ФИЗИКА

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ПОЛУЧЕНИЕ И ИССЛЕДОВАНИЕ МИКРОСТРУКТУРНЫХ СВОЙСТВ ОБЪЕМНЫХ СПЛАВОВ $Pb_XSn_{1-X}Te$

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PREPARATION AND INVESTGATION OF MICROSTRUCTURAL PROPERTIES OF $Pb_XSn_{1-X}Te$ BULK ALLOYS

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Цель работы – получение и характеристика поликристаллических объемных сплавов $Pb_XSn_{1-X}Te$, выращенных методом синтеза Pb, Sn и Te элементов в откаченных кварцевых ампулах. Элементный состав полученных образцов был определен методом энерго-диссперсионной рентгенографии (ЭДР). Структурная характеристика была исследована рентгеновской дифракцией (РД). Порошковая рентгеновская дифракция сплавов показала единственную фазу – кубическую. Параметр решетки *a*, рассчитан на основе рентгеновских данных. Зависимость постоянной решетки от состава X, показывает линейное поведение и может быть описана законом Вегарда.

Ключевые слова*: теллурид свинца-олова, рентгеновская дифракция, энерго-диссперсионная рентгенография, микроструктура, параметр решетки.*

The aim of this work is the preparation and characterization of the polycrystalline $Pb_XSn_{1-X}Te$ bulk alloys, which isgrown 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: *lead tin telluride, X-ray diffraction, energy dispersive X-ray, microstructure, lattice parameter.*

Introduction

The lead-tin telluride alloys ($Pb_XSn_{1-X}Te$) system has been investigated for many decades and applied mainly in the fabrication of infrared photodetectors and diode lasers [1]–[3]. The quality of single crystals is of great importance in the fabrication of detectors in the range of 8 to 14 μm region [4]–[7]. Melt growth of lead tin telluride presents a challenge because this process is thermosolutally unstable in gravitational fields [2]. The technological importance of $Pb_XSn_{1-X}Te$ is 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 elemental compositions of $Pb_XSn_{1-X}Te$ can be accurately measured from the lattice parameter (α°) calculated from the peak positions by using Vegard' law [3]. These bulk alloys are used for preparation of thin films, because of this, the investigation of structural properties is important. Early investigations showed varied results in structural parameters of this system depending on technology of preparation. This work represents our result on preparation and investigation of $Pb_XSn_{1-X}Te$ bulk alloys.

1 Experimental

Polycrystalline $Pb_XSn_{1-X}Te$ ($0.0 < X < 1.0$) 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 ampoules. 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 ampoules were then placed into an electric furnace and kept at 450°C for 7 days and after that at 700°C for 10 days. The ampoules were gradually cooled with a cooling rate of about 20°C/h to room temperature in order to obtain polycrystalline $Pb_XSn_{1-X}Te$ bulk material.

The crystal structure of the $Pb_XSn_{1-X}Te$ alloys was observed by X-ray diffraction (XRD) using a Siemens D-5000 diffractometer with CuKα ($\lambda = 1.5418$ 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%.

2 Results and Discussion

Figure 2.1 shows the EDX spectra for the obtained $Pb_XSn_{1-X}Te$ alloys with different composition, illustrating the respective peaks of lead, tin and tellurium. For each element there are some peaks that can be distinguished, these are defined as K_α, K_β, and L –series (L_α, L_β и L_γ lines) alpha (α) and beta (β). These peaks originate from the relaxation of excited electrons [8]. In the spectra presented in figure 2.1, all elements show the K_α, K_β peaks which indicate both the final place of the electron and also the type of transition that has taken place. For example, K_α indicates that the electron has jumped to a K shell from the nearest outer L shell. Since the probability of transition from the nearest shell is greater the peak intensities for K_α transitions are larger than for K_β peaks. The energy dispersive analysis shows that the obtained alloys are homogeneous and the compositions are reproducible. This clearly shows that composition control can be easily achieved using the fusion method.

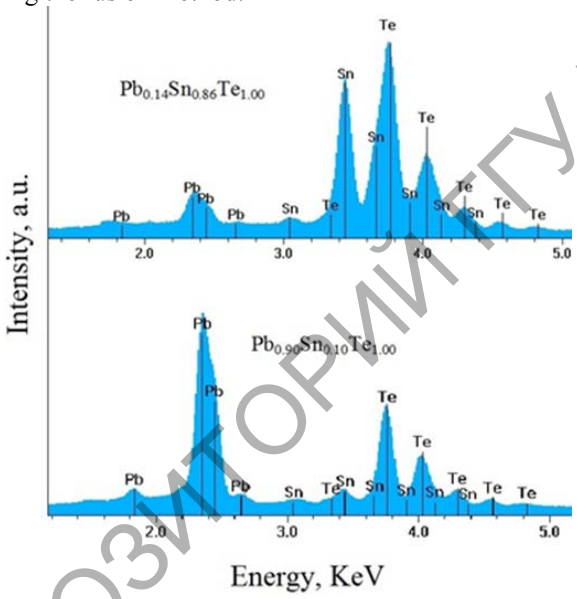

Figure 2.1 – Energy dispersive X-ray (EDX) patterns of $Pb_XSn_{1-X}Te$ bulk alloys

Table 2.1 shows the composition and lattice parameter of $Pb_XSn_{1-X}Te$ samples. Also the atomic percent of elements has been shown in the table. As shown in the table, by increasing the mole fraction of Pb in the composition, the linear increasing is observed in the lattice parameters of our samples.

X-ray diffraction measurements were performed to identify the crystal structure and phases in alloys. Figure 2.2 shows the XRD spectra patterns for $Pb_XSn_{1-X}Te$ alloys. Many methods are used for accurate determination of peak positions.

We used Gaussian method in software Origin. For achieving the lattice parameter to within 1.10^{-5} nm, we must calculate the peak positions to within 0.02°. Naturally there are different types of systematic errors associated with different x-ray instruments. We extrapolated peak positions to high 2θ using a function that minimizes the influence of systematic errors. There are different types of extrapolation functions for different types of systematic error in the lattice parameter [9]–[11]. We used the Nelson- Riley function to achieve accurate data. The Nelson-Riley Function (equation 2.1) is applied very well for Diffractometers, because the first term ($\cos^2\theta / \sin\theta$) corrects for sample displacement, typically the main error source. The second term ($\cos^2\theta / \theta$) may be correct for vertical divergence of the incident beam. Decreasing the window of the detector slit reduces the error due to vertical divergence [12]:

$$\frac{\Delta d}{d} = k\left(\frac{\cos^2\theta}{\sin\theta} + \frac{\cos^2\theta}{\theta}\right), \qquad (2.1)$$

where k is a constant.

The XRD spectra showed that bulk materials have polycrystalline nature with the cubic NaCl crystal structure and indicates the absence of other phases [6], [13]. All XRD peaks are shifted to lower angels with the increase of Pb content in the $Pb_XSn_{1-X}Te$ compounds. The JCPDS data files for SnTe and PbTe are card number of # 46-1210 and # 78-1905, respectively. The $Pb_XSn_{1-X}Te$ 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.

Table 2.1 – Composition and lattice parameter of $Pb_XSn_{1-X}Te$ alloys system

X, mol. fraction	Composition	Atomic %			Lattice parameter, Å
		Pb	Sn	Te	
0.1	$Pb_{0.10}Sn_{0.90}Te_{1.00}$	4.92	44.93	50.15	6.327
0.14	$Pb_{0.14}Sn_{0.86}Te_{1.00}$	6.80	42.78	50.42	6.331
0.35	$Pb_{0.35}Sn_{0.65}Te_{1.00}$	17.62	32.45	49.93	6.342
0.53	$Pb_{0.53}Sn_{0.47}Te_{1.00}$	26.83	23.57	50.60	6.383
0.85	$Pb_{0.85}Sn_{0.15}Te_{1.00}$	42.65	7.20	50.15	6.419
0.90	$Pb_{0.90}Sn_{0.10}Te_{1.00}$	44.86	4.97	50.17	6.436

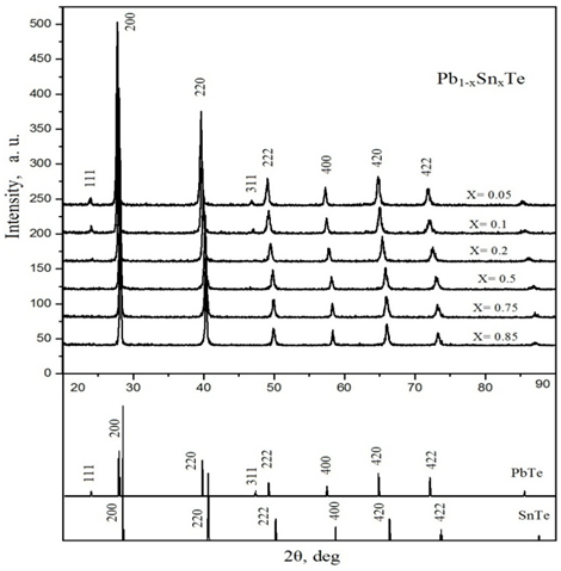

Figure 2.2 – X-ray diffraction patterns of $Pb_XSn_{1-X}Te$ bulk alloys and the JCPDS data for PbTe and SnTe

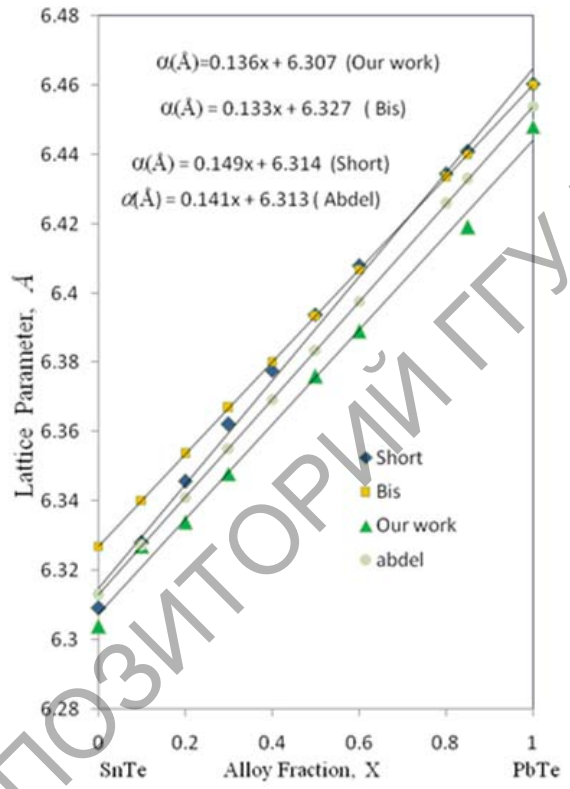

Figure 2.3 – Lattice parameter of $Pb_XSn_{1-X}Te$ alloys system

Just Short stated that the lattice parameter of $Pb_XSn_{1-X}Te$ system has a positive deviation from Vegard's law [14]. Bis et al. have showed that in small deviations from stoichiometry, Vegard's law is satisfied for PbSnTe alloys [15]. Also Abdel Rafea et al. showed the linearity of the lattice parameter of this system [13].

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 $Pb_XSn_{1-X}Te$ alloys are in agreement with the standard JCPDS data. It was found that the variation of lattice parameter versus composition (X) is virtually linear and followed to the Vegard's law behavior. This relationship was calculated mathematically by least-squares analysis. The general expression for these relations is

$$a\ (\text{alloy}) = 0.136X + a\ (SnTe), \qquad (2.2)$$

Where *a* (alloy) is the lattice constant of the alloy in *Å*; *X*, is the alloy fraction; and *a* (SnTe), is the lattice constant of SnTe.

Our investigations showed that the lattice parameter of the $Pb_{1-X}Sn_XTe$ bulk alloys prepared by the fusion method obeys Vegard's law.

Conclusions

We have successfully synthesized polycrystalline $Pb_XSn_{1-X}Te$ ($0.0 < X < 1.0$) alloys by the fusion method. The energy dispersive analysis showed that the obtained alloys are homogeneous and the compositions are reproducible. 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 $Pb_XSn_{1-X}Te$ compositions, the lattice parameter, in accordance to Vegard's law, is significantly linearly increased.

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