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Original Research Paper

Synthesis, characterization, structural and optical absorption behavior of $Sn_xSb_yS_z$ powders

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ABSTRACT

The $SnSb_4S_7$, $SnSb_2S_4$, $Sn_4Sb_6S_{13}$, $Sn_2Sb_2S_5$ and $Sn_3Sb_2S_6$ materials were synthesized by the Bridgmann technique. The five pellets were characterized by X-ray diffraction, Raman spectroscopy and diffuse reflectance spectroscopy. The changes observed in Raman spectra were due to the influence of the composition. Kubelka–Munk model and inversion method were used to determine the band gap energies of the samples. The absorption edges were determined using the first derivative of diffuse reflectance spectra. The band gap varies between 1.17 and 1.06 eV.

1. Introduction

Sulfosalt materials received considerable attention for their technological uses. They include photovoltaic, thermoelectric energy conversion, phase change memory, X-ray detectors [1], optical sensors, optical fibers, waveguides for telecommunication wavelengths and ultrafast optical signal processing [2–7]. Several methods have already been used to elaborate sulfosalt materials, thermal evaporation [3,8–11], pulsed laser deposition techniques [3,12] and RF magnetron sputtering [11,13,14]. Sn–Sb–S is a new ternary sulfosalt which attracts the attention of the researchers due to their potential use in solar cells [15]. $SnSb_2S_4$ has a band gap of 1.65 eV and an absorption coefficient of about 10^5 cm^{-1} in the visible region [16]. $SnSb_4S_7$ films have relatively high absorption coefficients, higher than 10^5 cm^{-1} in the visible and in the near-IR spectral region [17]. Comparing with Ge–As–Se/Te, the ternary Sn–Sb–S does not contain high toxic elements and this is one interesting opportunity. Sn, Sb and S elements are widely found in nature and cheaper than photovoltaic materials containing indium. To the best of our knowledge, all the published studies on the ternary Sn–Sb–S used the thermal evaporation technique for elaboration of thin films. Most of these researches have focused mainly on Sn–Sb–S thin films properties. In our knowledge Raman characterization of the $SnSb_2S_4$, $Sn_4Sb_6S_{13}$, $Sn_2Sb_2S_5$ and $Sn_3Sb_2S_6$ materials had never been published. The present study is the first attempt to investigate the structural and optical properties of the $Sn_xSb_yS_z$ powders. The optical properties of the five compositions were studied from measurement of diffuse reflectance which is a standard technique for the determination of the absorption properties and the band gap of materials [18–20].

2. Experimental details

The $SnSb_4S_7$, $SnSb_2S_4$, $Sn_4Sb_6S_{13}$, $Sn_2Sb_2S_5$, $Sn_3Sb_2S_6$ crystals have been synthesized by the horizontal Bridgman method of high-purity (99.999%) elemental tin, antimony and sulfur. Stoichiometric amounts of the elements Sn, Sb and S, corresponding to the compositions of the ternary compounds, were placed in a quartz ampoule. After pumping down to 10^{-5} Torr, the ampoules were sealed off and were transferred to a programmable furnace (Nabertherm–Allemagne). The temperature inside the furnace was increased from room temperature to 250 °C with rate of 75 °C per hour, than from 250 °C to 600 °C slowly (10 °C per hour). A complete homogenization could be obtained by keeping the melt at 600 °C for about 51 h. Thermal expansion of the melt on solidification was avoided by cooling the melt at a rate of 10 °C per hour until room temperature. Fig. 1 shows the scheme of these steps. The resulting ingots are opaque with gray color grades and contain

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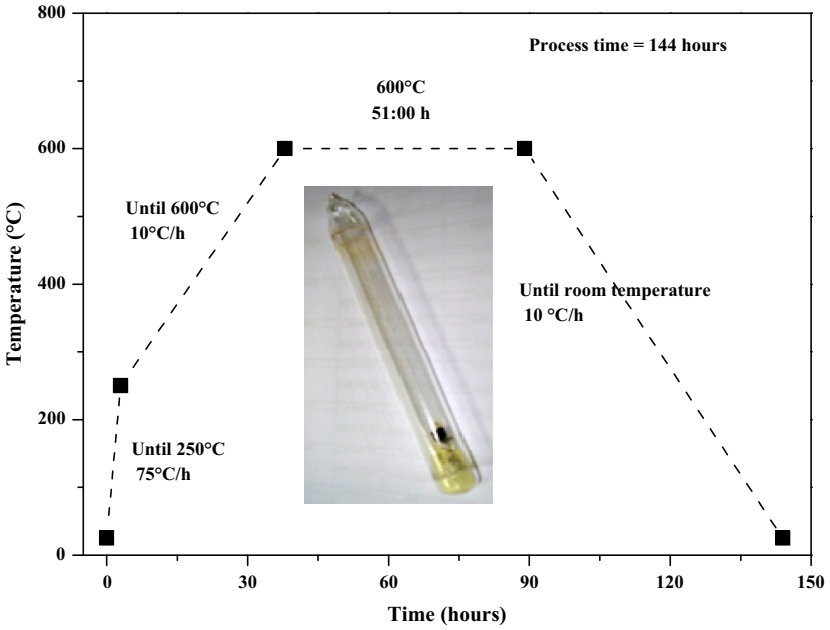

Fig. 1. The thermal profile used for the synthesis of the $Sn_xSb_yS_z$ ingots by the horizontal Bridgman method.

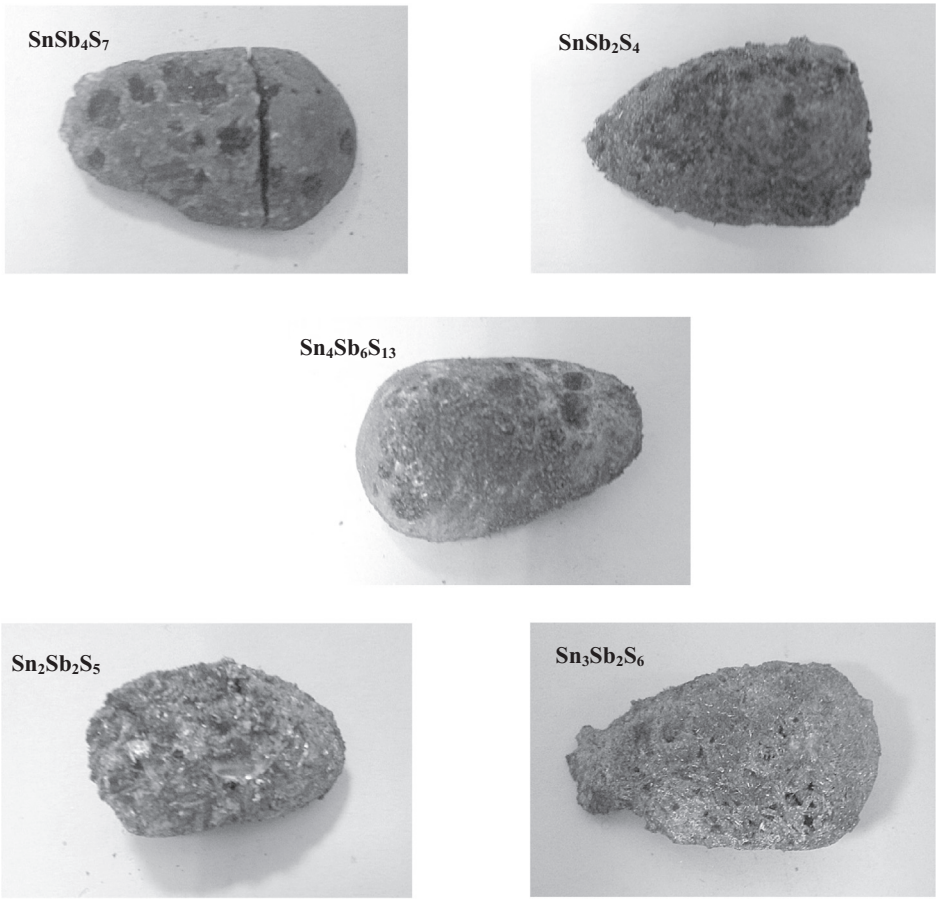

Fig. 2. Ingots of $Sn_xSb_yS_z$ materials.

pores (Fig. 2). The ingots were crushed during 10 min and the obtained powders were compacted to be easily characterized.

The powders have been systematically characterized after preparation. The phases of the $Sn_xSb_yS_z$ powders and crystal orientation were investigated by X-ray diffraction (XRD) using monochromatic CuK_α ($\lambda = 1.54056$ Å) radiation in 2θ range of 15–60°. The operation voltage and current used are, respectively, 40 kV and 30 mA. Raman scattering measurements was performed