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Synthesis, Crystal Structure and Thermal Properties of Cr-doped Bi_2S_3 Crystals by Gel Method

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ABSTRACT: Cr-doped Bi_2S3 crystals were grown in silica gel medium by single diffusion method. The grown crystals were characterized by thermo analytical techniques (TGA and DSC), X-ray powder diffraction (XRD), by powder X-ray diffraction analysis the crystal structure is confirmed to be Orthorhombic having lattice parameters a = 4.76 A°, b = 3.97 A°, and c = 12.39 A°. Thermal study reveals that Iron doped Bismuth Tri-Sulphide crystal is Di-hydrous. TGA and DSC analysis shows a remarkable thermal stability.

KEYWORDS: Gel method, Cr-doped Bi_2S_3 crystal, X-ray diffraction, TGA and DSC

I. INTRODUCTION

Several investigators used gel method for growing different crystals [1-4] Bismuth sulphide is V-VI group compound semiconductors application in optoelectronic devices [5]. Bismuth sulphide semiconductor with band gap energy ranges 1.3 eV to 1.7 eV. [6, 7].Optical and structural properties of Bi_2S_3 with various dopants are studied. Fe was reported [8-10] all these researchers have used different synthesis methods. The purpose of this paper is to report the synthesis of single crystals Cr doped Bi_2S_3 using gel method. The thermal properties of the grown crystals were studied using Thermogravimetric and differential scanning calorimetry thermal analysis. TGA and DSC analysis shows a remarkable thermal stability.TGA and DSC analysis suggest that the thermal stability of Bismuth tri- Sulphide crystal decreases due to Chromium doping.

II. LITERATURE REVIEW

Growth of crystal is an important aspect of materials science. The art and the science of crystal growth have developed very much like any other branch of science. Bismuth tri sulphide is a ferroelectric semiconductor having Para electric phase and exhibiting interesting photo conducting behaviour. Bi_2S_3 are narrow band gap semiconductor with layered structure are interesting, and important because of major contribution in solar cells, photo detectors, opto-electronic light amplifiers, Lasers, photo electronic etc. Nano crystals of Bi_2S_3 in different shape and sizes have applications in hydrogen storage, high-energy battery and catalytic field. By looking to wide applications of and importance of the Bi_2S_3 single crystals, these crystals were grown from melt, using modified Bridgeman Stock burger, Chemical Vapour Transport method, and Skeleton layered structured crystals were grown by hydrothermal method. Flower like nanobelts, Straw-tied nano wires are synthesized by facile decomposition and precursor solution method. There are very few reports available in the literature on single crystal growth of Bi_2S_3. Synthesis of nanomaterial has become a prolific area of investigation due to their wide range of applications.

III. METHODOLOGY

The required Silica gel medium was prepared by adding the Sodium-Metasilicate solution (specific gravity 1.04 g/cc) drop by drop with constant stirring into the 5 ml Acetic acid till the pH value was 4.4. Then add 15 ml H_2S Gas Water solution was as inner reagent. The gel was usually set within two to three days. It was left for two days for gel ageing and then the outer reagent, aqueous solution of Bismuth Chloride ($BiCl_3$) and $CrCl_3$ was added on to the top of the gel. The mixture was carefully poured along the walls of the tube with the help of pipette over the set gel, in order to avoid gel breakage. After addition of upper reactant, mixture small nucleation was observed within 36 hours.. The reaction between Bismuth Chloride, dopant and H_2S Gas Water solution in gel medium resulted in the growth of doped Bismuth tri-Sulphide crystals.

$$2\text{-}x\ BiCl_3 + xCrCl_3 + 3H_2S = Bi_{2-x}Cr_xS_3 + 6HCl$$

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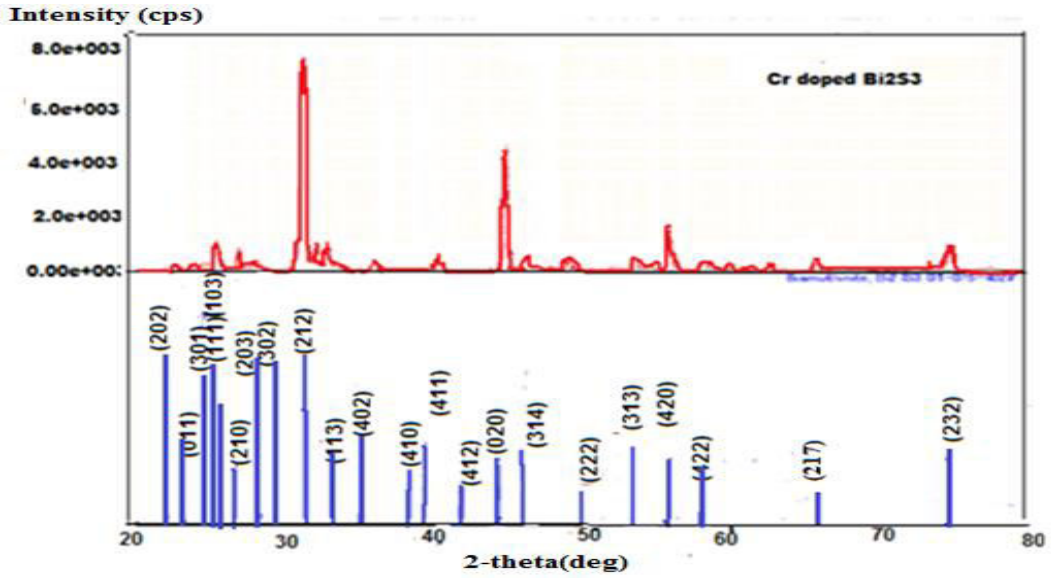
IV. EXPERIMENTAL RESULTS

X-Ray Diffraction Study



The grain size for grown crystals were derived using x-ray diffraction line broadening analysis based on the Scherrer formula

The crystallite size determine by using relation $D = \frac{0.9(\lambda)}{(\cos\theta)\beta}$

β is full width of half maxima in radian = 0.229 ^{0} in radian β =0.003839

$D = \frac{0.9\times1.54056}{0.003839\times\cos15.7}$ = 37.66 nm

Average grain size is 37.66 nm.

Grain size of Cr doped Bismuth tri sulphide crystals =37.66 nm

In Orthorhombic crystal structure the length of unit cells are different . i.e. $a \neq b \neq c$ & $\alpha = \gamma = \beta = 90^0$. Chromium doped Bismuth Tri-Sulphide crystals fulfil the condition of Orthorhombic structure, having lattice parameters a = 4.76 Å, b = 3.97 Å, and c =12.39 Å

TGA and DSC Measurements

Thermo gravimetric analysis (TGA)

Figure shows the TGA curve of Chromium doped Bismuth tri sulphide crystal. The different stages of decomposition of Chromium doped Bismuth tri sulphide crystals are presented in table

Stage Decom.	Decomposition Temperature range	%weight loss Observed	% weight loss Calculated	molecule loss in stage
1	29.58to 500	12.403	11.26	$2H_2O$ -- $2SO_2$ ↑
2	500 to 786.271	6.812	17.58	$4SO_2$ ↑
	Totalweight loss	19.215	28.84	
	Residue Stable[$2Bi_2O_3$] ↓	80.785	71.16	2 $CrBi_2O_3$ ↓

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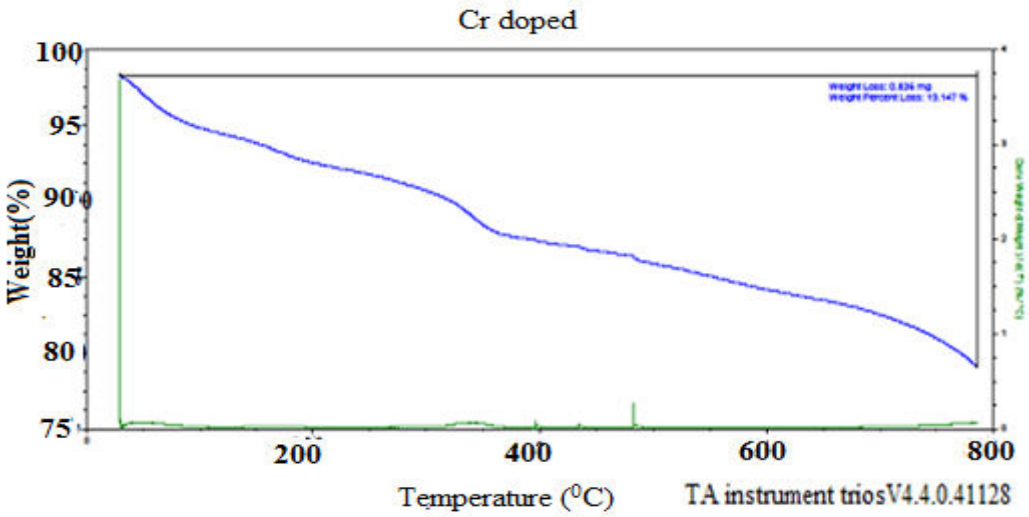


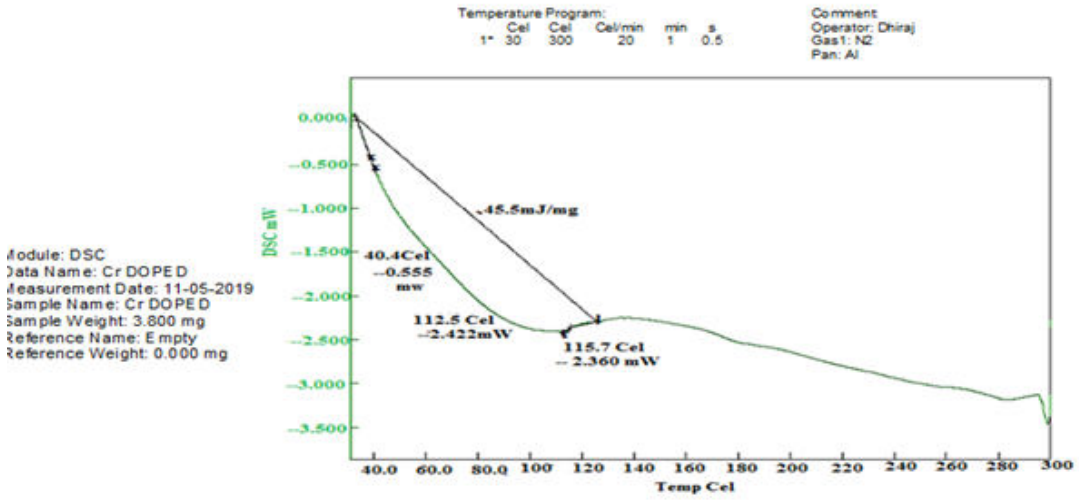
The stages of decomposition of Cr doped Bi_2S_3 crystals are given in table. The first stage of decomposition occurs at the temperature range 29.58 to 500 ^{0}C in which the observed weight loss was found to be 12.403 % which is good agreement with calculated weight loss 11.26 %. This weight loss is attributed to loss of [$2H_2O$ +$2SO_2$] and decomposition is in continuous manner.

Decomposition, at second stage occurs in the temperature range 500 to 786.271^{0}C in which observed weight loss of 6.812 % nearly agreed with calculated weight loss %. Here observed weight loss appear to be less as compared with calculated can be attributed due to incomplete decomposition of Bi_2S_3.. This weight loss is attributed to loss of $4SO_2$ and decomposition is in continuous manner. The remaining product finally turns into residue $CrBi_2O_3$ is confirmed at 790.112^{O}C.The observed residue weight was 80.785 %, which is nearly good agreement with calculated residual weight 70.005%. This confirms the present of Chromium and Bismuth in grown crystals.

Differential scanning calorimetry

Specimen material and reference material both are maintained at almost the same temperature during the conduct experiment. Here we can measure the transition state of the material.

The differentials scanning calorimetry analysis of the grown crystals was recorded between 30^{0}C to 300^{0}C in the nitrogen atmosphere using Metlae TA 4000. The initial weight of sample was 3.8 mg and heating rate was maintained at 20^{0}C/min. The DSC curve for Cr doped Bismuth tri sulphide gel grown crystals is shown in figure . The initialization temperature to start the thermo analysis of the grown crystals was from 31.22^{0}c and phase change ended at peak end-down temperature of 112.3^{0}c.The temperature at which the sample and the reference come to thermal balance by thermal diffusion. The peak appeared in the DSC curve at 140.05^{0}c indicates the phase transformation due to loss of water molecules and formation of stable anhydrous Cr-Bi_2S_3 crystals. This is the good agreement with the TGA curve.



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V. CONCLUSION

Chromium doped Bi2S3 crystals can be successfully grown by silica gel method. Gel setting period is strongly dependent on pH of a mixture of sodium meta silicate, acidic acid and density of sodium meta silicate. The results of TGA analyses indicated that the first stage of decomposition occurs at the temperature range 29.58 to 500 ^{0}C in which the observed weight loss was found to be 12.403 %. This weight loss is attributed to loss of [$2H_2O$ +$2SO_2$] and decomposition is in continuous manner. Decomposition, at second stage occurs in the temperature range 500 to 786.271^{0}C in which observed weight loss of 6.812 % nearly agreed with calculated weight loss %. Here observed weight loss appear to be less as compared with calculated can be attributed due to incomplete decomposition of Bi_2S_3. This weight loss is attributed to loss of $4SO_2$ and decomposition is in continuous manner. The remaining product finally turns into residue $CrBi_2O_3$ is confirmed at 790.112^{O}C. The peak appeared in the DSC curve at 140.05^{0}c indicates the phase transformation due to loss of water molecules and formation of stable anhydrous Cr-Bi_2S_3 crystals.

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