

Study of cryogenic properties of tin alloys for the development of a superconducting bolometer

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Neutrinoless double beta decay ($0\nu\beta\beta$ or NDBD) is the only known experimental probe to study the fundamental nature of neutrinos (Majorana or Dirac). Given its importance, several international experimental collaborations are searching for $0\nu\beta\beta$ using complementary experimental strategies. The India-based tin detector (*TIN.TIN*) [1] proposes to search for $0\nu\beta\beta$ in the isotope ^{124}Sn using an array of cryogenic Sn-based bolometers operated at $\sim\text{mK}$ temperatures. The choice of the absorber crystal is crucial for the performance and stability of the bolometer. In the search of $0\nu\beta\beta$, the absorber is usually made out of the $0\nu\beta\beta$ isotope or its compound. A central challenge in the fabrication of Sn-based cryogenic bolometers is the phenomenon of tin pest [2], which is a metal (β) to semiconductor (α) phase transition in Sn occurring near ambient conditions. Due to the sudden lattice expansion associated with the phase transition, the Sn sample cracks and deforms. This poses a significant risk to the longevity and performance of the Sn-based bolometers. Therefore, it is crucial that the risk of tin pest formation is suitably mitigated.

It is known that alloying Sn with an appropriate element can pin the dislocations in the crystal, thereby suppressing the lattice expansion associated with tin pest formation. The $\beta \rightarrow \alpha$ transition becomes kinematically unfavourable when the lattice expansion associated with the change of crystal structure is suppressed. This motivates the present work to qualify a Sn-rich alloy as a candidate for the fabrication of superconducting bolometers for *TIN.TIN*, on the basis of resistance to tin

pest, superconductivity and radiopurity of the alloys.

In order to find a suitable alloy candidate, several tin alloys (Sn-Bi, Sn-Cd, Sn-Cu, Sn-In, Sn-Pb and Sn-Sb) were fabricated using high purity Sn (99.99999% pure) and alloying elements ($\geq 99.999\%$ pure). These samples were then tested for resistance against tin pest. The Sn-Cu and Sn-Cd samples were found to be susceptible to tin pest. The Sn-Pb alloys performed better in comparison, but developed tin pest over long durations of time. The Sn-In, Sn-Bi and Sn-Sb samples showed promising results with respect to the inhibition of tin pest. However, the Sn-Sb and Sn-In samples were regarded as unsuitable due to the anticipated background from neutron activation channels in the region of interest (ROI). The Sn-Bi alloy was found to be the most suitable candidate.

Magnetization measurements were performed to check the superconducting critical temperature of the Sn-Bi alloys (0.08% - 1.69% Bi by weight). The alloys were found to be superconducting at a critical temperature within 2% of that of pure Sn. The robustness of the observed superconducting transition implies that Sn-Bi alloys are suitable for use in superconducting bolometers.

Although the phenomenon of tin pest has been studied for at least a century, there were several inconsistencies in the literature regarding the $\alpha \rightleftharpoons \beta$ transition temperature. Improved measurements of the $\alpha \rightarrow \beta$ phase transition temperature in Sn and Sn-Cu were performed in the present thesis work using various modern techniques, namely, differential scanning calorimetry, synchrotron x-ray diffraction studies and temperature resolved scanning electron microscopy. The phase transition was consistently found to occur between

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30°C and 34°C, instead of the often quoted value of 13°C. No dependence of alloying Cu with Sn was observed on the transition temperature. Since α -Sn is a material with novel topological properties and has potential applications for developing qubits, it may be interesting that the material is more stable at room temperature than previously believed. Based on these measurements [3, 4], it is suggested that the *TIN.TIN* bolometer array should be baked at 50°C for a few min (20 - 30 min) between thermal cycling to further reduce the risk of tin pest. It should be mentioned that this protocol would also be useful for critical electronic systems using lead-free solders and operating at low temperatures.

It is well known that the sensitivity of NDBD experiments depends on the background in the region of interest. Internal radiation background studies were performed for Sn-Bi bolometers in the region of interest around $Q_{\beta\beta}$ (2291 ± 25 keV). The Sn-Bi crystals were assessed for radiopurity in the TIFR low background experimental setup (TiLES) [5]. No additional γ lines or enhancements compared to the ambient background were found at the measured sensitivity level.

Neutron activation of Sn-Bi was performed at the Pelletron Linac Facility, TIFR Mumbai to investigate the short and long lived neutron-induced activity in Sn-Bi alloys. Impurities in the alloys can also be sensitively probed using this technique. The samples were irradiated with fast neutrons at the 6m target setup, generated through the ${}^9\text{Be}(p, n){}^9\text{B}$ reaction ($Q = -1.850$ MeV) by using proton beams with energy $E_p = 15 - 21$ MeV on a ${}^9\text{Be}$ target, upto a maximum flux of $\phi_n \sim 10^6$ n cm $^{-2}$ s $^{-1}$. The activated samples were counted offline in close geometry using HPGe detectors (TiLES and Bruker Baltic detectors D1 and D2). No activation channels from Bi or any possible impurities in the crystal were measured. Instead, all the activity could be attributed to reaction products arising from the neutron activation channels of Sn.

${}^{209}\text{Bi}$ undergoes a very rare α decay with a half-life of 2.0×10^{19} y which is comparable to the half-lives of some $2\nu\beta\beta$ emitters. The

surface events can become a source of internal background for Sn-Bi bolometers. GEANT4 simulations were developed to estimate the background from the decay of ${}^{209}\text{Bi}$ for various bolometer volumes (27, 64 and 125 cc) and Bi alloying concentrations (0.25%, 0.50%, 0.75% and 1.00%). The anticipated internal background from U/Th impurities was also simulated and compared to the background from ${}^{209}\text{Bi}$ α decay. The β decay from ${}^{214}\text{Bi}$ (product of the ${}^{238}\text{U}$ chain) was found to be the limiting background, while the radioactivity of ${}^{209}\text{Bi}$ had negligible effect on the background ($\sim 10^{-5}$ cts/(keV.kg.y)). Nevertheless, it was observed that the total background from these sources was within the background limit of 10^{-2} cts/(keV.kg.y), which is typical for the first generation bolometric experiment without particle discrimination.

The efficiency of Sn-Bi bolometers of different sizes for the $0\nu\beta\beta$ signal was also estimated to be 86.6% (27 cc), 89.0% (64 cc) and 90.7% (125 cc). The sensitivity of *TIN.TIN* for $0\nu\beta\beta$ was calculated for a range of enrichment percentages (natural - 99%) and detector sizes using these simulated efficiencies.

The present work provides important inputs towards the consideration of a suitable alloy for the bolometer absorber crystal. Based on this work, a dilute Sn-Bi alloy (0.22% Bi by weight) is suggested as a suitable candidate for the fabrication of superconducting bolometers for *TIN.TIN*. The future outlook includes detailed radiation background studies on the thermal neutron and cosmogenic backgrounds arising in Sn-Bi.

References

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