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《Original》

A Study on the Germanium Radiation Detector Compensated by Gamma-ray Irradiation

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Abstract

The n-type germanium crystals have been irradiated by ^{60}Co gamma-ray with 647 Mrad at room temperature for compensation. The Ge(γ) detectors were fabricated from the gamma-ray irradiated germanium single crystals. The detector characteristics of the Ge(γ) detectors were comparable to those of thin Ge(Li) detectors and high purity germanium detectors. The thermal stability of the Ge(γ) detector showed a feasibility for ambient temperature storage.

요 약

n형 Ge 결정체를 보상하기 위해 상온에서 ^{60}Co 감마선으로 647 Mrad 조사하였다. 감마선으로 조사된 Ge 결정체로 Ge(γ) 검출기를 제작하였다. Ge(γ) 검출기의 검출기 특성은 얇은 Ge(Li) 검출기나 고순도 Ge 검출기와 비슷하였다. Ge(γ) 검출기의 온도에 대한 안정도는 상온에서의 저장이 가능함을 보여 주었다.

1. Introduction

In the past decade, the development of germanium detectors for scientific applications has been rapid and widespread.[1,2,3] Especially the Ge(Li) detectors are commonly used in the radiation spectrometry because of their good energy resolution and detection efficiency.[4] But the Ge(Li) detectors must be stored at the liquid nitrogen temperature (77°K) under a back bias to prevent diffusion of the lithium and consequently the degradation of the detectors. The fabrication of the Ge(Li) detectors has met with major difficulties with regard to the yield of finished detector and the consistancy of their energy resolution.[4]

In order to find a thermally stable germanium detectors, many investigators have studied on the development of the high purity germanium detectors[5] and the gamma-ray compensated germanium detectors (denoted by Ge(γ) detector).[6-11]

The high purity germanium with impurity concentrations less than $10^{10}cm^{-3}$ has recently become available in rather small dimensions. The depletion layer of a few millimeters can be achieved at the liquid nitrogen temperature. Unlike the Ge(Li) detectors, the high purity germanium detectors are thermally stable and can be stored at room temperature without any deterioration in detector characteristics. But the high purity germanium detector is still in its developing stage.[5]

The compensation process depends on the creation of crystal defects by energetic particles or gamma-ray irradiation. By the ^{60}Co gamma-ray irradiation, the vacancies and interstitials are produced in the n-type germanium single crystal. Most of these crystal defects anneal out at very low temperature, but a small fraction of the lattice vacancies form complexes with impurities in the crystal. These complexes produce acceptor levels deep in the forbidden energy gap and are stable at room temperature. Thus the n-type germanium crystal becomes an intrinsic material.[12]

In 1965, Ryvkin has reported on a gamma-ray counter of germanium with radiation induced defects.[6] Since then there were a few experimental evidences to support the gamma-ray compensation of germanium crystal. Therefore a further detailed study on the germanium compensation by gamma-ray irradiation was initiated.

In this study, three different n-type germanium crystals of 1, 20 and 30 ohm-cm, which have dopant elements of antimony, phosphorous and arsenic respectively, were used for the fabrication of the Ge(γ) detectors and to study the detector characteristics.

The thermal stability of the Ge(γ) detector was also investigated for the ambient temperature storage.

2. Experimental Procedure

2.1. Detector Fabrication

The Ge(γ) detectors were fabricated from the n-type germanium crystals of the following specifications.

Table 1. Germanium crystal specifications.

Sample	Resistivity (ohm-cm) (at 300° K)	Dopant	Net donor density (atoms cm^{-3})	Dislocation density (cm^{-2})	Orientation
A	1	Sb	4×10^{15}	?	(111)
B	20	P	7×10^{13}	1800–3500	(111)
C-1	30	As	4.5×10^{13}	1500	(111)
C-2	30	As	4.5×10^{13}	1500	(111)

The germanium single crystals of (111) orientation obtained from the Oak Ridge National Laboratory were employed in the experiments. Sample A, B and C had different dopant materials and dislocation densities. All samples were cut from different crystals, lapped and polished to a thickness of 1.1mm and a diameter of 2.5cm. Initially the samples had a circular disc shape and these germanium samples were mounted on a light-tight wooden box and irradiated by ^{60}Co panoramic irradiator at room temperature.

Figure 1 shows the configuration of gamma-ray irradiation by ^{60}Co panoramic irradiator.

For the accurate gamma-ray irradiation, the dose rate of ^{60}Co panoramic irradiator was repeatedly calibrated by ferrous sulfate dosimeters, Victoreen's Radocon and γ-meter. The measured value of irradiation dose was within $\pm95\%$ accuracy. The ^{60}Co gamma-ray source intensity was 10,050 Ci on April 12, 1973, and the dose rate was 0.85×10^6 R/hr at 10cm from ^{60}Co source on March 1, 1974.

For the gamma-ray compensation, the compensation factor (CF) is defined as:

$$CF=\frac{\text{Integrated photon flux}}{\text{donor density}}\ \text{cm} \quad (1)$$

The compensation factor for the n-type germanium crystals is known to be within the range of 1.6×10^3 to 6×10^3cm. The following

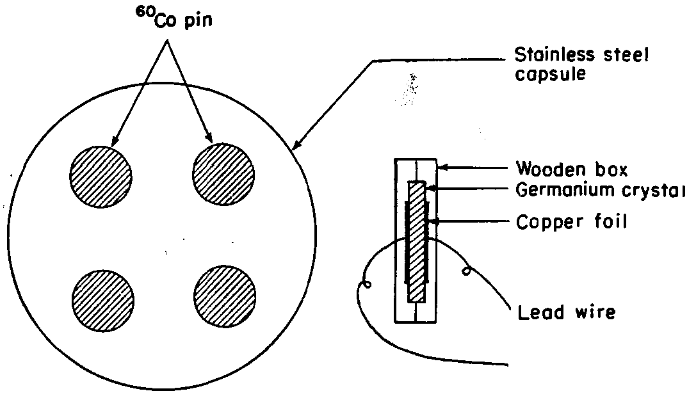

Fig. 1. Configuration of ^{60}Co gamma-ray irradiation.

equation approximates the relationship between the gamma-ray dose, D, in rad and the integrated photon flux, ϕ:

$$\phi = 0.9 \times 10^9 D \text{ photons/cm}^2 \qquad (2)$$

Inserting Eq. (2) into Eq. (1),

$$CF = \frac{0.9 \times 10^9 D}{\text{donor density}} \text{ cm} \qquad (3)$$

The dose required for a complete compensation of the n-type germanium crystal varies from a few hundreds Mrad to more than several thousands Mrad depending on the donor density. With the irradiation dose of 270 Mrad, the CF values for samples A, B and C-1 were 60cm, 3.5×10^3cm and 5.4×10^3 cm respectively. The irradiation dose for the sample A was raised to 1000 Mrad and the CF value was estimated to be 225cm, which was not enough for full compensation. Hence, the sample A had to be irradiated by gamma-ray dose of more than several thousands Mrad for compensation. The irradiation dose for the sample C-2 was 647 Mrad and the corresponding CF value was 13×10^3cm, and which indicated the complete compensation.

From the estimated CF values for each germaniun samples and the corresponding energy resolutions of the Ge(γ) detectors, the generally accepted range of CF values, $1.6 \times 10^3 \sim 6 \times 10^3$ cm, was not valid for the sample C-2.

Both surfaces and edges of the sample were carefully lapped with 1200 grade carborandum powder and then washed in an ultrasonic cleaner with deionized water, methanol and trichloroethylene. After washing, the samples were etched for 5 minutes in a HNO_3: HF= 10 : 1 etching solution.

The electrical contacts were applied on both surfaces of the sample. On one face of the sample, lithium was evaporated on and diffused into this face at 350°C for 2 minutes to provide an n contact.

On the opposite face, pure gold was evaporated on in high vacuum to a thickness of several hundreds Å to form a surface-barrier contact. This contact was circular and it had an equal area as that of lithium contact on the opposite surface.

The Ge(γ) detector thus made was left for approximately 40 hours for oxidation in a dry atmosphere and then clamped on a copper holder as shown in Fig. 2. A pressure contact of Au surface to the holdor material and an In-Ga alloy contact to n surface were provided.

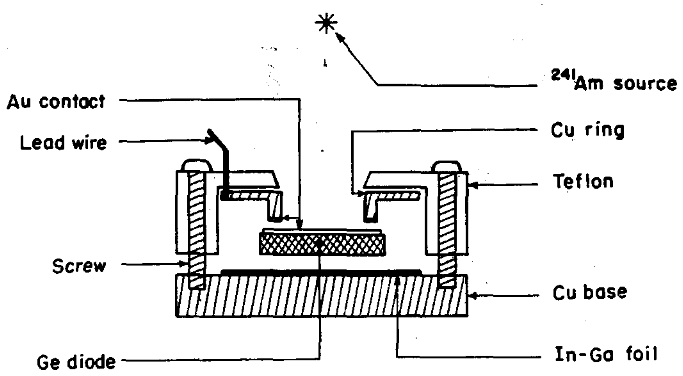

Fig. 2. The gamma-ray compensated germanium detector.

2.2. Measurements

In order to examine the process-rate of the gamma-ray compensation, the capacitance and resistivity changes were measured at intervals during the gamma-ray irrdiation without taking the samples out from the ^{60}Co irradiaton cell.

The Ge(γ) detector was clamped on a copper cold finger of a liquid nitrogen cryostat. An In-Ga foil was placed between the Ge(γ) detector and the copper base for good thermal contact. The capacitance vs. voltage characteristics were measured in vacuum at the temperature of 77°K using a LC meter. The reverse current vs. voltage characteristics were also measured in vacuum at the temperature of 77°K using a microammeter. During the measurements of detector characteristics, the reverse bias voltage was set at 30 volts for the samples A, B and C-1, and 20 volts for the sample C-2.

^{241}Am and ^{210}Bi alpha sources were used as radiation sources in the spectral measurements. The source was placed at 5cm from the Ge(γ) detector surface in the cryostat, 10^{-5} Torr. In order to examine the collected charge carriers, the signal currents were taken from the front and the rear contacts. The pulses from the Ge(γ) detectors were amplified by ORTEC-109A preamplifier and ORTEC-451 main amplifier and then the pulses were analyzed by TMC-256 channel analyzer.

The thermal stability of the Ge(γ) detectors was studied by leaving the Ge(γ) detectors for a long period at room temperature. During the ^{60}Co gamma-ray irradiation, the thermal effects on the Ge(γ) detectors at different temperatures were also studied.

The temperature maintained during the compensation procedure were at dry ice temperature and at room temperature. Two identical sample B detectors were fabricated by utilizing two different treatments for thermal effect study.

3. Results and Discussion

Figure 3 shows the measured capacitance vs. reverse bias voltage characteristics of the germanium detectors, sample B, C-1 and C-2. For these germanium detectors, full compensations were achieved at about 270 Mrad. The sample C-2 seemed to have achieved a complete compensation with the irradiation dose of 647 Mrad.

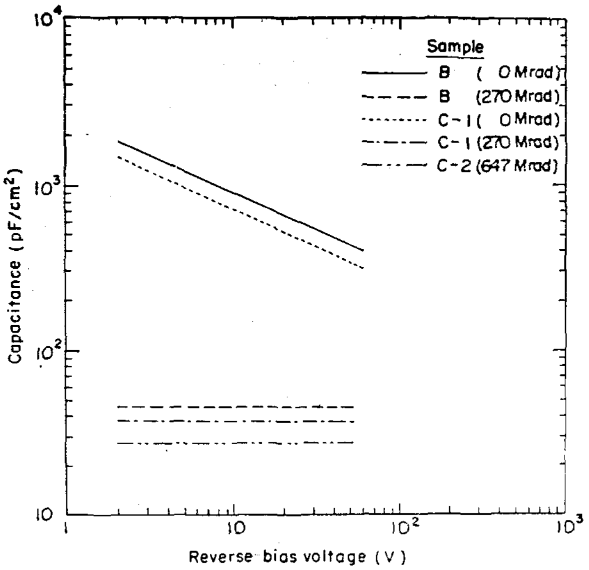

Fig. 3. Capacitance vs. voltage characteristics of the Ge(r) detectors (sample B & C-1) at 77°K.

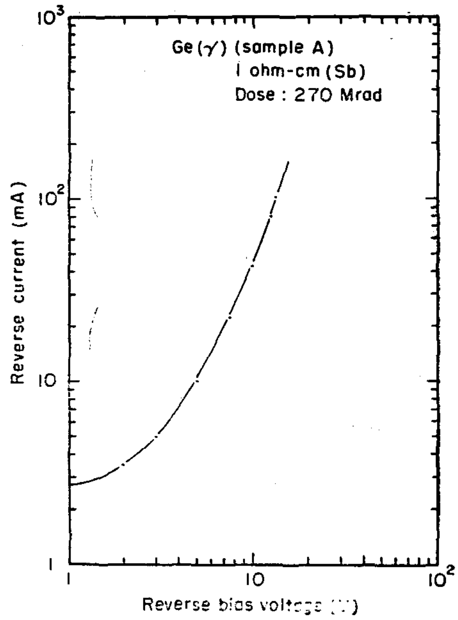

Fig. 4. Current vs. voltage characteristics of the Ge(r) detector (sample A) at 77°K.

Figures 4, 5 and 6 show the reverse current vs. reverse bias voltage characteristics of sample A, B and C-1 detectors respectively. The sharp rise in reverse current indicates the poor integrity of the back contacts of the Ge(r) detectors. The final surface treatments seemed rather rough, and the excessive reverse current could result in a poor detector resolution. The reverse currents were found to be approximately an order higher than the available data on other types of germanium detectors.[2)]

Figure 7 shows the reverse current vs. reverse bias voltage characteristics of sample C-2 detector.

One of the sample B detectors, which was compensated at dry ice temperature, showed no significant difference in I-V characteristics from the one irradiated at room temperature.

The spectral analysis was done by TMC-256 channel analyzer after the pulses from the

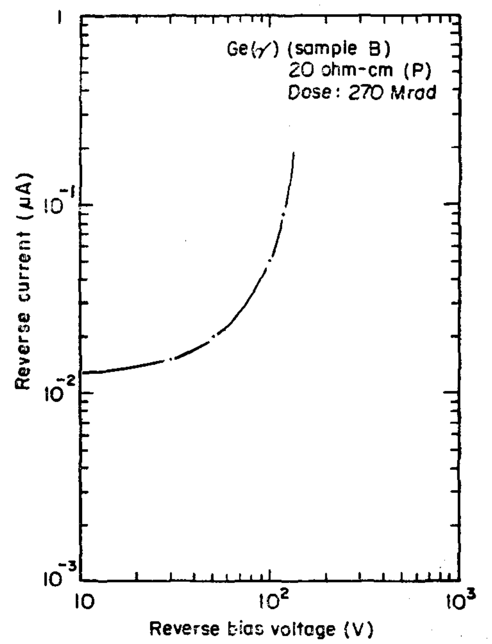

Fig. 5. Current vs. voltage characteristics of the Ge(r) detector (sample B) at 77°K.

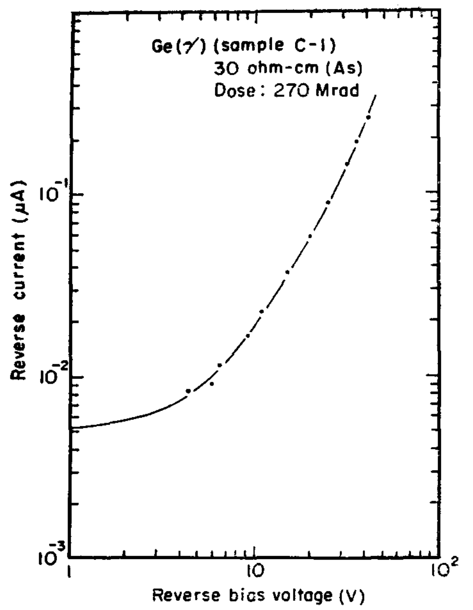

Fig. 6. **Current vs. voltage characteristics of the Ge(γ) detector (Sample C-1) at 77°K.**

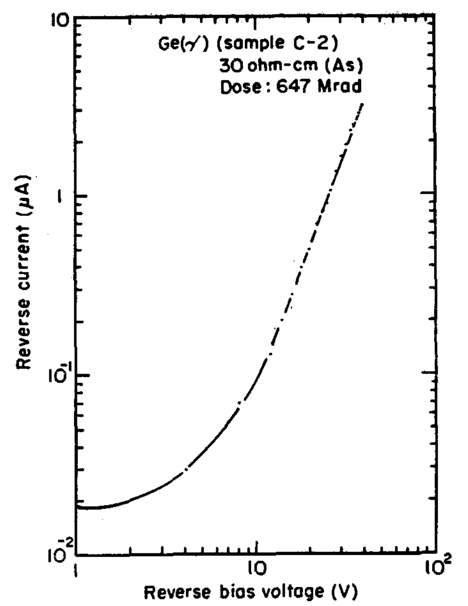

Fig. 7. **Current vs. voltage characteristics of the Ge(γ) detector (sample C-2) at 77°K.**

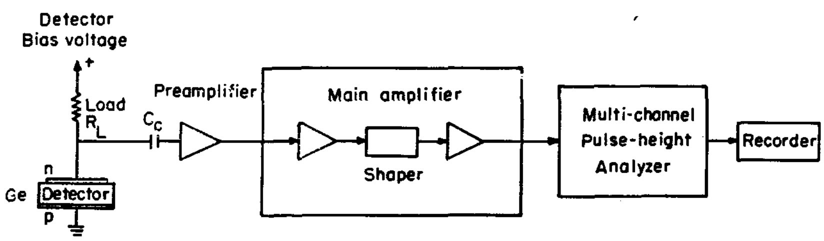

Fig. 8. **The germanium detector spectrometer systems.**

Ge(γ) detectors were amplified by ORTEC-109A preamplifier and ORTEC-451 main amplifier. Figure 8 shows the block diagram of the Ge(γ) detector spectrometer systems.

The spectral performances of the Ge(γ) detectors were examined after the gamma-ray irradiation. The spectra from the Ge(γ) detectors, sample B and C-1, are shown in Figs. 9 and 10. The spectra were taken from ^{241}Am alpha source. There was no difference between the spectra obtained by the pulse collection from p or n electrodes.

Figures 11 and 12 show the spectra of the Ge(γ) detector of sample C-2 for ^{241}Am and ^{210}Bi alpha sources respectively.

In Fig. 11, the 2.738 MeV pulser was taken for the energy calibration of the 5.477 MeV pulse from ^{241}Am alpha source.

The detector resolutions $(FWHM)_d$ of sample C-2 are 87 keV at 5.477 MeV of ^{241}Am alpha source and 198 keV at 4.65 MeV+4.69 MeV of ^{210}Bi alpha source. The Ge(γ) detector of sample C-2 showed the best detector resolution in these experimental results.

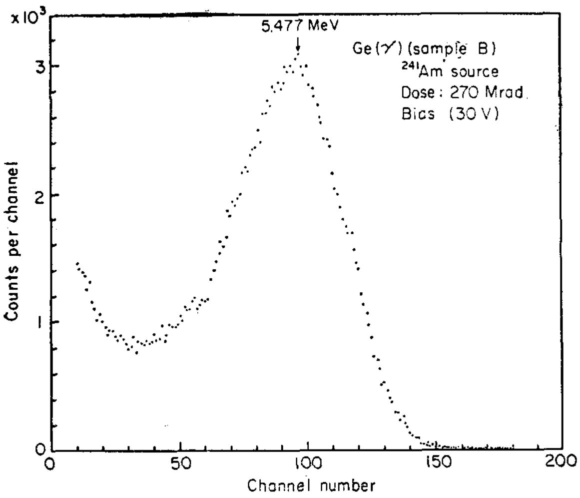

Fig. 9. ^{241}**Am alpha spectrum of the Ge(γ) detector (sample B) at 77°K.**

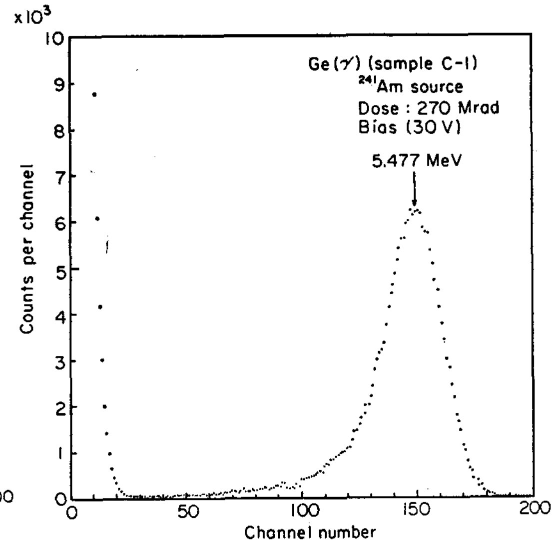

Fig. 10. ^{241}**Am alpha spectrum of the Ge(γ) detector (sample C-1) at 77°K.**

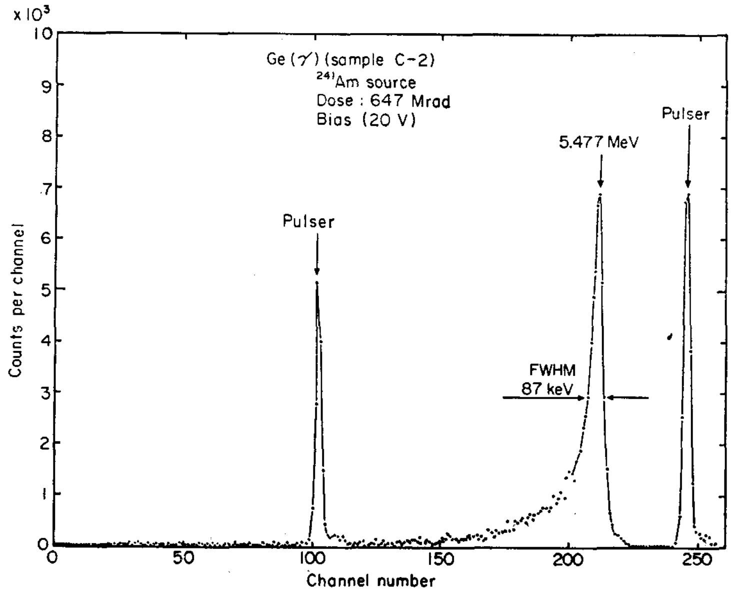

Fig. 11. ^{241}**Am alpha spectrum of the Ge(γ) detector (sample C-2) at 77°K.**

These detector resolutions are somewhat comparable to the thin Ge(Li) detector and the high purity germanium detector.

The preamplifier resolution $(FWHM)_c$ is derived from the following relation of ORTEC-109A preamplifier circuit.

$$(FMHM)_c = 2.3 + 0.0294 C_d \text{ keV} \quad (4)$$

where C_d is the detector capacitance of germanium crystal.

The detector capacitance C_d was calculated from the following relation.[7]

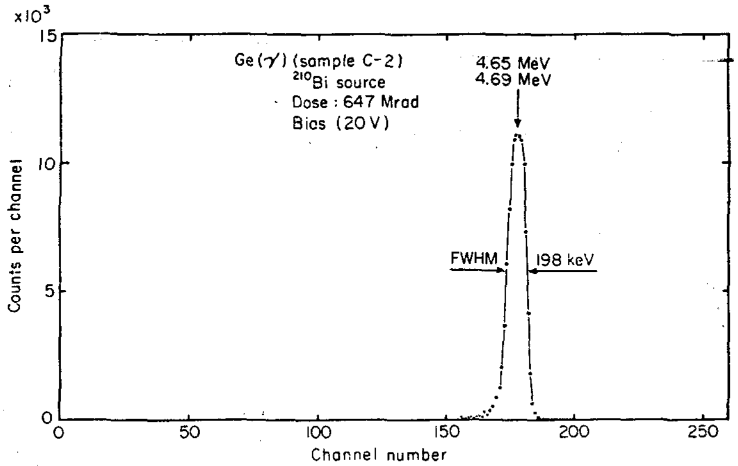

Fig. 12. ^{210}**Bi alpha spectrum of the Ge(γ) detector (sample C-2) at 77°K.**

$$C_d = 1.1\kappa A/(4\pi w) \quad \text{pF} \qquad (5)$$

where κ is the dielectric constant (16 for Ge), A is the detector area and w is the depletion layer thickness (0.1cm). Since the germanium crystal was a cylindrical planar type of 2.5cm diameter and 0.1cm height, the calculated detector capacitance is 68.75pF. Inserting this value in Eq. (4), the preamplifier resolution $(FWHM)_c$ is 4.3 keV. Therefore most of the observed detector resolution is the contribution from the detector itself.

The theoretical detector resolution $(FWHM)_d$ of the Ge(γ) detector was calculated from the following relation[2].

$$(FWHM)_d = 2.35(\epsilon EF)^{1/2} \text{ keV} \qquad (6)$$

where ϵ is the energy required for an electron-hole pair creation (2.98 eV/pair for Ge), F is Fano factor (0.129 for Ge), and E is the energy of incident radiation in keV. The calculated theoretical detector resolution for ^{241}Am alpha source at 5.477 MeV is 3.4 keV.

The calculated theoretical detector resolutions for ^{210}Bi alpha source at 4.65 MeV and 4.69 MeV are about 3.14 keV and 3.16 keV respectively.

Considering these theoretical and preamplifier resolutions, the observed detector resolution appears to be poor. This is attributed to the edge contamination[2] and imperfections introduced during the course of detector fabrication. The energy resolution may be improved by placing the source nearer to the detector with collimation.

For the fabrication of the Ge(Li) detector, a long term lithium-ion drifting time and high voltage are required in order to make a large intrinsic region, and the Ge(Li) detector has to be stored at the liquid nitrogen temperature at all the time. Considering these disadvantages in fabrication and storage of Ge(Li) detector, an uniform and completely compensated large volume Ge(γ) detector fabrication is quite promising, even though there is a room to improve the energy resolution by further study.

The thermal stability of the Ge(γ) detector was investigated during the compensation procedure and the temperature was maintained at room temperature for all germanium crystals except one sample B crystal. Two identical sample B detectors were fabricated by two different treatments at room tamperature and dry ice temperature.

But there was no significant differences in the detector characteristics by the annealing of radiation-induced defects, indicatings that the annealing on radiation defects occurs much below the dry ice temperature. In case of the room temperature irradiation, the samples were heated by gamma-ray flux, but the actual sample temperature was less than 60° C.

A long term storage of the Ge(γ) detector at room temperature did not show any deterioration in the detector characteristics for all detectors except for a slight increase in reverse current. The increase in reverse current after storage at room temperature may be attributed to the possible contamination of detector edges during storage. A slight etching of the detector edge with HNO_3 : HF =10 : 1 solution could recover the reverse current to original level. The temperature-cycle from room temperature to 77° K for an interval of an year had no influence on the detector characteristics.

The Ge(γ) detector had been successfully fabricated in the form of n-i-p diode with sample C-2. This detector is comparable to the thin Ge(Li) detector and to the high purity germanium detector (net acceptor concentration of $\sim 7\times 10^{11}/cm^3$) in their detector characteristics. In contrast with the Ge(Li) detectors,[4] the Ge(γ) detector may be stored at room temperature when not in use. In order to fabricate the Ge(γ) detector, the donor density of 10^{14} donors/cm^3 did not raise any problem for gamma-ray compensation while the high purity germanium detector requires the crystal donor density of 10^{11}-10^{12} donors/cm^3 or less.[5] In these respects, the germanium crystal for the Ge(γ) detector is much easier to obtain than the high purity germanium. The ^{60}Co gamma-ray seemed much better for homogeneous compensation of germanium crystal than the lithium-ion drifting method or the high purity germanium compensation.

By the gamma-ray compensation with 270 Mrad, the 20 ohm-cm (P doped) and 30 ohm-cm (As doped) n-type germanium crystals have shown fair detector characteristics, which is quite contradictory to the emphasis placed on the need for extreme crystal uniformity and to the idea that the nature of the center is strongly dependent on the kind of dopant introduced in making the n-type crystal[13]. It is safe to assume from the experimental results that only the donor density of original sample (i. e. resistivity) can affect the germanium detector characteristics. Therefore, the kind of dopant does not seem to affect the deep level gamma-ray compensation.

By the gamma-ray compensation with 647 Mrad, the 30 ohm-cm (As doped) n-type germanium crystal has shown quite satisfactory detector characteristics.

It is also apparent that the annealing during gamma-ray irradiation to the n-type germanium has no significant effect on the Ge(γ) detector characteristics.[14]

The evaporated gold on to the n-type silicon forms a good surface barrier; but in case of germanium, the direct evaporation of gold on the surface barrier has shown rather unstable detector characteristics. In order to avoid this instability, the gold evaporation has to be done on the germanium surface as well as the copper contact ring in vacuum.

4. Conclusions

The results of this study on the Ge(γ) radiation detector can be summarized as follows.

1) The degree of gamma-ray compensation to the n-type germanium crystal depends on the degree of impurity concentration and irradiation dose. An irradiation dose of 647 Mrad from ^{60}Co source seems to be quite adequate for complete gamma-ray compensation of 30 ohm-cm (As doped) n-type germanium single crystal at room temperature.

2) The energy resolutions of the Ge(γ) detector for ^{241}Am and ^{210}Bi alpha sources are comparable to the thin Ge(Li) detectors and the high purity germanium detectors.

3) During the gamma-ray compensation process, there ars no observable thermal effects on the n-type germanium crystals at the temperatures below 60°C.

4) The thermal stability of the Ge(γ) deector shows the feasibility for storage at ambient temperatures for more than an year.

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