PREPARATION AND INVESTIGATION OF THICK Si(Li) *p-i-n* GAMMA DETECTORS*

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Si detectors of about 3.2 cm³ volume and 10 mm compensated layer thickness were prepared by Li ion-drift process. The pulse amplitude spectrum of the detectors was measured in the temperature range -50 to +5 °C by irradiating the detectors with 323, 662, 1170 and 1330 keV energy of Cr⁵¹, Cs¹³⁷ and Co⁶⁰ γ radiating sources. Peaks characteristic for the energy of γ radiation did not appear in the spectra, as the γ radiation of the energy range investigated is absorbed by several subsequent Compton interactions in the Si. The position of the decay sections of the spectra obtained is characteristic for the energy of radiation. The absolute counting yield of the detectors for Co⁶⁰ γ radiation was found to be 4.8 and 4.5%, respectively. Storing the detectors at room temperature under reverse bias for several months, practically no changes in the parameters of the detectors could be found. The shape of the pulses produced by irradiation of the detectors in the temperature range -65 to -30 °C was also investigated.

Introduction

Si detectors prepared with the Li ion-drift method are extensively used for investigation of nuclear radiations, especially for measuring the energy spectra of α and β radiation. For investigating γ radiation, mainly Ge(Li) detectors are used, as the absorption coefficient of Ge for γ radiation is comparatively high, and so the detecting yield of the Ge detectors of big volume is high. These detectors are operated at liquid nitrogen temperature; at such low temperatures the reverse current of the detectors is very low, therefore the noise of the detectors is small, too, which permits very good energy resolution. In practice, however, a serious disadvantage of Ge(Li) detectors is the circumstance that they cannot be operated and stored above this temperature, as repeated heating to room temperature, even for short periods, results in deterioration of the detectors. (The recently developed intrinsic Ge detectors are free from this disadvantage, but also they can be used only at very low temperatures).

On the other hand, Si(Li) detectors with thick compensated layer (5 to 10 mm) can be stored at room temperature for unrestricted time, without observable deteriora-

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tion of their parameters [1-4]. The absorption coefficient for γ radiation of Si is comparatively low because its low atomic number; so the yield of a Si detector is less that of a Ge detector of the same volume. The absorption coefficient rapidly decreases with increasing energy of the γ radiation and therefore Si detectors detecting radiation of higher energies (above 300 keV) must have a great volume. With the method applied, we tried to prepare Si detectors which can be used for detection of radiation of higher γ energies with acceptable yield at temperatures not much lower than room temperature. The requirement of great volume was fulfilled by preparing detectors of 3.2 cm³ in volume with 10 mm compensated layer thickness. Detectors of great volume show, on the other hand, a high reverse current consisting of two components:

- i) the thermal generation current proportional to the thickness of the compensated layer and;
- ii) the leakage current proportional to the area of the detector.

Both components show a strong increase with increasing temperature of the detector. Owing to the high reverse current of the detectors of great volume, the energy resolution is limited by the detector noise [5]. In the investigated case of energies > 300 keVthe radiation is absorbed in the Si by Compton interactions, therefore there are no photopeaks in the signal spectrum of the detectors (so we cannot speak of energy resolution in the usual sense), and the functioning of the detectors, especially the maximum operating temperature, is limited by the detector noise. In preparing detectors, we aimed at reaching low leakage currents, without using, however, a guard ring [6] which would drastically reduce the leakage current. We did not extend the investigation to energy resolution in the region of lower γ energies, where photopeaks should appear, as for obtaining notable energy resolution, the detectors should have been cooled to lower temperatures than used in our experiments.

Preparation of the detectors

The Si(Li) detectors were prepared from a *p*-type Si single crystal of 1000— 1100 Ω cm resistivity, 7000—9000 cm⁻² dislocation density, and 1100—1300 µsec lifetime of the minority carriers. The diameter of the cylindrical ingot was about 22 mm, the orientation of its length axis was (111). As last step in the mechanical processing of the four cylindrical samples cut from the ingot, their surfaces were mirror-polished with diamond paste of 5 µ grain size on teflon foil. After carefully degreasing and washing the samples, the preparation of the detectors was continued with the steps described below.

The final diameter of all detectors was about 20 mm, the thickness of detector No. 1, 2 and 3 was 10 mm, that of detector No. 4 was 5 mm. Beside these detectors, rectangular samples of $4 \text{ mm} \times 15 \text{ mm} \times 1 \text{ mm}$ size were cut from the ingot. On these samples all steps of the detector preparation were performed together with the cylindrical samples, or at least under the same conditions, to study processes making necessary the damaging of the samples during the investigation.

In preparing the detectors, the methods described in [1] were used with small modifications.

1. Preparation of the n^+ contact by phosphorus diffusion. One of the contacts was prepared with phosphorus diffusion. The samples were etched in CP4A at

20 °C for 3 minutes. The etching was finished by continuous dilution and washing with de-ionized water (of 2 M Ω resistivity) with special care for not exposing the samples to contact with air before totally removing the etchant. Then the samples were dried in a stream of pure nitrogen gas. Subsequent etchings, except the stabilizing etching, were performed with the above method. Phosphorus diffusion was made in a quartz furnace of two temperature zones with the carrier gas method, using P_2O_5 vapour at 1025 °C temperature for 2 hours [7, 8]. In the lower temperature zone of the furnace, the $P_{9}O_{5}$ of analytical purity was sublimated and carried to the 1025 °C temperature zone by an Ar stream of 99.99% purity. The conditions and the quantity of P_2O_5 were adjusted in a way to keep the phosphorus concentration constant in the neighbourhood of the samples during the period of diffusion. The rate of heating the samples to 1025 °C and cooling down to room temperature was 5 °C min⁻¹. According to [7, 8], diffusion at 1025 °C for 2 hours results in a diffusion depth of 2 μ , the concentration of the phosporus atoms in the surface laver being 10²¹ cm⁻³. After removing with HF the phosphate glass covering the samples, the phosphorus concentration was determined by measuring the surface conductivity with a four probe method, and the diffusion depth by staining [9] after lapping the samples, diffused together with the detectors, at an angle of 5°. The values determined in this way were in good accordance with those found in the quoted literature.

After masking one of the base surfaces of the sample with picein, the phosphorusdiffusion layer was removed from the free surfaces by two subsequent etchings of 3 minutes, then the masking picein was carefully removed by dissolving in trichlorethylene and carbon tetrachloride.

2. Preparation of p^+ contacts by aluminium alloying. The samples were deoxidized with HF, washed and dried, then 200 µg cm⁻² Al of 99.99% purity was evaporated in 2×10^{-6} torr vacuum onto the base surface opposite to the n^+ contact. During the evaporation, the samples were covered with a mica plate provided with a circular aperture, permitting Al evaporation only to the surface wanted. Before evaporation, the Al was carefully degreased and etched. The evaporated Al layer on the samples was alloyed with the Si by heat treatment of 10 minutes in a furnace of 675 °C in dry inert atmosphere. The rate of heating and cooling the samples was $5 ^{\circ}$ C min⁻¹ in this case again.

3. Evaporation and diffusion of lithium. After repeated deoxidation with HF, lithium essentially exceeding the quantity necessary for compensation was evaporated onto the phosphorus diffused base plane in 2×10^{-6} torr vacuum. During the evaporation, the samples were covered by a mica mask with a circular aperture, as described above. The heating of the samples was started in the vacuum before beginning the evaporation, then after the evaporation of Li, the samples were held at 320 °C in the vacuum for 12 minutes before cooling. The Li diffusion took place during the heat treatment. Excess Li was removed from the samples by washing in ethanol, then, after masking the contacts with picein, the free surfaces were etched. After removing the picein the samples were boiled in carbon tetrachloride for 5 minutes, then stabilized in air for 24 hours. After stabilization, the reverse current of each detector at 120 V bias, measured at 20 °C was less than 10 μ A cm⁻².

Rectangular samples of 1 mm thickness evaporated and diffused together with the detectors were lapped at an angle of 5° and the depth of Li diffusion was determined with the staining method [9, 10]. A diffusion depth of 0.11-0.14 mm was

obtained in accordance with [11]. The Li concentration of the surface layer was estimated to about 8×10^{17} cm⁻³.

4. Lithium ion-drift. The compensation by Li of the detectors No. 1, 2 and 3 with 200 V reverse bias at 115 °C temperature lasted nearly 3 months. In the first hours of the drift, lower voltage was applied, which gradually increased to 200 V. The detector No. 4 was drifted in a similar way, but the drifting period was about 4 times shorter. Cooling of the detectors during the drifting process was performed by filtered and purified air stream. The drifting apparatus was secured against thermal runaway. For time to time the drifting process was interrupted, the detectors cooled to room temperature, and the drifted depth checked by capacity measurement.



Fig. 1. Capacity vs. reverse bias characteristic of the detector No. 2 before the drift process

The increase in thickness of the compensated layer with increasing drifting time was in good accordance with the values determined from the nomograms published in [1]. The compensation process performed at 115 °C was followed by a flattening



Fig. 2. Capacity vs. reverse bias characteristic of the detector No. 2 after the drift process

drift at 80 °C with 200 V reverse bias for one week; the aim of the latter was smoothing of the drift profile.

In Figs. 1 and 2 the capacity vs. reverse bias characteristics of the detector No. 2 are shown before and after the Li ion-drift, respectively. While the capacity before drift is very high and strongly voltage dependent, the capacity after drift is small and practically independent of voltage above 10 V; the measured capacity was equal to the geometrical capacity (~3.2 pF) of the detector to a good approximation; this shows that the detector was compensated in its full thickness of 10 mm. The C—U curves of detector No. 1 and 3 were practically the same as those shown in Figs. 1 and 2.

5. Stabilization of the detector. After the drift the detectors were subjected to the stabilizing etching described in [5] before measuring the C-U curve shown in Fig. 2. This process resulted in a stable surface; minimizing of the leakage current of the detectors and of their capacity by gas cycling was superfluous; the process proved to be well reproducible; repeated stabilizing etching always restored the

original I—U characteristics. It is worth mentioning that the leakage current minimized with the above method did not show observable increase even after repeatedly cooling down the detectors to -50 °C and warming up again to room temperature.

We tried also to wash the ready detectors with hot de-ionized water and to boil them in de-ionized water according to [5, 12]. The original reverse current of $10 \,\mu A \, cm^{-2}$ of the 1 mm thick detectors compensated in their whole thickness measured at 120 V at 20 °C temperature decreased to 1 µA cm⁻² 24 hours after boiling in de-ionized water for about 15 minutse. The reverse current of thicker detectors (5-10 mm) increased, however, by this treatment and the original value before treatment could only be restored by the stabilizing etching mentioned above.

6. Encapsulation of the detectors.



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Fig. 3. Diagram of the detector capsule: a) output connection of the detector; b) spring and electric contact; c) upper part of the Al capsule; d) silicon rubber seal; e) spring of the distance ring; f) tefion distance ring; g) 0.5 mm thick side-wall of the Al capsule; n^+ and p^+ denote the contacts of the detector

The ready detectors were placed in an airtight Al capsule in pure nitrogen atmosphere (Fig. 3). The thickness of the side wall of the capsule was 0.2 mm. This thin wall did not practically absorb the radiation investigated.

Testing of the detectors

The current vs. reverse bias characteristics of the detectors No. 2 and 4 measured at 25 °C are presented in Fig. 4. The very rapid increase of the current in the range 0 to 10 V is noticable. (A curve of similar character can be seen *e.g.* in [13].) At voltages exceeding 40 V the increase of both curves was nearly linear. Fig. 4 shows our general experience that in the case of detectors of identical diameter but of different thickness, prepared from the same ingot, it was always the current of the thicker detectors which was higher and these higher currents generally increased more steeply at higher voltages. This phenomenon can be accounted for only partly by the higher generation current of thicker detectors. While the breakdown of the detectors before compensation at room temperature generally began at 180—250 V reverse bias, depending on the detector, in the case of detectors with 10 mm depletion layer no sign of breakdown was observed up to 1100 V bias tested. It can be supposed that the breakdown occurs at considerable higher voltages.

In Fig. 5 the current vs. reverse bias characteristic of detector No. 1 is shown at different temperatures. It can be seen from the figure that the current of the detector



Fig. 4. Reverse current vs. voltage characteristics of detectors No. 2 and 4

strongly decreases with decreasing temperature, but at the same time below -10 °C the I—U curves become considerably steeper. The I—U curves of the detector No. 2 were in good accordance with those shown in Fig. 5, while the curves of detector No. 3 were somewhat different.

In order to study the shape of the pulses produced by γ photon absorption, the encapsulated detector was put in a thermostat which could be regulated between -10 to -70 °C. A lead plate of 20 mm thickness was placed immediately beside the detector, with the γ source of 1 µC activity behind it. The γ photons penetrated into the detector through a hole of 0.5 mm diameter in the lead plate and through the wall of the capsule, parallel to the plane of the detector contacts. A coaxial cable of low capacity served to connect the detector with the charge sensitive preamplifier which did not contain integrating and differentiating elements. The output of the latter was connected with the input of a high frequency oscilloscope. The pulses were observed and/or photographed on the screen of the oscilloscope. The





value of the load resistor of the detector was 50 MΩ. The 100 V reverse bias of the detector was supplied by an anode battery. The pulses obtained with the detector No. 2 by the γ radiation of 662 keV energy of a Cs¹³⁷ source at -65 °C detector temperature are shown in Fig. 6*a*, those obtained with the γ radiation of 1170 and 1330 keV energy of a Co⁶⁰ source in Fig. 6*b*. The smallest scale divisions in both figures mean 100 µsec and 10 mV on the horizontal and vertical axis, respectively. The number of the pulses in Fig. 6*a* is higher than in Fig. 6*b*, due to the fact that the absorption coefficient of Si for γ radiation of lower energies is higher than for radiation of higher energies. (Both sources were of nearly the same activity and the experimental conditions were identical in both measurements.) In Fig. 6b there are also higher pulse amplitudes than in Fig. 6*a* this can be attributed to the higher energy of the radiation.



Fig. 6a. The pulses of the detector No. 2 due to gamma radiation of a Cs^{137} source, obtained at -65 °C detector temperature



Fig. 6b. The pulses of the detector No. 2 due to gamma radiation of a Co⁶⁰ source, obtained at -65 °C detector temperature

By studying the pulses of detectors with higher time resolution, it was possible to determine the carrier collection time of the detectors, which resulted to be about 0.5 µsec at 100 V detector voltage near -60 °C detector temperature; this is near to the value calculated from the carrier mobility, the thickness of the detector, and from the field strength in the detector.

In Figs. 7*a*, 7*b* and 7*c* the pulses of the detector No. 2 due to γ radiation of a Cs¹³⁷ source, obtained at detector temperatures of -65, -50 and -30 °C, respectively, can be seen. This bias of the detector was 100 V, the value of the load resistor 50 MΩ, the smallest scale division on the horizontal axis 100 µsec, on the vertical axis 4 mV in all three cases. The figures show a strong increase of the detector noise with increasing temperature.

Performing the same investigations with the detectors No. 1 and 3, it was found that the rise and decay times of pulses of detector No. 1 due to γ radiation, as well

as the mean value of the amplitude maxima determined from numerous measurements were practically the same as obtained with detector No. 2 under identical conditions. In the case of detector No. 3 the main pulse amplitude proved to be about 20% less, while the rise time of the pulses was about 15% higher. This deviation of the parameters of detector No. 3 was attributed to the lower lifetime and mobility of the carriers.

The pulse amplitude spectra of the detectors were also measured with y radiation of Cr⁵¹. Cs137 and Co60 at different detector temperatures, detector voltages and load resistors. The block diagram of the measuring apparatus* is shown in Fig. 8. The detectors were connected with the preamplifier Tannelec type TC-135 by a cable of low capacity. The load resistor was varied between 1 and 47 M Ω . The signals obtained from the preamplifier were further amplified to the level necessary for the analyser by a main amplifier Canberra type 1417 B provided with an active filter (integrator and differentiator). The pulses were analysed with an amplitude analyser of 1024 channels, type ND-2200 of Nuclear Data. The information stored in the analyser was recorded on a tape, the spectra were plotted and the measurements evaluated. The correct functioning of the measuring system was checked by a pulse generator BNC type GL-3 connected immediately with the preamplifier.



Fig. 7a. The pulses of the detector No. 2 irradiated by gamma radiation of a Cs¹³⁷ source at -65 °C temperature



Fig. 7b. The pulses of the detector No. 2 irradiated by gemma radiation of a Cs¹³⁷ source at -50 °C temperature



Fig. 7c. The pulses of the detector No. 2 irradiated by gamma radiation of a Cs¹³⁷ source at -30 °C temperature

* These measurements were made in the Hungarian Bureau of Measurements

The method of measurement was as follows:

- i) the noise spectrum of the detector during a period t_m was recorded;
- ii) the pulse spectrum of the detector was recorded with γ irradiation for the time t_m again;
- iii) the noise counts were subtracted from the counts due to irradiation of the respective channels, and the spectra were plotted from these differences.



Fig. 8. Block diagram of the measuring apparatus.



Fig. 9 shows the pulse amplitude spectrum of detector No. 2 produced by 662 keV γ irradiation of a Cs¹³⁷ source. The temperature of the detector was $T_d = -50$ °C, the bias $U_d = 100$ V, the value of the load resistor $R_L = 10$ MΩ, the time constant of the integrator and differentiator in the main amplifier (denoted in the following shortly as time constant) $\tau = 1$ µsec, and the period of measurement

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 $t_m = 100$ sec. The numbers of the channels are indicated on the horizontal axis, the counts per channel on the vertical axis of the figure. The sharp peak characteristic for the energy of the radiation does not appear in the pulse spectrum, because the radiation of energy > 100 keV is absorbed mainly by Compton interaction with



Si [14]. In the spectrum shown in Fig. 9, four regions may be distinguished on the basis of the counts per channel with increasing channel number (or pulse amplitude): In region I (channel numbers from 0-49) a sharp increase is seen;

In region II (channel numbers from 49-178) a rapid decrease, gradually slowing down;

In region III (channel numbers from 178—272) the counts are nearly constant; In region IV (channel numbers higher than 272) the counts decrease again, approaching 0 at the end of the region.

In Fig. 10 the pulse amplitude spectrum of detector No. 2 irradiated by 323 keV γ rays of a Cr⁵¹ source is presented. The experimental conditions in the case of Figs. 9 and 10 were identical. The main difference between the curves of both figures is to be found in the fact that regions II, III and IV of Fig. 10 are shorter and the counts

11

reach the 0 level at a lower channel numbers, which is clearly due to the lower energy of the radiation.

Figs. 11, 12 and 13 show the counts per channel vs. channel number curves of the detector No. 1 irradiated under identical conditions by a Co⁶⁰, Cs¹³⁷ and Cr⁵¹



source, respectively. The parameters $T_d = -30$ °C, $U_d = 500$ V, $R_L = 10$ M Ω , $\tau = 1$ µsec, $t_m = 200$ sec were used in all three cases. The noise spectrum, or its part corresponding to higher channel numbers is also plotted in all these figures. The detector noise, compared with that for -50 °C, markedly increased (see Figs. 9 and 10). The spectral range IV, characteristic for the energy of radiation is influenced in a less degree in Figs. 11 and 12, while in Fig. 13 this influence is significant. From this it can be concluded that the detectors can be used at the higher temperatures, the higher the energy of the γ radiation.

Similarly as it was observed for the spectra shown in Figs. 9 and 10, the decay section IV of the pulse amplitude spectra gradually shifts towards higher channel numbers (pulse amplitudes) with increasing energy of the γ radiation; therefore the energy can be estimated from the position of the decay region of the spectra. The

decay is decidedly more sharp in Fig. 12 than in Fig. 11; this can be attributed to the circumstance that the radiation of Cs¹³⁷ is monochromatic, while that of Co⁶⁰ is not.

The pulse amplitude spectra of detector No. 2 irradiated with a Co⁶⁰ source at $+5 \,^{\circ}$ C and $-50 \,^{\circ}$ C detector temperature, respectively, are presented in Figs. 14





The absolute counting yield η of the detectors

$$\left(\eta = \frac{\text{total counts}}{\text{number of the incident photons}}\right)$$

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was determined at -30 °C and with a time constant of 1 µsec using a Co⁶⁰ source of 17.21 µC activity placed at a distance of 25 cm from the detector. Knowing the geometry of the detector, as well as the distance and the activity of the γ source, the number of the γ photons incident on the detector was determined supposing the formation of two photons per radioactive decay. The differences of the counts obtained during the period of measurement and the noise counts determined for



the same time were used as total counts. η was found to be 4.8 and 4.5%, respectively, for the detectors No. 1 and 2. The absolute counting yield showed practically no changes on varying the detector bias between 150 and 500 V and the values of load resistor between 1 and 47 M Ω .

In order to determine the optimum time constant of the integrator and differentiator in the main amplifier in connection with the detector used, the detector



No. 2 was irradiated by a Co⁶⁰ γ source and the time constant was set to 0.25, 0.5, 1, 2 and 4 µsec; the number of the total counts in 200 sec at 150 V detector bias was determined with each time constant (Fig. 16). The optimum time constant at -30 °C temperature resulted to be 0.5 µsec. As the optimum time constant depends also on the detector temperature through the carrier mobility, and the counts varied only slightly with the time constant, the time constant $\tau = 1$ µsec was used for all spectral measurements described in this paper.



Fig. 16. Total counts vs. time constant caractehristic of the detector No. 2

Discussion

Irradiating the detectors by γ rays between 323 and 1330 keV energy, no sharp peak characteristic for the energy of radiation appeared in the spectra. This can be attributed to the fact that radiation of the energy range investigated is absorbed by several subsequent Compton interactions in the Si. The first Compton interaction generates a hot electron and a γ photon of less energy than the incident one. The hot electron is slowed down in the detector by generating hole-electron pairs; the secondary photon either interacts repeatedly with the crystal, or leaves the detector without further interaction. The signal of maximum amplitude is produced if the γ photon loses its total energy in the detector by repeated Compton interactions or photoeffects. Beside the effects mentioned, also the detector noise plays a role in determining the shape of the pulse amplitude spectrum. Therefore the region IV pertaining to higher channel numbers is characteristic for the energy of the γ radiation. Due to this circumstance, the detectors studied are less suitable for measuring the energy of γ radiation, while they can be used for counting the γ photons of higher energies, especially at lower detector temperatures.

The absolute counting yield of the detectors for γ radiation of a Co⁶⁰ source was higher than 4%; it is clear that this value increases rapidly with decreasing energy of the radiation, owing to the increase of the absorption coefficient of Si for γ radiation.

The parameters of the detectors can be significantly improved by reducing the leakage current, using a guard ring or other suitable methods.

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ИЗГОТОВЛЕНИЕ И ИССЛЕДОВАНИЕ ТОЛСТЫХ Si(Li) p-i-n ГАММА-ДЕТЕКТОРОВ

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Изготовлены Si детекторы с обьёмом 3,2 см³ и 100 мм толстым компенсированным слоем при помощи диффузии Li в электрическом поле. Спектр амплитуды импульса детектора измерен в интервале температур от - 50 °C до + 5 °C при облучении детекторов радиоактивными источниками Cr⁵¹, Cs¹³⁷ и Co⁶⁰ с энергией 323, 662, 1170 и 1330 кэв. Характерные пики для энергии гамма-излучения в спектре не наблюдались, т. к. энергия излучения исследованного диапазона поглощалась в Si, вследствие нескольких комптоновских взаймодействий Положение затухающих участков полученных спектров характерно для энергии излучения. Абсолютная эффективность счёта детекторов, относящаяся к гамма-излучению Со⁸⁰, оказалась 4,8 и 4,5% соответственно. При хранении детекторов при комнатной температуре под маленьким обратным смещением в течение нескольких месяцев, параметры детекторов практически не изменялись. Также исследовалась форма возникшего импульса при воздействии на детекторы гамма-излучения в интервале температур от -65 до -30 °C.