Formation of Doped Nano-layers in CdTe Semiconductor Crystals by Nanosecond Pulse Laser Irradiation

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Abstract

The technological procedures of fabrication of CdTe-based diodes for room temperature X/γ-ray radiation detectors have been described and analysed. In addition to the surface processing of detector-grade Cl-compensated (111) oriented CdTe single-crystal wafers, particular attention has been drawn to the developed technique of laser-induced solid-phase doping of a nano-layer of the semiconductor. High-resistivity *p*-like CdTe crystals pre-coated with a relatively thick In film were irradiated from the In side with nanosecond laser pulses. The In film served as an *n*-type dopant source as well as an electrode after laser irradiation. Laser-induced doping allowed to overcome the phenomenon of dopant self-compensation in CdTe, introduce and activate an In impurity as donors with high concentration in the near In-CdTe interface nano-region and thus to form a built-in shallow and sharp *p-n* junction. The mechanisms of nanosecond laser pulse action on the In/CdTe structure, defect formation and stimulated doping in the semiconductor were associated with the laser-induced generation and propagation of stress and shock waves, baro-diffusion of In atoms into the thin CdTe region and creation of a large number of donor centers In_{Cd}. The formed In/CdTe/Au diodes demonstrated high rectification current-voltage characteristics (*I-V*) characteristics and were suitable for the fabrication of X/γ-ray radiation detectors.

Keywords: CdTe semiconductor, Laser irradiation, Doping, Nano-layer, *p-n* junction, Diode, *I-V* characteristics, X/γ-ray detector.

1. Introduction

High-resistivity cadmium telluride (CdTe) semiconductor is a basic material for fabrication of high energy radiation direct-conversion detectors operating at room temperature and covering a wide energy range from a few keV to many MeV [1-5]. An optimal set of physical properties of CdTe makes this material attractive and still promising for compact solid-state X/γ-ray radiation detectors which are widely used in industry, medicine, security, ecology, space astronomy and other important application fields [1-3]. Among the excellent features of this semiconductor, the following key advantages should be highlighted: large atomic numbers of the compound components (Z_{Cd} = 48, Z_{Te} = 52) provide a high photon absorption efficiency, power stopping and hence large attenuation coefficient; wide band-gap energy (E_g ~ 1.5 eV) hence quite high electrical resistivity (ρ > 109 Ω•cm) and good charge transport properties allow to operate CdTe-based detectors without cryogenic cooling and reach high detection efficiency, energy and spatial resolutions and also fast timing response [1-3].

At the same time, CdTe has inherent some disadvantages, such as rather low mobility and short lifetime of holes that results in an incomplete collection of radiation-generated carriers and hence the considerable amount of charge losses because holes can be trapped

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before reaching the cathode in a CdTe-based biased detector [4-7]. If the mean drift path of charge carriers, expressed as the product of hole mobility and lifetime and applied electric field in a detector, is less than its thickness, only a fraction of the photo-generated charge carriers is induced at the electrode. This reduces energy resolution and limits capability of detectors, particularly in X/γ-ray radiation spectroscopy and imaging [1-5].

An increase in bias voltage applied to the detector can provide an extended electric field profile and wider depletion layer up to the full thickness of the CdTe crystal that increases the charge collection efficiency and hence allows obtaining higher detection effectiveness and energy resolution [4-9]. However, although CdTe has high resistivity, application of much higher bias voltage to improve the charge collection increases leakage current (dark current which flows through the biased detector in the absence of a radiation signal) which is a source of electrical noise that decreases the energy discrimination of X/γ-ray radiation and deteriorates the functional detector characteristics [2-5]. Efforts to overcome an increase in leakage current in CdTe-based detectors even at high bias voltage have been made by developing them as diode structures [2-9]. Diodes based on a built-in *p*-*n* junction are preferred over diodes with a surface barrier (Schottky diode), because it is easier to form an extended depletion layer and improve the charge collection in a *p*-*n* junction as well as to form robust electrical contacts [7-9].

In order to create a shallow and sharp *p*-*n* junction in semi-insulating *p*-like CdTe, which is commonly used for X/γ-ray radiation detector fabrication, it is necessary to form a thin highly doped layer in the surface region of semiconductor crystals. One of the most suitable and frequently used *n*-type dopants for CdTe is indium because this impurity has a shallow transition energy level and relatively low defect formation energy [10]. However, high-concentration doping of CdTe with an In impurity, realized by conventional equilibrium methods, is rather problematic because of the self-compensation phenomenon which consists in spontaneous formation of compensating opposite charged native defects or complexes of the dopant, in particular dopant-native vacancy pairs (V_{Cd}-Cl_{Te}) (so called A-centers) [3,10-12]. Self-compensation limits the efficient doping (amount of donors) in CdTe to a value considerably less than the impurity concentration [10,11].

The basic idea of the present research is to utilize indium both as a dopant source and anode electrode for *p*-type CdTe crystals using nanosecond laser irradiation. Laser processing of CdTe has been successfully used to modify the structure and properties of surface nano-layers and change different characteristics of the semiconductor [12-14]. Of particular interest is application the laser-assisted doping technique to suppress the dopant self-compensation mechanism, and reach introducing and efficient electrical activation of In atoms in CdTe [8,9,15-17]. We have continued development and optimization the methods of chemical surface processing of CdTe crystals, laser-induced doping technique for creation of a built-in *p*-*n* junction by nanosecond laser irradiation of the CdTe crystals pre-coated with a relatively thick In film which serves both as an *n*-type dopant source and electrical contact (electrode) after the doping and deposition of the second electrode. The formation of a surface nano-layer highly-doped with In in semi-insulating detector-grade *p*-like CdTe crystals and creation of In/CdTe/Au diodes with a shallow and sharp *p*-*n* junction has been studied and mechanism of laser-induced doping is analyzed.

2. Experimental Details

2.1. Pre-treatment of CdTe crystals

To meet the requirements for the material for room temperature high-energy radiation detector fabrication, we have used high-resistivity CdTe semiconductor obtained by the Traveling Heater Method (THM) which has been known as an advanced technique to grow uniform single crystals with decreased number of native point and extended defects, and accidental impurities [1-5]. High-quality detector-grade CdTe single crystal wafers grown by THM were obtained from Acrorad Corporation [4,5]. Semi-insulating Cl-compensated CdTe semiconductor showed weak *p*-type conduction with the room temperature resistivity ρ = (2-4) × 10^9 Ω·cm that was close or even higher than the intrinsic value, so the investigated CdTe can be considered as an almost intrinsic semiconductor with *p*-like type conductivity. The grown CdTe ingot was sliced into wafers and then polished by the manufacturer. Parallelepiped-like (111) oriented CdTe single-crystal samples had the area of 5 mm × 5 mm and thickness of 0.5 mm.

Before doping and electrode deposition, the investigated CdTe crystals were subjected to the preliminary surface processing for cleaning and removing a damaged and disordered thin surface layer which was commonly formed after mechanical polishing and long-time storage of the samples in air. The samples were washed in acetone and methanol, and chemically etched in a polishing 5% Br-methanol solution. Then, the samples were quickly dipped into methanol to stop the etching process and finally were thoroughly rinsed in a few beakers with pure methanol. The chemically cleaned and polished samples were stored in a beaker with methanol to prevent oxidation of the CdTe surface and were dried in an argon flow just before the application of the next technological procedures, in particular deposition of a metal film.

2.2. Laser-induced doping and electrical contact formation

The technology of the laser-induced formation of an In doped nano-layer in the surface region of CdTe crystals and fabrication of In/CdTe/Au diodes with a *p*-*n* junction shown in Fig. 1. After the preliminary surface processing of CdTe crystals, an In film was evaporated on the CdTe(111)B crystal surface (Te-terminated) in a physical vapor deposition system at low pressure (< 6 × 10^{-4} Pa) without heating of the samples (Fig. 1(a)) . However, low temperature (~350-360 K) annealing of the CdTe crystals in vacuum was applied during several hours before the In deposition to remove a thin (~0.03 nm) Te film which is usually formed on the semiconductor surface under etching in Br-containing solutions and then it is oxidized with time [14,18]. The thickness of the deposited In film was selected to provide the optimal conditions for laser-induced doping of the underlying region of the semiconductor [16]. The In film was relatively thick (~500 μm) and it was not completely evaporated even at multiple irradiation, so the film served as an *n*-type dopant source during laser action and also as an electrode after laser-induced doping.

The second electrode was formed on the opposite side of the samples in the same manner, i.e. by vacuum evaporation of an Au film (~300-400 μm) on the CdTe(111)A surface (Cd-terminated) (Fig. 1(d)). Both the electrodes were formed as squares with 4 mm sides and centered on the sample sides of 5 mm × 5 mm. The deposition velocity and thickness of the metal films were controlled by changing voltage applied to the evaporation source and using an XTC Thin Film Deposition Controller.

The whole surface area of CdTe crystals pre-coated with an In film was entirely and uniformly irradiated with nanosecond laser pulses at room temperature (Fig. 1(b)). The

pulsed radiation source was a ruby laser (λ = 694 nm), or YAG:Nd laser (second harmonic with λ = 532 nm) or KrF excimer laser (λ = 248 nm) emitting single pulses with duration (FHWM) of 20 ns, 7 ns and 20 ns, respectively. The incident laser pulse energy density was varied in a wide range below as well as above the melting threshold of CdTe [19]. The set of lens, glass diffusers and homogenizers was used for uniform and controlled irradiation of the samples. The energy and time parameters of laser pulses were monitored and fluctuations in the laser output energy were less than 5%. The laser-induced doping was carried out in different environments (air, vacuum, argon pressure or water) to investigate and select the optimal ambient conditions.

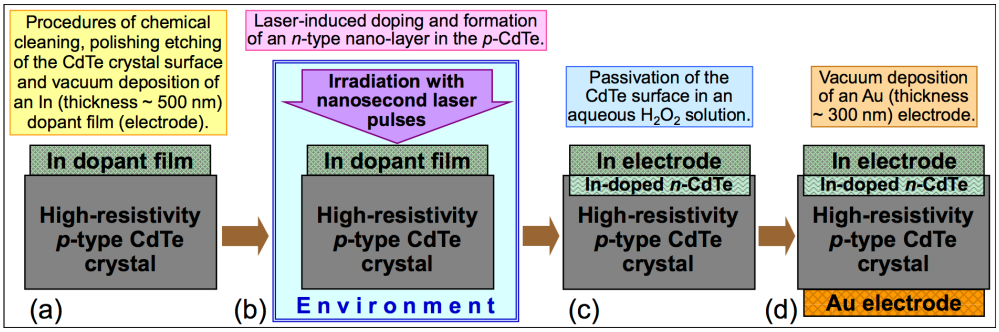

Fig. 1. Procedures of the fabrication of the In/CdTe/Au diode with a *p-n* junction: chemical surface processing of the high-resistivity *p*-like CdTe semiconductor crystal and deposition of an In dopant (electrode) film by vacuum evaporation (a), laser-induced solid-phase doping of the CdTe surface region with In atoms (donors) and formation of an n-type nano-layer (b), chemical passivation of the CdTe crystal surface (c), deposition of an Au electrode by vacuum evaporation (d).

After laser-induced doping of the surface CdTe layer and prior to the vacuum deposition of an Au electrode, the In/CdTe structures were subjected to passivation into an aqueous H_2O_2 solution for a few tens of seconds and after that, the samples were rinsed in methanol (Fig. 1(c)). The passivation was applied to minimize lateral leakage current and stabilize the electrical characteristics of the diode. Finally, the In/CdTe/Au diode structures with an In doped nano-layer and built-in *p-n* junction were obtained (Fig. 1(d)).

3. Result and Discussion

3.1. Mechanism of nanosecond laser pulse action on In/CdTe structure and stimulated doping

One of the key features of the laser-assisted technique to dope a very thin layer of *p*-like CdTe crystals with high concentration of n-type impurity is the use of a relatively thick In dopant film which was much large than the depth of radiation absorption and thermal diffusion length. Thus, laser-induced doping was provided without heating the underlying bulk In region and CdTe crystal that avoided a heat-induced change and deterioration of the semiconductor structure and properties.

The thickness of the deposited In was investigated in the range of 20-1000 nm and the value of ~400-500 nm was selected that allowed obtaining the diode In/CdTe/Au structures with high rectification [16,17]. The absorption coefficients of laser radiation (wavelengths of 694 nm, 532 nm and 248 nm) used in the experiments were α ~ 105-106 cm^{-1}, so absorption

of radiation occurred in a thin (tens of nanometers) surface layer of the In film. However, high doping was made in the under In-CdTe interface region, therefore the study of the mechanisms of laser action, modification of the defect structure and solid-phase doping in deep-seated layers of the In/CdTe sample is an important and interesting task for practical application.

Figure 2(a) illustrates the action of a laser pulse on the In/CdTe structure that was accompanied with superfast heating, melting and evaporation of a surface In nano-layer with the thickness of the order of the radiation absorption depth (~1/α). The temperature of the formed plasma, containing evaporated In and overheated environment medium, could reach thousands of Kelvins and higher at laser pulse energy densities that were used for the doping (70-150 mJ/cm^2). The rapid expansion of the laser-heated In surface layer and recoil momentum, arising from the formed plasma, resulted in the generation of high-amplitude stress waves which were transformed to the shock wave with the pressure of ~400 MPa and higher with increasing energy density or decreasing the duration of laser pulses [16,17]. Such stress and shock waves propagated through the In film and entered into the CdTe crystal involving In dopant atoms. Thus, the fast penetration of In atoms with high concentration into a thin layer of the semiconductor near the In-CdTe interface was caused by significant gradients of the elastic stresses, generation of stress and shock waves and superfast diffusion of In atoms under high pressure, i.e. baro-diffusion [16,17].

Despite the high temperature of the In surface under laser action, underlying deeper layers remained unheated. The deposited In film was much thicker than the laser-excited In surface region, therefore the temperature in the bulk of In and especially in CdTe was not increased. The use of thick In film as an dopant source under laser irradiation provided the conditions for the solid-phase doping of a thin nano-layer of the underlying CdTe crystal owing to rapid mass transfer of the impurity as result of action of stress and shock waves, transformation of the point defect structure of the semiconductor near the In-CdTe interface and baro-diffusion [16,17].

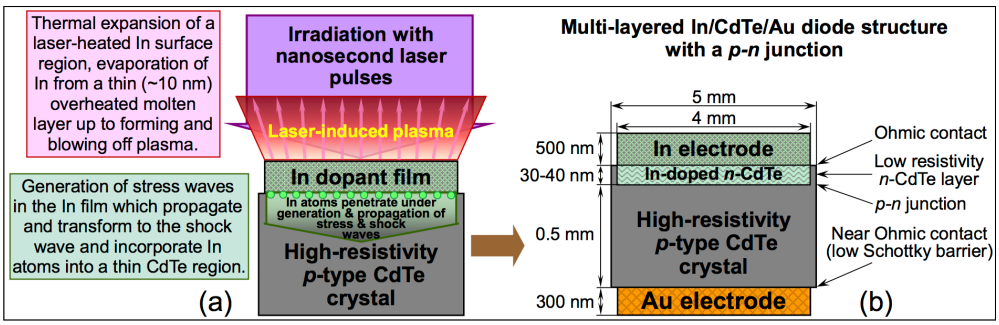

Fig. 2. Schematic illustration of the processes of laser pulsed irradiation of the In/CdTe structure from the In dopant film side and solid-phase doping of a CdTe nano-layer near the In-CdTe interface stimulated by laser-induced stress and shock waves (a), and graphic image of the multi-layered In/CdTe/Au diode structure with a built-in *p-n* junction (b).

The In/CdTe/Au diodes, created by the laser-induced solid-phase doping, represent a complex metal-semiconductor multi-layered structure consisting of the following parts: In electrode, In/n-CdTe Ohmic contact, thin low-resistivity heavily doped n-CdTe:In layer,

sharp built-in *p*-*n* junction, bulk part of semi-insolating *p*-like CdTe, Au/CdTe near Ohmic contact (low Schottky barrier) and Au electrode (Fig. 2(b)).

3.2. Laser-induced defect formation in CdTe and creation of a doped nano-layer

The atomic structure (a) of a surface layer of semi-insolating Cl-compensated CdTe crystal, transformation of its point defect structure during (b) and after only laser irradiation (c), and after laser-induced doping (d) are schematically shown in Fig. 3. High-resistivity CdTe:Cl semiconductor contains a rather large number of point defects both of intrinsic and impurity nature, in particular cadmium vacancy (V_{Cd}) and chlorine as an interstitial (Cl_i) or substitutional impurity in the tellurium site (Cl_{Te}). These defects are usually aggregated as complex defects (V_{Cd}-Cl_{Te}), (V_{Cd}-$2Cl_{Te}$) or (V_{Cd}-Cl_i) [3,11,12]. In detector grade CdTe:Cl or CdTe:In semiconductors typical complex acceptor defects called A-centers ((V_{Cd}-Cl_{Te}) and (V_{Cd}-In_{Cd})) are formed that determines *p*-type conductivity of the material (Fig. 3(a)). Moreover, spontaneous formation of compensating acceptors (V_{Cd}-In_{Cd}) is the general problem in n-type doping of CdTe with an In impurity [3,11].

As discussed above, the main reason of laser-induced modification of the CdTe structure and properties, in the case of irradiation of the CdTe crystals pre-coated with a relatively thick (much thicker than the radiation absorption depth) In film, was the action of laser-induced shock wave that could be considered as a stream of phonons which were being scattered by existing point and extended defects of the CdTe structure. Such action stimulated the dissociation of defect complexes, superfast diffusion (baro-diffusion) of an In impurity, desorption and segregation of interstitial atoms [16,17].

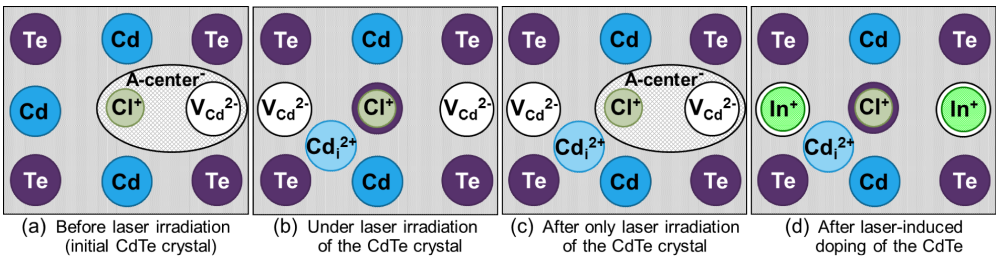

Fig. 3. Schematic diagrams of the atomic structure of the high-resistivity Cl-compensated CdTe semiconductor (a), transformation of its point defect structure under irradiation with a nanosecond laser pulse (b), after the laser processing (c) and after laser-induced doping with In atoms (d).

In the case of direct irradiation of CdTe, nanosecond laser pulse action resulted in annealing of the surface region of the semiconductor, formation of a large number of V_{Cd} and Cd_i as result of the dissociation of (V_{Cd}-X) complexes (X is a point defect) and stimulated desorption of Cd atoms from the CdTe crystal lattice (Fig. 3(b)) [12-14]. However, some amount of simple point defects formed complexes (particularly A-center (V_{Cd}-Cl_{Te}) after only laser irradiation with time (Fig. 3(c)) [12]. In the case of nanosecond laser irradiation of the In/CdTe structures, In atoms implicated by laser-induced stress and shock waves penetrated into the CdTe region near the metal-semiconductor interface [16]. Solid-phase high In concentration doping of a CdTe nano-layer occurred owing to the laser-stimulated processes of baro-diffusion and migration of In atoms at V_{Cd} and then very fast "freezing" of a large number of electrically active point defects (donors) In_{Cd}, Cd_i and Cl_{Cd}

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without formation of compensating acceptor complexes (V_{Cd}–X) in particular A-centers like (V_{Cd}–Cl_{Te}) and (V_{Cd}–In_{Cd}) (Fig. 3(d)) [15-17].

The laser-induced doping, performed by irradiation of the In/CdTe structure with nanosecond laser pulses, was the solid-phase process without heating of the bulk of CdTe crystals that avoided undesirable modification of the point defect structure and properties in the underlying layers and thus made it possible to obtain a shallow and sharp *p-n* junction in the CdTe surface region.

3.3. Electrical measurements of the In/CdTe/Au diode structures

The fabricated In/CdTe/Au diodes were exanimated by electrical measurements and samples with low leakage current were selected for high-energy radiation testing as X/γ-ray radiation detectors [8,9]. Current-voltage characteristics (I-V) were measured in dark conditions at room temperature using a microprobe I-V measurement system. The forward current was measured when the Au contact was biased positively with respect to the In contact.

Figure 4 shows the typical I-V characteristics of the In/CdTe/Au sample fabricated without laser irradiation of the In film (electrode) (a) and diodes after laser-induced doping performed by irradiation of the In/CdTe structure with nanosecond pulses of a KrF excimer laser with energy densities 88 J/cm^2 (b) and 114 J/cm^2 (c). High forward current and low reverse (leakage) current flowed in all the cases that were evidence of diode properties of the In/CdTe/Au structures. In the case of an unirradiated In/CdTe/Au sample with just deposited In and Au electrodes, the rectification properties were due to a Schottky barrier which is very general and typical for an electrical contact of In and semi-insulating CdTe [1-5].

The In/CdTe/Au diodes subjected to laser irradiation, i.e. after laser-induced doping demonstrated much higher rectification that was due to the formation of a sharp *p-n* junction in the CdTe surface region. It is clearly seen that laser irradiation of the In/CdTe structures from the In film side remarkably shifted the forward branch of the I-V characteristic to lower voltages and reduced leakage current in comparison with an unirradiated In/CdTe/Au sample (Fig. 4).

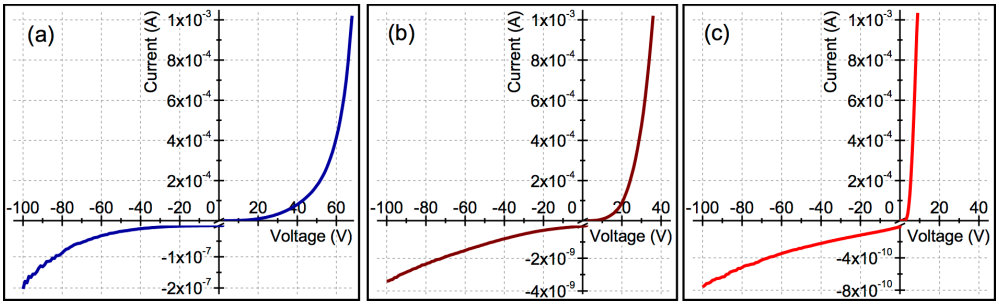

Fig. 4. *I-V* characteristics of the In/CdTe/Au diode structures before (a) and after (b, c) irradiation from the In film coated side by nanosecond pulses of a KrF excimer laser with different energy densities: 88 J/cm^2 (b) and 114 J/cm^2 (c).

A low leakage current (< 1 nA at 100 V) and steep increase in the forward biased current indicated high rectification properties of the fabricated diodes (Fig. 4(c)). This was evidence of a high barrier for the reverse current due to formation of an *n*-type nano-layer in the *p*-like

CdTe crystal as a result of laser-induced solid phase doping with In atoms and a high barrier *p-n* junction was created.

The performance of the In/CdTe/Au diodes depended on the environments (ambient conditions) used under laser-induced doping. Irradiation of the CdTe crystals pre-coated with an In film in vacuum resulted in intense and nonuniform evaporation of a surface In layer that excluded repeated or multiple irradiation of the In/CdTe structure. The same problem was in the case of irradiation of the samples in atmospheric pressure air, though lower laser pulse energy density was enough to obtain a diode structure with a *p-n* junction. To reach high doping level and good rectification in the diodes, higher laser energy densities were required. Quite suitable diode characteristics were attained when laser-induced doping was carried out in high-pressure argon environment [8]. Very high rectification, especially very high forward current at relative low leakage current was obtained for In/CdTe/Au diodes fabricated by laser-induced doping in water. The diodes showed good stability of electrical properties and high detection ability and energy resolution for X/γ-ray radiation [9]. The promising technique of laser-induced doping of CdTe and creation of In/CdTe/Au diodes with a built-in *p-n* junction has been recently developed irradiating the In/CdTe structure from the CdTe side by nanosecond laser pulses with λ = 1064 nm. CdTe is transparent for this laser wavelength and it is possible to directly irradiate the In-CdTe interface through the CdTe bulk and provide direct laser processing of the In inside the In/CdTe structure [20].

4. Conclusions

The efficient laser-assisted technique of solid-phase doping of a nano-layer in semi-insulating *p*-like CdTe crystals with an In impurity (donor) of high concentration has been developed. The use of a relative thick In dopant film provided the conditions for laser-induced generation of stress and shock waves which penetrated into the CdTe and introduced In atoms to the crystal region near the In-CdTe interface. This process was accompanied with laser-stimulated baro-diffusion and fast migration of In atoms at cadmium vacancies which were formed during the laser action. It was also important to employ the appropriate surface processing (chemical polishing etching, low temperature vacuum annealing and efficient passivation) to provide the corresponding surface states of the CdTe crystals prior the deposition of an In doping film (electrode) and Au electrode. The fabricated In/CdTe/Au diodes with a built-in shallow and sharp *p-n* junction have demonstrated high rectification *I-V* characteristics and are promising as X/γ-ray radiation detectors with high energy resolution.

Acknowledgments

This paper contains the results obtained under implementation of the following international collaborative research projects: (1) “A Sensor Network for the Localization and Identification of Radioactive Sources” (SENERA) of the NATO Science for Peace and Security Programme (SPS), Grant NUKR.SFPP 984705; (2) “Development of Methods and Techniques of Fabrication of Cd(Zn)Te Semiconductor-based X/gamma-ray Detectors with High Resolution for Security and Diagnostics Instruments” of The State Fund for Fundamental Researches of Ukraine (SFFR) and The Japan Society for the Promotion of Science (JSPS), Grant No Ф68/54-2016; (3) “Development of Cd(Zn)Te-based X- and

Gamma-ray Detectors for High-performance Medical Imaging Devices" of 2016 Cooperative Research at RIE, Shizuoka University, Grant No 2055.

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