P: ISSN NO.: 2394-0344 RNI No.UPBIL/2016/67980 VOL-3* ISSUE-11*(Part-1) February 2019
E: ISSN NO.: 2455-0817 *Remarking An Analisation*

Growth and Characterization of PbTe Bulk Compound

Abstract

PbTe compound in bulk form was synthesized using high purity lead and tellurium by vertical directional solidification method. High purity lead (99.999%) and tellurium (99.999%) weighted in meter balance model AE 50 with a precision of +0.0005g the high purity elements were taken in stoichiometric proportions and loaded in a quartz ampoule (conical in shape). Ampoule with cone angle below 20 degree was preferred to provide nucleation site in the growth experiment. The conical quartz in which the elements have been taken in stoichiometric proportions was initially cleaned by dilute solution of Hydrofluoric acid and water (1:50) and then washed well in acetone. Powder x-ray diffraction technique was employed to identify the phase of the as grown bulk PbTe compound. Surface morphology and the stoichiometry of the bulk compound was carried out by using scanning electron microscope (SEM) with attachment of energy dispersive spectrometer (EDS).

Keywords: PbTe Compound, Energy Dispersive Spectrometer.

Rajeev Kumar
Assistant Professor,
Dept. of Physics,
S.M.P. Govt. Girls P.G. College,
Meerut, U.P., India

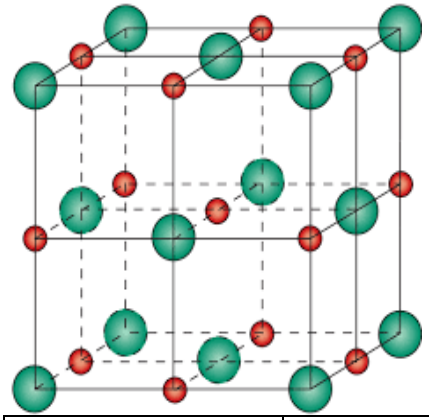
Mukesh Kumar
Assistant Professor,
Dept. of Physics,
M.M. Degree College,
Modinagar, U.P., India

Introduction

In view of global energy needs and global warming problem, research and development has been promoted in the field of thermoelectric power generation as a means of recovering the huge amount of waste heat emitted by automobiles, factories [1,2]. The thermoelectric devices convert heat into electrical energy. Among the IV-VI compounds, the lead chalcogenides with narrow energy gap are the promising materials to be used as the photoconductors infrared detectors, and thin film transistors [3-5]. It also has good performance as a thermoelectric material partly due to a low thermal conductivity. Lead telluride is a compound of lead and tellurium it is a narrow gap semiconductor. It occurs naturally as mineral altaite. It is often alloyed with tin to make lead tin telluride, which is used as an infrared detector material. Due to its cutoff wavelength λ_c=5.9μm at 77K, PbTe is an interesting option for the mid wavelength infrared region. In the present study efforts have been made to grow good qualitystoichiometric bulk compound by VDSmethod under high vacuum conditions. Detailed microscopical and composition investigations have been carried on the as gravenbulk PbTe in order to assess it for various device applications

LeadTelluride (PbTe)

Lead telluride is a compound of lead and tellurium (PbTe). It crystallizes in the NaCl crystal structure with Pb atoms occupying the cation and Te forming the anionic lattice. It is a narrow gap semiconductor with a band gap of 0.32 eV. It occurs naturally as the mineral altaite.PbTe has proven to be a very important intermediate thermoelectric material[6-7]. The performance of thermoelectric materials can be evaluated by the figure of merit, in which is the Seebeck coefficient, is the electrical conductivity and is the thermal conductivity. In order to improve the thermoelectric performance of materials, the power factor needs to be maximized and the thermal conductivity needs to be minimized.PbTe is a well-known narrow-gap semiconductor. This material is widely used for mid-infrared lasers and detectors. Moreover, PbTe has attracted a lot of interest due to its thermoelectric properties, and the material is used for small-scale cooling applications as well as for power generation in remote areas. The efficiency of a thermoelectric device is described by the dimensionless thermoelectric figure-of-merit parameter *ZT*. In the currently used thermoelectric devices based on PbTe, Si-Ge, or Bi_2Te_3 alloys, *ZT* reaches 1. This value imposes limitation to possible applications of semiconductor thermoelectric devices [8-11], and a lot of effort is put to increase the parameter.

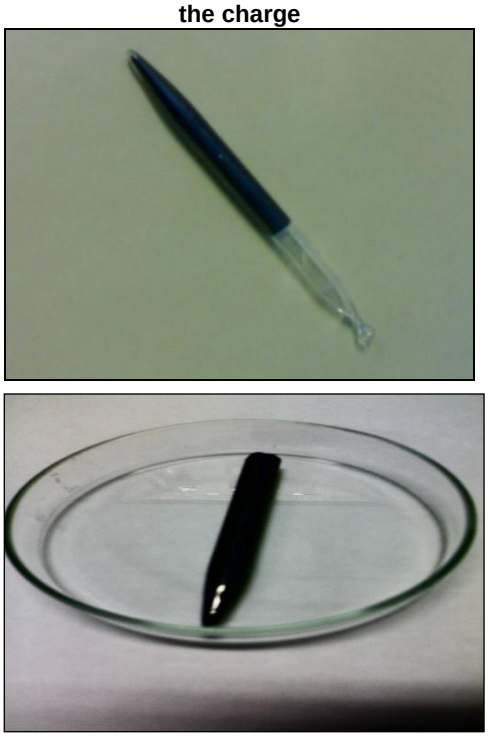


Chemical Formula	PbTe
Molecular Weight	334.8 g/mol
Group	Pb-14 Te-16
Crystal Structure	Cubic
Lattice Constant	6.454 Å
Electrical Property	
Band Gap	0.25 (0K), 0.32eV(300K)
Electron Mobility	1600 cm^2/Vs(0K)
	6000 cm^2/Vs(300K)
Hole Mobility	600 cm^2/Vs
Heat of Formation	393 kJ/mol
Thermal Conductivity	2.30 W/mK
Mechanical Property	
Density	8.16 g/cm^3
Melting Point	924°C
Mohs Hardness	3

Experimental Procedure

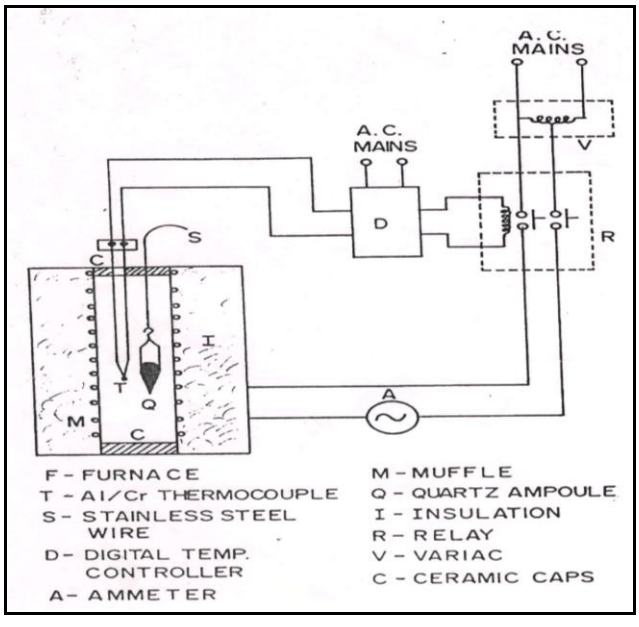
PbTe compound in bulk form was synthesized using high purity cadmium and tellurium by vertical directional solidification method. High purity lead (99.999%) and tellurium (99.999%) weighted in meter balance model AE 50 with a precision of +0.0005g the high purity elements were taken in stoichiometric proportions and loaded in a quartz ampoule (conical in shape) of 1cm diameter and about 12 cm in length. Ampoule with cone angle below 20 degree was preferred to provide nucleation site in the growth experiment. The conical quartz in which the elements have been taken in stoichiometric proportions was initially cleaned by dilute solution of Hydrofluoric acid and water (1:50) and then washed well in acetone. The ampoule was loaded with weighted amount of Pb (2.86g) and Te (2.70g) and then evacuated up to a pressure of 10^{-5} torr and sealed. Later this ampoule was heated in a vertical single zone resistance furnace which is fully temperature controlled. The PbTe charge was melted by keeping the ampoule in the constant temperature zone of the furnace as shown in fig(2). The temperature of the furnace was raised up to 380°C initially so as to melt Pb first (m.p.321°C) and kept at the same temperature for 30 minutes .The temperature was further raised to 500°C so as to melt Te (m.p.449.5°C) for another half an hour, the temperature was now raised slowly to 1100°C the temperature where the eutectic point of PbTe as shown in the phase diagram of the PbTe

Fig 1(a,b) Picture of vacuum sealed ampoule with the charge



(Fig.6). This temperature was maintained 1100°C for two hours for homogeneous mixing of the melt. After two hours temperature of the furnace was lowered to 700°C and kept at this same temperature for half an hour. The ampoule was lowered at the rate of 1cm per half till the whole length of ampoule lowered. Finally, the furnace was switched off and let it for the overnight. The graphite on the sample was cleaned thoroughly, dried and kept in the covered and dry container.

Fig 2: Schematic diagram of the VDS Technique



Growth of the Compound

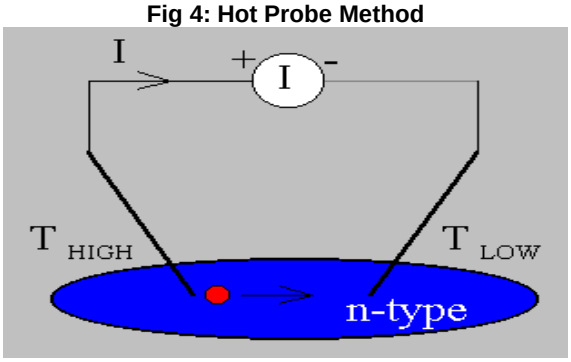
A good quality crystalline PbTe bulk compound has been grown by using vertical directional solidification technique as shown in Fig. About 30cm long and 10mm in diameter bulk compound is formed. A dark grey, dull appearance having no pits on the surface of the as grown crystal is obtained. In a good quality growth, the ingot can be removed from the quartz ampoule easily showing that the melt does not stick to the wall of the ampoule. This has been achieved by coating the inner walls of the ampoule with graphite before loading the charge and vacuum sealing.

Fig 3. Photograph of the as grown PbTe

Type of PbTe Bulk Material

Carrier type measurement of the as grown PbTe compound has been carried out by the hot probe method. This is simple, frequently used method, unencumbered by the necessity for the preparation of a special sample. One simply touches the unknown semiconductor surface by two identical metal probes, between which a galvanometer is connected as shown in the figure 4.One of the probes is heated while the other is at the room temperature. The hot probe heats the semiconductor immediately under it, with a consequent rise in the kinetic energy of the free carriers there. These carriers then move with higher thermal velocities than their cooler neighbors. These carriers therefore diffuse out of the hot region faster than their slower neighbors can diffuse back into it from the vicinity. These results in the hot region becoming slightly depleted of majority carriers and acquiring the potential of the ionized impurities there, while the vicinity of the cold probe remains neutral. Current therefore will flow in the galvanometer, the direction of which depends on the ionized impurity. Thus, on an N-type semiconductor, the hot probe is the more positive one, while on a P-type semiconductor it is more negative. The cold probe polarity therefore indicates the type.

Fig 4: Hot Probe Method

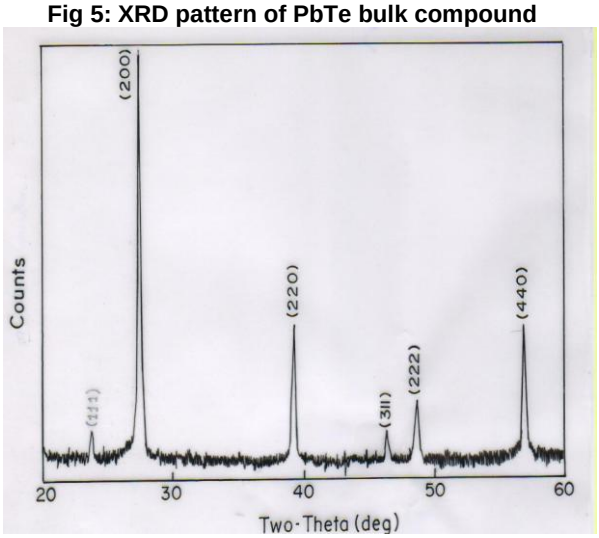


Carrier type measurement of the as grown PbTe compound has been carried out by using hot probe P/N type tester. During the investigations the type of the as grown PbTe compound was found to be P type.

XRD Analysis

Powder X-ray diffractometer model Bruker Axes D4 has been used to characterize the PbTe compound and to identify the phase present in the as grown compound. Powder X-ray diffraction pattern of the powdered as grown lump shows very sharp and well resolved peaks as shown in fig 5. The powder data are in good agreement with the JCPDS file of PbTe (cubic) JCPDS (38-1435).

Fig 5: XRD pattern of PbTe bulk compound



On analyzing the diffraction pattern, it was observed that the as grown compound consists of (111), 200, 220, 311, 222, 440 planes of PbTe having cubic structure. However, some reflections of un reacted Pb, and Te are also observed as indicated in the diffraction pattern (Fig.5).

SEM with EDS Analysis

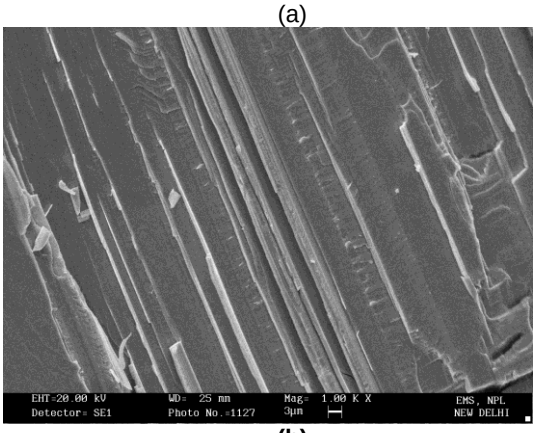
Scanning Electron Microscopy (SEM) with EDS provides the information about high-resolution image, Elemental microanalysis and particle characterization, Grain size, shape and distribution; With EDS to added analytical capability to present SEM, Compositional analysis, Elements distribution in material.

Morphology and Structure of PbTe compound

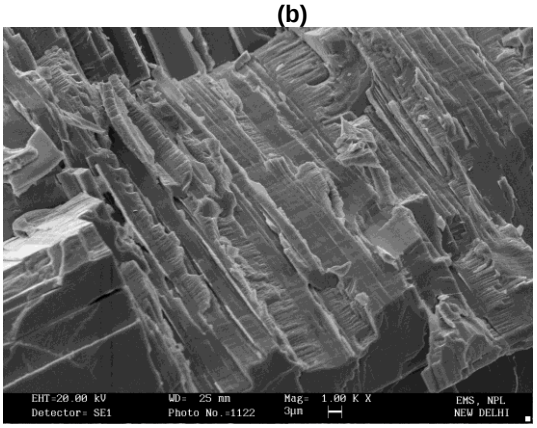
A good quality PbTe compound was obtained after synthesis. Surface morphology and the elemental compositional analysis of the as grown compound was carried out using scanning electron microscope (SEM) and energy dispersive spectrometer (EDS) system attached with SEM respectively. Small broken pieces taken from the compound were thoroughly examined under SEM model Leo 440 at different areas and suitable magnifications. Fig 6(a) and 6(b) represents the SEM micrographs recorded at different areas and different magnifications. From the micrograph in Fig 6(a) and 6(b), presence of set of cleavage planes revealing the formation of ordered structure of Pb Te compound has taken place during the synthesis.

Fig 6(a,b) SEM Images of PbTe Bulk Compound

(a)



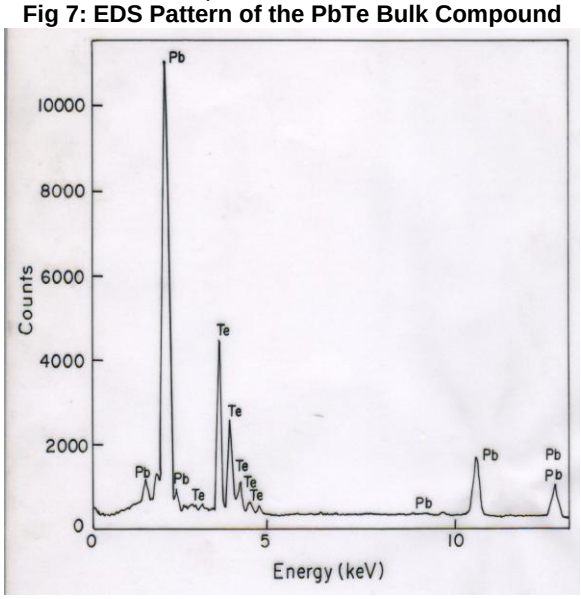
(b)



Elemental Compositional Analysis of the Compound

Elemental compositional analysis of the PbTe compound was also carried by using energy dispersive spectrometer attached with SEM.EDS pattern of the as grown compound revealed that the stoichiometry of the PbTe are maintained throughout the compound after the growth and no other element is found in the compound.

Fig 7: EDS Pattern of the PbTe Bulk Compound



Conclusion

A good quality PbTe bulk compound having no voids and pits on the surface of the lump and have smooth surface and grayish finish was grown using VDS method. The compound was found to be stoichiometric and consists of single phase(cubic) XRD data of as grown compound matches with the JCPDS file of PbTe (cubic) JCPDS (38-1435). From the SEM micrograph the presence of set of cleavage planes revealing the formation of ordered structure of PbTe compound has taken place during the synthesis. Further this bulk compound will be used to make the nano structured thin films for various device applications.

References

A.S Barros, E. Abramof and P.H.O RapplBrazillian Journal of Physics vol. 36, no 2A, (2006) 474

Akram N. Al-Mashhadany and Noor S. SadeqEngg& Tech. Vol.25.no 10 (2007) 1171.

Goldsmid, H. J. Introduction to Thermoelectricity. Vol. 121 (Springer, 2010). [8]Nolas, G. S., Sharp, J. and Goldsmid, H. J. Thermoelectrics : basic principles and new materials developments. (Springer, 2001).

Jeong, C., Kim, R. and Lundstrom, M. S. On the best bandstructure for thermoelectric performance: A Landauer perspective. Journal of Applied Physics 111, (2012).

Kanatzidis, Mercouri G. (2009-10-07). "Nanostructured Thermoelectrics: The New Paradigm†"(http://pubs.acs.org/doi/pdf/10.1021/cm902195j).Chemistry ofMaterials.22(3):648–659.doi:10.1021/cm902195j https://doi.org/10.1021/cm902195j).

Liao, B. L., Zebarjadi, M., Esfarjani, K. and Chen, G. Cloaking Core-Shell Nanoparticles from Conducting Electrons in Solids. Physical Review Letters 109, (2012).

Rowe DM 1996 CRC Handbook on thermoelectric (Boca raton, Fl:CRC press)

Rowe DM and Bhadari CM 1983, Modern thermoewlectric (London; Holt-Saunders)

Shi, X., Yang, J., Salvador, J. R., Chi, M. F., Cho, J. Y., Wang, H., Bai, S. Q., Yang, J. H., Zhang, W. Q. and Chen, L. D. Multiple-Filled Skutterudites: High Thermoelectric Figure of Merit through Separately Optimizing Electrical and Thermal Transports. Journal of the American Chemical Society 133, 7837-7846, (2011). 64

U.P.Khairnar, P.H. Pawar and G.P.Bhavasar, Crst. Res. Technol 37, 12 (2002) 1293

Zhang, Q., Cao, F., Liu, W. S., Lukas, K., Yu, B., Chen, S., Opeil, C., Broido, D., Chen, G. and Ren, Z. F. Heavy Doping and Band Engineering by Potassium to Improve the Thermoelectric Figure of Merit in p-Type PbTe, PbSe, and PbTe1-ySey. Journal of the American Chemical Society 134, 10031-10038, (2012).