

# Artikel 6\_3

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**THE EFFECT OF TELLURIUM ATOMIC FRACTION ON THE CRYSTAL  
STRUCTURE AND CHEMICAL COMPOSITION OF Pb (Se<sub>1-x</sub>, Te<sub>x</sub>)  
SEMICONDUCTOR MATERIALS PREPARED USING BRIDGMAN  
TECHNIQUE**

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**Abstrak**

This study aimed to determine the effect of atomic fraction  $x$  of tellurium (Te) on the lattice parameters and the crystal structure of the Pb (Se, Te) compound prepared using Bridgman technique. Further characterization of the materials used X-Ray Diffraction to determine the lattice parameters and the crystal structure. The chemical composition was determined using Energy Dispersive Spectroscopy (EDS), and the surface morphology shown by Scanning Electron Microscope (SEM). The results showed that all compounds crystallized in a cubic crystal structure and the lattice parameters of the crystal were a function of the fraction  $x$  of Tellurium atoms. EDS results showed that all samples were non stoichiometric but approached ideal composition. All compounds had identical surface because it had a cubic structure with homogeneous materials.

Keywords : Bridgman technique, lead , selenium and Tellurium

**Introduction**

Semiconductor materials have electrical conductivity between insulators and conductors. They are widely used in electronic circuits since their electrical properties can be easily changed by adding some impurities. These impurities are called dopant and created by adding different material using several technique like diffusion, ionic implantation, melting, etc. Each semiconductor material has certain physical, optical and magnetic characteristics, so its application should consider these characters. For an example, in ultraviolet sensor application which need high sensitivity, materials with wide energy gap are chosen such as gallium nitride with 3,4 eV energy gap [1].

Research in semiconductors, especially in silicon (Si) and germanium (Ge) application, has already been well established. Research on other semiconductor materials is also rapidly underway. It is aimed at producing variation in semiconductor materials for various application. One of the research is on the lead chalcogenides group IV-VI compounds such as PbS, PbTe, PbSe, PbSeTe which their material properties and also their application are heavily studied [2, 3].

Research on ZnO, GaN, AlGaN, AlGaSb, PbS, PbTe and PbSe are widely conducted. The last three have similarity, that is n-type semiconductor produced from combining group IV (Pb) with group VI (Se, S, Te). PbS, PbTe, and PbSe each has energy gap of 0.41 eV, 0.32 eV and 0.29 respectively [4, 5] which is well suited to control application: non-contact temperature

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measurement, humidity control, spectrophotometry, medical imaging, infrared detector, laser semiconductor and thermoelectric [6].

This research focus on the study of massive Pb(Se, Te) system material prepared using Bridgman technique. The concept of preparation is temperature adjustment by considering the melting point of the basic materials. In this process, several samples with different heating patterns were used. It was aimed to determine the quality and lattice parameter of crystal, and to determine chemical composition and the best surface morphology structure of the samples. The choice of Pb(Se, Te) in this research is meant to be used as the basic materials in building optoelectronic devices for infrared detectors, laser semiconductors and thermoelectric.

### Methods

Massive semiconductor preparation process, which was crystal growth process, was supposed to give perfect crystals with high purity which will improve the device quality prepared. In preparing massive alloys, there are two crystal growth methods: Czochalski and Bridgman method. Crystal growth process using Czochalski method is quite complex and need expensive apparatus in university's laboratory scale. On the other hand, the Bridgman method, first introduced by Lowman using closed quartz capsul, is cheap and the apparatus is easier to be made. The superiority of Bridgman technique to Czochalski technique is that this technique can produce massive semiconductor materials with affordable cost for the laboratory. In addition, its temperature can be controlled easily, the materials freezing rate can be adjusted, the rate of temperature decrease from liquid phase can be accurately controlled, and mechanical stress in the material can be reduced to avoid the crack in the preparation capsul in order to reduce the explosion hazard produced by certain substances at their critical temperature. This method can be conducted in two ways: the horizontal Bridgman method and the vertical Bridgman method. Their difference is in the position of the capsul inside the furnace in heating process.

Variables in this preparation stage were the vacuum pressure (in Torr) and time (in second) as the controlled variables, Pb(Se, Te) materials preparation temperature with maximum value of 500 °C and the heating pattern as the independent variables, and the crystal structure, lattice parameter, chemical composition, and surface morphology of massive Pb(Se, Te) alloy as the dependent variables.

The research consisted of several steps:

### Materials Preparation

This process consisted of tube washing and material weighing. Material weighing was done for Pb, Se and Te using molar ratio. It started with Te mass, then for Pb ( $\text{Se}_{1-x}\text{Te}_x$ ) compound the mass for lead (Pb) and selenium (Se) can be calculated using

$$\text{Mass of Pb} = \left( \frac{\text{mass of Te}}{\text{atomic weight of Te}} \cdot \text{atomic weight of Pb} \right) \cdot \frac{1}{x} \text{ gram}$$

$$\text{Mass of Se} = \left( \frac{\text{mass of Te}}{\text{atomic weight of Te}} \cdot \text{atomic weight of Se} \right) \cdot \frac{1-x}{x} \text{ gram}$$

Materials in accordance with the molar ratio were put into a pyrex tube with inner and outer diameter of 16 mm and 12 mm respectively. Then the tube was vacuumed to  $10^{-5}$  mbar and sealed to give a capsul containing Pb, Se and/or Te. Next, it put inside the furnace which can reach temperature of 1100 °C. Heating was done at the maximum temperature above the melting

point of all substance, which is 500 °C with temperature pattern was chosen according to the available time in the laboratory. One of the heating pattern in the material preparation was that the material was heated until it reached 250 °C and kept for 4 (four) hours. Next, it heated until 500 °C and kept for 5 (five) hours and then the furnace was switched off so it would underwent temperature decrease until it reach room temperature in 6 (six) hours.

#### **Materials characterization**

To determine the crystal structure of the alloys using X-Ray Diffraction (XRD), Shimadzu XRD-600 was used. The data obtained in the XRD characterization were in the form of diffractogram. It showed the intensity as a function of diffraction angles ( $2\theta$ ). How the XRD works was started by putting the samples on the stationary holder, then X-ray was struck to the samples at certain wavelength so that diffraction waves were produced for certain crystal planes. The diffracted beam resulting in diffractogram followed the Bragg's diffraction law  $2d \sin q = n \lambda$ , with  $n$  is integers (1, 2, 3, ...),  $d$  is the distance between crystal planes,  $q$  is the diffraction angle and  $\lambda$  is the x-ray wavelength. In this research, the source of the XRD was Cu with wavelength of 154060 Å. Parameters used in the XRD were operational voltage of 40 kV, current of 30 mA, and angles  $2\theta$  between 0° to 90°.

The diffractogram was then compared to the JCPDS (*Join Committee on Powder Diffraction Standard*) data, so the  $hkl$  planes from the samples were obtained. The value of lattice constants ( $a$ ,  $b$ ,  $c$ ) were determined using analytical method [7] by looking for the same values in the  $\sin^2 q$  value. This can be done since for the cubic system, the distance between two crystal planes ( $hkl$ ) can be stated by  $1/d^2 = (h^2+k^2+l^2)/a^2$ . If the Bragg's law is substituted into the equation, it produces  $\sin^2 q = A (h^2+k^2+l^2)/a^2$  with  $A = \lambda^2/4a^2$ . So  $a = 1/2\sqrt{A}$  which is the same value in  $\sin^2 q$  for various diffraction angles  $q$  [7]. Next, the molar ratio, which is the real chemical composition, was determined directly from the analysis of the Energy Dispersive Analysis of X-ray (EDAX) results, and the surface morphology of the sample was determined from the Scanning Electron Microscope (SEM) test. Both analysis were done using integrated device the Jeol JSM-636 OLA.

#### **Results and Discussion**

XRD characterization was used to determine the characteristic of the crystal, that is, the structure and the lattice constant  $a$ ,  $b$ ,  $c$  of the massive or ingot produced in the Pb(Se,Te) heating using Bridgman technique. Data obtained from it were in the form of diffractogram, graphs describing the relation between intensity ( $I$ ) of the spectrum peak of the crystal and the diffraction angles ( $2\theta$ ). The distance between planes ( $d_{hkl}$ ) determined from the diffraction peaks is listed in Table 1.

Diffraction intensity from Bragg's law requires that the path difference from the diffracted rays must be in the integer of the X-ray wavelength, but also depends on atomic and crystalline structure. From this condition, the type of Bravais lattice can be determined. It will be in the simple cubic Bravais lattice if the diffraction peaks gives all the available ( $hkl$ ) planes. It will be in the body-centered cubic (bcc) Bravais lattice if the diffraction intensities show the summation of integers  $h+k+l$ , and if  $h$ ,  $k$ ,  $l$  are totally odd or totally even then the crystal has a face-centered cubic (fcc) Bravais lattice.

The XRD results showed that the massive Pb(Se, Te) compound system produced take the polycrystalline form and has the 100% dominant peak on the peaks of Miller indices (200).

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This peak relates to  $2\theta$  angle around  $29,24^\circ$  and this value decreases to the domination of Se atom, which is in the PbSe the value become  $27,51^\circ$ . The same thing happens to the other diffraction peaks. For example, the second dominant peak at the  $2\theta$  angle of  $41,77^\circ$  relates to plane (220) in PbTe and decreases to  $39,36^\circ$  in PbSe and relates to the same plane, which is (220).

Table 1. Diffraction angles and (hkl) plane related to  $\text{Pb}(\text{Se}_{1-x}\text{Te}_x)$ 

x	Compound	$2\theta$ (degree)	Int. (c/s)	hkl	x	Compound	$2\theta$ (degree)	Int. (c/s)	hkl
0	PbSe	23,7481	5	111	0,6	$\text{Pb}(\text{Se}_{0,4}\text{Te}_{0,6})$	23,9646	95	-
		27,5184	100	200			24,9758	753	111
		39,3625	84	220			27,7000	2829	200
		46,5252	4	311			39,5454	842	220
		48,7343	28	222			41,2200	122	-
		57,5556	11	400			48,9456	210	222
		23,7481	5	111			51,1497	109	222
0,2	$\text{Pb}(\text{Se}_{0,8}\text{Te}_{0,2})$	25,0190	1569	111	0,8	$\text{Pb}(\text{Se}_{0,2}\text{Te}_{0,8})$	25,3606	29	111
		27,7414	6174	200			29,3401	100	200
		39,5720	1633	220			41,8786	60	220
		48,9441	443	311			49,4909	15	311
		51,2076	270	222			51,8546	19	222
		57,1225	777	-			60,5901	11	400
		71,6263	297	-			68,6312	26	420
							76,2297	16	422
0,4	$\text{Pb}(\text{Se}_{0,6}\text{Te}_{0,4})$	23,9646	95	-	1,0	PbTe	25,2622	1143	111
		24,9758	753	111			29,2405	5214	200
		27,7000	2829	200			41,7767	2619	220
		39,5454	842	220			49,4131	621	311
		41,2200	122	-			51,7508	786	222
		48,9456	210	222			60,5062	573	400
		51,1497	109	222			66,5981	190	331

hkl planes in Table 1 are the data from diffractogram produced in XRD characterization, which are then compared to JCPDS data so the Miller indices (hkl) from each peaks can be determined. It shows that all  $\text{Pb}(\text{Se},\text{Te})$  have cubic structure. Since (hkl) planes in

the diffractogram follows all odd or all even, thus the crystal follows the face-centered cubic Bravais lattice. Pb(Se,Te) produced in this research formed the cubic structure polycrystal with lattice constants are shown in Table 2, which is in a good agreement with literatures [8, 9]. The lattice constant is more dominant to Te or Se and obeys the 4th order polynomial as a function of Tellurium atom x:

$$a (\text{\AA}) = 6,1301 - 0,183 x + 0,354 x^2 + 1,613 x^3 - 1,426 x^4$$

Table 2. Lattice Parameters for massive Pb(Se<sub>1-x</sub>Te<sub>x</sub>) compound prepared using Bridgman technique

x	0,0	0,2	0,4	0,6	0,8	1,0
Lattice parameter	6,1281 Å	6,1282 Å	6,16 Å	6,331 Å	6,4415 Å	6,4895 Å
Structure	cubic	cubic	cubic	cubic	cubic	cubic

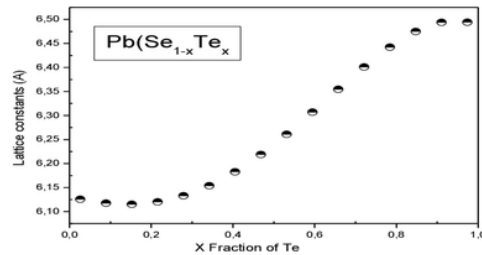


Figure 1. Lattice constants relation to Te atomic fraction x in Pb(Se<sub>1-x</sub>Te<sub>x</sub>) compound

The chemical composition of the materials is shown in Table 3, and the SEM results are shown in Figure 2.

Table 3. Chemical composition for Pb(Se<sub>1-x</sub>Te<sub>x</sub>) compound

x	ideal atomic percentage			compound	Atomic percentage In the sample			compound
	Pb(%)	Se(%)	Te(%)		Pb(%)	Se(%)	Te(%)	
0,0	50	50		PbSe	52,15	47,85	0	PbSe <sub>0,92</sub>
0,2	50	40	10	Pb(Se <sub>0,8</sub> Te <sub>0,2</sub> )	44,93	37,24	17,83	Pb(Se <sub>0,8</sub> Te <sub>0,4</sub> )
0,4	50	30	20	Pb(Se <sub>0,6</sub> Te <sub>0,4</sub> )	41,88	25,87	32,45	Pb(Se <sub>0,6</sub> Te <sub>0,8</sub> )
0,6	50	20	30	Pb(Se <sub>0,4</sub> Te <sub>0,6</sub> )	44,11	9,26	46,67	Pb(Se <sub>0,2</sub> Te)
0,8	50	10	40	Pb(Se <sub>0,2</sub> Te <sub>0,8</sub> )	50,24	1,15	48,60	Pb(Se <sub>0,02</sub> Te <sub>0,97</sub> )
1,0	50	0	50	PbTe	47,89	0	52,11	PbTe <sub>1,08</sub>

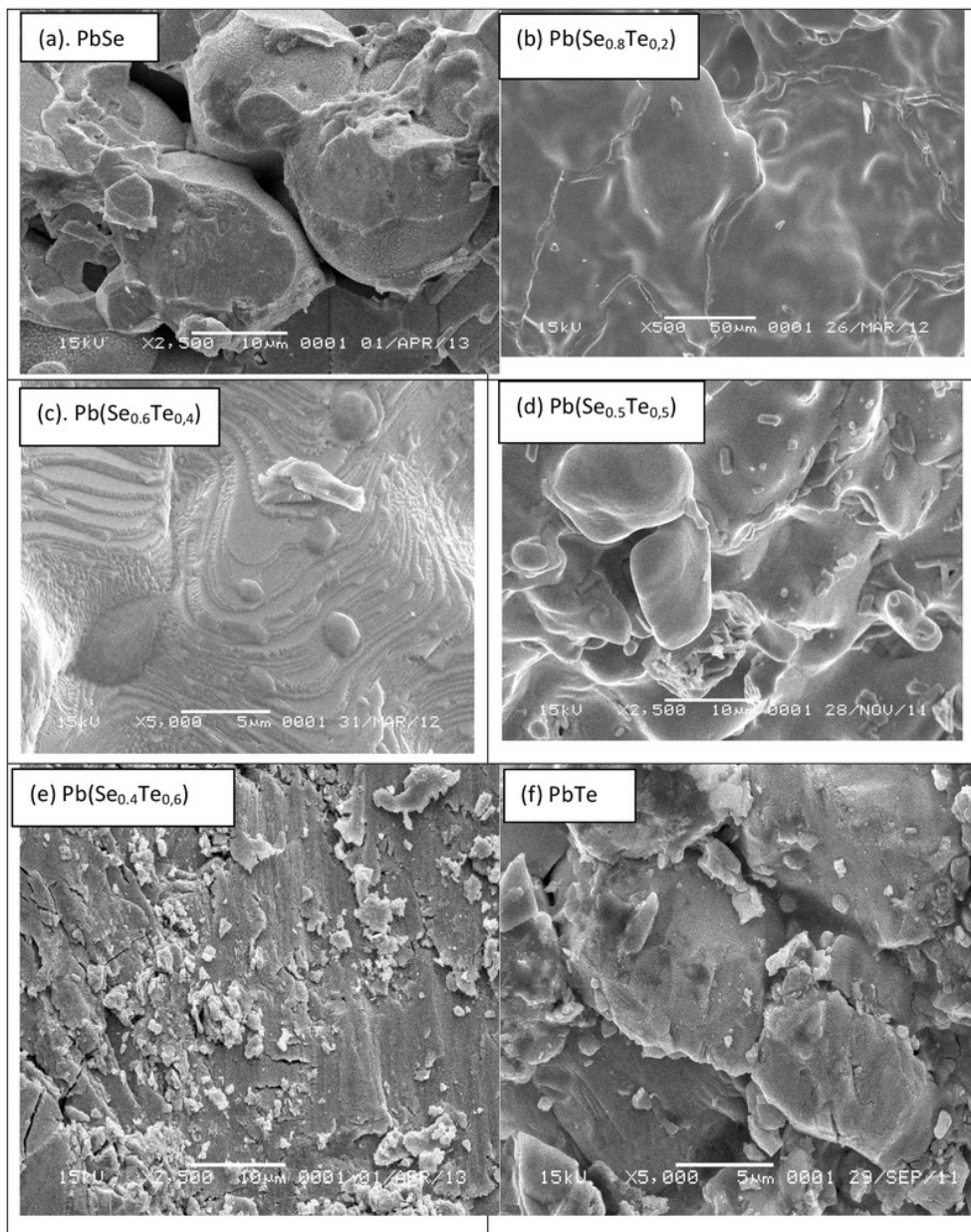


Figure 2. SEM results in surface morphology (a). PbSe, (b).  $Pb(Se_{0.8}Te_{0.2})$ , (c).  $Pb(Se_{0.6}Te_{0.4})$ , (d).  $Pb(Se_{0.5}Te_{0.5})$ , (e)  $Pb(Se_{0.4}Te_{0.6})$  and (f) PbTe

EDS results shows that all samples are non stoichiometric and rich in tellurium or lack in Pb. From EDS results, the produced crystal grains size is unclear because of the technical problem so it cannot be zoomed in to 40,000 times. But the surface condition showed the homogeneity of the sample since all surface have the same colors.

### **Conclusion**

From this research on  $\text{Pb}(\text{Se}_{1-x}\text{Te}_x)$  semiconductor material preparation and characterization, we can conclude that:

1.  $\text{Pb}(\text{Se},\text{Te})$  semiconductor has cubic structure, following face-centered cubic Bravais lattice with lattice constant  $a$  as a function of Te atomic fraction  $x$  given by  
$$a (\text{\AA}) = 6,1301 - 0,183 x + 0,354 x^2 + 1,613 x^3 - 1,426 x^4.$$
2. Results from EDAX showed that all samples had non-stoichiometric composition which was rich in Te with on some samples lack of Pb. Results from SEM showed homogeneity in composition and crystal patterns were emerged but no grains were visible on all sample since the zoom out until 40000 times was not done yet.

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