

Detectors

5

Status of Indigenous Development of HPGe Detectors in BARC

Shreyas Pitale¹, Manoranjan Ghosh¹, S. G. Singh¹, G. D. Patra¹, A. K. Singh¹, M. Sonawane¹, Shashwati Sen¹, R. S. Shastrakar³, Tushar Kesarkar³, K. M. Sudheer³, V. B. Chandratre³, K. G. Bhushan², S. M. Rodrigue², S. Malhotra², L. M. Pant¹ and S. M. Yusuf^{1*}

¹Technical Physics Division, Bhabha Atomic Research Centre (BARC), Trombay – 400085, INDIA

²Electromagnetic Application & Instrumentation Division, Bhabha Atomic Research Centre (BARC), Trombay – 400085, INDIA

³Electronics Division, Bhabha Atomic Research Centre (BARC), Trombay – 400085, INDIA



Fabricated diode

ABSTRACT

For the last 5 decades High Purity Germanium (HPGe) Detectors have played key role in nuclear physics experiments and nuclear fuel cycle application in DAE. These detectors are imported in large numbers that are quite expensive and difficult to maintain. A planned program was initiated in DAE in order to develop the technology of HPGe detectors indigenously. The project aims to build gamma ray detectors starting from commercially available raw Ge material, process it by zone refinement to 13 N purity level and grow in-house crystals by Czochralski technique for fabrication of detectors with various configuration and geometries. Currently, P and n-type planar HPGe diodes have been developed by forming suitable electron and hole blocking electrical contacts. A cryostat is developed to maintain the temperature of the detector below 100K and mount the electronics for readout of the HPGe detector. Finally, a complete HPGe detector with indigenous cryostat assembly, readout electronics and software for high resolution spectroscopy are realised which meet the requirements of DAE's activities.

KEYWORDS: High-purity germanium (HPGe), Czochralski technique

Introduction

High-purity germanium (HPGe) detectors are preferred in high resolution gamma and x-ray spectroscopy for the identification of closely spaced radionuclides. Compare to silicon, Ge shows high electron and hole mobility, higher atomic number and lower average energy required to create an electron-hole pair. Thus Ge based radiation detectors are best choice for high-energy spectroscopy and no alternatives are found without substantial loss of resolution and efficiency [1].

DAE has been using HPGe detectors for decades in programs ranging from nuclear physics experiments, fuel production to nuclear medicine due to its high energy resolution. These radiation detectors are imported without service support and face export control hurdles. It is the need of the hour that DAE develops the technology to fabricate HPGe detectors indigenously. This project is a step in that direction to mitigate these issues, build capabilities in long run and be self-sufficient.

Basic Principle of HPGe

Most high-energy photons (100 keV–10 MeV) interact with the electrons in the high pure Ge material. The density of electrons is proportional to atomic number (Z). Thus the absorption coefficient and detection efficiency of gamma ray photon is higher for a given volume of high Z germanium. There are three main processes by which gamma rays lose energy in the detecting media: the photoelectric effect, Compton scattering, and e^+e^- pair creation. The photoelectric process is dominant at low energies (<100 keV approximately) and is

related to emission of electrons from the atomic shells. This process depends on the energy of the photons and the atomic number of the detecting media. The energy of a photon is absorbed by an inner-shell electron and leads to its emission from the atom. Subsequently, the photoelectrons lose their kinetic energy in the semiconductor by electron-hole pair generation. In the intermediate energy range, Compton effect dominates, hence the absorption of the photon becomes a multistep process with Compton electrons (<1 MeV) being absorbed after traveling a short distance, while Compton photons are created and absorbed in subsequent steps. Depending upon the size and geometry of the detector, photo-peak can be obtained with high resolution [2].

A planar HPGe detector using a p-type crystal is shown in Fig.1. In this configuration, the electric contacts are created on the two flat surfaces of a germanium crystal. A lithium evaporation and diffusion method is used to form the n^+ contact over one of the surface. The thickness of lithium-diffused layer is several hundred micrometer thick. The other

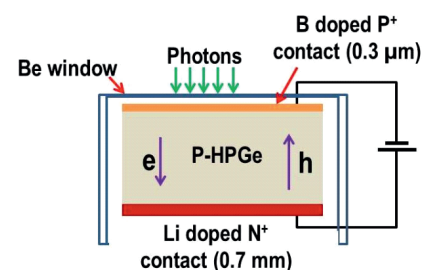


Fig.1: Planar HPGe detector (p type).

*Author for Correspondence: S. M. Yusuf
E-mail: smyusuf@barc.gov.in

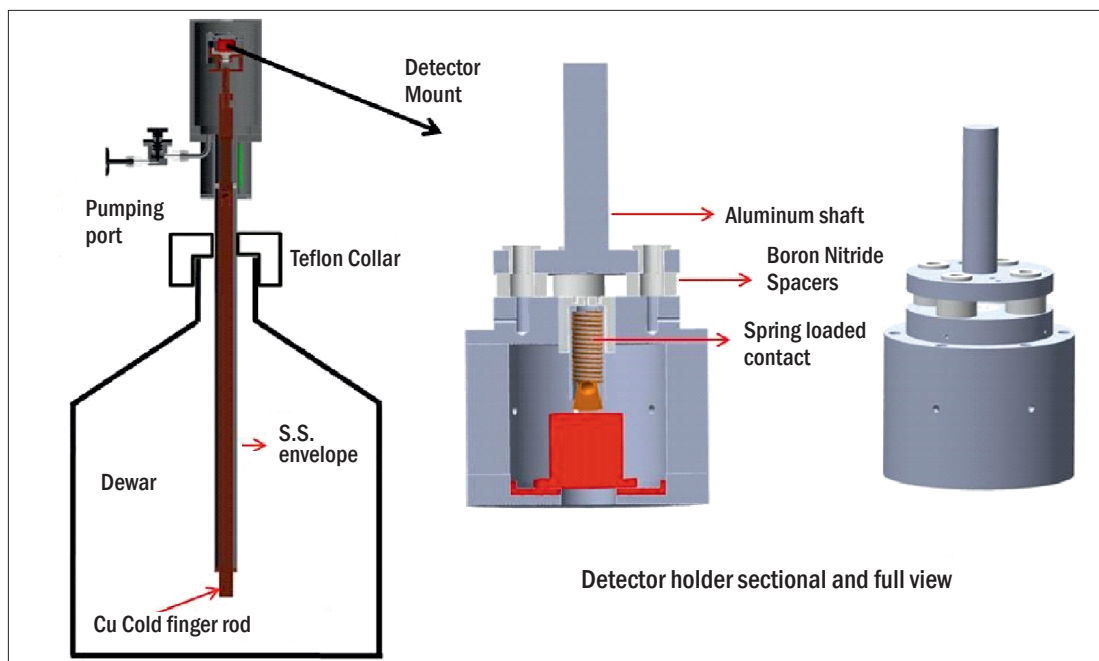


Fig.2: A complete detector system showing the cryostat and detector holder.

side is $0.3\ \mu\text{m}$ thick boron implanted p^+ layer that provides ohmic electrical contact to collect the charge carriers created by the radiation otherwise would be lost in natural recombination process. The depletion region is formed by reverse biasing the p/n junction. The small thickness of the implanted boron layer makes it suitable entrance window for low energy photons. For reverse biasing, a positive high voltage is applied to the n^+ contact with respect to the p^+ surface. The depletion region is formed at the region close to the n^+ contact and then expanded deeply into the p-side as the bias voltage is raised. Once the detector is fully depleted, further increase of the bias (over voltage) does not make any effect on the active volume. However, it makes the electric field stronger, which subsequently shortens carrier collection times and reduces the risk of carrier losses through processes such as recombination and trapping [3].

HPGe crystals were first developed in the mid-1970s. The starting material was chosen as industrial grade germanium. For the purpose of a detector required in high resolution spectroscopy, the material is further purified using zone refining technique. In this process, germanium is melted in a crucible using radio-frequency (RF) heating coils. The underlying principle is that impurities concentrate in the liquid phase leaving the solid purer than the original melt as the liquid freezes and solid appears. As the RF coils are slowly moved along the length of the crucible, the molten zone moves with them. Thus, the germanium melts as the coil approaches and freezes as the coil moves away. The impurities tend to remain in the molten section, which leads to a higher concentration of impurities in the liquid than the solid. In this way, the impurities are swept to one end. 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. The improvement or reduction in impurity concentration actually realized is about a factor of 100 or more at the completion of this process [4].

Large single crystals of germanium are grown using the Czochralski technique [5]. 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. The type of conductivity is determined by the donor or acceptor nature of the low-level impurities present in the grown crystal. In order to reach optimum resolution of semiconductor detectors, defect density of the crystal needs to be decreased, in particular for those that are electrically active. In addition to point defects and impurities, dislocations play a major role as they behave like a sink for impurities. This gives a rule of thumb for the dislocation density that should not exceed $10^4\ \text{cm}^{-2}$ and go below $10^2\ \text{cm}^{-2}$ for detector-grade material [6].

Because germanium has relatively low band gap, these detectors must be cooled in order to reduce the thermal generation of charge carriers to an acceptable level. Otherwise, leakage current induced noise degrades the energy resolution of the detector [7]. Therefore, HPGe detectors are usually equipped with a cryostat as shown in Fig.2. Germanium crystals mounted within a metal container referred to as the detector holder (indicated in Fig.2) are maintained at temperature 90 to 100 K. The holder is generally made of aluminium and is kept within a vacuum envelope. The detector holder as well as the "end-cap" is thin to avoid attenuation of low energy photons. The HPGe crystal inside the holder is in thermal contact with a Copper metal rod called a cold finger. The cold finger extends past the vacuum boundary into a dewar flask that is filled with liquid nitrogen and transfers heat from the detector assembly to the liquid nitrogen (LN2) reservoir. The combination of the detector holder, vacuum envelope, the cold finger and the liquid nitrogen dewar is called the cryostat. The preamplifier of the germanium detector is normally included as part of the cryostat package and is installed nearest possible distance from the detector to minimize the overall capacitance. The input stages of the preamp are also cooled.

On-going Research in the Field of High purity Germanium Detector at DAE

Owing to its dependence on import for the need of high energy resolution gamma ray detector based on High purity germanium, DAE, under XII plan started a project to



Fig.3: Chemical laboratory in CTL, TPD showing installed fume hoods, water purifier, racks and working table.

indigenously develop the full technology for the fabrication of HPGe detector. The project covers the full technology spectrum including material purification, crystal growth, detector fabrication and readout electronics with spectrum analysis software. For that purpose, well directed and concerted efforts have been put forward towards development of state of the art facilities for the in-house growth of HPGe single crystal and fabrication of detector in Crystal Technology Section of Technical Physics Division, BARC. The readout electronics and software work was undertaken by ED, BARC. A brief description of created facility and ongoing research activity are presented here.

Processing of Ge Crystal for detector fabrication

Well equipped Chemical laboratories (Fig.3) and Ge crystal processing facilities were created for crystal cutting,

cleaning, etching and mechanical polishing. Custom designed holders made of PTFE are used to handle various geometries of crystals during chemical etching. An inventory of high purity semiconductor grade concentrated acids and solvents such as HF, HNO₃, Acetic Acid, Methanol, Acetone, Trichloroethylene, H₂O₂ etc having impurity metal ion concentration (per element) approx. 10-50 ppb and particle concentration < 250/ml, are maintained for chemical treatment of ultra-pure germanium.

The high purity Ge crystal needs to be cut as per the desired size and shapes. A wire saw (Fig.4a) and diamond wheel cutting machines are commissioned for this purpose. A complete process is developed for cutting of the high purity crystals in different geometries. Fig.4 (b-c) shows a high purity Ge single crystal cut in top hat geometry. Also circular grooves were fabricated using a diamond coated core drill bit [Fig.4d] on one face of planer Ge crystal [Fig.4 (e-f)] using SiC slurry mediated core drilling technique. SiC abrasive of grit 220 (particle size ~1000 μm) to 1500 (particle size ~ 10 μm) are used successively to lap the crystal for removal of mechanical damages created while cutting. Further planarization is performed by polishing the crystals with Al₂O₃ powder of particle sizes 9 μm, 3 μm and 0.3 μm. To remove the residual damages created by the polishing process, chemical etching is performed with a mixture of concentrated nitric acid and hydrofluoric acid.

Facilities for qualification of detector grade Ge Crystal

The Ge crystal needs to be characterised before fabrication of gamma detector. A Ge crystal is selected for diode fabrication only if the electrically active impurity is found to be around $\sim 10^{10}/\text{cm}^3$, the carrier mobility is higher than 40,000 cm²/V. sec, carrier lifetime is higher than 10³ sec and dislocation density is within 3000-7000/cm² [8]. Enormous amount of efforts have been devoted to create and maintain facilities, develop the knowhow and expertise for characterization of these critical parameters.

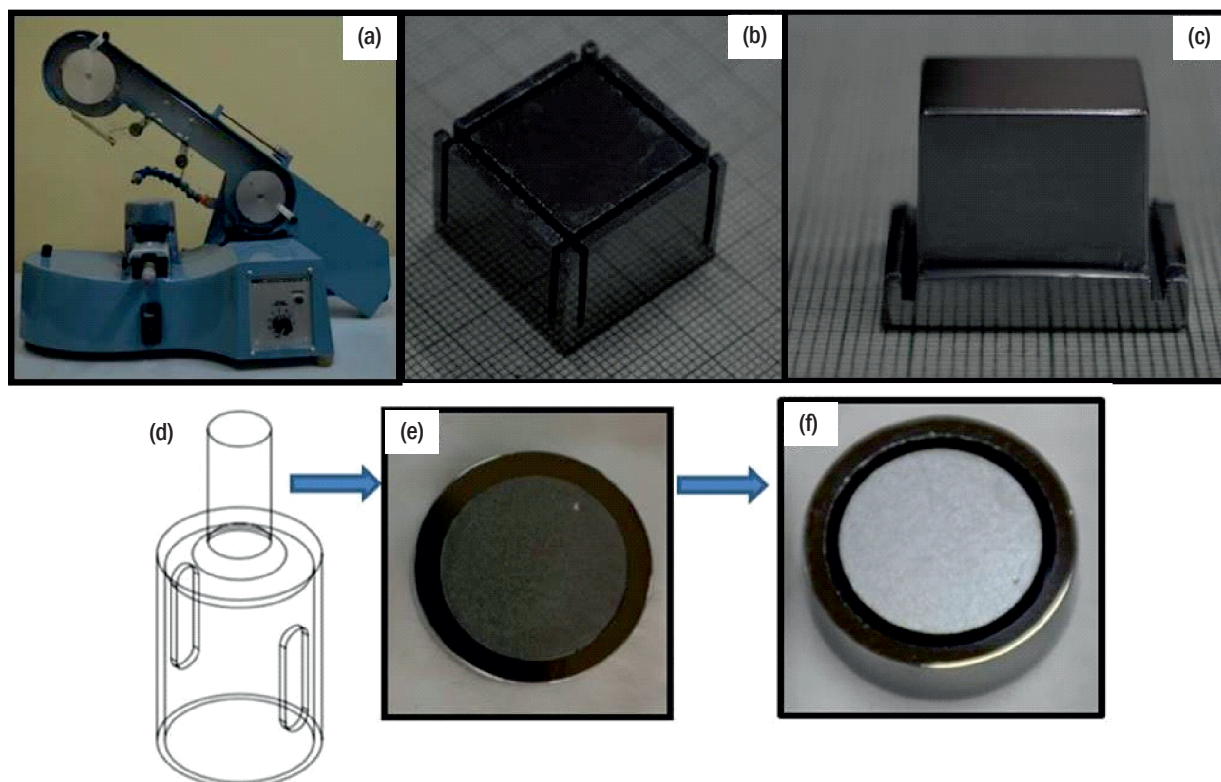


Fig.4: (a) Wire saw cutting machine, (b-c) Crystal processed to various shapes and geometries by cutting and grinding, (d) Schematic of diamond coated core drill bit, (e - f) Circular groove on Ge crystal performed by SiC slurry mediated Core drilling.

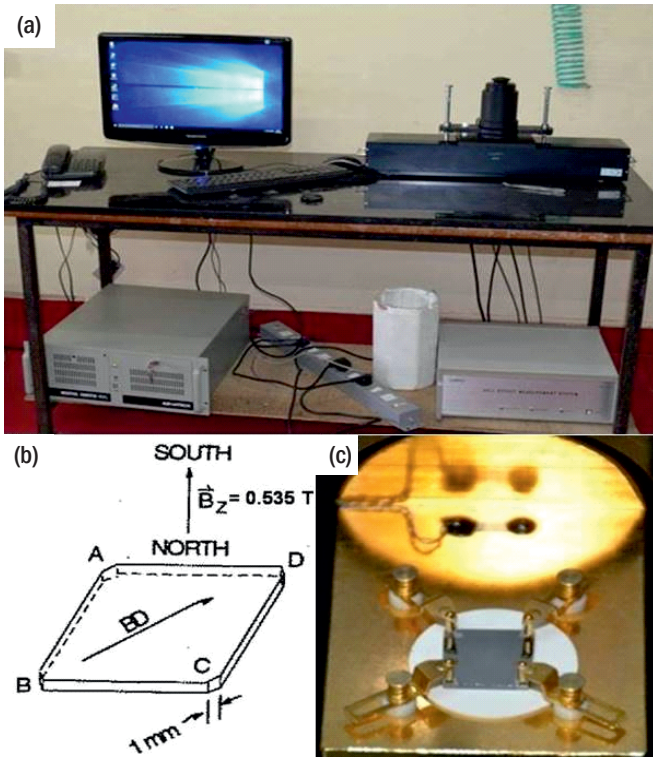


Fig.5: (a) Hall measurement system installed in CTS, TPD (b) Contact arrangement on a square sample and magnetic field for Hall measurement (c) Actual photograph of Ge crystal mounted in four probe configurations on the sample stage.

Facilities for determining crystallinity, dislocation density, band gap and transparency in IR region: Single crystalline nature of Ge crystal is characterized by Laue diffraction and High Resolution X-ray diffraction measurement facilities maintained at CTS, TPD. The optical microscope is used to

visualize and measure the etch pit density of Ge crystal exposed by appropriate chemical etching. A Shimadzu make transmission measurement system for wavelength range 180-3000 nm is also used to determine the band gap and transparency in the IR region of grown Ge crystal.

Hall and I-V measurement systems for determining the purity of Ge crystal: Net carrier concentration, mobility and resistivity of the grown as well as procured Ge crystal and zone refined Ge ingot are ascertained in-house following IEEE standard [9] by Hall measurement set up (Fig.5a). The system is equipped with measurement of Hall co-efficient, resistivity, mobility and net carrier concentrations and current-voltage characteristics within the temperature range of 80-350 K. Transport property measurements were carried out in four probe configuration using Van der Pauw method [10]. For this measurement Ecopia make HMS5000 Hall measurement system is employed. IEEE protocol [9] has been followed for sample preparation. Thin Ge sheet of dimension $10 \times 10 \times 1 \text{ mm}^3$ were prepared from the sliced wafer and were mechanically lapped to a surface finish up to 0.5 micron. Finally the samples were etch-polished using 3:1 HF-HNO₃ solution. Four contacts were attached on the corners of polish etched square samples at 90° spacing using In-Ga eutectic [Fig. 5(b-c)].

Facility for room temperature collinear sheet resistance measurement has been created that provides first-hand information about the purity of the material. This set up consists of one Keithley make nano-voltmeter, current source and a sample holder with height adjustable four collinear probes. Sheet resistance measurement is found useful for detecting lithium diffusion and boron implantation in Ge.

Transport properties measurement as a function of temperature can give substantial information about the material purity, as the resistivity and mobility show strong dependencies on impurities in a semiconductor material. The carrier concentration (CC) in a semiconductor consists of two parts; (i) intrinsic charge carriers and (ii) extrinsic charge carriers. In an n-type semiconductor, with N_d donor density, the total electron density n can be defined as

$$n = (N_d / 2) + [(N_d^2 / 4) + n_i^2]^{1/2} \tag{1}$$

$$\text{where } n_i = (N_v N_c)^{1/2} \exp(-E_g / 2k_b T) \tag{2}$$

$$\text{and } N_d = N_c \exp(-E_g / k_b T) \tag{3}$$

N_d being the extrinsic carrier density and n_i is intrinsic carrier density, while N_v and N_c are effective density of states of valence and conduction band respectively [11]. Calculated carrier concentration over the temperature range of 78-330K for different extrinsic impurity in Ge is given in Fig.6a.

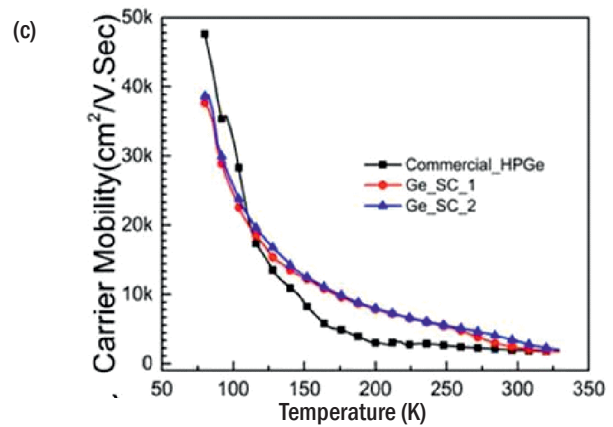
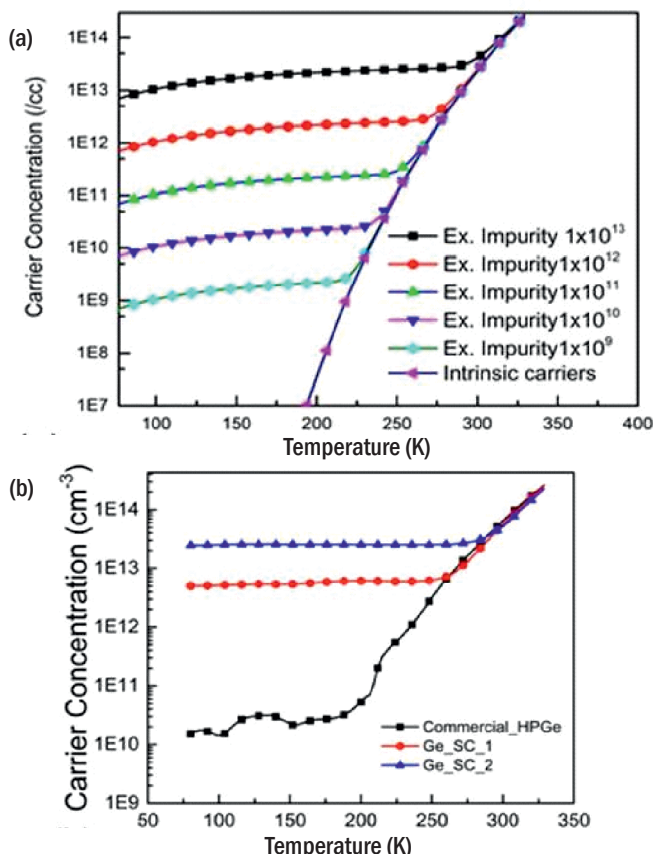


Fig.6: (a) Calculated carrier concentration (eq. 1) profile for different extrinsic bulk carrier concentration (b) Experimentally measured carrier concentration and (c) Carrier mobility of grown crystals (Ge_SC_1 and Ge_SC_2) as well as commercial HPGe crystal.

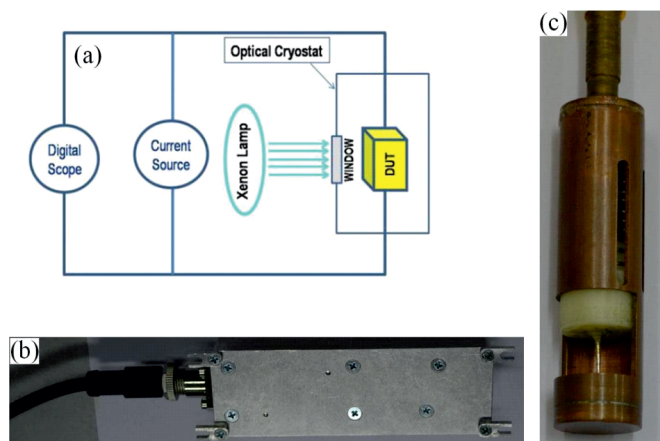


Fig.7: (a) Scheme for determining minority carrier lifetime through decay of voltage pulse after optical excitation of DUT (device under test) (b) IR (1532 nm) pulsed laser source head having repetition frequency as low as 5 Hz procured for excitation of Ge crystal (c) Ge crystal holder fabricated for carrier lifetime measurement by photoconductive decay method.

The surface states in high purity germanium plays an important role in the transport properties measurement as it affects the charge carrier recombination velocity and consequently the carrier mobility measurement [12]. The process for the surface preparation was optimized for the bulk transport properties measurement. The measured bulk carrier concentration of two single crystals grown in CTS, TPD and one commercially procured crystal are shown in Fig.6b which is in good agreement with the theoretically calculated values of carrier concentration for similar extrinsic impurity as shown in Fig.6a. The carrier concentration of the in-house grown crystal is in the range of 5×10^{12} /cc to 5×10^{13} /cc as compared to 2×10^{10} /cc for the commercial HPGe crystal as the in-house grown crystals were 10-12 N pure. The charge carrier mobility as shown in Fig.6c indicates that the mobility of the charge carriers of the grown crystals measured over the temperature range of 78-330 K are approaching the theoretical limit in germanium (~ 50000 cm²/V. Sec) [13] indicating that the defects are well controlled during growth.

Minority carrier lifetime (MCLT) measurement: The laboratory grown germanium crystal and HPGe (both p and n-type)

crystals are further characterized through Minority Carrier Lifetime (MCLT) measurement using Edinburgh FLP 920 instrument and a Keithley 6221 current source (Fig.7a) as per standard procedure [14]. One IR (1532nm) nano-second pulsed laser source (Fig.7b) is also procured and used as a source for MCLT measurement of Ge crystal. Measurements can be performed both at room temperature and 80 K. The samples mounted in a holder (Fig.7c) are placed in an optical cryostat and excited using pulsed laser or xenon source below 50Hz frequency. Signals were recorded on a Tektronix digital oscilloscope according to the scheme as shown in Fig.7a.

Facilities for in-house fabrication of electrical contacts on high purity Ge crystals

As discussed earlier an HPGe detector is a diode. To fabricate this diode hole blocking (p^+) and electron blocking (n^+) contacts are fabricated on two opposite faces of Ge crystal. Lithium diffusion is conducted for fabricating n^+ contact whereas boron ion implantation is carried out for p^+ contact.

Custom designed thermal evaporation system for lithium evaporation and diffusion

A specially designed thermal evaporation system equipped with a furnace inside the chamber for diffusion of lithium in Ge under Ar environment has been fabricated (Fig.8). The system is capable of depositing lithium on Ge crystal of diameter up to 25 mm followed by thermo-diffusion at 300°C. After the lithium coating is done, the chamber is filled with Argon gas and the crystal can be inserted in a furnace (Fig.8b) to undergo lithium diffusion as per selected diffusion temperature. After the completion of diffusion cycle, the crystal can be retracted out of the furnace and cooled rapidly to room temperature using Argon gas jets shown in Fig.8e to achieve maximum lithium concentration at achieved diffusion depth. Required fixtures for holding Ge crystals, shadow masks and suitable boats are also fabricated. Lithium doped Ge contact (n^+) having sheet resistance below 1Ω at room temperature has been repeatedly formed using this system.

To address the difficulties and challenges of handling lithium in air environment, a Glove Box equipped with thermal evaporation system along with heater (Fig.9) has been installed. The system is found useful for making large area lithium doped n^+ contact on Ge as well as metallization of fabricated contacts.

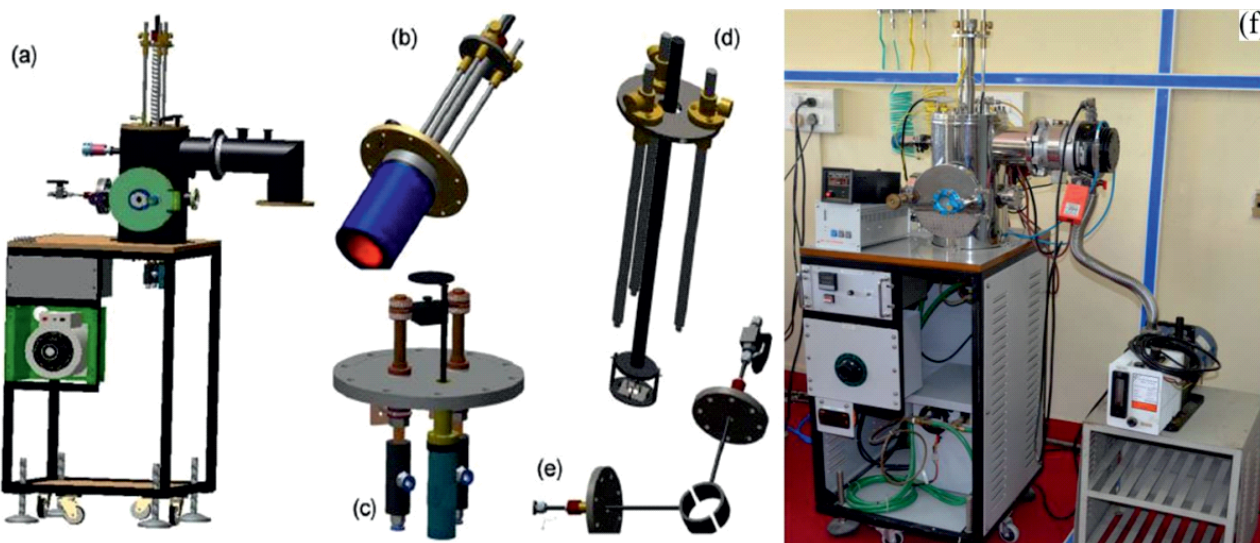


Fig.8: Thermal evaporation system for lithium evaporation and diffusion (a) Completely assembly (b) diffusion heater with sample retracting arrangement (c) Tantalum baffle box source with shutter (d) Sample holder (e) Gas shower jet for rapid cooling after diffusion (f) Actual image of the system installed.



Fig.9: Glove box equipped with thermal evaporation system.

Boron ion implantation system for making p^+ contact on Ge: P^+ contact is formed by boron ion implantation using the Danfysik (1080-30) ion implantation system (Fig.10). The ion source essentially consists of the discharge chamber and a directly heated oven which is charged with solid substances containing the material to be ionized ($BN + B_2O_3$ in the present case). A concentrically wound tungsten filament forms the cathode for electrode emission inside the discharge chamber. One positively charged anode ring of tungsten with a central bore is used as an inlet aperture for the source material evaporated in the oven. In the discharge space between the cathode and anode, ions are formed when electrons are emitted from the cathode, i.e., the tungsten filament. The discharge chamber on the cathode side is linked to a tungsten plate having a narrow central bore of about 0.5 mm diameter. Through this bore, the ions are extracted from the ion plasma by means of a highly negatively charged electrode, connected to a potential of at least 10,000 V, and are subsequently fed to the acceleration path and the analysing magnet, respectively. Once extracted, the ions travel through a mass separator where the defined m/q is selected by a sectorial magnetic field. An electrostatic scanning system is used to deflect the ion trajectories. This allows uniform irradiation of the sample (better than 3%) with low current density (of the order of $1 \mu A cm^{-2}$). The implantation is carried out along the [100] direction. The dose is measured by the current integrator, which integrates the current obtained from the four faraday cups.

Surface Passivation and Encapsulation of HPGe diode

The inter-contact region of HPGe diode need to be passivated by chemical treatment followed by dielectric encapsulation for achieving surface leakage current as low as 100 pA at applied field of 1 KV/cm as well as long term stability of the device.

RF sputtering system and required gas supply line: A RF sputtering unit with required accessories along with gas supply line have been set up for deposition of Silicon oxide, amorphous Si, amorphous hydrogenated Ge etc. on the pristine Ge surface (Fig.11). Targets for all required materials have been arranged. The system is easily maintainable and allows quick access to the sample. It is also found useful for making good quality metal contacts showing superior adhesion.

3D Optical Profilometer for measuring film thickness: Thickness of the sputtered deposited and evaporated films are measured by Filmetrics make Profilim 3D Optical Profiler. The

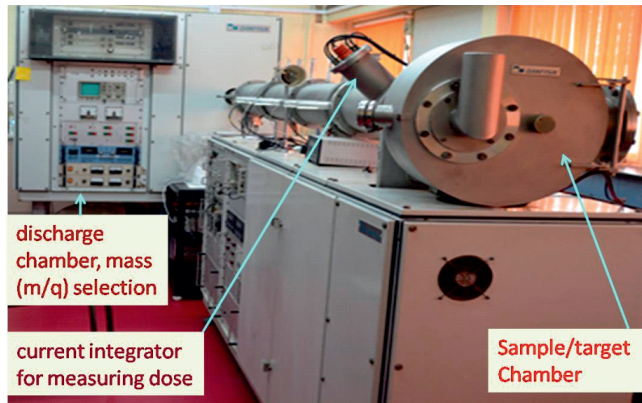


Fig.10: Danfysik (1080-30) 30 KeV ion implantation system.

system works with the principle of coherence interferometry and provides reliable film thickness in the range of 1 nm to 10 μm .

Cryostat for Testing Fabricated HPGe Diode at Near Liquid Nitrogen Temperature

Quick access cryostat for routine current-voltage measurement of HPGe diode: The current voltage measurement of HPGe diode is carried out at temperature nearly 80 K to reduce the population of thermally generated carriers. For relatively quick changeability of devices between successive measurements, a cryostat having shorter temperature cycle is developed in-house (Fig.12a). The basic function of the cryostat is to cool the germanium detector to its near liquid nitrogen operating temperature. The cryostat essentially consists of a double walled hollow chamber containing a solid copper rod. Ceramic to metal sealed high voltage low current vacuum feed through has been used for applying bias and collecting currents. The current path is shielded, ground loops are avoided and cold-end electronics (FET, RC feedback) were embedded (Fig.12b). As per requirements, two such cryostats and specimen holders for maintaining HPGe diode at 80K have been developed. Turbo pump based pumping units are procured for creation of vacuum insulation around liquid nitrogen chambers.

Vacuum envelope with cryo-finger for indigenous HPGe detectors: A liquid nitrogen dipstick is designed and fabricated for housing and maintaining the HPGe crystal near liquid

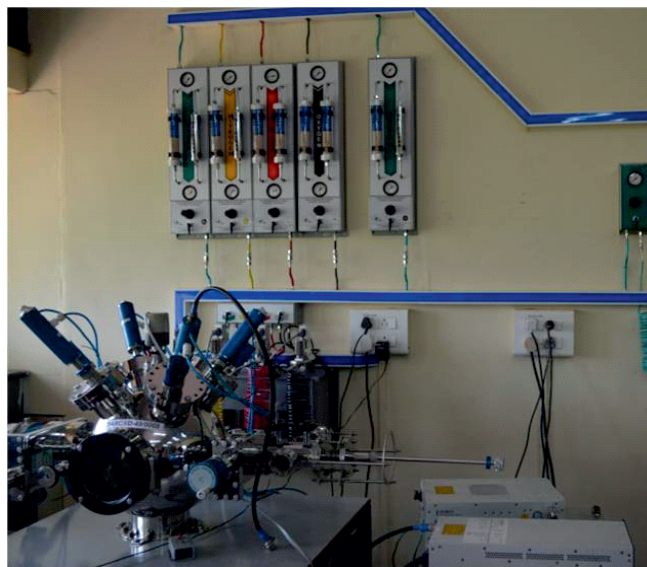


Fig.11: The RF sputtering unit along with gas supply line.

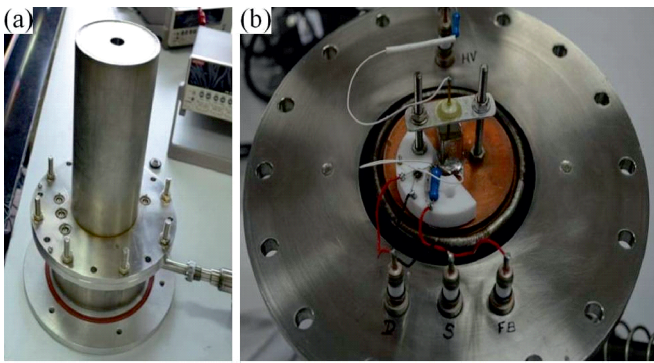


Fig.12: Quick access cryostat for routine current-voltage measurement at 80K (a) Complete assembly (b) Sample mounting arrangement.

nitrogen temperature during detector testing (Fig.13a). This consists of fully indigenous construction with multi-pin glass to metal seals having electron beam welding of dissimilar metals and other high vacuum compatible materials such as Zeolites, NEG modules, Vacuum key, etc as shown in Fig.13(b-c). The vacuum envelope allows for high voltage electrical insulation of the crystal while allowing full thermal conductivity to maintain the crystal at low temperature. The crystal reaches 88 K and remains at this temperature for at-least 15 days or higher with one fill of LN₂ Dewar. No sweating or change in temperature is observed indicating very high vacuum levels maintained in the sealed-off device. A combination of cryo-sorption and NEG (non evaporable getter) pumping modules is implemented to maintains ultrahigh vacuum levels in the sealed off device. Loss of temperature is observed only after LN₂ level falls below half the volume in the container (typically after 3 weeks).

Detector Fabrication and Preliminary Testing

For indigenous development both n-type and p-type HPGe detectors were fabricated and tested. The n-type HPGe detector fabrication started with a cylindrical n-type HPGe monocrystal with the dimensions of 26 mm diameter and 26 mm height. The HPGe crystal was procured from Umicore, Belgium having impurity concentration of $0.85 \times 10^{10} \text{ cm}^{-3}$ and $1.2 \times 10^{10} \text{ cm}^{-3}$ at the top and bottom planes, respectively. The crystal was cut in to sample of 26 mm diameter and 16 mm length using a diamond impregnated blade saw for further processing. The cut sample was lapped from all side to a surface finish of ~ 10 micron using SiC emery papers of grit size

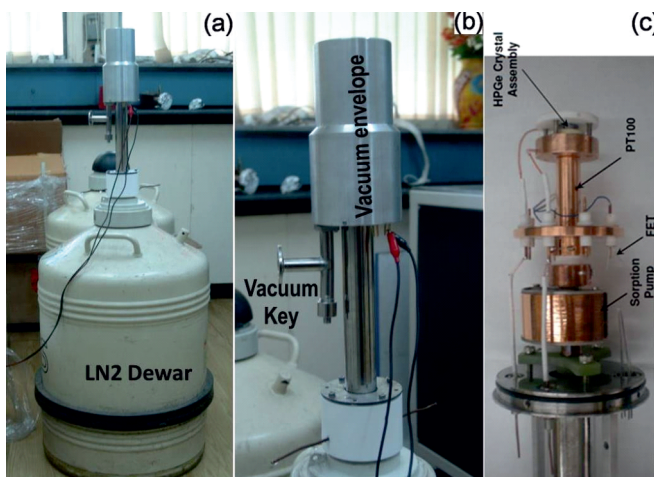


Fig.13: (a) The dipstick cryostat with LN₂ dewar (b) The vacuum envelope and (c) Inside assembly of the cryofinger.

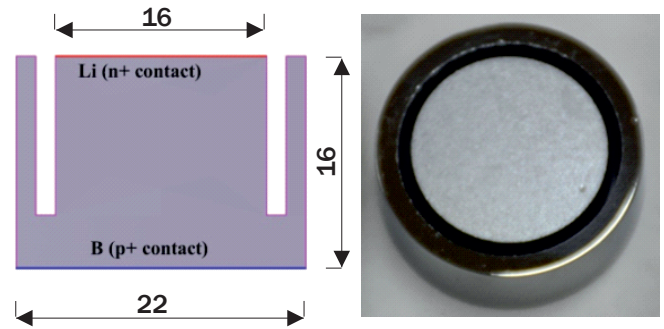


Fig.14: Cross sectional view of the planar n-type HPGe diode with grooved structure (left) and actual photograph of fabricated diode (right).

from 220 to 1500 followed by lapping using 9 micron alumina slurry on a polishing cloth. All the lapping and polishing was done in wet condition to minimize the surface damages during the process. Mechanically processed sample was etch-polished using a solution of HF-HNO₃ mix in 1:3 ratio. Etching time and etchant concentration was optimized to obtain damage free surface. For fabrication of n+ contact (hole blocking), Li was thermally evaporated on one of the flat surface followed by diffusion at 300° C for 20 min (Fig.14). To obtain a clean surface and good electrical contact after Li diffusion the crystal was again etched in the 3:1::HNO₃:HF mixture to remove any excess of lithium. After taking Li contact, a 10 mm deep groove of 16 mm ID and 1 mm width was cut in the sample around it using a diamond bonded core drill tool (Fig.14). After fabrication of groove the n+ contact was protected by Apeizon wax and the sample was again etched for 10 min in the 3:1 HNO₃:HF solution to remove the mechanical damages on the groove surface. The p+ contact (electron blocking) was created on face opposite to n+ contact by ¹¹B Boron implantation. Danfysik (1080-30) ion implantation system was used for the purpose. The implantation was performed with ions of energy: 25 keV, with dose of 10^{14} ions/cm² [15].

Implantation was done at room temperature and no annealing of the sample was performed after implantation [16]. After fabrication, both the contacts were metallized by thermal evaporation of Au ($40 \mu\text{g}/\text{cm}^2$). Finally, the sample was etched in a 8:1 HNO₃:HF solution for chemical passivation of the inter-contact surface. After complete process the diode was assembled in a dedicated crystal holder as shown in Fig.2 and integrated with cryostat (Fig.2) for testing detector performance and diode characterization at 100 K.

For fabrication of a p-type HPGe detector a p-type crystal (Umicore Inc., Belgium) having net carrier concentration $\approx 10^{10}/\text{cm}^3$ was cut using diamond impregnated wheel saw in

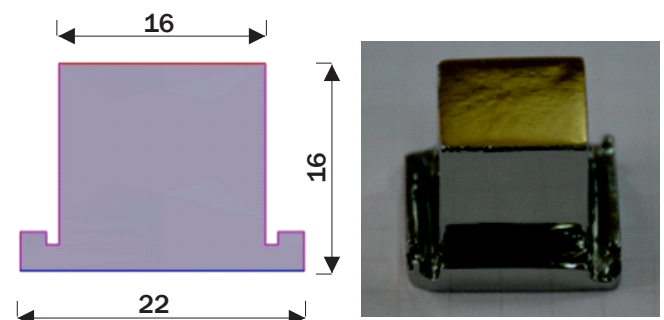


Fig.15: Cross sectional view of the planar p-type HPGe diode in top-hat geometry (left) and actual photograph of fabricated diode (right).

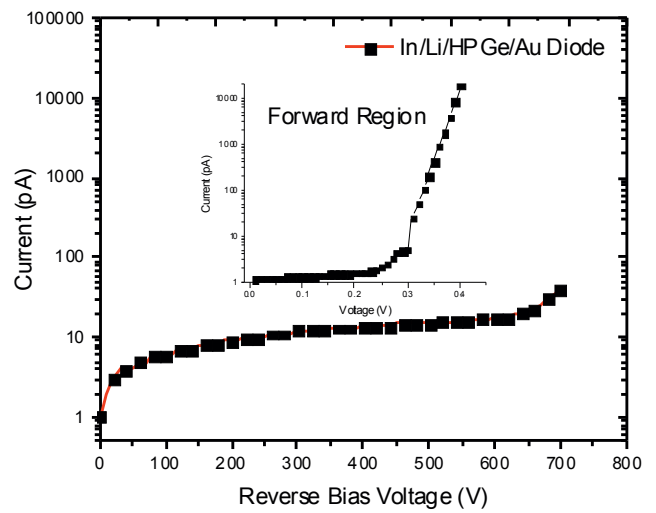
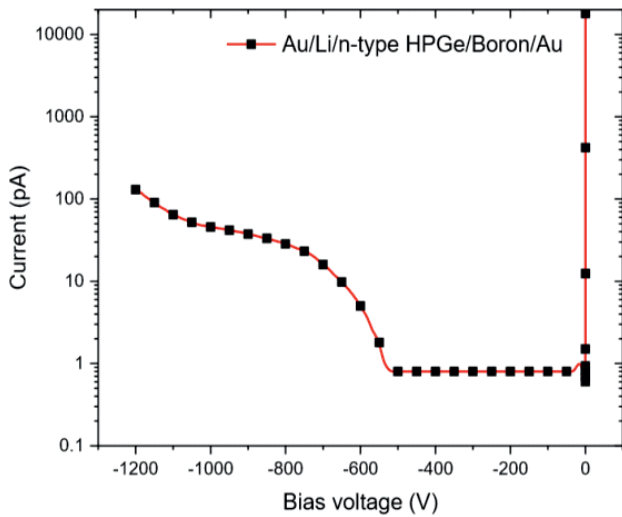


Fig.16: Diode characteristics of n-type (left) and p-type (right) HPGe diode under reverse bias.

top hat geometry (Fig.15). The top hat geometry facilitates crystal handling during various processing steps and also enables the pinching off of surface channels that makes passivation less critical.

The cut sample was lapped from all side to a surface finish of $\sim 10 \mu\text{m}$ using SiC emery papers of grit size ranging from 220 to 1500 in wet condition to avoid heating related surface damage. Final mechanical surface finish was given by lapping the crystal using $9 \mu\text{m}$ alumina slurry. Crystals were chemically polished in a solution of HF:HNO₃ mixture (1:3) until a smooth damage free surface with mirror finish was obtained. The etch was rapidly quenched in DI water and crystal was rinsed multiple times. n+ rectifying contact was produced by thermal evaporation of Li metal on one of the flat surface followed by diffusion at 300°C for 15 min in Argon in a custom built thermal evaporation-diffusion chamber as discussed earlier (section 2.3). The base vacuum of the chamber was 5×10^{-6} m barduring lithium deposition. The crystal is cooled within 30 mins to achieve a saturated Li concentration. Diffusion depth and sheet concentration of Li was found to be 250 μm and $10^{17}/\text{cm}^3$ respectively [17]. Excess Li was removed by applying methanol and crystal was subjected to 30 sec short etch to achieve a clean lithiated surface.

The lithium contact is masked using etch resistant Apeizon wax and the crystal is subjected to oxidation etch in HF:H₂O₂ mixture as described in Ref.[2]. $40 \mu\text{g}/\text{cm}^2$ Au is deposited on the oxidized surface which adheres strongly on the chemically grown oxide layer. Finally, protecting both contacts, the device is etched for 3 mins in HF:HNO₃ (1:8) mixture and loaded in a vacuum cryostat cooled to 100 K for leakage current and detection response tests. Current voltage characteristics of the fabricated p and n-type diode were measured at 80 K with an Electrometer. Both the diodes (with

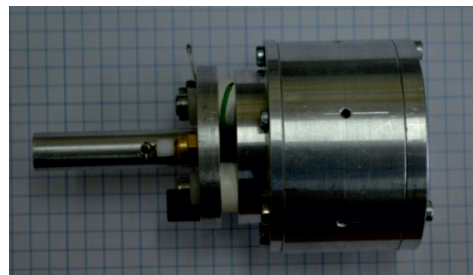


Fig.17: The aluminium crystal holder with the shaft for mounting on the cryo-finger of the dipstick cryostat.

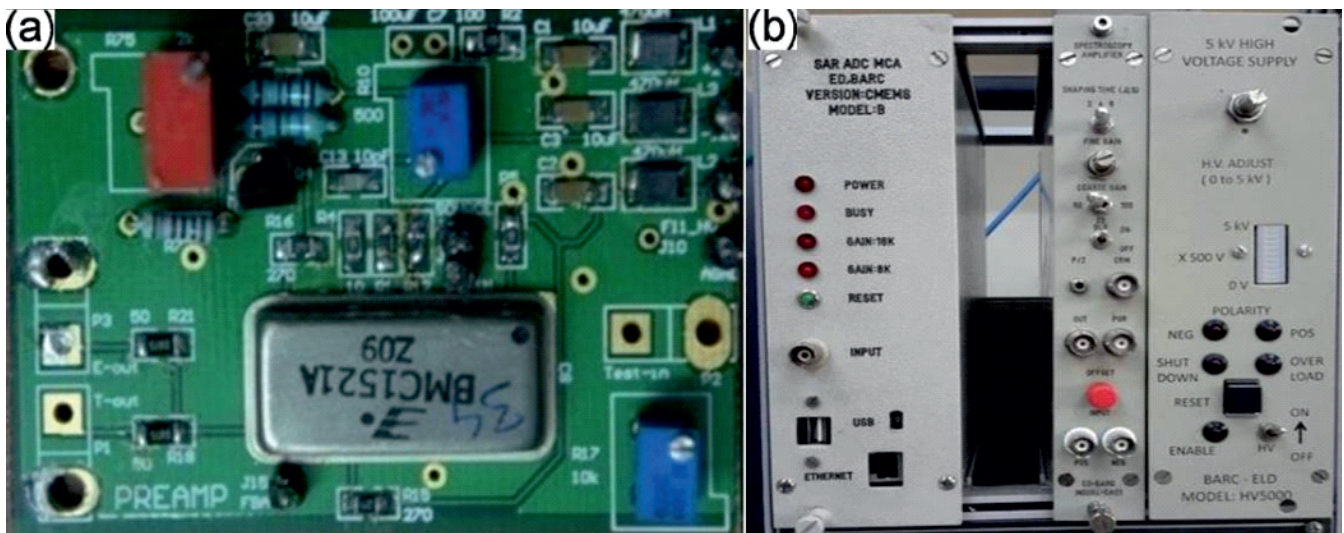


Fig.18: (a) BMC1521 HMC based preamplifier card and (b) NIM based HPGe readout electronics.



Fig.19: (a) 16k portable MCA module and (b) Integrated Multi-channel Analyzer (IMCA).

n-type and p-type crystal) exhibit less than 100 pA leakage current at 700 volt reverse bias (Fig.16). The forward currents of both diodes increases quickly after the set in voltage around 0.3 V and give rise to high rectification ratio ($>10^{-7}$). Diodes exhibiting leakage current less than 100 pA at 700 V are selected for detector spectroscopic tests.

For testing the spectroscopic performance of the detector, the fabricated diode is mounted in crystal holder made of aluminium (Fig.17) which is placed on the copper cold finger of the cryostat as shown in Fig.2. Sample holder is designed and fabricated to hold the crystal and apply high voltage reverse bias to it without damaging the diode properties. The materials are suitably selected so that the temperature of the HPGe crystal can be maintained below 90 K when the sample holder is mounted on the cold finger of the dipstick.

HPGe readout electronics & test results

In order to get good resolution from HPGe detector, it is crucial to have readout electronics that has ultra-low noise and



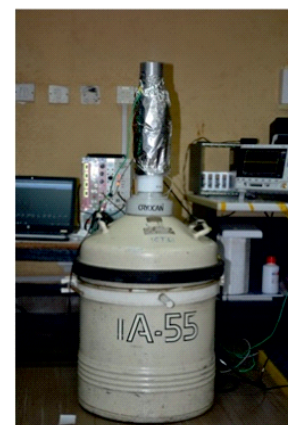
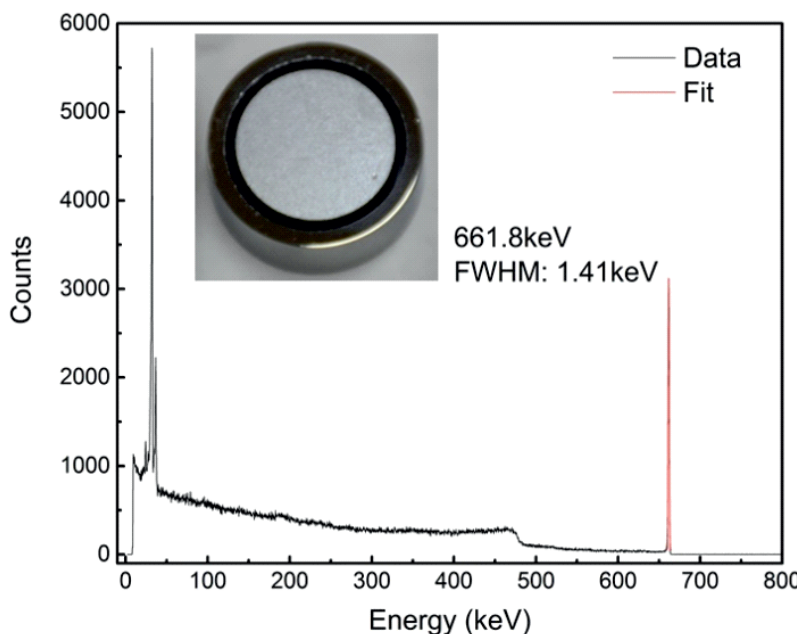
Fig.20: FWHM resolution of ~1.5 keV @ 661.6 keV Cs-137 peak.



Fig.21: FWHM resolution of ~1.95 keV @ 1332.5 keV Co-60 peak.

high linearity over wide dynamic range. The system noise is systematically minimised by first lowering the noise component in the charge sensitive amplifier (CSA) stage by cryo-cooled input FET and feedback resistor and employing low ripple detector bias, linear low noise spectroscopy amplifier with good Pole-zero (PZ) cancellation & base line restoration (BLR) interfaced to a high resolution nuclear ADC & MCA having good linearity.

Electronics Division (ED), BARC has been involved in indigenous development of electronics for readout of high resolution HPGe detectors. ED has developed BMC1521 HMC



137Cs Gamma spectra Recorded employing indigenously developed HPGe detector
Gain: 15
Shaping: 3µs
Bias: +1000 V

Fig.22: Spectra of ¹³⁷Cs recorded using HPGe detector (3 cc active volume) fabricated at CTS, TPD employing indigenously made cryostat (EmA&ID) and electronics (ED).

Table 1: Comparison of FWHM of BARC HPGe detector with commercial HPGe detector using lab sources

BARC HPGe - Preamplifier Assembly, p - type, Planar, 3 cc, BARC IMCA/commercial module: HV: +1000 V, Shaping time 6 us, Gain = 30		Commercial (BSI make GCD 50190 p - type, coaxial) HPGe - preamplifier assembly, 50 % efficiency ~ 250 cc, BARC IMCA module: HV: + 2 kV, Gain: 15, Shaping time: 6 μs (RSSD certified)	
Energy (keV)	FWHM (keV)	Energy (keV)	FWHM (keV)
121.5	0.97	121.5	0.916
661.6	1.5	-	-
1332.5	1.95	1332.5	1.835

based low noise compact charge sensitive preamplifier cards (Fig.18a) along with HPGe readout electronics for gamma ray spectroscopy in standard NIM as well as compact bench-top form factors. The instrumentation developed in the HPGe readout chain is: High Voltage bias supply (HV), Spectroscopy Amplifier (SA), 16k Nuclear ADC and Multichannel Analyzer (MCA), in NIM (Fig.18b) and 16k portable SAR ADC MCA (Fig.19a) & Integrated Multi-channel Analyser (IMCA) (Fig.19b) in compact standalone form factors.

The IMCA comprises of HV supply, SA and 16k SAR nuclear ADC, MCA along with LV power supply. The key specifications are: up to ± 5 kV low ripple HV bias supply, SA with selectable shaping time constant (2 μs/6 μs) & adjustable gain (10-150), 16 k fast Nuclear ADC & MCA with INL & DNL of < ± 0.025 % & < ± 1.5 %, respectively and LCD display for continuous monitoring of IMCA parameters like HV value, temperature etc. The IMCA has advanced features like automatic gated Base-line Restorer (Auto BLR), semi-automated PZ assist, automatic HV shutdown & over current protection which are essential for high-resolution gamma spectroscopy. ED has developed ANUSPECT Gamma Spectrum Analysis (GSA) software Package which provides uniform interface to all ED MCA modules through Ethernet.

The BARC p-type HPGe detector & pre-amplifier assembly has been tested with IMCA module. The resolution of ~1.5 keV FWHM at 661.6 keV Cs-137 peak (Fig.20) & ~1.95 keV FWHM at 1332.5 keV Co-60 peak (Fig.21) has been achieved with this indigenous readout electronics. Also, at low energy 32 keV and 36 keV X-ray peaks were well resolved as shown in Fig.22.

Further the detector performance was compared with commercially available HPGe detectors in BARC (Table 1). The results show that the FWHM of the indigenously developed detector is comparable to commercial detectors and can be used for departmental program.

Conclusion

In conclusion, the full know how of the indigenous technology for the fabrication of HPGe detector has been developed in-house. Detector performance was demonstrated and a FWHM of 1.41 KeV with energy resolution of ~0.2 % at 662 keV was achieved. Efforts are going on to increase the detector active volume up to 50 cc with better energy resolution.

References

[1] Kai Vetter, Annu. Rev. Nucl. Part. Sci. 57 (2007) 363–404
 [2] J. Ebertha, J. Simpson, Progress in Particle and Nuclear Physics 60 (2008) 283–337
 [3] Looker, Q. (2014). PhD Thesis, Fabrication Process Development for High-Purity Germanium Radiation Detectors with

Amorphous Semiconductor Contacts, University of California, Berkeley.

[4] Gang Yang, JayeshGovani, Hao Mei, Yutong Guan, Guojian Wang, Mianliang Huang and Dongming Mei, Cryst. Res. Technol. 49 (2014), No. 4, 269–275.
 [5] G. Wang, M. Amman, H. Mei, D. Mei, K. Irmscher, Y. Guan, G. Yang, Mater. Sci. Semicond. Process. 2015, 39, 54-60.
 [6] E.E. Haller, W.L. Hansen, F.S. Goulding, Proc. of the Mat. Res. Soc. 1982 Annual Meeting, November 1-2, LBL-14916, 1982.
 [7] N. Fourches, M. Zielińska, and G. Charles, 'High Purity Germanium: From Gamma-Ray Detection to Dark Matter Subterranean Detectors', Use of Gamma Radiation Techniques in Peaceful Applications. IntechOpen, Oct. 02, 2019. doi: 10.5772/intechopen.82864.
 [8] Eugene E. Haller , William L. Hansen & Frederick S. Goulding (1981) Physics of ultra-pure germanium, Advances in Physics, 30:1, 93-138.
 [9] IEEE Standard Test Procedures for High-Purity Germanium Crystals for Radiation Detectors," in IEEE Std 1160-1993 , vol., no., pp.1-36, 25 May 1993, doi: 10.1109/IEEESTD.1993.115139.
 [10] Oliveira, F.S., Cipriano, R.B., da Silva, F.T. et al. Simple analytical method for determining electrical resistivity and sheet resistance using the van der Pauw procedure. Sci Rep 10, (2020) 16379
 [11] Boldrini, Virginia &Maggioni, G. & Carturan, Sara Maria & Raniero, W. & Sgarbossa, Francesco & Milazzo, Ruggiero & Napoli, D. & Napolitani, Enrico & Camattari, Riccardo & De Salvador, Davide, Characterization and modeling of thermally-induced doping contaminants in high-purity Germanium, Journal of Physics D: Applied Physics. 52, 3(2018) 035104.
 [12] J.P. Ponpon, Nucl. Instrum. Methods Phys. Res., A, 457 (2000), p. 262
 [13] C. Claeys, E. Simoen, Extended Defects in Germanium: Fundamental and Technological Aspects, Springer Series in Materials Science, Vol. 118, Springer, Berlin, Heidelberg 2009, Ch. 2, pp. 65–136.
 [14] Richard L. Mattis and A. James Baroody, Jr., Carrier Lifetime Measurement by the Photoconductive Decay Method, National Bureau of Standards Technical Note 736 Nat. Bur. Stand. (U.S.), Tech. Note 736, 52 pages (Sept. 1972)
 [15] Jones, K.S. & Haller, E. Ion implantation of boron in germanium. Journal of Applied Physics. 61 (1987) 2469 - 2477.
 [16] J.P. Ponpon, J.J. Grob, R. Stuck, P. Burger, P. Siffert, Chapter: Boron Implanted Contacts on High Purity Germanium, Ion Implantation in Semiconductors, 1971, ISBN : 978-3-642-80662-9
 [17] B. Pratt, F. Friedman, Diffusion of Lithium into Ge and Si, Journal of Applied Physics 37, (1966) 1893–1896.