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Transformation of native defects in bulk GaAs under ultrasonic vibration

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Abstract. The effect of high-intensity ultrasonic vibration on the spectrum of deep electron traps in bulk n-type GaAs has been studied by means of deep-level transient spectroscopy. The ultrasonic treatment results in a drastic reduction of the EL6 trap concentration and a generation of three other traps, suggesting an ultrasound-driven transformation of defects associated with the traps. It is argued that the traps EL6, EL5 and EL18 are associated with the following native defects: As_{Ga}–V_{As}, V_{Ga}–V_{As} and V_{Ga} respectively. The defect transformations are described by two reactions involving emission of arsenic interstitials.

1. Introduction

Bulk gallium arsenide usually contains a high number of deep-level centres in the concentration range exceeding 10^{15} cm^{-3}. More than a dozen various deep levels, commonly observed in as-grown GaAs crystals, were characterized and catalogued some 15 years ago [1]. Over the years the traps have been intensively studied, showing that most of them are associated with native lattice defects, although their atomic structure has not yet been unambiguously resolved. The defects in melt-grown crystals have their origin in small deviations from the stoichiometry at a temperature of crystal growth. Both liquid-encapsulated Czochralski (LEC) and horizontal Bridgman (HB) GaAs belong to melt-grown crystals which are normally grown under arsenic-rich conditions. Results of precise measurements of the lattice parameter in LEC GaAs indicated that the main defect responsible for non-stoichiometry in As-rich crystals is the As interstitial [2]. Large concentrations of isolated Ga vacancies are unlikely to exist in as-grown crystals because they are easily annihilated by highly mobile As interstitials, thus forming As antisites. However, positron annihilation studies have shown that vacancy-related defects are also present in as-grown crystals of both HB and LEC, probably in the form of complexes with other defects or impurities [3]. It has also been found that radiation produces more of such defects [3].

It is a feature of II–VI and III–V semiconductor compounds that ultrasonic vibration, even at low temperature, can induce some defect reactions in these materials. The ultrasonic treatment resulted in the transformation and, in some cases, the elimination of existing point defects and their complexes in CdS and other II–VI semiconductors; cf [4] and references therein. The transformation of some structure defects or reduction in their concentration was also proposed to account for the observed improvement, as a result of ultrasonic treatment, of electrical and optical parameters of several types of electronic devices based on GaAs [5].

In an effort to obtain some new information on the structure of deep-level centres in bulk n-type GaAs we have studied the effect of high-intensity ultrasound vibration on the spectrum of deep electron traps revealed using deep-level transient spectroscopy (DLTS). The first results of our study were presented at the Jaszowiec '93 Meeting [6], and a thorough report is given here.

2. Experiment

The investigated samples were prepared from an n-type HB-grown single crystal of commercial origin with an electron concentration of 3.6×10^{16} cm^{-3}. Three (001)-oriented samples, labelled A, B and C, were cut in the form of round slabs of thickness 500 μm and diameter 10 mm. Sample A was taken as the reference sample, and samples B and C were subjected to ultrasonic vibration in an ultrasonic resonator combined with a high-pressure vessel. Special efforts were made to avoid the possible damage of the sample surfaces; the samples were separated from an adjacent waveguide (beryllium copper) at their faces with a thin (120 μm) Teflon foil and from a pressure medium (ethyl alcohol) at their sides with a thin rubber layer. A vibration of frequency 17.5 kHz was applied for 600 s at a temperature of 0 °C, which was stablized by immersing the vessel in iced water. Since GaAs is a brittle material at low temperature the ultrasonic experiment was performed under a hydrostatic pressure of 1 GPa in order to avoid the possible fracture of the samples. Two amplitudes of vibration giving rise to normal stresses in the samples of 100 MPa (sample B) and 200 MPa (sample C) were applied. They were measured with a piezoelectric sensor placed in the close vicinity of the sample centre.

After the ultrasonic treatment, several gold discs of diameter 1 mm were evaporated onto each sample to form Schottky diodes. The diodes were characterized by current–voltage and capacitance–voltage measurements, revealing no effect of the ultrasonic treatment on the barrier height of the diodes and their reverse current. DLTS spectra of all the diodes were recorded in the temperature range from 80 to 400 K using a DLTS system operating at 1 MHz with a rate window realized by lock-in detection. The trap concentrations were calculated taking into account the correction due to the effect of the non-ionized region at the edge of the depletion layer.

3. Results

The electron concentration in samples B and C was only slightly reduced, by an amount not exceeding 10% of the initial value, by the ultrasound, as obtained from the capacitance versus voltage measurements of the diodes. On the other hand, drastic changes in the DLTS spectra of the diodes were revealed as a result of the ultrasonic treatment.

Three deep electron traps, labelled EL2, EL3 and EL6 according to [1], were revealed in the DLTS spectra of the reference (as-grown) sample, as shown in figure 1 (diode no 1). In the samples subjected to vibration the concentration of the EL6 traps was strongly reduced and two new traps, EL5 and EL18, appeared, and the concentration of the EL3 traps increased. DLTS parameters of the traps, i.e. their electron emission activation energies and electron capture cross sections, obtained from the Arrhenius plot of the thermal emission rate for each trap, are listed in table 1.

The trap concentrations measured in various diodes of samples B and C were dependent on the diode position on the sample area, whereas the traps were homogeneously distributed over the area of the reference sample. The DLTS spectra of four diodes of samples B and C, numbered from 2 to 5, are presented in figure 1. Diodes 3 and 5 are placed in the centres of samples B and C respectively, and diodes 2 and 4 are representative of half-way between the centre and the periphery of samples B and C respectively. The inhomogeneity of the trap concentrations over samples B and C is thought to be the result of an inhomogeneous axially symmetrical distribution of ultrasonic stress in the resonator, being highest at the centre of a sample and lowest at its periphery. The measured amplitudes of the vibration-induced normal stresses in the samples of 100 and 200 MPa correspond to diodes 3 and 5 respectively.

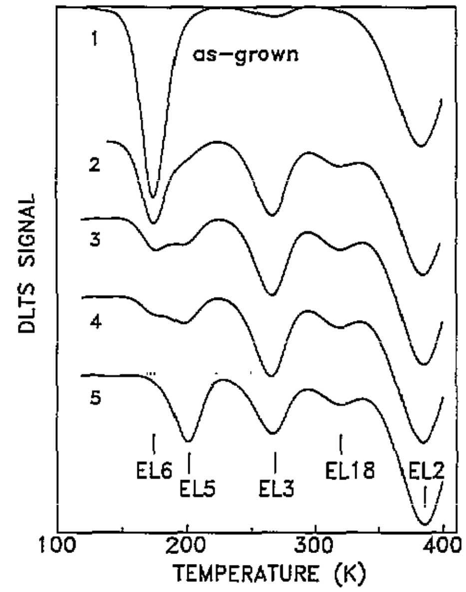

Figure 1. DLTS spectra of the reference sample A (diode 1) and the samples B (diodes 2 and 3) and C (diodes 4 and 5) subjected to ultrasonic treatment, recorded at the rate window 87 s^{-1}.

In figure 2 the concentrations of deep traps are presented as a function of the diode number, where diode 1 represents the reference sample and the numbers from 2 to 5 correspond to the diodes subjected to an increasing amplitude of ultrasonic vibration. The trap concentrations measured in various diodes are also listed in table 1. Only the EL2 concentration was unaffected by the ultrasonic treatment. The EL6 concentration decreased monotonically with increasing applied ultrasonic stress, and the concentrations of three other traps, EL5, EL3 and EL18, increased. At the maximum stress used, 200 MPa (diode 5), the EL6

Table 1. Electron emission activation energies and electron capture cross sections of the traps, and trap concentrations in various diodes.

Trap name	Activation energy[a] $E_c - E_t$ (eV)	Cross section σ_∞ (10^{-13} cm^2)	Trap concentration in diodes (10^{14} cm^{-3}) 1	2	3	4	5
EL6	0.35	2	32	11.5	4.7	3.2	—[b]
EL5	0.42	1	—	2.0	3.8	4.0	9.2
EL3	0.58	1	1.6	13	13.5	14	11
EL18	0.68	2	—	4.8	6.1	6.3	6.0
EL2	0.8	1	35	35	35	34	36

[a] Activation energy is T^2 corrected but not corrected for the activation energy of the capture cross section.
[b] Below the detection limit.

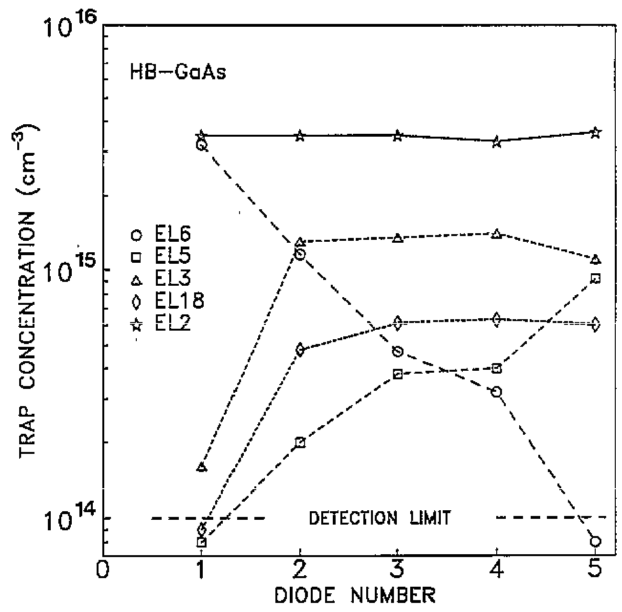

Figure 2. Concentrations of deep traps measured with DLTS in the reference diode (number 1) and the diodes subjected to increasing amplitude of the ultrasonic vibration (numbers 2 to 5). Diodes 3 and 5 correspond to ultrasonic vibration amplitudes of 100 and 200 MPa respectively.

concentration was reduced below the detection limit and the concentrations of the other traps were increased so that the total trap concentration remained almost unchanged. The results indicate a transformation of the EL6 traps into EL5, EL3 and EL18 traps driven by the ultrasonic vibration.

4. Discussion

Taking into account the present findings together with the experimental data reported so far in the literature, we propose the microscopic structure of the defects associated with the observed traps. Additionally, we shall take into consideration the energy levels of the simple point defects and their complexes in GaAs calculated theoretically by Baraff and Schlüter [7] using the self-consistent Green function technique.

4.1. The EL2 trap

The EL2 trap, being the most studied native trap in GaAs, is still a matter of controversy (for a recent review see [8]). However, there is now a growing conviction that an isolated arsenic antisite, As_{Ga} (As atom on a Ga site), is responsible for this trap, as was proposed over ten years ago [9]. Also, the latest results of some of the present authors [10] on the electric field enhancement of the thermal emission rate of holes from the rarely investigated doubly ionized charge state of the EL2 centre are consistent with this identification. Recently, Feenstra *et al* [11] studied the EL2 centres in low-temperature-grown molecular beam epitaxial (LT-MBE) GaAs by means of scanning tunnelling microscopy (STM) combined with tunnelling spectroscopy. They concluded that the centre had the structure of an isolated As_{Ga} in a tetrahedral environment. The results of recent theoretical calculations, examining the electronic structure of several As_{Ga}–interstitial complexes [12] and the pressure dependence of deep levels of the As antisite [13], are also in favour of this identification.

4.2. The EL6 trap

EL6 is a very common electron trap existing in both LEC and HB materials and sometimes its concentration is higher than that of EL2 [14, 15]. The EL6 trap, like EL2, was shown to be related to excess arsenic in GaAs [14, 16]. Also, a similarity between the optical properties of EL6 and EL2 was pointed out by Chantre *et al* [17] from their deep-level optical spectroscopy (DLOS) studies. The generation of EL6 was reported to occur in GaAs crystals subjected to ion implantation [18], neutron irradiation [19, 20] and plastic deformation [21].

Martin *et al* [19] observed the disappearance of the EL6 traps in fast-neutron-irradiated samples of both LEC and HB GaAs under annealing at temperatures between 450 and 500 °C. They described the process as a recombination of a close pair complex, possibly corresponding to a change in the Ga sublattice since defects due to the As sublattice anneal at lower temperatures (about 220 °C). On the other hand, Fang *et al* [15] observed, in HB-grown n-type GaAs, a reduction of the EL6 concentration by an amount of 6×10^{15} cm^{-3} and an increase in the EL2 concentration by about the same amount under heat treatment at 800 °C for 1 h. The authors suggested that EL6 defects were transformed into EL2 ones. Later on, the same group [22] observed, as a result of short (25 s) annealing at 900 °C of HB GaAs, a transformation of the EL6 traps into the traps termed by the authors ECX with a DLTS signature similar to that of EL5.

Results by Dobaczewski [23] on the electric field dependence of electron thermal emission from the EL6 trap (termed E3 by the author) could be described by the classical Poole–Frenkel effect with an isotropic Coulombic barrier. Our own measurements [24] of this dependence, carried out for the EL6 traps in the as-grown crystal employed in the present study, have shown very similar results to those obtained by Dobaczewski. They point out a single donor level (0/+) associated with the trap. On the other hand, the electron capture cross section of the EL6 trap was found to be thermally activated with a large activation energy of about 0.2 eV [25, 26]. This implies that the energy level associated with the trap lies at about $E_c - 0.15$ eV (where E_c is the conduction band edge) and is the same as the dominant donor level revealed very often by the temperature-dependent Hall effect in n-type bulk GaAs crystals [26, 27]. A similar energy level ($E_c - 0.14$ eV) was found for the $0 \rightarrow +$ transition of a complex involving an As vacancy from recent temperature-dependent positron-annihilation studies in a number of n-type HB- and LEC-grown GaAs crystals [28].

Levinson [29] investigated the site symmetry of the EL6 defect by means of both DLTS and photoionization under uniaxial stress. Although his results were consistent with a point defect having the full symmetry of the lattice, the author proposed a model of EL6 as a complex defect involving As_{Ga} and an electronically shallower centre which could account for the very large Franck–Condon shift of about 0.6 eV found for EL6 by Chantre *et al* [17].

To explain our present results we postulate that a complex As_{Ga}–V_{As} (As antisite–As vacancy) is responsible for EL6, which can arise from the nearest-neighbour hop of an As atom onto a Ga vacancy. According to the theoretical calculations [7], V_{As} is a shallow donor and the complex As_{Ga}–V_{As} gives rise to a single donor level at about $E_c - 0.2$ eV which, taking into account the theoretical uncertainty of a few tenths of an eV, accounts very well for the energy level of EL6.

Bourgoin *et al* [30] reported an electron paramagnetic resonance (EPR) observation of the As_{Ga}–V_{As} complex in its positive charge state in electron-irradiated GaAs. By measuring the population of the centre as a function of the Fermi level position in the crystal, they estimated positions of the energy levels (+/2+) and (0/+) associated with the complex to be at $E_c - 0.76$ eV and shallower than $E_c - 0.35$ eV respectively. They tentatively ascribed the deeper level to the E4 trap observed with DLTS in electron-irradiated n-type crystals. The shallower level can thus account for EL6. Very recently, Fang and Look [31] suggested the identification of the As_{Ga}–V_{As} complex with the electron trap T_6^* at $E_c - 0.14$ eV revealed, by means of thermally stimulated current (TSC) spectroscopy, in annealed LT-MBE GaAs. The trap is likely to be the same as EL6 revealed by DLTS in melt-grown GaAs.

4.3. The EL3 trap

The EL3 trap has also been commonly observed in melt-grown GaAs and is often considered to be associated with a native defect. However, recent infrared absorption measurements of localized vibration modes (LVM), carried out by two different groups [32, 33], allowed for identification of the trap to be associated with an oxygen-related defect. Its microscopic structure has been determined as an off-centre substitutional oxygen on arsenic site, O_{As}, exhibiting a negative Hubbard correlation energy (negative U); for recent review see [34]. As a consequence of the negative-U property, the O_{As} centre at thermal equilibrium can be occupied either with two electrons, giving rise to the occupied EL3 level and a negative charge state of the defect, or with zero electrons in its positive charge state. In a DLTS experiment, during the filling pulse, each of the O_{As} centres is filled with two electrons and during the emission period both electrons are emitted to the conduction band. As a result, the measured DLTS peak height should correspond to twice the concentration of the centres.

4.4. The EL5 trap

EL5 also belongs to the deep traps frequently observed in as-grown bulk GaAs crystals grown by both LEC and HB methods [14–16, 35], and sometimes was found to be the dominant recombination level in the crystal [35]. The trap was also reported to be created by ion implantation [18]. Look *et al* [36] carried out systematic studies of the EL5 centre in oxygen-doped LEC GaAs by a combination of temperature-dependent Hall effect measurements, spark-source mass spectroscopy (SSMS) and secondary-ion mass spectroscopy (SIMS). The conclusion was that the centre is an intrinsic defect not associated with either oxygen or any other impurity. Kuzuhara and Nozaki [37] observed generation of the EL5 traps during long-term annealing of SiO_2-capped GaAs crystals, i.e. under conditions of Ga out-diffusion. They ascribed EL5 to a defect complex associated with Ga vacancy.

Marrakchi *et al* [16], investigating the stoichiometry dependence of deep levels in LEC-grown n-GaAs, found a decrease of the EL5 concentration in the crystal with the increase of the Ga/As ratio in the melt, but the decrease was slower than in the case of EL6. The authors proposed a divacancy complex, V_{Ga}–V_{As}, as a possible origin of EL5. On the other hand, Taniguchi and Ikoma [14] reported only negligible seed-to-tail variation in the EL5 concentration in LEC GaAs, whereas at the same time the EL6 concentration decreased by a factor of 3 to 5; the seed-to-tail variation in defect concentration is considered to be caused by a small stoichiometry change during LEC growth due to a slight loss of arsenic [38].

We postulate, in line with [16], that the defect responsible for EL5 is a divacancy complex, V_{Ga}–V_{As}. The theoretical calculations [7] for this complex predict four energy levels in the bandgap, with a separation of roughly 0.2 eV between them. These levels cover the energy range that includes the EL5 level.

4.5. The EL18 trap

In contrast to the traps described above, EL18 has been rather rarely observed in as-grown bulk GaAs. The label EL18 was used for the first time by Mircea and Bois [39] for the deep electron trap found in as-grown vapour phase epitaxy (VPE) GaAs layers grown under As-rich conditions. Most probably the same trap had been reported earlier by Wada *et al* [40] (termed T_2 by the authors) to be determined in various as-grown n-type VPE layers. Further, the same trap was found to be created in n-type bulk crystals of both LEC and HB by ion implantation and to anneal out around 500 °C [18].

We suggest that EL18 is associated with an isolated Ga vacancy, V_{Ga}. The existence of a high concentration of isolated vacancies in as-grown crystals is rather improbable, but they can be generated by radiation at low temperatures. Goltzene *et al* [41] ascribed a Ga vacancy in its doubly negative charge state to a paramagnetic centre responsible for an EPR singlet line appearing in the spectra of semi-insulating GaAs as a result of fast neutron irradiation. Later on, examining thermal recovery of the EPR spectra of neutron-irradiated material, the same group [42] found that the centre completely anneals out at 450 °C. The energy level position near the midgap was predicted theoretically [7] for the (3−/2−) level of V_{Ga} in accordance with the interpretation of those EPR results as well as with the thermal activation energy obtained for the EL18 trap from DLTS measurements.

4.6. Defect reactions

The transformation of the EL6 traps into three other traps occurring under ultrasonic treatment can be explained by the following defect reactions:

$$As_{Ga}\text{–}V_{As} \rightarrow V_{Ga}\text{–}V_{As} + As_i \qquad (1)$$

$$As_{Ga}\text{–}V_{As} + O_i \rightarrow O_{As} + V_{Ga} + As_i. \qquad (2)$$

The EL5 traps are produced in reaction (1), and both the EL3 and EL18 traps are produced in reaction (2), where a limiting factor is the concentration of oxygen interstitials, O_i, in the crystal. Because of the small size of oxygen atoms, O_i interstitials are expected to have a high diffusion coefficient in GaAs. They are not electrically active but are commonly present in melt-grown crystals and could be identified by their LVM spectrum [34]. In the HB crystal employed in our study a possible source of oxygen contamination was a quartz boat utilized for the crystal growth. Arsenic interstitials, As_i, which are produced in both reactions, are highly mobile, even at low temperature, and can diffuse to sinks, such as the sample surface and dislocations, during the ultrasonic experiment.

Taking into account the negative-U property of the centres responsible for the EL3 trap, the concentration of this trap, determined from the DLTS spectra and presented in table 1, should be corrected by a factor of 1/2, as was pointed out in section 4.3. Thus, the concentrations of EL3 and EL18 traps produced during ultrasonic treatment would be roughly the same, in accordance with the supposition that both traps are produced in the same defect reaction. On the other hand, the total concentration of the traps, revealed with DLTS, decreases slowly with the increase of the ultrasonic stress applied (from diode 1 to 5). This, however, can be understood in the framework of the proposed model if we take into account an annihilation of some defects or a generation of defects, giving no rise to the deep traps observed with DLTS.

Finally, the possible electrical compensation of the samples as a result of the ultrasonic treatment needs a comment. The EL6 traps, which disappeared during the treatment, are deep donors and the newly produced traps, EL3 and EL18, are assumed to be deep acceptors. However, according to the theoretical calculations [7] As interstitials, produced in both defect reactions (1) and (2), are shallow donors and can supply electrons to occupy the new traps. Moreover, a small reduction of the electron concentration (by about 3×10^{15} cm^{-3} in diode 5) was, in fact, observed in the samples subjected to the ultrasonic treatment.

5. Conclusions

Our DLTS investigations of the n-type HB GaAs samples subjected to high-intensity ultrasonic vibration pointed out a transformation of the EL6 deep electron trap, commonly observed in bulk GaAs, into three other traps: EL5, EL3 and EL18. Using the obtained results and the data reported in the literature we have discussed the microscopic nature of these traps in terms of the native defect complexes formed in crystals grown under As-rich conditions and proposed the identification of the defects responsible for EL6, EL5 and EL18 as As_{Ga}–V_{As}, V_{Ga}–V_{As} and V_{Ga} respectively. Finally, we proposed two defect reactions, which can account for the observed trap transformations under ultrasonic vibration, involving emission of arsenic interstitials which are the most mobile native defects in GaAs at low temperature.

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