Research Paper

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Characterization of Structural, Morphological and optical studies of Nano Crystal by chemical route

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ABSTRACT

In present study, we were able to successfully synthesize single-crystalline $BaSnO_3$ nano crystal using a straightforward Chemical precipitation technique. $BaSnO_3$ have a length of up to several micrometers and a diameter of 25-50 nm and are single-crystalline, structure obtained from XRD and Scanning electron microscope (FE-SEM) and Optical properties also used to study about the morphological structure of nanocrystal.

Keywords: XRD, UV-Visible Spectroscopy, FESEM, Chemical precipitation techniques etc.,

INTRODUCTION

The architecture of electronic devices demands the multifunctionality of the basic materials used in it. TCOs are such class of materials that can be insulating, semiconducting and metal depending on its chemical constituents and these materials displays various properties like superconductivity, magnetism and ferroelectricity etc. Perovskites are the materials that falls in the category of Transparent Conducting oxide (TCO) or Transparent oxide Semiconductors(TOS) due to their richness in multifunctional properties. But mobilities in TCOs are strongly dependent on the temperatures, phase purity and electron effective mass. One of the key issues in mobility driven devices is that it is temperature dependent (μ is proportional to $T^{-3/2}$)due to lattice scattering. It is very efficient to address both factor

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simultaneously, however in very interesting Perovskites material such manoeuvring is possible. Thus research advancements in Perovskites .materials are carried extensively to explore materials whichhave higher carrier mobilities. Alkaline earth stannates with chemical formula $ASnO_3$ (A= Ca,Sr and Ba) are the class of materials that has high mobility (up to ~ 350 $cm^2V^{-1}s^{-1}$) reported at room temperature (RT). Generally at room temperature, the binary oxides TCOs show very low mobility ~ 1 to 3 $cm^2V^{-1}s^{-1}$ but ternary oxides like alkaline earth stannates having dispersed conduction has high RT mobilities. La doped Barium stannates ($Ba_{1-x}SnLa_xO_3$) has shown the highest mobility of 320 $cm^2V^{-1}s^{-1}$ in degenerate semiconducting TCOs [1-3]. Thus these features motivate us to explore $BaSnO_3$ system. In this chapter detailed study of $BaSnO_3$ system is extensively studied from synthesis to structural, optical, composition, electrical studies and annealing studies. Also, Al doped $BaSnO_3$ system is studied structurally,morphologically and optically.

Experimental Techniques

Materials and Methods

SnCl2.2H2O and BaCO3, the precursor for NH3.H2O , NaOH the oxide source, Ethanol, Deionized Water were purchased from Merck. Because the chemicals were of analytical regent grade with 99 percent purity, they were utilized exactly as received. Simple chemical route was used to synthesize $BaSnO_3$ nano crystal materials. X-ray powder diffraction method UV were used to characterize structural, morphological and optical respectively

SYNTHESIS PROCEDURES

The synthesis was done through facile chemical route keeping the focus on the concern of phase purity. In the synthesis of $BaSnO_3$ by chemical co precipitation method the optimization of pH for phase purity was done by using two different routes as is described below:

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method1: Precursors $SnCl_4.5H_2O$ and $BaCO_3$; NH_3 as base

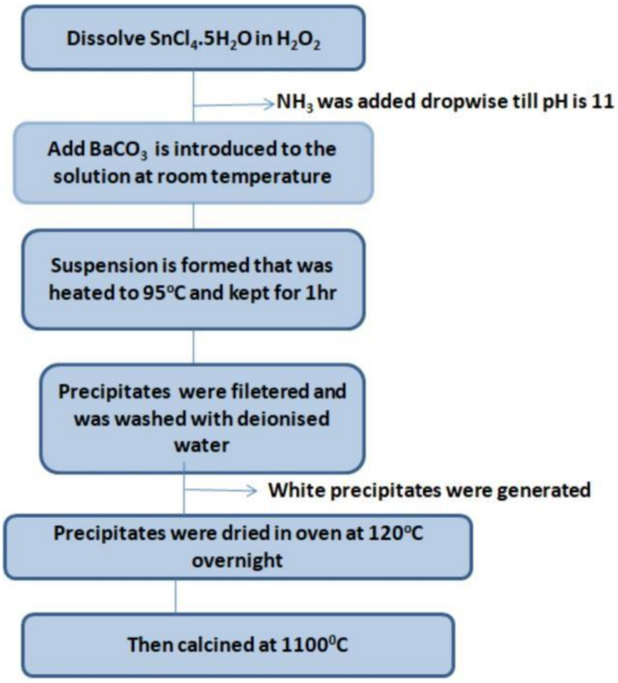

Figure 1.1: Schematic diagram of Sample preparation by Route 1

In this $SnCl_4.5H_2O$ was dissolved in H_2O_2 and NH_3 was added drop-wise till it reaches pH 11.Many experiments were performed on different pH in range 8, 9 and 10. When the desired pHis reached then $BaCO_3$ was added precipitates were formed. Then those suspensions of mixed solutions were heated to 95°C for 1 hr. The precipitates were washed with deionized waterand were dried in the oven for 24 hrs. Then the dried white colored precipitates werecalcined at temperature 1100°C. The steps followed are shown below in the form of flowchartin figure 1.1.

RESULTS AND DISCUSSION

XRD STUDIES

Through the XRD pattern shown in figure 1.2 few important observations are drawn. Firstly,at pH above 11.5 there is SnO_2 impurity as we can see the peak centred at 2θ values 26^o , 32^o. as compared to BSO which have higher phase purity. Secondly, BSO-13 and Al doped BSO-
13 has significantly different XRD pattern. Thus these two observations points towards important conclusions regarding the role of pH and dopants in deciding the phase purity of BSO.

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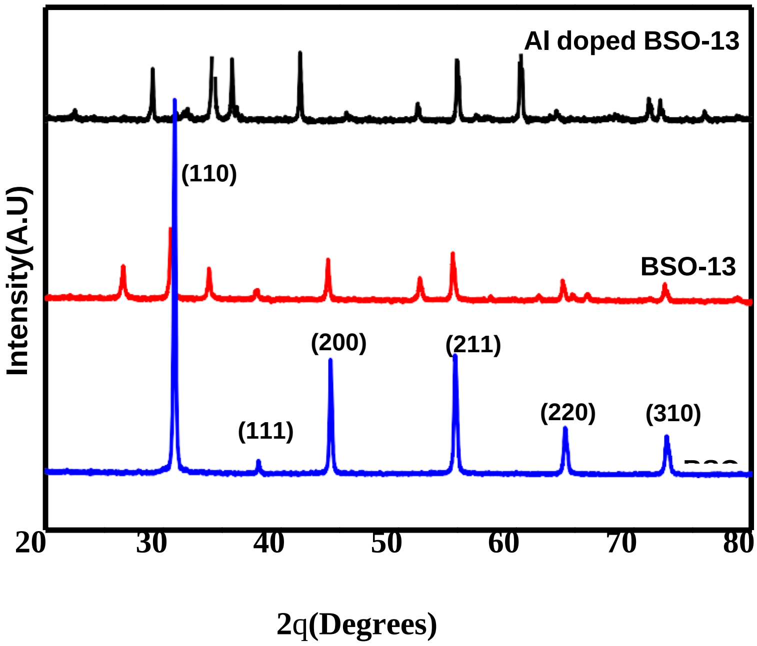

Figure 1.2: XRD pattern of BSO, BSO-13 and Al-doped BSO

Table 1: Ionic radii of ions

Ions	**Ionic radii (Å)**
Sn^{4+}	0.69
Ba^{2+}	1.35
O^{2-}	1.4
Al^{3+}	0.53

As discussed earlier that pH changes the morphology. At pH -13 instead of micro rods agglomerated 3-D flakes are observed as shown in figure 1.3 . From XRD observation again the emphasis is on dominant role of pH in phase also. As pH values prompts the change in solubility, at higher pH solubility decreases and hence it affects the whole process of precipitate formation by affecting the phase purity and morphology. Comparison of BSO-13 and Al doped BSO-13 shows that Al doped sample has multiple phases. It also has SnO_2peaks with high intensity Al peak at 38° of 2θ is observed and is shown in the magnified XRDimage in figure 4.16. $BaSnO_3$ phase is present but it is suppressed effectively due to the presence of other phases which are present due to changes corresponding to pH. The ionic radii of Al, Ba, Sn and O are tabled below in table 4.4.We observed that there is difference between ionic radii of Al, Ba & Sn. Thus, Al doped in the BSO-13 system makes it highly unstable and suppresses the BSO-13 phase drastically.

MORPHOLOGICAL STUDIES: FESEM

The morphological analysis of BSO-13 and Al doped BSO-13 is carried out using FESEM and is shown in figure 1.3. It is clear that Al doping affects the morphology as well. Both the samples have agglomeration of flakes. Al-doped sample has sharp edges, while BSO-13 has rough edges.

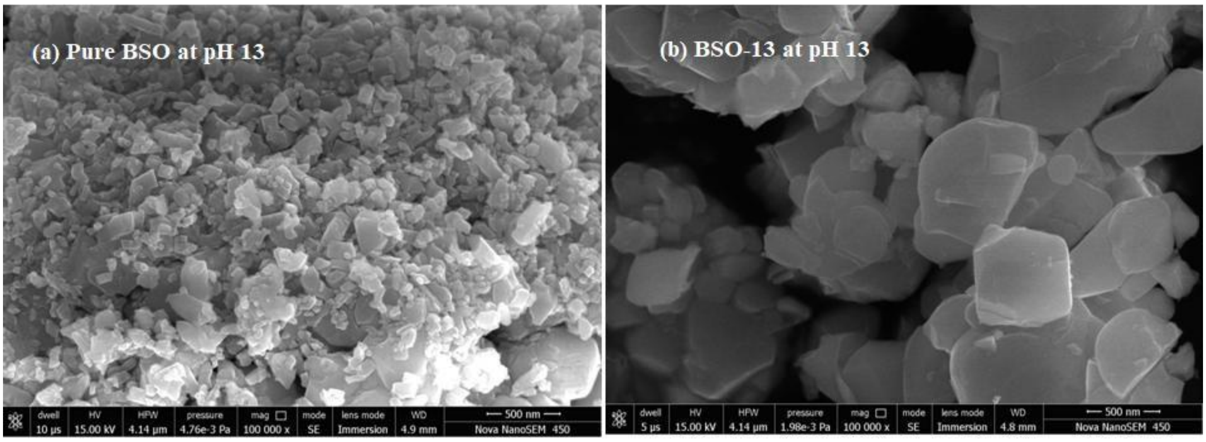

Figure 1.3: FESEM of (a) pure BSO-13 powder sample (b) Al doped BSO-13 at pH13

UV/VISIBLE SPECTROSCOPY

The optical studies of Al-doped BSO-13 and BSO-13 have been conducted using UV-Vis spectroscopy. Figure 1.4 shows the absorption spectra of both samples and figure 1.5 is the tauc plots of both sample. From Tauc plots it can be conferred that both samples are indirect band gap material.

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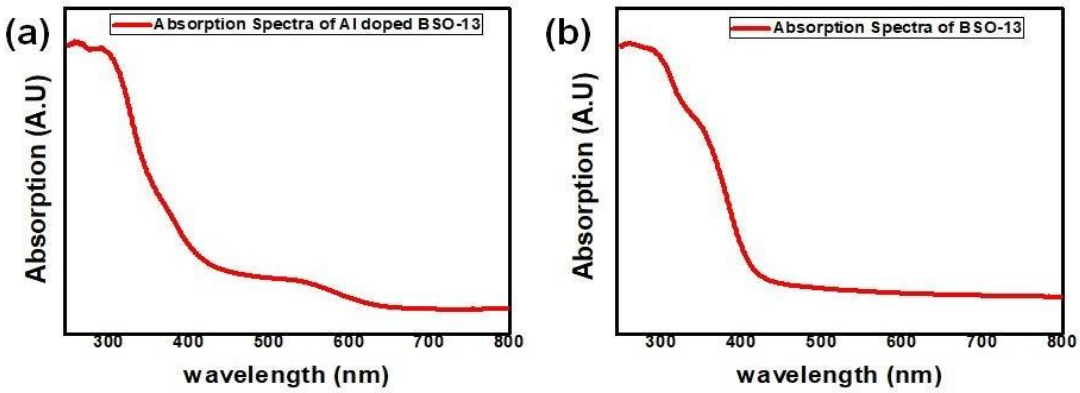

Figure 1.4: Absorption spectra of (a) Al doped BSO-13 (b) BSO-13

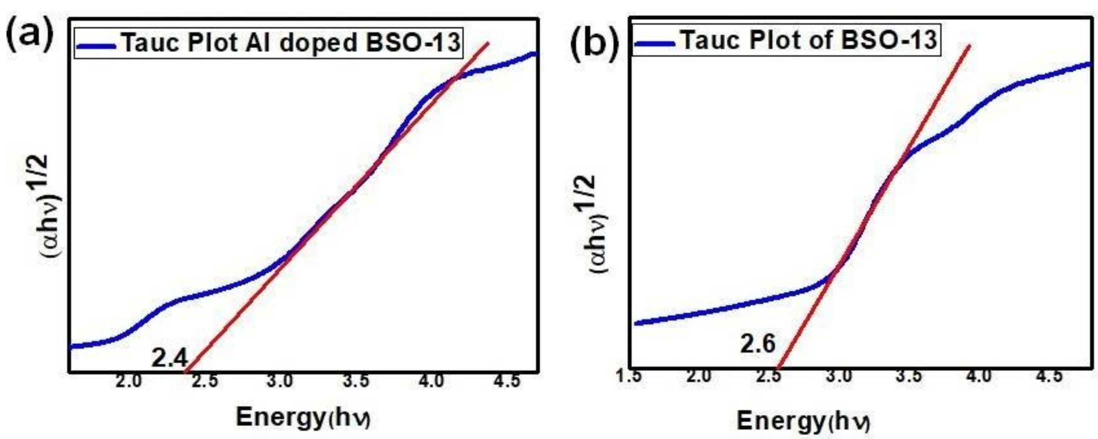

Figure1..5: Tauc plots showing indirect band gap of (a) Al doped BSO-13 (b) BSO-13

Table 2 Band gap description of samples

Samples	Direct (eV)	Indirect (eV)
BSO-13	2.9 ±0.01	2.6 ±0.01
Al doped BSO-13	3.3 ±0.01	2.4 ±0.01

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CONCLUSION

The pH plays the crucial role in deciding the purity of phase and affects the morphology of $BaSnO_3$ synthesized via chemical precipitation route. Al doping of BSO significantly affects the Al doping of BSO significantly affects the structural, Morphological and optical properties. The BSO microrods synthesized were porous and are transparent in the visible range having direct band gap of 3.1 eV.

References :

[1] Sergiu T. Shishiyanu, Teodor S. Shishiyanu, Oleg I. Lupan. Sensors And Actuators B, 107 (2005) 379-386.

[2] F.M. Filho, A.Z. Simoes, A. Ries, I.P. Silva, L. Perazolli, E. Longo, J.A varela, Ceramics International, 30 (2004) 2277-2281.

[3] Tacettin Yildirim, Emre Gur, S. Tuzemen, V. Bilgin, S. Kose, F. Atay, I. akyuz, Physics E, 27 (2005)290-295.

[4] Won Jae Moon, Ji Haeng Yu, Gyeong Man Choi, Sensors And Actuators B, 80 (2001) 21-27.

[5]. Xiangdong Lou, Xiaohua Jia, Jiaqiang Xu, Shuangzhi liu, Qiaohuan gao, Materials Science and Engineering A, 291 (2006) 258-261.

[6] Zirang Li, Xueling, Xiaoxi Zhang, Yitaiqian. Journal of Crystal Growth, 291 (2006)258-261.

[7] J.X. Wang, S.S. Xie, H.J. Yuan, X.Q.Yan, D.F. Liu, Y. Gao, Z.P.Zhou, L. Song, L.F. Liu. Solid State Communications, . 131 (2004) 435-440.

[8] Yong Su, Li-ang Zhu, Liang Xu, Yiqing chen, Haihue Xiao, Qingtaozhou, Yi Feng. Materials Letters, . 61 (2007) 351-354.

[9] Yongsheng Zhang, Ke Yu, Guodong Li, Deyan Peng, Qiuxiang Zhang, Feng Xu, Wei Bai, Shixi Ouyang, Ziqiang Zhu, Materials Letters, 60 (2006) 3109–3112.

[10] P.A. Santos, S. Maruchin, G.F. Menegoto, A.J. Zara, S.A. Pianaro, Materials Letters. 60 (2006) 1554–1557.

[11] M. Parthibavarman V. Hariharan, C. Sekar (2010) J Optoelect Adv Mater

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12:1894-1898

[12] S.M Sedghi, Y. Mortazavi , A. Khodadadi (2010) Sens. Actuators B: Chem. 145:7-12. doi: 10.1016/j.snb.2009.11.002.

[13] Ai Z, Lee S, Huang Y, Wingkei, Ho, Zhang L, (2010) J Hazardous Mater.179:141-150.

[14] M. Jayalakshmi, K. Balasubramanian, *Int. J. Electrochem. Sci*, *3* (2008) 1196-1217.

[15] R. Ramachandran, S. Felix, G. M. Joshi, B. P. Raghupathy, S. K. Jeong, A. N. Grace, *Materials Research Bulletin*, *48* (2008) 3834-3842.