

Role Of pH In the Pure Phase Formation of Barium Stannate Nanostructure Via Chemical Precipitation Route

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Abstract. Mobility of a TCO (transparent conducting oxide) depends on purity of phase, temperature and effective mass of charge carriers. Phase purity and operating temperature are the two controllable parameters to enhance the mobility. In this study firstly we have analyzed the role of pH on pure phase formation of BaSnO₃ by using two different bases NaOH and NH₃. For this, the experiment is performed at various pH values i.e. from pH-8 to pH-11 using NH₃. Secondly at pH -11 the chemical precipitation synthesis was performed by using Base NaOH and chlorides as precursor. It is observed that Pure phase formation of stannate nanostructure i.e. BaSnO₃ through Chemical precipitation is very much controlled by pH of the precursor solution. The pH affects the solubility of a compound and thus further decides the precipitation of desired hydroxides of metals. In this study pure phase formation was analyzed through XRD studies. It is evident through our studies that in chemical synthesis of BaSnO₃, NaOH is better choice of base than NH₃. Also, Barium Chloride is better precursor than Barium carbonate in synthesizing BaSnO₃ nanostructures. XRD study also revealed that while using Barium carbonate, SnO₂ phase appeared as impurity but when Barium chloride is used, no such impurity is observed. SEM studies revealed the micro rod like morphology of Barium stannate. The RT mobility of these Barium stannate microrods comes out to be 49 cm²/V-sec.

1. Introduction

The multifunctionality is demand of today's electronic devices. Barium stannate (BaSnO₃) falls in the category of wide band gap Transparent Conducting oxide (TCO) due to its richness in multifunctional properties. Functional oxides like Perovskites (i.e. having formula ABO₃) have gained much attention in research due to their structures and physical properties. BaSnO₃ is a perovskite which falls in the category of wide band gap semiconductors. At room temperature BaSnO₃ is transparent as well as has high mobility. The la-doped BaSnO₃ has depicted 320 cm²/V-sec but this is for single crystalline. The

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highest mobility of undoped BaSnO_3 has been reported to be $183 \text{ cm}^2/\text{V}\cdot\text{sec}$ which is grown epitaxially [1].

To improve the mobility of these optoelectronic materials i.e. BSO there is necessity of pure phase formation in the material. Precipitation route is one the facile method to synthesize nanomaterials. Pure phase formation is very much dependent on the various kind of synthesis parameters of precipitation route. In case of precipitation route the different parameters are pH of the solution, calcination temperature, time for which the temperature is maintained etc. Optimization of these synthesis parameters are the main deciding factor for the formation of the phase [2].

This article focuses on the role of pH on the formation of pure phase of BSO which further affects the RT mobility of it. For this the sample preparation was done at various pH values and was then characterized. It was observed that pure phase formation is very much dependent on the values of pH pointing towards its significant role. The pH decides the solubility of the species and controls the rate of reaction which further assist in precipitation of desired hydroxide.

2. Synthesis Method

In optimization of pH for pure phase of BaSnO_3 several experiments were performed. Basically, two different methods were followed.

In Method 1- $\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$ was taken in H_2O_2 and was continuously stirred on magnetic stirrer. Dropwise NH_3 was added to set a particular pH of the solution at a particular pH BaCO_3 was added and was heated simultaneously upto 95°C for 1 hr. Then washing the precipitates with Deionised water till the pH reaches 7. Then drying the precipitates in oven for 24 hrs. After drying, calcinating the sample at 1100°C for 5 hrs.

In Method 2- $\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$ was taken in H_2O_2 but instead of NH_3 strong base NaOH was taken and also BaCO_3 was replaced by BaCl_2 . The flow chart is as shown below.

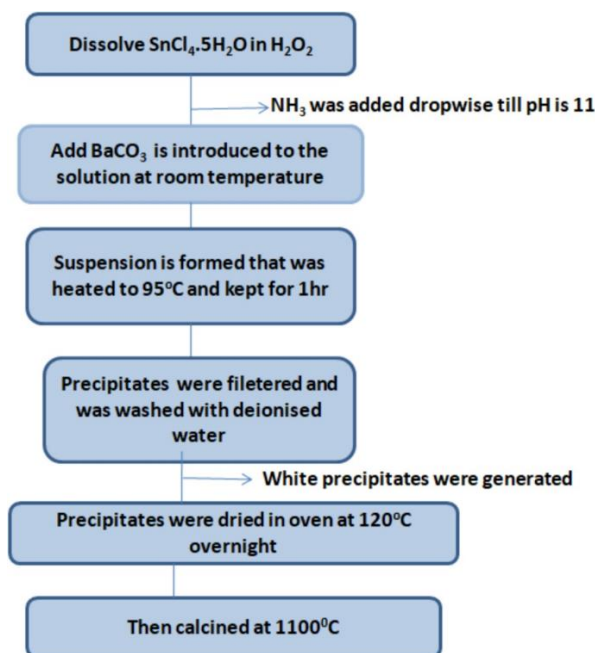


Fig. 1. Schematic Diagram of Sample preparation by Method1

3. Experimental Characterizations

To study the phase formation at different pH values XRD study was performed. The desired Pure phase BaSnO₃ was then characterized by FESEM and electrical characterization was done through Hall-Probe method.

3.1 XRD studies

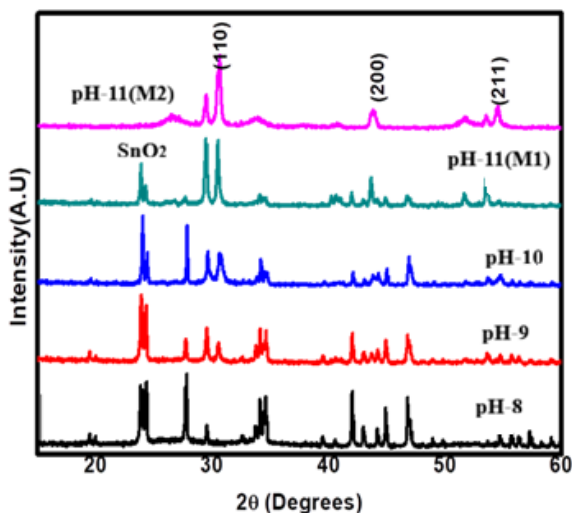


Fig. 2. XRD of different samples prepared at different pH

It is observed from XRD that at low pH SnO₂ phase is more easily formed and appears as impurity but at high pH BaSnO₃ phase dominates. The samples were also prepared at pH-12 with method-1. Fig.3.1.1 depicts that at pH-12 more pure phase of BaSnO₃ was obtained.

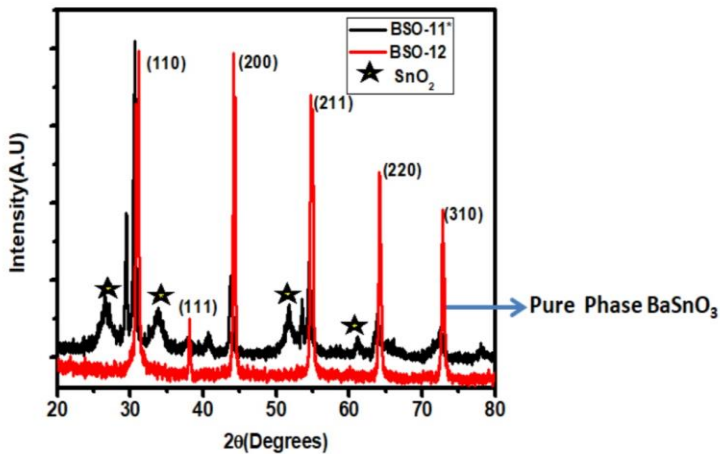


Fig. 3. XRD of different samples prepared at pH-11 and pH-12

3.2 Morphological Studies

FESEM studies revealed that at pH-13 agglomerated nanostructures were formed while at pH-12 rod like morphology was observed. The average length of these micro rods were observed to be 25 μm .

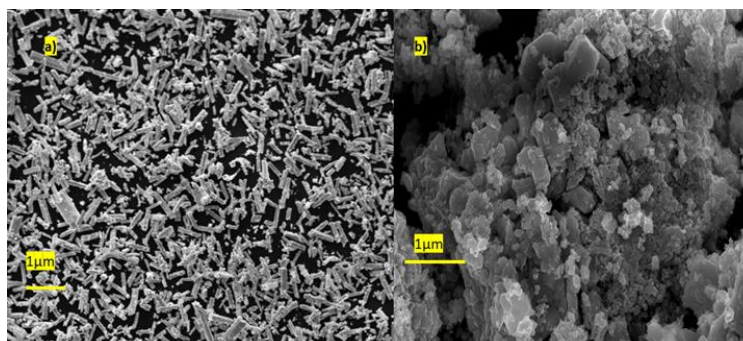


Fig. 4. FESEM Image a) BaSnO_3 at pH-12 b) BaSnO_3 at pH-13

4. Conclusion

In hydrothermal synthesis the optimization of pH is one of the key parameters in phase purity of nanostructures. Also the kind of base used for a desired pH also affects the formation of phase.

1. XRD studies reveals that, phase formation of BaSnO_3 strongly depends on the choice of precursors and pH.
2. BaSnO_3 is a complex ion which forms its hydroxide at high pH. At low pH simple ions are formed.
3. At high pH the morphology changes from micro rods to flakes i.e. agglomeration occurred at high pH.

1-D hollow micro rods can further assist in DSSCs in more dye loading. The electrical mobility of these rods comes out to be $49 \text{ cm}^2/\text{V}\cdot\text{sec}$. The efficiency of DSSC prepared by using these micro rods comes out to be 3.3%.

References

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