CHAPTER IV

THERMOLUMINESCENCE
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CHAPTER IV

THERMOLUMINESCENCE

4.1 Introduction:

The electrons escape from traps because of thermal jostling by nearby crystal atoms, this characteristic of traps is helpful for learning more about traps. The chance of an electron's escape is an exponential function of the trap depth and the temperature. Thus by raising the temperature, the electrons can be released first from the shallow traps and at higher temperature from the deeper ones. The glow experiment, thus provides probably the most elegant technique for the fundamental studies of traps, particularly their populations and depths and as such is widely used(1).

In practice, thermoluminescence is measured by exciting the phosphor usually at a low temperature and allowing the emission to phosphoresce to a low intensity. This is followed by simultaneously measuring the emission intensity and temperature as the phosphor is heated, preferably at a slow linear rate which is made as uniform as possible. Thus the traps filled during excitation are emptied releasing the frozen-in luminescence. The intensity of emission first
increases with temperature, reaches a maximum and then decreases. Such a plot of intensity versus temperature is known as a thermoluminescence curve or more popularly a 'glow curve'. A glow curve may show many well resolved peaks. The position of each peak on the temperature scale is a measure of the energy depth of trapped electrons in the phosphor, while the area under the glow peak is proportional to the number of electrons that left the traps by the radiative process. The shape of the glow peak is determined by the rate at which electrons are released from the traps. Thus a glow curve for traps of one depth has the same shape and peak temperature regardless of the number of electrons trapped. The nature of the glow curve also depends on the warming rate used in the experiment as well as the composition and structure of the phosphor.

Ellickson and Urbach (5,6) found that the number of light quanta emitted during the stimulation of certain alkaline earth sulphide phosphors by infrared radiation is much greater than those obtained as thermoluminescence after similar excitation. They have suggested that during thermoluminescence, the energy of some electrons thermally ejected from the traps is dissipated non-radiatively and therefore the thermal glow curve must be corrected if it is to provide a true picture of trap distribution in the phosphor under study.
4.2 Methods of Measuring Thermoluminescence:

The experimental techniques used for measuring thermoluminescence are essentially the same as those employed for the decay study with an additional accessory for heating the phosphor at a uniform rate. Rate of heating is an important factor to be taken into account. Slow rate of heating increases the resolution of different peaks in a glow curve \((7,8)\) while the higher heating rate gives rise to a sharp maximum which reduces the uncertainty in its position and the corresponding temperature of maximum glow \((9,10)\). Rates of heating as low as \(0.025^\circ K/\text{sec.}\) have been used by Johnson and Williams \((11)\), compared to a high rate of \(5^\circ K/\text{sec.}\) used by many other workers. A summary of the arrangements used by different workers is given below.

In the arrangement of Randall and Wilkins \((9)\), a thin layer of powdered phosphor was spread on the surface of a copper box smeared slightly with glycerol. An electric heater coil was arranged inside the box and the temperature was measured using a copper-constantan thermocouple soldered to its surface. For working below the room temperature, the whole apparatus was kept inside a Dewar flask containing liquid air. The emission during warming was measured with a photomultiplier, and a recorder arrangement. An electric bell connected to a clock was used to record time accurately. The main drawback of this apparatus was that the
rate heating often varied with temperature. Garlick(12) removed the above defect by further modifying this apparatus and was able to obtain high warming rates of the order of 5° K/sec.

Arbell and Halperin (13) have studied the different glow peaks separately by using filters. The alkali halide phosphor crystal was mounted in a vacuum cryostat which could be heated from liquid air temperature to about 600°K or above with the help of a heating element. The glow emission of the crystal was measured with RCA IP 28 photomultiplier detector unit. The output from the amplifier was fed to a 50- mV Brown recorder used to record the glow curves. The temperature was measured with a calibrated copper-constantan thermocouple, one junction of which was fixed near the crystal and the other in an ice-bath. The heating rate could be varied from 10° K/min to 20° K/min and with a high speed of the recorder an accuracy of about 0.2°K was achieved in the measurement of temperature differences within a glow peak.

Joshi and others (14−16) mounted the phosphor crystal on the flattened end of a thick copper rod kept inside Dewar flask containing liquid nitrogen to cool the phosphor to a desired low temperature. The phosphor was heated by passing current through a wire surrounding the lower end of the rod. A stream of dry nitrogen obtained by
boiling liquid nitrogen in another Dewar flask was circulated in a transparent quartz cylinder to prevent condensation on the specimen at low temperature. A spark produced between two aluminium rods was used as the excitation source. The glow intensity was measured with RCA 931 photomultiplier and galvanometer unit, while the temperature was recorded with a copper-constantan couple.

Recently Brunner (17) has devised a double beam spectrometer for the measurement of thermoluminescence. The main features of his instrument are:

(i) the measured 'black body' radiation is suppressed by the double beam technique and
(ii) the strong demagnification of the beam permits the use of small phosphor quantities.

Ghosh (18) has also devised an automatic rapid scanning spectrometer to study thermoluminescence spectra.

4.3 Present Method:

The apparatus used for the present study (shown in Fig. 4.1) has been designed and prepared in this laboratory. It consists of a cubical metal box made of copper which is divided into two chambers. The lower chamber has a copper cylinder provided with a circular cavity at the top which can be heated at a convenient uniform rate by passing a suitable constant current through a small coil embedded in
FIG 4.1
APARATUS FOR THERMOLUMINESCENCE MEASUREMENT

V = CAVITY; U = COPPER CUP;
W = WINDOW; R = SCREEN
J, K = SHUTTERS; C = BRASS CYLINDER ATTACHED TO RJ
T1, T2 = TERMINALS TO EE = WATER INLET AND OUTLET HEATING COIL
G, H = AIR CIRCULATION TUBES
\( \text{insulation} \)}
insulating material packed round the cavity. A small copper cup for holding the phosphor powder for the purpose of exciting and heating it, just fits into the cavity. It is also provided with a handle to lift it up from the cavity. The lower half of the cylinder consists of a ballow chamber around the cavity fitted with two tubes at its bottom for water circulation. The inner cavity is held in position by packing asbestos in the intervening space and the whole assembly is encased in a light-tight box. Water circulation facilitates a rapid cooling down of the cavity to room temperature after the phosphor has been heated. Two additional tubes are also provided to circulate compressed air from an air compression pump through the outer jacketing space so as to expel any water drops left inside. The upper chamber of the cubical box carries a tightly fitting slide door. The top of the cubical box is provided with a part for surmounting the photomultiplier tube. A hollow metallic cylinder open at both ends can be slid in between the phototube and the phosphor by means of a side shutter so that when the cylinder is in position, phosphorescent emission can reach the photocell. However, when the cylinder is in out position an extension plate provided as a continuation on the side of the top of the cylinder, automatically shuts off the emission from the phosphor to the phototube, providing a safeguard for the phototube.
4.4 Experimental Procedure:

The arrangement for the measurement of glow intensity as well as the source of excitation were exactly the same as used for the decay studies described in the previous chapter. Using the above apparatus, the temperature and glow intensity cannot be recorded simultaneously as a function of time with sufficient accuracy. Hence a calibration curve of the heater was first obtained in the following manner to get the exact values of temperature.

The phosphor holder containing some mercury was put in the heater cavity. One junction of a copper-constantan thermo-couple was dipped well in mercury and the other one was kept immersed in ice taken in a beaker. The desired current to be used in final studies was passed through the heater coil. The galvanometer deflection was recorded as a function of time and then a graph was plotted between the two. The hot junction was then taken out from the cavity and transferred into another hard glass test-tube containing mercury, which itself could be heated in a sand-bath. A thermometer was inserted inside the test-tube to record the temperature. Now the deflection of the galvanometer was noted as a function of temperature of the hot junction and the deflection versus temperature graph was plotted. With the help of these two graphs, a third graph between temperature and time was obtained which gave the necessary calibration curves shown in Fig.4.2
and Fig. 4.3 for the two warming rates used in the present work.

The phosphor to be studied was packed in the holder up to a fixed mark so as to have a uniform thin layer and was excited for four minutes. The holder was then placed in the cavity and the brass cylinder was aligned with the phototube by sliding the side arm. After the phosphor had decayed to a negligibly low value of intensity as shown by the galvanometer reading, the heater current was switched on and the galvanometer deflection was recorded as a function of time. The apparatus was then cooled to room temperature and another sample was studied in a similar manner. In this way, the majority of the samples of the two series were studied at both the warming rates and the corresponding glow curves were plotted, using the calibration graphs.

4.5 (A) Theory:

Randall and Wilkins (9) were the first to investigate thermoluminescence theoretically. They used a model in which electrons in metastable states are raised thermally to an excited state from which they return to the ground state with the emission of thermoluminescence. Their theory also assumes monomolecular mechanism and the absence of re-trapping.

If a phosphor contains traps of one depth E only and at any time t, these contain n electrons, the luminescence
intensity $I$ is determined by the rate at which electrons escape from traps and is represented by the equation,

$$I = - \frac{dn}{dt} = ns \exp\left(-\frac{E}{kT}\right) \ldots \ldots \ldots (4.1)$$

$$= - \frac{n}{\tau} \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots (4.2)$$

where $\frac{1}{\tau} = p = s \exp\left(-\frac{E}{kT}\right) \ldots \ldots \ldots (4.3)$

The symbols have their usual meaning stated earlier. From equation (4.1),

$$\frac{dn}{n} = -s \exp\left(-\frac{E}{kT}\right) \cdot dt \ldots \ldots \ldots \ldots (4.4)$$

If the phosphor is now heated at a uniform rate of $B$ degrees per second, then

$$dT = B \cdot dt \ldots \ldots \ldots \ldots (4.5)$$

Putting this value of $dt$ in eq. (4.4) and integrating we get,

$$\log\frac{n}{n_0} = -\int_0^T \frac{1}{B} s \exp\left(-\frac{E}{kT}\right) - dT$$

or

$$n = n_0 \exp\left[-\int_0^T s \exp\left(-\frac{E}{kT}\right) \cdot \frac{dT}{B}\right] \ldots \ldots \ldots (4.6)$$

where $n_0$ represents the number of filled traps at $t=0$ i.e. at the instant heating is started. The thermoluminescence intensity $I$, at the temperature $T$ is given by,

$$I = n_0 \cdot s \exp\left(-\frac{E}{kT}\right) \cdot \exp\left[-\int_0^T s \exp\left(-\frac{E}{kT}\right) \cdot \frac{dT}{B}\right] \ldots \ldots (4.7)$$

This expression represents the glow curve for a phosphor containing traps of one depth only.
However, Randall and Wilkins have shown that the maximum intensity of a glow curve occurs at a temperature somewhat below the temperature at which the probability of an electron escaping from the trap is one second. Hence,

\[ s \exp\left[ -\frac{E}{kT} \left( 1 + f(s, B) \right) \right] = 1 \quad \ldots \quad \ldots \quad (4.8) \]

where \( f(s, B) \) has a small value, \( T_G \) is the temperature of maximum glow and a function of \( E \). Therefore,

\[ E = kT_G \left( 1 + f(s, B) \right) \log s \quad \ldots \quad \ldots \quad (4.9) \]

Neglecting \( f(s, B) \), since it has a small value compared to unity, e.g. (4.9) becomes,

\[ E = kT_G \log_e s \quad \ldots \quad \ldots \quad \ldots \quad \ldots \quad (4.10) \]

or \[ E = 2.303 \, kT_G \log_{10} s \quad \ldots \quad \ldots \quad \ldots \quad \ldots \quad (4.11) \]

This equation has been extensively used to determine the distribution of traps because in practice the trap depth \( E \) is not single valued but varies over wide ranges.

4.5 (B) Value of \( s \):

The frequency factor \( s \) is a function of temperature but it is assumed to be independent of trap depth and the mean life-time of an electron in a trap. If the trap is regarded as a potential box, \( s \) should be equal to the product of the frequency with which the electron strikes the sides of the box and of the reflection coefficient. The value of \( s \) should therefore be less than the thermal vibration frequency of a crystal i.e. \( 10^{12} \) sec\(^{-1} \). For different
phosphors, various values of the constant $s$ have been reported by different workers (8,9,19 - 21).

The value of $s$ can be determined from decrease in photoconductivity at low temperatures, phosphorescence decay, dielectric relaxation time measurements and from the cross-section for luminescence capture of photo-electrons. But all these methods are more or less tedious and the value obtained therefrom cannot be taken to be sufficiently trustworthy. Therefore, one prefers to preassume a certain value of $s$ rather than determining it experimentally. However, the investigations of Bube (8) on ZnS phosphors together with previously published results of other investigators, favour that the probable range of values for $s$ is between $10^8$ and $10^{10}$ sec$^{-1}$. The minimum value it can have is about $10^6$ sec$^{-1}$.

In thermoluminescence, many workers have used the shift of glow peak temperature with different warming rates to estimate the value of $s$ (22 - 24). From the measurements of glow curves after various periods of decay, assuming a distribution of traps with the same $s$, Randall and Wilkins have recommended the value of the order of $10^{8.5}$ sec$^{-1}$ for zinc sulphide and alkaline earth sulphide phosphors. Hence the value of $s$ chosen for the present investigation has been taken as $10^9$ sec$^{-1}$. 
4.6 Results:

(a) Glow Curves:

Practically all the samples of both the series were studied at the two warming rates of \(0.9^\circ\) K/sec. and \(0.35^\circ\) K/sec. Glow curves for all the samples studied were plotted. A few curves corresponding to these two different warming rates are shown in Figs. 4.4 and 4.5. The study was carried out from room temperature to about \(500^\circ\)K. The general characteristics of the glow curves observed are summarised below -

(i) The glow curves for all the sample show only one peak at both the warming rates. However, the thermoluminescence intensity was found to be much higher for samples of series A than for those of the series B (Table No.4.3).

(ii) The higher temperature peak oscillates between \(381^\circ\)K and \(431^\circ\) K for samples of series A while between \(379^\circ\)K and \(397^\circ\) K for those of series B. Moreover, there does not seem to be any marked and regular variation of peak temperature with activator concentration.

(iii) The thermoluminescence intensity shows a definite variation with the activator concentration.

(iv) The shape of the glow peaks also appears to depend on the activator concentration.

(v) The low temperature peak is found to be very sharp for fast decaying samples but broad and diffused in the case of slow decaying ones.
Figure 4.12
Calibration Curve for Heater
Current = 2 Aamps
Average Heating Rate 0.35 K/Sec.
Determined

Figure 4.18
Calibration Curve for Heater
Current = 8 Aamps
Average Heating Rate
Determined = 0.9 K/Sec.
(vi) From Figs. 4.4 and 4.5 it is obvious that the form of the glow curve is altered little by changing the warming rate except that the glow curve for higher warming rate shows a sharper maximum than that for the slower warming rate and (as expected from theoretical considerations) the glow curve is slightly displaced as a whole towards lower temperature side when the rate of warming is decreased (9).

(b) Trap Depths:

Trap depths for nearly all the samples were calculated by putting $a = 10^9$ sec$^{-1}$ in the Randall and Wilkins formula i.e. equation (4.11). The calculated values for both the warming rates are given in table Nos. 4.1 and 4.2. There does not appear any dependence of the trap depth on the activator concentration.

(c) Variation of Thermoluminescence Intensity with Activator Concentration:

As shown in Fig. 4.6, addition of cerium to CaS:Zr phosphor reduces the thermoluminescence intensity to a marked extent. The quenching effect becomes more and more pronounced as the concentration of cerium increases. In Fig. 4.6, Curve I represents the thermoluminescence of CaS:Zr (Zr = 0.0036 gm). When $8.05 \times 10^{-6}$ gm of cerium is added to it, the thermoluminescence intensity falls to nearly 50% of its original value (Curve III) and for cerium concentration of
**FIGURE No. 4-6**

VARIATION OF THERMOLUMINESCENCE INTENSITY WITH CERIUM CONCENTRATION. OPTIMUM AMOUNT OF Zr IN EACH SAMPLE = 0.036 g

CONC OF Ce-A23 = 0 gm

A23 = 6.05 x 10^{-6} gm
A19 = 6.44 x 10^{-6} gm
A17 = 2.415 x 10^{-4} gm

**FIGURE No. 4-7**

VARIATION OF THERMOLUMINESCENCE INTENSITY WITH CERIUM CONCENTRATION. OPTIMUM CONCENTRATION OF Zr IN EACH SAMPLE = 0.036 g

CONC OF Ce-A17 = 2.415 x 10^{-4} gm
A15 = 5.635 x 10^{-4} gm
A14 = 6.5 x 10^{-4} gm
A13 = 5.635 x 10^{-3} gm
FIGURE No. 4-b

VARIATION OF THERMOLUMINESCENCE INTENSITY WITH ZIRCONIUM CONCENTRATION OPTIMUM CONC. OF CERIUM

IN EACH SAMPLE 0.0161 gm
Zr CONC. → B12 = 3.6 x 10^{-4} gm.
      B10 = 7.2 x 10^{-4} gm.
      B6 = 1.4 x 10^{-3} gm.
      B16 = 7.2 x 10^{-5} gm.
      B18 = 7.0 x 10^{-5} gm.
      B2 = 0.72 x 10^{-5} gm.

INTENSITY

TEMP. IN °K
1.127 \times 10^{-2} \text{ gm.} the intensity is more or less completely quenched (Curve VII - Fig. 4.7).

However, for the samples of series B, maximum intensity is obtained for a particular value of the zirconium concentration (Ce = 0.0161 gm., Zr = 3.6 \times 10^{-4} \text{ gm.}) as shown by the Curve I of Fig. 4.8 while it falls on both the sides when the concentration is increased or decreased (Curves II and V).

(d) Thermoluminescence after Various Periods of Decay:

Sample A_{14} (Zr = 0.0036 \text{ gm}; Ce = 8.05 \times 10^{-4} \text{ gm}) was excited for four minutes and then allowed to decay for the designated period. The glow curves obtained after different periods of decay are shown in Fig. 4.9. Thermoluminescence has been obtained even after a decay of two hours (Curve IV). The peak of the glow curve shifts continuously to higher temperatures with increasing periods of decay. After two hours of decay the peak shifts approximately to 4650 K. These curves represent the distribution of electrons in traps at various stages of phosphorescence decay.
FIGURE No. 4-9

THERMOLUMINESCENCE AFTER VARIOUS PERIODS OF DECAY

SAMPLE - A14

I DECAY AFTER 30 SECS.

II " " 120 "

III " " 15 MINS.

IV " " 2 HOURS
### TABLE NO. 4.1
Peak Temperature and Trap Depths

Slower Warming rate = \(0.35^\circ\) K/sec.
Higher Warming Rate = \(0.9^\circ\) K/sec.
Room Temperature = \(295^\circ\) K.
\(s = 10^9\) sec\(^{-1}\).

#### SERIES A

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<th>Sample No.</th>
<th>(T_1) in (^\circ)K</th>
<th>(E_1) at slower warming rate in eV</th>
<th>(T_2) in (^\circ)K at higher warming rate</th>
<th>(E_2) Trap Depth in eV</th>
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### TABLE NO. 4.2

Peak Temperature and Trap Depths

- Slower warming rate = 0.35° K/sec.
- Higher warming rate = 0.9° K/sec.
- Room Temperature = 295° K
- \( s = 10^9 \text{ sec}^{-1} \)

#### SERIES B

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<th>( E_1 ) in eV</th>
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<th>( E_2 ) in eV</th>
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<td>0.63</td>
<td>386.0</td>
<td>0.69</td>
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<tr>
<td>B18</td>
<td>342.2</td>
<td>0.61</td>
<td>390.7</td>
<td>0.70</td>
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<tr>
<td>B19</td>
<td>346.0</td>
<td>0.62</td>
<td>379.0</td>
<td>0.68</td>
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</table>
TABLE NO. 4.3

Variation of Thermoluminescence Intensity
With Activator Concentration.

Warming Rate = 0.90° K/sec.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>SERIES - A</th>
<th>Series - B</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Zr = 0.0036</td>
<td>Ce = 0.0151</td>
</tr>
<tr>
<td>Conc. of Ce</td>
<td>gms.</td>
<td>Conc. of Zr</td>
</tr>
<tr>
<td></td>
<td>in gms.</td>
<td>intensity in arb. units.</td>
</tr>
<tr>
<td>A_4</td>
<td>2.415 x10^{-2}</td>
<td>1.0</td>
</tr>
<tr>
<td>A_6</td>
<td>1.127 x10^{-2}</td>
<td>3.0</td>
</tr>
<tr>
<td>A_8</td>
<td>5.635 x10^{-3}</td>
<td>4.5</td>
</tr>
<tr>
<td>A_{14}</td>
<td>8.050 x10^{-4}</td>
<td>10.5</td>
</tr>
<tr>
<td>A_{15}</td>
<td>5.635 x10^{-4}</td>
<td>36.0</td>
</tr>
<tr>
<td>A_{17}</td>
<td>2.415 x10^{-4}</td>
<td>145.0</td>
</tr>
<tr>
<td>A_{18}</td>
<td>8.050 x10^{-5}</td>
<td>455.0</td>
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<tr>
<td>A_{19}</td>
<td>6.440 x10^{-5}</td>
<td>520.0</td>
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<tr>
<td>A_{21}</td>
<td>3.220 x10^{-5}</td>
<td>670.0</td>
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<td>A_{23}</td>
<td>0.000</td>
<td>1200.0</td>
</tr>
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4.7 References:

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<th>Author(s)</th>
<th>Journal/Book Details</th>
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