Fabrication of a Large Volume Ge(Li) Detector

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Synopsis

This is a technical report on the fabrication of a large volume Ge(Li) detector of single open-ended type. We could minimize the material loss compared with FIEDLER's method. Furthermore, the heat-up process was interposed during the drift procedure which distinctly improved the drift efficiency. The volume of the final detector is 35 cc, and the energy resolution is 5.2 KeV at 2.754 MeV gamma-ray from ^{24}Na.

I. Introduction

There are some difficulties in fabricating and utilizing a large Ge(Li) detector of planar or double open-ended coaxial type. The reasons are found in the increase of the surface leakage current, very long drift time and the troubles in the etching procedure. The advantages and disadvantages of the single open-ended coaxial type, compared with other types, are as follows:

Advantages

a) Large intrinsic region can be obtained in reasonable drift time.
b) Chemical etching and quenching is easy.
c) Large active volume can be got without increasing the surface leakage current.

Disadvantages

a) The charge collection field at the closed end is non-uniform.
b) So, it shows intrinsically poorer timing resolution in gamma-ray coincidence experiment and also the energy resolution is inferior to the other types.

We think that the difficulty in the fabrication of Ge(Li) detector lies only in the chemical etching, quenching and washing procedures of the intrinsic region. So, we adopt the single open-ended type.

II. Fabrication of Ge(Li) detector

(1) Germanium crystal.

Used germanium single crystal is of "Hoboken", Belgium. It is in the form of pulled, gallium-doped p-type, grown in ⟨111⟩ orientation, of about 40∼43 mm in diameter and about 130 mm in length. The resistivity is in the range of 24 to 33 ohm-cm, dislocation density is $2300\sim2700/cm^2$ and the minority carrier life time is $650\mu sec$. A piece of 45 mm in length is cut out using a diamond cutter*.

(2) Lithium diffusion and drift.

Surface of the piece was lapped about 0.5 mm by a lapping machine with 600 mesh emery paper or by 600 mesh polishing powder on the glass. Then, 1000 mesh polishing was added. The sharp edge of the n^+ side was also lapped roundish to prevent clack formation which might be induced by a small mechanical shock. The crystal was etched in a 3:1 HNO_3/HF mixture until the crystal surface became mirror-like, and then quenched and washed by the deionized water of about 10^6 ohm-cm. Then, it was put on a clean filter paper and dried at room temperature. Prior to the diffusion of lithium, the crystal was carefully checked in order to ensure the absence of clack, scratch by locally high speed etching. When we found these defects, the lapping and etching procedures were repeated. These steps are indispensable in ensuring the uniformity of the lithium diffusion which promises a good diode.

The p-n junction was formed by lithium diffusion onto the germanium crystal. For this purpose, we adopted the lithium electroplating method. The 1:1 LiCl/KCl mixture (the melting point is approximately 380°C) was prepared in a graphite crucible. The germanium crystal placed on a graphite plate hanged by nickel wire was immersed in the melting mixture (Fig. 1 and 2). The temperature was raised slowly upto about 450°C, then lithium was electroplated allover the germanium crystal surface at a current density of about $40\,mA\sim60\,mA/cm^2$ for five minutes. Then, the crystal was removed from the crucible and cooled at room temperature within about twenty minutes by a fan. Thereby, the p-n junction having a good diode characteristic was obtained. Our method can minimize the loss of the crystal material compared with FIEDLER's method[1]. However, the lithium contamination on the p-type surface must be removed about 1 mm by lapping.

The lithium diffusion depth (n^+ type) was measured by the Cu-plating must have the uniformity to prevent the breakdown during the drift process. The drift process was performed in the boiling n-pentane (at 36°C).

* We wish many thanks to Tokyo Denshi Yakin Co., Ltd.

The essential points of the etching procedure will be described in the next paragraph (3). Fig. 3 show the current vs time characteristics. Region I shows low voltage-large current characteristic which sometime appears in the initial drift, and region II, after heated at about 420°C for several minutes, show high voltage-low current characteristic, which may be attributed to the re-resolving of most of the precipitated lithium in the n^+ region. When the intrinsic region grew to about 10 mm depth after seven weeks drift, we re-diffused new lithium to obtain the Li-riched layer. After the short time drift, about 100 hr, the diode was cleaned-up for three weeks at −10°C. The typical condition of the clean-up process was 10 mA at 400 V. The final clean-up was performed for about 24 hr at the dry-ice temperature which condition was

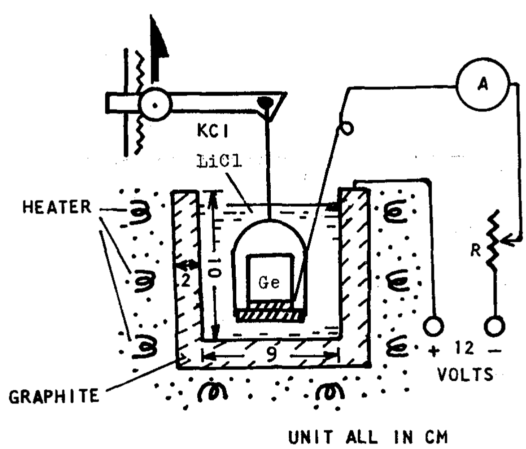

Fig. 1. Apparatus for Electroplating.

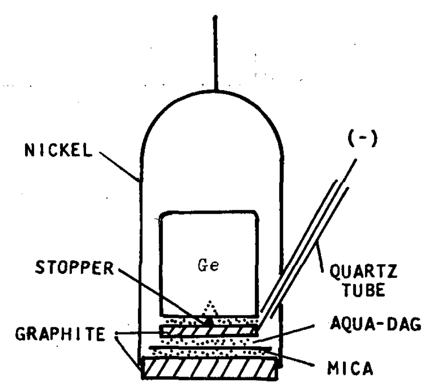

Fig. 2. Crystal Hanger.

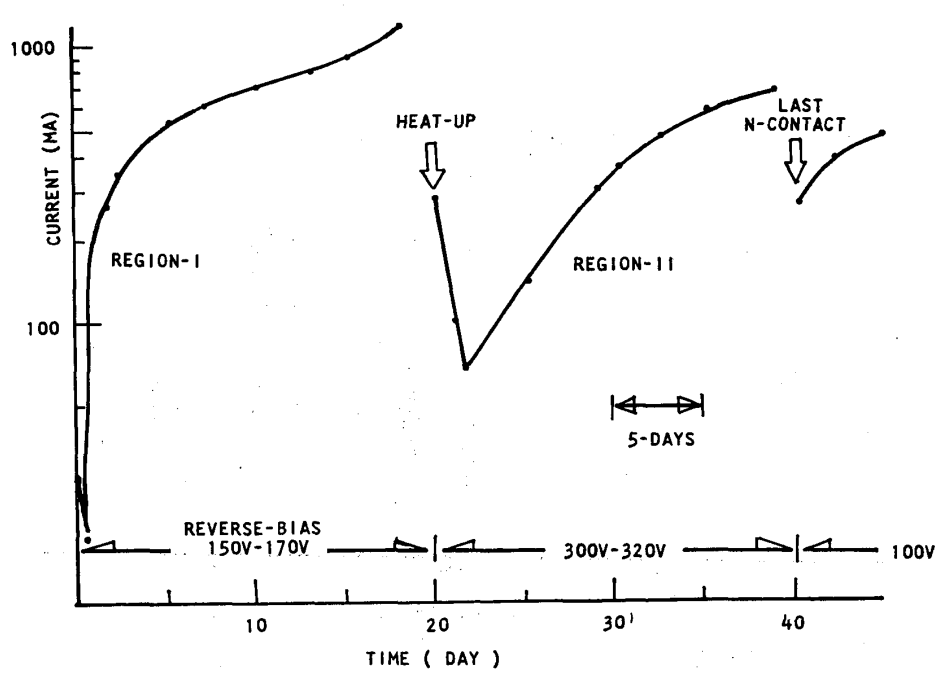

Fig. 3. Current vs time characteristic in Drifting.

0.3 mA at 1000 V.

(3) Mounting

The greatest care was needed for mounting[2]. The open ended section which was an exposed n-i-p junction was lapped about 0.5 mm. Furthermore, the fine polishing powder was used to ensure the flatness of this surface. The washing and de-greasing was carried out with KOH(aq). Then, n^+ surface

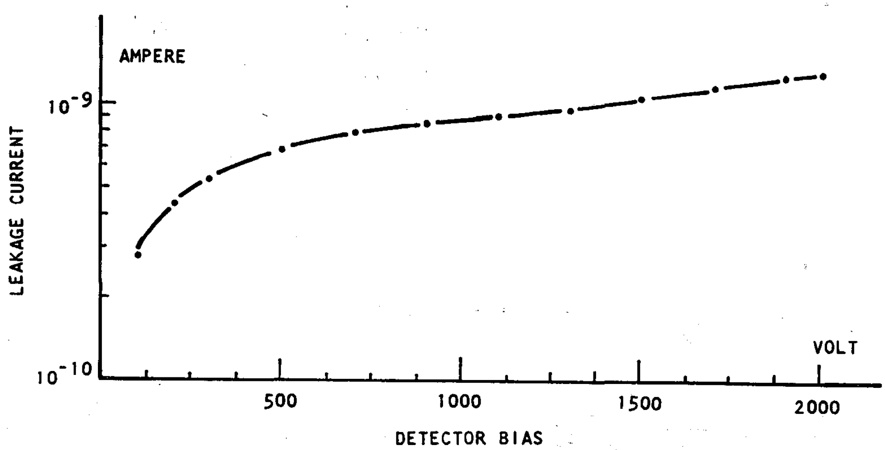

Fig. 4. Leakage current vs bias voltage.

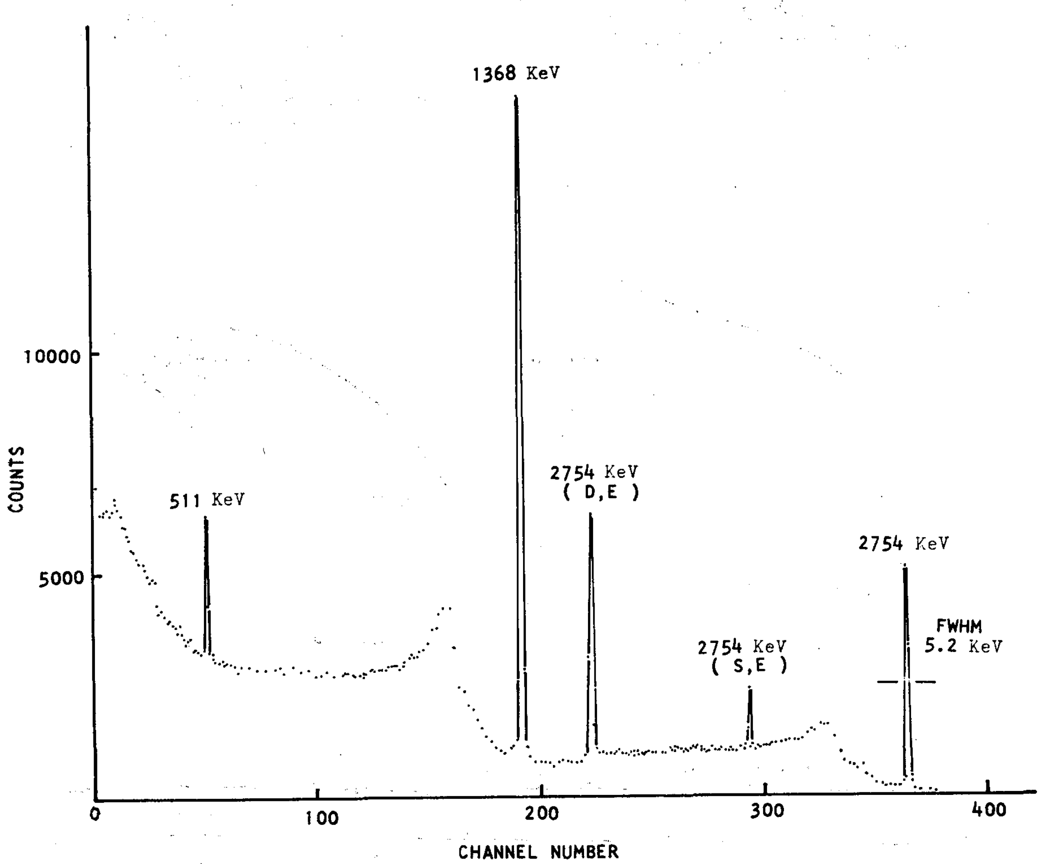

Fig. 5. Gamma-ray spectrum from ^{24}Na source.
Bias: 2000 V Preamp: ortec 118 A

was masked with the plastic tape*. To avoid the separation of this tape from n^+ surface, we usually wound a vinyl tape around it.

The diode was then etched in 3:2 HNO_3/HF mixture for long enough time until the visible n-i junction line and mirror-like surface were observed. Adequate etching temperature was 20 to 25°C.

After peeling off the masking tape by tweezers, the diode was lightly etched by 5:1 HNO_3/HF, one or two times. At that time, we must not touch

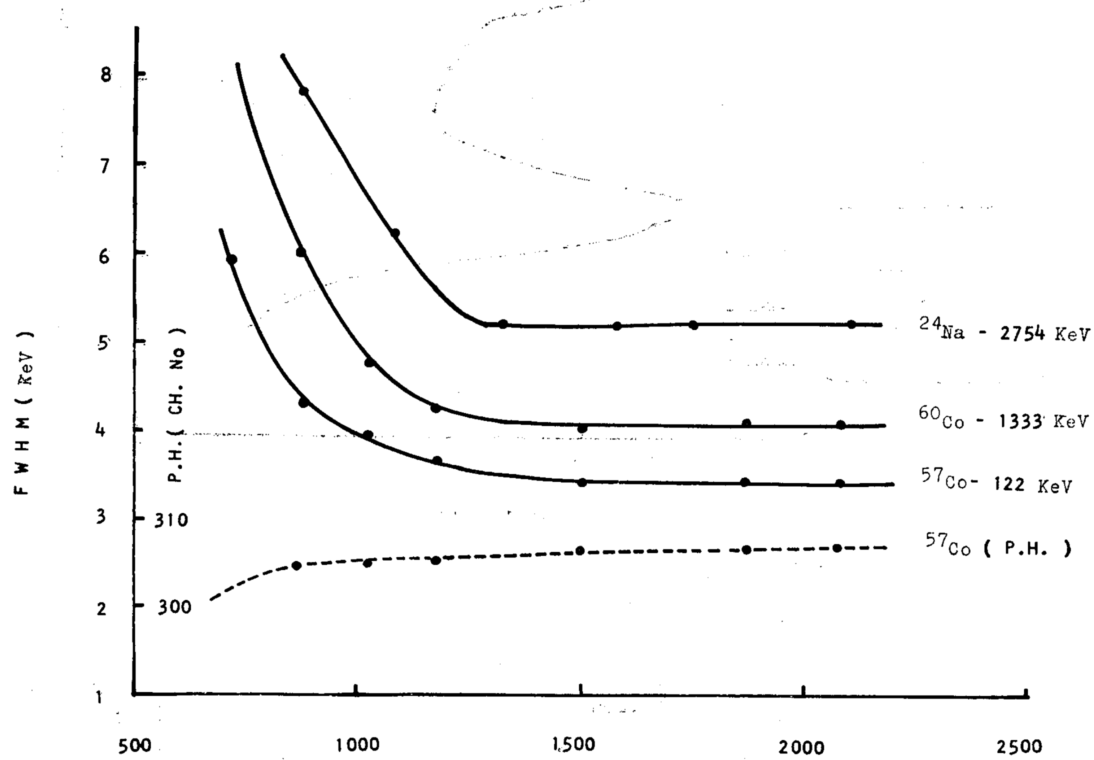

Fig. 6. Variation of detector properties vs bias voltage.

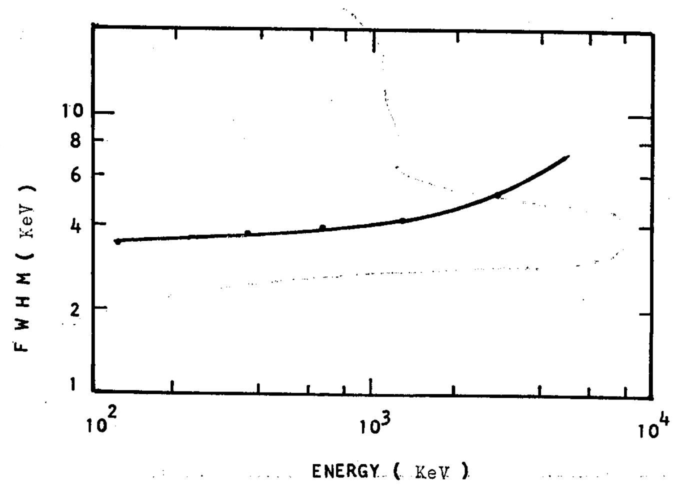

Fig. 7. Resolution vs gamma-ray energy.

* Sumitomo three M plastic tape, No. 470.

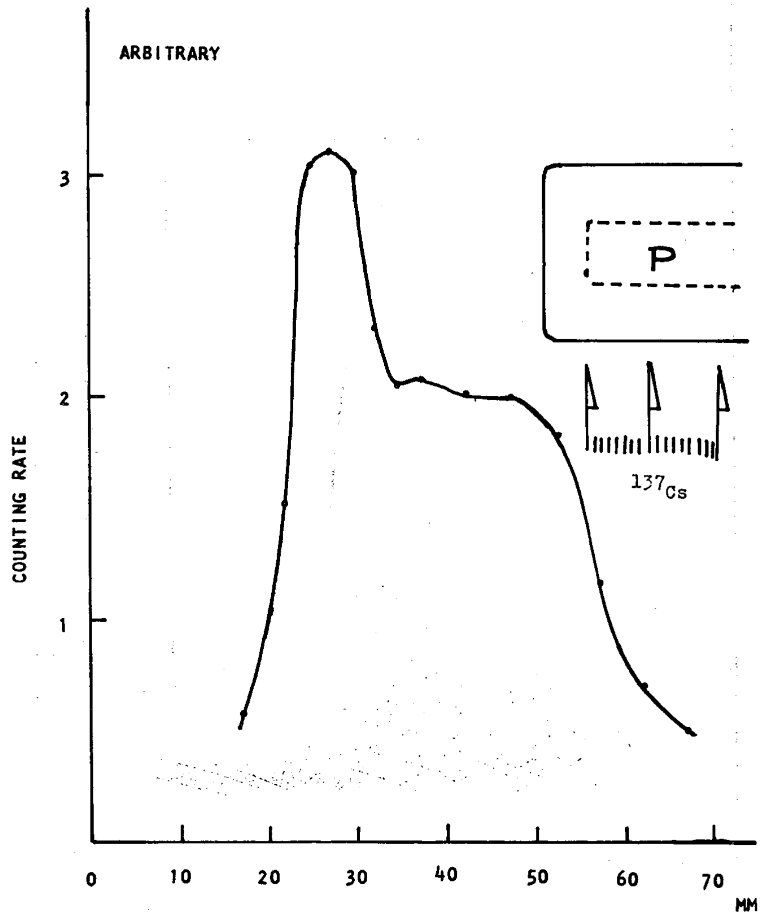

Fig. 8. Colliminated Gamma-ray beam scanning.

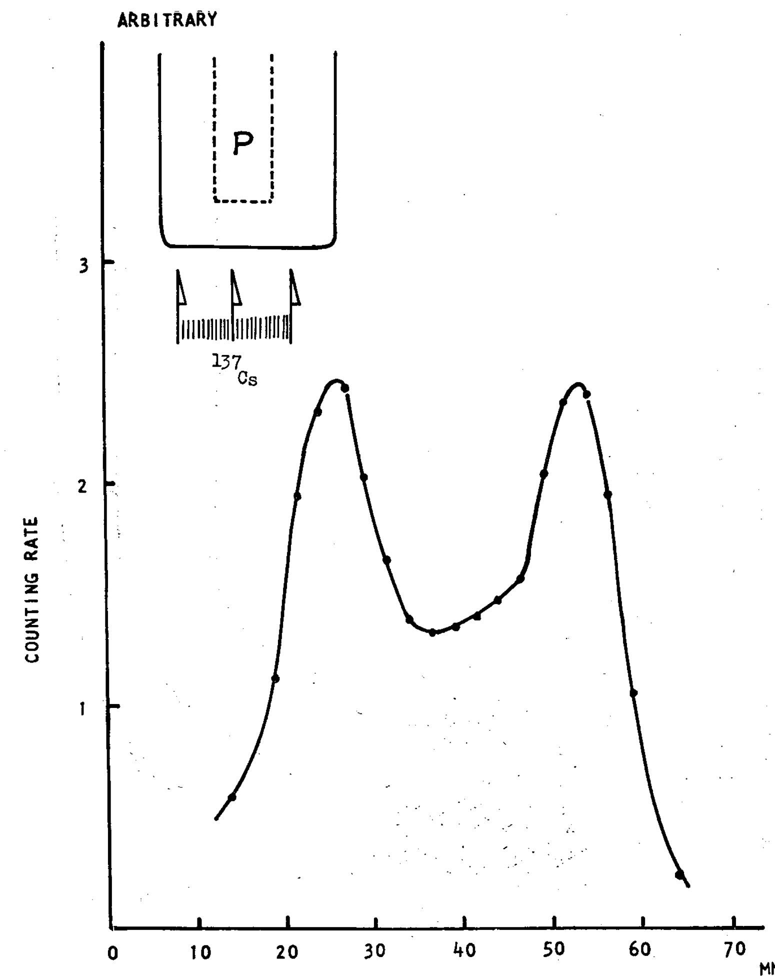

Fig. 9. Colliminated gamma-ray beam scanning.

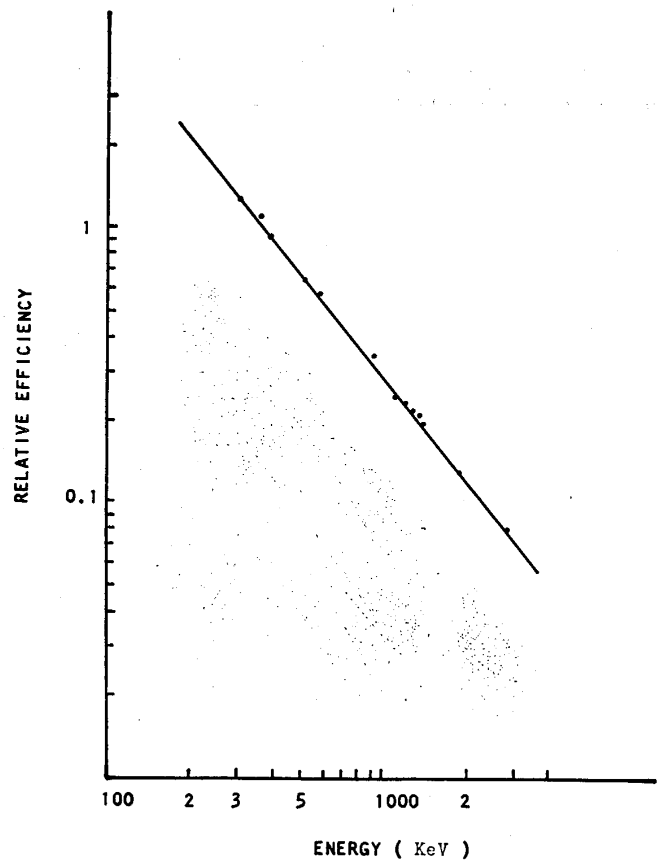

Fig. 10. Relative efficiency vs Gamma-ray energy.

the exposed n–i–p surface. The quenching and washing was performed by diluting the etchant with methyl alcohol. After dried on a filter paper, it was mounted on the cold finger sideways. Then, the cryostat was evacuated upto about 1×10^{-5} mmHg and cooled down to liquid nitrogen temperature.

III. Performances

Leakage current vs bias voltage, gamma-ray spectrum from ^{24}Na, resolution and pulse height vs bias voltage, and resolution vs gamma-ray energy are shown in Fig. 4 to 7, respectively. Effective volume of this detector is 35 cc. Energy resolution is 4.2 KeV at 1.333 MeV and 5.2 KeV at 2.754 MeV. Estimated capacitance is about 80 pF. Fig. 8 and 9 show the results of the collimated gamma-ray scanning, and Fig. 10 is the efficiency vs gamma-ray energy curve at one source distance.

Acknowledgements

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References

1) H. J. FIEDLER, et al., Nucl. Instr. and Method, 40, 229, (1966).
2) M. ISHII, et al., J. A. E. R. I., No. 1131, (1966).