Defect engineering using microwave processing in SiC and GaAs

Oleg Olikh1!, Petro Lytvyn2

**Abstract.** The influence of microwave radiation (2.45 GHz, 1.5 W/cm2 , up to 80 s) on defects was studied in single crystals of n-6H—SiC, n-GaAs, and epi- GaAs. The capture cross section of the charge carrier was found to change, and defect complexes were reconstructed because of the growing number of interstitial atoms in the near-surface layer. The correlation between the changes in the defect subsystem and deformation of the near-surface layer was analyzed. Possible mechanisms of the revealed effects are also discussed.

1. Introduction

Microelectronics is currently a field of primary importance, and investigation of how semiconductors and their structural properties change under the action of various external factors has become one of the most important tasks in material science. Numerous theoretical and experimental studies have aimed to reveal the degradation mechanisms of microelectronic devices and develop new technologies for their production. The influence of certain factors, for example, radiation, has been studied quite well — see, for instance, [1, 2, 3]. At the same time, new agents begin to attract more attention, such as ultrasound loading (USL) [4, 5], or microwave treatment (MWT) [6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19]. The MWT has been widely applied owing to its capability to heat solid bodies [6, 7]. This approach is unique because of its high efficiency and capability to increase the temperature of a sample as a whole or at selected locations with extremely high heating speeds [6]. As a result, MWTs are widely used to synthesize various compounds, and semiconducting compounds including [6, 8]. However, this type of external influence also causes changes in the characteristics of semiconductor materials and device structures. For instance, it has been found that irradiation by superhigh-frequency electromagnetic

| Author to whom any correspondence should be addressed. waves causes relaxation of internal stresses and modification of near-surface regions in GaAs and InP structures [11, 10, 12, 13, 15, 18, 19], leveling of surface microrelief in SiC/SiO2 structures [9], redistribution of impurities [9, 16, 18], change in charge state in the complexes [12] and generation of defects [16]. One of the consequences of structure-admixture ordering is a decrease in the range of Schottky diode parameter spread [12, 16]. Moreover, MWT has been found to induce changes in the properties of Ti, Gd, and Er films deposited on silicon carbide [17], as well as in the reconstruction of GaAs photoluminescence spectra [13, 15, 16], and the effect depends on both the type of dopant and the crystal structure orientation of the samples. Overall, these facts allow us to consider MWT as one of the most promising ways to modify semiconductor devices.

However, it is well known that the properties of semiconductor structures are strongly influenced by their defect subsystem. Defects in SiC and GaAs have been extensively investigated [20, 21, 22, 23]. At the same time, more detailed information about the MWT influences the deep-center parameters is practically unknown. The aim of this study was to investigate the impact of MWT on the parameters of deep centers located in the near-surface region of n—6H--SiC and n--GaAs single crystals, as well as on GaAs epitaxial structures, by means of acoustoelectric transient spectroscopy.

1. Experimental details

It has been reported [11, 12, 13, 14, 15] that the impact of the MWT on semiconductor structures depends on many factors. The primary factors were the initial level of structural perfectness, conductivity, dielectric permittivity, and structural topology. To estimate the effect of the MWT on the defect parameters, we selected different samples based on the doping degree, initial level of residual mechanical stress, and structure. They were as follows.

1. Single-crystal n-6H-SiC wafers were grown using the Leli method and were doped with nitrogen. The samples were 490 pm thick plates with dimensions of 5 x 10 mm2 and carrier concentration (3 — 6) x 1018 cm-3 (further on SIC1 and SIC2); and 460 pm thick wafer of the same dimensions and with concentration of carriers (1 — 3) x 1018 cm-3 (SIC3).
2. GaAs single-crystal plates with thickness of300 pm. The plates were (100) oriented, doped with tin, and the concentration of electrons was (1.5 — 2.5) x 1018 cm-3 for sample GAS1 and (3 — 5) x 1016 cm-3 for sample GAS2. GAT denotation is used for wafer (111), which was doped by tellurium, n = (1 — 2) x 1018 cm-3.
3. Epitaxial n-n+ structures of GaAs which were 300 pm thick single crystal substrates n = 2x 1018 cm-3 covered with 6 pm thick layer with carrier concentration 3.9 x 1015 cm-3 (sample GAE1), 3.5 x 1015 cm-3 (GAE2), 5.0 x 1015 cm-3 (GAE3). The substrate and epitaxial layers were doped with Te.
4. Epitaxial n-n+-n++ structures of GaAs:Te with a buffer layer. They were made from a single-crystal (100) substrate (300 pm, n = 2x1018 cm-3) subsequently covered with a 1 pm layer with n = 8x 1016 cm-3 and a 2 pm layer with n = 7x 1015 cm-3. Two samples (GAB1 and GAB) were cut from different wafers and used in this study.

Epitaxial systems have been produced using gas-phase epitaxy. The samples used in the experiments are shown in figure 1.

The MWTs of the samples were performed in free space at room temperature in a magnetron at a frequency of 2.45 GHz and a specific power of 1.5 W/cm2. The epitaxial structures were irradiated onto the side of the epitaxial layer. The total exposure time tMWT varied in the range of 20 - 80 s for the different samples. To avoid essential heating, the maximum single irradiation exposure time was no more than five seconds.

The parameters of deep centers, such as the efficient cross section of electron capture *an* and location of the center energy level with respect to the conductivity band bottom Ec - Et were determined before and after MWT. For this purpose, we used acoustoelectric transient spectroscopy [24, 25, 26, 27]. A schematic representation of this method is shown in figure 2. The samples were placed on a LiNbO3 piezoelectric plate in which acoustic waves were excited as impulses. After ultrasound impulse termination, relaxation of the transverse acoustoelectric voltage (TAV) occurs according to

Vtav (t) = Vtav,o exp(-t/T). (1)

A simple exponential dependence according to equation (1) is observed in cases where only one type of deep center is effective in acoustoelectric interactions. For n-type semiconductors, the characteristic relaxation time is described by the equation [24, 27]

1 / Ec - E- \

where uth,n is the electron thermal velocity and *Nc* is the density of states in the conduction band.

The experimental measurements of the TAV relaxation at different temperatures and further approximation of the results according to equation (1) allowed us to obtain the t*(T*) dependence. Ec — Et was determined from the slope of t dependence on *(kT*)-1 in semi-logarithmic scale and then, using equation (2), *an* was calculated. The measurements were performed in the temperature range (290 - 350) K except for the GAB samples, the TAV for which was high enough to be measured only after heating to above 310 K.

For single-crystal samples before and after MWT, we also determined the curvature radius Rcur and deformation <^cur of the near-surface crystallographic planes. The value of <^cur was estimated by the X-ray method from the change in the angle of the diffraction maximum location during sample translation [28], and the curvature was measured using the profilometer DekTak 3030 Veeco Instruments. Rcur and <^cur were measured with a relative error no more than 2 %. For GaAs single crystals, we also analyzed the distribution of structural defects over the area using the Borman X-ray projection topography method, and estimated the distribution of dislocation densities and microstresses from the analysis of the intensities of the Friedel reflection pairs hkl and *hkl*

1. Results and discussion

Figure 3 presents the typical temperature dependencies of t for the samples before and after the MWT. The above data show the change not only in the curve slopes (which is directly related to the level location in the gap), but also in the absolute value of the characteristic time of the relaxation TAV after MWT. The character of the MWT impact (the decrease or increase in relaxation time) depends not only on the exposure time and degree of doping but also on the internal structure of the samples under study. The results are presented in table 1. In the silicon carbide samples, there are two deep levels, labeled ESC1 and ESC2, while in gallium arsenide, there are six (EGA1-EGA6).

The presented data show a number of characteristic features:

1. The value of the carrier capture cross-section is much more sensitive to the MWT than the energy location of the levels. For example, *an* was found to change by an order of magnitude when the level location displacement was no more than 20%, and the capture cross-section was modified at lower exposition times. For instance, the value of (Ec - Et ) for GAB1 practically did not change after 20 s of MW exposure, while *un* grew approximately four times.
2. In single crystals, the MWT-induced changes become stronger as the free charge carrier concentration decreases (see data on samples GAS1 and GAS2) and the relative deformation increases (the increase in surface curvature).
3. After durable MWT of single-crystal samples (tMWT > 40 s for GaAs, tMWT > 80 s for SiC), the TAV signal essentially decreased. This fact correlates with the data from [16], who reported a decrease in the concentration of centers with levels in the upper half of the band gap as a result of MW annealing.
4. The irradiation dose required to essentially change the parameters of the centers in the epitaxial structures is higher than that required for single-crystal samples. In particular, table 1 provides data for the GA and GAB series samples after 20 s of MWT which supports this fact. It should be noted that the doping levels of the GAB and GAE substrates were the same as those of samples GAS1 and GAT, and the doping level of the GAB epitaxial layer was similar to that of GAS2. In addition, GAB, GAE, and GAT contain the same doping impurities. Thus, the observed differences were determined by the structure of the samples but not by the difference in their conductivities.
5. The characteristics of changes in single-crystal wafers and epitaxial structures are opposite: for SIC, GAS, GAT, *un* and (Ec — Et) were found to decrease after MWT, while for GAE and GAB, both parameters increased.

We now consider the possible configurations of the centers discovered in the structures under study. For this purpose, we should consider that the reported data for the main trap parameters vary over a wide range; in particular, the difference between the values of capture cross sections can be as large as four orders of magnitude [29]. One of the possible reasons for such a large spread is the essential dependence of the thermal charge emission speed on the electric field strength [30, 31] caused by a) decrease in ionization energy due to the Pool-Frenkel effect or, for example, Coulombic interactions of centers [32]; b) change in the *un* value [33]. As a rule, the change in (Ec — Et) comprises several tens of meV, and the change in the capture cross-section reaches several orders of magnitude. For instance, according to Bourgoin and Angelis [33], at room temperature, *un* for the *EL2* center in GaAs decreases 200 times at an intensity of 105 V/cm. Consequently, the different methods used for investigating defects yield different parameters for the same centers. As an example, we can compare, from reviews on various traps in gallium arsenide, the data obtained by deep-level transient spectroscopy [34] and thermally stimulated currents [29]. Data were obtained for defects with closely located levels and very different capture cross­section values. Generalizing the above-mentioned, we should note that it is the energy location of traps that we shall be oriented toward in our research.

The position of the ESC1 level (Ec — (0.33 — 0.34) eV) observed in the initial crystals of silicon carbide can be compared with the position of the S-center (Ec — 0.35 eV) [35, 36, 37], EK-center (Ec — 0.34 eV) [38], or (—/+) level center E1 (Ec — 0.34 eV) [35]. S-center is responsible for non-radiative recombination, and in 6H-SiC it is a self-interstitial defect [35]). According to the results reported in [36, 37], the S-center and R-center (Ec — 1.27 eV) are associated with two different charge states of one and the same defect, while according to Lebedev et al . [39], the R-center is a divacancy VSiVC . It should be noted that a divacancy is a typical defect in 6H-SiC [40, 20]. On the other hand, the level Ec — 0.39 eV is more often associated with center E1 [21, 41]. Thus, in our opinion, ESC1 level is related to complex VSiVC .

After the MWT, the position of the level responsible for TAV generation in SiC moved to Ec — (0.26 — 0.29) eV (level ESC2). And this situation is also ambiguous: closely located are donor level (0/+) of center E1 (Ec — (0.27 — 0.28) eV [42], Ec — 0.26 eV [21, 41]) and center X1 (Ec — 0.3 eV [43]). The authors of the latter publication reported the essential dependence of X1 concentration on crystal structural perfection. They stressed that this center was not identical to that of center E1. In turn, level E1 has been identified as the center of the negative correlation energy [43, 21] and dominating intrinsic point defect in n-type 6H-SiC [44]. According to Refs. [44, 21], E1 is related to carbon vacancy. Considering the difference between the X1 and ESC2 energy locations, the configuration VC was associated with the ESC2 level.

The data for every level revealed for gallium arsenide are listed in table 2. The presented data show that the centers are associated with intrinsic vacancy-related defects.

Several factors caused the trap parameters to change. They are as follows.

1. Transformation of the defect complex due to decay, involvement of additional components, change in the distance between defect components, etc.
2. Defect recharging.
3. Changes in the trap environment, which can result, for instance, in the modified strength of the electric field around the defect.
4. An increase in the concentration of a given type of defect; for instance, the change in ionization energy was reported [32] to be proportional to the cubic root of the defect concentration.

Analysis of the observed phenomena should consider the probable mechanisms of the impact of microwave radiation on the crystals. First, the effect of the temperature increase should be analyzed. It is believed that the structural modification in the MWT result is mostly caused by the change in the defect charge state and elastic stress fields arising in instantly heated defect regions. However, these processes are known to intensify with increasing free charge carrier concentrations [6], whereas in our case, the effects weaken with the concentration increase (samples GAS1 and GAS2). Moreover, the applied irradiation mode did not imply long-term continuous exposure to MW oscillations, which reduced the overall heating of the structure. However, numerous studies have shown that the observed effects of MWT cannot be explained only by the mechanisms of fast annealing; therefore, non-thermal factors should also be considered. In recent research, more attention has been paid to the non-thermal mechanisms of MWT action (see, for example, [68] and the references it contains), which cause dislocation generation and result in smaller clusters of point defects in semiconductor wafers [69] or even trigger recrystallization processes [68]. Possible non-thermal processes causing changes in the structural characteristics of binary semiconductors have been reported in [69]. In particular, the processes of dislocation oscillations under the action of an electric field were analyzed, and the decorating impurities were found to influence the behavior of the dislocation segments. On the one hand, the available impurities decrease the resonance frequency of oscillations and provide the presence of electric charge, on the other hand, at high oscillation amplitudes they can escape the dislocation, which causes new chemical defects to arise. In turn, the point defects can perform superhigh-frequency oscillations and diffuse as a result of the MWT.

The observed modifications of deep-level parameters are the result of the above­mentioned structural reconstruction in semiconductor near-surface regions owing to the MWT. The results of X-ray investigations show that MWT increases the convexity of single-crystal samples, which indicates the aggregation of interstitial defects in the near-surface layer, particularly because of the generation of separate dislocations [11, 19]. The defect accumulation effect caused by the MWT in the near-surface region was reported in [11, 18]. To a certain extent, only the SIC3 sample was excluded; however in this case, a rather strong deformation of the near-surface region was also observed prior to irradiation. Researchers have reported [9, 10, 11, 12, 13] that in this stressed state, MWT causes redistribution as well as certain weakening of elastic deformations, which is what occurs in SIC3. The profilometry data correlated with X-ray measurements. Structural investigations show that in the initial GaAs samples, the density of dislocations has a W-like distribution over the plate area; the dislocation density over the plate diameter varied from 2 • 104 cm-2 to 2 • 105 cm-2. This inhomogeneity in the dislocation density distribution is evidence of considerably strong elastic deformation in the sample.

The analysis showed that the ESC1 and ESC2 centers are complexes of carbon vacancies, EGA1 is associated with VAs, and EGA3 — with VAs or VGaGaiVAs complexes. The MWT stimulates the diffusion of point defects, which are mostly intrinsic interstitial atoms, resulting in trap modifications. The ESC1 center in silicon carbide transforms into ESC2 under the influence of closely located interstitial silicon:

Vsi Vc + Si i Vc .

Further modification of the ESC2 parameters in SIC3 is caused by the enhanced electric field of the dislocation. In the GAS2 samples at tMWT = 20 s, the VAs transformed into complex VAsAsi (EGA2 center) because of the increased number of interstitial atoms in the near-surface layer:

VAs + As i VAs As i .

A similar process in GAS1 is more complicated because of its higher charge carrier concentration. It has been reported [7] that with a growth in resistance, the depth of microwave penetration increases, and thus, the volume from which defect gettering begins in the near-surface layer grows as well. In addition, the cause of the weak (in comparison with GAS2) influence of MWT on trap parameters in GAS1 is the absence of pressure stresses, which intensifies the MWT-simulated complex formation process in the system's intrinsic defects. This is supported by data for silicon carbide single crystals. In the GAT sample, which is also characterized by a high concentration of free electrons, the transformation of EGA3 to EGA4 (VGaGaAs complex) occurs in the reaction described in [60]:

VGa Ga i VAs GaGa VAs GaAs VGa

Accumulation of a large number of interstitial atoms in the near-surface layer at high doses of radiation (tMWT « 40 s for gallium arsenide and tMWT > 80 s for silicon carbide) causes complete annihilation of vacancies (or transformation into antisite defects, whose levels are filled in the crystals with electron conductivity); therefore, the TAV signal disappears (samples GAS1, GAS2, SIC1):

SiC : Vsi Vsi + Sii + Sii 0;

GaAs : Vas + As i 0 ; Vas + Ga i GaAs•

It is believed [27, 25, 24] that the TAV that arises in epitaxial structures is mostly caused by defects located at the epi-layer and substrate interface. This difference in the deep-level location is the cause of the difference between the dose-dependent modification of the defect parameters in the epitaxial and single-crystal samples.

In epitaxial structures n-n+-GaAs and n-n+-n++-GaAs, MWT induced increase of curvature radius reported in [11, 19] is the result of forming single dislocations and their further propagation deep into the structure along the glide planes. As a result, the strength of both the electric and stress fields changes, which causes defect transformation and, thus, the shift of the respective deep levels. As shown in table 2, EGA5 and EGA6 levels were associated with the complexes VGaVAs and VAsAsi, respectively. Traps such as EGA2 and EGA4 have also been previously found in epi­structures [48, 55, 34, 61, 62, 66, 67]. The observed MWT-stimulated transformations are caused by an increasing number of interstitial atoms, which are described by the following reactions:

VGa VAs + Ga i + As i VAs As i

for GAE1 and GAE2 and

VGa GaAs + As i GaGa VAs + As i VAs As i for GAB1 and GAB2. The increase in activation energy EGA6 in sample GAE3 is probably caused by the change in Coulombic interaction of interstitial-vacancy complexes, which is due to a decrease in their concentration, while the growth of capture cross-section EGA4 in GAB1 at tMWT = 20 s and EGA6 in GAE3 are associated with the growth of the electric field strength caused by charged dislocations.

1. Conclusion

The influence of microwave radiation on the parameters of point defects (cross-section of electron capture and energy levels in the gap) was studied experimentally in single crystals of n-6H -SiC and n-GaAs, as well as in gallium arsenide epitaxial structures. The investigation showed that the traps available in the near-surface layer are associated with intrinsic vacancy-related defects. The microwave radiation-induced change in the trap energy level and capture cross-section was caused by the growing number of interstitial atoms in the near-surface layer. The radiation-induced process involving the transformation of defect complexes intensifies under stress conditions.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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