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FABRICATION TECHNIQUES FOR REVERSE ELECTRODE COAXIAL GERMANIUM NUCLEAR RADIATION DETECTORS

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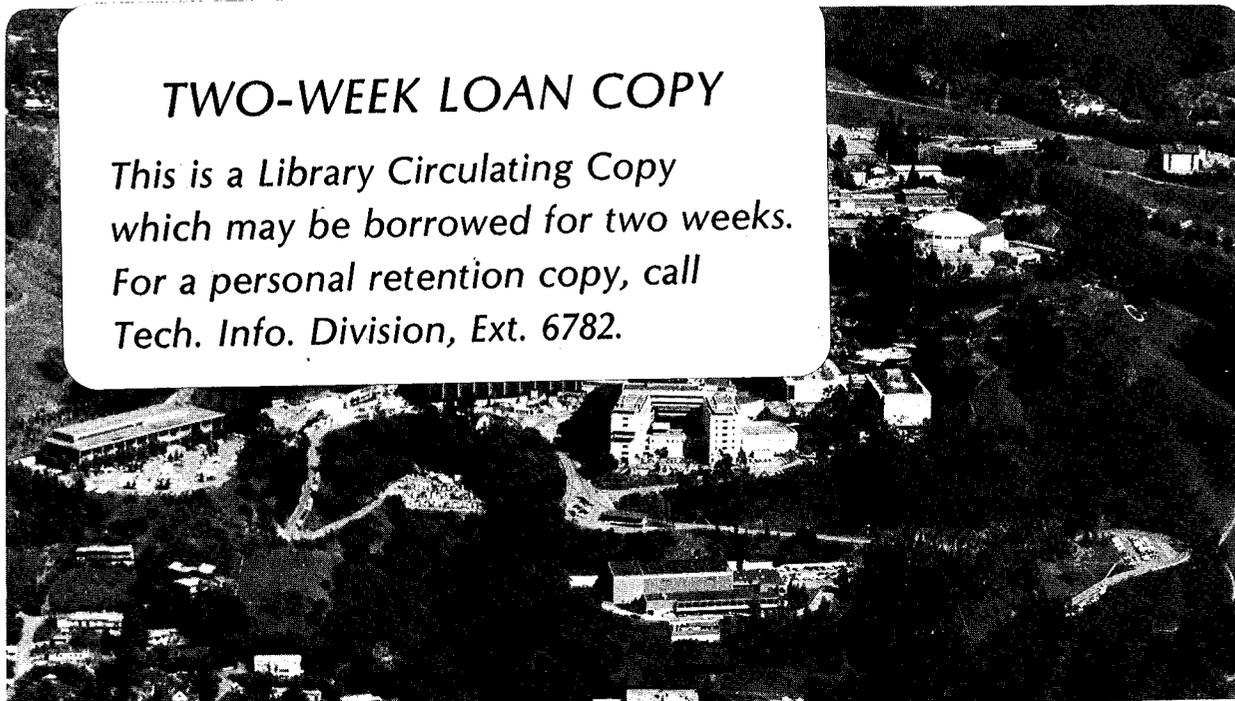
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### Summary

Germanium detectors with reverse polarity coaxial electrodes have been shown to exhibit improved resistance to radiation damage as compared with conventional electrode devices<sup>1</sup>. However, the production of reverse electrode devices involves the development of new handling and fabrication techniques which has limited their wider application. We have developed novel techniques which lead to a device which is simple to fabricate, environmentally passivated and surface state adjusted.

1) n+ Contact. The n+ contact in the central hole in the crystal is formed by lithium, electroplated from a LiNO<sub>3</sub>-KNO<sub>3</sub> eutectic molten salt bath held at the diffusion temperature. The outside surface of the crystal is protected during electrolysis by teflon pipe tape. Electrolysis has proven to be fast and convenient and it provides a rugged surface for handling during further processing.

2) p+ Contact. The p+ contact on the outer surface of the crystal is formed by boron ion implantation. The crystal is held by a shaft through the central hole and implanted with 10<sup>15</sup>cm<sup>-2</sup>, 25 KeV boron ions. No post-implant anneal is needed.

3) Passivation and Surface State Adjustment. The open ends of the coaxial detector have their surfaces passivated and the surface states adjusted to flat-band condition by sputtering germanium in an argon-hydrogen mixture<sup>2</sup>. A special sputtering fixture is described for conveniently mounting the detector during sputtering.

Due to the combination of thin external contacts and interelectrode surface state adjustment, the detector has complete charge collection over nearly 100% of the original crystal volume.

### Introduction

It has been demonstrated<sup>1</sup> that coaxial germanium nuclear radiation detectors made from high purity n-type crystals with a reverse electrode structure exhibit superior resistance to degradation from radiation damage as compared to conventional devices. The electrode configuration of the conventional and reverse detectors is illustrated in Fig. 1. For the conventional detector the n+ contact is usually formed by diffusing lithium deposited by vacuum evaporation and the p+ contact by an evaporated metal Schottky barrier. This process results in a detector that is simple to fabricate but which has high sensitivity to radiation damage and which uses a delicate Schottky barrier contact. Techniques have been developed recently that allow simple fabrication of reverse electrode detectors which have durable contacts and are environmentally passivated.

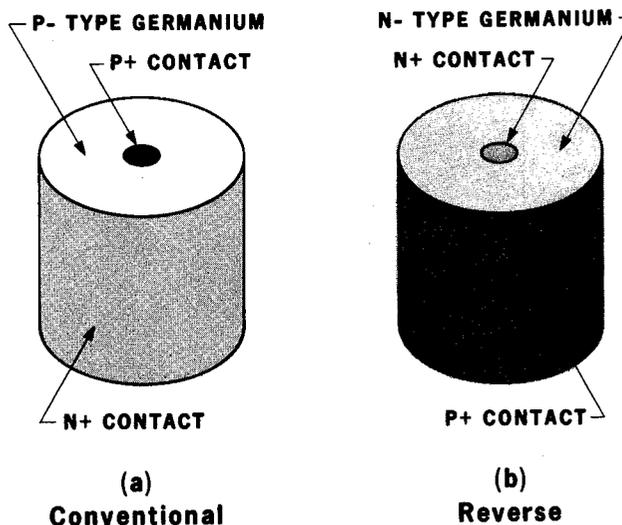


Fig. 1. Electrode configuration for conventional and reverse coaxial detectors.

### Lithium Contact

Because of the small bore hole (4 to 8 mm) of the coaxial detector, it has not been found feasible to produce the n+ contact of the reverse electrode device by vacuum evaporation and subsequent diffusion of lithium. Our usual procedure has been to use lithium in oil suspension. This involves a rotation fixture extending into a tube furnace, a low temperature oil evaporation step (250°C) and finally a higher temperature diffusion step. The lithium does not reliably attack the germanium uniformly unless the diffusion step is carried out at a minimum of 400°C and this temperature is too high to avoid the introduction of copper-related trapping centers. These problems have been solved by forming the lithium contact by electrolysis from a molten salt bath--a method which has proven to be fast, convenient and reliable.

The lithium electrolysis cell (Fig. 2) is constructed of spot-welded stainless steel sheet with a piece of Pt-10% Rh thermocouple wire for the anode, held in place by ceramic insulators. The electrolyte, which was first suggested by Robertson and Kennett<sup>5</sup>, is a eutectic mixture of LiNO<sub>3</sub> and KNO<sub>3</sub> (35 wt.% LiNO<sub>3</sub> - 65 wt.% KNO<sub>3</sub>) which melts at 130°C.<sup>2</sup> The apparatus is lowered into a 500 cm<sup>3</sup> Pyrex resin pot which is contained in a Glas-Col<sup>3</sup> resin pot heater. The bath is maintained at 220°C to keep it dry and so that excessive amounts of salt do not freeze on the device when it is inserted. In order to prevent the lithium from depositing on the outer surface of the detector, it is covered with 3 or 4 layers of 2 mil teflon plumbers tape. A fused silica thermocouple well is provided adjacent to the detector (the soda-lime glass of normal thermometers is attacked by the molten salt).

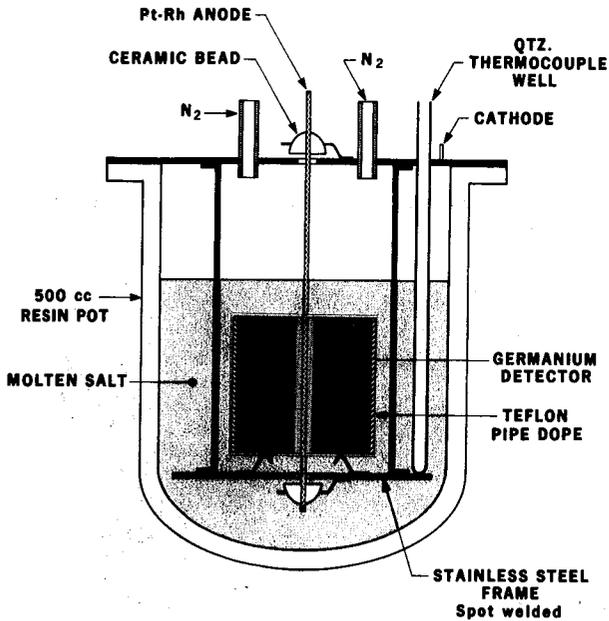


Fig. 2. Molten salt bath lithium electroplating cell.

The lithium diffusion sequence is as follows:

- (1) insert electrolytic cell in molten salt at 220°C,
- (2) raise bath temperature to desired diffusion temperature,
- (3) using the Pt-Rh wire as the anode and the stainless structure as the cathode, perform lithium plating at 500 mA. Too slow an electrolysis rate will result in the lithium being consumed by residual water and oxygen. NO<sub>2</sub> gas is evolved by the anode,
- (4) after selected diffusion time, remove cell, tilt to drain excess molten salt and allow to cool in room air.

Using a diffusion time of 30 minutes and a bath temperature of 320°C, the diffusion depth is about 0.50 mm. The frozen salt left on the device after cooling is weak and does not unduly stress the crystal. No fumes are evolved by the molten salt without electrolysis and the salt does not migrate to cooler parts of the apparatus. The end surfaces of the detector are not protected from the lithium and so must be removed by lapping or sawing. A device ready for lithium plating is shown in Fig. 3.

#### P+ Contact

After the device has been lithium diffused and the lithium diffused ends removed by lapping, the entire device is polish etched for two minutes in 3:1 HNO<sub>3</sub>:HF, quenched and rinsed in CH<sub>3</sub>OH, rinsed in 1% HF and blown dry. The p+ contact is formed by implanting 25 KV boron ions with a dose of about 10<sup>15</sup> cm<sup>-2</sup>. During implanting the device is held by the central hole with two teflon cones on a stainless steel shaft which is motor driven. The device as implanted has sufficient boron activation so that no post implant anneal is required.

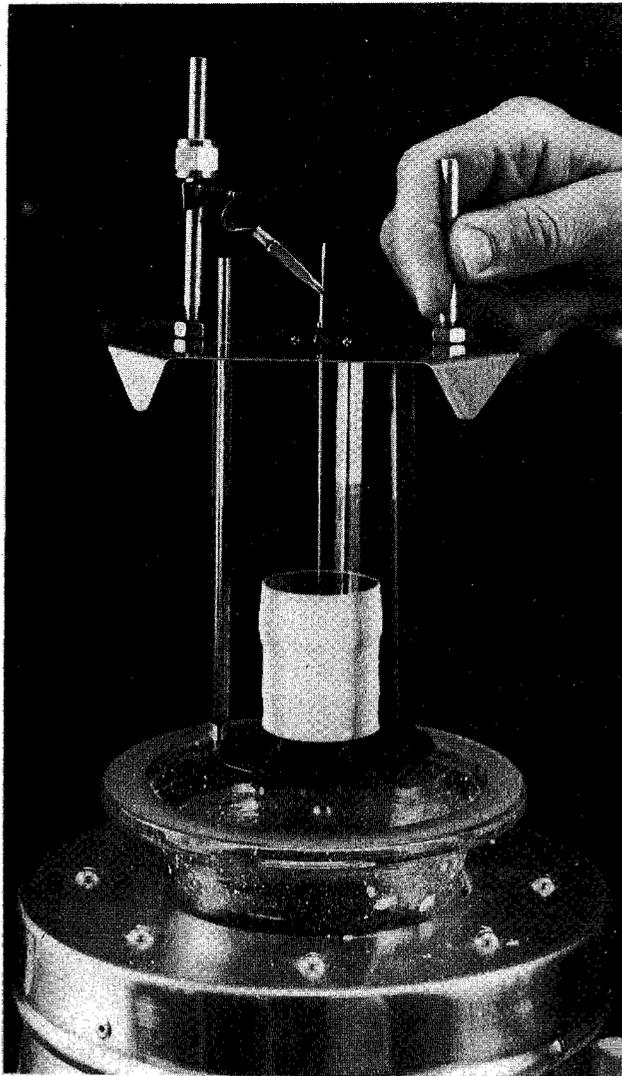


Fig. 3. Device ready for lithium plating.

#### Passivation and Contacts

After boron implantation, the ends of the detector have their surface states adjusted and passivated with a deposit of hydrogenated amorphous germanium (a-Ge:H).<sup>4</sup> For this purpose the boron implanted outer surface is protected with vinyl tape and the device is again briefly etched in 3:1 HNO<sub>3</sub>:HF, quenched and rinsed in CH<sub>3</sub>OH and blown dry. Using a shaft through the central hole as a handle, the tape is removed and the device inserted in a special sputtering fixture as shown in Fig. 4. Typical sputtering conditions are:

- sputtering gas - Ar+7%H<sub>2</sub> at 7μ pressure;
- target - eight in. diameter germanium;
- power - 300W at 13.56 MH;
- target spacing - 4 cm;
- sputtering time - five minutes;
- coating thickness - about 3000Å.

Further details of the sputtering conditions can be found in Reference 4.

## REFERENCES

1. R. H. Pehl, N. W. Madden, J. H. Elliott, T. W. Raudorf, R. C. Trammell, and L. S. Darken, Jr., "Radiation Damage Resistance of Reverse Electrode Ge Coaxial Detectors", *IEEE Trans. Nuc. Sci.* NS-26, No. 1 (1979).
2. E. M. Levin, C. R. Robbins, and H. F. McMurdie, *Phase Diagrams for Ceramists*, Amer. Ceramic Soc., Fig. No. 1069 (1964).
3. Glas-Col Apparatus Co., Model No. TM-570.
4. W. L. Hansen, E. E. Haller and G. S. Hubbard, "Protective Surface Coatings on Semiconductor Nuclear Radiation Detectors", *IEEE Trans. Nuc. Sci.* NS-27, No. 1, 247-51 (1980).
5. R. Robertson and T. J. Kennett, *Nuclear Inst. and Meth.* 98, 599-600 (1972).

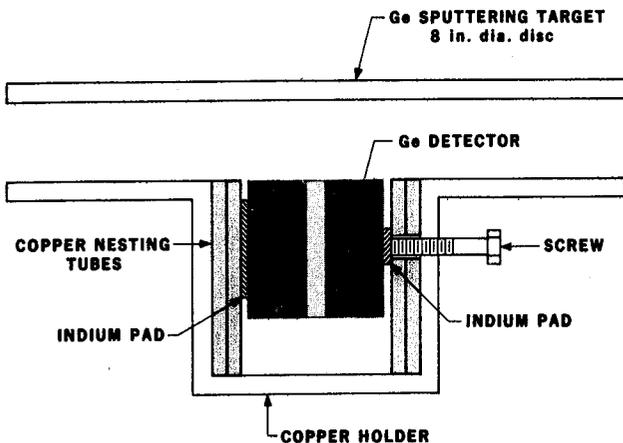


Fig. 4. Sputtering fixture for passivating ends of coaxial detector.

The finished detector has durable layers on all surfaces and is stable during ambient storage. Radiation damaged detectors can be annealed at 150°C in an inert gas or vacuum with no requirement for new surface treatment.

The detector needs only contacts to the highly doped n+ and p+ contacts for operation. This is most conveniently accomplished by rubbing a dab of In-Ga eutectic (25 wt.%In) on the implanted and diffused layers and using pressure contacts. When using liquid metal contacts it is important to apply a very thin layer to avoid local stress on cooling.

In practice, the reverse electrode structure coaxial detector has proved simpler to fabricate by these techniques than the conventional electrode detector mainly because it avoids the delicate Schottky barrier contact. The thickly diffused core provides a rugged surface for handling and at the same time uses a very small volume of expensive germanium. The a-Ge:H passivated ends are nearly neutral and allow a 100% charge collection throughout the whole detector volume. With the thin implanted p+ contact, the entire external surface of the detector presents a very thin window and allows detection of low energy  $\gamma$ - and x-rays.

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