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# Growth Mechanism and Characterization of $PbTe_{0.5}Se_{0.5}$ Thin Films Used by Closed-Space Vapor Transport in a Vertical Reactor

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**Abstract**

*A simple method for growing thin film of semiconductor material $PbTe_{0.5}Se_{0.50}$ has been designed using the vapor transport (CSVT) method in a vertical reactor. The objectivity of this method is to study thin film growth formation due to chemical reactions during the deposition process in the reactor. In this study will describe some formations the vapor transport mechanism of $PbTe_{0.5}Se_{0.50}$ semiconductor material using iodine gas ($I_2$) to accelerate the etching reaction on the substrate surface. Next, we will describe how the mechanism of the reaction in the reactor zone for growing thin films on the substrate. The thin films were characterized by structural, morphology properties and its composition. The film structure is a cubic structure with the maximum diffraction intensity at peak (222). The surface morphology of the thin film has a microcubes shape with a grain size~10 to 20 $\mu$m.*

**Keywords:** *etching reaction; micro-cube; $PbTe_{0.5}Se_{0.50}$; close-spaced vapor transport*

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## INTRODUCTION

The CSVT method using horizontal reactors was first used to grow semiconductor crystals in the 1980s. This model can grow semiconductor material for III-V GaAs elements that were published in 1983 (Chávez *et al.*, 1983), other researchers have examined the equilibrium and the growth rate of thin films of $As_2$ and $As_4$, $Ga_2O$. (Cote *et al.*, 1986). Yanuar *et al.*, 2018 using the CSVT in a vertical reactor method have succeeded in growing thin layers of group I-III-VI2 elements $CuIn(S,Se)_2$. GaAs photovoltaic devices from group III-V elements have been fabricated using steam transportation methods in the form of GaAs powder material in a horizontal reactor developed by Ritenour *et al.*, 2015.

Naeemullah *et al.*, 2014 has reported that material semiconductor PbS, PbSe and PbTe have energy gap 0.41 to PbS, PbSe and 0.3 to 0.28 for PbTe. Synthesis of PbSeTe nanocubes has been reported Quan, *et. al*., (2011). $PbTe_xSe_{1-x}$ material is one of the ternary lead chalcogenides functional materials, this material is extensively studied for applications as thermoelectric materials (Korkosz *et al.*, 2014), infrared photoelectric detection (Song *et al.*, 2018) and solar cell (Leschkies *et al.*, 2009). Furthermore, this material is very prospective for infrared light emitting diodes (Yan *et al.*, 2015), and the optical properties are very effective in mid-infrared regimes. The application of lead chalcogenides material has also been reported by Yamini *et al.*, 2014 as a functional material for infrared detectors, light emission devices, infrared lasers in optical fibers and thermoelectric materials.

The technique for growing $PbSe_xTe_{1-x}$ thin films have been used many researchers in the last decade is vacuum evaporation (Kumar *et al.*, 2003), alternative techniques such as thermal evaporation PbTe and PbSe condensation (Fedorov *et al.*, 1999), physical vapor deposition (Arivazhagan, *et al.*, 2012), RF magnetron sputtering (Song *et al.*, 2018 and Feng *et al.*, 2015). Strelsov *et al.*, 1997 grew a thin layer of PbS, PbSe and PbTe alloys using a cathodic electro-deposition technique. According to Smith *et al.*, 2011, there are still few publications on the development of the high-quality synthesis of PbSe-PbTe alloy material available in published publications, perhaps because of the tendency of alloys of lead chalcogenides compound to diffuse through mixed boundaries in the pseudo-binary phase of PbSe-PbTe compounds. Today, the researchers are considered the most effective method for preparation PbSeTe including low cost, low vacuum, large area, and high-quality thin film products. The CSVT also provides a simple method to grow the metal chalcogenide elements to results in the proper structure and crystal properties. In this study, we present the CSVT in a vertical reactor use to study of $PbTe_{0.5}Se_{0.5}$ thin films grow and mechanism of the formation reaction that occurs during the deposition process. The deposition parameters such as source temperature ($T_{so}$) material alloy and substrate temperature ($T_{su}$) plays an important role in the mechanism of the reaction in the reactor. The influence of different temperature deposition on the structural and morphology properties has been investigated. Thin films of $PbTe_{0.5}Se_{0.50}$ obtained are characterized.

## METHOD EXPERIMENT

### Preparation of $PbTe_{0.5}Se_{0.50}$ ternary alloy

The $PbTe_{0.5}Se_{0.50}$ (nPb: nSe: nTe = 50:25:25) ternary alloy source material (99.99 % purity Sigma Aldrich) from a mixture of stoichiometric proportion were grown by Bridgman Horizontal Furnace method in evacuated ($10^{-5}$ Torr) quartz tube. The ampoule was placed in a horizontal furnance through a temperature of 900°C for 24 hours the procedure has been described in the literature (Yanuar *et al.*, 2018). The $PbTe_{0.5}Se_{0.50}$ polycrystalline ingots (black metallic in color) was crushed using a mortar to powder form and then compressed with a pressure of 300 kg/cm$^2$ using a hydraulic press with a diameter of 1.8 mm and a thickness of 0.5 mm. The $PbTe_{0.5}Se_{0.50}$ pellet is ready to be used as source material for deposition processes in the CSVT.

### Design of CSVT in a Vertical Reactor

CSVT is fast and simple processes for deposition semiconductor materials. They do not require a very high vacuum; the equipment is not complicated and low cost. The reactor used for $PbTe_{0.5}Se_{0.50}$ thin films deposition is shown in Figure 1. Yanuar *et al.*, 2018 has been reported the CSVT consist of a quartz tube 20 cm high and 20 mm in diameter. The reactor tube is connected to a diffusion pump ($10^{-3}$-$10^{-4}$ Torr) to vacuumed before the thin film deposition. The $PbTe_{0.5}Se_{0.50}$ pellet is carried out in the reactor tube where the material source of $PbTe_{0.5}Se_{0.50}$ and substrate are placed facing each other at the base of the tube which is separated by a spacer made of quartz glass with a distance (*d*) which can be varied from 0.3 to 1 mm. Solid iodine ($I_2$) as a reagent is placed at the upper part of the reactor to accelerate the reaction for deposition (closed under vacuum by a valve). Finally, the reactor tube is placed above a heating element of silicon carbide (SiC) material. In this deposition, a slide glass microscope was used as the substrate.

Before the deposition process, the substrate was heated using a substrate heater (heating coil) with a temperature interval of 60-90°C for 10 minutes.

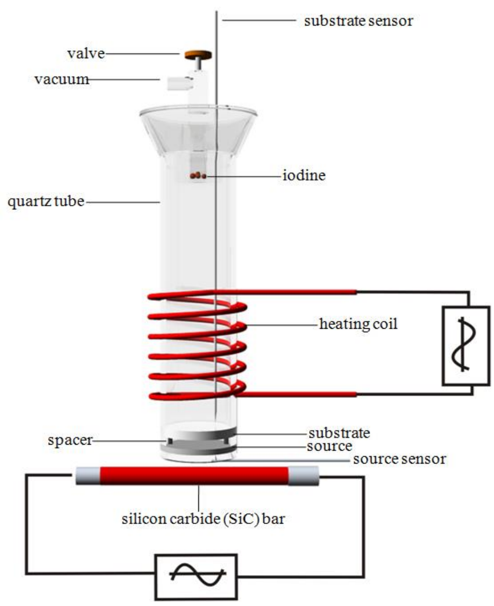


Figure 1. Schematic diagram of CSVT vertical reactor

The $PbTe_{0.5}Se_{0.50}$ source material and substrate temperature were measured by sensors put inaccessible point (out of the reactor tube deposition zone). Because the distance between the substrate and the source is very close (*d*=1 mm) to accept sensor, the sensor for the source is placed under the reactor and above the substrate.

In this study, two samples were grown with the following labeled: sample 380/320 with the temperature deposition ($T_{so}$=380°C; $T_{su}$=320°C) and sample 650/560 ($T_{so}$=650°C; $T_{su}$=560°C). The temperature gradient between the source material and the substrate separated by spacers 1 mm which is the driving force for transporting the source material in the growth of $PbTe_{0.5}Se_{0.50}$ thin films. The deposition time for the growth of source material on substrates in the reactor is 10 minutes. When deposition is finished, vacuum pumps are closed. Finally, the reactor cooled by opening the lid of the reactor. A $PbTe_{0.5}Se_{0.50}$ thin film as samples is characterized to observe structural, morphology properties and their composition.

**Characterization**

Characterization of the structure source material $PbTe_{0.5}Se_{0.50}$ as powder and thin films of the sample used x-ray diffraction (XRD), Shimadzu XRD-700 Japan with radiation sources $CuK_{\alpha}$= 0.15406 nm. Morphology and elemental composition of the sample using a scanning electron microscope (SEM) and energy dispersive x-ray spectroscopy (EDS), SEM, JEOL JSM-6360 LA, Japan (the energy of the electrons is 15 keV).

## RESULTS AND DISCUSSION

**The Mechanism of Growth $PbTe_{0.5}Se_{0.50}$ thin films**

A scheme in Figure 2 describes the mechanism of the growth of $PbSe_{0.5}Te_{0.5}$ thin film in the reaction zone CSVT in vertical reactor method. The iodine gaseous species reacts between two zones (source-substrate) will be produced $PbI_2$ gaseous by diffusion.

In the reaction zone is divided into two regions i.e the region between the source ($T_{so}$) and the substrate temperature ($T_{su}$) is not very large, the formation of the material are transported only by diffusion reaction between the two regions.

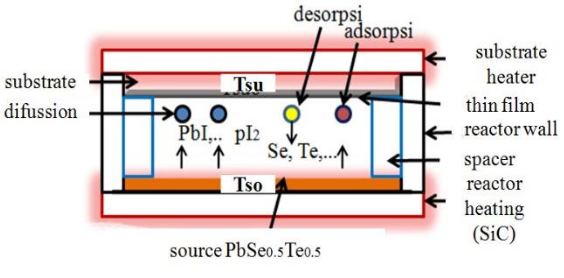


Figure 2. The mechanism of growth of $PbSe_{0.5}Te_{0.5}$ thin film in the reaction zone by the CSVT method in a vertical reactor

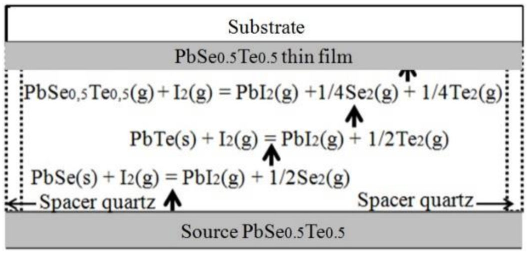


Figure 3. The reactions in the reaction zone for source material deposition on the substrate

The temperature gradient between the source and the substrate govern mass transportation for the gaseous $Se_2$ and $Te_2$ desorption and further undergone adsorption for the deposit on the substrate temperature ($T_{su}$) colder than on the source ($T_{so}$). Figure 3 describes the reaction mechanism grown on the thin film of $PbSe_{0.5}Te_{0.5}$ on a substrate in a separate reaction zone 1 mm spacer (quartz ring) to the source of $PbSe_{0.5}Te_{0.5}$ polycrystalline material.

The reaction mechanism for mass transport solid compound polycrystal $PbSe_{0.5}Te_{0.5}$ in the form of pellets into the surface of a substrate by gas iodine is as follows: first, the elements of the metal is converted into gas phase metal iodide ($PbI_2$), by the etching reaction where the phenomenon of evaporation occurs along the diffusion. Iodide and elemental gaseous species chalcogenide $Te_2$ and $Se_2$ will be transported from the surface of the source in the formation of desorption. Then, the reaction of mixing gaseous iodide chalcogen together with lead (Pb) compound adsorption on the surface of the substrate. The controller parameters on the reaction mechanism are the temperature of the source material ($T_{so}$), the distance between the source and the substrate (*d*) with the iodine gas pressure in the reaction zone. Finally, the decomposition reaction of metal with chalcogenide gaseous $Te_2$ and $Se_2$ led to the growth formation of a $PbSe_{0.5}Te_{0.5}$ thin film.

Therefore, the surface etching is one of the important reaction in the zone of reaction for the growth of thin film metal chalcogenides in the CSVT in a vertical reactor system. The question is how to control excess Te (Telluride). First, set the heater rod (SiC) for heating source material to increase the temperature as quickly as possible. With this way, the time used to reach the point of decomposition of PbTe must be minimized and resulting in less excess to Te in the final film product. The second way, excess Pb in the source material to offset the excess Te and reduce the non-stoichiometric composition of the thin film.

**Characterization of thin films $PbTe_{0.5}Se_{0.50}$**

For source $PbSe_{0.5}Te_{0.5}$ polycrystal sample, the XRD shows that the compound is composed completely of PbSeTe. Another phase is not detected.

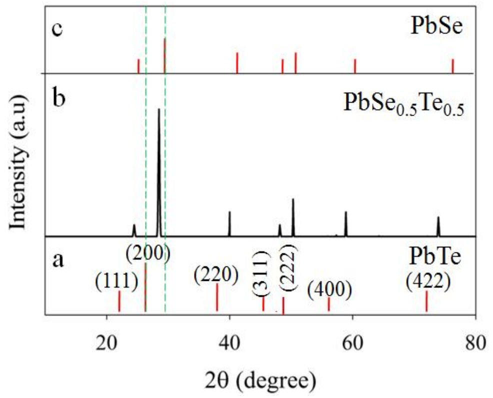


Figure 4. XRD pattern of $PbSe_{0.5}Te_{0.5}$ polycrystal for source material deposition comparison with reference PbSe (JCPDS No. 08-0028) and PbTe (JCPDS No. 06-0354)

The source $PbSe_{0.5}Te_{0.5}$ sample has an orientation along a direction (200) at the angle of reflection 2θ=28.4° between 2θ=27.5° for PbTe and 2θ=29.1° for PbSe standard reference, presented in dot green line (Figure 4b) and additional reflection from (111), (220), (311), (222), (400) and (422). These results are in good agreement between PbTe (JCPDS No. 06-0354) in Figure 4a and PbSe (JCPDS No. 08-0028) in Figure 4c and XRD standard reference as shown in Table 1. Table 1 shows the XRD measurement for $PbSe_{0.5}Te_{0.5}$ source material with the comparison to PbTe and PbSe standard XRD reference.

Figure 5 (a) shows the XRD patterns of a reference standard $PbI_2$ (JCPS-07-0235). The 380/320 sample has an orientation along a direction (002), (003) and (004) with very high relative intensities (Figure 5b). These results are in good agreement in the reference (JCPS-07-0235).

Table 1. XRD data for $PbSe_{0.5}Te_{0.5}$ source material

| PbTe ref. | | $PbSe_{0.5}Te_{0.5}$ | | PbSe ref. | | *hkl* |
|---|---|---|---|---|---|---|
| 2θ | I | 2θ | I | 2θ | I | index |
| 23.7 | 10 | 24.4 | 20 | 25.1 | 30 | 111 |
| 27.5 | 100 | 28.4 | 100 | 29.1 | 100 | 200 |
| 39.4 | 80 | 40.6 | 40 | 41.6 | 7 | 220 |
| 46.9 | 10 | 47.8 | 16 | 49.3 | 18 | 311 |
| 48.9 | 30 | 50.1 | 20 | 51.6 | 20 | 222 |
| 57.2 | 20 | 58.9 | 19 | 60.4 | 14 | 400 |
| 64.5 | 50 | 66.2 | 23 | 68.4 | 25 | 420 |
| 71.7 | 40 | 73.7 | 18 | 76.0 | 16 | 422 |

ref.=reference; 2θ=degree; I=Intensity of XRD reflection

Experimental results confirm that this temperature CSVT method in the source and substrate temperature is very low to deposit a thin film $PbSe_{0.5}Te_{0.5}$. It shows the reaction mechanism between metal Pb and iodine gas and the formation of $PbI_2$ grown on a substrate. This can be described that the diffusion reaction between $Se_2$ and $Te_2$ gas with Pb does not occur if the source material and substrate at the low-temperature deposition.

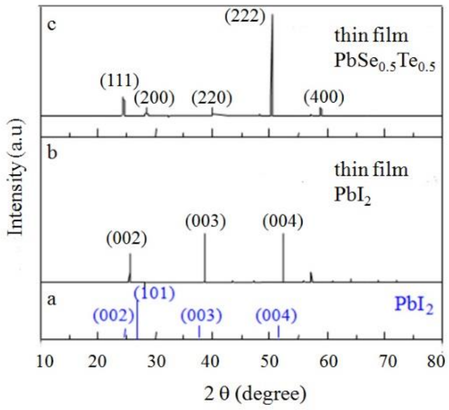


Figure 5. XRD pattern of (a) reference $PbI_2$ (JCPDS-07-023, (b) sample thin film of 380/320, and (c) 650/560

XRD pattern thin film sample 650/560 deposited on the higher source material and the substrate temperature free of iodine as shown in Figure 5 c. The film $PbSe_{0.5}Te_{0.5}$ sample has an orientation along a direction (222) with high relative intensity and additional reflection from (111), (200), (220), and (400). These result observations are shifted to higher angle diffraction angle by increasing the composition (x=0.5) and in good agreement between PbSe (JCPDS No. 08-0028) and PbTe (JCPDS No. 06-0354) XRD standard reference as shown in Figure 4. The diffraction pattern is very similar to that of the $PbSe_{0.5}Te_{0.5}$ source material. The maximum intensity of spectral peak (222) at the angle of reflection 2θ=50.3°, presented in Table 2. This film is different from the source material that peak with a maximum intensity diffraction peak (200) at an angle of reflection 2θ=28.4° still in accordance with the standard reference between PbSe and PbTe. The differences in peak intensities between them caused by variation in the scattering intensity of their lattice parameters *(a)* arrangement in the cubic structure in the film *(rock salt type)*. Table 2 presents the XRD analyses of sample 380/320 and 650/560 grown under different temperature deposition.

Table 2. XRD data for $PbI_2$ standard reference, sample 380/320 and 650/560

| $PbI_2$ ref. | | $PbI_2$ film (380/320) | | $PbSe_{0.5}Te_{0.5}$ film (650/560) | | *hkl* index |
|---|---|---|---|---|---|---|
| 2θ | I | 2θ | I | 2θ | I | |
| 25.5 | 6 | 25.4 | 11 | | | 002 |
| 38.6 | 6 | 38.7 | 67 | | | 003 |
| 52.3 | 6 | 52.4 | 62 | | | 004 |
| | | | | 24.5 | 30 | 111 |
| | | | | 28.4 | 27 | 200 |
| | | | | 40.6 | 7 | 220 |
| | | | | 50.3 | 100 | 222 |
| | | | | 59.7 | 15 | 400 |

ref.=reference; 2θ=degree; I=Intensity of XRD reflection

Shifting the angle of reflection led to the expansion of the lattice parameter *a*=6.459 Å for source material into *a*=6.286 Å for thin film. These relative structural parameters in accordance with the model are calculated using the approach of generalized gradient approximation via first-principles calculations obtained a=6.27 Å (Naeemullah *et al*., 2015). Strelsov *et al*., 1998 has been observing that thin film amorphous $PbSe_xTe_{1-x}$ by cathodic electrodeposition method where the maximum of angle reflection 2θ~31° cubic lattice structure with lattice parameter *a*=6.241 Å.

Accordingly (Yamini *et. al*., 2017) in Figure 6 shows the comparison of our lattice parameter (*a*) and energy gap (Eg) as a function of composition x=0.5 with previous studies of lead chalcogenides (PbQ, Q=Te, Se, S) alloys. In these studies show that the lattice parameters (a) decrease as a function of composition (x) and nonlinear variation of the energy band gap ($E_g$). Although the differences in our $PbSe_{0.5}Te_{0.5}$ thin film lattice parameter (*a*=6.286 Å) from linear Vegard's law value for composition x=0.5 is small (*a*=6.290 Å). Vegard's law describes that the relationship between lattice parameter PbSe and PbTe correspond to the linear interpolation as seen a solid line in Figure 6.

In this work, we did not measure the optical properties of the $PbSe_{0.5}Te_{0.5}$ thin film, using the data (Yamini *et. al*., 2017) for the energy gap at 300K obtained is 0.295 eV compared to these reference the energy gap is 0.299 eV a slightly different of 0.004 eV as seen the empty circle and red square in Figure 6. The relation between lattice parameters and energy gap as a function of the lead chalcogenide alloys composition (x) can be perspectives for tuning the band gap energy in the optical application.

Figure 7(a) shows the foto thin film and source material of $PbSe_{0.5}Te_{0.5}$ polycrystal. Figure 7(b) and (c) SEM micrographs of the surface and cross-section of $PbSe_{0.5}Te_{0.5}$ film (sample 650/560) on the slide glass microscope substrate, respectively.

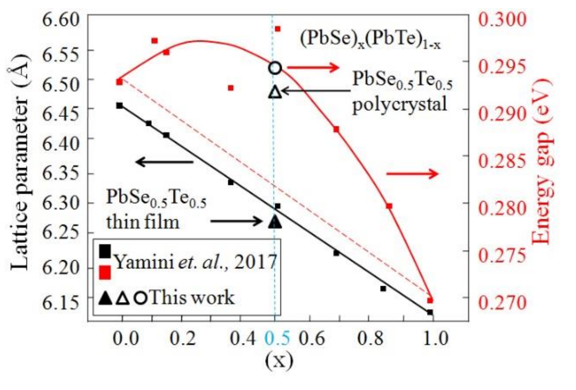


Figure 6. The relationship between the lattice parameter (*a*) and energy gap ($E_g$) as a function of composition (x) in literature data for $(PbSe)_x(PbTe)_{1-x}$ (Yamini *et. al*., 2017) with the comparison to our work

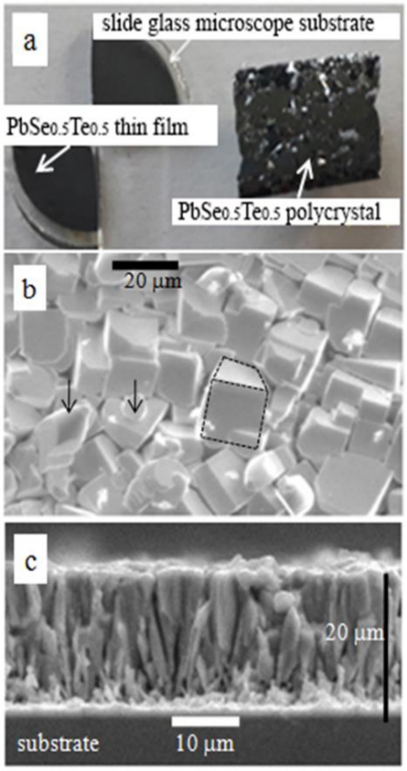


Figure 7. (a) Foto of thin film and $PbSe_{0.5}Te_{0.5}$ polycrystal, (b) SEM micrographs of sample 650/560 morphology, and (c) cross-section view

Microcubes grains with a size of about 10-20 μm are observed homogenous on the surface. The trigonal shapes grains are non-homogeneous and distributed on the $PbSe_{0.5}Te_{0.5}$ surface with a grains size of 5-15 μm as shown by the black arrow in this micrograph. The cross-sectional micrograph observation of the $PbSe_{0.5}Te_{0.5}$ film shows that the columnar grains grow on the substrate with the thickness of about 20 μm. Trigonal grains that have covered the surface $PbSe_{0.5}Te_{0.5}$ film because of excess Te (see Table 3) which has a trigonal crystal structure (*a*=*b*=0.4457 Å and *c*= 0.5929 Å) in the space group *P3121* (Li *et al*., 2015). Te excess plays an important role as doping to improve conductivity in the thermoelectric material application

Table 3 shows the comparison of composition measurements the source material and thin films samples by EDS. It is observed that the source material of $PbSe_{0.5}Te_{0.5}$ polycrystal is near-stoichiometric with a composition x = (Te/Te+Se) = 0.51 with slightly excess Te. For sample 380/320 shows a thin film obtained is $PbI_2$ with composition Pb=33.61% and $I_2$=66.39%. The absence of elements of Se and Te in source temperature ($T_{so}$=380°C) in the reaction zone of the deposition mechanism of etching only. Mixing and decomposition reactions has not occurred to deposit metallic Pb gas compounds with chalcogen $Te_2$ and $Se_2$ gas to substrate at the temperature of 320°C.

Sample 650/560 composition of thin film deposition has shown that in the source temperature ($T_{so}$=650°C) occurs deposition mixing between $PbI_2$ gas with chalcogen $Se_2$ and $Te_2$ on the substrate. This indication is shown from the results of the compositional elements of Pb=50.24%; Te=25.72%, Se=24.24%, a composition x=(Te/Te+ Se)=0.52 and Pb/(Te+Se) ratio=1.01. The composition of the Se increases from source material $PbSe_{0.5}Te_{0.5}$ polycrystal 23.98% to 24.04% in thin film sample 650/560 due to the increase in gas diffusion $Se_2$ on the substrate. Excess Te on thin films that have been deposited relatively easier for the decomposition of PbTe compared PbSe where there is always a small percentage of elements Te at both the source material and cause a thin film of excess Pb.

Table 3. EDS analyses of source material, sample 380/320 and 650/560

| Sample | Elemental composision (% atomic) | | | |
|---|---|---|---|---|
| | Pb | Te | Se | I |
| Stoichiometric | 50.00 | 25.00 | 25.00 | |
| Source material | 50.39 | 25.63 | 23.98 | 0.00 |
| Sample-380/320 | 33.61 | 0.00 | 0.00 | 66.39 |
| Sample-650/560 | 50.24 | 25.72 | 24.04 | 0.00 |

Although the time of deposition $PbSe_{0.5}Te_{0.5}$ thin film is 10 minutes, excess Pb exists due to the temperature of the source material ($T_{so}$) is higher than the temperature of the substrate ($T_{su}$) in CSVT method. The study of thermodynamic models in CSVT method for growing thin film alloys metal chalcogenides M=Sn,Pb; X=S,Se,Te very interesting to study further.

## CONCLUSION

In summary, we have deposited the microcubes $PbSe_{0.5}Te_{0.5}$ film on slide glass microscope substrates by using CSVT in a vertical reactor method. The reaction mechanism of thin film growth using this method in the reaction zone is dominated by surface etching reaction, mixing reaction and decomposition reaction of metal (Pb) with chalcogenide gaseous $Te_2$ and $Se_2$. The thin film of $PbSe_{0.5}Te_{0.5}$ is a cubic structure with the morphology of grain size varying between 10-20 μm. The composition of this film is near stoichiometric (Pb-excess) with a ratio of metal-chalcogenides is 1.01, without iodide. The CSVT a in vertical reactor method can grow a $PbSe_{0.5}Te_{0.5}$ thin film in a short time and produce a high-quality film. These findings can develop to further devices in tuning the composition (x) ternary $PbSe_xTe_{1-x}$ lattice parameters (*a*) and the optical band gap (Eg) for optoelectronic applications.

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