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Synthesis and Structural Analysis of Bi_2S_3 Crystals by Gel Method

K. B. Patil

Physics (Electronics) Department

J. D. M. V. P. Co-Op. Samaj's

Shri S. S. Patil Arts, Bhausaheb Shri T. T. Salunkhe Commerce & Shri G. R. Pandit Science College, Jalgaon

Abstract: *Bi_2S_3 crystals were grown in silica gel medium at room temperature by single diffusion method. The optimum growth conditions were established by varying various parameters such as pH of gel solution, gel concentration, gel-setting time, concentration of reactant etc. Gel was prepared by mixing sodium metasilicate ($Na_2SiO_35H_2O$), Acetic acid (CH_3COOH), inner reactant Na_2S water solution and Supernant Bismuth chloride ($BiCl_3$) at pH value 4.3. The effects of parameters such as gel aging, gel pH, the density of gel on growth of crystals were studied. The grown crystals characterized by Surface morphology of materials were studied by SEM. It shows grown crystals are nano wires in shape, flat and the plates with the sharp edges were observed. And nanoscale rod like structure of the grown crystals and not affected significantly by the doping. XRD analysis the crystal structure was confirmed as Orthorhombic having lattice parameters a = 10.86Å, b = 4.06 Å, and c = 10.22 Å both elements Bi and S elements can be seen in grown crystal confirmed by EDAX. Fourier transforms infrared spectroscopy (FTIR), the confirmation of the crystal formation was done by carrying out XRD study. FTIR study gives the information of functional groups in a crystal.*

Keywords: Bi_2S_3 Crystal, growth, XRD, EDAX SEM and FTIR

I. INTRODUCTION

Gel method is very simple and useful method to grow the crystals, which are insoluble or slightly soluble. This method can be controlled [1-3] by various parameters Bismuth tri sulphide has been attracting a considerable interest owing to its potential application in thermoelectric, electronic and optoelectronic devices and IR spectroscopy. In addition, it has energy band gap of 1.3 to 1.7 eV, which is suitable for making photodiode arrays and photovoltaic. A band gap can be tuned depending on the size of the subcomponents [4] Bi_2S_3 exhibits pronounced positive photoconductivity upon visible light exposure, and are a good candidate for optical switches.[5-6] Bi2S3 is a layered semiconductor that crystallizes in the orthorhombic system .

II. METHODOLOGY

To grow bismuth Tri-Sulphide crystals, the desired silica gel medium was ready by adding Sodium-Met silicate 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 until the pH value 4.4 was set for the mixture. To the above sodium Meta silicate solution of pH 4.3, Take 15 ml the aqueous solution of Na2S was added as inner reactant with constant stirring. This mixture was then transferred to the test tube. To keep the solution free from dust and impurities, care was taken to cover the test tube with cotton. The gel was typically set within 4 days. It was left for 48 to72 Hours for gel ageing and then the outer reactant, aqueous solution of 0.5M bismuth Chloride (BiCl3) added as Supernant over the set gel. The outer reagent was added down the sides of the test tube using a pipette and ultimately on to the gel medium. The diffusion of the outer reactant into the gel medium. Its reaction with inner reactant, Nucleation was observed within 48 Hours of addition of the outer reactant. The experiment was carried out at an ambient temperature of about 28^0C. The various optimum conditions for the growing Bi2S3crystals were found. The reaction between Bismuth Chloride and Na2S solution in gel medium resulted within the growth of Bi_2S_3 crystals. Shown in Fig. 1

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2.1 Chemicals Used

1. Sodium metasilicate powder (A.R. grade) Na2SiO3, 9H2O (M.W.284.20)
2. Acetic acid (A.R. grade) CH3COOH
3. Sodium sulphide ((A. R. grade)) Na2S
4. Bismuth chloride (A. R. grade) BiCl3 (M.W. 315.33)
5. Double distilled water

The crystals were grown using following chemical reaction.

$2XCl3 + 3Y2S \rightarrow X2S3 + 6Y (Cl)$ Where X=Bi and Y= Na.

2.2 Process PARAMETERS OPTIMUM CONDITIONS

Condition Bi2S3
Conc. of Na2S 0.5 M
Conc. of Bismuth chloride 0.5 M
Conc. of Acetic acid 1N
Gel setting period 4 days
Gel aging period 3 days
Period of growth 34 days
Temperature 28^0C (Room temperature)
Gel pH 4.3
Gel density 1.04 gm/cm^3

III. RESULTS AND DISCUSSION

3.1 X-Ray Diffraction Study

Fig 1

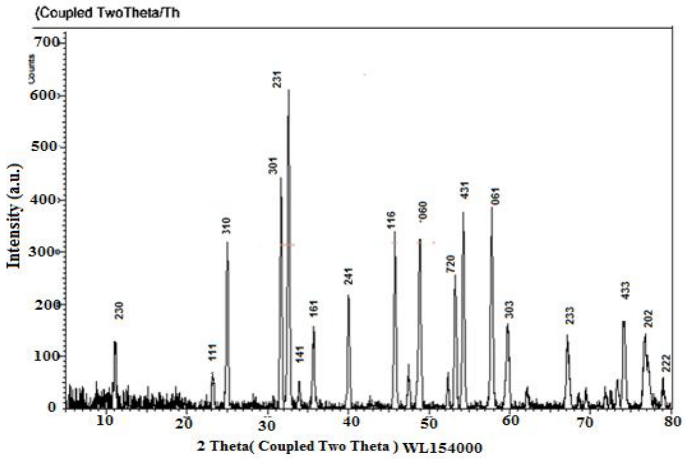

Fig 2 Powder X-ray diffraction pattern of Bismuth tri sulphide Crystal

Figure.2 shows XRD spectrum of Bi2S3 sample. It found that no peak from impurities can be observed in XRD spectrum of Bi2S3 sample, proving that none of the other different crystalline phases was formed. Grain size calculated from broadening of XRD peaks using the Scherer's formula

$D = K\lambda/\beta\cos\theta$

Where K is constant equal to 0.9, λ is wavelength of CuKα radiation (λ= 1.5409Å), β is the full width at half maxima (FWHM) of XRD peaks. In the present work, the crystallite size of the Bi2S3 estimated from X-ray line broadening of the maximum intensity peak. The crystalline grains mainly oriented along the (231) plane. The crystalline size calculated using Scherer's formula

D = 0.9×1.54056 *A*°= 0.9X0.1506/ .005758X COS 16.29
= 26.21.66nm

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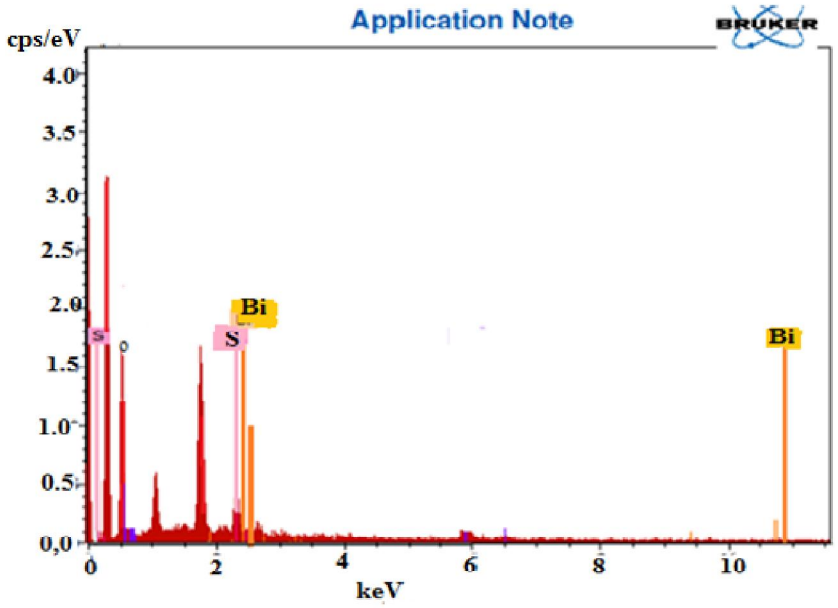
D is grain size (i.e. the diameter of the crystal particle in the material) the calculated average particle size is 26.21nm. From standard data and XRD data, the calculated lattice parameters are a=2.502 Å, b=12.44 Å and 41.30 Å i.e. $a \neq b \neq c$ and $\alpha = \gamma = \beta = 90^0$ these are the condition for orthorhombic crystal structure.

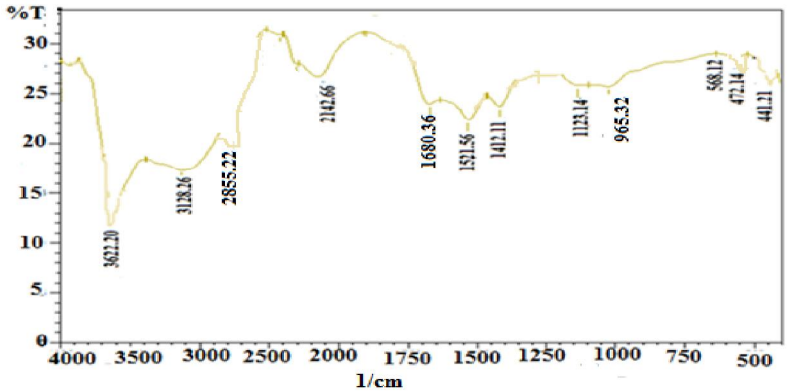
3.2 EDAX

Elemental analysis of gel grown Bismuth tri sulphide crystals was done using Bruker instrument at NMU Jalgaon. Figure indicates EDAX spectra of Bi2S3 grown crystal.. During EDAX measurement different area were formed and the correspondence peaks are shown in figure. Both elements Bi and S elements can be seen in grown crystal.



Experimental and theoretical calculated composition obtained from EDAX for various constituent elements present in Bismuth tri sulphide.

3.3 Fourier Transform Infrared Spectral Analysis FTIR



The FTIR Spectrum identifies chemical bonds in a molecule by producing an infrared absorption spectrum. Therefore, an important use of FTIR is the Recognition of unknown functional group present in the chemical compounds. The FTIR Spectra of Bismuth tri sulphide (Bi2S3) is as shown in fig. From the observed result of FTIR spectra of grown Bi2S3 crystals, Intensity peaks, Band assignments and band range .

A few of the major vibration modes are empirically assign here the bands around 3700 to 3400 cm-1 recognized to asymmetric and symmetric O-H stretching. The O-H stretching frequency appeared between 3603.15 to 3064.99 cm-1 is possibly due to stretching vibration of Hydroxyl group-H bonded possibly will be due to O-H stretching or Si-OH

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bond. The band appearing at 2855.22 cm-1 can be recognized to C-H stretching due to alkyl group. It may be the present due to Bismuth acetate impurity. The band appearing at peak 1680.36 cm-1 can be recognized to C-O stretching. In the range 970 to 920 cm-1 band appearing can be attributed to C-H group. The band appearing at 471.14 and 441.21 can be recognized to metal Bismuth - Sulphur bond in same plane.

3.4 SEM Analysis

Grown sample of Bi2S3 was Scanning Electron Microscopy characterization taken at Department of Chemical technology, Kavayitri Bahinabai Chaudhari North Maharashtra University, Jalgaon. Sample images were taken magnifications of 400.00 nm and 200.0 nm. Grown Bi2S3 crystals Scanning Electron Microscopy images demonstrated in Figure 4.3. It was seen from SEM images that as growing crystal exhibit the aggregation of the particles at room temperature. It may be due to the smaller particle size. An entire images surface show is enclosed with figs of unlike shapes and size. Some of them are roughly observe to be spherical and triangle shape

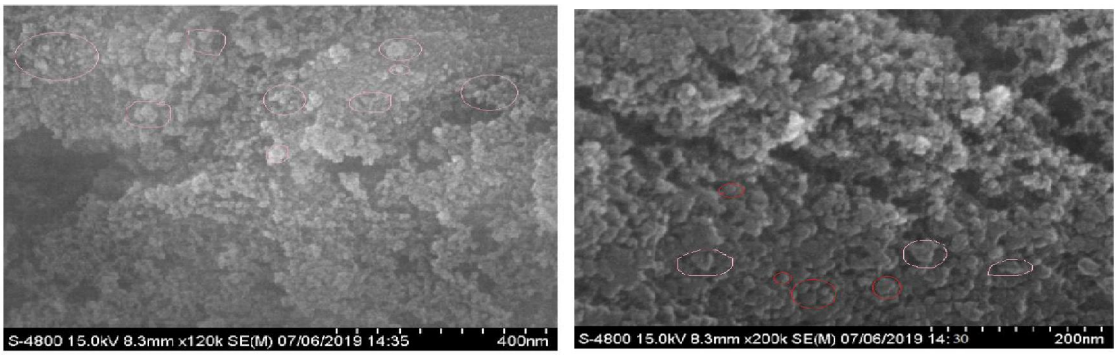

Figure (a, b): SEM picture Bismuth tri sulphide crystals

IV. CONCLUSION

The Bi2S3 crystals can be successfully grown by silica gel method. The gel setting period is strongly dependent on the pH of a mixture of sodium meta silicate, acidic acid and density of sodium meta silicate. X-ray diffraction pattern shows that the sample was crystalline in nature. FTIR study suggests A few of the major vibration modes are empirically assign here the bands around 3700 to 3400 cm-1 recognized to asymmetric and symmetric O-H stretching. The O-H stretching frequency appeared between 3603.15 to 3064.99 cm-1 is possibly due to stretching vibration of Hydroxyl group-H bonded possibly will be due to O-H stretching or Si-OH bond. from SEM images that as growing crystal exhibit the aggregation of the particles at room temperature. It may be due to the smaller particle size. An entire images surface show is enclosed with figs of unlike shapes and size. Some of them are roughly observe to be spherical and triangle shape. The elemental composition was determined by EDAX studies, which show that the Presence of Bismuth and sulphur.

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