Preparation and Microstructure Characterization of Pb$_{1-x}$Sn$_x$Te Bulk Alloys

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Abstract: In this paper we describe the preparation and characterization of the polycrystalline Pb$_{1-x}$Sn$_x$Te bulk alloys, which is grown by synthesis via a reaction of Pb, Sn, Te elements in sealed quartz ampoules. The elemental composition of the obtained samples was determined from energy dispersive X-ray (EDX). The structural characterization was investigated by X-ray diffraction (XRD). The powder of X-ray diffraction of the alloys showed a single phase only, which was found to be cubic. The lattice parameter $a$ is calculated from the peak positions of X-ray diffraction data. The dependence of the lattice constant on composition $X$ exhibits a linear behavior and may be described by the Vegard’s law.

Keywords: Pb$_{1-x}$Sn$_x$Te, X-ray diffraction, Energy dispersive X-ray, Microstructure, Lattice parameter.
1. Introduction

The lead-tin telluride alloy system has been investigated for many decades and applied mainly in the fabrication of infrared photodetectors and diode lasers [1-4]. Also the quality of single crystals has significant importance in the fabrication of detectors in the range of 8 to 14 μm region [5-9]. Melt growth of lead tin telluride presents a challenge because this process is thermosolutally unstable in gravitational fields [4]. The technological importance of PbSnTe based on its band gap versus composition diagram which has a zero energy crossing at approximately 40% SnTe.

A convenient way of determining the composition of an alloy from a complete solid solubility is by measuring its lattice parameter. The tin compositions can be accurately measured from the lattice parameters α calculated from the peak positions using Bragg’s Law [5]. It’s necessary to state that, this bulk material will be use as raw material to make thin films in our research, because of this, the investigation of structure of these compositions is important for our work. The old investigations have varied results in structural parameters of this material and that’s the reason why we needed to make new experiment and extract new results. This work represents our result on preparation and investigation of Pb_{1-x}Sn_{x}Te bulk compounds.

2. Experimental

Polycrystalline Pb_{1-x}Sn_{x}Te (0.0 < X <1.0) alloys were synthesized by the fusion method. In this method, the reaction between the sulfur vapors and molten metals were allowed to take place gradually in evacuated silica tubes. High purity (99.999%) mixtures of constituent elements (Pb, Sn, and Te) in stoichiometric proportions (with an accuracy of 5×10^{-4}g) were sealed into evacuated silica tubes at the pressure of 10-3 Torr. The evacuated tube was then placed into an electric furnace and kept at 450°C for 7 days and after that at 700°C for 10 days. In order to avoid explosions due to the telluride vapor pressure, the tube was heated slowly. The tube was gradually cooled with a cooling rate of about 20°C/h to room temperature in order to obtain polycrystalline Pb_{1-x}Sn_{x}Te compounds.

The crystal structure of the Pb_{1-x}Sn_{x}Te alloys was observed by X-ray diffraction (XRD) using a Siemens D-5000 diffractometer with CuKα (λ = 1.5418 Å) radiation. The observed phases were determined by comparing the d-spacing with the Joint Committee on Powder Diffraction Standard (JCPDS) data files. The elemental composition of the obtained materials was determined from energy dispersive X-ray (EDX) data, using Scanning electron microscope Stereoscan-360 with EDX spectrometer AN 10000 with an accuracy of about 2%.

3. Results and discussion

The energy dispersive analysis of all alloys shows that the obtained Pb_{1-x}Sn_{x}Te compounds are homogeneous and the compositions are reproducible. This clearly shows that composition control can be easily achieved using the fusion method. the EDX spectra for the obtained Pb_{1-x}Sn_{x}Te alloys with different composition, illustrating the respective peaks of lead, tin and tellurium Fig.(1).

X-ray diffraction measurements were performed to identify the crystal structure and phases in alloys. Figure 2 shows the XRD spectra patterns for Pb_{1-x}Sn_{x}Te Te alloys. The XRD spectra showed that bulk materials have polycrystalline nature with the cubic NaCl-crystal structure and indicate the absence of other phases [8]. All XRD peaks are shifted to lower angels with the increase of Pb content in the Pb_{1-x}Sn_{x}Te compounds. The JCPDS data files for SnTe and PbTe are card number of #
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46-1210 and # 78-1905, respectively. The Pb$_x$Sn$_{1-x}$Te alloys exhibited a (200) peak representing the preferable orientation. The spectrum also showed other peaks in addition to the (200) peak. The most intensive additional peaks corresponds to (220), (222), (400), (420), (422) and (440) orientations.

The cell parameters were evaluated using the standard equation for a cubic crystal structure. The evaluated interplanar spacing ($d$-values) and the lattice parameter of Pb$_{1-x}$Sn$_x$Te alloys are in agreement with the standard JCPDS data. It was found that the variation of lattice parameters versus composition ($X$) is virtually linear and followed to the Vegard’s law behavior. These relationships calculated mathematically by least-squares analysis are presented in figure 3.

Just Short stated that the lattice parameters of Pb$_{1-x}$Sn$_x$Te system has a positive deviation from Vegard’s law [1]. Bis et al. have showed that in small deviations from stoichiometry, Vegard’s law is satisfied for PbSnTe alloys [2].

4. Conclusion

Pb$_{1-x}$Sn$_x$Te alloy system prepared by the fusion method. The XRD spectra showed the polycrystalline nature with the cubic NaCl crystal structure and indicated the absence of other phases. It was established that by increasing the content of Pb in the near stoichiometry Pb$_{1-x}$Sn$_x$Te compositions, the lattice parameter, in accordance to Vegard’s law, is significantly linearly increased. Evidence exists that Vegard's law holds in general for this alloy system are confirmed our results.

5. References
Fig. (1): Energy dispersive X-ray (EDX) patterns of Pb$_{1-x}$Sn$_x$Te bulk alloys.
Fig. (2): X-ray diffraction patterns of Pb$_{1-x}$Sn$_x$Te bulk alloys and the JCPDS data for PbTe and SnTe.
Fig. (3): Lattice parameters of Pb$_{1-x}$Sn$_x$Te alloy system.