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STUDY OF THE DEEP LEVELS INDUCED BY THE HIGH ENERGY PROTON AND NEUTRON IRRADIATION IN THE STRUCTURES OF HIGH RESISTIVITY Si, SiC AND GaN

Summary of doctoral thesis
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VILNIAUS UNIVERSITETAS
PUSLAIDININKIŲ FIZIKOS INSTITUTAS

Vidmantas Kalendra

DIDELĖS ENERGIJOS PROTONAIS IR NEUTRONAIS SUKURTŲ GILIŲJŲ CENTRŲ TYRIMAS DIDŽIAVARŽIO Si, SiC IR GaN DARINIUOSE

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Santrauka

Pagrindinis šio disertacinio darbo tikslas - išanalizuoti giliuosius centrus didžiavaržėse Si, SiC ir GaN medžiagose, sietinus su didelės energijos protonų bei neutronų spinduliuote sudarytais defektais, atskleisti radiacinių defektų transformacijas po iškaitinimų, didelių energijų spinduliuotės įtaką krūvio pernašai ir pagavai medžiagose, tinkamose jonizuojančiosios spinduliuotės detektoriams, tiriamiems pagal Europos branduolinių tyrimų centro (CERN) projektus.

Disertaciją sudaro įvadas, 5 skyriai, išvados ir cituojamos literatūros sąrašas. Įvade yra aprašoma tiriamoji problema, darbo aktualumas ir naujumas, iškeltas tikslas, sprendžiami uždaviniai bei pateikiami ginamieji teiginiai.

Pirmame skyrelyje apžvelgti tyrimų rezultatai, skelbti literatūroje, analizuojant egzistuojančias nagrinėjama tema problemas bei motyvuojant šio darbo tyrimus. Jau dabar žinoma, kad CERN‘e sumontuoti standartinio silicio jonizuojančiosios spinduliuotės detektoriai nėra pakankamai atsparūs itin dideliems spinduliuotės įtėkiams. Todėl buvo paruošti projektai sukurti atsparesnius spinduliuotės detektorius, pasitelkiant naujas medžiagas. Dėl didelio medžiagos tankio ir plataus draustinių energijų tarpo GaN, SiC galėtų būti tinkamos medžiagos jonizuojančiosios spinduliuotės detektorių gamybai, išvysčius auginimo, legiravimo ir elektrinių charakteristikų tikslingo valdymo metodus. Šios medžiagos yra alternatyva Si detektoriams aukštųjų energijų spinduliuotei registruoti, kai Si atsparumo jonizuojančiajai spinduliuotei didinimo galimybės yra beveik išsemtos. Tačiau gerai išvystytos Si detektorių technologijos įgalina formuoti subtilesnius detektorių darinius, tokius kaip erdvines trijų dimensijų struktūras. Todėl visų apžvelgtų medžiagų tyrimai yra labai aktualūs, sprendžiant artimiausio penkmečio jonizuojančiosios spinduliuotės detektorių, naudojamų itin intensyvių spinduliuočių eksperimentuose, kūrimui.

Antrame skyriuje trumpai aptarta eksperimentų įranga ir pasitelktų metodų pagrindai. Darbe buvo kombinuojama keletas metodų giliųjų lygmenų spektrui ir jo pokyčiams po apšvitų aukštųjų energijų spinduliuote įvertinti, krūvininkų pagavos ir rekombinacijos centrų tankio ir aktyvacijos energijų vertėms surasti, dalelių detektorių svarbiems parametrams, tokiems, kaip nuotėkio srovės stiprio, krūvio surinkimo efektyvumo dydžiams nustatyti. Matavimų jautrio padidinimui buvo pasirinkti nuostoviųjų parametrų tyrimo būdai: srovės stiprio, srovės stiprio temperatūrinių kitimų bei fotosrovės spektrinių priklausomybių matavimai. Buvo analizuojami medžiagos būdingųjų

parametrų kitimai, esant mažiems nuokrypiams nuo pusiausviros stacionariajame arba kvazistacionariajame režimuose, esant mažiems sužadinimo lygiams.

Silicio karbido (SiC) tyrimų rezultatai aptariami trečiame skyriuje, kur analizuojamos elektrinės charakteristikos neapšvitintose medžiagose ir jų kitimai, apšvitinus SiC darinius 24 GeV/c protonais. Iš šiluma skatinamųjų srovių spektrų buvo nustatytos šiluminės aktyvacijos energijų vertės, kurios paaiškintos šilumine krūvininkų generacija iš gilių gaudyklių arba draustinio tarpo potencialo fluktuacijomis, keičiančiomis krūvininkų pernašą. 4H-SiC bandiniuose, apšvitintuose 24 GeV/c energijos protonais, kurių įtėkiai siekė iki 10^{16} cm^{-2}, buvo įvertintas skirtingų izotopų kiekis ir parodyta, kad apšvita didelės energijos protonais pablogina 4H-SiC detektorių voltamperines charakteristikas.

Ketvirtame skyriuje nagrinėjamas spinduliuotės poveikis galio nitrido detektoriams, apšvitinus juos reaktoriaus neutronais ir didelės energijos protonais. Neapšvitintose GaN plokštelėse buvo gauta plati šiluma skatinamųjų srovių spektro juosta, kurios forma nepasikeitė net bandinį pakartotinai šildant. Tai parodo, kad tokią priklausomybę lemia ne šiluminė krūvininkų generacija, bet jų judrio kitimas. Apšvitinus neutronais buvo nustatyti dominuojančių defektų energetiniai lygmenys. Visas defektų ir nevienalytiškumų rinkinys medžiagoje sąlygoja krūvininkų pernašą, kuri gali būti paaiškinama tik įskaitant defektų ir krūvininkų sąveiką ir defektų elektrinio aktyvumo transformacijas, priklausomai nuo temperatūros ir sužadinimo sąlygų. Apšvita 24 GeV/c protonais, kurių įtėkiai buvo keičiami 10^{14} cm^{-2} - 10^{16} cm^{-2} intervale, žymiai pakeitė GaN jonizuojančiosios spinduliuotės detektorių krūvio pernašos charakteristikas. Po apšvitų susidarė ^{7}Be, ^{22}Na ir kiti ilgaamžiai radionuklidai, kurių atominis skaičius $A < 70$.

Didžiavaržio silicio (Si) detektorių tyrimai po apšvitos reaktoriaus neutronais aprašyti penktame skyriuje. Iš fotolaidumo spektrų ir tamsinės srovės temperatūrinių priklausomybių nustatyta, jog dėl apšvitinimo susidarė visa eilė radiacinių defektų, veikiančių, kaip taškiniai krūvininkų pagavos centrai, ir jų kompleksų su kristalo defektais. Šiems defektams priskirtini gilūs energijos lygmenys yra žemiau draustinės energijos juostos vidurio. Nustatyta, kad iškaitinimas temperatūrose iki 120 ºC padidina akceptorių tankį. Tuo tarpu po iškaitinimo aukštesnėse temperatūrose buvo pastebėtas donorų tankio padidėjimas.

Introduction

Research problem. The main requirements for materials in production of the particle detectors capable to function within a harsh vicinity of particle colliders are addressed to: i) purity of material, when semi-insulating materials are desirable to increase thickness of the depleted particle trackers and to enhance charge collection efficiency, ii) to density of material exploited for fabrication of detectors, when radiation energy loss *dE/dx*, due to ionisation processes, and, consequently, radiation damage of detectors can be reduced, iii) to band-gap of semiconductor material, when wide-gap materials are preferential for improvement of the signal-to-noise ratio of the particle detectors, iv) to carrier diffusion parameters of the material and to design of the detector circuitry, to enable functionability of detectors at fast shaping cycles (in time scale of 12- 80 ns) of particle bunches in modern high luminosity colliders. Modern semiconductor particle detectors installed within CERN Large Hadron Collider (LHC) are based on silicon material. However, these detectors are anticipated to be functional only during 5 years, while the LHC experiments, started on 2008, are foreseen for 10 years. The main problem arises due to low radiation tolerance of the Si detectors under irradiation fluences of hadrons above 10^{15} particle/cm^2. Thus, an expensive procedure of the detectors replacement is inevitable. Therefore, several research and development projects (e.g., rd39, rd40, rd50 and etc. [1]) have been arranged in CERN, where hundreds of institutions and researchers are involved in search of new materials and in improvement of the radiation hardness of modern particle detectors. These devices should be capable to handle the high voltages, to collect charges efficiently, to be functional at low temperatures and at high timing rates together with high tolerance of large fluences of particles. Several trends have been developed to accept the mentioned challenges, as the search and characterization of new materials, the design of 3-D detectors, the improvements of circuitry of planar detectors etc.

As the most promising materials, the diamond, SiC and GaN semiconductors have been selected for the fabrication of advanced particle detectors [2,3], being a wide-band gap and high density materials. However, diamond appeared to be a non-stable material in the sense of nuclear transmutations when carbon in a diamond crystal transforms into nitrogen by increasing contamination of the initial detector material with detrimental impurities [4, 5]. Therefore, together with device type matching solutions based on silicon, the material science developments are addressed to replacement of the silicon material by the wide-band gap semiconductors as SiC and GaN. However, a reached level of crystal growth and of doping technologies of the latter materials does not yet meet the high requirements for the wider applications of the novel material based structures. Thus, the state-of-the–art technological level of the commercial production of the semi-insulating SiC and GaN materials also appears to be insufficient for immediate production of the particle detectors based on these wide-band gap materials. Therefore, an in depth study of SiC and GaN materials is necessary to pave basis in design of particle detectors on the wide-band gap materials. The main problems are identification and characterization of the grown-in and radiation induced imperfections of the crystalline structure, those are the most detrimental defects, and finding of the methods for managing of the electrical passivation of defects to improve functioning of particle detectors. The complementary problem is a selection of the most efficient techniques of the complex characterization of materials, when standard methods employed for the evaluation of Si are not simply applicable to SiC and GaN. The main carrier killers in the detector structures are known to be the deep centres ascribed to extended crystal growth defects and to radiation induced ones. Therefore, the spectroscopic techniques should be developed in modern technology of detectors fabrication, based on SiC and GaN. Simultaneously, the specific spectroscopic techniques should be evolved for the characterization of the heavily irradiated Si detectors, when standard measurement methods, such as DLTS (deep level transient spectroscopy), TSC (thermal stimulated currents), and other ones are not applicable due to the high densities of radiation defects which exceed those of dopants. Therefore, a lot of problems within material science and fabrication technology should be solved to optimize characteristics of the state-of–the-art industrial implementations in fabrication of particle detectors of the new generation.

Objectives of research. The aim of this work is namely addressed to developments in material science, defect engineering and measurement technology approaches in optimization of the particle detector structures based on silicon and as well as SiC and GaN.

The main objectives are:

1. Study of the electrical characteristics of the tentative SiC detector structures irradiated by high energy protons to clarify an impact of the intrinsic and radiation induced defects.
2. Evaluation of the impact of ionizing radiation on the changes of the electrical and charge transport characteristics in the tentative particle detectors made of the semi-insulating GaN.
3. Analysis of the deep level spectra in Si detectors, irradiated by high fluencies of reactor neutrons, and examination of variations of the deep centres density upon thermal treatment of the irradiated material.

Relevance and scientific novelty of these investigations consist in separation of the carrier trapping and scattering parameters ascribed to growth and radiation induced defects in wide-band gap SiC and GaN materials for fabrication of the radiation tolerant modern particle detectors. Comparative analysis of the deep level spectra and the electrical characteristics of SiC and GaN under irradiations by alpha particles, protons and neutrons comprises another aspect of scientific novelty in this work. Development of the enhanced sensitivity techniques by the steady-state probing of the photo-ionisation spectra of deep centres in neutron irradiated Si detector structures contains a scientific novelty in characterization technology of heavily irradiated materials.

Practical importance. Investigations made on new materials and their structures for production of particle detectors based on semi-insulating SiC and GaN comprise the technological and applied importance of this study. Innovations in defect control technology, especially, in recognition of extended defects and percolative carrier transport in heavily irradiated detector structures are considered or applied for scientific implementations. These investigations have been performed in the framework of CERN rd50 programme.

Statements to defend:

1. The radiation induced defects in SiC and GaN materials containing large densities of the intrinsic growth defects determine the qualitative changes of carrier transport and recombination properties. At low irradiation densities, these properties, being a

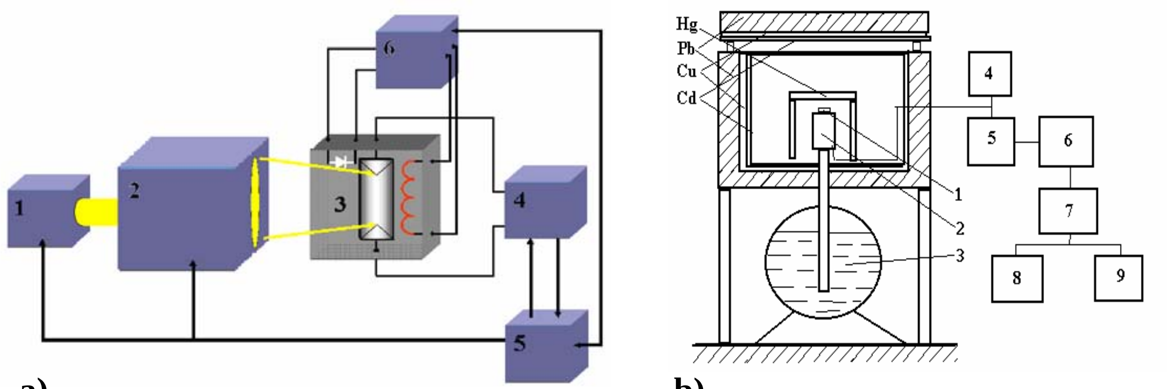
characteristic of the crystalline structures, are modified due to introduction of the radiation-induced point and extended defects. Meanwhile in heavily irradiated materials the percolative carrier transport becomes determinant, which is inherent to the disordered structures.

2. A wide spectrum of radio-isotopes with integral densities proportional to the irradiation fluence is formed in SiC and GaN under hadron irradiations. The steady-state background radiation caused by these isotopes in the irradiated material does not impact considerably the electrical characteristics of the particle detectors and their functionality up to the fluences of $< 10^{16}$ cm^{-2}.
3. Density of the neutron radiation induced point and extended defects in Si increases linearily with fluence by featuring an invariable spectrum of deep levels. These findings are explained by simultaneous production of vacancy associated point complexes as well as of extended clusters of nano-pores under cascade processes of neutrons scattering within silicon particle detectors. Heat-treatments of the post-irradiated material change both the density and the spectrum of deep centres when only point defects are transformed under anneals.

Structure of dissertation. The dissertation consists of introduction, 5 chapters, conclusions, and the reference list (128 titles). The text is written in Lithuanian language on 112 pages with illustrations presented in 47 figures and 5 tables.

A review of the relevant scientific publications is given in the **first chapter** to motivate the present study and to unveil the problems and challenges of the items under investigation. Nowadays SiC and GaN are the main two materials which represent the most promising alternatives for silicon to meet the requirements for radiation-hard detectors. The epitaxial SiC material currently matches to the demands for quality suitable for implementations of the particle detectors, in terms of low equilibrium carrier densities, of carrier lifetime and mobility parameters. However, such layers are not yet available as the semi-insulating free-standing samples of suitable thickness. In order to achieve sufficient signal-to-noise ratio for MIP (Minimal Ionising Particle) detection, it will be necessary to produce material bulks approaching 300 μm thickness for the most specific applications [6-10]. The similar situation exists also concerning GaN, as a vapour phase epitaxy is the preferred growth method for the production of thick layers, when high densities of dislocations are inherent. Fortunately, various companies are developing hydride vapour phase epitaxy (HVPE) technologies for growth of ultra-low dislocation density material, and are now starting to release commercially available free-standing thick material [11-15]. However, parameters of these materials are examined insufficiently. Czochralski technology pulled (Cz) and epitaxially grown n-type silicon had been shown to be the most radiation tolerant materials for fabrication of particle detectors, when comparing these materials with the CERN standard float-zone (sFZ) silicon in respect to the radiation induced changes in effective doping. The fact that both materials do not undergo space charge sign inversion, Cz and epitaxial n-Si materials can be exploited to produce cost-effective and radiation-hard p-in-n detectors [16-20].

The experimental techniques and measurement instrumentation are briefly described in **chapter 2.** A generalized setup of the equipment for measurements of the spectra of thermally stimulated currents and of photo-ionization is sketched in Fig. 1a. Densities and species of radio-active isotopes have been examined by using a gamma spectrometer based on Ge(Li) gamma radiation detector, as sketched in Fig1b.



a) **b)**

Fig. 1. Experimental setup for TSC and photo-ionization spectroscopy of deep centers (a), and a sketch of gamma radiation spectrometer for analysis of radio-isotopes (b). a: 1 – light source, 2 – monochromator DMR4, 3 - liquid helium closed cycle cryostat with sample inside, 4 – DC source with pA meter, 5 – PC, 6 – temperature controller. b: 1 - sample, 2 – Ge(Li) detector, 3 – liquid nitrogen cryostat, 4 – high voltage rectifier, 5,6 – signal amplifier, 7 – signal analyzer, 8 – printer, 9 – PC.

The electrical characteristics and the results of the thermally stimulated currents (TSC) and the gamma spectroscopy of the pristine SiC samples and particle detectors irradiated by 24 GeV/c protons are discussed in **chapter 3.** SiC particle detectors were produced from the bulk vanadium-compensated semi-insulating single crystal 4H-SiC.

The detectors were supplied with a titanium ohmic electrode on the rear surface and a nickel Schottky barrier on the front surface. The dominant defect levels have been unveiled by means of the thermally stimulated current (TSC) and the thermally stimulated depolarization (TSD) techniques. The advanced modification of these techniques is a multiple heating method. In 4H-SiC:V the following thermal activation values have been deduced: 0.18 – 0.19 eV, 0.20 – 0.22 eV, 0.33 – 0.41 eV, and 0.63 eV. The most intensive spectral band within a TSC spectrum (Fig. 2) with an activation energy of 0.33 – 0.41 eV appears below 125 K. It is most probably caused by the thermal generation of carriers from defect levels. Two spectral peaks with the lowest activation energies, which nevertheless appear at higher temperatures, are likely associated with material inhomogeneities due to potential fluctuations of the band gap. Existence of different polarization sources in different temperature ranges have been also corroborated by TSD spectroscopy. Irradiation by 24 GeV protons varying fluence up to 10^{16} cm^{-2} deteriorates rectifying properties of the 4H-SiC particle detectors. The observed changes have been attributed to the appearance of different potential barriers due to destruction of the crystalline structure of material by the high-energy particle bombardment, as illustrated in Fig. 3. This leads to formation of the disordered structure, i.e. to creation and redistribution of different potential barriers in the sample bulk, which inevitably invokes the percolative transport of carriers.

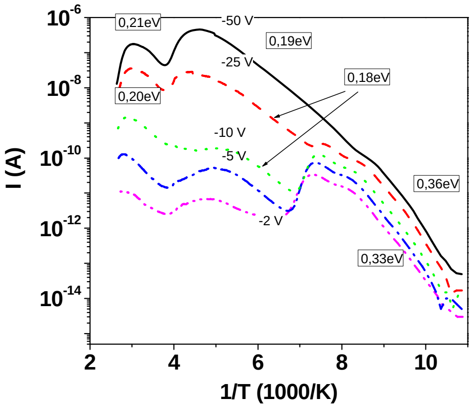


Fig.2. TSC spectra measured in 4H-SiC sample at various reverse voltages. Numbers in rectangles indicate the thermal activation energy values.

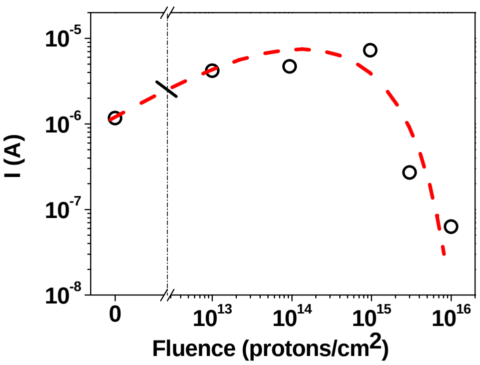


Fig.3. Reverse current as a function of irradiation fluence in 4H-SiC particle detectors irradiated by 24 GeV/c protons.

Table 1. Isotopes produced in SiC particle detectors irradiated by 24 GeV/c protons depending on the irradiation fluence.

Fluence, cm^{-2}	^{11}B $\times10^{9}$	^{10}B $\times10^{9}$	^{9}Be $\times10^{9}$	^{7}Li $\times10^{9}$	^{6}Li $\times10^{9}$	^{4}He $\times10^{10}$	^{3}He $\times10^{9}$	^{2}H $\times10^{9}$	^{1}H $\times10^{9}$
1×10^{13}	0,1	0,15	0,15	0,49	0,77	0,49	0,97	0,89	0,63
1×10^{14}	1,2	1,8	1,8	3,5	9,0	5,8	11	11	7,4
1×10^{15}	8,1	12	12	23	60	38	76	70	50
3×10^{15}	31	45	45	51	230	150	290	270	190
1×10^{16}	75	110	110	230	560	360	710	650	460

Fluence, cm^{-2}	^{28}Si $\times10^{7}$	^{27}Al $\times10^{8}$	^{26}Mg $\times10^{7}$	^{25}Mg $\times10^{10}$	^{24}Mg $\times10^{10}$	^{23}Na $\times10^{10}$	^{22}Ne $\times10^{7}$	^{21}Ne $\times10^{10}$
1×10^{13}	0,16	0,17	0,26	1,3	0,43	0,12	0,66	0,16
1×10^{14}	1,3	1,3	2,1	9,7	3,4	1,0	5,2	1,3
1×10^{15}	8,1	8,4	13	66	27	6,4	25	8,2
3×10^{15}	28	29	45	220	76	21	120	28
1×10^{16}	75	78	110	580	190	56	290	75

Fluence, cm^{-2}	^{20}Ne $\times10^{10}$	^{19}F $\times10^{10}$	^{18}O $\times10^{9}$	^{17}O $\times10^{10}$	^{16}O $\times10^{9}$	^{15}N $\times10^{10}$	^{14}N $\times10^{10}$
1×10^{13}	0,66	0,21	0,59	0,28	0,82	0,35	1,0
1×10^{14}	5,1	1,7	4,5	2,2	6,4	2,8	8,0
1×10^{15}	34	9,8	29	14	41	18	51
3×10^{15}	110	37	100	49	140	62	180
1×10^{16}	310	98	270	130	380	160	470

Different isotopes produced during the irradiation by protons have been revealed by gamma spectroscopy measurements. The isotopes of B, Be, Li, He and H are produced in proton nuclear spallation reactions with carbon. Meanwhile, the Al, Mg, Na, Ne, F, O and N isotopes are produced in proton reactions with silicon, as listed in Table 1. The total amount of the stable isotopes formed in the samples irradiated by 10^{13} protons/cm^{-2} is 1.2×10^{11} cm^{-2}, and it grows up to 5.9×10^{13} in the samples irradiated by 10^{16} protons/cm^{-2}. Activities and amounts of the ^{7}Be and ^{22}Na atoms produced in SiC detectors under irradiation have been measured experimentally, with good repeatability statistics. Activities of other radio-nuclides, in a period of 390 days after the irradiation, have been calculated with reference to these data. Activities of ^{7}Be and ^{22}Na were proportional to the total irradiation dose and ranged from 1.3 to 890 Bq and from 1.9 to

950 Bq, respectively. The production rate of electrons with different energies due to radio-activity of material reaches values from 1 to 600 per second in the 4H-SiC samples irradiated with 1×10^{13} and 1×10^{16} protons/cm^2, respectively, for the 390 days time instant after the irradiation.

In **Chapter 4** investigations of TSCs, of gamma spectra, and of electrical characteristics of the GaN irradiated by high energy protons and reactor neutrons, are described. Single crystal and semi-insulating epitaxial GaN radiation detectors have been examined by means of the TSC and TSD techniques. The complicated carrier transport features have been revealed in GaN structures by the TSC spectra measurements in the multiple heating regime at different applied reverse voltages. In the non-irradiated GaN material, no expressed structure of the TSC spectrum has been observed in the temperature range from 100 to 350 K. Only a wide and flat TSC band (Fig. 4a) has been detected, and shape of this spectrum band did not change even under multiple temperature scans. This is an evidence that such a behaviour is caused by the carrier mobility temperature variations. A numerical analysis of this TSC signal has been performed by taking into account the carrier scattering processes by ionized impurities and phonons. It has been found that mobility varies as a power function T^{α} of temperature with index α=2.8 for scattering by ionized impurities while lattice scattering is characterised by α=-3.5, respectively, Fig.4b. The highest mobility values have been reached in the light-excited samples. An impact of the impurity scattering increases in the course of thermal de-excitation, evidencing an appearance of the potential inhomogeneities within crystal.

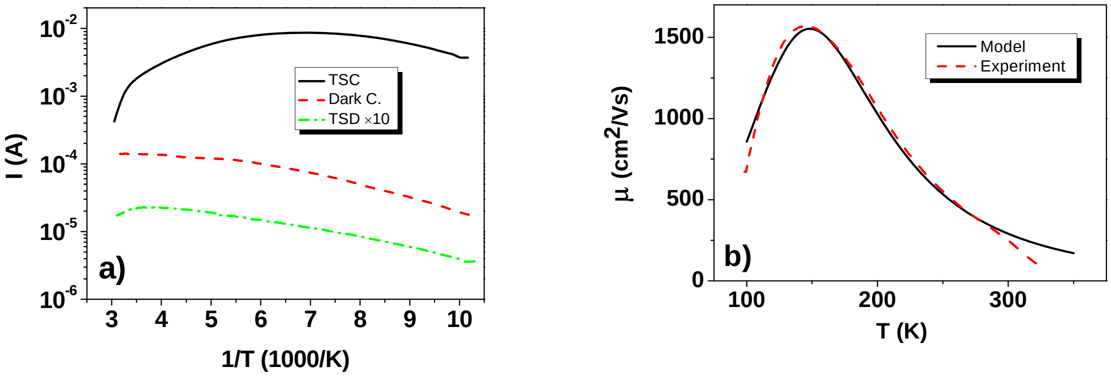


Fig. 4. TSC spectra (a) and simulated temperature variations of mobility (b) in the initial material.

A significant decrease of the TSC current values, clearly dependent on fluence and varied by several orders of magnitude, have been observed in GaN irradiated by reactor neutrons. No mobility dependence on temperature has been observed in the detectors irradiated by 5×10^{14} neutrons/cm^2 (Fig. 5a). In these samples the current instabilities take place. Such an effect could be caused by the percolation effects in the highly disordered material. An increase of the irradiation fluence up to 10^{16} n/cm^2 results in the re-appearance of the TSC structure (Fig. 5b), and the dominant TSC spectrum peaks have been separated with the thermal activation energy values of 0.16–0.2, 0.27–0.32, 0.36–0.45 and of 0.73–0.74 eV. The complete set of defects and the inhomogeneities of crystal structure emerge within the carrier transport characteristics by a specific behaviour, and these effects can be only explained by taking into account the mutual interactions and transformations of defects dependent on temperature and excitation conditions.

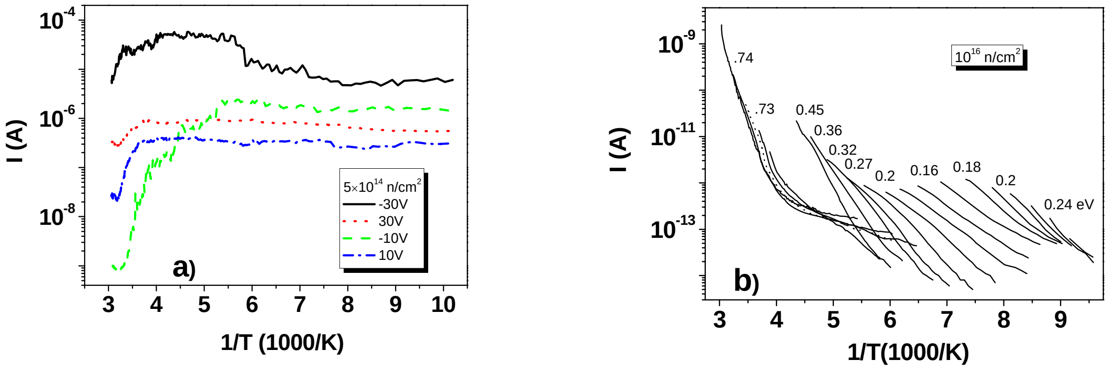


Fig. 5. TSC spectra in neutron irradiated GaN by different fluences: 5×10^{14} n/cm^2 (a), and 10^{16} n/cm^2 (b).

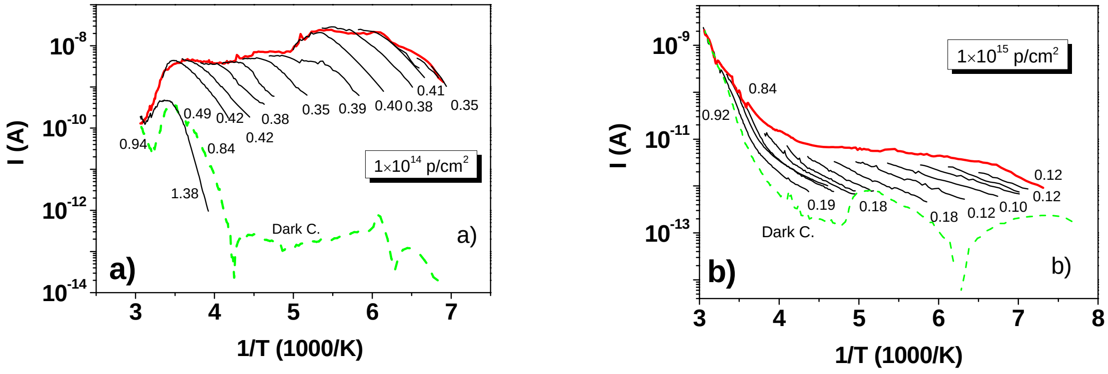


Fig. 6. TSC spectra in proton irradiated GaN by different fluences: 10^{14} p/cm^2 (a), and 10^{15} p/cm^2 (b).

The TSC spectral bands with trap thermal activation energy values of 0.35-0.42 eV and of 0.84-0.94 eV have been estimated in the GaN detectors irradiated by 24 GeV/c protons of 1×10^{14} p/cm^2 fluence, Fig. 6a. Probably these values characterize the heights of the drift barriers created by irradiation and associated with the destruction of the crystalline structure. Meanwhile, in the GaN samples irradiated with $>1\times10^{15}$ p/cm^2 fluence, the TSC spectral band with lower values of thermal activation energy of about of 0.10-0.19 eV prevails in the temperature range below 180-250 K (Fig. 6b). However, the latter value seems to be determined by the height of the drift barriers. The drift barriers appear upon irradiation and could be associated with the destruction of the crystalline structure of material. In the heavily irradiated samples, no structure of the TSC spectrum could be discriminated on the background of the dark current (Fig. 7), and this change in TSC indicates appearance of the fast recombination centres. The structureless TSC and TSD variations seem to be associated with creation of the complex ensemble of point defects and of the extended inhomogeneities, ascribed to the polycrystalline structure.

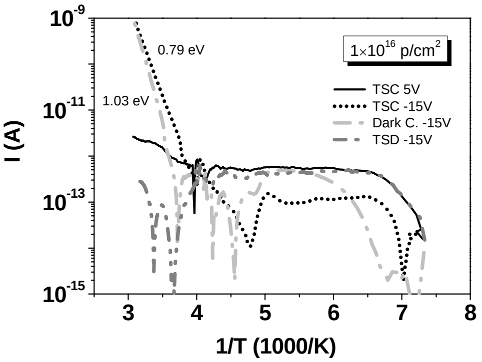


Fig. 7. TSC and TSD spectra in GaN irradiated by protons with fluence of 10^{16} p/cm^2.

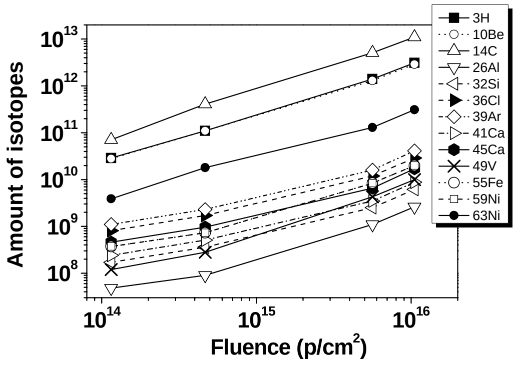


Fig.8. Fluence dependent variations of the amount of radioactive isotopes in proton irradiated GaN.

The isotopes of ^{7}Be, ^{22}Na and other long-living radio-nuclides with atomic numbers of A < 70 have been identified from gamma spectroscopy measurements in proton irradiated GaN detectors. A set of 19 radio-nuclides and of 70 isotopes produced in reactions with Ga nuclei have been evaluated. The fluence dependent variations of the amount of isotopes in 24 GeV/c protons irradiated GaN are illustrated in Fig.8.

Results of the photo-ionisation spectroscopy in neutron irradiated and heat-treated high resistivity Si detectors are presented in **chapter 5**. The samples were irradiated in TRIGA reactor at Ljubljana University by the reactor neutrons with fluencies in the range from 10^{13} to 10^{16} n/cm^2. The isochronal anneals for 15 h were performed at different temperatures in the range from 80 ^{o}C up to 180 ^{o}C. Spectra of the photo-ionisation of deep centres measured by recording variations of dc photocurrent in neutron irradiated p+-n-n+ Si particle detectors are illustrated in Fig. 9. The photo-ionisation spectral "steps", characteristic for the carrier generation from the traps, are employed to analyse variations of a cross-section for the photon-electron interaction at the specific deep levels. The spectral scans were performed first by increasing photon energy and afterwards by decreasing it. The photocurrent spectra in the neutron irradiated samples are presented in Fig. 9a. A drop of the intrinsic photocurrent obtained for the photon energies above ~ 1.2 eV in the irradiated samples is caused by the reduction of the free carrier lifetime reciprocal to irradiation fluence. Meanwhile, for the defect-related extrinsic photoconductivity in the photon energy spectral range of <1.1 eV, a photo-current with inherent step-like structure increases with enhancement of a photon energy in the range of 0.55 - 1.1 eV. The photo-ionisation spectra have been analyzed by simulation of the spectral steps using the Lucovsky deep centre model with δ-potential [21]. The optical activation energy values obtained from the photoconductivity spectra can be related with the excitation of electrons from the filled deep centres or generation of free holes by excitation of electrons from the valence band into the empty deep levels. The shallowest level with photo-ionisation energy of the 0.5 eV is ascribed to the defect states nearly the Fermi level in the irradiated material. Additionally, two groups of the overlapped spectral steps associated with the levels characterized by the optical activation energy values in the ranges of 0.49-0.52 eV and of 0.77-0.91 eV below the conduction band have been discriminated. These levels probably are related with the defects in the vicinity of clusters. A wide scattering range of the photo-ionisation energy values of single spectral "steps" indicates an extended spectral distribution of defect states, beeing specific for the cluster-type defects.

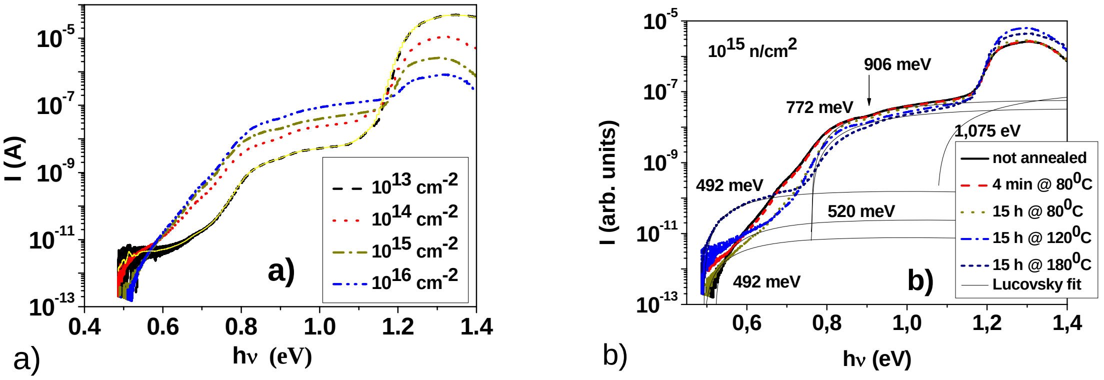


Fig. 9. Photo-ionization spectra measured on neutron irradiated MCz Si particle detectors: (a) after the irradiation, and (b) after heat treatments at different temperatures.

The defect densities, estimated from the relative amplitudes of the photo-ionisation spectral steps, vary with irradiation fluence and under procedures of isochronal heat treatments (Fig. 9b). Appearance and fadeaway of several spectral constituents have been observed for the heat treated samples, those can be associated either with a creation of definite deep centres resulted from transformation of radiation defects and/or from anneal of some other defects. However, densities of most of the separated defects vary non-monotonously with the annealing temperature, by indicating the statistical processes of the intricate transforms of different defect complexes.

Conclusions

1. The thermal activation energy values of 0.18 eV, 0.21 eV, 0.37 eV and of 0.63 eV have been evaluated in the initial 4H-SiC material by the TSC spectroscopy. The structure of TSC spectra and amplitudes of the TSC spectral bands depend on the applied reverse bias voltage due to simultaneous effect of thermal generation of carriers from the deep levels and due to fluctuations of the potential barriers within inhomogeneous relief of the band gap edges..

2. In the proton-irradiated 4H-SiC, the isotopes of B, Be, Li, He and H are produced in proton nuclear spallation reactions with carbon, meanwhile Al, Mg, Na, Ne, F, O and N isotopes are produced in proton reactions with silicon. In the proton irradiated GaN, the isotopes of ^{7}Be, ^{22}Na and other long-living radio-nuclides with atomic number of

$A < 70$ have been identified from gamma spectroscopy measurements. In both materials: 4H-SiC and GaN, activities and amounts of isotopes vary linearly with the total irradiation dose.

3. In the non-irradiated high resisitivity GaN the mobility changes determine the structure of the TSC spectrum, causing a wide and flat spectral band. Values of the TSCs are reduced by several orders of magnitude after irradiation by reactor neutrons due to enhancement of the density of recombination centres, those decrease efficiency of carrier trapping into the shallow levels. The TSC spectra for the neutron irradiated GaN material are comprised of the clearly distinguishable spectral bands characterized by activation energies of 0.18 eV, of 0.29 eV, of 0.40 eV, and of 0.73 eV.

4. The dominant radiation defects with activation energy values of 0.38 eV and of 0.89 eV have been determined in the epitaxial GaN layers for fluence range $\leq 10^{14}$ p/cm^2 of 24 GeV/c proton irradiations, while shallower centres with activation energy of 0.15 eV prevail for irradiation fluences $>10^{15}$ p/cm^2, that are most probably related with the fluctuations of the energy gap edges.

5. The photo-activation energy values of 0.60 eV, of 0.65 eV, of 0.77 eV, and of 0.89 eV have been determined for levels located below the mid-gap of MCz n-Si, irradiated by reactor neutrons, by using experimental photo-ionisation spectra simulated by Lucovsky‘ model. Thermal activation energy values for shallow levels located below an edge of conduction band have been extracted from the TSC spectroscopy data and found to be of 0.10 eV, of 0.25 eV, of 0.34 eV, and of 0.41 eV. The comparative analysis of the activation parameters in this study and data from literature features that centres with activation energy of 0.25 eV and of 0.41eV can be ascribed to ($V_2^{=/-}$, $V_2^{-/0}$) divacancy related radiation defects, while centres with activation energy of 0.34 eV, of 0.60 eV, and of 0.65 eV could be associated with clusters of interstitials and multi-vacancy pores. Other the shallower levels can be interpreted by existence of $C_{i,s}$ and thermo donors related defects.

6. Isochronal heat treatment of the neutron-irradiated MCz Si detectors in the temperature range below 120 ºC enhances the density of the acceptor-type vacancy-related defects, while annealing at the elevated temperatures >150 ºC leads to an increase of density of the donor-type defects.

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Author's contribution. The author of this thesis made all the experiments and preliminary analysis of the results published in the articles [S2-S13] listed below, he had also contributed to the experimental investigations described in [S1, S14]. Author had prepared and presented himself the results at 12 scientific conferences.

Publications. The main results of this study were published in 14 scientific articles [S1-S14] and presented in 30 talks [K1-K30] at several conferences. Publications are listed below.

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S1. J. Vaitkus, A. Blue, W. Cunnigham, E. Gaubas, J. Grant, K. Jarašiūnas, A. Kadys, **V. Kalendra**, V. Kažukauskas, P. Pobedinskas, V. O'Shea, K. Smith, J. Storasta, A. Žukauskas. CERN large hadron collider projects to improve the radiation hardness of ionizing radiation detectors: the role and control of defects in Si potential of GaN. Lithuanian Journ. of Physics, **45** (2005) 437-443.

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