



IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 6 Issue: VII Month of publication: July 2018

DOI: http://doi.org/10.22214/ijraset.2018.7102

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Growth of Bismuth Trisulfide (Bi₂S₃) Single Crystals using Horizontal Zone Leveling and Gel Methods

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Abstract: Ferroelectric semiconductors of the A_2B_3 having the Para electric phase are currently being studied intensively. Bi_2S_3 of one of such semiconducting crystals exhibiting interesting photo conducting and being isomorphic with orthorhombic lattices. There crystals are grown from the melt using modified Bridgeman [1] and chemical vapor Transport method [2]. Skeleton, layered structured crystals are grown by hydrothermal method [3]. Flower like multibranched Nano belts, straw-tied nanowires are synthesized by facile decomposition and precursor solution method [4] but no attempt have been made to grow single crystals of Bi_2S_3 using simple and inexpensive methods like zone refining method and Gel technique. Present paper is an attempt in this direction. These sulfides have unique electrical or optical properties as a major point of concern.

Crystals of Bi_2S_3 were grown by a simple Gel technique using single diffusion method. The optimum growth conditions were established by verifying various parameters such as PH of the gel solution, gel concentration, gel setting time, concentration of reactance etc. Crystals having different morphologies and habits. Bi_2S_3 single crystals are also grown by Horizontal Zone Leveling method. The container was a Pyrex tube, 10 cm in length and 1 cm in diameter. A trolley furnace having a temperature gradient traveled with speed of 1 cm/Hr. and after several passes good quality single crystals were obtained. Key Word: Bi_2S_3 single crystals, zone refining method, Gel method, Growth of Bi_2S_3 , Quality of Crystal.

I. INTRODUCTION

Bismuth is a semimetal with very small band overlap. Bismuth is very useful in thermal fuses, catalyst for making acrylic fibers, fusible boiler plugs and low melting solder. Bismuth expands 3.32% on solidification, this property makes bismuth alloys suited to making of sharp carting and it's also form low melting alloys by mixing Sulphur in it. Bismuth sulfide is a chemical compound of Bismuth and Sulphur. Semiconducting chalcogenide compounds (A₂ B₃) have been receiving much attention because of their wide range of application such chalcogenide compounds are narrow band gap semiconductor with homogenous layered crystal structure and are useful for solar cells, photo detectors, optoelectronic, light amplifiers.

II. EXPERIMENTAL

A. Zone Leveling Method

5N pure Bismuth material was obtained from Nuclear Fuel complex, Hyderabad. High purity (99.999%) bismuth was square rod type. Small slices of this material are uniformly mixed with Sulphur powder. This mixed powder was weighted proportional ratio and leaded into thoroughly cleaned thick walled Pyrex tube. 10cm length and 1 cm in diameter. The ampule was then evacuated to 10^{-5} Torr and sealed and then placed in horizontal quarts tube 1 m long and 1-inch diameter. The boat (Pyrex tube) is set in quartz container tube. A movable trolley furnace having a temperature gradient of 52°C/cm traveled with speed of 1cm/6Hrs, 1.5 cm/6Hs and 0.5 cm/6Hrs. unidirectional passes are given. (Fig. 1) The charge is prepared at 630°C. After several passes good quality single crystals were obtained. The crystals were cleaved at liquid nitrogen temperatures in conventional manner. After 10 passes with different speed of motors, the Furness was cooled at room temperature and then boat was taken out from the container and broken at liquid nitrogen temperature which reacted in the growth of single crystals of Bi₂S₃ in shape of needles. The shape and length of these crystals are shows in Fig. 2.

B. Gel Method

Crystals of Bismuth Trisulfide (Bi_2S_3) were grown by Gel method by using single diffusion technique. The gel medium prevents turbulence and it provides a three-dimensional crucible which permits the reagents to diffuse at a desirable controlled rate. Test tube of diameter 2.5cm and 25cm length were used as crystallizing vessels. The required silica gel medium was prepared by adding the



International Journal for Research in Applied Science & Engineering Technology (IJRASET) ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 6.887 Volume 6 Issue VII, July 2018- Available at www.ijraset.com

sodium-meta silicate (NO₂SiO₃.5H₂O) of specific gravity 1.04 gm/cc drop by drop with constant stirring by using stirrer into the 5ml (2N) glacial Acetic Acid the pH value 4.4 was set for the mixture to the above solution meta silicate solution of pH 4.4.15 ml aqueous solution of 0.1M Bismuth chloride (BiCl₃) was added as inner reagent with constant stirring. This mixture was then transferred to the test tube. The Gel was usually set within 30 days. All chemicals used for growth are of AR grade. The crystals were grown using following chemical reaction.

 $2XCl_3+3Y_2S \rightarrow X_2S_3+6Y(NO_3)$

Where X=Bi and Y=K or Na. The grown crystals are shown in Fig.3.

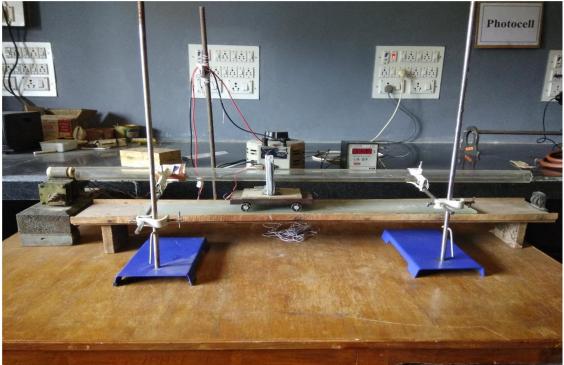


Fig.1 Zone Leveling Method

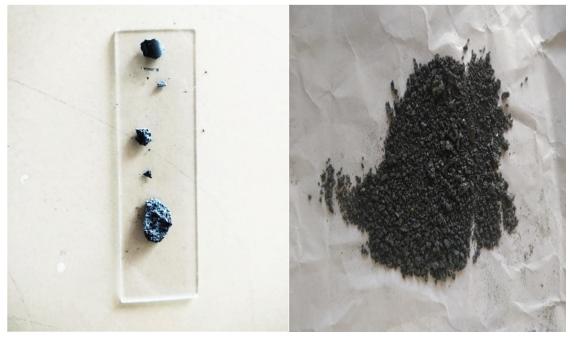


Fig. 2 Grown Crystals using Zone Leveling Method





Fig.3 Gel Method and Grown Crystals

II. RESULTS AND DISCUSSION

The various optimum conditions for growing crystals were established and are given in Table-1 for Zone Leveling Method and Table-2 for Gel Method.

Sr.No.	Conditions
1	Boat: Pyrex Tube 1- Cm length and 1 Cm diameter
2	Motor speed :1.5 cm/6Hrs,1.0 cm/6 Hrs. and 0.5 cm/6Hrs.
3	Evacuation Pressure: 10 ⁻⁵ Torr
4	Cleaved after: Unidirectional 10 passes.
5	Approx. Time: 60 Days.

Table-I Optimum Conditions for Zone Leveling Method.

Table-II Optimum Conditions for Gel Method

Sr. No.	Conditions	Bi ₂ S ₃
1	Density of Sodium Metasilicate	1.04 gm/cm^3
2	Amount of 2N Acetic Acid	5ml
3	pH of Gel	4.40
4	Temperature	Room Temperature
5	Concentration of Bicl ₃	0.5 ml, 1M
6	Gel Setting Time	15 days
7	Gel Ageing Time	90 Hrs.
8	Period of growth of crystals	30 days



International Journal for Research in Applied Science & Engineering Technology (IJRASET)

ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 6.887 Volume 6 Issue VII, July 2018- Available at www.ijraset.com

V. CONCLUSION

Crystals of Bi_2S_3 were grown by a simple and inexpensive Gel technique using single diffusion method. The optimum growth conditions were established. Crystals having different morphologies and habits. Crystals are quite transparent and Bi_2S_3 single crystals are also grown by Horizontal Zone Leveling method. good quality single crystals were obtained. Crystals are opaque and not in shape.

VI. ACKNOWLEDGEMENT

The author would like to acknowledge her guide and Head Prof. K.C.Poria for providing laboratory facilities and encouragement for this work.

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