



## Growth of Cr- doped Bismuth Trisulfide ( $\text{Bi}_2\text{S}_3$ ) Single Crystals using Gel method

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**ABSTRACT:** Cr-doped  $\text{Bi}_2\text{S}_3$  crystals were grown in silica gel method. By varying the growth parameters such as pH of gel, concentration of supernatant and reactant etc., the Optimum condition to grow doped  $\text{Bi}_2\text{S}_3$  crystals has been obtained. If the crystals were Grown at constant temperature, it is established that the transparency and size of the grown Crystals are increased.

**Keywords:** gel method, optimum conditions, growth, doped  $\text{Bi}_2\text{S}_3$  crystals

### Introduction

$\text{Bi}_2\text{S}_3$  is one of the important materials whose band gap energy 1.7 eV lies in visible solar energy spectrum which makes it useful for solar energy conversion devices.  $\text{Bi}_2\text{S}_3$  belongs to the members of the V-VI group of compound semiconductors which are technically important materials in view potential applications in thermoelectric conversion, optoelectronic devices and IR spectroscopy and excellent properties like photosensitivity, photoconductivity and solar cell.  $\text{Bi}_2\text{S}_3$  exhibits pronounced positive photoconductivity upon visible light exposure, and is a good candidate for optical switches. [1– 2]. Due to its significant thermoelectric effect, this material is important in thermoelectric applications these materials, in which the magnetic dopant concentration is typically a few percentage points, are known as diluted magnetic semiconductors (DMS). The main cause of the new physical properties owned by these DMS materials is attributed to the sp-exchange interaction between the host nonmagnetic semiconductor sp-band and the partially occupied transition metal-state [3– 4]. Synthesis of several kinds of transition metal doped semiconductor nano crystals are reported in the literature, such as Mn-doped  $\text{Bi}_2\text{S}_3$  [5] The Bismuth Tri-sulphide ( $\text{Bi}_2\text{S}_3$ ) is a quite interesting compound and hence some attempts Have been made to grow. Here Cr-doped Bismuth Tri-Sulphide is grown by gel technique in Silica gel medium and the grown crystals analyzed under the various characterizations.

### Experiment method

To grow the Bismuth Tri-Sulphide [ $\text{Bi}_2\text{S}_3$ ] crystals, the required Silica gel medium was prepared by adding the Sodium-Metasilicate solution of specific gravity 1.04 g/cc drop by drop with constant stirring by using magnetic stirrer into the 5 ml (2N) Acetic Acid till the pH value 4.4 was set for the mixture. To the above Sodium MetaSilicate solution of pH 4.4, 15 ml the aqueous solution of  $\text{H}_2\text{S}$  Gas Water solution was added as inner reagent with constant stirring. This mixture was then transferred to the test tube of length 15 and 2.5 cm diameter. To keep the solution free from dust and impurities, care was taken to cover the test tube with cotton. The gel was usually set within 13 days. It was left for 66 to 72 Hours for gel ageing and then the outer reagent, aqueous solution of 0.5 M Bismuth Chloride ( $\text{BiCl}_3$ ) and 0.5 M  $\text{CrCl}_3$  was added on to the top of the gel. The outer reagent was added down the sides of the test tube using a pipette and not directly on to the gel medium. Owing to the diffusion of the outer reagent into the gel medium and its reaction with the inner reagent, crystals started growing. Nucleation was observed within 48 Hours of addition of the outer reagent. Circular shaped,

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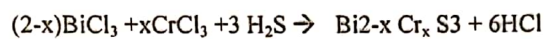
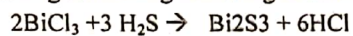
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opaque and brittle crystals were observed. The experiment was carried out at an ambient temperature of about 28 °C. The reaction between Bismuth Chloride, do pent and H<sub>2</sub>S Gas Water solution in gel medium resulted in the growth of Circular shaped Bismuth Tri-Sulphide [Bi<sub>2</sub>S<sub>3</sub>], crystals. The reaction that takes place in the gel medium.

Chemicals used:-

- 1) Sodium metasilicate powder (A.R. grade) Na<sub>2</sub>SiO<sub>3</sub>·9H<sub>2</sub>O (M.W.284.20)
- 2) Acetic acid (A.R. grade) CH<sub>3</sub>COOH
- 3) H<sub>2</sub>S Solution
- 4) Bismuth chloride (A. R. grade) BiCl<sub>3</sub> (M.W. 315.33)
- 5) Double distilled water
- 6) Chromium chloride (A.R.grade) CrCl<sub>3</sub>·6H<sub>2</sub>O (M.W.210.35)

The crystals were grown using following chemical reaction.

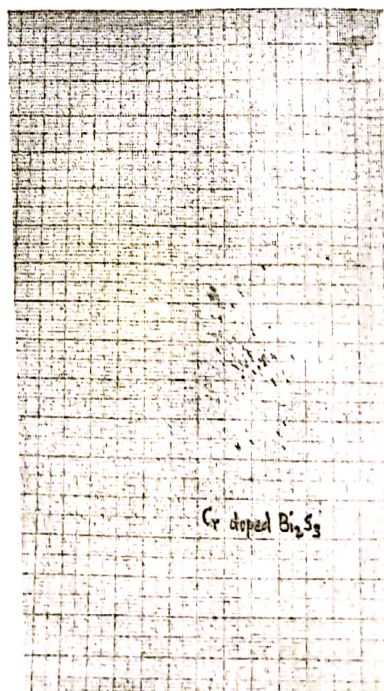


The grown crystals are shown in Fig..

Fig 1 Crystal's growth in test tube Cr- doped Bi<sub>2</sub> S<sub>3</sub>



Fig 2 Few Crystals of Cr- doped Bi<sub>2</sub> S<sub>3</sub>



Optimum Conditions for Gel Method:-

Table I

Condition	doped Bi <sub>2</sub> S <sub>3</sub>
H <sub>2</sub> S GAS water solution	---
Conc. of Bismuth chloride	0.5 M
Conc. of CrCl <sub>3</sub> 6H <sub>2</sub> O	0.5M
Conc. of Acetic acid	2N
Gel setting period	3 days
Gel aging period	2 days
Period of growth	30 days
Temperature	Room temperature
Gel pH	4.4
Gel density	1.04 gm/cm <sup>3</sup>

**Results and discussion**

Crystals in little mm size were obtained. Study of kinetics of growth parameters reveals some interesting information. These types of Circular rings of crystals were reported by Liesgang. The optimum growth conditions for various parameters were found and are reported in Table 1. Different parameters such as gel density, gel setting time, gel aging time, Concentrations of reactants, pH of gel etc have the considerable effect on the growth rate

**Effect of gel density:** - It was observed that the transparency of the gel decreases as the gel Densities increases .Gels with higher densities set more rapidly than the gels with lower densities.

**Effect of pH of gel:**-It was observed that as the pH increased the transparency of the gel Decreased..The crystals growing at higher pH values were not well defined. This was due to contamination of the crystals with silica gel. It was observed that as the pH of gel increased the number of crystals decreased.

**Effect of gel aging time:** -Gels were allowed to age for different period before adding the feed Solutions. It was observed that as aging time of gel increased, the number of crystals decreased. This is because gel aging reduces the cell size and consequently the rate of diffusion of ions into the gel .

**Effect of concentration of supernatant:-** Bismuth chloride and Chromium chloride is used as supernatant with different concentration from 0.2M to1.2M is added over the set gel. It was observed that at 0.2M of concentration of supernatant very few nucleations were observed with very small size of the crystals and crystals were not well defined.

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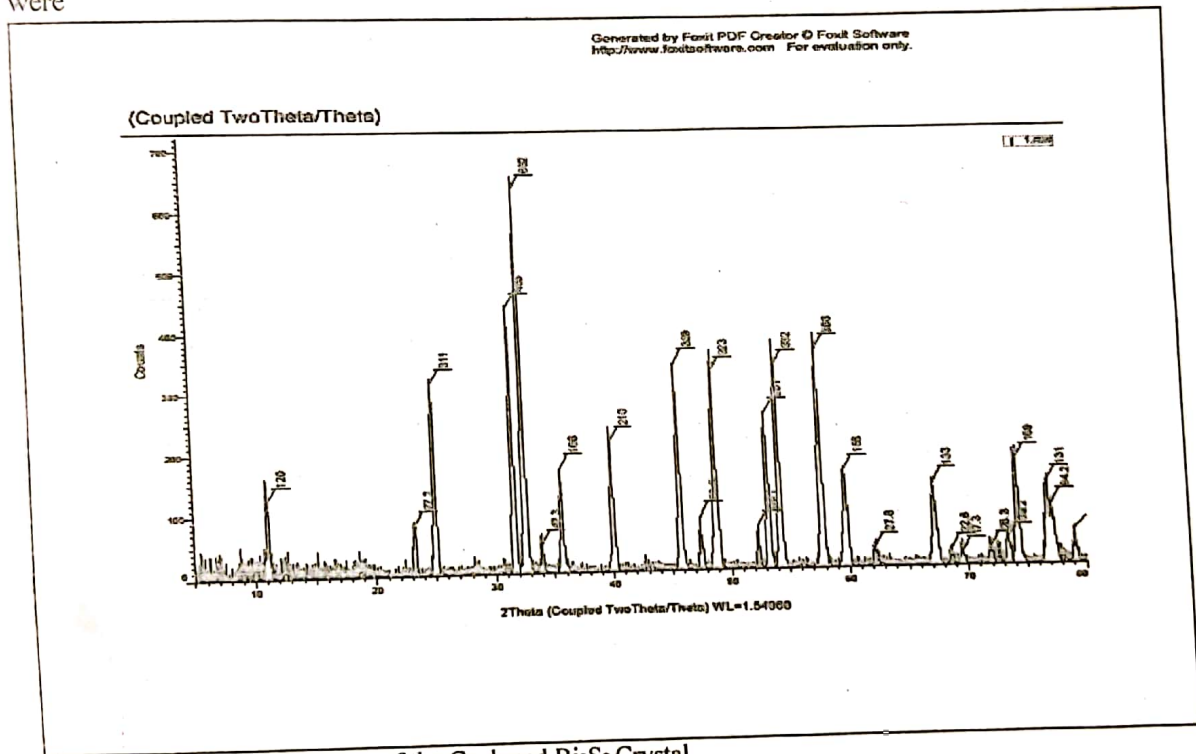
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**Effect of concentration of reactants:-**

The reactant used for the growth of  $\text{Bi}_2\text{S}_3$  in gel is  $\text{H}_2\text{S}$  gas as the inner reactant. Initially a gel solution (sodium silicate) of density 1.04 g/ml was prepared and  $\text{H}_2\text{S}$  gas was passed through the gel solution for different time intervals (say 5, 10, 15 minutes etc). This gel solution after the impregnation of the inner reactant was acidified with 2N acetic acid for gelation and was taken in straight tubes. This reactant on diffusion into the gel medium reacts with the inner reactant ( $\text{H}_2\text{S}$ ) changing the entire gel medium black in colour After a week's time nucleation sites were observed.



XRD spectrum of the Cr-doped  $\text{Bi}_2\text{S}_3$  Crystal

From XRD spectrum no peak from impurities can be observed in the XRD spectrum of the Cr-doped crystal that is no other different crystalline phases was formed.

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December 2018 Volume 8 Number 12  
UGC Approved Journal

1/12/2018



International journal of basic and applied research

www.pragatipublication.com

ISSN 2249-3352 (P) 2278-0505 (E)

Cosmos Impact Factor-5.86

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