ORTEC®

High-Purity Germanium (HPGe) Detector Manufacturing

For 50 years ORTEC has been responsible for most major innovations in silicon charged-particle and germanium gamma-ray detectors (see Timetable). ORTEC was the first company to develop its own germanium crystal growth technology. The ensuing availability of the world's best detector-grade germanium has made ORTEC the world's primary supplier of high-purity germanium detectors.

The production of ORTEC HPGe photon detectors involves complex manufacturing processes. The description of these processes in this section emphasizes the tangible benefits to both customer and manufacturer of having crystal and detector manufacturing plus quality control "under one roof."

Process

The many steps required to convert polycrystalline germanium into a gamma-ray spectrometer are depicted in Fig. 1. The description of these steps follows the flow of germanium through the process, some steps being more intricate than others.

The initial starting material, electronic grade polycrystalline germanium "metal" (because of its metallic appearance), is zone refined in a quartz "boat" having a pyrolytic graphite coating. A zone refiner uses the principle that most impurities concentrate in the liquid phase as the material begins to freeze. The rf heating coils of the zone refiner melt a small section of the germanium ingot or bar held in the quartz boat.

The rf coils are slowly moved along the length of the ingot, causing the liquefied portion beneath the coils to move also. Thus, the ingot is continuously melting at the advancing solid-liquid interface and freezing at the receding interface. The impurities tend to remain in the molten section and hence are "swept" to one end of the ingot by this process. This "sweeping" operation is repeated many times, until the impurities are concentrated at one end of the ingot. This end is then removed, leaving the remaining portion much purer than the original starting material. Figure 2 shows a three-coil zone refiner in operation. The improvement or reduction in impurity concentration actually realized is about a factor of 100 or more at the completion of this process. Figure 3 shows a zone-refined ingot. The tapered end contains the high concentration of impurities and is cut off. The impurity concentration of the remaining portion is then determined by a Hall effect measurement, and the ingot is sliced into pieces suitable for loading into the crystal-growing equipment.

Timetable of ORTEC Innovations in Semiconductor Radiation Detectors

Silicon Surface Barrier detectors	1961
Ge(Li) Lithium-Drifted detectors	1965
Si(Li) Lithium-Drifted detectors (X-ray)	1968
Ge(Li) LEPS Low-Energy Planar detectors	1973
In-House Ge(Li) material	1973
HPGe LEPS with First Ion-Implanted Contact	1975
High-Purity Coaxial Germanium detectors	1976
In-House HPGe material	1977
N-Type Germanium detectors (GAMMA-X)	1978
HPGe Well detectors	1979
Streamline cryostat	1979
Portable HPGe detector (Gamma Gage)	1979
Low-Energy Coaxial (LO-AX) detector	1979
DUET BGO-HPGe and "Golf Club" detectors	1983
Electrically Cooled HPGe detector	1985
70% Efficiency P-type HPGe detector	1986
70% Efficiency N-type HPGe detector	1987
PopTop Transplantable detector	1987
80% Efficiency P-type HPGe detector	1987
100% Efficiency P-type HPGe detector	1988
150% Efficiency P-type HPGe detector	1992
175% Efficiency P-type HPGe detector	1994
IGLET-X Soft X-Ray detector	1994
"Safeguards Tuned" HPGe detector	1998
X-COOLER low-cost electrically cooled detector	2000
200% Efficiency P-type HPGe detector	2000
SMART-1 Authenticated HPGe detector	2001
PROFILE Series GEM HPGe detector	2001
trans-SPEC HPGe Spectrometer	2004
Interchangeable Detector Moduel (IDM)	2007
trans-SPEC-DX-100 HPGe Spectrometer	2009
Micro-trans-SPEC Portable HPGe Spectrometer	2009

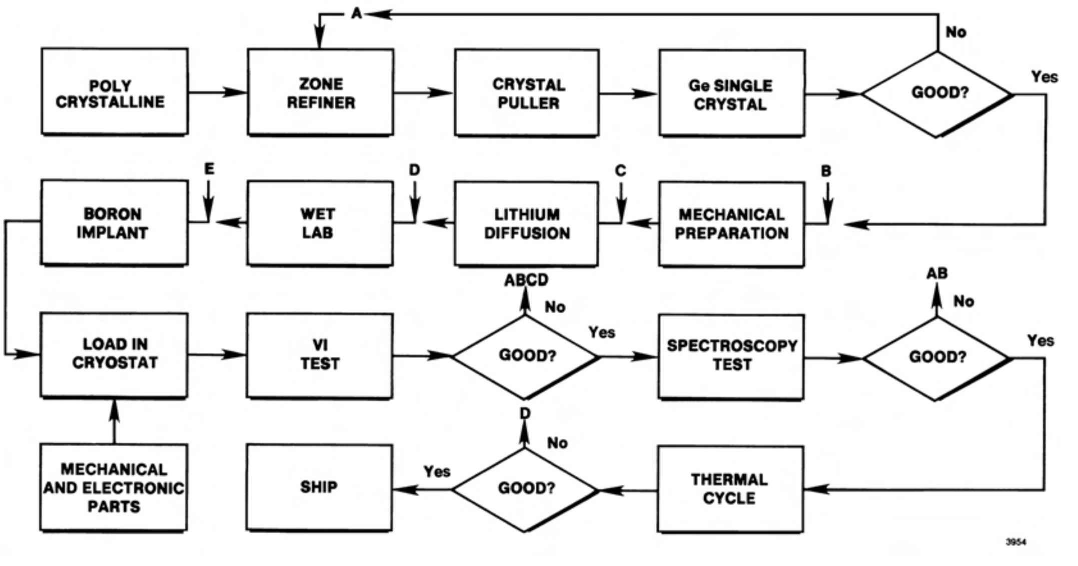

Fig. 1. Germanium Detector Manufacturing Process.

High-Purity Germanium (HPGe) Detector Manufacturing

Large single crystals of germanium are grown using the Czochralski technique, which is schematically illustrated by Fig. 4. A precisely cut seed crystal is dipped into the molten germanium and then withdrawn slowly, while maintaining the temperature of the melt just above the freezing point. The rate of crystal withdrawal and temperature of the melt are adjusted to control the growth of the crystal.

Figure 5 shows a crystal during the growth process. High-purity germanium crystals suitable for detector fabrication are almost always grown in a quartz crucible under a hydrogen atmosphere. Near the completion of the growth process, the crystal is tapered gradually at the tail to minimize thermal strain. It is imperative that the crystal be grown to the exhaustion of the melt, because germanium both wets quartz and expands on freezing. The valuable quartz crucible might be fractured if any germanium were left after completion of the crystal growth.

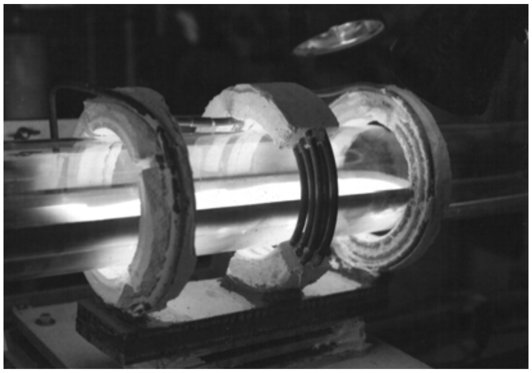

Fig. 2. A 3-Coil Zone Refiner.

After the crystal is grown and cooled, it is mounted in a Plaster-of-Paris cast for slicing. Figure 6 depicts a mounted crystal during the slicing process. The completed crystal is cut by an ORTEC-designed string saw that causes virtually no damage to the crystal. A slurry of water and silicon carbide is pulled along by a wire, resulting in a sawing action. Sections of the crystal from both top and bottom are checked by Hall effect measurements to determine the impurity concentration and type (n or p). On the basis of the Hall effect results, that part of the crystal which contains detector-grade material is selected. The rejected material is returned to the zone refining operation.

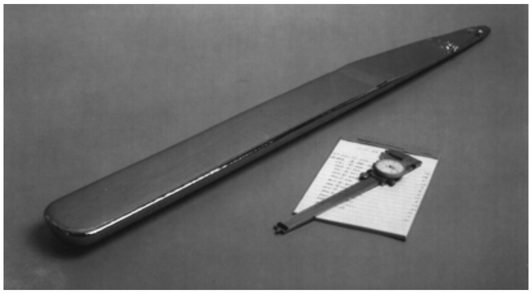

Fig. 3. A Zone-Refined Ingot.

The section of crystal which has both adequate purity and crystallographic perfection for coaxial detector fabrication is then ground perfectly cylindrical. The edge at one end is beveled to a radius (“bulletized”) to improve charge collection and timing performance. Figure 7 illustrates the grinding operation. Afterwards, a hole is machined into the unbeveled end so that the central contact of the device may be made later. The detector subsequently is hand lapped all over to remove damage caused by the machining processes.

A lithium diffusion to form the n^+ contact is then performed over the entire outer surface except the flat, unbeveled end for p-type coaxial detectors and on the “walls” of the central hole for n-type coaxial detectors. This lithium-diffused layer is about 600-µm thick. After the lithium diffusion operation, the detector is lapped once more, chemically polished, and a surface protective coating applied. The coating is amorphous germanium hydride deposited by a sputtering process, similar to that described by Hansen, *et al.,* (Ref. 1). Next, the p^+ contact is formed by the ion implantation of boron ions. This last step completes the fabrication process for the coaxial detector element itself. Figure 8 shows schematically the structure of both p-type and n-type coaxial detectors.

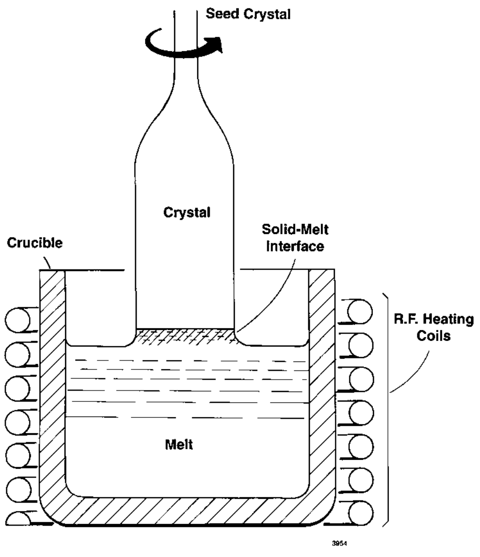

Fig. 4. Czochralski Technique Schematic.

At this point the detector is ready to be mounted in a cryostat. The basic function of a cryostat is to cool the germanium detector to its near-liquid-nitrogen operating temperature. For best performance the first stage of the preamplifier is also cooled to low temperature, the entire cold assembly being maintained by the cryostat under high vacuum for both thermal insulation and protection of the internal components from contamination. Figure 9 shows the construction of a typical cryostat system. Cryosorption material (such as selected zeolite or activated charcoal) is used as a residual gas getter or pump to maintain the vacuum for long periods of time. After being loaded into the cryostat, the detector is tested for several parameters, including leakage current and energy resolution. If the device fails a test, it is returned to some previous stage of the process. Another important cryostat design consideration is allowing for the convenient positioning of the detector element. This usually means that a copper cooling rod, needed to conduct the heat from the detector element, is routed from a point outside the dewar to the liquid nitrogen inside the dewar. All of the cryostat materials around the detector should be as low Z as possible to reduce photon scatter. Hence, aluminum, magnesium, beryllium, Teflon, and Mylar are used whenever possible.

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Because many of the steps in the manufacturing process (Fig. 1) have less than a 100% yield, a detector element may spend an extended time in a “loop” before being shipped.

The Past and the Future

When ORTEC started production of germanium detectors in the late 60’s, a detector of 5% relative efficiency (compared to a 3” x 3” NaI detector) with 3-keV energy resolution at 1.33 MeV was considered typical. In 1975 the “VIP 10” offered 10% efficiency at 2 keV. In 1980 the Ge(Li) technology in use was obsoleted by high purity germanium detectors which could be cycled to room temperature. Recently detectors with 170% efficiency and <2 keV resolution have been produced.

ORTEC pioneered these technologies and others, such as GAMMA-X coaxial detectors, streamline cryostats, and PopTop transplantable detector capsules.

Development work toward larger crystals, application-matched performance parameters, ultra-low background cryostats, and exceptional reliability continues. We welcome suggestions for new, more application-tuned detectors.

References

1. W.L. Hansen, E.E. Haller, and G.S. Hubbard, “Protective Surface Coatings on Semiconductor Nuclear Radiation Detectors,” *IEEE Trans. on Nucl. Sci.* **NS**-27, No. 1, 1980.

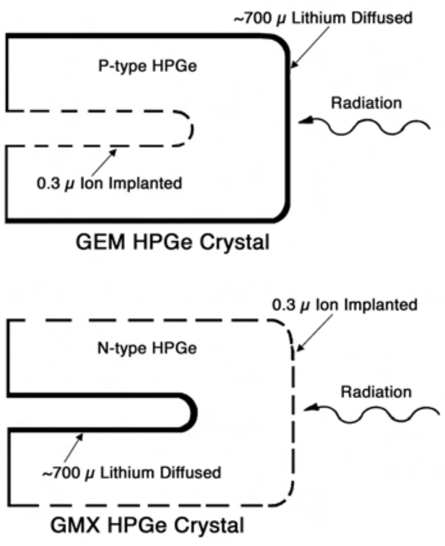

Fig. 8. Schematic of Both P-Type and N-Type Coaxial Detectors.

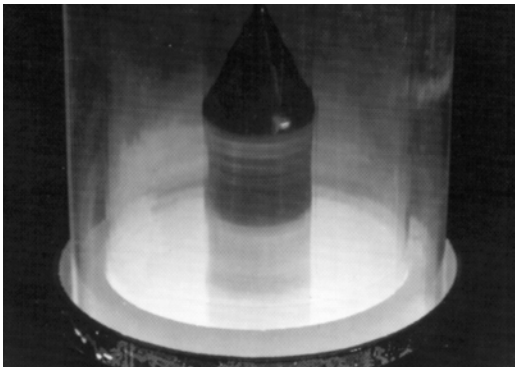

Fig. 5. A Germanium Crystal Being Grown.

Fig. 6. A Mounted Crystal Being Sliced.

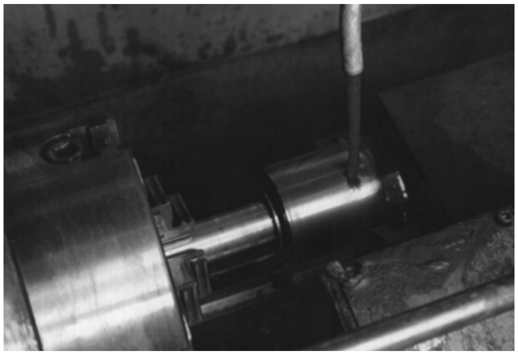

Fig. 7. Grinding the Germanium Crystal.

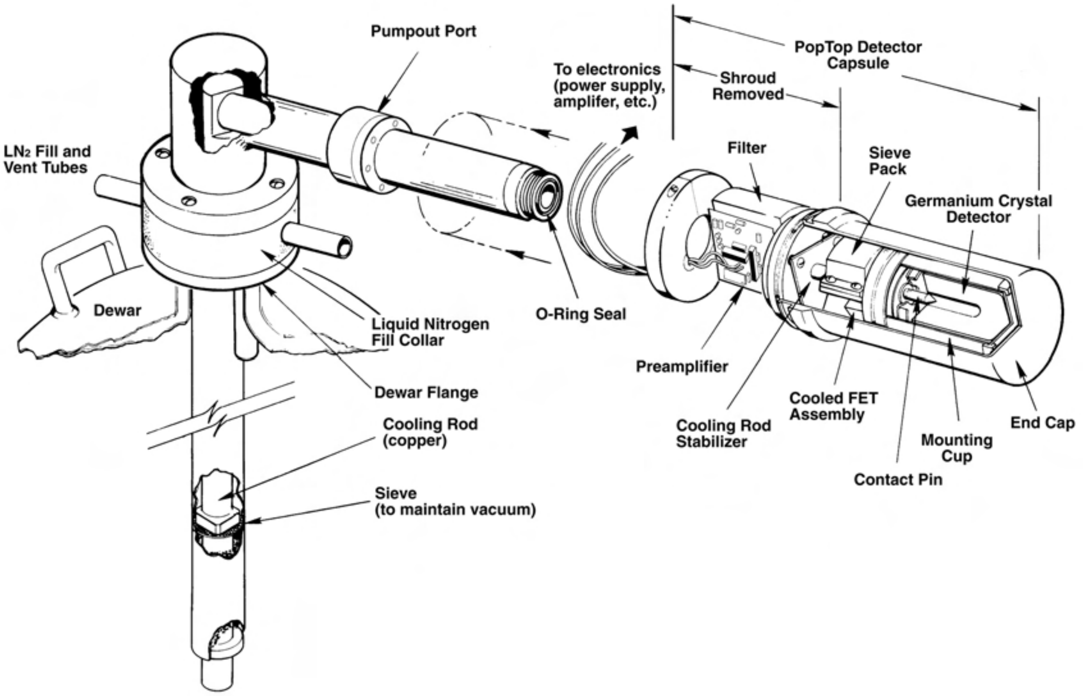

Fig. 9. Exploded View of PopTop Detector Capsule with Horizontal Dipstick Cryostat and 30-Liter Dewar.

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