Effect of reducing agent on the phase and morphology of Sb_2Te_3 powder using cost-effective solvothermal route

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***Abstract*: Improving the thermoelectric (TE) properties of materials, that are promising candidates for low- temperature thermoelectric applications, is by nanostructuring. These materials are increasingly becoming preferred alternatives over other energy resources. Nanostructured TE materials proved to be efficient ones with high figure of merit (ZT). For p-type Sb_2Te_3 compound, a simple solvothermal route has been employed using water as solvent and effect of variable quantity of reducing agent is studied through phase characterization. The final products with different concentrations of reducing agent are further investigated through microstructural studies.**

***Keywords*: Thermoelectric, figure of merit, solvothermal, nanostructuring, reducing agent**

Introduction

Thermoelectric devices have the ability to directly convert heat energy into electricity. These materials possess various advantages such as compactness along with low maintenance and have low levels of noise and vibrations. The development of high-performance thermoelectric materials has a lot of scope as an alternative for energy resources. The efficiency or performance of a thermoelectric material is measured in terms of a dimensionless factor known as the figure of merit, ZT, given by:

$ZT = (S^2/\rho\kappa)T$ where, S is the Seebeck coefficient, ρ is the electrical resistivity, κ is the thermal conductivity, and T is the absolute temperature [1-3]. Over the past decades, several attempts have been made to improve efficiency of thermoelectric materials through nanostructuring [4]. A promising candidate for low temperature applications, apart from bismuth telluride, is antimony telluride (Sb_2Te_3) [5-6]. For the synthesis of nanostructured Sb_2Te_3, various methods are available in the literature e.g. vapor-liquid-solid growth method [7-8], chemical vapor deposition [9-10], mechanical alloying [11], microwave-assisted wet chemical [12], electrochemical deposition [13-15], hydro/solvothermal [16-19] routes. Out of all these methods, solvothermal route has proved to be comparatively easy and effective in synthesizing nanostructured Sb_2Te_3. There are various processing parameters involved in solvothermal route. These processing parameters are synthesis time, synthesis temperature, amount and nature of reducing agent as well as solvent, pH of reaction media etc. Appropriate tuning of these parameters is required to obtain desired phase and morphology. In the present case, solvothermal route is being employed for synthesis of nanostructured Sb_2Te_3 and the effect of reducing agent of varying quantities on the final product, is given while keeping all other parameters fixed.

Experimental

For the synthesis, $SbCl_3$ and Te powder are mixed in stoichiometric ratio of 2:3. This mixture is put in a Teflon beaker filled with 300 ml water, taken as solvent. After that different quantities of sodium borohydride ($NaBH_4$), is dissolved in whole aqueous mixture. Stirring is done continuously for proper mixing. Then sodium hydroxide (NaOH) is added slowly to the mixture while stirring to get required pH. Once it is achieved, beaker is placed inside the autoclave. Then the autoclave is sealed immediately, heated up to 563 K and maintained for 20 h. After that it is allowed to cool to room temperature. Once the reaction is over, the obtained precipitate is washed several times with distilled water, and then dried at 373 K in inert atmosphere. The final powder is then characterized using X-Ray diffractometer for phase determination and scanning electron microscopy for structural investigations. The phase determination was done by collecting XRD patterns using CuKα radiation in Rigaku MiniFlexII X-Ray diffractometer. Relevant patterns were recorded over a 2θ range of 10-80°. Microstructural characterization of the as-synthesized powder sample was carried out by recording images at different magnifications under Scanning Electron Microscope (SEM) for all samples.

Results and Discussion

The synthesis of final powder is done by taking 1.8g, 2.1g and 2.3g of reducing agent ($NaBH_4$). The XRD plots of as-synthesized powders are given in Fig.1.

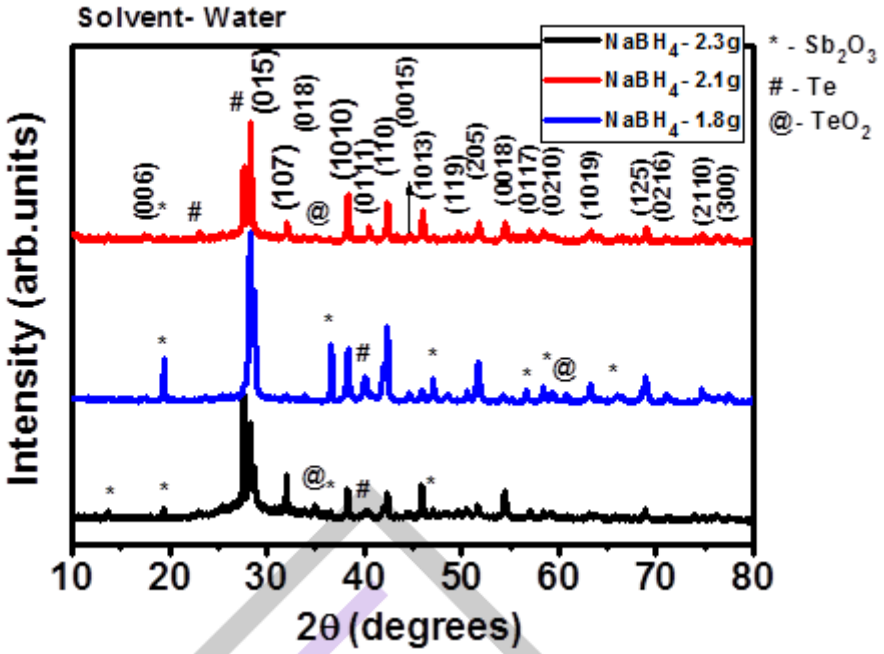

Fig.1: XRD plots of as-synthesized powder prepared using different quantities of $NaBH_4$ and water as solvent

When 1.8 g of $NaBH_4$ is used, mixed phases are observed along with peaks corresponding primarily to Sb_2Te_3 hexagonal phase. The peaks corresponding to Sb_2O_3, elemental Te and TeO_2 are also observed. As quantity of $NaBH_4$ is increased from 1.8 g to 2.1 g, the intensity of oxide peaks is decreased while that of Sb_2Te_3 is increased. Only few peaks are observed corresponding to Te, TeO_2 and Sb_2O_3. Phase purity is much enhanced for 2.1 g of $NaBH_4$. Further increasing $NaBH_4$ to 2.3 g reduced the intensity of hexagonal Sb_2Te_3 phase while some more secondary peaks are found. Thus, the quantity of $NaBH_4$ is optimized to 2.1 g to get predominantly hexagonal Sb_2Te_3 phase as compared to other quantities. Therefore, the appropriate amount of reducing agent is crucial in obtaining the require phase. Further purity of phase can be achieved by fine tuning the other synthesis parameters. This powder sample is taken further for structural characterizations. To study the morphology of synthesized powder, SEM images are taken as shown in Figs. 2(a)-(c). The image (Fig. 2(a)) depicts overall morphology of sample synthesized with 1.8g of $NaBH_4$. It is composed of numerous plate-like structures piled one over the other. The edge to edge distance of plate is tens of micrometers. The nanoparticles of diameter 150-250nm are observed in sample prepared using 2.1g of $NaBH_4$ as shown in Fig.2(b). Further the sample with 2.3g of reducing agent resulted in wire like morphology with diameter around 200nm (Fig.2(c)).

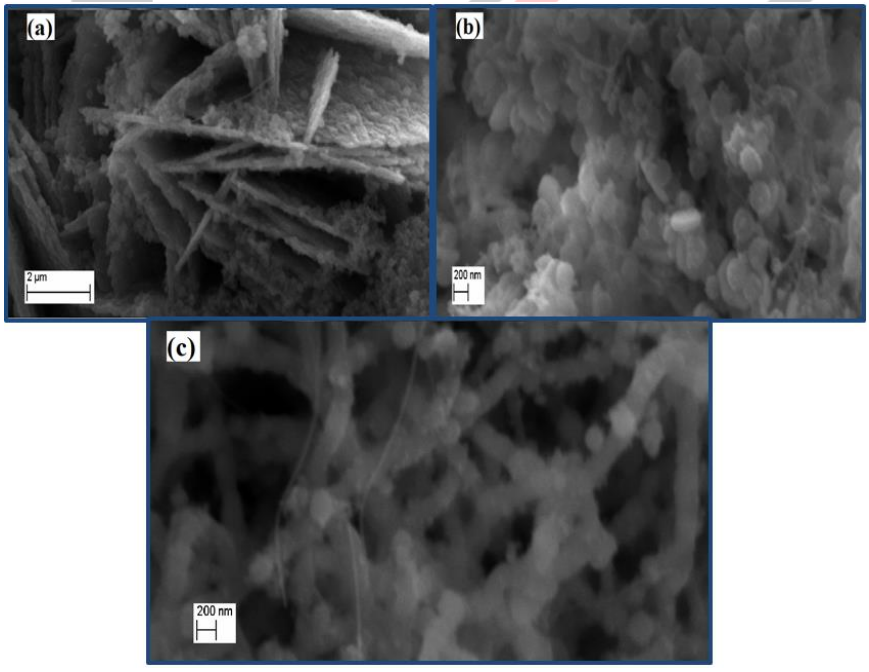

Fig.2: SEM images of powder samples with (a) 1.8g; (b) 2.1g; (c) 2.3g of reducing agent $NaBH_4$

Conclusion

The solvothermal route has been successful in synthesizing nanostructured Sb_2Te_3. The role of varying concentrations of reducing agent ($NaBH_4$) on the end product is studied. Water has been used as solvent. It is evident from the phase characterization that powder prepared by water with 2.1 g of $NaBH_4$ results in better phase purity of hexagonal Sb_2Te_3 as compared to other concentrations. The other synthesis parameters are required to be optimized to reduce the oxide peaks in 2.1g sample powder. The structural investigations show that using different amounts of reducing agent leads to distinct morphologies in powder samples. These nanostructured powders could prove to be efficient in reducing the thermal conductivity of material through phonon scattering, thereby improving its figure of merit and overall TE performance.

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