

Ing. Arpád Kósa

Author's report on the dissertation thesis

**DEEP LEVEL TRANSIENT SPECTROSCOPY
STUDY OF EMISSION AND CAPTURE PROCESSES
IN MULTILAYER SEMICONDUCTOR STRUCTURES**

for the acquisition of: academic title philosophiae doctor, PhD.

in study programme: **Electronics and Photonics**
and study field: 5.2.13 Electronics

Date and place: In Bratislava, July 2016

**SLOVAK UNIVERSITY OF TECHNOLOGY
IN BRATISLAVA
FACULTY OF ELECTRICAL ENGINEERING
AND INFORMATION TECHNOLOGY**

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at the Institute of Electronics and Photonics, Slovak University of Technology
in Bratislava, Faculty of Electrical Engineering and Information Technology

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1. Introduction, current state of research

Development and production of high quality semiconductor materials are crucial steps of technical advancement. Concentrated effort is focused on the fabrication of perfect crystal structures, despite the fact that it is very hard to ensure precise growth conditions such as temperature, pressure and high component purity. During growth processes different types of impurities are formed, which can highly affect the function and electrical properties of final structures. Electrically active defects also called as deep energy levels from energetic point of view are allowed states localized in the band gap of the semiconductor material. By capturing charge carriers these are directly influencing generation and recombination processes, usually with non beneficial effects.

In order to identify inadequacies of growth technologies, the manufacture of high quality semiconductor materials has to be supported by appropriate diagnostic approaches. One of the most important methods of electrically active defect investigation is Deep Level Transient Spectroscopy (DLTS). The standard DLTS method was founded by David Vern Lang in 1974 with the objective of semiconductor material characterization [1]. During the following decades many variations of the standard method were developed like Deep Level Transient Fourier Spectroscopy - DLTFs, which processes measured data by Fourier analysis [2] - [6]. DLTS has a key role in defect characterization satisfying basic requirements of diagnosis, especially accuracy, non-destructivity and sensitivity [7], however in certain cases reliability of evaluated data is highly affected by the presence of mutually interacting complex impurities. It is difficult to effectively separate these responses therefore calculated parameters are obtained with reduced precision. Investigation of complicated multilayer semiconductor structures also induces difficulties, since different materials and several semiconductor junctions are involved. Various experimental and analytical evaluation methods are needed to be carried out and compared to establish a complex evaluation and defect investigation.

Actual research is focused on multilayer structures utilized in photovoltaic applications, since solar energy as a renewable energy source is more and more important in everyday life. It is an efficient and pollution free method of electricity generation, rapidly grown with high importance in the last decade. Attention is devoted to promising semiconductor compounds on GaAs basis, such as InGaAs, GaAsN and InGaAsN alloys. These materials, due to their unique physical properties are suitable candidates for multi junction solar cells and other semiconductor and photovoltaic applications. Dilute-nitride InGaAsN based solar cells lattice matched to GaAs were demonstrated as working prototypes since 1998 [8]. Sub cells with adequate performances for 3 junction cells were reported in 2007 [9]. World record efficiency 43.5% has been achieved by multi-junction solar cells employing GaInNAsSb junctions [10]. New approaches for InGaAsN based structures are continuously studied to realize 50% efficiency by 4- or 5- junction solar cells lattice matched to GaAs and Ge substrates [11]. Increasing requirements on quality and efficiency of solar cells, not only in space technology but also in terrestrial utilizations, needs to be supported by various experiments and characterization procedures. Successful application requires the understanding of generated electrically active defects. Investigation must be correlated with the growth process in order to develop the fabrication, since it is hard to determine proper material compositions, especially in the case of InGaAsN where defect states are associated with the indium and nitrogen content [12], [13].

One of the most difficult parts of the DLTS investigation is the interpretation of experimental results. Different factors are needed to be taken into account in order to establish a reliable evaluation procedure. On one hand we can have a comprehensive result, although on the other a non evaluable experiment. In some cases the opposite is true, deep energy level parameters are obtained with high reliability, but structural and geometrical properties are indicating a questionable investigation [14] - [16]. The accuracy of the method is mainly affected by complex situations, in which different defect states are interacting, while a more complicated situation rises, when the object of the investigation is a multilayer structure, containing several p-n or p-i-n junctions not only in homogenous but also heterogeneous compositions [17], [18]. To state the layer origin of such a DLTS spectrum is rather difficult. Without knowing the exact layer and material type where the defect is localized, the investigation loses its point. There is no 100% proven approach for the DLTS evaluation of multilayer structures, therefore application of referent structures and different evaluation and experimental methods is essential.

The dissertation thesis deals with proposition of an effective approach of high reliability and complex deep energy level parameter investigation in complicated multilayer structures by the DLTS method. Main attention is focused on InGaAsN based tandem solar cells and selected referent samples to solve challenges of analysis, such as limiting factors of evaluation: involvement of undoped semiconductor layers, different material compositions and structural properties, composition and growth parameter defect dependencies.

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2. Goals of the dissertation thesis

Quality of multilayer semiconductor structures based on A3B5 utilized in photovoltaic applications, and also investigation of their emission-capture processes dependent electrical properties are constantly at the center of basic research. Following goals of the dissertation thesis have been established:

- To analyse basics of the DLTS method and according to this knowledge to propose an effective approach of high reliability and complex deep energy levels parameters investigation in complicated multilayer structures.
- To investigate emission and capture processes in multilayer structures with several homogeneous and heterogeneous semiconductor junctions (p-n, n-p, p-i-n) by DLTFs method and its modifications. Focus of attention on defect distribution in multilayer (tandem) solar cell structures based on InGaAsN/GaAs.
- To analyse structure composition and growth process dependent defect states (different material concentrations - different band gaps, undoped semiconductor layers, defect complexes) affecting the trap parameter calculation, defect type identification and origin determination.

3. Experiments and sample preparation

The investigated InGaAsN tandem solar cell samples, including the referent structures were grown by Atmospheric Metal Organic Vapour Phase Epitaxy (AP-MOVPE) using AIX 200 R&D horizontal reactor on a n-type GaAs (Si doped) substrate at the Wroclaw University of Science and Technology. As dopant sources silane (SiH_4 in H_2) and diethylzinc (DEZn - $\text{Zn}(\text{C}_2\text{H}_5)_2$) were used for n-type and p-type dopants respectively. High purity hydrogen was employed as carrier gas (99.9999 %). Trimethylgallium (TMGa, $(\text{Ga}(\text{CH}_3)_3)$), trimethylindium (TMIn - $\text{In}(\text{CH}_3)_3$), tertiarybutylhydrazine (TBHy - $(\text{C}_4\text{H}_9)\text{HN}_2\text{H}_2$) and arsine (AsH_3 : 10% mixture in H_2) were applied as growth precursors. Fabricated structures were prepared in two metallization configurations. In the first case as a typical detector ring structure with an inner diameter $\varnothing = 150 \mu\text{m}$ (circle shaped active area), where the metallic

p-type (Pt/Ti/Pt/Au) and n-type (AuGe/Ni/Au) contacts were deposited under vacuum conditions on the top and around the mesa, respectively. The second configuration was prepared with p-type AuBe transparent and n-type AuGeNi standard metallization by deposition on the top and bottom side of the structure (Fig.). The transparency was determined as 40 % [19].

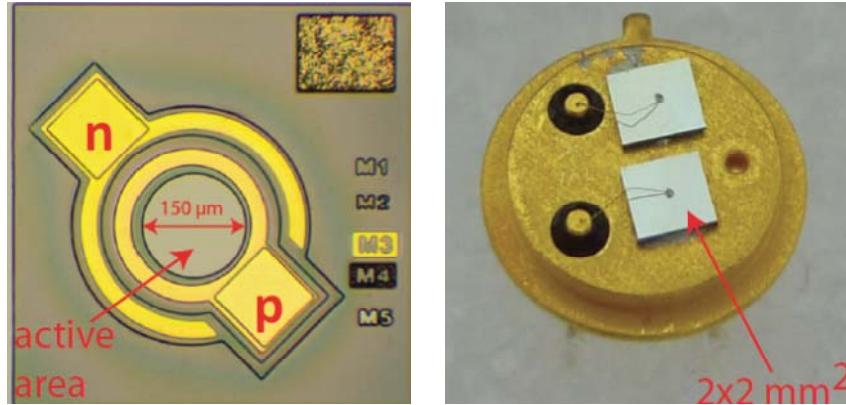


Fig. 3.1 Images of the fabricated, test solar cell structures. Typical detector configuration with circle shape active area (left) and structures with transparent top electrodes (right).

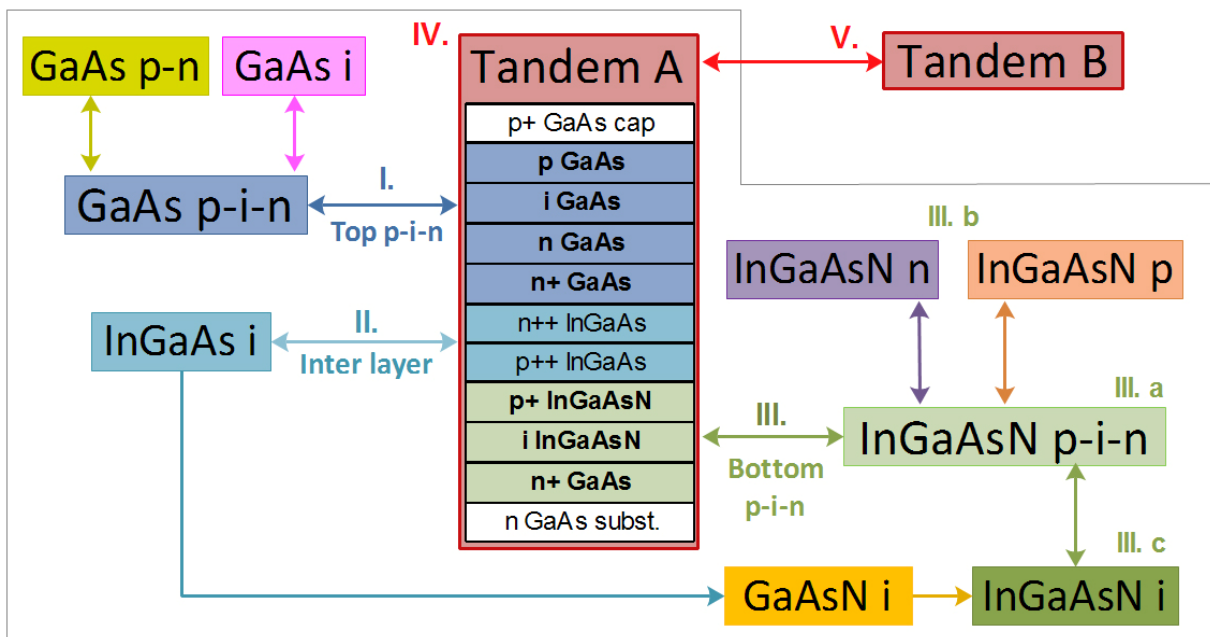


Fig. 3.2 Mind-map of DLTS analysis, plan of the tandem solar cell evaluation.

The examined tandem solar cell sample consisted of three sub-junctions: GaAs top p-i-n InGaAs n-p interlayer and InGaAsN bottom p-i-n. In order to establish a complex defect investigation, each sub solar cell junction (top p-i-n, bottom p-i-n and the InGaAs n-p interlayer) should be investigated and compared by referent structures prepared in the same aperture with the same technology. According to available referent samples and the structure scheme the first tool of complex investigations, an evaluation diagram was created, simply to define the basic tasks of analysis (Fig. 3.2). The following steps are relevant:

- I. Examination of the top GaAs p-i-n solar cell (**Chyba! Nenašiel sa žiaden zdroj odkazov.**, blue), by comparison with referent GaAs p-i-n, p-n and i structures. Attention devoted to intrinsic layer induced trap levels, reliability and defect type statement, addressing the problem of intrinsic layer involvement in DLTFs analysis.
- II. Analysis of In concentration influences on DLTFs responses in InGaAs referent samples to precisely identify In trap origins for the InGaAs tunnel junction (Fig. 3.2, turquoise).
- III. Evaluation of the bottom InGaAsN p-i-n structure (Fig. 3.2, green):
 - a. DLTFs experiment and analysis of emission and capture processes in the InGaAsN p-i-n bottom solar cell referent sample.
 - b. Evaluation of correlations between InGaAsN p and n type referent samples and the InGaAsN p-i-n structure, to state the conduction type of defects (p, n).
 - c. Comparison of InGaAsN intrinsic referent sample with the bottom p-i-n structure to locate the intrinsic layer response, overall comparison of undoped referent structures GaAs i, InGaAs i at different percentages of In, GaAsN i at different contents of N and InGaAsN i to state and distinguish between defect level origins (GaAs, In or N induced). Assessment of result in accordance with the InGaAsN p-i-n referent sample.
- IV. DLTFs analysis of the InGaAsN tandem solar cell structure Tandem A by referent sample comparison (each sub-junction I, II, III). Identification of probable defect origins and generation conditions.
- V. DLTFs evaluation of Tandem A and Tandem B multilayer InGaAsN samples with different growth conditions to identify dependent electrically active defect states. Investigation of relations between these two samples and generated defect states.
- VI. Overall InGaAsN tandem solar cell DLTFs evaluation and conclusion. Discussion about identified defect states, possible origins and conditions of elimination or reduction. Specification of a more suitable structure for a high efficient solar cell.

4. Results and discussion

Main achievement of the thesis is a complex DLTFs investigation of emission and capture processes in multilayer InGaAsN solar cells, proposing an effective way of defect recognition in complicated semiconductor structures. This adequate electrically active defect study is particularly important, since it supports the up to date DLTFs and InGaAsN solar cell research. Stated results represent essential information for future DLTFs investigations and the InGaAsN solar cell growth process optimization. Outcome of the analysis should assist the growth of InGaAsN solar cells with higher efficiencies. All the results of the present work

can be summarized according to the stated sub tasks of the InGaAsN tandem solar cell investigation (Fig. 3.2).

I.

DLTFS analysis of the top GaAs p-i-n sub-junction of the multilayer solar cell structure in terms of reliability improvement, addressing the problematic of a dual type majority/minority carrier defect complex and the intrinsic layer induced defect conduction type statement difficulty (Fig. 4.1). This type of trap state, typical for undoped structures, highly influences the reliability of standard DLTFS evaluations. A unique analytical approach of data sorting was proposed, capable to overcome standard DLTFS evaluation limitations thereby ensuring a high precision defect parameter calculation. Based on the definitions of transition areas (Fig. 4.1 right shaded areas) low classified spectral points were filtered out and defect parameters were recalculated (Fig. 4.1 right). This chapter includes a detailed discussion of many available analytical and experimental techniques and their limitations in connection with such a complex defect state.

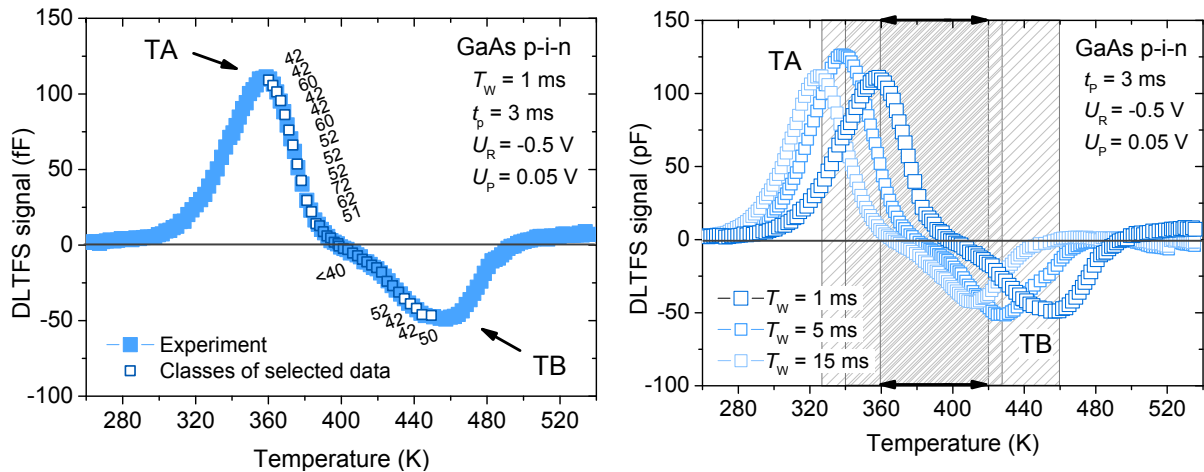


Fig. 4.1 Transient classifications of the GaAs p-i-n measured DLTFS curve (left) and defined curve transition areas (right).

The approach was confirmed by all the available experimental methods such as: DLTFS experimental parameter variations, Isothermal Transient Spectroscopy, Minority carrier DLTFS, DLTFS spectrum simulations, comparison by referent samples and literature. It should be highlighted, that the Minority carrier DLTFS an important part of the experiment capable to filter out majority carrier responses (Fig. 4.2), was the first time utilized and optimized for the DLTFS measurement system DL8000. Standard procedures of these optical experiments and sample contacting were specified assessed and are used since then. Spectral results confirmed the validity of the proposed analytical approach, and the presence of the interacting defect complex. Probable defect and intrinsic layer conduction types were stated, in addition insufficiencies of the GaAs top p-i-n solar cell were discussed in detail. The realized high precision defect investigation showed two dominative defect states of the dual type complex: a nickel related electron type defect (0.48 eV [20], [21]) and a possible hole

like oxygen associated meta-stable state of the arsenic antisite EL2 (0.63 eV [22]). Both the presence of oxygen and Ni were connected to a possible growth process source.

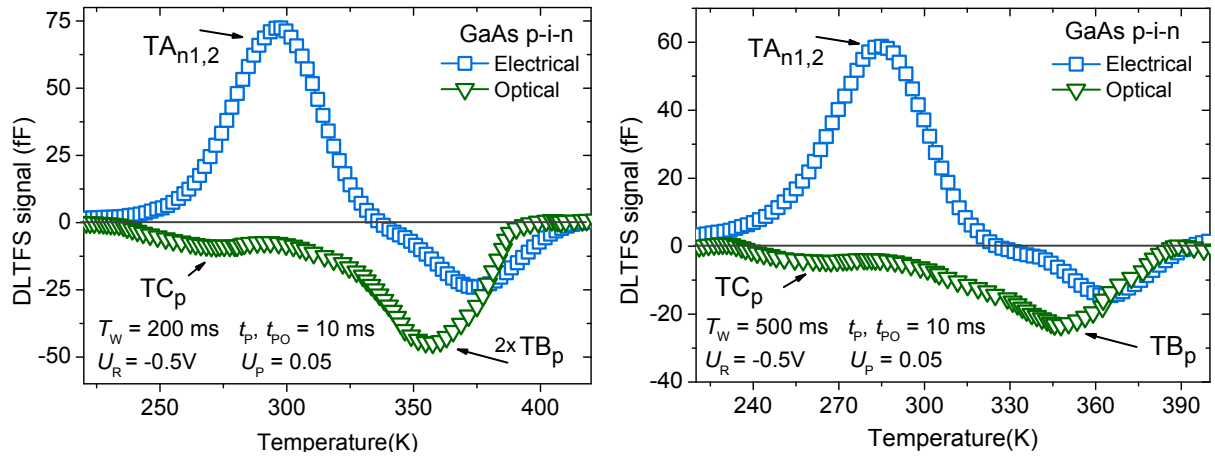


Fig. 4.2 Comparison of standard DLTFs and MCDLTFs measurements of GaAs p-i-n at two set of parameters. Optical excitation of the minority response.

II.

DLTFs analysis of InGaAs based referent structures at various In concentrations supporting the InGaAs n-p tunnel junction investigation of the multilayer InGaAsN solar cell, regarding In dependent defect states (Fig. 4.3). The most important achievement of this chapter is the definition of proper indium concentrations for InGaAs as a high purity sample. Four referent structures with In concentrations 3.4 %, 8.9 %, 10.5 % and 12.8 % were investigated addressing the gradient composition DLTFs question of activation energy calculation. Three significant affects of the In composition were concluded: shifting DLTFs spectrum peaks, hence fluctuating activation energies at different band gaps of the associated In content (Fig. 4.4), higher In concentration around 10.5 % reducing low temperature point defects (Fig. 4.3), presence of a more substantial In dependent dual state defect confirmed experimentally and by calculations (Fig. 4.4).

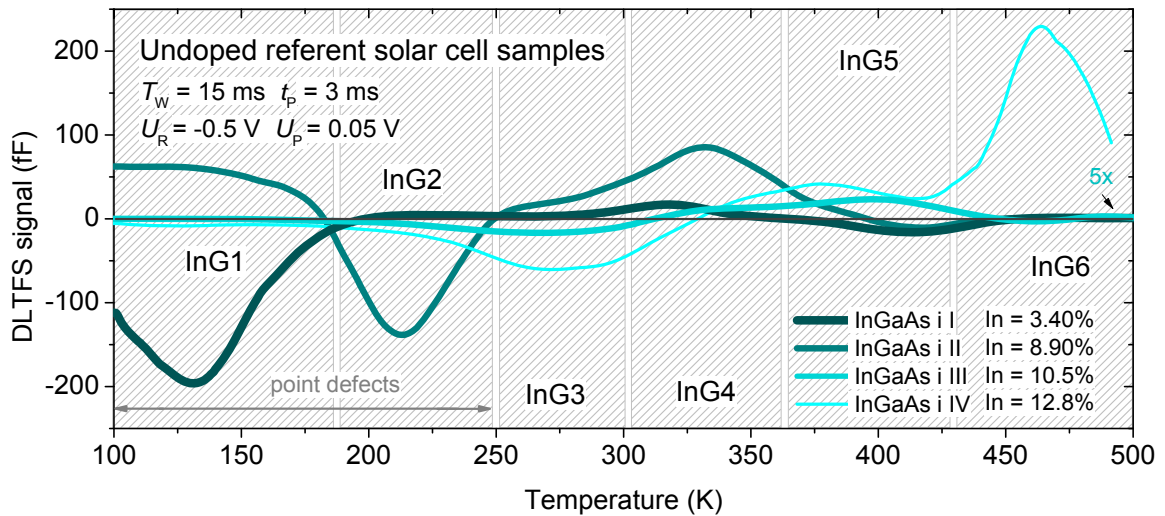


Fig. 4.3 DLTFs spectra of InGaAs undoped referent samples at different In concentrations.

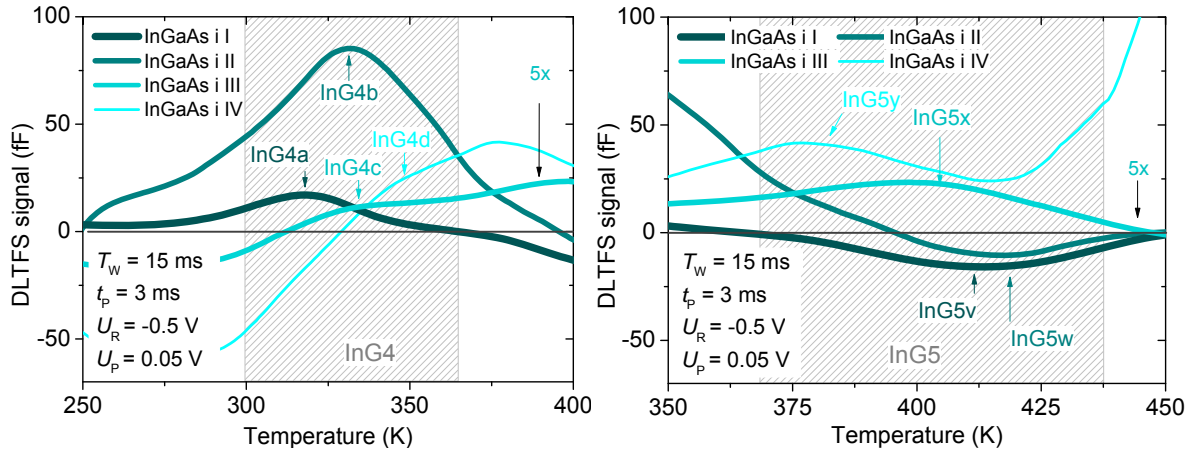


Fig. 4.4 Detailed DLTFs of trap group InG4 (left) and InG5 (right).

DLTFs experiments ensured the identification of two significant deep energy levels InG4 as a Cu(~ 0.48 eV [23]) related hole trap visible in all samples (Fig. 4.4 left) and InG5 as EL2 (~ 0.77 eV [24]) and its meta stable double donor state EL²²⁺ (~ 0.57 eV [25]). According to these results there is a high possibility that the discussed formation of the identified EL2 and its meta-stable state is connected to the In concentration, since a clear transition was experimentally supported by examined DLTFs spectra (Fig. 4.4 right). As far as the study of research information is correct and up to date, the double donor state defect transition of EL2 was not related to indium content until now. By the observed number and concentration of trap states, sample with 10.5 % of In was stated as the purest, nevertheless a hypothetical value of 9.5 % was assumed, high enough to eliminate or reduce point defects, further on to ensure an equilibrium of the dual state deep energy level.

III.

DLTFs analysis of InGaAsN based p-i-n referent sample a structural equivalent of the bottom p-i-n multilayer solar cell sub-junction. The most important achievement of this chapter is the definition of proper nitrogen concentrations for GaAsN as a high purity sample. Five referent structures with N concentrations 0.9 %, 1.15 %, 1.5 %, 1.6 % and 1.85 % were investigated addressing the gradient composition DLTFs question, and growth condition dependence of electrically active deep energy levels, since each sample was grown at different temperatures (585 °C, 605 °C, 595 °C, 565 °C, 575 °C - GaAsN i I - V). These experiments together including task II were carried out in order to support the InGaAsN p-i-n analysis, since different In and N gradient concentrations were involved. Three significant affects of the N composition and growth temperature parameter pair were concluded: shifting DLTFs spectrum peaks, meaning fluctuating activation energies at different band gaps of the associated N content (Fig. 4.6), higher N concentration around 1.0 % eliminating low temperature dislocations (Fig. 4.5), probable presence of more substantial N concentration and growth temperature dependent defect complex confirmed experimentally (Fig. 4.6).

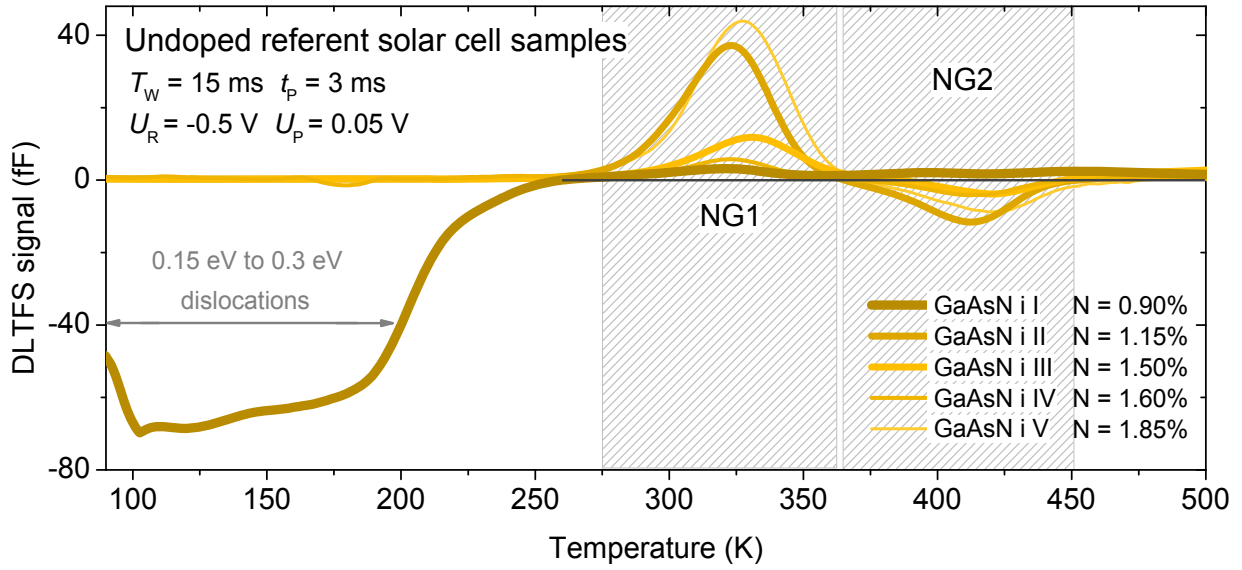


Fig. 4.5 DLTFS spectra of GaAsN undoped referent samples at different N concentrations.

DLTFS experiments ensured the identification of two significant defect complexes NG1 and NG2, possibly containing a nitrogen and GaAs related state in both cases: NG1 was identified as a complex of a hole like GaAs trap (NG1* \cong 0.46 eV [23]) together with a nitrogen induced defect (NG2** \cong 0.54 eV [26]), while NG2 as an opposite type trap complex including a possible nitrogen related electron like negative signal (NG2 \cong 0.66 eV [27]) with a GaAs assumed hole like positive one (NG2 \cong 0.71 eV [28]). Each complex was significant at specific growth conditions.

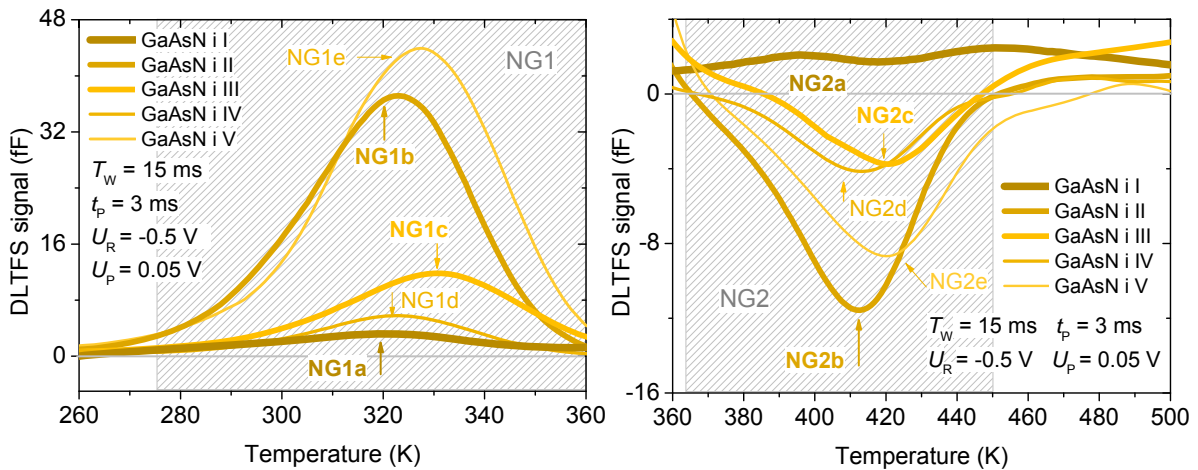


Fig. 4.6 Detailed DLTFS of trap group NG1 (left) and NG2 (right).

As concluded, NG1 defect concentration was increased by both growth factors (temperature, N content Fig. 4.6 left), while the temperature sensitive positive signal of NG2 interacting with the N content dependent negative spectrum showed dominant behaviours as follows: low N content and high temperature was favourable for GaAs related NG2 (Fig. 4.6 right NG2a) and high N content lower temperatures for the negative N related NG2 (Fig. 4.6 right NG2b). Referent sample with 1.60 % at 565 °C (GaAsN i IV) could be considered as the most balanced structure where NG1 and NG2 were the lowest and at the same time dislocations

were suppressed. An optimized growth temperature lower as 585 °C was proposed for a high purity sample avoiding the generation of growth temperature sensitive GaAs defects, if possible with N content around 1 % efficient enough to ensure a suitable nitrogen concentration eliminating dislocations.

IV.

Assessment of tasks I - III for the tandem solar cell defect distribution investigation. All the previous achievements were processed to analyse the Tandem A solar cell structure. DLTFs spectrum origin was localized as the intrinsic layer of the bottom InGaAsN p-i-n sub-junction, and an adequate defect investigation was carried out accordingly (Fig. 4.7). Experimentally obtained DLTFs spectrum of the investigated multilayer solar cell structure was compared step by step with all of the examined referent solar cell structures, which made possible to precisely identify relevant defect states.

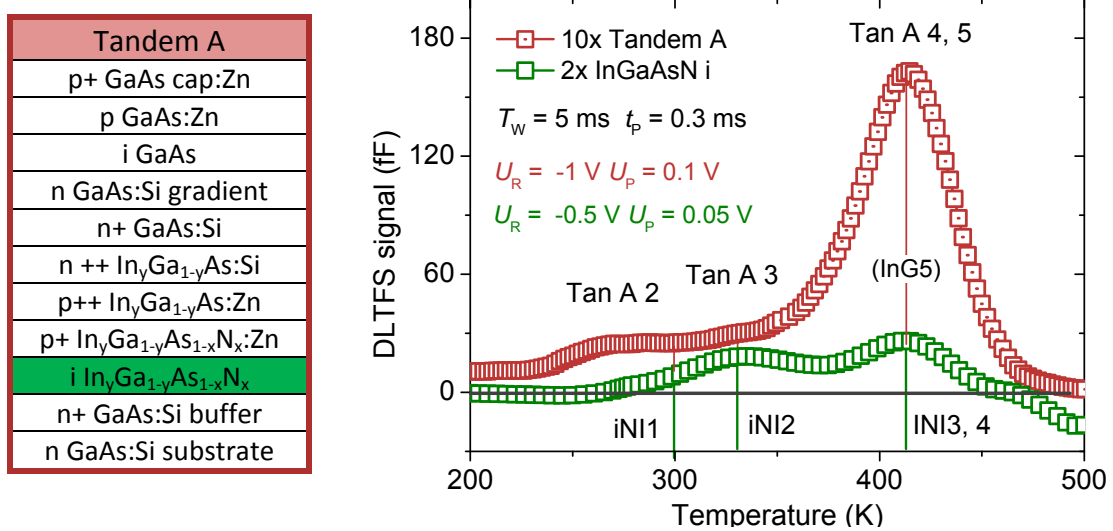


Fig. 4.7 Schematic description of the Tandem A InGaAsN multilayer solar cell with marked InGaAsN i reference of the bottom p-i-n sub junction (left) with compared DLTFs spectrums (right).

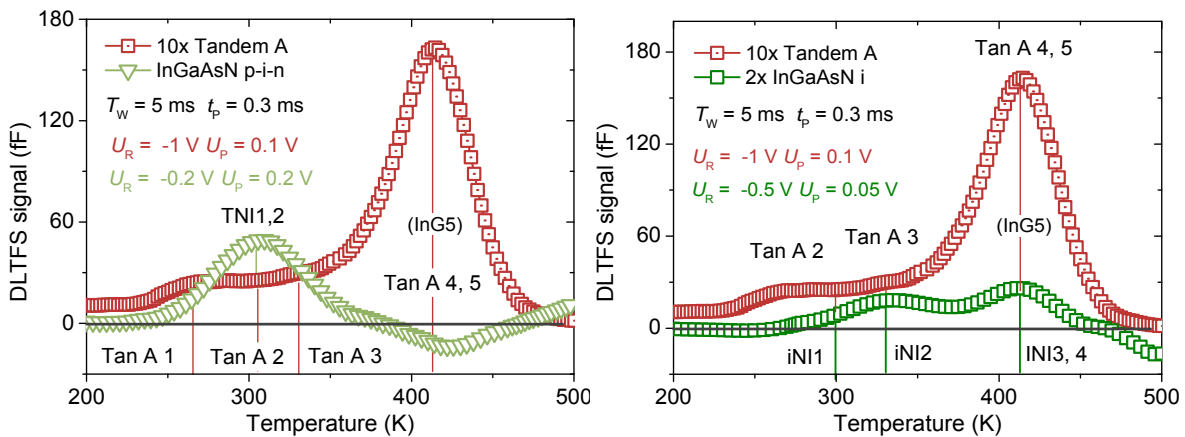


Fig. 4.8 DLTFs comparison of InGaAsN p-i-n, i and Tandem A solar cell samples.

Electrically active defect differences in the InGaAsN intrinsic referent, InGaAsN bottom p-i-n referent and the tandem experiment were observed (Fig. 4.8). These deviations were attributed to the In sensitive dual defect state of InG5 (chapter II.) indicating an another growth condition dependence (the only difference in these samples), thus the hydrogen flow rate of the AP-MOVPE gallium source (TMGa). In the light of discussed achievements, for comparison purposes another tandem solar cell structure Tandem B was examined, in the light of TMGa growth condition correspondences: Tandem A with InGaAsN intrinsic referent sample (7 mil/min) and Tandem B with InGaAsN bottom p-i-n referent structure (10 mil/min). Further growth condition dependent defect state investigations were carried out by the next chapter of the dissertation thesis.

V.

Analysis of growth dependent deep energy levels in tandem solar cell structures. Significant achievement of the Tandem A and Tandem B comparison (Fig. 4.9) is the evidence of InG5 dual defect state and its growth condition dependence probably capable to change the conduction type of the bottom InGaAsN p-i-n intrinsic layer. Identical but inverse DLTFs spectra were measured for Tandem B (Fig. 4.9 right).

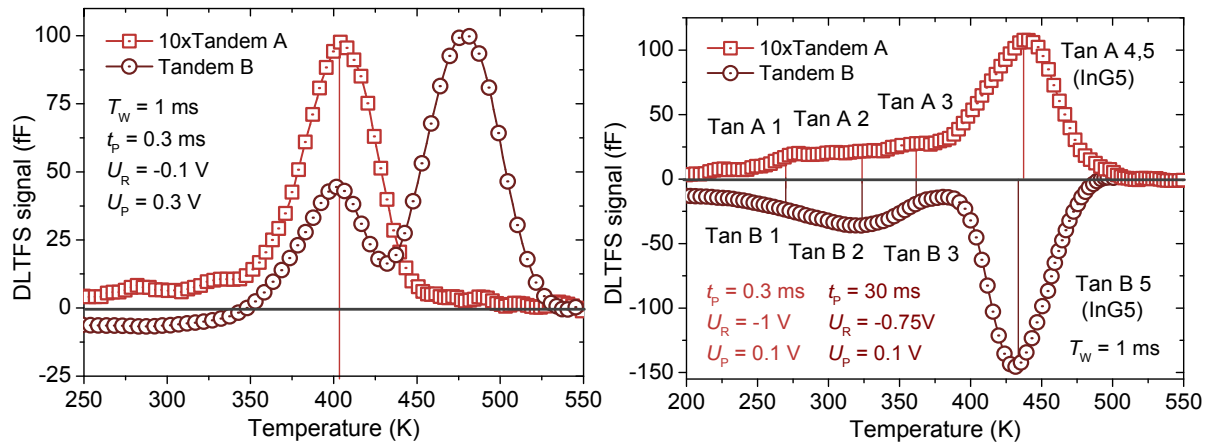


Fig. 4.9 DLTFs spectra comparison of Tandem A and Tandem B InGaAsN multilayer solar cell structures at different experimental conditions.

Several DLTFs experimental outputs and analysis of spectral simulations confirmed the conduction type change tendency of InG5. According to the observed relations the 7 mil/min TMGa flow rate assists the concentration of the p type trap state (InG5 at 0.57 eV of Tandem A Fig. 4.9 right, light red), which can become significant enough to result a p type conduction. On other hand the 10 mil/min TMGa flow rate increases the n-type InG5 concentration, becoming high enough to induce the conduction type change, hence the undoped layer of Tandem B acts as n type (Fig. 4.9 right, dark red). InG5 can co-exist in both forms due to the indium sensitive property (chapter II.) and gradient concentration of InGaAsN layers. This trap state was observed at different DLTFs conditions both in Tandem A and Tandem B samples (Fig. 4.9).

VI.

Specification of a more suitable structure for a high efficient solar cell. Comparison of Tandem A with 7 mil/min and Tandem B with 10 mil/min resulted the assumption of the InGaAsN bottom p-i-n conduction type change and increased concentration about 10 times higher (Fig. 4.9). According to all the evaluated relations Tandem A sample is a more proper candidate for higher multilayer solar cell efficiencies, nevertheless all the proposed In/N factors (tasks II and III) should ensure a multilayer sample of a higher quality. In means of a pure growth process, provision of an oxygen gas purifier should be applied, able to eliminate the oxygen defect relation of the top GaAs p-i-n active layer (task I).

5. Achievements of the dissertation thesis

Main achievements of the dissertation thesis can be summarized as follows:

- proposal of a new Deep Level Transient Spectroscopy evaluation approach of emission and capture processes in multilayer semiconductor structures to increase the effectiveness and reliability of standard DLTFs analysis,
 - implementation of the proposed Arrhenius data sorting method in the evaluation process of a complex dual conduction type defect state typically observed in multilayer semiconductor referent structures,
 - calculation of deep energy level parameters with higher reliability and reproducibility by the utilization of proper DLTFs methods and evaluation procedures (DLTFs with electrical and optical excitation, Isothermal transient spectroscopy, DLTFs experimental parameter variation and optimization, direct and indirect evaluation methods),
- verification of the proposed approach by DLTFs evaluation of emission and capture processes in selected semiconductor structures based on GaAs, InGaAs, GaAsN, InGaAsN,
- implementation and optimization of Minority carrier DLTFs for the DLTFs measurement system DL8000 workplace, extension of InGaAsN minority carrier defect analysis,
- acquirement of new knowledge about emission and capture processes and related defect states in multilayer solar cell structures based on InGaAsN in order to support the growth process,
 - more than 20 deep energy levels were identified in referent structures, many of these corresponding with the tandem solar cell experiment,
 - based on spectral comparison and analysis probable type and origin of identified undoped layer defect states were stated,
- specification of structural properties and growth conditions to achieve higher solar cell efficiencies of fabricated multilayer structures,
 - 10.5 % of indium in InGaAs samples decreases/eliminates low temperature point defects

- 1.6 % of nitrogen at growth temperature 565 °C of GaAsN samples suppresses dislocations and reduces GaAs/N complex defect amplitudes,
- indium content and growth condition dependence of a dual type complex (EL2/EL2²⁺) was confirmed experimentally and by calculations,
- nitrogen content related and growth temperature dependent GaAs defect complexes were evaluated,
- according to the analysis and defect distributions in tandem solar cell samples the gallium dopant source with 7mil/min flow rate, indium around 9.5 % and nitrogen with 1 % at growth temperature lower as 585 °C were stated as preferable,

6. Conclusion

Development of high quality semiconductor materials is an important factor in the technical advancement of semiconductor structures. The production has to be supported by appropriate diagnostic methods, in which Deep Level Transient Fourier Spectroscopy has a key role. Although DLTFs satisfies basic requirements of diagnostics like other techniques it has its limitations. The most difficult parts of the DLTFs investigation is the interpretation of experimental results. The reliability of the method is mainly affected by complex situations, like the involvement of multilayer structures. Utilization of various DLTFs defect recognition techniques, as well as analytical approaches and referent structures is crucial in complicated structure analysis.

Objects of this study, InGaAsN based tandem solar cells were chosen fulfilling all the structural properties, which require complex evaluations. These are: involvement of several semiconductor junctions (p-n, n-p, p-i-n) in homogeneous and heterogeneous compositions complicating the localization of DLTFs response origins, layers of different doped and undoped materials (GaAs, InGaAs, GaAsN, InGaAsN) questioning the defect type determination, gradient compositions (different In and N concentrations - different band gaps) affecting the trap parameter calculation, and last but not least the growth condition dependence having the potential to generate different defect complexes.

As this study shows, the defect distribution DLTFs investigation of complicated semiconductor structures is essential to reveal defect behaviours of composition, and growth condition sensitive meta-stable and complex deep energy levels. It has the potential to support the growth process and the understanding of generated defects. Comprehensive analysis is crucial, since the standard DLTFs evaluation is not sufficient and adequate enough to observe reliable results. Several analytical and experimental methods are needed to be exploited and processed to ensure a reliable defect parameter calculation. Special demands of multilayer samples need an effective DLTFs guide of electrically active trap investigations to ensure a pure technological process and mass utilization in praxes.

7. Resumé

Vývoj vysoko kvalitných polovodičových materiálov je dôležitým faktorom pre dosiahnutie technického pokroku v oblasti polovodičových štruktúr. Výroba musí byť podporovaná vhodnými diagnostickými metódami na skúmanie kvality, medzi ktoré patrí spektroskopia hlbokých hladín (DLTS), univerzálna metóda na identifikáciu parametrov elektricky aktívnych porúch v polovodičových bariérových štruktúrach. Táto dizertačná práca sa zaoberá návrhom efektívneho skúmania emisno-záchytných procesov vo viacvrstvových polovodičových štruktúrach. Komplikované štruktúry obsahujúce nielen rôzne materiály ale aj viaceré homogénne a heterogénne polovodičové prechody (n-p, p-n, p-i-n) sú príčinou nejednoznačných výsledkov DLTS experimentov a následne náročnej interpretácie elektricky aktívnych porúch. Tieto defekty môžu byť nielen kompozične ale aj technologicky závislé, čo znamená, že rôzne koncentrácie prvkov zlúčenín, ale aj rôzne rastové podmienky, ako je napr. teplota, môžu byť pôvodom generovaných komplexných porúch. Fourierova spektroskopia hlbokých energetických hladín (DLTFS), digitálna modifikácia štandardnej DLTS, je v súčasnosti jedna z najvyužívanejších metód DLTS diagnostiky, ktorá sa vyznačuje vysokou citlivosťou, nedeštruktívnym charakterom a adaptabilitou pre rôzne materiály. Z analytických a experimentálnych výstupov metódy DLTFS je možné určiť pôvod a základné parametre identifikovaných defektov. DLTFS rovnako ako ostatné experimentálne metódy, má ale svoje obmedzenia.

Cieľom výskumu prezentovaného v tejto práci bolo riešiť jednotlivé výzvy DLTFS metódy súvisiace s viacvrstvovými polovodičovými štruktúrami ako sú: skúmanie presnosti danej metodiky z hľadiska vyhodnotenia vplyvu nedopovaných štruktúr pre určenie typu vodivosti defektov, analýza kompozične závislých hlbokých energetických hladín rôznych materiálov (rôzne šírky zakázaného pásma) a vyšetrenie vplyvu rastových podmienok, ktoré majú priamy dosah na presnosť výpočtu parametrov porúch.

Súčasný výskum sa orientuje na vývoj vysokoúčinných viacvrstvových solárnych článkov, v ktorých InGaAsN predstavuje perspektívny materiál. Pridávaním malého množstva india alebo dusíka do tejto zlúčeniny je možné dosiahnuť výhodné vlastnosti, ako sú napríklad rôzne šírky zakázaného pásma optimalizované pre rôzne vlnové dĺžky slnečného žiarenia. S cieľom dosiahnuť vysokú účinnosť, sú z tohto materiálu vytvorené takzvané tandem solárne články obsahujúce viaceré aktívne vrstvy teda aj polovodičové prechody a heteroprechody. Skúmanie týchto štruktúr má potenciál vyriešiť jednotlivé DLTFS výzvy, preto predmetom predloženej práce bolo vykonanie komplexnej diagnostiky InGaAsN tandemových solárnych článkov s viacerými polovodičovými štruktúrami vytvorených pri rôznych rastových podmienkach.

Výsledky dizertačnej práce a jednotlivé prínosy boli roztriedené do šiestich bodov podľa plánu analýz, dostupných referenčných vzoriek a štruktúrnych vlastností InGaAsN tandemových článkov pozostávajúcich z troch štruktúr: hornej GaAs p-i-n diódy, InGaAs n-p tunelovacej diódy a dolnej InGaAsN p-i-n diódy. Veľká pozornosť bola venovaná presnosti

vyhodnotenia nedopovaných vrstiev, rôznych kompozícií a rastových podmienok referenčných štruktúr.

I.

DLTFS analýza hornej GaAs p-i-n štruktúry z hľadiska zvýšenia presnosti štandardného výpočtu parametrov hlbokých energetických hladín, a to hlavne v prípade komplexného defektu s duálnym vodivostným typom (majoritné/minoritné) v nedopovanej polovodičovej vrstve. Tieto špecifické podmienky majú za následok nejednoznačnosť výsledkov analýzy a určenia typu a pôvodu porúch. V nameraných DLTFS spektrách je často pozorovaný prudký pokles spojený so zmenou z kladného na záporný signál (Fig. 4.1). Bol navrhnutý unikátny analytický prístup na triedenie spektrálnych dát (Fig. 4.1), ktorý je schopný prekonať obmedzenia DLTFS štandardného vyhodnotenia a zabezpečiť presnejšie určenie poruchových stavov. Na potvrdenie správnosti navrhnutej metodiky a adekvátnosť získaných výsledkov boli využité relevantné experimentálne metódy: variácia experimentálnych meracích parametrov, Izotermálna spektroskopia hlbokých energetických hladín, DLTFS s optickou excitáciou (MCDLTFS), komparácia s referenčnými štruktúrami a simulácie DLTFS spektier. Je potrebné vyzdvihnúť, že dôležitá časť experimentu, teda využitie DLTFS metódy s optickou excitáciou vhodnej na skúmanie minoritných porúch bolo na meracom pracovisku DL8000 implementované prvýkrát (Fig. 4.2). Boli špecifikované a zaznamenané optimalizované postupy DLTFS experimentov s optickou excitáciou, ako je nakontaktovanie a umiestnenie štruktúr, nastavenie optického zdroja (optický zdroj: GaAs laserová dióda s vlnovou dĺžkou $\lambda = 850$ nm) a vhodných meracích podmienok. Stali sa základom bežných DLTFS experimentov. S vysokou pravdepodobnosťou bol identifikovaný pôvod a vodivostný typ porúch v intrinzickej GaAs vrstve. Vyhodnotenia realizované so zvýšenou presnosťou potvrdili prítomnosť dominantných defektov duálneho komplexu, a to prítomnosť niklu (0.48 eV [20], [21]) elektrónového typu a pravdepodobne dierového meta-stabilného stavu známeho GaAs defektu EL2 (0.63 [22]) súvisiaceho s kyslíkom. Bolo stanovené, že vznik týchto porúch bol priamym následkom rastového procesu.

II.

Vyhodnotenie priameho vplyvu veľkosti koncentrácie india na generáciu alebo zánik elektricky aktívnych porúch v InGaAs štruktúrach na podporu optimalizácie tunelovej InGaAs medzivrstvy tandemového solárneho článku (Fig. 4.3). Hlavným prínosom tejto časti práce je zadefinovanie vhodnej koncentrácie india na dosiahnutie štruktúry s najnižšou aktivitou porúch. Skúmané boli štyri referenčné vzorky s koncentraciami 3,4 %, 8,9 %, 10,5 % a 12,8 %. Zistené boli tri významné vplyvy veľkosti koncentrácie india: posunutie DLTFS pík a spektier prisudzované rozdielom v šírke zakázaných pásiem spôsobených rôznou veľkosťou koncentrácie In, zníženie koncentrácie bodových porúch pri vyšších obsahoch India okolo 10,5 % (Fig. 4.3), experimentálne ale aj výpočtom potvrdená prítomnosť závažnej komplexnej poruchy s duálnym stavom (Fig. 4.4). Boli určené dve významné hlboké energetické hladiny, a to InG4 ako Cu (0,48 eV [23]) dierového typu prítomná vo všetkých vzorkách (Fig. 4.4 vľavo) a InG5 ako komplex s duálnym stavom

(Fig. 4.4 vpravo) elektrónového typu EL2 (0,77 eV [24]) spolu s dierovým meta-stabilným komplexom $EL2^{2+}$ (0,57 eV [25]). Zo získaných výsledkov vyplýva, že s veľkou pravdepodobnosťou je prechod tejto poruchy medzi dvoma stavmi priamo spojený s koncentráciou India. Pokiaľ sú informácie výskumných štúdií adekvátne, meta-stabilný charakter poruchy EL2 nebol doteraz zaznamenaný v súvislosti s indiom, avšak diskutovaný prechodový jav bol jednoznačne potvrdený DLTFs experimentálnymi výsledkami. Na základe počtu a koncentrácie porúch bola určená ako najčistejšia vzorka s 10,5 % india. Tiež bola stanovená hypotetická hodnota 9.5 % ako postačujúca na elimináciu bodových defektov, pri ktorej je InG5 duálny komplex pravdepodobne v rovnováhe.

III.

Podrobná analýza dolnej InGaAsN p-i-n referenčnej štruktúry tandemového solárneho článku. Najdôležitejším prínosom tejto časti je vyhodnotenie priameho vplyvu koncentrácie dusíka na generáciu alebo zánik elektricky aktívnych porúch v GaAsN štruktúrach. Získané výsledky spolu s InGaAs experimentmi (bod II) sú nevyhnutné na podporu DLTFs vyhodnotenia dolnej InGaAsN p-i-n štruktúry. Bolo skúmaných päť referenčných vzoriek s koncentraciami dusíka 0,9 %, 1,15 %, 1,5 %, 1,6 % a 1,85 %, čo umožnilo vyšetriť otázku DLTFs problematiky gradientných kompozícií a závislosti defektov od rastových podmienok. Každá vzorka bola narastená pri inej teplote, aby bola dosiahnutá iná koncentrácia dusíka (585 °C, 605 °C, 595 °C, 565 °C, 575 °C). Boli určené tri významné vplyvy dusíka a rastovej teploty: posunutie DLTFs píkov (Fig. 4.6), teda fluktuácia aktivačných energií vyvolaná rôznymi koncentraciami dusíka t.j. rôznymi šírkami zakázaných pásem GaAsN, zníženie koncentrácie dislokačných defektov pri vyšších obsahoch dusíka okolo 1 % (Fig. 4.5), pravdepodobná prítomnosť poruchového komplexu čiastočne závislého od N koncentrácie ale aj rastovej teploty. Pomocou DLTFs experimentov a analýz boli určené dva komplexy NG1 and NG2 s interagujúcimi poruchami GaAs a N pôvodu, ktoré je možné interpretovať nasledovne: NG1 bola identifikovaná ako komplex dvoch dierových porúch, a to GaAs typu Cu ($NG1^* \cong 0.46$ eV [23]) spolu s dusíkom indukovaným defektom ($NG1^{**} \cong 0,54$ eV [26]). Ďalej NG2 ako komplex s duálnym vodivostným typom s komplexnými členmi elektrónového dusíkového ($NG2 \cong 0,66$ eV [27]) a dierového GaAs typu ($NG2 \cong 0,71$ eV [28]). Toto tvrdenie bolo stanovené podľa charakteru DLTFs spektier a píkov NG1/NG2, ktoré mali nelineárne vlastnosti, teda v prípade NG1 maximálna amplitúda bola pozorovaná nielen pri najvyššej koncentrácii dusíka, ale aj pre najvyššiu rastovú teplotu rôznych vzoriek. Podľa toho je možné predpokladať, že s veľkou pravdepodobnosťou každý komplexný člen má svoj dominantný charakter pre daný štruktúrally/rastový parameter. Znamená to, že pri vyššej rastovej teplote GaAs porucha sa stáva dominantnou, zatiaľ čo vyššia koncentrácia N je priaznivá pre dusíkom indukované poruchové stavy (Fig. 4.6). Referenčná vzorka s parametrami N = 1,60 % pri 565 °C bola najvhodnejšia z hľadiska vyváženosti elektricky aktívnych porúch, pri ktorom amplitúdy komplexov NG1 a NG2 boli najnižšie a v rovnakom prípade boli odstránené elektricky aktívne dislokácie. Technológom bola na zväzanie navrhnutá optimalizovaná rastová teplota nižšia ako 585 °C, ktorá by mohla zabrániť vzniku

teplotne senzitivných GaAs porúch. Pokiaľ by bola vzorka narastená s koncentráciou okolo $N = 1 \%$ je predpoklad, že bude zabezpečená eliminácia dislokácií.

IV.

Zhrnutie výsledkov I až III s účelom vykonať relevantné a detailné skúmanie poruchových stavov viacvrstvého tandemového solárneho článku na základe systematického porovnávania referenčných experimentov. Na základe získaných výsledkov bola lokalizovaná DLTFs odozva viacvrstvého solárneho článku, ktorej pôvod je pravdepodobne z InGaAsN nedopovanej vrstvy dolnej p-i-n štruktúry (Fig. 4.7). Zároveň boli pozorované závažné rozdielne elektricky aktívne poruchové stavy referenčných vzoriek InGaAsN i a InGaAsN p-i-n v porovnaní s tandemovou štruktúrou (Tandem A, Fig. 4.8). Rozdiely boli preskúmané v súvislosti s InG5 komplexom duálneho stavu (bod II) indukujúcim ďalší možný faktor ovplyvňujúci meta-stabilný stav tohto defektu. Jediný rozdiel medzi týmito vzorkami okrem In/N gradientu bol jeden z APMOPE technologických parametrov, teda pomer toku vodíka gálium zdroja (TMGa). Na základe týchto skutočností bola zahrnutá do ďalšieho výskumu druhá viacvrstvá štruktúra (Tandem B) s identickými rastovými podmienkami ako InGaAsN p-i-n referenčná vzorka. Podľa korešpondujúcich TMGa parametrov boli vykonané nasledovné porovnávania: Tandem A s InGaAsN intrinsickou referenčnou vzorkou (TMGa = 7 mil/min) a Tandem B s InGaAsN dolnou p-i-n referenčnou štruktúrou (TMGa = 10 mil/min). Podrobné vyhodnotenie bolo vykonané ako hlavná úloha ďalšej kapitoly dizertačnej práce.

V.

DLTFs analýza vplyvu rastových podmienok na hlboké energetické hladiny tandemového solárneho článku. Najdôležitejším výstupom porovnania Tandemu A a Tandemu B je dôkaz prítomnosti poruchy InG5 duálneho komplexu, ktorá je schopná pri daných rastových podmienkach zmeniť typ vodivosti nedopovanej vrstvy InGaAsN dolnej p-i-n štruktúry (Fig. 4.9). Viaceré experimentálne, analytické ale aj simulačné výsledky podporili túto hypotézu a ukázalo sa, že pomer toku gália TMGa = 7 mil/min vyvolá výraznejšiu koncentráciu dierového stavu duálneho komplexu (InG5 = 0.57 eV), pričom TMGa = 10 mil/min zvyšuje prítomnosť InG5 defektov opačného, elektrónového typu (Fig. 4.9 vpravo). Pravdepodobne v oboch prípadoch rastovo indukovaná koncentrácia porúch sa stáva takou výraznou, že dokáže zmeniť vodivosť. InG5 môže koexistovať v oboch stavoch vzhľadom na In citlivosť (bod II) a gradientnú kompozíciu InGaAsN vrstiev, teda bolo možné pozorovať prítomnosť týchto energií pri rôznych DLTFs meracích podmienkach.

VI.

Špecifikácia najvhodnejšej viacvrstvovej štruktúry solárneho článku na dosiahnutie vyššej účinnosti a návrh faktorov pre vylepšenie štruktúrnych vlastností. Porovnanie výsledkov získaných z DLTFs spektier nameraných na tandemových solárnych článkoch Tandem A (TMGa = 7 mil/min) a Tandem B (TMGa = 10 mil/min) umožnilo stanoviť rôzne poruchové stavy závislé nielen na kompozičné vlastnosti ale aj rastové podmienky. Vo vzorke Tandem B bola pozorovaná zmena vodivostného typu aktívnej vrstvy dolnej p-i-n InGaAsN štruktúry

a približne desať násobné zvýšenie koncentrácie porúch (Fig. 4.9). Na základe dosiahnutých výsledkov vzorka Tandem A je viac vyhovujúca pre vyššie účinnosti, avšak navrhnuté štrukturálne zmeny podľa In/N faktorov porúch (body II, III) by mali zabezpečiť solárny článok s vylepšenými vlastnosťami. Z hľadiska zvýšenia čistoty technologického procesu, by bolo žiaduce zavedenie vysoko kvalitného čistiacieho modulu kyslíka na elimináciu poruchových stavov súvisiacich s kyslíkom aktívnej vrstvy hornej GaAs p-i-n štruktúry.

Prínosy a výstupy dizertačnej práce poukázali na nevyhnutnosť skúmania distribúcie poruchových stavov metódou DLTFs vo viacvrstvových komplikovaných polovodičových štruktúrach. Aby bolo možné identifikovať rastovo ale aj kompozične závislé meta-stabilné a komplexné poruchové stavy je dôležité využiť rôzne experimentálne, ale aj analytické metódy, ktoré sú schopné prekonať limitujúce faktory a nedostatky presnosti štandardného DLTFs vyhodnotenia, a to hlavne pre špecifické prípady nedopovaných vrstiev. Vysoké požiadavky na kvalitu viacvrstvových štruktúr vytvárajú potrebu efektívneho vyhodnotenia elektricky aktívnych porúch na podporu čo najčistejšieho technologického procesu a možnosti širšieho zavedenia výsledných viacvrstvových polovodičových prvkov do praxe.

Hlavné prínosy dizertačnej práce sú zhrnuté v nasledujúcich bodoch:

- navrhnutie nového prístupu na vyhodnocovanie emisných a záchytných procesov spektroskopiou hlbokých energetických hladín vo viacvrstvových polovodičových štruktúrach s cieľom zvýšiť efektívnosť a spoľahlivosť DLTFs analýz,
 - návrh analytického riešenia na triedenie Arrheniových dát za účelom implementácie do štandardného DLTFs vyhodnotenia v prípade komplexného defektu s duálnym vodivostným typom typickým pre viacvrstvové štruktúry,
 - zvýšenie spoľahlivosti a reprodukovateľnosti vypočítaných parametrov identifikovaných hladín využitím vhodnej kombinácie modifikácií DLTFs metód a vyhodnocovacích techník (DLTFs s elektrickou a optickou excitáciou, Izotermálna spektroskopia hlbokých energetických hladín, optimalizácia vstupných meracích parametrov, priama a nepriama evaluácia...),
- verifikácia návrhu prístupu vyhodnocovania emisných a záchytných procesov metódou DLTFs vo vybraných polovodičových štruktúrach na báze GaAs (InGaAs, InGaAsN a GaAsN),
- implementácia a optimalizácia metodiky DLTFs s optickou excitáciou (MCDLTFs) na meracom pracovisku DL8000, rozšírenie možností identifikácie minoritných porúch v InGaAsN,
- získanie nových poznatkov o emisných a záchytných procesoch vo viacvrstvových polovodičových solárnych článkoch na báze InGaAsN spektroskopiou hlbokých energetických hladín s cieľom optimalizácie výrobného procesu,

- identifikovali sme v referenčných štruktúrach viac ako 20 porúch, z ktorých viaceré korešpondovali s poruchami v skúmanom tandemovom solárnom článku,
- na základe porovnania sme určili pravdepodobný charakter a pôvod porúch v intrinzičných vrstvách,
- špecifikácia štrukturálnych vlastností a rastových podmienok pre dosiahnutie vyššej účinnosti viacvrstvových solárnych článkov,
 - pri koncentrácii india 10,5 % v InGaAs štruktúrach dochádza k zníženiu koncentrácie a eliminácii bodových porúch,
 - v GaAsN štruktúre s koncentráciou dusíka 1,60 % rastené pri 585 °C bola zistená eliminácia dislokácií, zníženie amplitúd GaAs/N komplexov v nameraných DLTS spektrách,
 - experimentálne a výpočtom bola potvrdená závislosť poruchového komplexu s duálnym stavom (EL2/EL2²⁺) od rastových podmienok a od veľkosti koncentrácie india,
 - bola vyhodnotená závislosť poruchových komplexov od koncentrácie dusíka a rastovej teploty,
 - z analýzy distribúcie porúch v tandemových štruktúrach bol ako výhodnejší stanovený prietok gália zo zdroja v rámci rastového procesu 7 mil/min, s koncentráciou india okolo 9,8 % a dusíka 1 % pri rastovej teplote nižšej ako 585 °C.

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