EXPERIMENTAL DETAILS

CHAPTER II

Experimental Details

2.1 Preparation of Samples :

The samples are prepared using quenching method. The multicomponent systems consists of elements Se, Te and In. The molecular weights and melting points of above elements are as given below:

Element	Atomic Weight	Melting Point Oc
Selenium	78.96	217
Tellurium	127.6	449
Indium	114.8	156.2

Six compositions of Se-Te, with In in different percentage are prepared. The following formula is used to calculate the weight of each element, to prepare the samples in the required ratios.

1)
$$Se_{70} - Te_{30}$$

= atomic weight of Se x 70 + atomic weight of Te x 30 = $\frac{78.96 \times 70}{930}$ + $\frac{127.6 \times 30}{930}$ (ten grams weight of the total sample) = 5.9432 + 4.1161

= 10.059 gm

Similarly for Se_{70} - $Te_{30-x}In_x$ (where x = 1,3,5,7,9% atomic weight)

= atomic weight of Se x 70 + atomic weight of Te_{30-x} + atomic weight of In

Using above formula the individual weight of each element is given below.

Sample	Weight of 'Se' in gm.	Weight of 'Te' in gm.	Weight of In in gm.
Se ₇₀ - Te ₃₀	5.943	4.116	-
Se ₇₀ - Te ₂₉ In ₁	5.943	3.979	0.124
Se ₇₀ - Te ₂₇ In ₃	5.943	3.705	0.370
Se ₇₀ - Te ₂₅ In ₅	5.943	3.430	0.617
Se ₇₀ - Te ₂₃ In ₇	5.943	3.156	0.864
Se ₇₀ - Te ₂₁ In ₉	5.943	2.881	1.111

While preparing the samples, the contents were kept in Silica tubes (length 22cm, inner diameter 0.92cm and outer diameter 1cm). The tubes are cleaned three times with teepol chemical and after that with distilled water. The cleaned tubes are dried at temperature 80° C in order to remove the moisture. The tubes with the contents are sealed under vacuum. The silica tubes were fitted to vacuum system, with the help of gas welding. The elements were taken in the form of granules in order to avoid the sucking. With the help of rotatory and diffusion pumps the tube are evacuated to 10^{-6} torr for three hours. Then the silica tubes with the contents were again sealed with the help of gas welding. The silica tubes are heated in an electric furnace. The tubes were kept in a circular disc made up of fire bricks as shown in Fig.(2.1). At a time six tubes were kept in the furnace. A steel rod fixed at the centre of the disc is used for rotating the samples in the furnace. The rod is rotated through 360° for homogeneous mixing of samples. Samples are heated for 30 hours as given below:

Duration in Hours Temperature Range, °C

0 - 2	l to 200
2 - 6	200 to 400
6 - 10	400 to 600
10 - 12	600 to 850
12 - 30	850

After heating the tubes for the required period, they were taken out of furnace and suddenly quenched in ice cold water. These samples are used to prepare pellets of required dimensions.

Initially, the powder was just mechanically blended in agete morter using acetone base. To prepare the pellet about one gram powder was taken. The powder was filled in to the hole of the die having one cm. diameter, Fig(2.2). Hydraulic pressure of 2 tonnes/inch² was applied to the die for 2 minutes. The thickness of every pellet was measured after polishing, with the help of travelling microscope. These pellets are used to study I-V characteristics, d.c. conductivity and thermoelectric powder.



2.2. X-RAY DIFFRACTION

The X-ray diffraction study is used to confirm the amorphous structure of the samples. Consider the notation of Kastner, Adler and Fritzsche for bond formation in Te-Te glass, [2.1].

 Te^+ bonds itself with another Te_- or se_- forms either Te_3^+ or Se_3^+ centre. When it forms Se_3^+ center by the reaction

 Te^+ + : Se \rightarrow Te^- Se⁺ The total defect reaction becomes

 $\operatorname{Se}_{2}^{\bullet} + \operatorname{Te}_{2}^{\circ} \xrightarrow{} \operatorname{Se}_{3}^{+} + \operatorname{Te}_{1}^{-}$

The net result: is that a weaker Te-Te bond is replaced by stronger Se-Te bond. The bond energy difference is recovered, and hence formation of the conjugate pair of defect namely Te_1^- and Se_3^+ require only lone pair energy. This defect model is discussed in chapter I.

To know the amorphous structure, sample was crushed into fine powder and the powder was taken on glass plate; tooformathin film of uniform thickness. The glass plate with powder is introduced in path of monocromatic beam of X rays. Since the fine grains of the powder are randomly oriented the incident X ray beam finds some plane which satisfies the Bragg Law [2.2,2.3]. In X ray diffractometer a counter is mounted instead of photographic film. The counter gives graphical record, proportional to intensity of diffracted radiation. The X ray diffraction patterns of the samples are shown in Fig.(2.3-2.8). We have used the Hitachi X-ray diffractometer at BARC. The ranges of angular changes were restricted between 10° to 60°. The rate of change of angular displacement of goniometer was adjusted to 2°/min. The voltage of 34 KV is applied, which produces a current of 18 mA across the instrument.

All the samples used, for the study of I-V characteristics, electrical conductivity, thermoelectric powder and magnetic susceptibility are annealed. Samples are heated upto 383°K, and then the study is done.

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2.3. I-V CHARACTERISTICS :

A crystal holder consisting of two circular copper discs of perfectly smooth surface and same dimensions Fig.(2.9) is used to study - I. - V. characteristics. Among the two discs, one is in fixed position, while the other can be adjusted forward or backward. The pellet is fixed between these two discs as shown in Fig.(2.9). The electrical circuit used to study I-V characteristics is as shown in Fig.(2.10). The potential across the sample (pellet) was applied from an electronically regulated power supply. The readings were taken for different concentrations of the samples. The potential applied across the pellets of different concentrations was in the range 70 to 170 volts. The current was recorded with the help of a multimeter (RS 260 Simpson)

The observations are reproducible and they are listed in tables(2.1). The current voltage characteristics are plotted for readings at room temperature, 27°C. These curves are shown in Fig.(2.11). The nature of curves is discussed in chapter III.

*

I-V For Different Concentrations

Current(mA) Voltage(volts)	Sero-Te ₃₀	Se ₇₀ -Te ₂₉ In ₁	Se ₇₀ -Te ₂₇ In ₃	Se ₇₀ -Te ₂₅ In ₅	Se ₇₀ -Te ₂₃ In ₇	Se ₇₀ -Te ₂₁ In ₉
0	0.0	0.0	0.0	0.0	.0.0	0.0
5	0.4	0.3	0.2	0.2	0.1	0.1
10	0.8	0.6	0.5	0.4	0.2	0.2
15	1.1	0.8	0.6	0.5	0.4	0.3
20	1.5	1.1	0.8	0.8	0.6	0.4
25	1.8	1.5	1.1	6,0	0.7	0.6
30	2.2	1.8	1.4	1.1	0.8	0.7
35	2.6	2.0	1.5	1.2	0.9	80 8°0
40	3.0	2.1	1.6	1.4	0.9	0.9
50	3.5	2.6	2.0	1.6	1.0	1.0
60	4.0	3.0	2.2	L.8	1.2	1.1
70	5.2Breakdo	wn 3.5	2.4	2.0	1.4	1.3
80		4.0	2.6	2.5	1.5	1.4
06		4.8Breakdow	m 3.1	3.2	1.8	1.6
100			4.0	4.0	2.0	1.7
110			4.8Breakdown	6.0Breakdow	n 2.1	1.8
120					2.5	2.0
130					3.0	2.2
140					3.8	2.4
150					5.0Breakdo	wn 2.8
1 70						3.4 4.7 th
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2.4 D.C. ELECTRICAL CONDUCTIVITY

A flat circular pellet was fitted between the two discs of a crystal holder. For good ohmic contacts, thin silver foils are placed at the interfaces between pettet and discs. The whole arrangement is placed inside an electric furnace, so that external changes of temperature do not affect the sample. The experimental arrangement is shown in Fig. (2.12). With the help of a regulated power supply (Aplab 7111) constant voltage of 1 volt is applied across the sample. The change in current across the sample was noted with the help of Pla DM-14B Ammeter at different temperatures. The temperature cf the sample is recorded with the help of digital DC microvoltmeter (Vasavi Electronics Secunderabad). Keeping temperature constant with the help of temperature controller, currents are noted for different field strengths, for all samples. The readings for each sample are listed in Table (2.7-2.11).

From the observations, resistance, resistivity and conductivity is calculated using the following formulae.

1)	R = V/I	Where	V = Voltage applied.
			I = Current across sample.
			R = Resistance of sample.
2)	$\rho = \frac{RA}{R}$		
	Li li		A = Cross sectional area of
			sample
			L = Thickness of sample
3]	$\sigma = 1/\rho$		ρ = Resistivity of sample.
			σ = Conductivity of sample.

The values of $\log \sigma / \ln \sigma$, and 1/T are listed in Table (2.2-2.6). The graph of $\log \sigma / \ln \sigma$, vs 1/T was plotted. The procedure was repeated for all samples. The curves are reproducible as shown in Fig.(2.13).

The conductivity is given by equation (2.4,2.5),

$$\sigma = \sigma_0 \exp\left(-\frac{E_\sigma^*}{kT}\right) \qquad \dots (2.1)$$

Where σ is conductivity, σ_{0} is constant and $E_{\sigma}\star$ is activation energy.

$$\ln\sigma = \ln\sigma_0 - \frac{E_\sigma^*}{kT}$$

From the above equation

Slope =
$$E\sigma * / k$$

 $E_{\sigma} * = Slope X k$
= Slope X 0.8626 X 10^{-4} eV.

Using above equation activation energy for each sample is calculated which is listed in Table (3.1). The different graphs from conductivity are shown in Fig.(2.14 to 2.18). The results of these graphs are discussed in chapter III.

The variation of conductivity with applied field is as shown in Fig.(2.27 to 2.31). The result of these graphs are discussed in chapter III.

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CONDUCTIVITY MEASUREMENT OF Se70 -Te30

Fixed potential across sample = lvolt. Diameter of Sample = 1.012 cm.

Thickness of the sample = 0.213 cm. Area A = 0.8044 cm², A/L = 3.7765 cm.

	10 ³ /T°K ¹	I (mA)	$R = V/I(\Omega)$	$\rho = RA/L (\Omega cm)$	$\sigma = 1/\rho (\tilde{\Omega}^{1} cm^{-1})$	$\log_{10}(\bar{n}^{1}c\bar{m}^{1})$	$lro(\Omega^{-1} cm^{-1})$
	3.3557	0.03	3333.33	125884.98	7.9437×10 ⁶	-5.1001	-11,7453
	3.3003	0.08	12500.00	47214.00	21.18 x 10 ⁶	-4.6741	-10.7644
ε	3.2467	0.11	06.000	34340	29.12 x 10 ⁶	-4.5359	-10.4461
8	3.1948	0.14	7142.85	26983	37.06 x 10 ⁶	-4.4311	-10.2048
	3.1446	0.19	5263.15	19880	50.30×10^{-6}	-4.2985	-9.8994
	3,0959	0.27	3703.70	13989	71.48 x 10 ⁻⁶	-4.1459	-9.5480
	3.0487	0.33	3030.30	11406	87.67 x 10 ⁻⁶	-4.0572	-9.3437
	3.0030	0.40	2500.00	9442.8	105.9×10^{-6}	-3.9752	-9.1475
	2.9585	0.53	1886.79	7126.5	140.32×10^{-6}	-3.8529	-8.8732
	2.9154	0.59	1694.91	6401.6	156.21×10^{-6}	-3.8013	-8.7543
	2.8735	0.68	1470.58	5553.7	180.04×10^{-6}	-3.7447	-8.6240
	2.8328	0.80	1250.00	4721.2	211.81 x 10 ⁻⁶	-3.6741	-8.4614
	2.7932	0.91	1098.90	4150.4	240.94×10^{-6}	-3.6181	-8.3324
	2.7548	1.01	60.099	3739.5	267.41 x 10 ⁻⁶	-3.5729	-8.2283
	2.7173	1.05	952.38	3597.1	278.00 x 10 ⁻⁶	-3.5560	-8.1894
	2.6890	1.08	925.92	3497.0	285.95 x 10 ⁻⁶	-3.5438	-8.1613

CONDUCTIVITY MEASUREMENT OF Se₇₀ -Te₂₉ In₁

Fixed potential across sample = lvolt. Diameter of sample = l.012 cm.

Thickness of the sample = 0.198 cm. Area A = 0.8044 cm². A/L = 4.0626 cm.

	0³/T°K	I (mA)	$\mathbf{R} = \mathbf{V}/\mathbf{I}(\Omega)$	$\rho = RA/L(\Omega cm)$	$\sigma = 1/\rho \left(\frac{3\tau^1 \operatorname{cm}^1}{x} \right)$	1oga (ភ ¹ cmً ¹)	lng (\mathfrak{A}^1 cm 1)
	3.3557	0.05	20000.00	81252.0	12.30	-4.9101	-11.31
	3.3003	0.08	12,500.0	50,782.0	19.68	-4.7060	-10.83
7 8	3.2467	0.12	8,333,33	33,854.9	29.53	-4.5298	-10.83
	3.1948	0.17	5,882.35	23,897.6	41.84	-4.3785	-10.08
	3.1446	0.22	4,545.40	18,466.1	54.14	-4.2665	-9,83
	3.0959	0.27	3,703.70	15,046.6	66.45	-4.1776	-9.62
	3.0487	0.34	2,941.17	11,948.7	83.68	-4.0774	-9.39
	3.0030	0.38	2,613.57	10,618.01	93.52	-4.0291	-9.28
	2.9585	0.53	1,886.79	7,665.3	130.44	-3.8846	-8.95
	2.9154	0.61	1,639.34	6,659.9	150.13	-3.8236	-8.81
	2.8735	0.73	1,369.86	5,565.2	179.66	-3.7456	-8.63
	2.8328	0.80	1,250.00	5,078.3	196.89	3.7058	-8.53
	2.7932	0.92	1,086.95	4,415.6	226.43	-4.6451	-8.39
	2.7548	1.10	60*606	3,693.3	270.73	-3.5675	-8.22
	2.7173	1.22	819.67	3,330.1	300.26	-3.5226	-8.11
	2.6809	1.27	787.40	3,198.9	312.57	-3.5051	-8.07

	ייי די די די גר גר גר גר גר גר גר גר גר גר	CONDUCTIVITY N CONDUCTIVITY	TABLE NO. 2.4 1EASUREMENT OF Se 1 1 volt Diame	70 -TC ₂₇ In ₃ tar of Sample		
	Thickness of	sample = 0.204	cm, Area A = 0.80	344 cm^2 , $A/T_1 =$	3.9431 cm.	
) ³ /T°K ⁻¹	I (mA)	$R = V/I (\Omega)$	$\rho = RA/L (\Omega cm)$	$\sigma = \frac{1}{\rho} \left(\frac{\Omega^{1} c \bar{m}^{1}}{10^{-6}} \right)$	logr(N ⁻¹ cm ¹)	lng (2 ⁻¹ cm ¹)
3.3557	0.02	50,000.0	197,230.0	5.071	-5.2949	-12.19
3,3003	0.04	25,000.0	98,577.5	10.14	-4.9939	-11.50
3.2467	0.06	16,666.6	65,718.1	15.21	-4.8178	-11.09
3.1948	0.09	11,111.1	43,812.2	22.82	-4.6417	-10.69
3.1446	0.13	7,692.3	30,331.5	32.96	-4.4820	-10.32
3.0959	0.18	5,555,5	21,905.8	45.64	-4.3407	-9.996
3.0487	0.24	4,166.6	16,429.3	. 60.86	-4.2157	-9.7L
3,0030	0.28	3,571.4	14,082.4	71.01	-4.1488	-9.55
2.9585	0.36	2,777.7	10,952.7	91.28	-4.0396	-9.30
2.9154	0.44	2,272.7	8,961.5	111.57	-3.9525	-9.10
2.8735	0.53	1,886.8	7,439.8	134.39	-3.8717	-8.92
2.8328	0.61	I,639.3	6,463.9	154.68	-3.8106	-8.78
2.7932	0.64	1,562.5	6,161.1	162.29	-3.7898	-8.73
2.7548	0.71	1,408.5	5,553.6	180.04	-3.7447	-8.64
2.7173	0.85	1,176.4	4,638.4	215.54	-3.6665	-8.44
2.6809	0.89	1,123.6	4,430.5	225.68	-3.6465	-8.40

	lng(^{Ω⁻¹cm⁻¹})	-11.80	-11.52	-11.11	-10.82	-10.60	-10.34	-10.07	-9.81	-9 . 57	-9.44	-9.26	-9.06	-8.91	-8.82	-8.64	-8.58
ple = 1.012cm. /L = 4.002 cm.	logσ(Ω ¹ cm ¹)	-5.1252	-5.003	-4.8242	-4.6993	-4.6024	-4.4884	-4.3719	-4.2599	-4.1552	-4.0972	-4.0226	-3.9492	-3.8700	-3.8315	-3.7511	-3.7273
.5 DF Se ₇₀ -Te ₂₅ In ₅ Diameter of Sam = 0.8044 cm ² A/	$\sigma = 1/\rho \left(\Im^{-1} \ c \mathring{\Pi}^{-1} \right)$ x 10-6	7.49	66.6	14.99	19.98	24.98	32.48	42.47	54.96	69.95	79.95	94.94	112.43	134.91	147.41	177.39	187.38
TABLE NO. 2. Y MEASUREMENT C nple = lvolt, .01cm. Area A	$\rho = RA/L(\Omega cm)$	133,399.9	100,050.0	66,699.7	50,027.0	40,020.0	30,784.6	23,540.9	18,190.7	14,292.7	12,506.2	10,531.6	8,893.2	7,411.3	6,782.9	5,636.4	5,335.9
CONDUCTIVIT ttial across sar of sample = 0.2	$R = V/I (\Omega)$	33,333.3	25,000.0	16,666.6	12,500.5	10,000.0	7,692.3	5,882.3	4,545.4	3,571.4	3,125.0	2,631.6	2,222.2	1,851.9	1,694.9	1,408.4	1,333.3
Fixed poten Thickness	I (mA)	0.03	0.04	0.06	0.08	0.10	0.13	0.17	0.22	0.28	0.32	0.38	0.45	0.54	0.59	0.71	0.75
	10 ³ /T°K ¹	3.3557	3,3003	8 3.2467	3.1948	3.1446	3.0959	3.0487	3.0030	2.9585	2.9154	2.8735	2.8328	2.7932	2.7548	2.7173	2.6809

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CONDUCTIVITY MEASUREMENT OF Seq $_{70}$ -Te $_{23}$ In $_{7}$

Thickness of sample = 0.214cm. Area $A = 0.7644cm^2$, A/L = 3.4278cm. Fixed potential across sample = lvolt, Diameter of sample = 0.997cm.

	10³/T°K ⁻¹	I (mA)	$R = V/I (\Omega)$	$\rho = RA/L (\Omega cm)$	$\sigma = \frac{1}{\rho} \left(\frac{\alpha^{-1} \tilde{cm}^{1}}{x} \right)$ × 10 ⁻⁶	logg(A ⁻¹ cm ⁻¹)	.lnơ(Q ⁻¹ cm ¹)
4	3.3557	0.01	1000,00.0	342,780.0	2.91	-5.5351	-12.75
18	3.003	0.01	100,000.0	342,780.0	2.91	-5.5351	-12.75
	3.2467	0.015	66,666.6	228,519.7	4.37	-5.3590	-12.34
	3.1948	0.02	50,000.0	171,390.0	5.83	-5.2341	-12.05
	3.1446	0.03	33,333.3	114,259.0	8.75	-5.0580	-11.65
	3.0959	0.05	20,000.0	68,556.0	14.58	-4.8361	-11.14
	3.0487	0.06	16,666.6	57,129.7	17.50	-4.7570	-10.96
	3.0030	0.08	12,500.0	42,847.5	23.33	-4.6320	-10.67
	2.9585	0.11	6'060'6	31,161.8	32.08	-4.4937	-10.35
	2.9154	0.13	7,692.3	26,367.7	37.92	-4.4212	-10.18
	2.8735	0.15	6,666.6	22,851.8	43.75	-4.3590	-10.04
	2.8328	0.19	5,263.2	18,041.2	55.42	-4.2564	-9.80
	2.7932	0.22	4,545.4	15,881.1	64.17	-4.1927	-9.66
	2.7548	0.24	4,166.6	14,282.3	70.00	-4.1549	-9.57
	2.7173	0.26	3,846.2	13,184.0	75.84	-4.1201	-9.49
	2.6809	0.28	3,571.4	12,242.0	81.67	-4.0880	-9.41



Fig. 2.13



Fig. 2.14



Fig.2:15





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Fig.2.18

VARIATION OF DC CONDUCTIVITY WITH FIELD

		1)	88	12	2	8	9	17	
213 cm.	100°C	1090 (1 ¹ cm	-3.54	-3.524	-3.512	-3.506	-3.501	-3.504	
.let) = 0.3		current (mA)	1.08	1.61	2.32	2.94	3.57	4.11	
ample (pel	Q - J - C	1090 (N ⁻¹ cm ¹)	-3.5447	-3.6851	-3.6763	-3.6888	-3.6835	-3.6801	
kness of s	75°	current mA	0.68	0.92	l.56	1.99	2.32	2.74	
Thic		<u>log</u> a (¹ cm ¹)	-4.1459	-4.1321	-4.1223	-4.1178	-4.1405	-4.1169	
	50°0	Current (mA)	0.27	0.36	0.57	0.72	0.85	1.10	
	5 ° C	logo (î ^{lcm-1})	-5.1001	-4.9992	-4.9752	-4.9338	-4.8782	-4.8425	
	29	Current (mA)	0.03	0.05	0.08	0.11	0.15	0.19	
°70 ^{-1'e} 30	Field	M∕/m	4.69 x 10 ²	7.04×10^{2}	9.38 x 10 ²	11.73 x 10 ²	14.08 x 10 ²	16.43 x 10 ²	
ample St	Applied	Voltage V(volts)	1.0	1.5	2.0	2.5	3.0	3.5	
S	Ĩ			88					

Thickness of sample = 0.198 cm.

Sample - Se₇₀-Te₂gIn₁

		والافتقادية والمتعادين والمساولة والارتباط والمتعاولة والمتعادية والمتعادية والمتعادية								والمرابقة والمرابقة والمحافظة والمحافظة والمحافظة والمحافظة والمحافظة والمحافظ والمحافظ والمحافظ والمحافظ
	Applied	Field		25°C	50	ی°C	75	S°C	. 10	0°C
	Voltage	w∕m	Current	loga	Current	loga	Current	logo	Current	loga
68	V(volts)		(mA)	(² , c ¹)	(mA)	(^{Ω⁻¹ c^{m⁻¹})}	(mA)	(I^1 cm 1)	(mA)	(1 ¹ cm ¹)
I										
	1.0	5.05×10^{2}	0.05	-4,9101	0.27	-4.1776	0.73	-3.7456	1.27	-3.5051
	1.5	7.57×10^{2}	0.08	-4,8821	0.39	-4.1939	1.10	-3.7436	1.88	-3.5108
	2.0	10.1×10^2	0.11	-4.8688	0.54	-4.1776	1.48	-3.7397	2.54	-3.5051
	2.5	12.65 x 10 ²	0.14	-4.8607	0.68	-4.1743	1.98	-3.7102	3.16	-3.5072
	3.0	15.15×10^{2}	0.18	-4.8308	0.82	-4.1722	2.32	-3.7205	3.75	-3.512Q
	3.5	17.67 x 10 ²	0.20	-4.8519	0.98	-4.1617	2.75	-3.7137	4.41	-3.5085

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Thickness of sample = 0.204 cm.

sample - $\text{Se}_{70}^{-\text{Te}_{27}} \text{In}_3$

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Sample

	Applied	Field	. 2	5°C	ũ	0°C	75	ວຸ	10(ວ。(
	Voltage V (Volts)	m∕/n	Current (mA)	Loga (R ⁻¹ cm ¹)	Current (mA)	Lọgσ (Ω ¹ cm ⁻¹)	Current (mA)	Loga (^{Ω 1} tm ^{- 1})	Current (mA)	Log _d (^Ω ^{tun1})
1 16	1.0	4.97 x 10 ²	0.03	-5.1246	0.13	-4.4884	0.38	-4.0226	0.75	-3.7273
	1.5	7.46 x 10 ²	0.06	-5.0003	0.22	-4.4360	0.60	-4.0003	1.08	-3.7150
	2.0	9.95 x 10 ²	0.09	-4.9492	0.31	-4.4120	0.83	-3.9843	1.55	-3.7131
	2.5	12.43 x 10 ²	0.11	-4.9403	0.41	-4.3875	1.11	-3.9550	1.97	-3.7058
	3.0	14.92×10^2	0.14	-4.9324	0.50	-4.3805	1.32	-3.9589	2.36	-3.7066
	3.5	17.41×10^{2}	0.17	-4.9160	0.62	-4.3540	1.56	-3.9533	2.84	-3.6931

Sample :- $Se_{70} - Te_{23} In_7$. Thickness of sample = 0.199cm.

00°C	Loga (A ¹ cm ¹)	والمراجع	-4.0880	-4.0210	-4.0438	-4.0410	-4.0255	-4.0300
10	Current (mA)		0.28	0.49	0.62	0.78	0.97	1.12
	Loga (M ⁻¹ cm ¹)		-4.3590	-4.2798	-4.3590	-4.3533	-4.2880	-4.2798
75°C	Current (mA)		0.15	0.27	0.36	0.44	0.53	0.63
0°C	Loga ((1 ст 1)		-4.8361	-4.8081	-4.7570	-4.7869	-4.7570	-4.7570
2	Current (mA)		0.05	0.08	0.12	0.14	0.18	0.21
5°C	Loga (M ⁻ tm ¹)		-4.9101	-4.8821	-4.8688	-4.8607	-4.8308	-4.8579
2	Current (mA)		0.01	0.015	0.02	0.02	0.03	0.04
Field	M∕m		5.02×10^{2}	7.53×10^{2}	10.05 x 10 ²	12.56×10^{2}	15.07 x 10 ²	17.58 x 10 ²
Applied	Voltage V (Volts)	76	1.0	1.5	2.0	2.5	3.0	3.5

| 76









Fig. 2.30



2.5 THERMOELECTRIC POWER

To determine thermoelectric power of a given amorphous sample, arrangement was made as shown in Fig.(2.19). The sample is fixed between two brass rods of equal dimensions. To know the temperature difference between the two junctions, thermocouple (Alumel-chromel) was used keeping one end of the sample hot than the other end. The temperature across one end was measured in mV of DM-14--B meter (PLA Electronic, Bombay). The temperature across other end was measured with digital dc microvoltmeter (Vasavi Electronics Secunderabad). The thermo e.m.f. developed across the sample was measured with the help of dc microvoltmeter. The temperature of both junctions is converted into degree Kelvin with the conversion factor $(1mV = 25^{\circ}C)$.

The observations for each sample are listed in Table (2.12) to (2.15). The graph of $\triangle V$ against $\triangle T$ is plotted as shown in Fig.(2.20).

Using formula $s = \Delta V / \Delta T$, thermoelectric power is calculated. The graph between S and 1/T is as shown in Fig.(2.21). The nature of the graph is linear and from this graph, the activation energy is calculated using formula, [2.6].

$$S = \frac{\Delta V}{\Delta T} = \frac{k}{q} \left[\frac{Es^*}{kT} + A \right] \qquad ..(2.2)$$
$$= \frac{k}{q} \left[\frac{Es^*}{kT} + 1 \right] \qquad Where A = 1.$$
$$S = \frac{Es^*}{q} \frac{1}{T} \qquad ..(2.3)$$

This equation gives,

Slope = Es^*/q

$$Es^* = Slope x q$$

Where Es* is activation energy and q is charge of the carrier. The graph of lno against S, Fig.(2.22) yields value of σ_0 , and the graph of Q(T) against 1/T, Fig.(2.23) gives value Qo. The values are listed in Tables (2.16-2.18) and (3.1).

We know the equations, [2.7], [2.8], [2.9]

$$Q(T) = \ln_{\sigma}(T) + \frac{q}{K}S(T)$$
$$Q(T) = Qo - \frac{EQ}{kT}$$

and

$$\sigma_{0} = 0.03 \frac{q^{2}}{m^{2}} l_{1}$$

$$= 0.03 \times \frac{q^{2} 4 \pi^{2}}{h^{2}} l_{1}$$

$$\sigma_{0} = 0.8806 \times 10^{8} l_{1}$$

$$l_{1} = \sigma_{0} \times 1.355 \times 10^{-8} \text{ cm}.$$

Where lis called inelastic diffusion length.



TERMOELECTRIC MEASUREMENT OF Se70-Te30

	$S = \Delta V / \Delta T$ $(mV k^{-1})$	0.648	0.631	0.615	0.600	0.584	0.570	0.554	0,540
	10 ³ /T (°K ⁻¹)	3.208	3.120	3.037	2.958	2.883	2.812	2.745	2,681
	$T = T_1 - \frac{\Delta T}{2}$	311.75	320.50	329.25	338.00	346.75	355,50	364.25	373.00
-	ΔV (mV)	1.62	3.15	4.60	6.00	7.31	8.60	9.70	10.80
	ΔТ (°С)	2.5	5.0	7.5	10.0	12.5	15.0	17.5	20.0
	Temp. of OTHER END T2°C	37.5	45.0	52.5	60.0	67.5	. 75.0	. 82.5	0.06
	Temp. of ONE END T ₁ °C	40.0	50.0	60.0	70.0	80.0	0.02	100.0	110.0
	1	1							

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Thermoelectric Measurement of Se_{70} -Te29- In_1

$S = \frac{\Delta V}{\Delta T}$ (mV k ⁻¹)	0.672	0.654	0.637	0.621	0.604	0.587	0.570	0.554	
10 ³ /T (°K ⁻¹)	3.208	3.120	3.037	2.958	2.883	2.812	2.745	2.681	
$T = T_{l} - \Delta \frac{T}{2}$ (°K)	311.75	320.50	329.25	338.00	346.75	355.50	364.25	373.00	
Δ V (mV)	1.68	3.27	4.78	6.21	7.55	8.81	9.97	11.08	
∆ T (°C)	2.5	5.0	7.5	10.0	12.5	15.0	17.5	20.0	
Temp. of OTHER END T2°C	37.5	45.0	52.5	60.0	c./٥	75.0	82.5	0.06	
Temp, of ONE END T ₁ °C	40.0	50.0	60.0	70.0	0.00	0.06	100.0	110.0	

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Thermoelectric Measurement of $\text{Se}_{70}\text{-Te}_{27}\text{-In}_3$

$S^{-} = \Delta V / \Delta T$	(mV K ⁻¹)	0.704	0.684	0.667	0.644	0.622	0.605	0.585	0.566
10 ³ /T	(° K̃ !)	3.208	3.120	3.037	2.958	2.883	2.812	2.745	2.681
$T = T_{i} - \frac{\Delta T}{2}$	(X °)	311.75	320.5	329.25	338.0	346.75	355.5	364.25	373.0
Δ	(m/)	1.76	3.42	4.98	6.44	7.81	6.07	10.2	11.3
ΔT	(J°)	2.5	5.0	7.5	10.0	12.5	15.0	17.5	20.0
Temp. of OTHER END	T2°C	37.5	45.0	52.5	60.0	67.5	75.0	82.5	0.06
Temp. of ONE END	Tl°C	40.0	50.0	60.0	70.0	80.0	0.06	100.0	110.0

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P. Of ER END 2.5 2.5 2.5 2.5 2.5 2.5 2.5 2.5 2.5	Termoelectric Measurement of	5e ₇₀ Te ₂₅ In ₅ and 5e ₇₀ Te ₂₃ In ₇	$\Delta T \qquad \Delta V \qquad T = T_{1} - \frac{\Delta T}{2} \qquad 10^{3}/T \qquad S = \Delta V/ \Delta T \qquad \Delta V \qquad S = \Delta V/ \Delta T$	(°C) (mV) ($\circ K$) ($\circ K^{-1}$) ($mV K^{-1}$) (mV) (mV) ($mV K^{-1}$)	2.5 1.81 311.75 3.208 0.725 1.9 0.760	5.0 3.52 320.5 3.120 0.704 3.68 0.737	7.5 5.11 329.25 3.037 0.682 5.37 0.713	10.0 6.59 338.0 2.958 0.660 6.90 0.690	12.5 7.97 346.75 2.883 0.638 8.34 0.667	15.0 9.24 355.5 2.812 0.616 9.66 0.644	17.5 10.4 364.25 2.745 0.595 11.9 0.621	
			mp. of AT	T ₂ °C (°C)	37.5 2.5	45.0 5.0	52.5 7.5	60.0 10.0	67.5 12.5	75.0 15.0	82.5 17.5	

TABLE NO. 2.16

Se₇₀-Te₃₀

 10 ³ /T/°K	$ln\sigma(T) \Omega^{-1} cm^{-1}$	S(T) mV/°K	Q _T =ln ₀ (T)+ll.608 x S(T)
 2.7	-7.7926	0.548	-1.4315
2.8	-8.1845	0.568	-1.5912
2.85	-8.3802	0.577	-1.6824
2.90	-8.5923	0.587	-1.7785
2.95	-8.7902	0,597	-1.8607
3.0	-9.0290	0.607	-1.9830
3.1	-9.2813	0.627	-2.0031

 $se_{70} - re_{29} - in_1$

10 ³ /T/°K	lng(T) Ω^{-1} cm ⁻¹	S(T) mV/°K	Q _T =ln ₀ (T)+11.608 x S(T)
2.7	-7.9914	0.564	-1.4445
2.8	-8.4059	0.585	-1.6153
2.85	-8.6132	0.595	-1.7065
2.90	-8.8435	0.606	-1.8091
2.95	-9.0507	0.617	-1.8886
3.0	-9.2810	0.627	-2.0028
3.1	-9.6726	0.648	-2.1509

 $\operatorname{Se}_{70} - \operatorname{Te}_{27} - \operatorname{In}_{3}$

 10 ³ /T/°K	$\ln_{\rm C}$ (T) Ω $^{-1}$ cm ⁻¹	S(T) mV/°K	$Q_{\rm T} = \ln_{\sigma}({\rm T}) + 11.608$ x S(T)
2.7	-8.1986	0.578	-1.4892
2.8	-8.6362	0.603	-1.6365
2.85	-8.8665	0.615	-1.7276
2.9	-9.0968	0.628	-1.8070
2.95	-9.3271	0.640	-1.8950
3.0	-9.5374	0.654	-1.9658
3.1	-9.9950	0.679	-2.1132

Se₇₀ -Te₂₅ -In₅

10 ³ /T/°K	$ln\sigma(T) \Omega^{-1} cm^{-1}$	S(T) mV/°K	Q _T =lnσ(T)+11.608 x S(T)
 2.7	-8.4980	0.590	-1.6493
2.8	-8.9356	0.616	-1.7851
2.85	-9.1889	0.630	-1.8759
2.90	-9.4192	0.643	-1.9553
2,95	-9.6495	0.656	-2.0347
3.00	-9.8798	·0.670	-2.1025
3.10	-10.3404	0.696	-2.2613

TABLE NO. 2.17

Se₇₀-Te₂₃-In₇

10 ³ /T/°K	$ln\sigma(T)\Omega^{-1}$ cm ⁻¹	S(T) mV∕°K	Q _T =ln ₀ (T)+11.608 x S(T)
2.7	-9.1889	0.612	-2.0849
2.8	-9.6726	0.641	-2.2319
2.85	-9.9029	0.656	-2.2881
2.90	-10.1792	0.670	-2.4019
2.95	-10.4095	0.685	-2,4581
3.0	-10.6628	0.700	-2.5372
3.1	-11.1465	0.730	-2.6727

TABLE NO. 2.18

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Sample	Inelastic diffusion length (m)	$\sigma_0 \ \overline{\Omega}^1 \ cm^{-1}$	Qo
Se ₇₀ -Te ₃₀	27.8 x 10^{10}	20.83	1.875
Se ₇₀ -Te ₂₉ -In ₁	26.6 x 10^{-10}	19.86	1.80
Se ₇₀ -Te ₂₇ -In ₃	23.9 x 10^{10}	17.9	1.76
^{Se} 70 ^{-Te} 25 ^{-In} 5	22.9 x 10 ¹⁰	17.1	1.55
Se ₇₀ -Te ₂₃ -In ₇	20.8 x 10^{-10}	15.6	1.53



Fig. 2.20





Fig. 2.22



Fig. 2.23

2.6 MAGENETIC SUSCEPTIBILITY MEASUREMENT

GOUY METHOD

This is the most common and simplest method used for the measurement of magnetic susceptibility of the materials, [2.10]. It has the advantage that the apparatus is simple, Fig. (2.24) and robust and can be used to measure a wide range of susceptibilities. In this technique a tube filled upto a certain height with the magnetic sample is suspended from an arm of a sensitive balance, such that the bottom part is in a strong magnetic field, while the top part is in a zero field. The whole set up is housed inside a drought free enclosure. Usually an electromagnet giving a constant magnetic field in the range 5,000 to 10,000 gause is used.

A small volume of sample dv of volume susceptibility k will experience a force dF which is given by

$$dF = H.K.dv \frac{dH}{dx}$$

where dH/dx is the gradient of magnetic field. Taking A as cross-sectional area and dx as the small height of the sample we have

$$dF = H.K.A.dx. \frac{dH}{dx}$$
 ...(2.4)

dF = H.K.A.dH. ...(2.5)

Considering the magnetic field between H and H $_{\rm o}$ and integrating E $_{\rm g}$ (2.5) we have



$$F = \int dF = \int_{H_0}^{H} H \cdot K \cdot A \, dH$$

= A.K. $\frac{1}{2} (H^2 - H_0^2)$
= A $\chi_g^{0} \frac{1}{2} (H^2 - H_0^2)$
= $\chi_g \frac{m}{1} \frac{1}{2} \cdot (H^2 - H_0^2)$...(2.6)

where m and l represents the mass and the length of the sample specimen respectively. If H_{O} is made negligible then

$$F = \chi_g - \frac{.m}{21} H^2$$
 ...(2.7)

The force experienced by the sample is measured by the change in weight, thus :

$$\chi_{g} = \frac{21 \text{ F}}{\text{m H}^{2}}$$

$$\chi_{g} = \frac{21. \Delta \text{ W}. 981}{\text{mass of sample x H}^{2}} \dots (2.8)$$

where ΔW is change in the mass of sample.

Experimental Procedure :

The experimental procedure is based on finding the susceptibility of a sample relative to the known susceptibility of a reference. The reference liquid used is benzene.

1) The empty glass tube is suspended between the poles of the magnet with its lower tip in the centre of the pole gap. It is weighed, and the balance beam is left in the released position. A suitable current say 2 ampere is passed for a minute or two and the weight is noted. A decrease in weight due to the predominating diamagnetism of glass is readily observed on the optical scale. Care must be taken to avoid prolonged passage of current, which produce heating effect.

The procedure is repeated at other field strengths obtained by passing higher currents say 4,6,8,10,12 ampere. The change in weight ΔW_t corresponding to each amperage is noted.

- 2) A reference liquid is enclosed in the sample tube up to a mark and is weighed as before without the magnetic field. The sample is weighed in magnetic field corresponding to the exact amperages, used previously for the empty tube. The change is weight ΔW_{t+r} corresponding to each setting is noted.
- 3) The tube is dried and is filled upto the same mark with the sample. The weighing procedure with the magnetic field off and on is repeated as before. This gives the change in weight ΔW_{t+s} corresponding to the same fields used before for the reference.

Measurement on Powdered Solids.

The powder is packed in the tube. Finely ground powder is used so that a maximum filling is obtained in a fixed volume. Experiments show that under normal conditions of working a loose packing or tight ramming of the powder in the tube does not appreciably affect the susceptibility measurement, provided corrections are made for the presence of air pockets.

Basic for Calculation of Magnetic Susceptibility.

The following symbols are used :

W = Weight, d = density, K = Volume susceptibility, χ = gram susceptibility, Δ W = change in weight on applying the magnetic field, V = actual volume up to the mark.

The subscripts r,s,t corresponds to the reference, sample, and tube.

 $K_{air} = 0.029 \times 10^{-6}$ cgs units.

For measurement at a fixed field corresponding to a known current, which has fix value of 12 amp.

 $\Delta W_{r} = W_{t+r} - W_{t}$ $\Delta W_{s} = W_{t+s} - W_{t}$

If the sample and reference are filled to the same mark in a tube of cross-sectional area A and are subjected to the same magnetic field H, under identical conditions of the Gouy experiment the following equations hold when the tube is surrounded by air.

> Force (s) = $g \Delta W_s = \frac{1}{2} A H^2 (K_s - K_{air})$ Force (r) = $g \Delta W_r = \frac{1}{2} A H^2 (K_r - K_{air})$ $\frac{\Delta W_s}{\Delta W_r} = \frac{K_s - K_{air}}{K_r - K_{air}} = \frac{\chi_s \mathbf{d}_s - K_{air}}{\chi_r dr - K_{air}}$

Hence

$$\chi_{s} = \left(\frac{\chi r d_{f} - 0.029 \times 10^{-6}}{\Delta W_{r}}\right) \frac{\Delta W s}{ds} + \frac{0.029 \times 10^{-6}}{ds} \dots (2.9)$$

or

or

$$\chi_s = (\text{Tube constant}) \frac{\Delta Ws}{dS} + \frac{0.029 \times 10^{-6}}{dS}$$

used for liquid sample.

With solids it is almost impossible to powder them to the same particle size, and to pack them uniformly up to a mark in the tube. This introduces variation in the volume of paramagnetic air held in the pocket of the sample. Hence the contribution K_{alr} (1-Ws/V.ds) is included, due to air enclosed per c.c. of the solid air mixture. The susceptibility of a powdered solid relative to a liquid reference is then computed from the following relationship, [French and Harrison].

$$X_{s} = \left(\frac{Xr dr - 0.029 \times 10^{-6}}{\Delta Wr}\right) \cdot \frac{\Delta Ws}{Ws} \frac{Wr}{dr} + \frac{0.029 \times 10^{-6}}{ds}$$

$$\chi_{s} = (\text{Tube constant}) \frac{\Delta Ws}{Ws} \frac{Wr}{dr} + \frac{0.029 \times 10^{-6}}{ds} \dots (2.10)$$

For higher degree of accuracy, another correction must be included to account for the situation that the volume of the solid packed upto a mark will be less than that of reference liquid by an amount equal to the volume of the miniscus. In this case V.dr = Wr - C

Where C is correction for miniscus of height 'h' for a tube of diameter 3mm.

$$C = 0.054 \frac{Ws}{n} - 0.0037$$

$$\chi_{s} = (\text{Tube constant}) \quad \frac{\Delta Ws}{Ws} \quad \frac{(Wr - C)}{dr} + \frac{0.029 \times 10^{-6}}{ds} \quad ..(2.11)$$

The calculated values of X_s at room temperature are listed in table (2.19).

Magnetic Susceptibility Variation with Temperature.

The experimental arrangement used is as shown in Figure (2.25). The temperature of sample is measured with the help of sensitive thermocouple (Alumel-Chromel). At different temperatures the change in weight of the sample was noted. The magnetic field was kept constant, (current 12 amp.). The same procedure is repeated for each sample. Using the above formula the magnetic susceptibility for that perticular temperature is calculated. The values of χ_s at perticular temperature are listed in table (2.20). The diamagnetic susceptibility is found to increase with temperature, Fig.(2.26).

When the sample is heated above room temperature then upto 353°K the diamagnetic susceptibility nearly remains same, but after that temperature, considerable increase in $\chi(T)$ is observed.

TABLE NO. 2.19

Room temperature = 25°C

Sample	Weight of Sample Ws in gm.	Change in weight of sample ∆Ws in gm.	Diamagnetic Susceptibility X _s (cgs units)
Se ₇₀ -Te ₃₀	1.0529	0.00022	-0.3082×10^{-6}
Se ₇₀ -Te ₂₉ In ₁	0.92305	0.00019	-0.3035×10^{-6}
Se ₇₀ -Te ₂₇ In ₃	0.9351	0.00019	-0.2999×10^{-6}
Se ₇₀ -Te ₂₅ In ₅	0.9732	0.00019	-0.2874×10^{-6}
Se ₇₀ -Te ₂₃ In ₇	0.9343	0.00018	-0.2511×10^{-6}
Se ₇₀ -Te ₂₁ In ₉	0.8762	0.00015	-0.2511×10^{-6}

Observations

- 1. Density of reference liquid (benzene) dr = 0.877 gm/cm^3 .
- 2. Density of sample (amorphous) ds = 3.7827 gm/cm^3 .
- 3. Tube Constant = -0.253×10^{-2} .
- 4. Correction C = 0.004608 gm.
- 5. Wr C = 0.5254 gm.
- Applied constant magnetic field current passing through the electromagnetic coils = 12 ampere.

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Magnetic Susceptibility-Temperature Variation.

6							
S€70 -Te21 In X(T)	-2.3267	-1.7508	-1.4513	-1.7508	-0.8677	-0.2841	-0.5769
Se ₇₀ -Te ₂ 3 In ₇ X(T)	-0.9741	-0.3170	-0.8041	-0.9741	-0.9741	-0.3170	-0.4794
Se ₇₀ -Te ₂₅ In ₅ X(T)	-0.5963	-0.4453	-0.5963	-0.5963	-0.4453	-0.4453	-0.2843
Se ₇₀ -Te ₂₇ In ₃ X(T)	-0.4758	-0.9592	-0.6369	-0.6369	-0.4758	-0.3146	-0.1535
Se ₇₀ -Te ₂₉ In ₁ X(T)	-0.7360	-0.5501	-0.5501	-0.5501	-0.5501	-0.5501	-0.1783
Se70-Te30 X(T)	-0.4369	-0.2887	-0.4369	-0.2887	-0.4369	-0.2887	-0.2887
Temp. of sample (°K)	313	323	333	343	353	363	373



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