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Structural study of the quaternary $Se_{68}Te_{19-x}Sn_{13}Bi_x$(x=8, 9, 10, 11, 12) chalcogenide crystals through X-ray Diffraction

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ABSTRACT

The X-ray diffraction technique is used to study the structure of the material. The quaternary system $Se_{68}Te_{19-x}Sn_{13}Bi_x$ (x=8, 9, 10, 11, 12) is analyzed with the powder diffraction method which is known as the finger print identification of the material. The material is heterogeneous and has the different phases. The various parameters regarding the material viz. miller indices (h, k, l values), volume, density, lattice spacing, FWHM, particle size and Lorentz's polarization factor have been calculated. The system taken is showing both the cubic and hexagonal structure. This confirms the polycrystalline behavior of the system showing the presence of a multiphase structure. The material has same phases in all compositions as the peaks are almost at the same positions in all the samples, However there is slight variation in their intensity. The samples are prepared by the melt quenching technique. The Lorentz polarization factor gives the lower value of intensity of reflection at the intermediate angles compared to those in forward and backward directions. The particle size in the material found to be increasing up to x=11 thenafter decrease. In average the distance of closest approach for cubic system is found to be 9.62 Å and for the hexagonal system it is found to be 5.02 Å. The density of material is found to be more in the hexagonal system as that of cubic system

Keywords: Chalcogenide crystals;XRD ;Miller indices, particle size ;density

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INTRODUCTION

Crystalline structures occur in all classes of materials, with all types of bonds. Most of the materials are polycrystalline; they are made of a large number of single crystals called crystallites. After the discovery of X-rays these materials are being studied at the world level to find the internal structure and properties of these materials.Chalcogenide materials have attracted much interest in the last couple of decades because of their interesting physical properties which can be controlled by changing the chemical composition , The addition of a third elemental impurity such as Ge, Sn, In, Pb, etc has a pronounced effect on structural, physical, optical, electronic and thermal properties [1-5]. Selenium in the pure state has disadvantages because of its short lifetime and low sensitivity. This problem can be overcome by alloying Se with some impurity atoms (Bi, Te, Ge, Ga, Sb, As, etc), which gives higher sensitivity, higher crystallization temperature and smaller ageing effects. Several workers have studied the effect of bismuth on the optical and electrical properties of chalcogenide materials [6-8]. The lattice perfection and the energy band gap of the material play a major role in the preparation of the device for a particular wavelength, which can be modified by the addition of dopants [9-10]. Since all of the crystals are highly degenerate semiconductors, it is reasonable to assume that structural defects have an insignificant effect on their electrical properties. The doping with Sn has a strong effect on the temperature dependences of the thermoelectric power and electrical conductivity of the crystals.

MATERIALS AND METHODS

Material synthesis: - $Se_{68}Te_{19-x}Sn_{13}Bi_x$ (x = 8, 9, 10, 11, 12) polycrystalline material are prepared by air quenching technique. The exact proportions of high purity (99.999%) Se, Te, Sn and Bi elements, in accordance with their

atomic percentages, are weighted using an electronic balance (LIBROR, AEG-120) with the least count of 10^{-4} gm. The material is then sealed in evacuated (~10^{-5} Torr) quartz ampoule (length ~ 5 cm and internal diameter ~ 8 mm). The ampoule containing material is heated to 800^{o} C and held at that temperature for 10 hours. The temperature of the furnace is raised slowly at a rate of 3 - 4^{o} C per minute. During heating, the ampoule is constantly rocked, by rotating a ceramic rod to which the ampoule was tucked away in the furnace. This is done to obtain homogeneous alloy. After rocking for about 10 hours, the obtained melt is quenched in air. The quenched sample is then taken out by breaking the quartz ampoule and grinded to the fine powder. The nature of the alloy is ascertained by powder X-ray diffraction technique. For this, X-ray diffraction (XRD) patterns of sample are taken at room temperature by using an X-ray diffractometer (Philips, PW 3050/60). The copper target is used as a source of X-rays with λ = 1.54 Å (Cu $K_{\alpha 1}$).

RESULTS AND DISCUSSION

We have prepared the Se, Te, Sn and Bi quaternary crystalline material by the air quenching technique, then after the materials are analyzed by the X-Ray diffraction technique and various parameters regarding the structural information from the XRD are studied as :-

3.1 $Se_{68}Te_{11}Sn_{13}Bi_8$ System

The XRD of this system is shown in fig3.1(a). From the Fig 3.1(a) we can see peaks which are sharp and distorted. This material is showing the behavior of cubic as well as of the hexagonal crystalline system. Various (h, k, l) values corresponding to the peaks are shown in the Fig 3.1(a) and are tabulated in Table 3.1(a) at their corresponding positions.

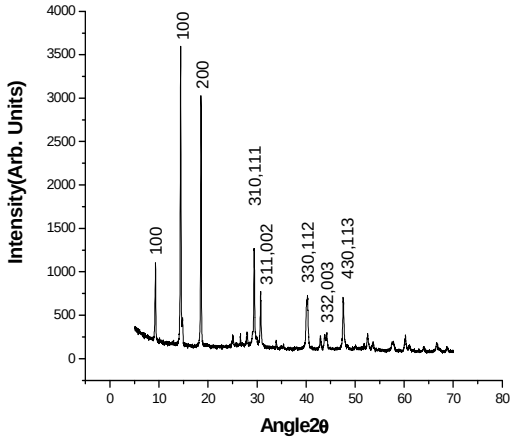

Fig 3.1(a)

Table 3.1(a)

2θ	hkl	Crystal System
9.31	100	cubic
14.29	100	hexagonal
18.56	200	cubic
29.28	310,111	Cubic, hexagonal
30.72	311,002	Cubic, hexagonal
40.23	330,112	Cubic, hexagonal
44.01	332,003	Cubic, hexagonal
47.60	430,113	Cubic, hexagonal

The various values of FWHM and d-spacing which are obtained from the XRD fig. for the different peaks are shown in the Table 3.1(b).

Table 3.1(b)

Pos. [°2Th.]	FWHM [°2Th.]	d-spacing[Å]
9.31	0.18	9.50
14.29	0.18	6.20
18.56	0.17	4.78
29.28	0.19	3.05
30.72	0.20	2.91
40.23	0.44	2.24
44.01	0.17	2.08
47.60	0.27	1.92

From these values we have calculated the lattice parameters for cubic system and hexagonal unit cell.

For Cubic:

As we know

$$a = (\lambda/2)\ (N/\ Sin^2\theta)^{1/2}$$

Also

$$a = d\ (h^2 + k^2 + l^2)^{1/2}$$

For this system this gives a = 9.64 Å and volume is, $V = a^3 = 895.84$ Å^3

Distance of closest approach = a = 9.64Å.

Density is given by the relation [11,21]

$$\rho = \frac{1.66020 \sum A}{V} \quad \ldots (1)$$

Where $\sum A$ is the sum of the atomic weights of the atoms in the unit cell, ρ is the density in (g/cc) and V is the volume of the unit cell (Å^3).Here the substance is a compound whose composition can be represented by the simple chemical formula, then

$$\sum A = n_1 M \quad \ldots (2)$$

Where n_1 is the number of the molecules per unit cell and M is the molecular weight, here in cubic unit cell n_1 =1. The density of the material in this case by cubic consideration of unit cell is found to be 18.50 g/cc.

For Hexagonal:
There are two variables in hexagonal unit cell which are given as

$$a = \sqrt{\lambda^2/_{3A}} \quad \text{and} \quad c = \sqrt{\lambda^2/_{4B}}$$

Therefore value of a=7.18Å, c=5.75Å. And the volume is, $V=0.866a^2c=256.70$Å^3.

Distance of closest approach is = $\sqrt{\frac{a^2}{3} + \frac{c^2}{4}}$ = 5.04 Å

In the hexagonal unit cell the value of n_1=6 and hence the density of the material is found to be 387.54 g/cc.So from the cubic and hexagonal unit cells average density is 203.02 g/cc.

The particle size is found by [11,21]

$$t = \frac{0.9\lambda}{B Cos\theta} \quad \ldots (3)$$

Where B is the broadening of the diffraction line measured at half its maximum intensity (radians), t is the diameter of the crystal particle and λ is the wavelength of X-rays used (1.54Å).

Lorentz polarization factor is a purely geometric factor which is the combination of Lorentz factor and polarization

factor and given as:

Lorentz polarization factor = $\frac{1+cos^2 2\theta}{sin^2\theta \cos\theta}$

The Lorentz polarization factor decreases the value of intensity of reflection at the intermediate angles compared to those in forward and backward directions.

Table3.1(c)

θ (degree)	Cosθ	B (radian)	Particle size t (A°)	Lorentz factor	Lorentz pol. factor
4.65	0.996	0.0016	869.72	39.22	309.05
7.14	0.992	0.0016	875.12	16.47	127.14
9.28	0.986	0.0014	1004.05	9.90	74.84
14.64	0.967	0.0016	895.81	4.05	28.51
15.36	0.964	0.0017	845.74	3.69	25.53
20.11	0.939	0.0037	398.93	2.25	14.22
22.01	0.927	0.0014	1067.96	1.92	11.59
23.80	0.915	0.0023	658.58	1.68	9.74

The particle size, Lorentz polarization factor and the Lorentz factor's variation calculated using XRD data are shown in Table 3.1(c) From the calculated values the mean value of the particle size is found to be 0.0826 μm.

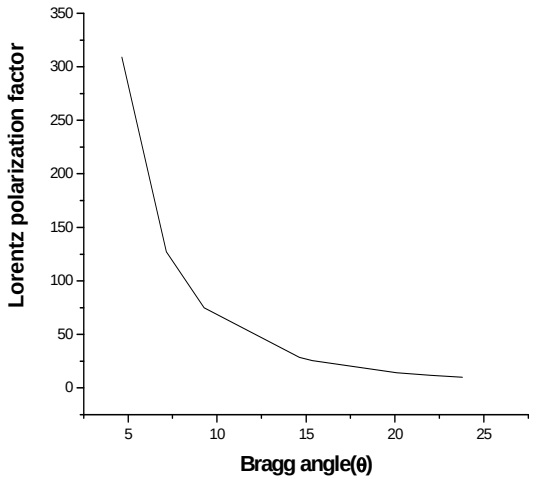

Fig 3.1(b): Lorentz pol. Factor variation with Bragg's angle for $Se_{68}Te_{11}Sn_{13}Bi_8$

3.2 $Se_{68}Te_{10}Sn_{13}Bi_9$ System

The XRD of this system is shown in fig3.2(a)

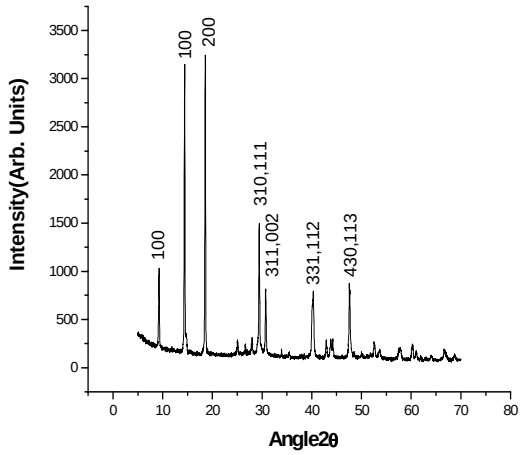

Fig3.2(a)

The material is showing the same trends. The cubic phase together with the hexagonal one is due to the polycrystalline behavior of the compound tending to present a multiphase structure. The Various (h, k, l) values corresponding to the peaks obtained in the fig3.2(a) are shown in Table 3.2(a). The various values of FWHM and d-spacing which are obtained from the XRD graph for the different peaks are shown in the Table 3.2(b)

Table.3.2(a)

2θ(degree)	hkl	Crystal System
9.12	100	Cubic
14.44	100	hexagonal
18.48	200	Cubic
29.40	310,111	Cubic, hexagonal
30.64	311,002	Cubic, hexagonal
40.27	331,112	Cubic, hexagonal
47.45	430,113	Cubic, hexagonal

Table 3.2(b)

Pos.[°2Th.]	FWHM [°2Th.]	d-spacing[Å]
9.12	0.18	9.74
14.44	0.16	6.16
18.48	0.16	4.81
29.40	0.19	3.04
30.64	0.19	2.91
40.27	0.42	2.23
47.45	0.24	1.95

In $Se_{68}Te_{10}Sn_{13}Bi_9$ system parameters for cubic unit cell are a = 9.71 Å, volume V = a^3 = 915.49 $Å^3$ and the density of the material is found to be 18.26 g/cc. Distance of closest approach for cubic system = a =9.71Å.The system parameters for the hexagonal unit cell are a = 7.09 Å, c=5.868 Å, volume V=255.80 $Å^3$ and the density of the material is 392.10 g/cc.Distance of closest approach for hexagonal system = 5.03 Å. Average value of density of both types of crystal systems is 205.18 g/cc.

Table 3.2(c)

θ (deg)	Cosθ	B (rad)	Particle size t (Aº)	Lorentz factor	Lorentz pol. factor
4.56	0.997	0.0016	868.85	40.44	318.66
7.22	0.992	0.0013	1027.33	16.05	123.90
9.24	0.987	0.0013	1032.54	9.89	74.76
14.70	0.967	0.0016	895.81	4.01	28.07
15.32	0.964	0.0016	898.59	3.71	25.82
20.13	0.939	0.0035	413.45	2.24	14.15
23.22	0.919	0.0020	754.08	1.74	9.86

The particle size, Lorentz polarization factor and the Lorentz factor's variation calculated using XRD data are shown in Table 3.2(c). The average value of the particle size for this sample is found to be 0.0841 μm.

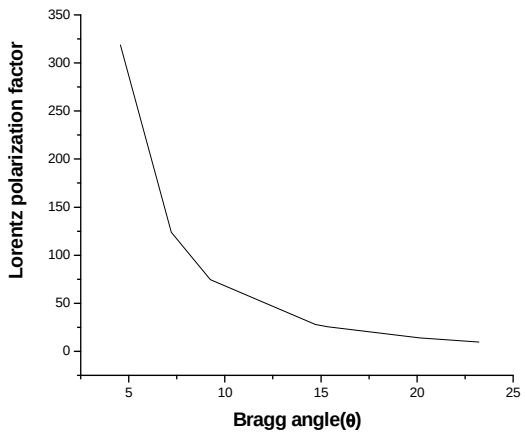

Fig3.2 (b): Lorentz pol. Factor variation with Bragg's angle for $Se_{68}Te_{10}Sn_{13}Bi_9$

3.3 $Se_{68}Te_9Sn_{13}Bi_{10}$ System

The XRD of this system is shown in Fig.3.3(a)

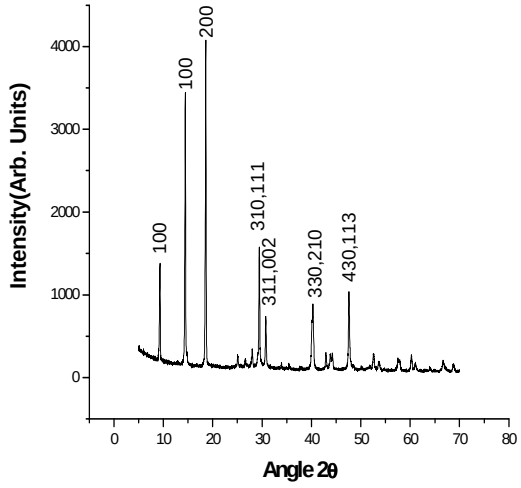

Fig3.3(a)

The Various (h, k, l) values corresponding to the peaks obtained in the fig.3.3(a) are shown in Table 3.3(a) The various values of FWHM and d-spacing which are obtained from the XRD graph for the different peaks $Se_{68}Te_9Sn_{13}Bi_{10}$ are shown in the Table 3.3(b)

Table.3.3(a)

2θ	hkl	Crystal System
9.46	100	Cubic
14.44	100	hexagonal
18.78	200	Cubic
29.70	310,111	Cubic, hexagonal
30.94	311,002	Cubic, hexagonal
39.97	330,210	Cubic, hexagonal
47.75	430,113	Cubic, hexagonal

Table 3.3(b)

Pos.[°2Th.]	FWHM [°2Th.]	d-spacing[Å]
9.46	0.18	9.39
14.44	0.17	6.16
18.78	0.16	4.75
29.70	0.19	3.01
30.94	0.19	2.89
39.97	0.41	2.25
47.75	0.24	1.90

In $Se_{68}Te_9Sn_{13}Bi_{10}$ system parameters for cubic unit cell are a = 9.51 Å, volume V = a^3 = 860.08 $Å^3$ and the density of the material is found to be 19.59 g/cc. Distance of closest approach for cubic system = a = 9.51Å. The system parameters for the hexagonal unit cell are a = 7.09 Å, c=5.64 Å, volume V=246.08 $Å^3$ and the density of the material is found to be 410.82 g/cc. Distance of closest approach for hexagonal system = 4.97Å. Average value of density due to both types of crystal systems is 215.20 g/cc.

Table3.3(c)

θ (deg)	Cos θ	B (rad)	Particle size t (A^o)	Lorentz factor	Lorentz pol. factor
4.73	0.996	0.0016	869.72	37.46	295.18
7.22	0.992	0.0014	997.98	16.05	123.96
9.34	0.986	0.0013	1032.54	9.71	73.40
14.85	0.966	0.0016	896.74	3.94	27.58
15.47	0.963	0.0016	899.53	3.65	25.25
19.98	0.939	0.0035	424.14	2.27	14.34
23.87	0.914	0.0020	758.20	1.67	9.68

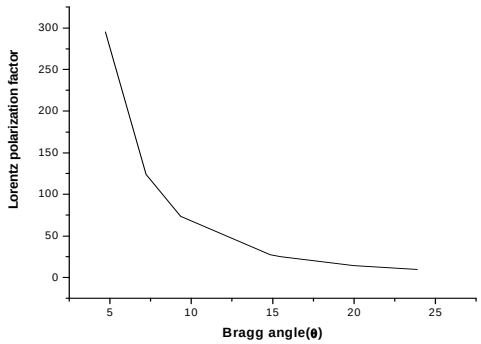

fig 3.3(b): Lorentz pol. Factor variation with Bragg's angle for $Se_{68}Te_9Sn_{13}Bi_{10}$

The particle size, Lorentz polarization factor and the Lorentz factor's variation calculated using XRD data are shown in Table 3.3(c). The average value of the particle size for this sample is 0.0839 μm.

3.4 $Se_{68}Te_8Sn_{13}Bi_{11}$ System

The XRD of this system is shown in fig3.4(a) The hkl values for different peaks calculated from the XRD and the crystal system they have shown in the Table 3.4(a)

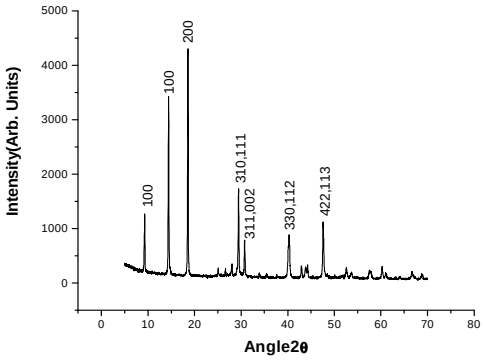

Fig3.4(a)

The FWHM and d-spacing values obtained for $Se_{68}Te_8Sn_{13}Bi_{11}$ are given in the Table 3.4(b).The values of FWHM and plane spacing are used to calculate the particle size and Lorentz factor for $Se_{68}Te_8Sn_{13}Bi_{11}$ sample.

Table.3.4(a)

2θ	hkl	Crystal System
9.46	100	Cubic
14.44	100	hexagonal
18.48	200	Cubic
29.40	310,111	Cubic, hexagonal
30.94	311,002	Cubic, hexagonal
39.97	330,112	Cubic, hexagonal
47.75	422,113	Cubic, hexagonal

Table 3.4(b)

Pos.[°2Th.]	FWHM [°2Th.]	d-spacing[Å]
9.46	0.17	9.74
14.44	0.17	6.16
18.48	0.16	4.81
29.40	0.18	3.04
30.64	0.17	2.91
40.27	0.41	2.23
47.45	0.24	1.91

Table 3.4(c)

θ (deg)	Cosθ	B (rad)	Particle size t (A°)	Lorentz factor	Lorentz pol. factor
4.73	0.996	0.0014	993.97	37.46	295.18
7.22	0.992	0.0014	997.98	16.05	123.90
9.24	0.987	0.0013	1032.54	9.89	74.76
14.70	0.967	0.0016	895.81	4.01	28.07
15.32	0.964	0.0014	1026.97	3.71	25.67
20.13	0.939	0.0034	424.15	2.24	14.15
23.72	0.915	0.0020	757.37	1.68	9.74

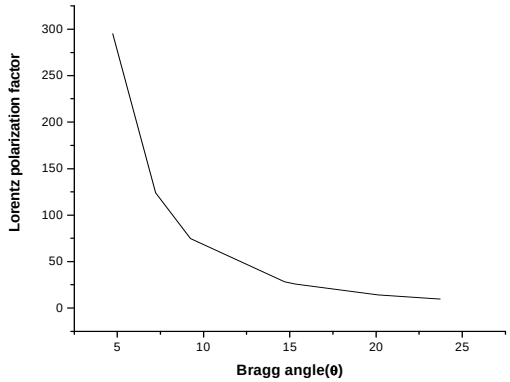

Fig3.4(b): Lorentz pol. Factor variation with Bragg's angle for $Se_{68}Te_8Sn_{13}Bi_{11}$

In $Se_{68}Te_8Sn_{13}Bi_{11}$ system parameters for cubic unit cell are a = 9.57 Å, volume V = a^3 = 876.46 $Å^3$ and the density of the material is found to be 19.38 g/cc. Distance of closest approach for cubic system = a = 9.57 Å. The system parameters for the hexagonal unit cell are a = 7.09 Å, c=5.86 Å, volume V=255.80 $Å^3$ and the density of the material is found to be 398.40 g/cc. Distance of closest approach for hexagonal system = 5.03Å. Average value of density due to both types of crystal systems is 208.89 g/cc.

In the Table 3.4(c) we calculate the particle size, Lorentz polarization factor and Lorentz factor's variation. The average particle size comes out to be 0.0875µm.

3.5 $Se_{68}Te_7Sn_{13}Bi_{12}$ System

The XRD of this system is shown in fig3.5(a) This system is showing the behavior of cubic as well as of the hexagonal system. Here also both the phases are present in the composition which shows the polycrystalline nature [11-18].

The various (h, k, l) values corresponding to the peaks obtained in the XRD figure and the crystal system they have is shown in Table 3.5(a) The plane spacing and FWHM values for different peaks obtained is given in the Table 3.5(b). From these values we have calculated the lattice parameters for cubic crystal systems and hexagonal crystal system [11, 19].

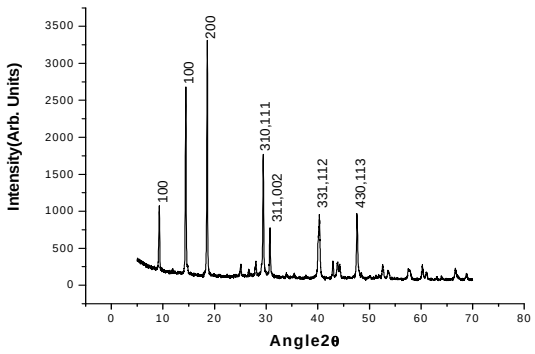

Fig3.5(a)

Table 3.5(a)

2θ	hkl	Crystal System
9.12	100	Cubic
14.10	100	hexagonal
18.78	200	Cubic
29.40	310,111	Cubic, hexagonal
30.64	311,002	Cubic, hexagonal
39.97	331,112	Cubic, hexagonal
47.75	430,113	Cubic, hexagonal

In $Se_{68}Te_7Sn_{13}Bi_{12}$ system parameters for cubic unit cell are a = 9.67 Å, volume V = a^3 = 904.23 $Å^3$ and the density of the material is found to be 18.93 g/cc. Distance of closest approach for cubic system = a = 9.67Å

Table 3.5(b)

Pos. [°2Th.]	FWHM [°2Th.]	d-spacing[Å]
9.12	0.20	9.74
14.10	0.18	6.31
18.78	0.19	4.72
29.40	0.19	3.04
30.64	0.19	2.91
39.97	0.40	2.25
47.75	0.24	1.90

The system parameters for the hexagonal unit cell are a = 7.05 Å, c=5.95 Å, volume V=256.51 $Å^3$ and the density of the material is found to be 400.50 g/cc. Distance of closest approach for hexagonal system = 5.04Å. Average value of density due to both types of crystal systems is 209.71 g/cc.

Table 3.5(c)

θ (deg)	Cosθ	B (rad)	Particle size t (Aº)	Lorentz factor	Lorentz pol. factor
4.56	0.997	0.0017	817.74	40.44	318.66
7.05	0.992	0.0016	873.23	16.91	131.22
9.39	0.986	0.0016	878.54	9.49	71.74
14.70	0.967	0.0016	895.81	4.01	28.07
15.32	0.964	0.0016	898.59	3.71	25.82
19.98	0.939	0.0034	434.13	2.27	14.34
23.87	0.914	0.0020	758.20	1.67	9.68

Table 3.5(c) shows the calculated values for Particle size, Lorentz factor

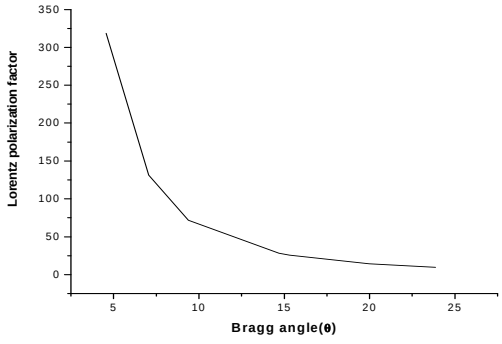

Fig3.5(b): Lorentz pol. Factor variation with Bragg's angle for $Se_{68}Te_7Sn_{13}Bi_{12}$

Table-3.6

Sample	Particle Size(μm)	Density(g/cc)
$Se_{68}Te_{11}Sn_{13}Bi_8$	0.0826	203.02
$Se_{68}Te_{10}Sn_{13}Bi_9$	0.0841	205.18
$Se_{68}Te_9Sn_{13}Bi_{10}$	0.0839	215.20
$Se_{68}Te_8Sn_{13}Bi_{11}$	0.0875	208.89
$Se_{68}Te_7Sn_{13}Bi_{12}$	0.0793	209.71

The average values of the particle size and densities for all the samples are shown in Table3.6

The average value of all the particle sizes of all the samples is 0.0834 μm and the density is the 208.40 g/cc. The average distance of closest approach for cubic system is found to be 9.62 Å and for the hexagonal system it is found to be 5.02 Å. In this work the structure of the aggregate is studied, using the term in the wide sense to mean the relative size, perfection and orientations of the particles or grains making up the aggregate. Whether these particles are large or small, strained or unstrained, oriented at random or in some preferred directions, frequently has very important effects on the properties of the materials. If the material contains more than one phase, its property naturally depend upon the properties of each phase considered separately and on the way these phase occur in the aggregate. Such materials offers wide structural possibilities since the size, perfection and orientations of the particles or the grains of one phase may differ from those of the other phases. Here the polycrystalline material is prepared by the air quenching, so here in this case the materials are under the many kinds of the crystal imperfection such as non uniform strain because it is so characteristics of the cold worked states of metals and alloys, that is why in this case distorted hexagonal structure are obtained. Here we can say that a cold worked metal or the alloy have the deformed texture, twinned annealed crystals or become polygonal in shape.Addtion of Bi may cause Nonlinear refractive index to increases linearly with linear refractive index as reported by Ishu [20].

CONCLUSION

In the present work the structure of $Se_{68}Te_{19-x}Sn_{13}Bi_x$ (x=8, 9, 10, 11, 12) system is studied and is found to be showing both cubic as well as hexagonal system. It shows that the material has polycrystalline nature, tending to present in multiphase structure. The effect of the Bi ,Sn doping is very small, systems showing the cubic as well as the hexagonal phase but with some distortion due to the strain and deformation effects of the cold work or the annealing The Lorentz polarization factor gives the lower value of intensity of reflection at the intermediate angles compared to those in forward and backward directions. The average value of particle size for all the samples is 0.0834 μm. In average the distance of closest approach for cubic system is found to be 9.62 Å and for the hexagonal system it is found to be 5.02 Å.

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