# Development of NTD Ge sensors for low temperature thermometry (for Neutrinoless Double Beta Decay)

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## **Plan of the talk**

- Cryogenic bolometer for Neutrino less Double Beta Decay (NDBD)
- NTD Ge sensor development for bolometer
- Hall Effect Measurement carrier concentration
- Defect studies in NTD Ge
  - Positron Annihilation Lifetime Spectroscopy (PALS)
  - Channeling
- Low temperature resistance measurement of NTD Ge sensor

### Summary

## Search for NDBD in 124Sn



#### Significance of 0vββ:

 Only experiment which can tell whether neutrino has its own antiparticle (Majorana particle) or not (Dirac particle)

#### **Experiments will look at:**

 Constancy of sum energy of two simultaneously emitted electrons, which requires high energy resolution and large scale detector

## **Schematic of bolometer**



- When DBD occur, energy of electrons heats the crystal
- \* Thermalization leads to rise in the crystal temperature T+δT
- The detector will be operated at an optimum temperature of 10mK, in order to get a reasonable  $\delta$ T.

## **NTD Ge sensor development**

- Low neutron capture cross-section homogenous doping. Melt-doped Ge with large concentration will be inhomogeneous and can have different characteristics
- Dopant concentration can be controlled by changing the neutron dose
- Low specific heat, fast rise time are essential to detect bolometer pulse.

Isotope	Products formed (half-	Decay into(decay	Туре
	life)	mode)	
<sup>70</sup> Ge	<sup>71</sup> Ge* (11.43 d)	<sup>71</sup> Ga(e⁻ capture)	р
<sup>72</sup> Ge	<sup>73</sup> Ge (stable)	<sup>73</sup> Ge	-
<sup>73</sup> Ge	<sup>74</sup> Ge (stable)	<sup>74</sup> Ge	-
<sup>74</sup> Ge	<sup>75</sup> Ge* (82.78min)	<sup>75</sup> As (β-decay)	n
<sup>76</sup> Ge	<sup>77</sup> Ge* (11.3,38.8hrs)	<sup>77</sup> As*, <sup>77</sup> Se	n
		(β <i>,</i> β-decay)	

### Dopant details of irradiated Ge

For a natural Ge, the neutron doping results in p-type due to its isotopic abundance and the thermal neutron capture cross section.

## **Irradiation details of NTD Ge**

- Ge sample irradiated with thermal neutron fluence at Dhruva reactor, BARC, Mumbai.
- Samples exposed to both thermal and fast neutrons.
- Fast neutron induces damage to the crystal lattice.
- The samples are etched with HF to remove oxide layers.
- Scanning Electron Microscope (SEM) and Energy Dispersive X-ray Analysis (EDAX) are done before and after etching.
- Etched samples are free of oxides.



(After H	F etching)
С	
0	
•	
□ 10 µm	σ

Sample	Values
Plane	<100>
Thickness (mm)	1.0
Resistivity (Ωcm)	>35
Thermal neutron fluence (<0.625eV) from the reactor data	7 x 10 <sup>18</sup> /cm <sup>2</sup>
Fast neutron fluence (.625eV - 0.821 MeV) from the reactor data	1.2 x 10 <sup>18</sup> /cm <sup>2</sup>
Calculated carrier concentration/cm <sup>3</sup> ( Acceptor – Donor )	1.7 x 10 <sup>17</sup> /cm <sup>3</sup>

#### SEM images of NTD Ge sample

## Hall Effect Measurement

- For Hall effect measurement six Au (88 %) Ge (12 %) were made using standard Vander Pauw method.
- The samples were rapid thermally annealed at 400°C for 2 min.
- Electrical connections were made by wedge bonding 25 μm Al wires to the contact pad.
- The Hall voltages were measured at both 77 K and at 300 K.
- The hall voltage is measured with 100µA current in the varying magnetic field of -1 to 1 tesla.
- Both Hall voltage and longitudinal voltage were measured simultaneously with two lockin amplifiers.
- The carrier concentration were estimated by assuming single carrier approximation using the following formula:



- $V_H$  = Hall voltage
- $B_z$  = Applied Mag field  $I_x$  = Supplied current
- *t* = thickness of sample
- n = carrier concentration
- e = charge





#### **Actual sample**

## Hall Effect Measurement cont..



- Carrier concentration was estimated using the Hall voltage value.
- The contribution of magneto-resistance was taken into account during estimation.
- The Hall factor (Hall mobility / Hall conductivity) is close to unity [1] below 100K.
- Hall effect measurement can be used as tool to estimate thermal neutron dose by estimating the carrier concentration at 77K.
- Estimated thermal neutron fluence is about
  4.6 x 10<sup>18</sup>/cm<sup>2</sup>.



Sample	<b>Carrier concentration (x 10<sup>17</sup>/cm<sup>3</sup>)</b>				
	77K	300K	Calculated using neutron dose		
NTD Ge	1.11	2.13	1.70		

At 300K the intrinsic carriers due to thermal agitation can participate in conductivity, hence the values are higher than the actual dopant concentration.

[1] F.J.Morin, Physical Review, 93 (1954) 62.

## Defect studies – Positron Annihilation Lifetime Spectroscopy

- PALS will quantify the defect size through the positron lifetime measurement
- A positron is allowed to interact with the sample, where it experiences both attractive (due to e<sup>-</sup>s) as well as the repulsive force (due to core ions)
- In a defect free crystal, the positron interacts with the valance electron and two γ rays of 511 keV are generated as a result of annihilation. This positron lifetime gives the bulk lifetime
- When a positron interacts with a crystal having defects, it is more likely to be trapped in the defect due to repulsive force of ions. This will lead to increase in lifetime of positron. Thus larger the defect, longer will be the lifetime of the positron



## **PALS Results**

- Irradiated sample, irradiated-annealed at 600°C for 2hrs (in vacuum sealed quartz tube) and virgin sample are studied using PALS at RCD, BARC, Mumbai.
- PATfit software is used for acquiring and analysing data
- The irradiated sample shows monovacant defects and vacancy clusters.
- The annealed samples had the positron lifetime close to that of the un-irradiated (bulk) lifetime.

Samples	φ <sub>th</sub> (n/cm²)	Τ <sub>β+</sub> (ps)	Ref (ps) [2]	
Virgin Ge	-	232 ± 0.3	228 (Bulk)	
Irradiated Ge	4.57 x 10 <sup>18</sup>	294 ± 0.3	293 (Monovacant)	
		401 ± 30	401 (Vacancy Clusters)	
Annealed NTD Ge	4.57 x 10 <sup>18</sup>	233 ± 0.4	228 (Bulk)	

[2] Reinhard Krause-Rehberg, Hartmut S. Leipner, *Positron Annihilation in Semiconductors: Defect Studies*, Springer, 1998.

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### **Channeling studies**

- NTD Ge crystal probed with an energetic alpha beam (~2MeV) along the axial direction at PARAS facility, IUAC, New Delhi.
- Defects lead to dechanneling of the beam, results in higher Rutherford Back Scattering (RBS) yield
- Minimum current avoids damage produced during measurement.



Artist view of ion channeling along the crystal axis



2D Image of Ge crystal <100> cleavage plane

### **Channeling results**

- The irradiated sample higher RBS yield compared to virgin sample
- Annealed sample close to virgin sample.
- Indicates recovery of the defects in the crystal after annealing.
- Defect density calculated to a depth of 316nm just below the surface



✤ The defect density is calculated by,  $N_D = N\left(\frac{\chi_{min}^{irr} - \chi_{min}^0}{1 - \chi_{min}^0}\right)$ Where  $\chi_{min}$  = channelled RBS/random RBS

Samples	316 nm
Virgin	$0.042 \pm 0.001$
Irradiated	$0.058 \pm 0.001$
Annealed	$0.040 \pm 0.001$
N <sub>D</sub> (/cm³)	(7.3 ± 0.4) x 10 <sup>20</sup>

#### Channeled spectra and random spectrum of NTD Ge sample

### Low temperature resistance measurement in dilution refrigerator



#### Gas Handling system along with the dilution unit

dilution unit



V. Singh, S. Mathimalar, N. Dokania, V. Nanal, R. G. Pillay, S. Ramakrishnan, Pramana 81 (2013) 719.

### Low temperature resistance measurement cont..

- The samples used in Hall probe measurement were tested for low temperature resistance measurement.
- The resistance values are compared with that of commercial  $RuO_2$  sensors.
- Clearly the NTD Ge sensor showed higher dR/dT compared to RuO<sub>2</sub>.
- The dR/dT is about ~2.3k Ω/mK at 100mK.
- Below 75mK the NTD Ge shows saturation, which might be due to thermal link or due the noise.



• Logarithmic sensitivity $\alpha = \left  \frac{d(\log R)}{d(\log T)} \right $ • For a semiconductor thermistor will			
range between 1 to 10.			
Samples	Logarithmic		
	consitivity at 100mK		
	Sensitivity at LOONIN		

 Clearly the figure of merit is higher for NTD Ge as compared to that of RuO<sub>2</sub>.

0.7

RuO<sub>2</sub>

## **Summary**

- Ge wafers are doped by neutron transmutation doping technique.
- Hall probe measurement was done in order to verify the carrier concentration formed due to doping.
- From the estimated carrier concentration at 77K, the neutron dose has been recalculated.
- Samples were studied for the defects due to fast neutron during irradiation.
- Both PALS and Channeling confirmed that annealing the irradiated samples at 600°C for 2hrs in a sealed quartz tube can reduce the defects.
- Low temperature resistance measurement showed better dR/dT compared to that of the commercial RuO<sub>2</sub>.

## **Collaborators**

Mr. V Singh Ms. Neha Dokania Prof. V. Nanal Prof. R.G. Pillay Dr. Sanjoy Pal Prof. S. Ramakrishnan Dr. A. Shrivastava Dr. Priya Maheswari Dr. P.K. Pujari Mr. Sunil Ojha Dr. D. Kanjilal Mr. K.C. Jagadeesan Dr. S.V. Thakare

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# Back ups

10 <sup>10</sup>	1K 500mK 200mK 100mK 62.5mK	Sample	Hole Conc. (x10 <sup>16</sup> cm <sup>-3</sup> )	K	ρ <sub>0</sub> (Ω cm)	Т <sub>о</sub> (К)
- 10 °	NTD 14	NTD 2	0.03	0.32	200	82.9
-E	НТО 16-	NTD 5	1.5	0.32	0.47	77.6
9 10 6		NTD 14	2.7	0.32	0.11	49.0
g "	Strate Strate	NTD16	4.2	0.32	0.1	26.5
2 10 4		NTD 18	5.3	0.32	0.15	15.9
<u>5</u> "	NIO23	NTD 28	6.3	0.32	0.09	7.84
¥	NTD 12	NTD 12	6.8	0.32	0.02	7.84
		NTD 25	8.6	0.32	0.008	2.74
۲ ۳		70 <sub>Ge-3.30</sub>	3.02	<0.001	0.34	364.8
יי	NID 2	<sup>70</sup> Ge-2.98	8.00	<0.001	0.0074	247.6
0.01		70 <sub>Ge-1.90</sub>	9.36	< 0.001	0.0019	201.4
0	05 1 15 2 25 3 35 4	70 <sub>Ge-1.65</sub>	14.5	<0.001	0.0006	100.3
	T <sup>-1/2</sup> (K <sup>-1/2</sup> )	70 <sub>Ge-2.15</sub>	17.7	< 0.001	0.0215	20.7