A program to develop low temperature (mK) sensors with neutron transmutation doped Ge for rare event studies with a cryogenic bolometer has been initiated. For this purpose, semiconductor grade Ge wafers are irradiated with thermal neutron flux from Dhruva reactor at BARC, Mumbai. Spectroscopic studies of irradiated samples have revealed that the environment of the capsule used for irradiating the sample leads to significant levels of $^{65}\text{Zn}$, $^{110}\text{Ag}$ and $^{182}\text{Ta}$ impurities, which can be reduced by chemical etching of approximately $\sim 50 \mu m$ thick surface layer. From measurements of the etched samples in the low background counting setup, activity due to trace impurities of $^{123}\text{Sb}$ in bulk Ge is estimated to be $\sim 1 \text{Bq/gm}$ after irradiation. These estimates indicate that in order to use the NTD Ge sensors for rare event studies, a cool down period of $\sim 2$ years would be necessary to reduce the radioactive background to $\leq 1 \text{mBq/gm}$.

**Keywords:** Neutron transmutation doping, radioactive impurities, $\gamma$-rays

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1. Introduction

Neutron Transmutation Doped (NTD) Ge thermistors have been widely used as low temperature sensors (in mK range) for bolometric detectors in dark matter searches and neutrino physics \[1, 2\]. Compared to the conventional metallurgical methods, neutron transmutation doping yields good uniformity and is found to show good reproducibility \[3, 4\]. The exposure to high neutron dose can also lead to radioactive contamination of Ge sensors \[5\] even if starting material is of high purity. Such trace radioactivity in sensors can produce significant background for rare event studies like double beta decay. It is therefore important to study and minimize the production of relatively long lived impurities in NTD Ge prior to sensor development. A significant cool down period for sensors may be needed depending on activity levels \[5\].

A program to develop low temperature (mK) sensors with NTD Ge for neutrinoless double beta decay studies with cryogenic bolometer has been initiated. Presently a prototype Tin cryogenic bolometer is under development \[6\], which will be later housed at the upcoming underground laboratory INO \[7\]. Semiconductor grade Ge wafers are irradiated with thermal neutrons from Dhruva reactor at BARC, Mumbai. The Ge samples of varying sizes wrapped in Aluminium were irradiated at designated ports. A detailed spectroscopic study of the NTD Ge samples has been carried out in a low background counting setup \[8\] to estimate the radioactive impurities. Chemical etching has been employed to remove the radioactive impurities implanted/diffused close to the surface and an assessment of trace radioactivity in bulk Ge has been carried out. An estimate of the cool down period has been made based on these measurements.

Dependence of radioactive impurities on neutron dose has been investigated. Effect of environment like wrapping material has also been explored within permissible constraints. Section 2 describes experimental details, while Section 3 highlights the results of spectroscopy measurements. Conclusions are given in Section 4.

2. Experimental details

Semiconductor grade Ge wafers of \(< 111 > (0.4 \text{ mm thick, } \rho \sim 30 \, \Omega \text{ cm})\) and \(< 100 > (1 \text{ mm thick, } \rho \geq 35 \, \Omega \text{ cm})\) were used in the present studies.
The actual isotopic composition was obtained using Secondary Ion Mass Spectrometer (SIMS) measurement \(^9\) and is given in Table 1 together with details of n-capture products \(^\text{10}\). Although overall composition is similar, small differences are present in the composition of \(< 111 >\) and \(< 100 >\) oriented wafers, which may depend on the crystal growth condition \(^4\) or the composition of the raw material.

Table 1: Measured isotopic abundances of Ge in the wafers used, n-capture reaction and the stable end products are listed. The decay mode and half-lives of products \(^\text{10}\) are also listed. Errors are \(< 0.2 \%\).

| Isotope | Abundance(%) | \(<111>\) | \(<100>\) | \((n, \gamma)\) reaction product & decay mode (T\(_{1/2}\)) |
|---------|-------------|----------|----------|--------------------------------------------------|
| \(^{70}\text{Ge}\) | 21.5 | 21.9 | \(^{70}\text{Ge} \rightarrow ^{71}\text{Ge}\) \((\text{EC}(11.4 \text{ days})) \rightarrow ^{71}\text{Ga}\) |
| \(^{72}\text{Ge}\) | 26.8 | 27.0 | \(^{72}\text{Ge} \rightarrow ^{73}\text{Ge}\) |
| \(^{73}\text{Ge}\) | 10.8 | 8.8 | \(^{73}\text{Ge} \rightarrow ^{74}\text{Ge}\) |
| \(^{74}\text{Ge}\) | 34.1 | 35.1 | \(^{74}\text{Ge} \rightarrow ^{75}\text{Ge} \rightarrow ^{75}\text{As}\) \((\beta^-(82.8 \text{ min})) \rightarrow ^{75}\text{As}\) |
| \(^{76}\text{Ge}\) | 6.8 | 7.2 | \(^{76}\text{Ge} \rightarrow ^{77}\text{Ge} \rightarrow ^{77}\text{Se}\) \((\beta^-(11.3 \text{ hr})) \rightarrow ^{77}\text{Se}\) |

Prior to irradiation, samples were cleaned in an ultrasonic bath with electronic grade isopropyl alcohol for about 15 min and blow dried with dry N\(_2\). Samples were loaded in a specially designed capsule as per the mandatory procedure for irradiation in Dhruva reactor. Different mounting arrangements permissible within operational constraints of the irradiation process in the reactor were tried out to assess the effect of wrapping material and irradiation environment. These consisted of a single sample wrapped in Aluminium, stacked samples (3 no.s) wrapped in Aluminium and stacked samples (3 no.s) inside a quartz tube. The quartz tube was also wrapped with Aluminium. Both Aluminium and quartz are permissible materials at Dhruva reactor as the flux attenuation is minimal and there is no resultant long term activity in the wrapping material. In most cases, the maximum permissible sample size of 30 mm x 10 mm was used. The irradiation details like neutron fluences, sample sizes and wrapping materials are given in Table 2. Samples A, B, C are of \(< 111 >\) type, while D, E, F are of \(< 100 >\) type.
Table 2: Details of estimated thermal neutron fluence ($\Phi_{th}$) for different Ge samples. The mean irradiation date as well as the duration of irradiation ($t_{irr}$) are also listed in the table.

| Sample Details | Wrapping | Size of irradiation ($x 10^{18}$ mm$^2$) | Mean date of irradiation ($t_0$) | $t_{irr}$ (days) | $\Phi_{th}$ (n/cm$^2$) |
|----------------|----------|----------------------------------------|---------------------------------|-----------------|-----------------------|
| A              | Al       | 10 x 10                                | 03/07/2011                      | 4.0             | 1.9                   |
| B              | Al       | 10 x 10                                | 11/08/2012                      | 4.1             | 14.0                  |
| C              | Al       | 10 x 10                                | 11/08/2012                      | 4.1             | 9.1                   |
| D(T1, M1, B1)  | quartz   | 30 x 10                                | 13/09/2013                      | 5.7             | 4.6                   |
| D(T2, M2, B2)  | Al       | 30 x 10                                | 13/09/2013                      | 5.7             | 4.6                   |
| E(T3, M3, B3)  | quartz   | 30 x 10                                | 16/11/2013                      | 4.1             | 2.1                   |
| E(T4, M4, B4)  | Al       | 30 x 10                                | 16/11/2013                      | 4.1             | 2.1                   |
| F(T5, M5, B5)  | quartz   | 30 x 10                                | 21/11/2013                      | 6.8             | 3.5                   |

After a cool down period of $\sim$ 45 days, individual samples were removed from the irradiation capsule and carefully transferred to separate plastic pouches for spectroscopic measurements. In case of stacked samples, the label M refers to middle sandwiched sample while T and B refer to the outer samples. Some of the larger samples were cut into $\sim$ 10 mm x 10 mm size pieces after irradiation, which were labelled as L (left), C (center) and R (right). Measured activity of the sample with the highest neutron dose was $\sim$ 3 $\mu$Sv/hr. There are three possible sources of radioactive impurities: 1) neutron induced reaction products of impurities in bulk Germanium, 2) neutron induced reaction products from wrapping material which can get recoil implanted in Ge and 3) deposition and thermal diffusion of radioactive contaminants from the surrounding environment in the sample capsule resulting from long exposures at high temperatures during irradiation ($\sim$ 80$^\circ$ C). It should be mentioned that irradiated samples often showed a lack of the lustre and significant improvement was observed after cleaning the NTD samples with HF acid (40%).

A specially designed low background counting setup consisting of $\sim$ 70% HPGe detector surrounded by low activity Cu + Pb shield was used for detection of characteristic $\gamma$-rays of radioactive impurities in the irradiated targets. Data were recorded with a commercial FPGA based 100 MS/s digitizer (CAEN-N6724). Depending on the activity of the sample, counting
was done initially at 10 cm from detector face and later in a close geometry with the sample directly mounted on the detector face. Concentrations of radioactive impurities were obtained from the intensity of the observed γ-rays after correcting for efficiency, branching ratio and decay during time elapsed since irradiation. In close geometry, efficiency corrections due to coincident summing were taken into account. Spectra for the ambient background and virgin samples were recorded for reference. Spectra of the irradiated Aluminium wrapper and the quartz tube were also studied separately.

Figure 1: (color online) Gamma ray spectra of the NTD Ge E-T3L sample ($\Phi_{th} = 2.1 \times 10^{18}/cm^2$) for $E_\gamma = 0$ to 800 keV (top panel) and $E_\gamma = 800 - 1600$ keV (bottom panel) at $t = t_0 + 125$ days (pink solid line) together with that of the $<100>$ virgin Ge sample (black dotted line). The ambient background (red dotted line) is also shown for the comparison. All the spectra are normalized to 12 hours counting time.
Table 3: A list of radionuclides and characteristic $\gamma$-rays observed in NTD Ge samples before etching.

| Radionuclide | Half-life | $E_\gamma$ (keV) |
|--------------|-----------|------------------|
| $^{46}$Sc    | 83.79 d   | 889.3, 1120.5    |
| $^{51}$Cr    | 27.7 d    | 320.1            |
| $^{59}$Fe    | 44.5 d    | 1099.3, 1291.6   |
| $^{60}$Co    | 5.27 y    | 1173.2, 1332.5   |
| $^{65}$Zn    | 243.66 d  | 1115.5           |
| $^{110}$Ag   | 249.76 d  | 657.8, 884.7, 937.5 |
| $^{124}$Sb   | 60.2 d    | 602.7, 1691.0, 722.8 |
| $^{182}$Ta   | 114.74 d  | 1121.3, 1221.4, 1231.0 |

Figure 1 shows $\gamma$-ray spectra of the irradiated sample E-T3L and an unirradiated < 100 > Ge wafer (5 cm dia). The ambient background spectrum is also shown for comparison with suitable scaling. It is clear that no additional trace impurities could be seen in the virgin sample at the measured sensitivity. As can be seen from Table 1, most of the n-capture products of Ge are either stable or have relatively short half-life compared to the initial cool down period (45 days). The $^{71}$Ge has a half-life of 11.4 days and decays mainly by electron capture to the ground state of $^{71}$Ga. However, a small fraction that undergoes Radioactive Electron Capture (REC) [11] shows up as a continuous gamma spectrum of $^{71}$Ge, which is observed with an end point energy of 225 keV.

Table 3 lists the observed radionuclides in various samples together with prominent $\gamma$-rays and half-lives. It should be mentioned that for unambiguous identification, half-lives of the observed $\gamma$-rays were measured and have been found to be consistent within 10%. In addition, wherever applicable, relative intensities of multiple $\gamma$-rays of the given nuclide were also verified. As is evident from the table most of these nuclides are fairly long lived and hence a cause of major concern for low background studies. Radioactive impurities in irradiated samples resulting either from recoil implantation or from thermal diffusion of surface contaminants will be restricted to depths close to the surface in the sample. To investigate the depth dependence of radioactive impurities (10 - 50 $\mu$m), the NTD Ge samples were chemically etched in a controlled manner using $\text{H}_2\text{O}_2$ at 80°C [12]. Typical etching
rate observed was 0.3 \( \mu \text{m/min} \) and etched depth was estimated by accurate mass measurement of the sample assuming uniform etching from all sides. Samples were cleaned in HF before and after \( \text{H}_2\text{O}_2 \) etching to remove oxide layers.

It should also be mentioned that for n-induced reaction products from wrapping material to get implanted in Ge sample, reactions must take place close to surface of the wrapping material. Hence, the surface trace impurities in wrapping materials were separately studied (\( \sim \) few \( \mu \text{m} \) depth) using Energy Dispersive X-ray Analysis (EDAX) [13].

3. Results and Discussion

Figure 2 shows \( \gamma \)-ray spectra of NTD Ge sample D-B1 before and after 46 \( \mu \text{m} \) etching at \( t \sim t_0 + 222 \) days. The sample D-B1 corresponds to the highest neutron fluence (\( \Phi_{\text{th}} = 4.6 \times 10^{18}/\text{cm}^2 \)) in the \( <100> \) set and clearly shows significantly higher activity as compared to the E samples (see Figure 1). The REC continuum at low energy is not visible due to larger elapsed time since irradiation. In the spectrum of the etched sample, it is clearly seen that most of the prominent \( \gamma \)-rays from the surface impurities are below measurable limits, while \( \gamma \)-rays from the bulk impurities can be seen above the background. For most of the samples, the observed activity reduced significantly after etching away few \( \mu \text{m} \) surface layer and remained nearly constant thereafter.

No measurable activity was found in the NTD A sample, which had a nearly 3 years of cool down time. However, since a very small size sample (\( \sim 15 \) mg) was used for spectroscopic studies, no limits on radiopurity of the sample were extracted. Samples B, C from \( <111> \) and D, E, F from \( <100> \) showed different bulk impurities. The only measurable radioactivity present in \( <100> \) Ge after \( \sim 50 \mu \text{m} \) etching was \( ^{124}\text{Sb} \). While the \( ^{124}\text{Sb} \) activity was not seen in \( <111> \) Ge samples, they showed activities of \( ^{65}\text{Zn} (222 \pm 87 \text{ mBq/gm}) \) and \( ^{110}\text{Ag} (225 \pm 67 \text{ mBq/gm}) \) even after 50 \( \mu \text{m} \) etching and \( \sim 1.6 \) years of cool down period. This could be the effect of deeper diffusion at higher temperatures [14] corresponding to higher neutron fluence (\( \Phi_{\text{th}} \sim 10^{19}/\text{cm}^2 \)). It should be mentioned that even in case of D samples, which had highest neutron fluence amongst \( <100> \) set (\( \Phi_{\text{th}} \sim 4.6 \times 10^{18}/\text{cm}^2 \)), some traces of \( ^{110}\text{Ag} \) and \( ^{182}\text{Ta} \) could be seen till 40 \( \mu \text{m} \) depth.
Figure 2: (color online) Gamma ray spectra of the NTD Ge sample D-B1 before (red solid line) and after 46 µm etching (black dotted line) at $t \sim t_0 + 222$ days for $E_\gamma = 0$ to 800 keV (top panel) and $E_\gamma = 800 - 1600$ keV (bottom panel). The sample D-B1 was mounted in the quartz tube and was exposed to the neutron fluence of $\Phi_{th} = 4.6 \times 10^{18}$/cm$^2$.

Table I lists measured activities for various NTD Ge samples, 150 days after the irradiation. The etched samples from $< 100 >$ set did not show any measurable $^{110}$Ag activity. In the present setup this corresponds to $< 12$ cts/day for 657.8 keV gamma ray peak, which implies $< 2.2 \pm 0.1$ mBq/gm of $^{110}$Ag activity for a typical 30 mm x 10 mm sample. The estimated cool down time for activity to reduce to $\sim 1$ mBq/gm is also listed in the last column. Given the high level of radioactivity, the sensors from samples B and C will be unsuitable for low background measurements. On the other hand from the results of different $< 100 >$ samples (D/E/F), it is evident
that for the expected dose $\Phi_{th} \sim 1 - 5 \times 10^{18}/\text{cm}^2$, approximately 2 years of cool down period after irradiation is essential.

Table 4: Measured radioactivity and estimated cool down period ($T_{cool}$) for reduction of the radio-activity below $< 1 \text{ mBq/gm}$ for various NTD Ge samples.

| Sample | Activity (mBq/gm) | $T_{cool}$ |
|--------|-------------------|------------|
|        | $(t_0 + 150 \text{ days})$ | $^{110}\text{Ag}$ | $^{124}\text{Sb}$ |
| B      | 3018(465)         | -          | 9           |
| C      | 743(222)          | -          | 7           |
| D-B1   | -                 | 420(9)     | 1.9         |
| E-T3L  | -                 | 201(20)    | 1.7         |
| F-B5   | -                 | 344(12)    | 1.8         |

As mentioned earlier, Alessandrello et al. [5] have also measured the residual radioactivity in NTD thermistors for a similar neutron fluence, namely, $\Phi_{th} \sim 3.36 \times 10^{18}/\text{cm}^2$. They have reported several isotopes like $^{75}\text{Se}$, $^{74}\text{As}$ and $^{68}\text{Ge}$ resulting from fast neutron induced reactions during irradiation. Elliott et al. [15] have also reported the formation of isotopes like $^{65}\text{Zn}$, $^{54}\text{Mn}$ and $^{60}\text{Co}$ in high energy neutron induced reactions. In the present case though $^{65}\text{Zn}$ activity was seen at surface of the samples, the $\gamma$-rays corresponding to fast neutron induced reaction products (namely, $^{75}\text{Se}$, $^{74}\text{As}$, $^{68}\text{Ge}$, $^{54}\text{Mn}$, $^{60}\text{Co}$ and $^{65}\text{Zn}$) are not visible in the irradiated-etched samples at measured level of sensitivity. It should be mentioned that the commercial NTD Ge sensor (AdSem, Inc [16]) showed much higher levels of $^{65}\text{Zn}$ and $^{110}\text{Ag}$, possibly due to other materials used in contact fabrication.

The observed residual activity of $^{124}\text{Sb}$ results predominantly from the $^{123}\text{Sb}(n,\gamma)$ reaction with thermal neutrons. The contribution from the fast neutrons can be neglected since the flux for $E_n > 1\text{MeV}$ is smaller by a factor $\sim 5$ and the cross-section for n-capture is smaller by a factor of $\sim 50$. The concentration of the reaction product is related to that of the parent isotope ($N_{impurity}$) by the following relation:

$$N_{\gamma}^{product} = N_{impurity} \times \sigma_c \times \phi_{th} \times \frac{1 - e^{-\lambda t_{irr}}}{\lambda}$$

where $\sigma_c$ is the thermal neutron capture cross-section (to the ground state and/or excited state as the case may be) [17], $\lambda$ is the decay constant, $t_{irr}$ is
the duration of the irradiation and $\phi_{th}$ is the thermal neutron flux expressed in units of $\text{neutrons.cm}^{-2}.s^{-1}$. The $\phi_{th}$ is assumed to be uniform during the irradiation period, i.e. $\phi_{th} = \Phi_{th}/t_{irr}$.

The $N_{\gamma}^{\text{product}}$ is computed from the measured intensity of $\gamma$-ray ($N_\gamma$) during the counting time interval of $t_1$ to $t_2$ (measured with respect to end of the irradiation) and is given by

$$N_{\gamma}^{\text{product}} = \frac{N_\gamma}{\epsilon_\gamma \times I_\gamma \times (e^{-\lambda t_1} - e^{-\lambda t_2})}$$  \hspace{1cm} (2)

where $\epsilon_\gamma$ and $I_\gamma$ are the photo-peak detection efficiency and branching ratio of the $\gamma$-ray, respectively.

Table 5: Estimated trace impurities from the residual radioactivity of Sb in etched $<100>$ NTD Ge samples. The etched depth for each sample is indicated in the bracket.

| Parent isotope | $E_{\gamma}^{\text{product}}$ (keV) | Concentration (ppt) |
|---------------|-------------------------------------|---------------------|
| $^{123}$Sb    | 602.7                               | D-B1(46 µm)         |
|               |                                     | E-T3L(42 µm)        |
|               |                                     | F-B5 (52 µm)        |
|               |                                     | 115(2)              |
|               |                                     | 119(12)             |
|               |                                     | 123(4)              |

Table 5 lists the estimated bulk impurity concentration in $<100>$ Ge samples. Figure 3 shows a plot of relative neutron fluence (R) of sample D and F with respect to the E sample extracted from $^{124}$Sb activity (open square) and from the irradiation data (filled circle). The good agreement between these two indicate that observed bulk impurity concentration of $^{123}$Sb is similar in different samples of $<100>$. It is also possible to use the $^{124}$Sb activity as a neutron fluence monitor for these samples. It should be mentioned that the bulk impurity concentration of $^{125}$Sb in Ge quoted in Ref. [5] is $< 1$ ppt, which is significantly smaller than the present work. Therefore, it would be desirable to use detector grade Ge as a starting material instead of the device grade.

The EDAX analysis of Aluminium wrapper and the quartz tube had shown a purity level of $\sim 99\%$. The neutron induced reactions in the Aluminium resulted in either stable or short-lived products ($T_{1/2} \sim$ few sec to 15 hrs), which could not be observed in the present study. The irradiated Aluminium wrapper showed very high levels of $^{110}$Ag, $^{65}$Zn and $^{182}$Ta, while the quartz tube showed several additional $\gamma$-rays of $^{54}$Mn, $^{58}$Co and $^{134}$Cs. For a neutron fluence of $\Phi_{th} \sim 4.6 \times 10^{18}/\text{cm}^2$, the measured $^{110}$Ag activity
Figure 3: (color online) Relative neutron fluence (R) of samples D and F with respect to the sample E from the residual $^{124}$Sb activity (open square) and from the irradiation data (filled circle).

in the Aluminium wrapper and the quartz was $\sim 2.2$ kBq/gm (corresponding to $\sim 0.2$ ppm of $^{109}$Ag in $^{27}$Al) and $\sim 0.07$ kBq/gm (corresponding to $\sim 0.02$ ppm of $^{109}$Ag in SiO$_2$), respectively, after a cool down period of $\sim 150$ days. The surface activity of $^{110}$Ag in the corresponding Ge samples, namely, D-T2 (wrapped in the Aluminum) and D-T1 (in the quartz tube) was 56(2) Bq/gm and 7.2(0.4) Bq/gm, respectively. It is evident that the Ge samples wrapped in Aluminium showed higher surface activity as compared to those in the quartz tube. Further improvements like irradiation in a sealed quartz capsule to reduce effect of environment are under consideration.

4. Conclusions

The development of low temperature (mK) sensors with neutron transmutation doped Ge for rare event studies with a cryogenic bolometer has been initiated. For this purpose, semiconductor grade Ge wafers were irradiated with thermal neutrons at the Dhruva reactor at BARC, Mumbai. Irradiated Ge samples have been studied in the low background counting setup and all $\gamma$–rays were identified. Chemical etching of surface removes most of the long lived impurities, indicating that these impurities are probably diffused in Ge
samples during irradiation from the sample capsule environment. For the desired neutron fluence of $1 - 5 \times 10^{18}/\text{cm}^2$, removal of 50 $\mu$m surface layer is found to be adequate for this purpose. The samples loaded in the quartz tube are found to have lower radioactivity than those wrapped in Aluminium. The observed radioactive impurities $\sim 1 \text{ Bq/gm}$ in the bulk Ge, estimated after chemical etching of the samples, implies that a cool down period of $\sim 2$ years would be necessary before sensors made from these samples can be used in rare decay studies requiring ultra low background ($\leq 1 \text{ mBq/gm}$).

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