULATING GALLIUM ARSENIDE

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Abstract

Defects in semi-insulating gallium arsenide are reviewed, especially these contributing to compensation between donors and acceptors, which are responsible for the very high electrical resistivity of the material. Compensation mechanism - fine balance between native deep donors, called EL2, shallow acceptors,(C, Zn, Be, etc.) and shallow donors (Si, Te, Se etc.) is discussed in details. Special emphasis is put on two subjects: carbon as an impurity in GaAs, which is believed to be the most important shallow impurity, and b) extrinsic and intrinsic defects (yet unidentified) introducing deep traps in forbidden energy gap of GaAs, which do not only contribute to the donor-acceptor balance but also strongly influence some important opto-electronic properties of SI GaAs (photosensitivity, spectral photoconductive response, persistent photocurrents etc.).

1. INTRODUCTION

In the course of the whole fascinating history of the fast development and wider and wider use of semiconductors, defects in crystal lattice have been considered as unwanted, harmful and detrimental; affecting numerous electrical and optoelectronic properties. Only in recent years this attitude

started to change in the case of silicon, when it was found that some defects can play a beneficial role as well (hardening of wafers due to the presence of residual oxygen, effects of gettering of fast diffusing residuals at intentionally introduced surface defects etc.). Similarly, in gallium arsenide the intentional introduction of one native lattice defect, called EL2, enabled for the first time the growth of undoped semi-insulating crystals (SI-GaAs), which are now extensively used as a substrate for field-effect transistors and integrated circuits production. So again, the emphases is shifting from elimination of all defects from the crystal toward the control over defects and even its use for some well defined purposes.

Properties of EL2 and some other specific defects have been reviewed recently. New developments in the study of the EL2 defect in GaAs were presented in twelve papers of the Revue de Physique Appliquee(1). Excellent review of native defects in GaAs has been given by Burgoin and Bardeleben(2), while discussions on isolated residual impurities(3) and on substitutional donor impurity, which gives rise to an additional resonant level in GaAs(4) will appear soon in the same journal. In this paper we will present an overwue of our investigations of some other defects important in SI GaAs, with emphasis on two subjects: a) carbon as an impurity in GaAs, which is beloved to play a crucial role in the compensation mechanism, and b) (jet unidentified) defects introducing deep traps in the forbidden energy gap, which not only contribute to donor-acceptor balance, important for compensation, but also strongly influence some optoelectronic properties of SI GaAs (photosensitivity, persistent photocurrents, life-time of photo-generated carriers etc.).

2. COMPENSATION MECHANISM IN SI GaAs

GaAs is considered to be semi-insulating when its electrical resistivity exceeds 10^7 ohm cm. Room-temperature electron mobility in undoped material can easily exceed 5000 cm^2/V s, which means that free electron (and hole) concentration is lower than $10^8/cm^3$. We will first show that such a low free carrier concentration can be obtained only through the compensation mechanism, and not through perfection and purity of GaAs crystal: In GaAs there are 2×10^{22} atoms of Ga and As

in each cm^3, which is 10^{14} times higher than the free carriers concentration. If this extremely low free carrier concentration is to be obtained through crystal perfection, the concentration of foreign atoms, acting as shallow donors or acceptors as well as of native electrically active defects should be lower than $10^{10}/cm^3$, which is 4-5 order of magnitude better than what is achievable with today's technology and seems inconceivable even in distant future.

In previous generation of SI GaAs substrates the semi-insulating property used to be obtained by doping with chromium atoms, which are deep acceptors in GaAs. Mid-gap level of Cr in GaAs forbidden energy gap is deep enough so that only 10^{-8} fraction of Cr atoms is ionized at room temperature. Several times $10^{16}/cm^3$ chromium atoms used to be introduced intentionally in GaAs during the crystal growth, and they were dominant foreign atoms in the lattice. The main residual impurities, usually in $10^{15}/cm^3$ range, were silicon atoms, which are shallow donors. (In fact part of Si atoms, typically 25%, land at the As site in the lattice, where they act as shallow acceptors, but fraction of donor-like Si atoms at Ga places is always larger). The main and unavoidable source of silicon contamination was quartz crucible, in which the crystal growth was carried out. Silicon donors would compensate other, less numerous residual shallow acceptors, while Cr deep acceptors would compensate donors.

Recognition and control over native defect EL2, which acts as deep donor, and crystal growth in pyrolitic boron nitride (PBN) crucible enabled growth of SI GaAs crystals at considerably higher level of purity. The nature of EL2 defect is still unclear. There is a consensus that this defect include arsenic atom at gallium site (antisite arsenic, As_{Ga}). The other part of the defect could be interstitial arsenic atom, or As and Ga divacancy, or something else. There is a vast amount of work dealing with unusual properties of this defect, including metastability. These results won't be analyzed here, as they have been reviewed in detail recently(1). EL2 defects can be introduced in controlled way in several times $10^{16}/cm^3$ concentrations if As overpressure is maintained in the system during the crystal growth. The use of PBN crucible, instead of quartz, reduce drastically silicon contamination. Therefore in this new generation of SI GaAs crystals the main

contaminant seems to be carbon. Graphite heating elements and other graphite components in the growth chamber are possible sources of the carbon and/or carbon dioxide which contaminate the GaAs melt. The resulting concentration of carbon acceptors in typical liquid-encapsulated-Czochralski (LEC) is in the 10^{14}-10^{15}/cm^3 range, and is comparable to the total concentration of residual donor impurities. In such crystals carbon and other residual acceptors compensate all electrical activity of shallow donors, while surplus of shallow acceptors is compensated by deep native donors EL2. This fine balance has to be maintained both in microscale along the wafer and on macroscale along the GaAs boule in order to maintain the uniformity of high electrical resistivity and other relevant properties. Therefore radial and axial distribution of both carbon and EL2 need to be known in detail. In spite of its obvious importance, the data on the distribution of carbon in GaAs crystals have been inconsistent and contradictory, reporting values for k from 0.8 to 2.4.[5-10]. Results of detailed investigation on distribution coefficient of C in GaAs, which will be summarized in the next section, give new insight in possible role of carbon in compensation mechanism in SI GaAs.

3. CARBON IN GaAs

3.1 Distribution coefficient of carbon in GaAs

All GaAs ingots used in this study were grown by the LEC method using PBN crucibles. Crystals were pulled in <111> directions. The growth rate varied from 5cm/h for the fastest-pulled boule to only 0.625 cm/h for the slowest growth. The concentration of carbon was determined using FTIR (Fourier Transform Infrared Spectroscopy, where the sensitivity limit is in the range of 10^{14} C-atoms/cm^3. The loss or accretion of carbon from the melt and solid during the crystal growth was negligible, as analyzed in details in Ref.(10).

3.1.1. Results

The distribution of carbon along the pulling direction (axial distribution) was measured, and plotted against (1-g), where g denotes the fraction of GaAs melt which has been solidified. Changes of carbon concentration along the ingots are presented in Fig.1, with the pulling rate, R, as the

parameter. We have found that the concentration of carbon, Cs, decreases from the seed toward the tang end in all GaAs ingots . It is obvious that small puling rate causes more pronounced changes in carbon concentration, and vice versa, indicating that the measured, effective distribution coefficient of carbon depends strongly on the pulling rate.

For every pulling rate the effective distribution coefficient can be determined from the slope of log C_S vs. (1-g) plots, while equilibrium distribution coefficient, k_O, can be evaluated by extrapolation for R=0. Namely, the relationship between effective and equilibrium distribution coefficients can be expressed as:

$$k_{eff} = \frac{k_O}{k_o + (1-k_o)\exp(-Rd/D)} \qquad (1)$$

where d is the effective diffusion layer thickness and D is diffusion constant of the impurity in the dilute liquid. Consequently:

$$\ln\left(\frac{k_{eff}-1}{k_{eff}}\right) = \ln\left(\frac{k_O-1}{k_o}\right) - Rd/D \qquad (2)$$

Fig. 2. depicts the dependence of $\ln((K_{eff}-1)/k_{eff})$ on R. To the limits of experimental error this dependence is linear.

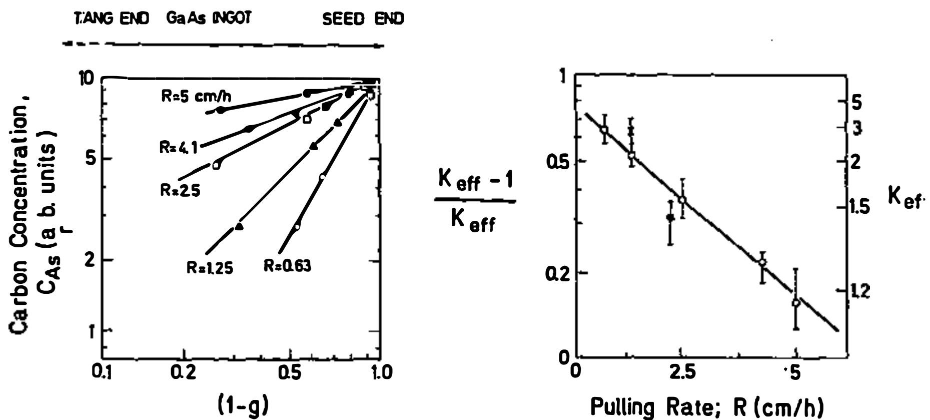

Fig.1 Carbon concentration (scaled to the same initial concentration in solid) along the LEC-grown GaAs ingots, pulled at different rates R (0.625÷5 cm/h)

Fig.2 Effective distribution coefficient of carbon in LEC-grown GaAs as a function of pulling rate. Full dot represents result from Ref. 7. The cross refers to the crystal grown from different charge material.

The extrapolation to R=0 yields a value for k_o of about 3.5.

3.1.2. Discussion

The value of 3.5 obtained for k_o is considerably higher than earlier published data of k_{eff}. However, we have shown that k_{eff} is strongly dependent on the pulling rate. The value of k_{eff}=1.44+0.08 obtained by Kobayashi and Osaka[7] at a pulling rate of 2cm/h is in good agreement with our results, as shown by the solid circle in Fig.2. The carbon distribution in the ingots grown by Chen, Holmeas and Asbeck[5] also showed that k must be larger than 1. Unfortunately, they did not specify their pulling rate, so a quantitative comparison with our results is not possible. Within the narrow range of the initial carbon concentration in the melt ($1-4\times10^{15}/cm^3$), we did not observe the influence of carbon concentration on effective distribution coefficient, as reported by Kirkpatrick et al.[8]. However, even if there is some concentration dependence for k_{eff} it is obviously insignificant in comparison with much stronger influence of pulling rate on carbon distribution along the ingot. The earlier published k_{eff} values of 0.8 and 0.9[6,11] were estimated from Hall-effect analysis. The presence of other donors and acceptors can easily mislead the interpretation of experimental data in Hall measurements.

In order to compare the experimentally obtained distribution coefficient of carbon with a predictions based on chemical trends, we plotted measured k (k_{eff} and k_o) for group IV elements in GaAs against their heat of sublimation values[10] and against their covalent radii[12]. In both cases extrapolation of these trends from Pb, Sn, Ge and Si to C indicated that one should indeed expect high k values for C in GaAs; in accordance with our experimental finding of k_o=3.5.

3.2 Consequences of high value of distribution coefficient of Carbon on its role in compensation mechanism in SI GaAs

A large equilibrium distribution coefficient of C in GaAs can have serious practical consequences for the compensation mechanism in SI GaAs and especially for a semi-insulating to p-type conversion. Wafers cut from the tang end of the ingot will have a considerably lower carbon concentration than wafers cut from the seed end, especially in ingots grown at low pulling rates. If carbon is really the main shallow acceptor,

and thus responsible for compensation of residual shallow donors, then its presence is crucial in obtaining semi-insulating GaAs. A considerable decrease in its concentration at the tang end should lead to n-type conductivity in this part of ingots. This effect should be further enhanced by the fact that all important shallow donor impurities (Si, Se, Te) have distribution coefficients lower than 1, and hence tend to accumulate toward the tang end of the ingot. Experimentally, however, just the opposite behavior is observed; LEC-grown GaAs ingots often tend to become p-type at the tang end. Our results obviously rule out the possibility that the accumulation of carbon at the tang end can contribute to the semi-insulating to p-type conversion.

We have to conclude that some other acceptors (native defects or B complexes, for example) have to play an important role in the compensation mechanism observed in GaAs, and that the question of main acceptors in SI GaAs is still (or better to say again) unresolved and open to further investigation.

4. DEFECTS WITH DEEP TRAPS IN SI GaAs

GaAs has relatively large forbidden energy gap (1.42 eV at room temperature). Some extrinsic and intrinsic defects introduce localized levels in this gap, contributing to donor-acceptor balanace and compensation mechanism. Beside that, these defects seem to be also responsible for some peculiar opto-electronic properties, which will be described and interpreted in this section.

If SI GaAs is illuminated at low temperature with photons in the 1-1.35 eV range, the decrease (quenching) of several opto-electronic properties has been observed, particularly that of photocapacitance[14], photoconductivity[15], optical transmission[16] and luminescence[17,18]. All these changes have been convincingly attributed to the metastability of EL2 defect center. However, in several very recent papers an increase in photoconductivity[19-26] and absorption[27,28] is reported for some specific illumination conditions. The consistent explanation for these phenomena still does not exist, and they have been tentatively interpreted either as a result of photogeneration of complex defects[19,23] or due to the change of population of EL2 level[27] or due to the switching of EL2 defect into its metastable state[22].

The other peculiar phenomenon, observed in SI GaAs after low temperature illumination is "persistent photocurrent" (I_{PP}). It was discovered first in nonhomogeneous configurations, like n-n^+ structures(29,30), and more recently in bulk SI GaAs as well(23). Basically, this effect consists of an anomalously long decay of photogenerated free carriers density after the illumination is switched off. Two conflicting interpretations presently exist(31). The first assumes macroscopic potential barriers, which spatially separate photogenerated electron-hole pairs and suppress their recombination(29). The second interpretation postulates microscopic barriers against recombination, either due to Coulombian repulsion(31) or due to large lattice relaxations(32). One interpretation(23) suggests that metastable states with the repulsive potential barriers are created by the very same photons which cause photoexcitation, if the energy of those photons is in the 1-1.25 eV range.

In our recent investigation(33) we have found that both effects, the photosensitivity and persistent current are connected with the presence of defects acting as electron or hole traps, and with their charge state. This type of defects is always present in wide-gap semiconductors, GaAs included(15,21,34-37).

We investigated deep traps and their trapping dynamics using thermally stimulated current (TSC) method. High quality wafers cut from commercial, Chochralski grown undoped SI GaAs material, were used in this study. All samples were slightly n-type, room temperature free carrier concentration was $2\text{-}3\times10^{8}$ cm^{-3}, and mobility around 5000 $cm^2V^{-1}s^{-1}$, with dark conductivity dominated by E_c-0.76 eV level, regularly ascribed to the EL2 defect(36). The non-ionized EL2 concentration (determined by standard absorption measurement in near infra-red) was $2\times10^{16}cm^{-3}$. Time evolution of photoconductivity (i.e. photo-generated current I_{pc}, per Volt), as well as TSC measurements after the filling of traps was carried out. The applied bias was 10 V, for which electrical contacts showed ohmic behavior, and the electric field was weak enough so that did not disturb trap filling(15). Light excitation was provided by a tungsten lamp and a set of narrow-band filters.

4.1 Results

Similarly to previously cited papers[19-25], we also observed the increases of photosensitivity (defined as photoconductivity per unit incident photon flux density), during the illumination with low light intensities[26].

Several characteristic curves depicting the time evolution of I_{pc} are presented in Fig.3, for two above-the-gap (curves a and b) and three below-the-gap photon energies (curve c, d and e). Photon flux density was around 1×10^{13} $cm^{-2}s^{-1}$, and temperature 85K. The above-the-gap photons induce an abrupt rise of I_{pc} followed by slow and continuous further increase (for another order of magnitude) during next 40 minutes of illumination. On the other hand, below-the-gap illumination causes continuous increase in I_{pc} for up to six orders of magnitude. However, this rise is not steady and several stages can be distinguished.

Persistent photocurrent, I_{PP}, was determined for different stages of photosensitivity in the following way:
The sample was cooled in dark from 330K to 85K, where it was illuminated for specific length of time; the light was switched

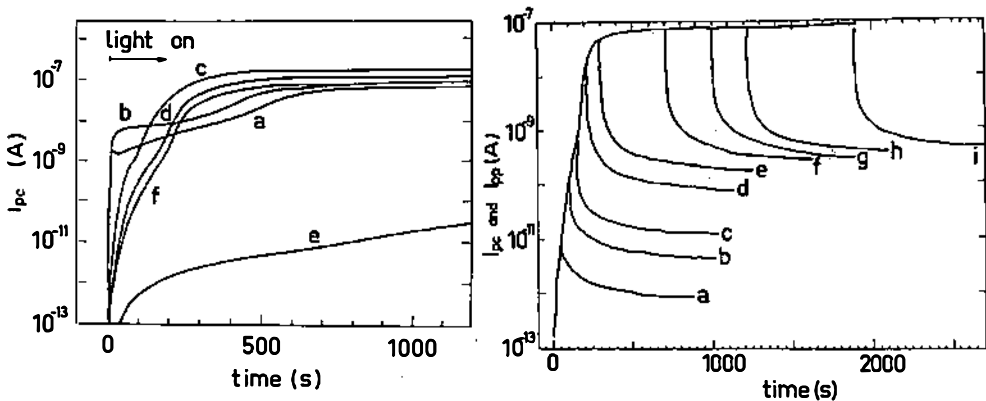

Fig.3 Time evolution of photoconductivity at 85K for illumination with 10^{13} $cm^{-2}s^{-1}$ flux density of photons with different energies: a- 1.77 eV; b- 1.55 eV; c- 1.38 eV; d- 1.18 eV; e- 0.70 eV. Curve f, corresponds also to 0.70 eV photon energy, but for 2×10^{14} $cm^{-2}s^{-1}$ flux density.

Fig.4 Time evolution of photoconductivity and persistent photocurrents for different times of illumination, t_i, at 85 K. Photon flux density was 2×10^{14} $cm^{-2}s^{-1}$, and photon energy 0.7 eV.
a- t_i=45s; b- t_i=100s; c- t_i=140s; d- t_i= 215s; e- t_i=300s; f- t_i= 700s; g- t_i= 1000s;
h- t_i= 1220s; i- t_i= 1900s.

off and decay of current was monitored up to several hours. The sample was then heated up to 330K with constant heating rate; during that sequence the TSC spectra were taken. The whole cycle was repeated again and again with different illumination times. An example of I_{PP} measurements performed in that way is shown on Fig.4. Decay of the current after the light was turned off regularly had two components - very fast initial decay, and than slower and slower further decrease, so that after several hours current reached almost constant value. It is obvious from Fig.4 that these persistent currents are always larger for higher photosensitivity stages.

An example of TSC measurements is shown in Fig, 5 for photon energy 1.38 eV; analogous curves were obtained for other photon energies. TSC spectra were taken after different illumination times in order to monitor trap filling rate (curves a to e). At least six different traps can be distinguish in Fig.5, denoted by T_1, T_2,..T_6. All of these 6 traps were found in all samples; relative amplitude of different peaks as well as total trap concentration varied only slightly from sample to sample (including those from different wafers). It is obvious from Fig.5 that traps filling rates, i.e. their free carrier capture cross sections, are quite different; for example trap T5 needs considerably shorter illumination time then T1 or T6 to be completely filled. Traps T_1, T_3, T_4, T_5 and T_6 correspond to those found by Lin et al.[15], denoted in their paper as a, d, e, f and g, respectively. By elaborate reasoning Lin et al.[15] deduced that all these traps should be electron traps. The total concentration of charge trapped in all traps, N_T, can be evaluated calculating the area under TSC curves, corrected for dark current. Maximal N_T, after longer illumination, was around 8×10^{14} cm^{-3}; most of this charge was trapped in T_1 - T_3 traps.

The time evolution of N_T (showing the filling rate of all traps) is presented in Fig.6 for several photon energies (circles). For each photon energy the absorption coefficient is different, therefore light intensities were adjusted to give comparable filling rate. (photon flux densities were selected to be 1×10^{13}, 3×10^{12} and $2\times10^{14} cm^{-2}s^{-1}$, for photon energies E_{ph}=1.55eV, 1.38eV and 0.7eV, respectively). These time dependences of N_T are compared with time evolution of I_{pc} (dashed lines); and with I_{PP}, (crosses); Fig. 6, (a),

(b) and (c). Similarities are obvious for all photon energies. Some differences can be observed only during the first minutes of illumination, but for longer illumination times both correlations are excellent. The time needed to reach steady-state I_{pc}, I_{PP} and N_T in different runs as well as their time evolutions were dependent on both photon flux and the energy of photons. However, in all cases the correlations between time evolutions of I_{pc}, I_{PP} and N_T were very good.

TSC, in principle, cannot distinguish electron traps from hole traps. However, if we adopt Lin's assignments(15), T_1 traps (in which most of N_T is trapped) are electron traps, as well as most of other observed traps. Under this assumption increases in photosensitivity and in persistent current during

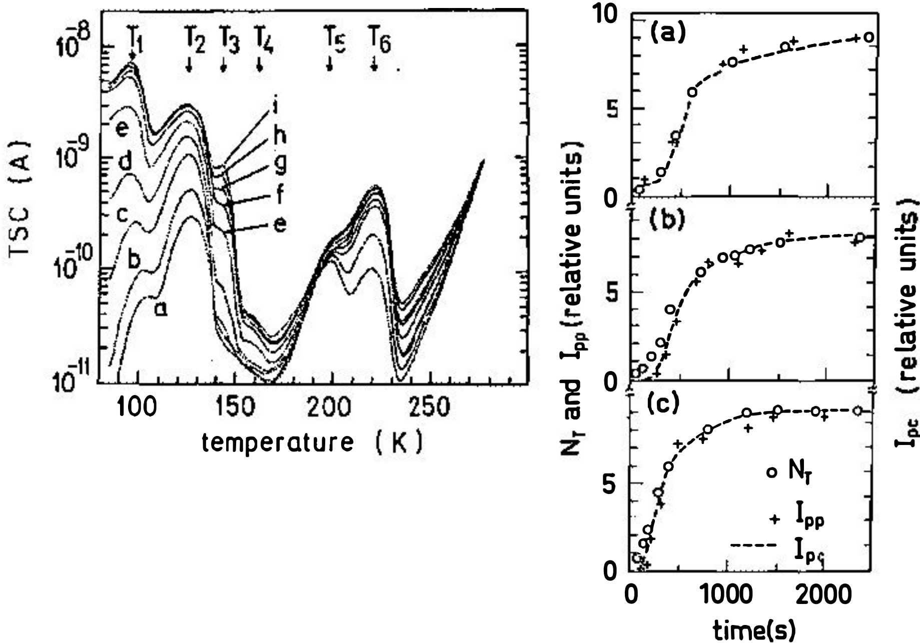

Fig.5 TSC spectra of sample illuminated with 3×10^{12} $cm^{-2}s^{-1}$ flux density of 1.38 eV photons at 85 K for different times of illumination:
a- 50 s; b- 100 s; c- 200 s; d- 300 s; e- 400 s; f-700 s; g- 1050 s; h- 1200 s; i- 1500 s

Fig.6 Correlations between time evolutions of the concentration of charge trapped in deep traps, N_T (open circles), persistent photocurrents, I_{PP} (crosses) and photosensitivity (broken line) for different photon energies, E_{ph}: (a)- 1.55 eV; (b)- 1.38 eV; (c)- 0.70 eV

the illumination can be tentatively explained in the following way:

a) above-the-gap illumination

When the light is turned on, some starting photoconductivity is established practically momentarily (in just a few ns, as typical free carrier lifetime in SI GaAs is in the 10^{-9}-10^{-8}s range(36)), reflecting an equilibrium between photogeneration of electrons and holes and their recombination. As long as the total concentration of trapped charge (in T_1-T_6) is small the concentration of free electrons in conduction band is practically equal to the concentration of holes in valence band and the photoconductivity also remains constant. However, as more and more electrons become trapped, the imbalance between concentrations of free electrons and free holes becomes larger and larger. Therefore more and more holes in valence band cannot be recombined and their lifetime increases, leading to the gradual increase in photoconductivity and gradual increase of persistent current.. When all traps are filled the photoconductivity and I_{PP} reach their steady-state values, which are considerably higher than the starting values, which correspond to the sample with empty traps.

b) Below-the-gap illumination

We believe that for these photon energies the same mechanism leading to the gradual increase of photosensitivity and I_{PP} is operational, but the dynamics of the process is much more complicated. At first mainly free electrons are created by EL2 photoionization process, but most of them get caught in traps, what may explain why the I_{pc} and I_{PP} increases lag behind the trap filling at the beginning of the illumination (Fig. 6 b and 6 c). Later, both electrons and holes are photogenerated through the two step process via EL2 centers(22,27). Finally crystal ends up again with all traps filled. As most of trapped charge are again electrons, some excess of free holes remain in the valence band which do not have free electrons available for recombination.

It is important to note that the previous reasoning would be equally valid if hole traps were dominant traps. In that case the filling of traps would lead to an excess of electrons in the conduction band which would cause an increase of their lifetime and again a consequent increases of photosensitivity and I_{PP}. Due to the charge neutrality requirement, total concentration of

free and trapped holes has to be equal to the sum of free and trapped electrons. If there is a significant imbalance between the concentrations of trapped electrons and trapped holes, the imbalance of concentrations of free electrons and holes is inevitable. The presence of traps would not affect photosensitivity only if the concentrations of trapped holes and electrons are similar, or if the total concentration of traps is small in comparison with the concentration of photo-generated free carriers in the material.

We believe that the same explanation can be successfully applied to the other published results on persistent photocurrents and optically enhanced photosensitivity. It seems that the photo-memory state or "on-state", as authors named it[23], could be interpreted as a state of SI GaAs crystal when traps are full, while the "off-state" would correspond to empty traps. In that case the assumption of low temperature photo-induced complex defect creation and annihilation may not be necessary.

Similar analysis and comparison with previous explanations for existence of I_{PP}[23,30,31] can be made. As we obtained the effect for both above-the-gap and below-the-gap photons, and later are absorbed throughout the bulk of the crystal, any type of surface connected, macroscopic potential barrier[30] does not seem plausible - at least not in bulk SI GaAs. Similarly, in our model I_{PP} depends on the existence of large enough concentration of electron or hole traps in the material and their charge states (i.e. whether those traps are full or not), so an assumption of photo-induced complex defect creation[15,23] is not necessary. In our model the prolongation of lifetime of one type of free carriers is caused by the fact that the other type of carriers, being trapped, is not available for recombination. Traps, by definition, communicate with one band only, and their probability to interact with the opposite band (i.e. to act as recombination center) is very low. That is especially true for traps which are far from mid-gap, which is the case with T_1-T_4 traps, in which most of trapped charge is stored. So in the present model there is no inherent need for any additional potential barrier[32] either, in order to explain very long recombination time constants. However, a detailed quantitative computation is needed to be able to evaluate whether observed

persistent currents could be completely explained in this way, or some sort of microscopic potential barrier is still needed.

In conclusion of this section, we have found a large increase of photosensitivity and persistent currents in SI GaAs during the illumination with low -intensity monochromatic light in the 0.7-1.8 eV range. For different photosensitivity stages the concentration of charge trapped in deep traps was determined by measuring thermally stimulated currents. Very good correlation between both photosensitivity and persistent current, and total concentration of trapped charge was found for most stages of photosensitivity and all photon energies. Assuming that most of trapped charge are electrons the increases of both photosensitivity and persistent current are interpreted as the increase of lifetime of optically created free holes in valence band. The process of hole recombination is slowed down due to the trapping of the electrons, which therefore are not available for recombination.

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