Vickers Micro Hardness of In: Bi$_2$Se$_3$ SINGAL CRYSTAL


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Abstract - In: Bi$_2$Se$_3$ single crystals have been grown by Bridgeman technique. The growth velocity was 0.5 cm / hour and freezing interface temperature was 65 °C/cm. The Vickers projection microscope was used for the study of micro hardness of the crystals. Vickers micro hardness tests were carried out on as cleaved, cold work annealed as well as quenched surfaces of the crystals, the results are discussed and reported in detail.

Keywords- Bridgeman technique, Vickers projection microscope, micro hardness

I. INTRODUCTION

In: Bi$_2$Se$_3$ is a narrow band gap material which has hexagonal structure and it is useful in the field of thermoelectric devices as solid state coolers or generators [1–3]. Both n- and p-type Bi$_2$Se$_3$ single crystals grown by the Bridgman technique is used to make excellent Hall effect magnetometers. It can be work in magnetic fields to at least 10 Tesla and over a wide range of temperature [4] The thermoelectric figure of merit is high [5] for Bi$_2$Se$_3$ and Bi$_2$Te$_3$ material. So these materials are useful for thermoelectric energy conversion. The band gap is very narrow, so it can be used for infrared radiation detectors [6]. The low temperature peaks found in the seebeck coefficients in Bi$_2$Se$_3$ show that materials that have strong spin orbit coupling and nearby dirac points [7,8], suggest that these characteristics may be related. In addition, processing of the microstructure of n- and p-type Bi$_2$Se$_3$ reduce the thermal conductivity to < 1 W/K-m. Bi$_2$Se$_3$, Bi$_2$Te$_3$ and Sb$_2$Te$_3$ are used as 3D topological insulators [9,10]. Many physical phenomena are predicted to emerge in low dimensional nanostructures of Bi$_2$Se$_3$ [11], ultra-thin Bi$_2$Se$_3$ down to a few (≤5) nano meters and is expected to exhibit topologically non-trivial edge. Recently, preparation of Bi$_2$Se$_3$ nano structured materials via wet chemical methods has attracted increasing attention due to low cost and convenience. Qiu et al.[12]. Therefore studies on various properties of Bi$_2$Se$_3$ are interesting for both basic and applied research. If we crystallize under proper controlled conditions Bi$_2$Se$_3$ is also easily cleavable.

II. SAMPLE PREPARATION

The stoichiometric amount of In, Bi and Se each of 99.999% purity were weighed accurately up to 10 μ gms using a semi micro balance. They were sealed in quartz tube under a pressure of 10–4 Pa. The tube was kept vertical in an alloy mixing furnace at a temperature of about 775 °C for 48 hr. Then .The ingot was then cooled to room temperature over a period of 24 hr at a rate of 200°C /hr. Single crystal In: Bi$_2$Se$_3$ grown by Bridgman method with a temperature gradient of 650°C /cm and a growth rate of 0.5 cm/hr.

III. TESTING SAMPLES

To measure the Vickers hardness of the material 1 to 2 mm thick cleaved flat surface cleavage is taken. The experiments were conducted at room temperature. The Vickers indenters were carried out at different loads for 35 second. The same process was done for similar samples. To study the effect of cold working on hardness of the material 4 to 5 mm thick cleaved flat surface plats of crystals were placed between two flat surface glass slabs and about 1.5 kg weight was placed over the top of slab for 24 hours. The experiments were conducted at room temperature and then the crystal was cleaved for 1 to 2 mm thickness. For annealing study the sample was sealed in a glass tube at 10$^{-4}$ pa. The glass tube is kept at 200 °C temperature in the furnace for 24 hours and gradually cooled down to room temperature. For quenching study the sample was sealed in a glass tube at 10$^{-4}$pa. The glass tube is kept at 200 °C temperature in the furnace for 24 hours and suddenly cooled down to room temperature. Then hardness tests were done on the cleavage surface of crystals with...
different loads for 35 second constant for as-cleaved cold-worked, annealed, and quenched samples. All necessary precautions were taken to see that the experimental error in the measurements were as small as possible.

IV. RESULT AND DISCUSSION

Figure 1 shows the plot of Vickers hardness versus applied load for In: Bi$_2$Se$_3$. The Vickers hardness number for normal sample HV is 150.38 MPa. The Vickers hardness number after cold work HV is 169.42 MPa, which shows that after coldwork process the value of hardness increases by 12.63 % w.r.t ascleaved sample. The Vickers hardness number after annealing HV is 117.49 MPa, which shows that after annealing process the value of hardness decreases by 21.88 % with respect to ascleaved sample. The Vickers hardness number after quenching is HV 191.57 MPa. This shows that after quenching process the value of hardness increases by 27.39 % w.r.t ascleaved sample.

V. CONCLUSION

Micro hardness is a load dependent quantity and prominent variations are observed in low load regions and is seems virtually independent of load in high applied load region. The HV value of ascleaved sample is 150.4 MPa, in the case of annealing the HV value is 117.5 which is less by 22 %. In the case of cold work the HV value is 169.4 MPa which is 12.6 % more as compared to ascleaved sample and after quenching the HV value is 197.6 MPa which is 27.4 % more with respect to ascleaved sample.

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