Journal of Nano- and Electronic Physics
Vol. **15** No 1, 01001(6pp) (2023)

Журнал нано- та електронної фізики
Том **15** № 1, 01001(6cc) (2023)

Laser-Induced Modification of the Morphology and Defect Structure of Heterostructures Based on Detector-Grade CdTe Crystals

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(Received 05 December 2022; revised manuscript received 17 February 2023; published online 24 February 2023)

The processes of laser manipulation of the defect-impurity system and laser transformation of the crystal morphology made it possible to increase the detection properties of CdTe-based structures with a Schottky barrier. The structural perfection of CdTe:Cl single crystals was assessed using high-resolution X-ray diffractometry. The contact efficiency depends both on the electrode material and the surface treatment of the CdTe crystal before its deposition, in addition, the characteristics of the formed electrode-semiconductor interface can be changed by various treatments. Irradiation of the surface of CdTe crystals or metal-CdTe structures with laser pulses led to a change in the morphology of the semiconductor surface, the formation and redistribution of defects in the surface region and modification of the characteristics of this region or interface. The effect of laser processing on the structure of impurity defects and electrical characteristics of X-ray/γ-detectors on Schottky diodes, developed by deposition of Ni and NiO on commercially available CdTe:Cl wafers, was studied. Using the methods of atomic force and scanning electron microscopy, the features of Ni and NiO thin films before and after laser irradiation were investigated. The effect of pulsed laser irradiation on Ni/CdTe and NiO/CdTe contacts and the mechanisms of transformation of their phase state has not been investigated; however, such processing of these Schottky contacts led to optimization of their electrical characteristics. It is shown that laser treatment of heterojunctions, both CdTe substrates and Ni and NiO films, can intentionally change the electrical properties and increase the sensitivity of Ni/p-CdTe/Au/Cu and NiO/p-CdTe/Au/Cu detectors. The effect of laser processing on the electrical and spectroscopic properties of CdTe-based Schottky diode X-ray/γ detectors is also discussed.

Keywords: CdTe crystal, Heterostructure, Laser surface treatment, Schottky diode, Reverse current, Charge transport, X/γ-ray detector, Emission spectrum.

DOI: 10.21272/jnep.15(1).01001 PACS numbers: 68.65. – k, 85.30.Hi, 85.30.Kk

1. INTRODUCTION

One of the efficient and attractive techniques of modification of the surface and superficial region of semiconductors, which has been widely and successfully used for various technological procedures in instrument making for more than three decades, is pulsed laser processing [1-3]. In particular, irradiation with strongly absorbed laser pulses has been studied and employed to modify the surface state of semi-insulating CdTe crystals, provide doping of their surface layer and create a p-n junction, form electrical contacts and other procedures during fabrication of X/γ-ray detectors [4-7]. Application of pulsed irradiation for irradiation of CdTe crystals pre-coated with a metal film allowed us to create p-n junction-diode X/γ-ray detectors operating at room temperature with extremely high energy resolution [7].

Furthermore, the parameters of the diode structures can be modified by laser treatment of the CdTe crystals during and after electrode deposition. It is practically important because we have recently developed CdTe Schottky-diode detectors with different electrodes and efficient optimization of the contact properties have been required [8-11].

In the present work, we continue to investigate the CdTe-based Schottky-diode detectors formed by deposition of different contact materials onto pre-cleaned commercially available detector-grade (111) oriented CdTe wafers [3, 4]. The similar CdTe crystals were also used in our previous research devoted to development of X/γ-ray detectors based on both p-n junction diodes formed by nanosecond laser-induced doping [4-6] and structures with a Schottky barrier [7-9]. The distinctive features of this study are the use millisecond laser pulses to modify the surface morphology and structure of the superficial region of CdTe before deposition of Ni and NiO contacts as well as optimize the electrical characteristics of the formed Ni/p-CdTe/Au/Cu and NiO/p-CdTe/Au/Cu heterostructures by irradiation of the Ni and NiO films deposited on the semiconductor crystals.

2. MATERIALS AND INVESTIGATION METHODS

High-resistivity commercial (111) oriented detector-grade CdTe single crystals produced by Acrorad Corporation were used in the investigations [10].

For fabrication of X- and γ-ray detectors we used (111) oriented CdTe single crystal wafers with sizes of $5 \times 5 \times 0.5$ mm^3 where 0.5 mm was the thickness of the crystal. The resistivity value of the samples, measured at room temperature, was $\rho \sim 10^9\ \Omega\cdot$cm. The Fermi level in the CdTe single crystals under study is located below the Fermi level in an intrinsic semiconductor, indicating

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the hole conductivity of the material, i.e., the CdTe crystals can be considered as an almost intrinsic semiconductor with *p*-like type conductivity [2, 5-9, 11-13].

Ni and NiO thin films were deposited onto the chemically etched in a $K_2Cr_2O_7 + HNO_3 + H_2O$ solution (20-30 s) B-face of *p*-CdTe single crystal substrates in the universal vacuum system Leybold-Heraeus L560 by means of DC-reactive magnetron sputtering.

The pure Ni target (a disc 100 mm in diameter and 1 mm thick) was placed on the table with a water-cooled magnetron. The NiO thin films were deposited by sputtering of Ni in an atmosphere of a mixture of argon and oxygen. During the deposition process, the partial pressure in the vacuum chamber was 4×10^{-3} mbar for argon and 4×10^{-3} mbar for oxygen. The power of magnetron was 100 W. The deposition rate of the thin films was 8 nm/min at substrate temperature of 300 K. The obtained NiO thin films possessed *n*-type of conductivity. The measured specific conductivity and the concentration of electrons were $0.34\ \Omega^{-1}\cdot\mathrm{cm}^{-1}$ and $4.3 \times 10^{18}\ \mathrm{cm}^{-3}$, respectively (295 K). For deposition Ni thin films, the same regimes were used as for oxide of nickel without oxygen.

The Au and Cu layers were successively deposited onto the modified back surface by means of the reduction from aqueous solutions of gold chloride and copper vitriol, respectively [4-8]. In order to modify and improve the structure and phase state of the Ni/CdTe and NiO/CdTe Schottky contacts, they were radiated by out of focus millisecond (1.5 ms) pulse laser beam using a diaphragm with tungsten. The yttrium aluminum garnet (YAG) laser with 1.064 μm wavelength in the single pulse mode was used. Laser treatment modes with energy density $E = 2$-$4.5\ \mathrm{J/cm^2}$ were selected so that the threshold of melting or destruction of the material in the process of heating the surface and near-surface layers was not reached.

The mechanical stresses and lattice deformations of the set of CdTe wafers in different geometry of reflections (111), (333), (331) were studied by X-ray diffraction (XRD) using the PANalytical Philips X'Pert PRO Material Research Diffractometer equipped with a standard $\mathrm{CuK}_{\alpha 1}$ X-ray tube. A high-resolution set with a parabolic mirror, located behind a standard $\mathrm{CuK}_{\alpha 1}$ X-ray tube, followed by a four-crystal monochromator Bartels (4×Ge220) and a point detector with a triple crystal analyzer (3×Ge220) was used for X-ray studies. The divergence of the primary beam and the angular acceptance of the analyzer used in front of the detector is $\Delta a_i, f \approx 12$ angular seconds[14].

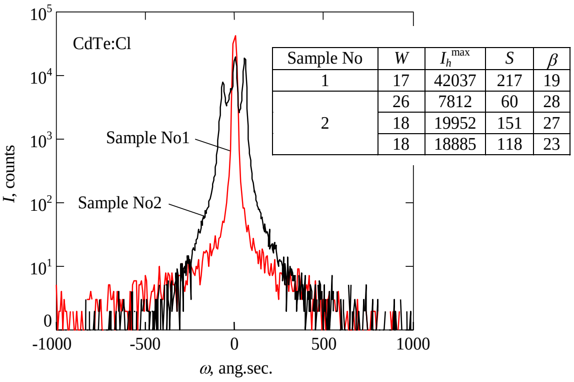
The degree of crystallinity and surface state of CdTe crystals were examined by reflection high-energy electron diffraction (RHEED) measurements at room temperature and a chamber pressure of 2×10^{-5} mbar. The RHEED patterns were obtained at room temperature at acceleration voltage of 80 keV. Rotating the sample around an axis perpendicular to the sampling surface during RHEED experiments allowed to choose the azimuth angle which satisfied the Laue equation describes the diffraction condition. The diffraction pattern was displayed on the CdS phosphor screen.

The morphology, structure, and phase state of the Ni/CdTe and NiO/CdTe detectors before and after laser treatment were monitored by SEM and electron probe microanalysis method on Zeiss EVO 50 XVP microscope at 20 keV voltage with standard image processing application and also by atomic force microscopy (AFM) on NT-206 set equipped with control software SurfaceScan and AFM-image processing application SurfaceView. Inspection of the scan area of 1 × 0.75 mm and the movement of the probe above the surface was provided by a real-time video camera when visualizing an image on a monitor of 640 × 480 pixels and a frame rate of 25-30 frames/s. The AFM measurements were carried out in contact mode using CSC38/AL BS probe manufactured by MikroMasch. The radius of the probe was 8 nm; the number of points on the scan matrix 256 × 256; the load on the probe was 10-12 units.

3. RESULTS AND DISCUSSION

The experimental high resolution XRD (HRXRD) intensity distributions $I_h(\omega)$ and $I_h(\omega, 2\theta\text{-}\omega)$ CdTe:Cl single crystals from symmetrical and asymmetrical reflections of $\mathrm{CuK}_{\alpha 1}$ radiation allow us to evaluate the structural perfection of the samples (Fig. 1). It was established that CdTe:Cl wafers are characterized by well-defined defect structure. This is evidenced by the presence of a strong diffuse background on $I_h(2\theta\text{-}\omega, \omega)$ distribution and blurring of the coherent scattering regions on the $I_h(\omega)$. The structural perfection of the CdTe:Cl crystals was evaluated according to the FWHM values *W*, the maximum intensity ($I_h^{\max}$), the integral intensity (*S*), the shape of the coherent scattering peak, as well as by the analysis of the diffuse component (Fig. 1).

To determine the dislocation pattern influence on the reciprocal space maps (RSM) formation two sets of dislocations were chosen: (i) the 60-degree dislocations with Burgers vectors $\vec{b}_1 = a/2[\bar{1}\bar{1}0]$ and $\vec{b}_2 = a/2[011]$ in the $\{1\bar{1}\bar{1}\}$ and $\{\bar{1}1\bar{1}\}$ planes, respectively; (ii) Franck partial dislocations with lines oriented in $\langle 0\bar{1}1|$ and $|\bar{1}01\rangle$ directions. The dislocations can also be placed at the small angular boundaries between separate blocks [14].



Sample No	W	$I_h^{\max}$	S	β
1	17	42037	217	19
2	26	7812	60	28
	18	19952	151	27
	18	18885	118	23

Fig. 1 – Experimental distributions $I_h(\omega)$ for two CdTe:Cl wafers with different degree of perfection; (333) reflection; $\mathrm{CuK}_{\alpha 1}$ radiation. The inset shows the FWHM values (*W*), maximum intensity ($I_h^{\max}$), integral intensity (*S*), and integral width (β)

If the crystal consists of blocks separated by small angular boundaries, formed by one system of dislocations, then the dislocation density N_L in the direction perpendicular to the dislocation lines can be estimated by the angle of disorientation $\Delta\theta$ between two blocks [15]:

$$N_{\mathrm{L}} = \frac{\Delta\theta}{3\left|\vec{b}\right| T_b}, \qquad (1)$$

where T_b is the average size of *a* block, $\vec{b}$ is the Burgers vector of the typical crystal dislocations.

In the case of chaotic distribution of dislocations, which is often found in the real crystals, the average dislocation density N_G can be estimated as

$$N_{\mathrm{G}} = \frac{W_G^2}{9\left|\vec{b}\right|^2}, \qquad (2)$$

where W_G is the stacking faults.

The estimated values of dislocation density N_G and N_L in CdTe:Cl crystals with different degree of perfection are presented in Table 1. As characteristic fragmentary (mosaic) structure appears on the dislocation distribution for sample No2 (Fig. 1), the value of N_G was determined for the most perfect fragment. The possible density of screw dislocation N_S

$$N_{\mathrm{S}} = \frac{\alpha^2}{4.35\left|\vec{b}\right|^2} \qquad (3)$$

estimated from Willamson-Hall plot by the slope angle α of mosaic grain was 4.8×10^5 cm^{-2} and 4.9×10^5 cm^{-2} for samples No1 and No2, respectively (Fig. 2).

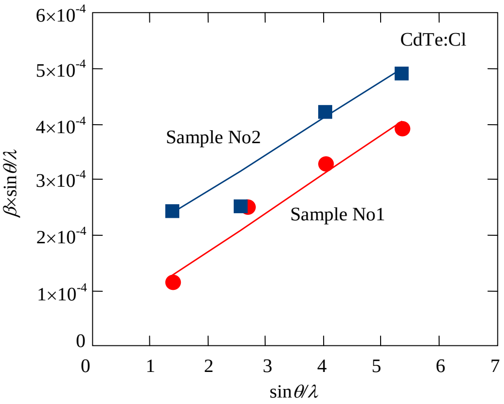


Fig. 2 – Williamson-Hall plot for a series of symmetric (*hhh*) reflexes of *ω*-scans for evaluation of in samples with different degree of perfection

Table 1 – Dislocation densities N_G and N_L in CdTe:Cl crystals with different degree of perfection

Reflection	Sample No1		Sample No2	
	$N_G \times 10^5$ cm^{-2}	$N_L \times 10^6$ cm^{-2}	$N_G \times 10^5$ cm^{-2}	$N_L \times 10^6$ cm^{-2}
111	2.6	–	17.9	2.1
333	3.4	–	5.8	5.1
331	1.3	–	13.1	5.6

The state of the CdTe crystal surface was examined by the reflection high-energy electron diffraction (RHEED) technique. The RHEED patterns were obtained at room temperature at an accelerating voltage of 80 kV. The experimental measurements were performed on the Te-terminated (*B*-face) of the CdTe (111) crystals. After mechanical polishing of the surface of the single-crystal CdTe sample, the diffraction pattern in the form of blurred rings indicates the presence of a thin surface layer with disordered or amorphous structure with plastic deformations which usually occurred after surface polishing treatment on the surface (Fig. 3a). A more perfect surface was obtained by etching of CdTe crystals in a $K_2Cr_2O_7 + HNO_3 + H_2O$ solution during 20-30 s at room temperature. The optimal surface of CdTe plates for spraying thin films was obtained by chemical etching to a depth of $d \geq 10$ μm. In the RHEED pattern, bright spots appeared for such sample (Fig. 3b). Low background intensity and bright spots in the RHEED pattern confirm perfect crystalline structure of the surface region of CdTe crystals after chemical processing.

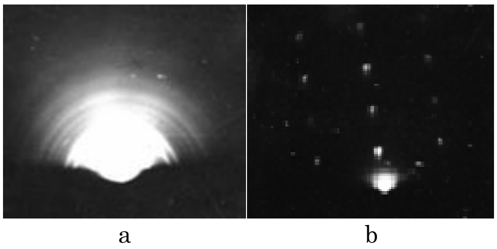
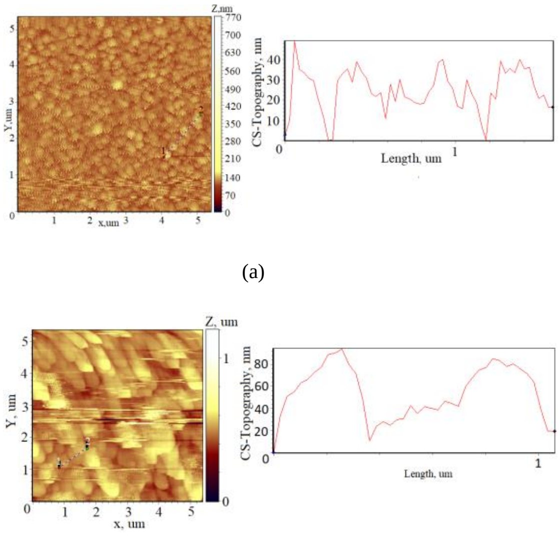


Fig. 3 – RHEED pattern of the CdTe (111) crystal: (a) after mechanical polishing, (b) after chemical etching

The Ni metal film strongly absorbs laser radiation by a system of free electrons. Due to the rapid heating in the Ni film, a significant increase in the lateral size of the crystallites was observed from 0.15-0.25 μm to 0.5-0.7 μm (Fig. 4b).



(a)

(b)

Fig. 4 – AFM-images of Ni film on CdTe single crystal substrates: (a) before laser treatment, (b) after laser treatment with the energy density of 3.6 J/cm². On the right – the scan profiles along lines 1-2

In this case, the area of thermal impact during laser treatment is concentrated near the interface Ni film – CdTe crystal. Therefore, the modification of the crystal is implemented in a relatively small surface layer of CdTe, which increases with the power density of the laser beam.

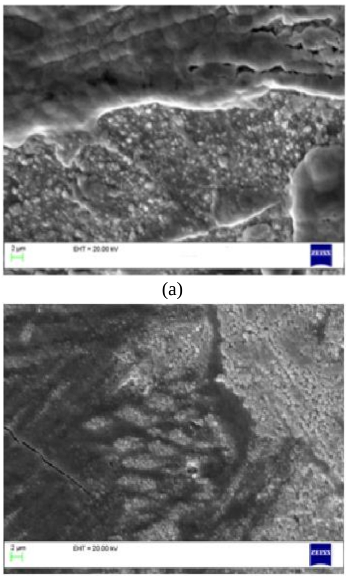
The analysis of scan profiles along lines 1-2 (Fig. 4, on the right) shows that the laser treatment induces an increase in the size of crystallites vertically from 35-38 nm to 80-92 nm. Significant laser-activated transformations of the Ni film structure take place. They are explained by the action of thermoelastic stress fields when reaching high-temperature gradients under the influence of laser radiation.

Upon reaching the threshold temperature melting the surface region at the center of the laser spot at critical energy values $E \geq 6.5$ J/cm^2 particularly significant change in the morphology of the Ni film is observed.

During the implementation of laser recrystallization from the liquid phase in the metal film – CdTe crystal system, large crystallites of different phase compositions are released (Fig. 5a). The morphology around the edges is smoother, but microcracks and photoinduced defects appear (Fig. 5b).

The NiO film is transparent to the laser wavelength $\lambda = 1.06$ μm. A slight increase of crystallites lateral size in NiO film from ~ 180 nm to ~ 230 nm is observed after laser treatment (Fig. 6b, c). Comparison of scan profiles along 1-2 lines also shows minor changes in their vertical dimensions (Fig. 6, on the right). This indicates a weak interaction of the laser beam with the NiO film and the passage of a significant part of radiation into the depth of the CdTe crystal.

As the CdTe crystal is also transparent to $\lambda = 1.06$ μm, the laser irradiation energy absorption occurs on the inhomogeneities and defects in the crystal volume. Because of the intensive penetration of laser energy into the depth of the crystal, solid-state reactions take place in the Ni/CdTe and NiO/CdTe heterojunctions.

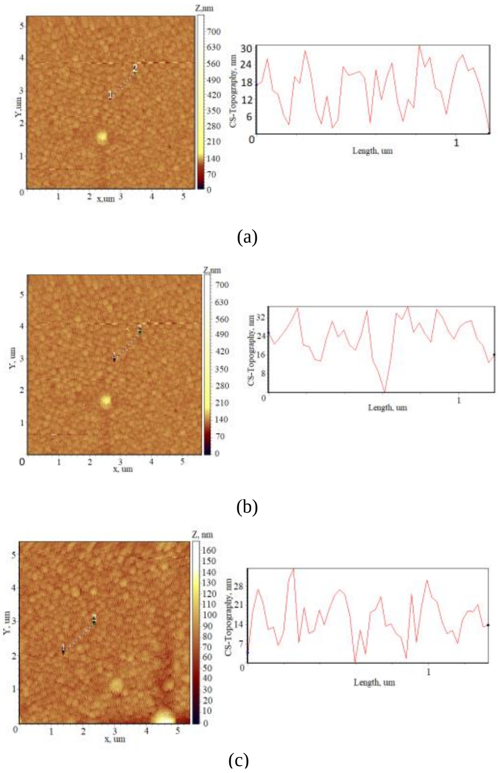

(a)

(b)

Fig. 5 – The morphology of the melted CdTe crystal after irradiation by the nanosecond laser with the energy density $E = 0.15$ J/cm^2: (a) in the center of the laser spot, (b) on the edge of the laser spot (study in SEM)

However, the area of the effect of the laser on the CdTe crystal through the NiO film extends to a much greater depth than through the Ni film. It contributes to the redistribution and relaxation of defects and better phase homogeneity.

Due to the absorption of thermal radiation in the CdTe crystal, an increased separation of donors Cl_{Te} and acceptors (V_{Cd}-Cl_{Te}) owing to the enhancement of the electron-phonon coupling occurs [14]. It can be explained by the thermogradient effect under which both Cl_{Te} and V_{Cd} drift towards the temperature gradient induced by the laser beam, i.e., towards the bulk of the irradiated CdTe crystal where they can create A-centers [15]. Taking into account the concentration of chlorine in the samples studied the probability of A-centers generation during the cooling is high.



(a)

(b)

(c)

Fig. 6 – AFM-images of NiO film on CdTe single crystal substrates: (a) before laser treatment, (b) after laser treatment with the energy density of 2.4 J/cm^2, (c) after laser treatment with the energy density of 3.6 J/cm^2. On the right – the scan profiles along lines 1-2

Thus, the concentration of the A-centers increases simultaneously with the concentration decrease of the isolated Cl_{Te} donors, which implies a reduction of the donor-acceptor pairs concentration and the corresponding increase of the mean distance between donors (Cl_{Te}) and acceptor (A-centers), and redistribution of defects and impurity-defect complexes in the temperature gradient induced by the laser beam.

The current-voltage (I-V) characteristics of the detectors were measured within a wide bias voltage range at different temperatures by a standard method with the use of a precise femto/picoammeter Keysight B2985A with a built-in source (± 1000 V) and Agilent 34410A were used as an amperemeter and voltmeter, respectively. The reverse current was measured when

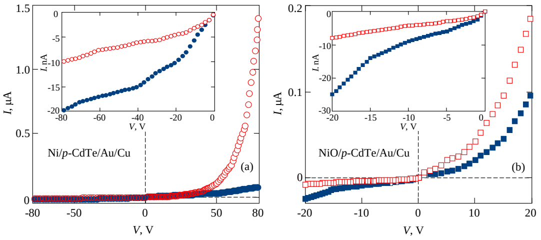


Fig. 7 – Room temperature dark *I-V* characteristic of the Ni/*p*-CdTe/Au/Cu (a) and NiO/*p*-CdTe/Au/Cu (b) heterostructures before (filled symbols) and after (blank symbols) laser treatment. The insets depict reverse *I-V* characteristics of the heterostructures

the Ni or NiO (Schottky-type) contact was biased positively with respect to the Au contact. As seen, the *I-V* characteristic of the Ni/*p*-CdTe/Au/Cu (Fig. 7a) and NiO/*p*-CdTe/Au/Cu (Fig. 7b) heterostructures show rectifying properties. The rectification significantly increased after laser irradiation of the structure. As seen, laser irradiation of the Schottky diode structure side remarkably shifted the *I-V* characteristic forward branch toward lower voltages (forward current increased) and reduced reverse current compared with the unirradiated sample (Fig. 7). From a practical point of view, it is important to note that the reverse current of the Ni/*p*-CdTe/Au/Cu and NiO/*p*-CdTe/Au/Cu detectors reduced by 2-3 times due to laser treatment of the Schottky contact standing at about 10 nA at $V = -80$ V and $V = -20$ V, respectively (Fig. 7a, b). This can be explained by an increased lifetime of charge carriers in the depleted region after laser treatment, more strongly in the NiO/*p*-CdTe/Au/Cu heterostructures than in the Ni/*p*-CdTe/Au/Cu one. Apparently, the reasoning is the fact that the NiO film is transparent to the laser wavelength used for irradiation. Therefore, the area of thermal impact during laser treatment extends deeply into the CdTe crystal and increases with the power density of the laser beam. So, the impact of laser irradiation increased the effective lifetimes of charge carriers in the depleted region, reduced the generation rate, and thus decreased the reverse dark current of Ni/*p*-CdTe/Au/Cu and NiO/*p*-CdTe/Au/Cu detectors with [13] Schottky contact.

It should be noted that not only electrical properties but the performance of the Ni/*p*-CdTe/Au/Cu and NiO/*p*-CdTe/Au/Cu detectors also has undergone changes after the laser treatment. In particular, the performance of the Ni/*p*-CdTe/Au/Cu detector before the laser treatment was so poor that the emission spectrum of ^{241}Am source was hardly detected against the background of the electrical noise and becomes clearly visible after the laser treatment. As for NiO/*p*-CdTe/Au/Cu detector, although the ^{241}Am source spectrum was visible even before the laser treatment, the height of the peak becomes three times higher after laser treatment.

4. CONCLUSIONS

The structural perfection of CdTe:Cl single crystals was estimated using high-resolution X-ray diffractometry. The analysis of HRXRD intensity distributions from symmetrical and asymmetrical reflections of CuK$_{\alpha 1}$ radiation shown that CdTe:Cl wafers are characterized by complex mosaic and defect structure. The dislocation density, obtained from the Williamson-Hall plot show that the most perfect crystal has the dislocation density of 4.8×10^5 cm^{-2}. It is shown that laser treatment using a millisecond (1.5 ms) YAG-laser with 1.064 μm wavelength both for the CdTe substrate and Schottky contact of Ni/*p*-CdTe/Au/Cu and NiO/*p*-CdTe/Au/Cu heterostructures, can intentionally decrease by 2-3 times the reverse current of the Ni/*p*-CdTe/Au/Cu and NiO/*p*-CdTe/Au/Cu detectors and improve their sensitivity to X/γ rays.

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Лазерна модифікація морфології та дефектної структури гетероструктур на основі кристалів CdTe детекторного класу

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Процеси лазерного маніпулювання дефектно-домішковою системою та лазерної трансформації морфології кристала дозволили підвищити детектуючі властивості структур на основі CdTe з бар’єром Шотткі. Методом рентгенівської дифрактометрії високої роздільної здатності оцінено структурну досконалість монокристалів CdTe:Cl. Ефективність контакту залежить як від матеріалу електрода, так і від обробки поверхні кристала CdTe перед його осадженням, крім того, характеристики сформованого інтерфейсу електрод-напівпровідник можуть бути змінені різними обробками. Опромінення поверхні кристалів CdTe або структур метал-CdTe лазерними імпульсами призводило до зміни морфології поверхні напівпровідника, утворення та перерозподілу дефектів в області поверхні та модифікації характеристик цієї області або межі розділу. Досліджено вплив лазерної обробки на структуру домішкових дефектів та електричні характеристики рентгенівських/γ-детекторів на діодах Шотткі, розроблених шляхом осадження Ni та NiO на комерційно доступні пластини CdTe:Cl. Методами атомносилової та скануючої електронної мікроскопії досліджено особливості тонких плівок Ni та NiO до та після лазерного опромінення. Обговорюється вплив імпульсного лазерного опромінення на контакти Ni/CdTe і NiO/CdTe та механізми трансформації їх фазового стану оскільки така обробка цих контактів Шотткі призвела до оптимізації електричних характеристик. Показано, що лазерна обробка гетеропереходів, як підкладки CdTe, так і плівки Ni та NiO, може навмисно змінити електричні властивості та підвищити чутливість Ni/*p*-CdTe/Au/Cu та NiO/*p*-CdTe/Au/Cu детекторів. Також обговорюється вплив лазерної обробки на структурні та спектроскопічні властивості рентгенівських/γ-детекторів на діодах Шотткі на основі CdTe.

Ключові слова: Кристал CdTe, Гетероструктура, Лазерна обробка поверхні, Діод Шотткі, Зворотний струм, Транспортування заряду, Рентгенівський детектор, Спектр випромінювання.