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Optimization technology of large-size Si(Li) p-i-n structures for X-ray detectors

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In this work, the features of manufacturing and investigating the electrophysical characteristics of Si(Li) p-i-n structures for detectors with a big volume of working area were considered. The technology of manufacturing of big-area (up to 110 mm) semiconductor detector structures with thickness of sensitive area up to 10 mm, from low-resistance silicon grown by the Czochralski process and with high-resistance silicon obtained by the floating zone melting method was developed. The I-V, C-V and noise *vs* voltage dependences of detectors produced from these crystals were compared. Moreover, the methods of double-sided diffusion and drift of lithium ions were applied to these crystals. Also, the methods of providing highly uniform compensated regions of detectors were considered and methods for laying uniform effective ohmic contacts on a big area of the structure were developed.

Keywords: Diffusion of lithium, Drift of lithium ions, p-i–n structure, Si(Li) detectors.

INTRODUCTION

It is well known that silicon detectors are widely used in the detection of various particles and radiations. One of the main advantages of such detectors is low energy, high braking power, highly efficient statistics set, good radiation resistance, etc. [1-2]. Due to these characteristics, silicon detectors are the object of research and production of various laboratories around the world. The literature data analysis [3-5] shows that every presented semiconductor detector material (Si, Ge, GaAs,), and detectors from this material, have significant disadvantages. Therefore, the important problem of semiconductor detector structures is to apply nontraditional, radiation-resistant and heat-stable materials in manufacturing semiconductor detectors that have similar characteristics with silicon, as silicon is the most useable material not only for detector manufacturing, but also for all semiconductor electronics. It is necessary to provide small reverse currents without using cooling, noises and to create extended sensitive area of a large volume. Moreover, the material should have high lifetime and mobility of charge carriers, small concentration of capture centers of recombination and should withstand a high electric field for obtaining good energy resolution.

Nowadays, in the world practice, detectors of relatively small size are sufficiently developed [6,7]. Simultaneously, there is an urgent need for development of semiconductor detectors of large sizes [8-10]. However, their development has their own physical, technical and technological peculiarities and difficulties. They are associated with the manifestation of the effects caused by parameters of initial crystals of big diameter and by manufacturing on their basis effective detectors of nuclear radiation. The technology of manufacturing semiconductor detectors of nuclear radiation is sufficiently complicated, and consists of mechanical, chemical and thermal operations, also it needs structural decorations. Every operation has its own purpose and needs certain control. Necessity of manufacturing of detectors of nuclear radiation with reproducible parameters and preserving the characteristics for a sufficiently long time determined the technology of manufacturing semiconductor detectors for detection of nuclear radiation. The most tested industrial detector materials of big-diameter silicon have significant heterogeneity of distribution of the electrical parameters in the crystal volume. Local and impurity strips existing in the sensitive volume of semiconductor detectors significantly impair their radiometric characteristics.

MATERIALS, TECHNOLOGY AND METHODS

Accuracy of compensation of initial semiconductor material is the most important characteristic of quality of Si(Li) p-i-n structures. However, despite the anomalously high mobility of lithium ions in silicon, they need long time for diffusion – drift compensation in big volumes (thickness ~ 10 mm, surface ≈ 110 mm). In order to significantly reduce the compensation process time of silicon in big volumes and to eliminate the

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negative effects of prolonged withstanding of crystal under high temperature and voltage, we developed the methods of creating Si(Li) p-i-n+ structures [11]. Reducing compensation time helps to minimize inhomogeneities in the detector crystal and improves the energy resolution of the detector.

For detector fabrication two types of silicon crystals were used. The first one was the dislocation-free monocrystalline silicon of p-type grown in an argon atmosphere by the Czochralski process, with diameter of 110 mm, high resistivity ρ = 10÷12 Ohm.cm and a lifetime τ ≥ 50 μs, chosen as an initial material. The concentration of oxygen was $N_o = 2{\bullet}10^{17}$ cm^{-3}. Second was the high-resistance silicon crystal obtained by the float-zone method, with a high resistivity ρ=1000÷5000 Ohm.cm and a lifetime τ ≥ 500μs.

Furthermore, by using these crystals, a p-i-n structure was created. The process of manufacturing p-i-n structures consists of the following stages:

- To remove damaged layer during the cutting, double sided grinding on a grinding machine with micropowders M-14, M-5 was used with a consequent reduction in the abrasive diameter. Wherein, at least 50 μm thick layer was removed from each side. After grinding, the plates were flushed with de-ionized water containing alkaline-free soap.
- The initial structure was obtained by diffusion of lithium. Lithium diffusion was carried out from two sides in a vacuum p ~ 10^{-5} mm Hg at a depth of 300 μm on the entire surface of the plate for t = 3 min at a temperature of 450°C. Detailed description of physical procedure of double sided diffusions of lithium atoms into silicon was shown in a work [12].
- Etching in a polishing etchant (1:3:1 mixture of $HF:HNO_3:CH_2COOH_2$) and in an aniline etchant.
- Drift of lithium ions in the electric field of the p-n junction should be carried out at a temperature of (70-80) ^{0}C and a reverse bias voltage of 100 ÷ 400 V during 10-20 days, depending on the thickness of the sensitive region.
- After finishing drift process, to detect the i-region, one of the sides of the crystal of the n + -region of n + -i-p structures was grinded on a glass disk with silicon carbide micropowder. The thickness of the layer to be removed was assessed taking into account the diffusion profile blurring. The thickness of the grinded layer ws usually 50 ÷ 400 μm. Reduction of the i-region was performed using a decorating etchant HNO_3:HF=1:1000. The i-region was considered completely obtained when its contours were close to a circle with a diameter equal to the diameter of the diffusion region. To obtain ohmic metal contacts, gold and aluminum coatings were used (Fig. 1).

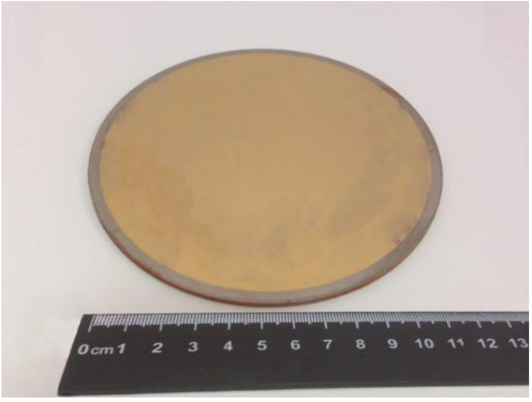

Fig. 1. General view of Si(Li) p-i-n detector structure

For the method of double-sided diffusion and drift of lithium ions it is possible to estimate the compensated area depth by using the well known equation proposed by Pell (1960)[13]:

$$W = 0.5\,(\rho U)^{1/2} \quad (1)$$

where ρ is resistivity in Ohm.cm and U – drift voltage.

The rate of growth of the compensated area depth is defined as:

$$R = \frac{dW}{dt} = \mu E = \mu U/W \quad (2)$$

or $$W = \sqrt{2\mu U t}, \quad (3)$$

where: μ - mobility of lithium ions, t – drift time.

In the case of bilateral drift the path traveled by the lithium ions is reduced by half.

$$\frac{W}{2} = \sqrt{2\mu U t} \quad (4)$$

Consequently, the expression for the time of compensation in the case of double sided drift is:

$$\sqrt{\frac{t_1}{t_2}} = 2 \qquad t_2 = \frac{1}{4} t_1 \quad (5)$$

The result shows, that in the proposed method the compensation time of specified volume of p-Si crystal is reduced 4 times. This is obvious from this empirical expression for the time dependence of compensated area depth in case of unilateral drift.

RESULTS AND DISCUSSION

The variation of characteristics of lithium distribution depending on the initial material parameters and basically on dislocation density and specific resistance in the Si(Li) p-i-n area has drawn particular interest. It is widely believed that first, at the process of drift all inhomogeneities of the initial material are corrected and the specific resistance of the compensated area is equal to the specific resistance of the initial material. Second, electric field distribution in the compensated area is almost uniform.

Consequently, during the drift process, the intrinsic conduction corresponding to the drift temperature is almost achieved. The difference between resistance of compensated area and initial material resistance is the result of influence of drift process of mobile thermal generated carriers. The temperature, necessary to exploit semiconductor detectors, is usually much lower than the drift temperature, consequently, under working conditions the quantity of thermally generated vapor is reduced because the equilibrium of mobile carriers, donor and acceptor impurities is violated. As a result, the basic area of Si(Li) p-i-n structure can be highly overcompensated with a notable excessive impurity gradient. Obviously, this influence will appear intensively on low-resistance materials. The effect can be reduced by using additional drift process under lower temperature, wherein a weakening of fixed spatial charge occurs that allows the lithium ions to be redistributed. Under uniform compensation conditions, the applied voltage of reversed bias leads to a static electric field at every point of the compensated region – it is the ideal state in terms of carrier formation. With the increase in specific resistivity of initial silicon the ratio of thermally generated carriers and donor and acceptor impurities varies, also the specific resistivity of compensated region increases.

During the work of semiconductor detectors, the values of current and capacitance play an important role when reverse bias voltage is applied. For the big-size semiconductor detectors the flatness of p-n junctions of the entire area of its sensitive surface is of great importance. The direct determination of the current-voltage characteristics (CVC) gives useful information. The CVC during application of reverse bias voltage to Si(Li) p-i-n structure were investigated. On Fig. 2 the typical CVC of Si (Li) p-i-n structures are shown, made with low-resistance silicon, grown by the Czochralski process (1) and with high-resistance silicon obtained by the float- zone method (2). It is obvious from the figure that the low-resistance silicon grown by the Czochralski process (1) has the advantages of manufacturing lithium drift detectors with a big volume, small reversed current and high exploitation characteristics. On a par with values of reversed current, the good characteristic of the structure is high breakdown voltage, indicating that the surface of the structure is sufficiently clean.

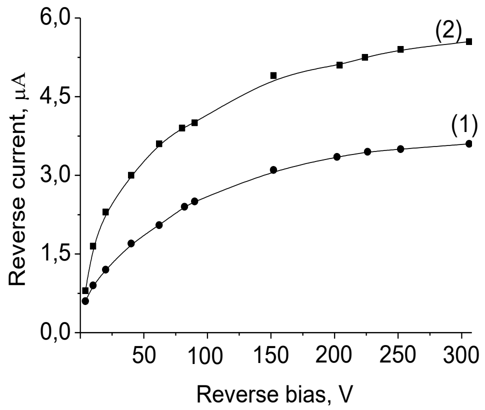

Fig. 2. The reverse branch of CVC of Si(Li) p-i-n structure grown by the Czochralski process (1) and by the float-zone method (2).

The capacitance of Si(Li) p-i-n structure is directly connected with thickness of the depletion layer and with a specific resistance of initial material. Therefore, by measuring this, it is possible to identify the specific resistance of compensated area of silicon in the prepared structure and predict the values of maximum energy of charged particle, under the conditions of its totally absorption in the depletion layer. The same samples, which were taken to investigate the CVC, were taken to study farad – voltage characteristics (FVC). On Fig. 3, the FVC of Si(Li) p-i-n structure are shown, made with low-resistance silicon, grown by the Czochralski process (1) and with high-resistance silicon obtained by float-zone method (2).

One of the main exploitation characteristics of semiconductor detectors of nuclear radiation is the energetic equivalent of noise value. This parameter determines the radiometric abilities and efficiency of detection.

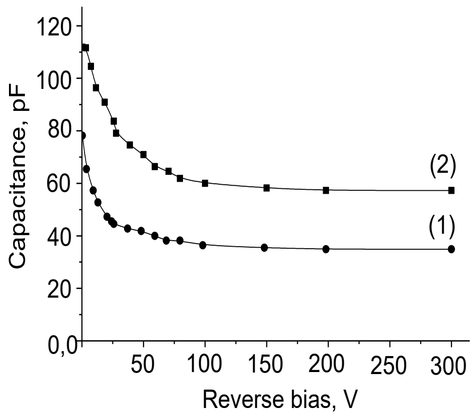

Fig. 3. FVC of Si(Li) p-i-n structure grown by the Czochralski process (1) and by the float- zone method (2).

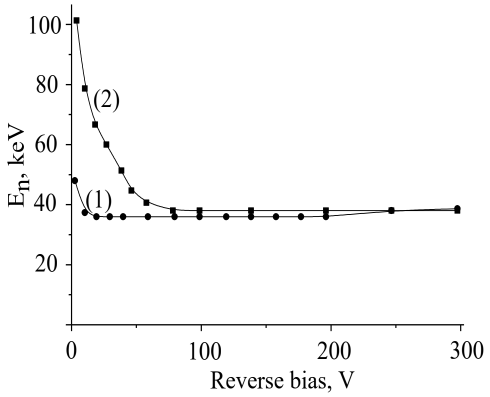

Fig. 4. Volt-noise characteristics of Si(Li) p-i-n detectors made of silicon, grown by the Czochralski process (1) and by the float-zone method (2).

On Fig. 4, curve (1) corresponds to the initial crystal with high resistivity - ρ = 10÷12 Ohm•cm grown by the Czochralski process, and curve (2) corresponds to the crystal with high resistivity ρ = 5000 Ohm•cm grown by the float- zone method.

On Fig. 5, the investigation of amplitude spectra of β-particles from the ^{207}Bi source is illustrated. The energy resolution of detectors for β-particles from the ^{207}Bi source is 1 MeV, R_β= 38 keV taken at T = 300 ^{0}C.

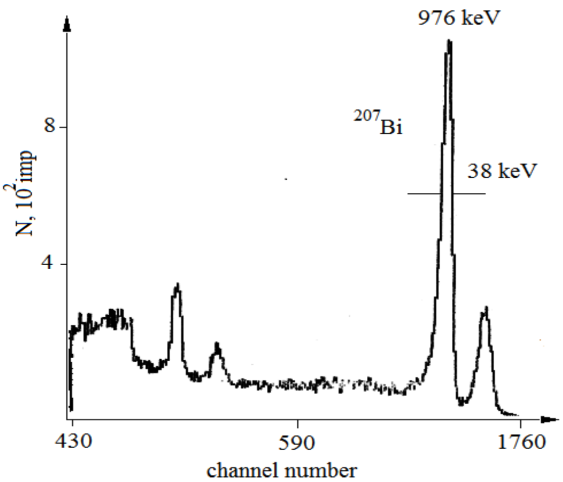

Fig. 5. The energy resolution of the detector for β-particles from ^{207}Bi.

CONCLUSIONS

In conclusion, it should be noted that all results of this work have scientific and practical value and can be used in broad areas of science and technology. In particular, the technology of manufacturing big-area (up to 110 mm) semiconductor detector structures with thickness of sensitive area up to 10 mm from low-resistance silicon grown by the Czochralski process and with high-resistance silicon obtained by the floating zone melting method was developed. From the CVC it is obvious that the reverse current in the crystals grown by the Czochralski process is lower than in the crystals grown by the float-zone method.

The method of double-sided diffusion and drift of lithium ions was proposed. It was shown that the proposed method of compensation of predetermined crystal volume reduces the time of drift 4 times and notably improves compensation quality. This is due to the suppression of the influence of free carriers generated at the drift temperature. The method of double-sided diffusion and drift of lithium ions can optimize the manufacturing process and improve electro-physical and radiometrical characteristics of Si(Li) p-i-n detectors.

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