CHAPTER III

DECAY STUDIES
I: INTRODUCTION

(1) Electron traps and Phosphorescence:

An electron on absorbing exciting radiation is raised to the excited state (into conduction band in the case of photoconducting phosphors). The electron in the excited state may undergo any of the following processes:

(a) Recombine with its original centre giving off fluorescence emission,

(b) Fall into a trap.

If an electron falls into a trap, then either it escapes after a certain lifetime and recombines with an empty centre giving off phosphorescence emission via its excited state or it can be retrapped thus causing delay in phosphorescence emission.

The activation energy $E$ required to liberate the trapped electron, is known as trap depth; this energy may be received by phonon interaction with the surroundings (1-9). If $p$ is the probability of liberating the trapped electrons per second, then

$$ p = \frac{1}{\tau} = se^{-E/kT} \cdots \cdots \cdots \cdots \cdots \cdots (3.1) $$

where $k$ is the Boltzmann constant and $s$ known as 'attempt-to-escape' frequency, is a constant which might vary slowly
with trap depth \( E \) and the temperature.

(ii) **Decay of Luminescence:**

Decay characteristics of the luminescence have received attention since the time of Becquerel (10) and two types of decay processes were recognized.

(a) **Exponential decay attributed to monomolecular mechanism,**

(b) **Hyperbolic decay attributed to bi-molecular mechanism.**

**First Order of Kinetics:** According to this kinetics the number of excited electrons 'n' decreases according to a constant probability law,

\[
\frac{dn}{n} = -pdt \quad \text{............................ (3.2)}
\]

where \( p \) is the transition probability. The above equation on solving reduces to,

\[
n = n_0 \exp (-pt) \quad \text{............................ (3.3)}
\]

Since luminescence intensity \( I \) is given by

\[
I = \frac{dn}{dt} \quad \text{............................ (3.4)}
\]

We have, \( I = I_0 \exp (-pt) \quad \text{............................ (3.5)} \)

here \( I_0 \) is the initial intensity and it is given by,

\[
I_0 = pno \quad \text{............................ (3.6)}
\]
where no denotes initial number of excited electrons.

**Second Order Kinetics**: According to this kinetics the probability of recombination is proportional to the number of available centres, apart from depending on the number of excited electrons. If 'n' electrons be excited leaving 'n' vacant centres, then we have,

\[
\frac{dn}{dt} = -p \frac{n}{n_0} dt
\]

(3.7)

So, \( n \) decreases hyperbolically with time, i.e.

\[
n = \frac{no}{1 + nopt}
\]

(3.8)

The luminescence intensity is given by,

\[
I = \frac{dn}{dt} = pn^2
\]

(3.9)

Hence, \( I = \frac{Io}{(I + at)^2} \)

(3.10)

where \( a = (Io)^{1/2} \), \( Io \) denotes the initial intensity.

Thus, the decay of luminescence becomes more rapid as excitation intensity is increased (11, 12). The intermediate process of trapping has not been considered in the above mentioned kinetics. So, these can only be applied to the case of fluorescence (13-15).

**Kinetics of Phosphorescence in Photoconductive Phosphors**:

The kinetics of phosphorescence can be discussed in the following two ways:
(a) If traps and centres are quite near to each other and their movement either way is very small, then 'each centre and neighbouring trap' can be treated as an independent unit giving a constant recombination with time. First order kinetics then applies and traps of mean life time $\tau$ release electrons according to the relation,

$$ n = n_0 e^{-t/\tau} \quad \text{........................................ (3.11)} $$

In phosphorescence emission of this type the number $dy$ of electrons recombining with a centre is equal to the number freed from traps, i.e.

$$ dy = dn \quad \text{........................................ (3.12)} $$

and resulting luminescence intensity (I) is given by,

$$ I = \frac{dy}{dt} = \frac{n_0}{\tau} e^{-t/\tau} \quad \text{................................. (3.13)} $$

and thus, an exponential decay results.

(b) If traps are spatially independent of centres and electron displacements are large enough for recombination with any of a large number of centres, recombination probability is proportional to the number of empty centres. In this case second order kinetics applies.

If $n_0$ be the total number of traps per c.c., $n$ be the number of filled traps, then the number of empty traps is equal to $(n_0 - n)$. Let $y$ be the number of conduction
electrons and \( N \) be the number of empty centres. Considering all centres normally occupied being emptied by excitation, we have,

\[
N = n + \nu \tag{3.14}
\]

and

\[
\frac{d\nu}{dt} = \frac{n}{\tau} - a_c N\nu - a_t (n_0 - n)\nu \tag{3.15}
\]

and

\[
\frac{dn}{dt} = a_t (n_0 - n)\nu - \frac{n}{\tau} \tag{3.16}
\]

and \( I = a_c N\nu \tag{3.17} \)

where \( a_c \) is effective cross section of centre multiplied by the thermal velocity of the electrons and \( a_t \) is similar product for empty traps.

Let, \( a_c = a_t = a \tag{3.18} \)

Then, Luminescence Intensity

\[
I = a (n + \nu)\nu \tag{3.19}
\]

which decays according to a simple law,

\[
I = \frac{\text{const}}{(t + t_0)^2} \tag{3.20}
\]

where \( t_0 \) is an integration constant.

According to the theory of Adirowitch (16,17), the approximate solution is given by,
\[ I = \frac{\text{const}}{(t + t_0)^p} \]  \hspace{1cm} (3.21)

The decay of a number of phosphors over a considerable range can be described by this type of formula. Various workers have treated similar differential equations but these are more complex in nature (18-22). The interpretation of phosphorescence decay is so complex that the decay cannot be used to draw inference about any specific mechanism involved.

Several workers have attributed the complexity of the mechanism of phosphorescence to a superposition of number of mono-molecular or bi-molecular mechanisms (23-27). The scheme to represent decay by a superposition of exponentials corresponding to different trap depths has been used by many workers (28-31). Randall and Wilkins (32) have found that in \( \text{znS} \) and alkaline earth sulphide phosphors the luminescence process could be explained by monomolecular kinetics but with a distribution of trap depths. They have considered the thermal release from the traps as the main rate determining process and assuming no retrapping, have calculated the form of the decay for several distributions of trap-depths. They found that if the distribution of trap depths is exponential, the decay follows a power law,

\[ I = \frac{\text{const}}{t^{(\alpha kT + 1)}} \]  \hspace{1cm} (3.22)
except for extremely short time of after glow. Using monomolecular theory and assuming absence of retrapping Garlick and Gibson have supported Randall and Wilkins's theory of phosphorescence decay (2).

II: EXPERIMENTAL TECHNIQUE FOR DECAY MEASUREMENTS

The decay of luminescence can be studied by measuring the decrease of residual light sum with time (26) or by determining the after glow intensity as a function of time. However, the choice of a method of measuring decay depends essentially on the actual duration of decay.

(a) Fast Decay (t < 10^{-5} sec.): - An oscilloscope can be used to measure the decay times of the order of 10^{-9} sec. This can be further improved by using co-axial cables suitable for transmission of short pulses and colour shifters to connect ultraviolet luminescence to a spectral region matching the spectral response of the detector (33).

The decay time of this order can also be measured with high accuracy by determining the phase angle between a sinusoidally modulated exciting intensity with a frequency of a megacycle/sec. or more and the luminescent intensity, the decay essentially being exponential. In the method used by Maercks (34) and Sharmann (35), the exciting radiation is modulated by an ultrasonic wave and the phase of emitted
radiation is changed by varying the path length between
the modulator and the phosphor. Relation between the phase
shift and the mean life time is given by,

\[ \tan \varphi = \frac{\omega t}{\tau} \]  

(3.23)

where \( \varphi \) is phase lag and \( \omega \) is the angular frequency of
the modulation. Birk and Little used modulated discharge
source for the measurement of fluorescent decay to replace
the supersonic cell (36). Electrical circuit techniques
are mainly used to determine the phase shift (37, 38).

(b) Slow Decay \((t > 10^{-5}\text{sec.})\) :- The phospho-
roscope invented by Becquerel (39) has been used to measure
decay times, by early workers. In this apparatus the lumines-
cent material is placed between the discs mounted on a
common axis. The discs have sector shaped apertures shifted
with respect to each other. The exciting light enters the
phosphoroscope and falls on the luminescent material through
the aperture in one of the discs. The luminescence is
observed through the aperture in the other disc. Time
interval between excitation and observation can be adjusted
by altering the speed of rotation. A decay time of the order
of \(10^{-4}\text{sec.} \) can be measured precisely when photomultiplier
and oscillograph is used in conjunction with the improved design
of Becquerel phosphoroscope (40).

Randall and Wilkins (41) have used a modified form of
phosphoroscope (42). In this apparatus the phosphor is taken
in an annular form along the periphery of a disc whose speed of rotation is kept constant. The phosphorescence emission is studied at different times by changing the angular position of the detector. Photographic recording method can be used and decay is studied by densitometric measurements. This method also makes possible the study of colour changes during phosphorescence (43).

For detection, a photomultiplier in conjunction with an oscilloscope or a sensitive galvanometer can be used efficiently (44, 45). The light source pulse can be obtained with a sector disc. The vertical component of oscilloscope trace gives the luminescence intensity and horizontal component represents the time scale. The entire decay curve at desired time after excitation is cut-off, can be recorded by adjusting the triggering of the sweep and its speed. Exponential curves of long decays have also been studied with oscilloscope having linear time base with means for taking logarithm of measured signal (46). The exponential decay can also be studied by comparison of the trace produced by the discharge of a condenser in a R.C. circuit with appropriately triggered oscilloscope (47). The two traces are matched by varying the component values and time constant is evaluated from the circuit data. A null method is also possible by mixing the two traces in opposition (48). For the measurement of long period decay exciting light is cut-off mechanically when desired and luminescence intensity is
recorded with a photomultiplier in conjunction with a galvano-
meter or an amplifier with a pen recorder (49, 50).

(iii) Present Method of Decay Measurement:

(a) Decay Apparatus:- The decay studies have been
carried out with the apparatus shown in fig. (3.1). A and B
are two close fitting cylinders. The outer metal cylinder A
is mounted on a heavy base C and the inner cylinder can be
rotated about a vertical axis. Two tubes D and E are hori-
zontally fixed to the openings 0₁ and 0₂ in the outer cylinder.
The exciting source is fitted in tube D and photomultiplier
is screwed to E. Another ebonite tube F is mounted hori-
zontally on a single opening in the inner cylinder. Top
plate H is fixed to the inner cylinder and a cap C is screwed
to H. On C the test tube containing phosphor sample can be
fixed. A vertical hole is drilled in the tube F to hold
the tube in position.

To excite the sample, the opening 0₃ and 0₁ are brought
in line with each other. On releasing a trigger (not shown
in fig.) the inner cylinder rotates through 180° due to the
action of two springs (S₁ and S₂). Now the opening 0₃ comes
in line with the opening 0₂ and consequently faces the photo-
multiplier. A stopper (S) ensures proper alignment of these
two openings. This stopper is padded with rubber to minimise
the displacement of phosphor particles. The sample is closely
packed while filling in the test tube. The displacement of
particles is likely to occur at the top layer which is not
Fig. 3.1

Decay Apparatus

A & B Cylinders; C - Cap; D - Base; E - Top plate; D & E - Tubes; S₁ & S₂ - Springs; F - Ebonite tube; S - stopper; O₁, O₂, and O₃ - Openings;
P - Photomultiplier tube
used for observations. Three sets of observations were
taken for each phosphor sample and were found to be consis-
tent.

(b) **Source of Excitation:** Phosphors were
excited with an 12 watt 6 volts tungsten filament lamp. The
stability of voltage was maintained by floating charge
method. The lamp circuit consisted of a rheostat and an
ammeter to ensure constant current.

(c) **Electron Multiplier Photometer:** The measure-
ment of luminescence intensity was done with the help of a
Ferrand Electron Multiplier photometer. The photometer
consisted of three units - (i) **Detector** (ii) **Power supply**
(iii) **Galvanometer** (Fig.3.2).

(i) **Detector Unit:** The photomultiplier tube used
was I_p21. It has a circular dynode arrangement with Sh-Cs
cathode type 931 A tube of improved sensitivity and reduced
dark current. The maximum spectral response peaks at
4720A (Fig.3.3) which is very near the peak position region
of the phosphors under investigation. The current in the
photomultiplier tube is proportional to the light intensity
for currents upto 150 microamperes and current amplification
is 2 x 10^6. The aperture of the detector unit is controlled
by a sleeve shutter operated by a knob at the top of the
detector housing.
Fig. 3.2

Ferrand Electron Photomultiplier

1. a. Detector
   b. Power Supply
   c. Rubicon Galvanometer
Fig. 3.3
Spectral Response Curve
(ii) **Power Supply Control:** The circuit diagram of this unit is shown in fig. 3.4. This consists of 30 dry batteries of 30 volts each. For balancing dark current a battery of 1.5 volts is also provided. It has a sensitivity control device.

(iii) **Galvanometer:** Rubicen galvanometer was used which has a resistance of 4800 ohms and sensitivity 0.0013 $\mu$amp/mm. It is provided with a built in lamp and scale arrangement. Its connecting cable has a 6 volt in-line transformer. The light spot can be shifted to any desired position with the help of a coarse and fine adjusting device.

(d) **Setting up of the Photomultiplier:** The three units were connected as shown in fig. 3.2. The phototube was adjusted for peak response. To compensate for dark current of the phototube, the shutter was completely closed and sensitivity control knobs were turned completely clockwise. The zero button was pressed and galvanometer deflection was noted. The zero button was then released and dark control knobs was adjusted so as to give the same deflection. The light spot was brought to zero position with the help of the coarse and fine adjusters. The time required to excite the phosphor samples for acquiring steady state of luminescence was determined and it was found to be about two minutes. So, for experimental studies the phosphors were excited for four minutes. As it was not possible to study
Fig. 3.4

Power Supply Circuit Arrangement
all the phosphors in the same sensitivity range, two ranges were chosen. Phosphor samples up to number 12 were studied with full sensitivity and the rest were studied with one tenth of the sensitivity. The response of the phototube being linear, the galvanometer reading was taken as measure of the intensity. The intensity was recorded as a function of time till it reduced to a low value.

(e) Measurement of the Decay of Red Phosphorescence Band:

Phosphorescence decay of the very weak red band was measured with Ferrand Electron Photomultiplier unit. The I p21 photomultiplier tube was coupled with the plate holder of Steinheil spectrograph (described in chapter V) ensuring that the arrangement was light tight. The luminescence intensity was measured by the usual method of galvanometric deflection as a function of time.

IV: RESULTS

(a) Theory: The probability $p$ of an electron escaping from a trap is given by,

$$
p = \frac{a}{s} \exp \left( \frac{-E}{kT} \right) \quad \text{................. (3.1)}
$$

$s$ being frequency factor - the attempt-to-escape frequency $s$ is generally assumed to be independent of temperature and it is interpreted as the number of times per second the crystal vibrations attempt to eject electrons from the
trap multiplied by the probability of transition from the
trap to the conduction band. Further assumptions have
been made to make practical use of the above equation.
They include that the retrapping is negligible, that radi-
ative rather than non-radiative transitions take place once
an electron is freed from the trap and that the trap depth
is independent of temperature. Then for a single trap depth
the decay of intensity is given by,

$$I = I_0 \exp (-pt) \quad \ldots \quad (3.24)$$

where

$$I_0 = n_0 \quad \ldots \quad \ldots \quad (3.25)$$

no being the number of trapped electrons at t = 0. If
there exists a distribution of trap depths, the decay
pattern will be a superposition of all intensities, each
of which decays exponentially with time. This results in
a hyperbolic decay.

(b) Decay Curves:— The measurements made at
room temperature (301K°) of the after-glow intensity as a
function as a function of time were plotted. Some repre-
sentative graphs are shown in fig. 3.5 The intensity in
arbitrary units are shown as ordinate while time in seconds
as abscissa. Earlier workers in this laboratory (51-54)
also reported the curves of the same nature for the alkaline
earth sulphide phosphors.
To investigate decay characteristics, the logarithm of afterglow intensity was plotted against time. Nonlinear curves were obtained showing that the decay did not follow an exponential law. The log of intensity was plotted against the log of time and the relation obtained was approximately linear which suggests a hyperbolic decay. Some representative graphs are shown in fig. 3.6. Co-relation coefficients for all the phosphors were also calculated to estimate the degree to which the relation between log I and log t is linear.

(c) Co-relation Co-efficient:– For the investigation of the relationship between variables x and y, based upon a set of n pairs of measurements, usually a graph is plotted. From this scatter diagram, the pronounced relationship between the two can be interpreted. When the relationship is approximately linear, the degree of linearity is estimated from the correlation coefficient given by:

$$r = \frac{n \Sigma x y - \Sigma x \Sigma y}{\sqrt{\{n \Sigma x^2 - (\Sigma x)^2\} \{n \Sigma y^2 - (\Sigma y)^2\}}}$$

The magnitude of r determines the strength of the relationship, whereas the sign tells whether tends to increase or decrease with x. The constant r has two properties:

(i) the value of r must satisfy the inequality $-1 \leq r \leq +1$

and
(ii) that the value of \( r \) will be equal to \( \pm 1 \) if the scatter points lie on a straight line.

The value of \( r \) computed for the phosphors is very nearly equal to one with a negative sign indicating a linear relationship between \( \log I \) and \( \log t \) and also that \( \log I \) decreases as \( \log t \) increases.

(d) **Calculation of Decay Constant** - The decay characteristics in the present case are hyperbolic and obey an equation of the form,

\[
I = I_0 t^b \quad \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots (3.27)
\]

where power 'b' is referred to as decay constant. It is evaluated by the method of least squares.

If the value of a certain variable to be estimated is given by \( \gamma \) and corresponding curve value is \( \gamma' \), then the error of estimation is given by \( (\gamma' - \gamma) \). For obtaining a good estimate of the parameter it is necessary that the sum of squares of this error shall be minimum. This minimisation determines the best fitting curve in the sense of least squares. The method of least squares for non-polynomial regression often gives rise to normal equations which are difficult to solve requiring tedious numerical methods. There are numerous other methods of fitting a curve to a set of points that can be employed, when the method of least squares gives rise to computational difficulties. The method followed here is to introduce new variables which are
functions of the old variables so as to obtain a more tractable relationship. Thus, taking the logarithm of equation 3.27, we get,

\[ \log I = \log I_0 + b \log t \]  \hspace{1cm} (3.28)

then, let \( y = \log I \), \( x = \log t \) and \( \log I_0 = c \)

The above equation reduces to,

\[ y = bx + c \]  \hspace{1cm} (3.29)

The problem is reduced to fitting a straight line to a set of points in the xy plane and this can be easily solved by the method of least squares.

'b' and 'c' can now be computed from the following relationships:

\[ b = \frac{\sum x \sum y - n \sum xy}{(\sum x)^2 - n \sum x^2} \]  \hspace{1cm} (3.30)

and \[ c = \frac{\sum y \sum x^2 - \sum x \sum xy}{n \sum x^2 - (\sum x)^2} \]  \hspace{1cm} (3.31)

The estimate of 'b' and 'c' obtained by this method differ from those obtained by solving the original equations but these differences are usually small.

The value of 'b' estimated for all the decay curves in the time interval from five seconds to the time when the intensity is negligibly small are given in table No. 3.1, along with the co-relation co-efficients. The value of 'b
comes out to be negative and hence the decay equation becomes,

\[ I = I_0 t^{-b} \]

\[ \text{(3.32)} \]

(e) 'Peeling-off' of Decay Curves:— Randall and Wilkins consider the hyperbolic decay as a result of superposition of exponentials corresponding to different trap-depthes. Following the method of Bube and others \(^{59,56}\) it has been possible to break up the decay curves into exponentials. Trap depths corresponding to these exponentials were calculated from the slopes of the straight lines on the semi log plot. Representative graphs are shown in fig. No. 3.7. The values of trap depths calculated are given in table No. 3.2.

(f) Decay of Red Phosphorescence:— From the measurements taken for the decay of the red band, it was possible to draw graph between luminescence intensity and time on semi log plot for sample Nos. 10, 11, 12 which is shown in fig. No. 3.8. The curve being a straight line suggests that the decay of red phosphorescence is exponential. The intensity of red phosphorescence becomes negligibly low after 15 seconds. No red phosphorescence emission was detected for samples which have a lower percentage of Mn-activator than sample No. 12. The intensity of red phosphorescence emission is given for various samples in
terms of galvanometric deflection in table No. 3.8. The red phosphorescence decays in a short time. Similar results have been obtained by Fonda (55) for the phosphor systems activated by Mn.
<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Mn Activator CaS. (in %)</th>
<th>Decay constant</th>
<th>Correlation coefficient</th>
</tr>
</thead>
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<tr>
<td>1</td>
<td>15.79</td>
<td>0.51</td>
<td>0.99</td>
</tr>
<tr>
<td>2</td>
<td>13.24</td>
<td>0.49</td>
<td>0.99</td>
</tr>
<tr>
<td>3</td>
<td>11.44</td>
<td>0.52</td>
<td>0.99</td>
</tr>
<tr>
<td>4</td>
<td>8.888</td>
<td>0.52</td>
<td>0.99</td>
</tr>
<tr>
<td>5</td>
<td>7.874</td>
<td>0.62</td>
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</tr>
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<td>11</td>
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<td>0.54</td>
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</tr>
<tr>
<td>12</td>
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<td>0.99</td>
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<td>0.98</td>
</tr>
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<td>0.99</td>
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<tr>
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<td>0.98</td>
</tr>
<tr>
<td>31</td>
<td>511</td>
<td>0.71</td>
<td>0.98</td>
</tr>
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<td>Sample No.</td>
<td>Mn Activator w.r.t. CaS (in %)</td>
<td>Trap Depths (eV)</td>
<td></td>
</tr>
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<td>-----------</td>
<td>-------------------------------</td>
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</tr>
<tr>
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<td>I Exponential</td>
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<td></td>
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<td>III Exponential</td>
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