In-situ observation of InSb and InGaSb crystal-melt interface and analysis of dendrite formations

Abstract:-- In this study, the effect of gradient freezing and grain boundary distribution on the formation of dendritic structures of InSb and InGaSb were analysed. The growth process of pure InSb and $In_{1-x}Ga_xSb$ alloy semiconductors were in-situ observed. The materials were crystallized under pre-determined cooling rates. The growth process was monitored and recorded using a microscope with video recorder. The growth rates of crystals were analysed from the t-x plot. The grain structures of the grown crystals were analysed after the in-situ experiments.

In-situ observation of crystallization process gives us deep insights about determination of microstructures, such as grain size, crystal orientation and some defect formations like twins and dendrites. This experimental investigation helps to understand how the polycrystalline semiconductor crystals are formed during melt growth.

The high purity source materials of In & Sb (99.99%) were cleaned by ultrasonication in acetone, etched in dil.HNO_3(HNO_3:H_2O=1:1) for 5 minutes and rinsed in distilled water followed by drying using N_2 gun. InSb ingots were prepared by melting In and Sb granules in a vacuum sealed quartz ampoule at 700˚C and cooling to RT slowly for 24h (28˚C/h). The prepared ingots were cleaned by etching the surface using HF : HNO_3 : CH_3COOH in 2:5:4 ratio. The crystallization of InSb from melt was in-situ observed at different cooling rates of 5, 10, 15, 30 K/min using specially designed furnace in which two independently controlled heaters were engaged to establish a temperature gradient of 6K/mm. The crystallization process was monitored and recorded using Keyence VHX-600 microscope. The grain structures of InSb ingots solidified at different cooling rates were analysed by Electron Backscatter Diffraction pattern (EBSP) using JEOL JSM-6610A. Similar procedure was followed for in-situ observing the crystallization process of $In_{1-x}Ga_xSb$.

During the in-situ observation of InSb, a thin layer of oxide layer was formed on the melt surface which makes it difficult to observe the growth process. Hence a thin quartz plate was placed on top of the crucible to avoid the oxide layer formation and the growth of InSb was in-situ observed [1]. At low cooling rates (< 15 K/min) no dendrites were formed on the InSb melt surface. At the higher cooling rate of 30 K/min, dendrites were formed with clear facets (Fig.1) and the dendrites were emerged from the facets of the grooves which were initially formed by the twin boundaries and led to faster growth as a result of twin plane re-entrant edge mechanism. The re-entrant corner created by the twins acted as a step source due to which continuous and faster growth was observed from its core. The growth behaviour of the crystal-melt interface with grooves and dendrites were analysed by t-x plot and in relation with change in growth rate as a function of time with isochrones. The grain structures and orientations of the grains around the dendritic structures were examined using EBSP analysis. During the experiments with the ternary $In_{1-x}Ga_xSb$ system, the vigorous oxidation and expansion on the surface of the ingot made it difficult to be observed through the microscope.

Furthermore, the results of the growth behaviour analysis will be compared with theoretical models and will be validated. The outcomes of the experiments paves way for designing an optimized growth conditions that leads to the growth of defect controlled InSb crystals. Such ingots can be used in various applications like IR sensing, photovoltaics, thermoelectrics and so on.

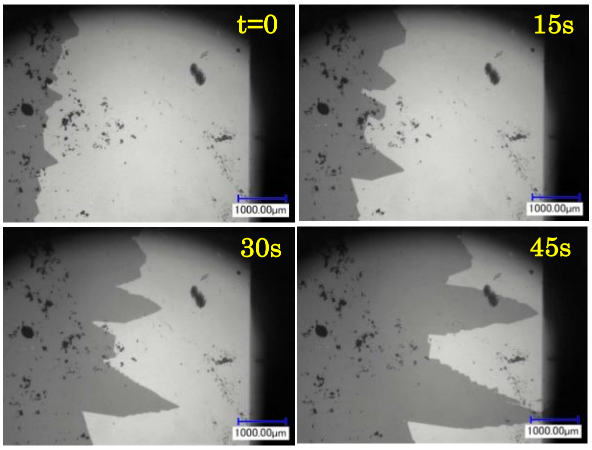

Fig.1 Faceted dendrites grown at crystal-melt interface.

References

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