I. INTRODUCTION

Recently, the rare-earth monopnictides (R-Sb/Bi) group have fascinated the researchers due to their novel electronic band structures. The novel electronic band structure in R-Sb/Bi leads to the observation of variety of exotic phenomena like non-saturating extremely large magneto-resistance (XMR), field-induced resistivity upturn with plateau, high carrier mobility, topological surface states and quantum oscillations\cite{1,2,3}. These salient features have raised immense interest in both fundamental understanding as well as in applied physics. XMR and field induced resistivity upturn with plateau are the hallmark phenomena of R-Sb/Bi which is quite similar to other trivial and non-trivial semimetals\cite{4–9}. The observation of ultrahigh mobility and XMR of $\sim 10^6\%$ in LaSb at $T=2\text{K}$ and $H=9\text{T}$, have triggered immense interest in these systems\cite{1}. Subsequently, similar results have been reported in other R-Sb/Bi and shown the non-trivial nature of band structure\cite{3,10}. DySb is a unique member among the R-Sb/Bi family due to its peculiar structural, magnetic and magnetotransport properties particularly at low temperatures. DySb hosts rock-salt type crystal structure having space group Fm$\overline{3}$m (225) of rock-salt type crystal structure. HRXRD on cleaved crystal confirms the single crystalline nature while rocking curve analysis reveals the high quality of the grown crystal. Temperature dependent resistivity and magnetisation measurements show a transition at 9.7 K from paramagnetic (PM) to antiferromagnetic (AFM) state.

II. EXPERIMENTAL

Single crystal of DySb has been prepared by self-flux method, which is quite different from earlier reported methods for the growth of DySb single crystal\cite{11,12,13}. Here, Dy (99.9\%) and Sb (99.999\%) granules are taken in 1:19 molar ratio, where the excess Sb acts as flux. As the constituent material (Sb) itself acts as a flux, it is termed as self-flux. The starting materials are homogeneously mixed for several hours using pestle and mortar. Thereafter, homogenously mixed powder of con-
stinent elements are vacuum sealed (~10^{-6} torr) in a quartz tube. The sealed quartz tube is placed in a programmable furnace, where it is heated up to 950°C and dwelled for 10 hours. The furnace is then cooled down to 630°C at the rate of 2°C/hr., where it is dwelled for 90 hrs. followed by natural cooling. The sealed quartz tube is carefully removed from the furnace and then broken in order to remove the grown crystal. Figure 1 illustrates the schematic diagram of heat treatment protocol for DySb single crystal growth along with the photograph of grown crystals. After mechanically removing the Sb flux and cleaving, tiny shiny crystals of DySb ranging from 0.5 mm-2 mm are obtained. Some of the cleaved crystals are crushed and homogeneously powdered in the mortar with the help of pestle for powder X-Ray diffraction (XRD) studies. The powder XRD spectra were recorded at room temperature by Bruker D8 Advance diffractometer using Cu-Kα source. The XRD data is recorded in the range of 10°-100° with step size of 0.02°. The obtained powder XRD data is fitted with FULLPROF software and then unit cell structure is generated using VESTA software. For determining the crystalline quality High Resolution X-Ray Diffraction (HRXRD) measurements (φ-scan and ω-scan) are performed in Bruker D8 Discover Diffractometer. Electrical transport and magnetisation measurements are performed in Physical Property Measurement System in the temperature range of 300 K to 2 K.

III. RESULTS AND DISCUSSIONS

A. Structural Characterization

Figure 2(a) displays the FESEM image of cleaved crystal. Figure 2(b) shows the out-of-plane XRD pattern of cleaved DySb crystal along (002) plane direction. The appearance of very sharp {002} set of planes suggest that the grown crystal is oriented along [002] plane, which is further characterised by HRXRD measurements. Inset shows the schematic of cubic crystal with [002] oriented. Powder XRD pattern of DySb crystal with Rietveld refinement. Red circles represent the experimental data, black solid line is calculated intensity, blue bar shows Bragg peak position and solid pink line is the difference between experimental and calculated data. (d) Unit cell structure of DySb, generated from VESTA software.

set represents a schematic of cubic crystal showing [002] is the out-of-plane direction as observed in XRD. DySb crystallises in NaCl-type crystal structure in cubic space group Fm\bar{3}m (225) [5, 11, 12]. The lattice parameter “a” calculated from Bragg’s law (2dsinθ=nλ) for the observed out-of-plane XRD pattern is a=6.15(4)Å. The calculated lattice parameter matches well with the JCPDS (15084) value of a=6.153Å for DySb crystal as well as with the reported values of a=6.143Å [11] and a=6.161Å [14]. Figure 2(c) displays the Rietveld refinement fit to the powder XRD pattern of crushed DySb crystal. All the peaks are well indexed and fitted on the basis of NaCl-type crystal structure with Fm\bar{3}m (225) space group. The absence of extra peaks in powder XRD pattern confirms the single-phase nature of grown crystal. The refined lattice parameters are a=b=c=6.155(1)Å, which resembles to the calculated lattice parameters for the out-of-plane XRD pattern as well as with the earlier reports [11, 14]. The occupancies obtained from refinement for Dy and Sb are 1.0:0.9, which confirms the proper stoichiometry of grown crystal. All the refined parameters are tabulated in Table 1. Figure 2(d) represents the rock salt type crystal structure of DySb crystal generated from VESTA software, where Dy and Sb atoms are represented by Blue and brown spheres respectively. The Wychoff position for Dy and Sb are 4a(0,0,0) and 4b(1/2,1/2,1/2) respectively.

The out-of-plane XRD shows only {002} set of planes i.e. <002> oriented. To see the in-plane orientation of crystallographic planes, in-plane θ-2θ XRD is performed.
for \{022\} planes. Figure 3(a) shows the XRD pattern of DySb crystal along \{022\} plane. The XRD data exhibits only two peaks corresponding to \{022\} and \{044\} parallel planes. Hence, the grown crystal has a periodic arrangement of planes for out-of-plane \{022\} direction as well as in-plane \{022\} direction, confirming single crystalline nature of crystal. DySb crystallizes in NaCl-type structure therefore it has four-fold crystal symmetry, which means in azimuthal \(\phi\)-scan, one can observe a peak separation of \(\sim 90^\circ\) for four-fold crystal symmetry. Figure 3(b) shows the azimuthal \(\phi\)-scan for \{022\} crystal plane. The \(\phi\)-scan exhibits two peaks which are separated by \(\sim 90^\circ\), suggesting the 4-fold \(360^\circ/90^\circ = 4\) symmetry of the grown crystal. The observation of only sharp peaks in \(\phi\)-scan indicates that the grown crystal has perfect orientation of azimuthal domains.

Figure 4(a) shows the rocking curve (RC) for \{002\} crystal plane. RC analysis is an important diagnostic tool to determine the crystalline perfection and disorder present in the crystal\cite{16, 17}. The full width at half-maxima (FWHM), asymmetry and kink in RC play a crucial role for determination of crystalline disorders. The RC of \{002\} plane has a small kink at lower \(\omega\) side of the main peak. To confirm this, RC (fig. 4(b)) is repeated for \{004\} plane where a similar type of kink is observed at lower \(\omega\) side. This confirms that kink is inherently from the crystal and is well characterized. The deconvoluted RC of \{002\} peak is shown in fig. 4(c), which gives two peaks. The appearance of an additional peak at a separation of \(0.03^\circ\) from main crystal domain indicates a very low angle grain boundary in the internal structure. The high intense deconvoluted peak with FWHM\(\sim 0.04^\circ\) represents the main crystal domain while the low intense deconvoluted peak with FWHM\(\sim 0.06^\circ\) is appearing from very low angle grain boundaries having misorientation of \(0.03^\circ\) with the main crystal domain.

The observed FWHM of main crystal domain in present work is smaller than that observed by Liu et al. in their DySb single crystal\cite{18}. Hence it is inferred that although the crystal has little defects, its small FWHM\(\sim 0.04^\circ\) of main crystal domain suggests a good crystalline nature of grown DySb single crystal\cite{16, 18, 20}.

### TABLE I. Rietveld refined parameters

| \(\alpha=\beta=\gamma\) | \(a=b=c\) | Volume (\(\text{Å}^3\)) | Wyckoff Position | Occupancy | \(\chi^2\) |
|-------------------------|-------------|----------------------------|-----------------|-----------|-----------|
| 90°                     | 6.155(1)    | 233.27(4)                  | 4a(0,0,0)       | Dy-1.0    | 1.35      |
|                         |             |                            | 4b(\(\frac{1}{4}\),\(\frac{1}{4}\),\(\frac{1}{4}\)) | Sb-0.9    |

B. Characterization of Phase transition by transport and magnetic measurements

Figure 5(a) shows the temperature dependence of electrical resistance (R-T) of DySb single crystal in the absence of magnetic field. The zero-field resistance decreases gradually with decrease in temperature, demonstrating the metallic nature of DySb. A small kink is seen near 10 K, whose enlarged view is shown in the upper inset of fig. 5(a). The enlarged view of R-T data shows a clear drop in resistance at \(\sim 9.7\) K showing PM to AFM transition. To find the appropriate transition point, the observed resistance data is differentiated with respect to temperature \(\frac{dT}{dR}\) which is shown in lower inset of fig. 5(a). The \(\frac{dT}{dR}\) data shows a sharp peak at \(\sim 9.7\) K that is well matched with the transition temperature reported in literature on DySb single crystals\cite{3, 11, 13, 15}. We have further characterised our grown crystal by magnetisation measurements. Figure 5(b) illustrates the temperature dependence of magnetisation (M-T) for DySb crystal in an applied magnetic field of 0.05 T. The M-T curve shows a sharp peak at \(\sim 9.7\) K corresponding to the PM to AFM transition. This confirms that the synthesized single crystal grown by self-flux method have has a good crystalline quality.
IV. CONCLUSION

The shiny single crystal of DySb has been successfully grown by self-flux method. The Rietveld refinement of powder XRD pattern at room-temperature reveals the single-phase nature while HRXRD confirms its single crystalline nature. The crystalline system was identified to be cubic Fm$\overline{3}$m space group. Rocking curve analysis confirms the high quality of grown crystal. Phase transition temperature is confirmed by PM to AFM transition as well as by magnetisation measurements, which further ascertains the good quality of crystal.

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