CHAPTER IV

EXPERIMENTAL DETAILS

The measurement of thermoluminescence (TL) intensity is amenable to a simple and relatively inexpensive instrumental set-up. The requirement for an experimental study of thermoluminescence of a phosphor can be divided mainly into four steps i) Excitation ii) Heating iii) Detection and iv) Recording with display. The TL - characteristics of an irradiated phosphor can be recorded in two ways a) TL intensity as a function of wavelength and b) TL intensity as a function of temperature. In the first case the emission spectrum of the TL material can be obtained by maintaining the temperature of the irradiated phosphor, a little below the reference TL glow peak temperature, and scanning the emission by means of monochromator. The recording of TL intensity as a function of temperature is important for the measurements of radiation dose and other TL applications. A simple experimental arrangement to measure thermoluminescence consists of a light - tight box in which a small sample of TL material facing the window of a photomultiplier tube is heated. The emitted thermoluminescence (TL) is collected by the photomultiplier. The output signal of the photomultiplier being proportional to the TL intensity, when plotted as a function of temperature, gives the TL glow curve of the irradiated material. This curve on analysing can

provide information about the dose received by the material, during irradiation and also about the other TL kinetics.

A. Preparation of Samples :

The NaCl:Ca samples used in the present experiments are prepared by using Sodium Chloride powder as the base material, wherein Calcium is introduced in the form of a Calcium salt. The 'Analar' grade Sodium Chloride obtained from BDH laboratory, is certified to have a purity of 99.9 %. The manufacturer's data indicates the major fraction of the impurities unavoidably present in the base material to be Potassium, Calcium and Magnesium. Extreme care has been taken for the cleanliness of the glassware used while preparing the specimens. All surfaces, which were to come in contact with the phosphor were cleaned with aquaregia. After cleaning, the surfaces are kept in contact with boiling distilled water for two hours and then dried thoroughly in an oven.

The dopent used in the present work is Calcium in the form of its salt viz : Calcium Chloride. It has been supplied by E. Merck Ag Darmstadt, Germany, and

is certified to have the purity of 99.9 %. The introduction of this impurity into the base material is accomplished by the usual method of recrystallization from the aqueous solution. The exact weight of the impurity, as determined by the molar fraction calculation, is dissolved in double distilled demineralized water. A weighed quantity of Sodium Chloride is then added to this solution. The solution is heated slowly on a hot plate until the excess water is completely driven out. Micro-crystals, are collected, dried at 40°C, powdered and mixed homogeneously. The micro-crystalline powder of the mesh size $< 80 > 120$ is then collected. The specimens collected in this way are designated "as - obtained from solution". Specimens with three different concentrations of Calcium (10^{-2} mf , 10^{-3} mf, 10^{-4} mf) have been prepared by this method.

B. Thermal Annealing Treatments :

NaCl:Ca specimens are annealed in a muffle furnace in batches at 550 and 750°C temperatures. A silica boat containing the NaCl:Ca specimen is kept in the furnace for two hours when a stable temperature of 550°C is attained. On completion of the annealing time, the specimen is quenched to room temperature by withdrawing the boat on to a block of aluminium and by a blast of

cold air. The second batch of the specimens is annealed at 750°C for two hours and quenched to room temperature in the similar manner. Such specimens are designated "annealed and quenched." The NaCl:Ca (10^{-3} mf) specimen quenched from 750°C designated as NaCl:Ca(T).

C. Mechanical Treatment :

A suitable quantity of the specimen, either as - received from solution or after thermal treatment is compressed into the form of a pellet by means of a stainless steel press. The pellet size is 1 cm in diameter and 0.1 cm in thickness. The pressure under which the specimens are compressed measures 5×10^3 kg/cm^2.

D. Sources of Irradiation :

i) UV - Irradiation Source :

The specimens are irradiated by the standard UV - wavelength of 253.7 nm from the Jarrel - Ash - Model - Mercury Lamp, supplied by Jarrel - Ash Co.U.S.A. The lamp is kept at a distance of 3.0 cm from the specimen. The output energy of 253.7 nm at 3.0 cm distance is around 100 $Jm^{-2}s^{-2}$. No external filter is used during irradiation.

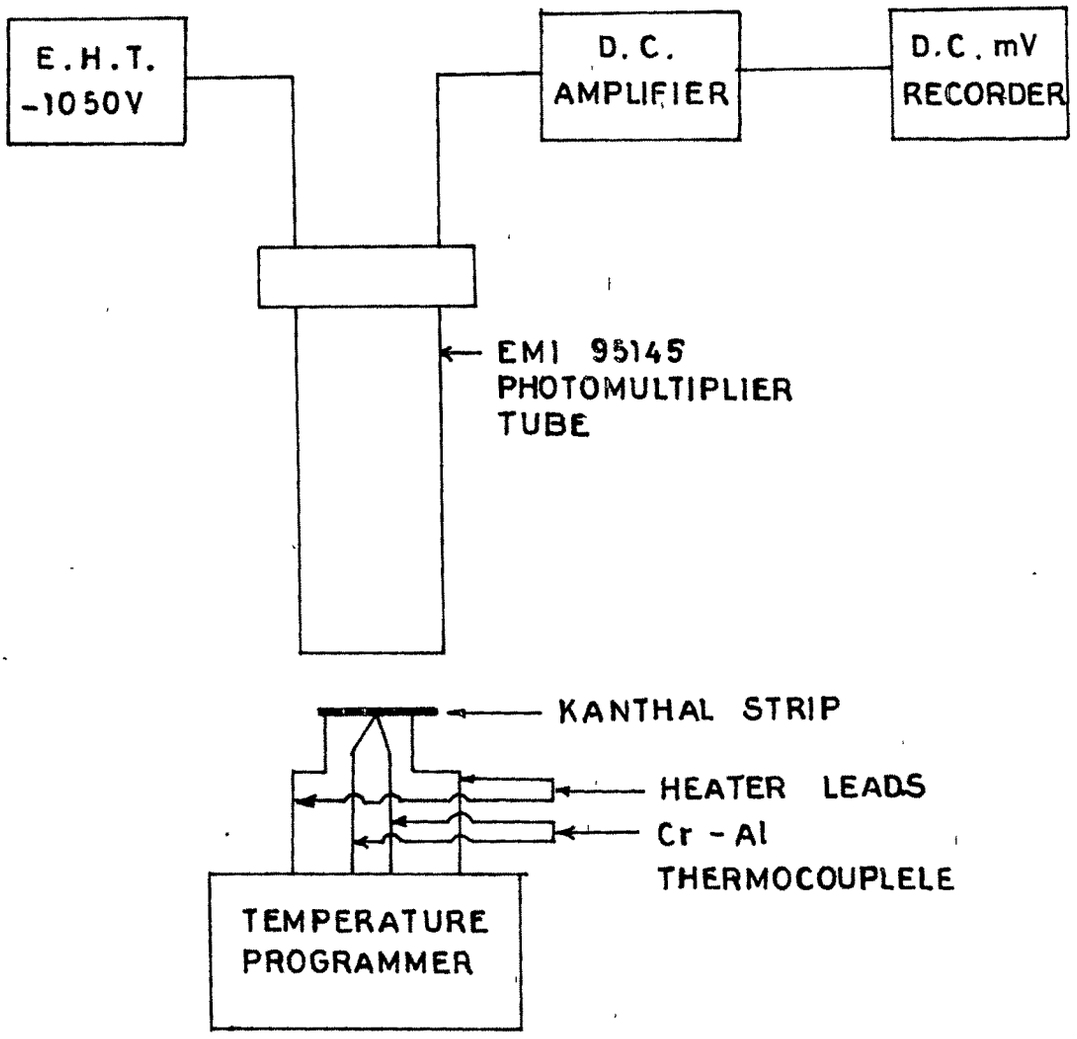
ii) Gamma - Irradiation Source :

For the gamma irradiation of the specimens ^{226}Ra - 9 MCi source having the dose rate of 6.8×10^2 rad min^{-1} (6.8 Gy min^{-1}) has been used.

E. TL Glow Curve Reader :

It consists of a photomultiplier tube, a high voltage unit, a dC amplifier, a temperature programmer and a strip chart recorder as shown in the block diagram (fig. IV-1). The specimen is spread uniformly on a metallic strip of Kanthal (an alloy having, Fe-72% ; Cr - 23% ; Al - 3% and Co - 2 %). The strip has the size of 30 x 5.5 x 0.25 mm^3 with a circular depression of 5 mm diameter and 0.5 mm depth at its centre. A cromel-alumel thermocouple records the temperature of the specimen. A linear relationship between the rise in temperature and time is maintained by the uniform heating rate. In the present work all the glow curves are recorded with specimen 20 mg in weight using a linear heating rate of 180°C min^{-1}.

A photomultiplier tube EMI 9514 S, with S_{11} response, is housed in a light-tight brass cylinder. A high voltage of 1050 volts is supplied to it. When the kanthal strip with the irradiated specimen is placed



BLOCK DIAGRAM OF PROGRAMMED LINEAR HEATING TYPE TL GLOVE CURVE RECORDER.

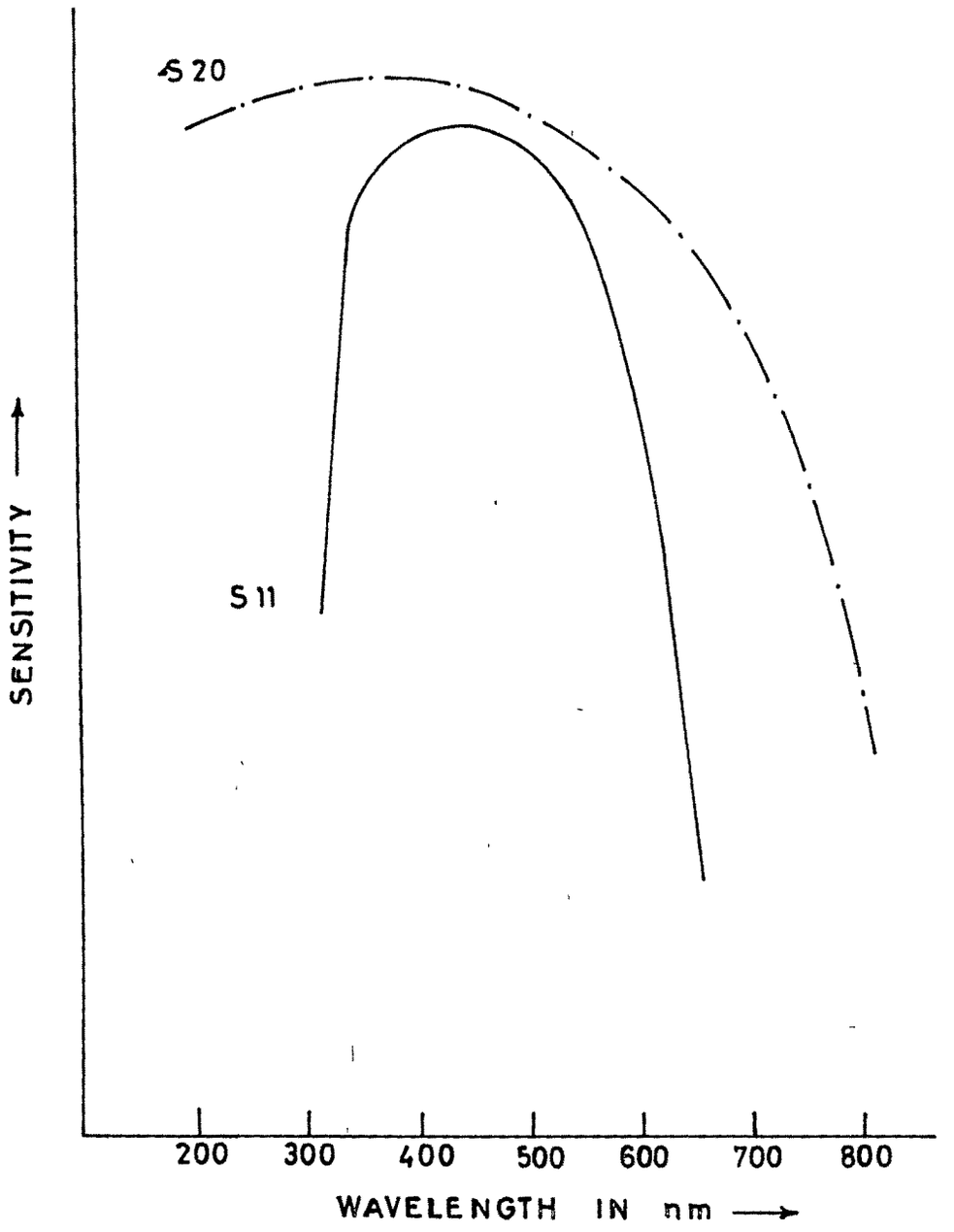
FIGURE : IV-1

infront of the photomultiplier window, the light emitted by the specimen during heating is recorded through the photomultiplier tube and a dc amplifier by a strip chart recorder. The strip chart recorder has a speed of 2 inch/min. The spectral response of the above photomultiplier is given in fig. IV-2.

F. TL - emission Spectra Recording System :

The TL-emission spectral measurements are carried out using a 0.25 meter Jarrel-Ash-Ebbert monochromator between the photomultiplier and the specimen on the heater strip (40 x 6 x 0.25 mm^3 kanthal). The 0.25 meter grating monochromator has an aperature ratio f/3.6 and a resolution of 0.5 mm at 313.1 nm. Two gratings blazed at 300 and 600 nm separately with 1180 grooves/mm over an area of 64 x 64 mm^2 are employed in the monochromator which gives a linear dispersion of 3 nm/mm. 2mm wide slits are used at entrance and exit ends of the monochromator. 300 nm blazed grating is used for recording the spectrum in the wavelength range 200 - 500 nm and for wavelengths higher than 500 nm, 600 nm blazed grating is used.

About 20 mg of the specimen is spread uniformly on the kanthal strip placed infront of the entrance



SPECTRAL RESPONSE CURVES OF PM TUBES USED FOR THE RECORDING OF TL-GLOW CURVES (S11) AND TL-EMISSION SPECTRA (S20).

FIGURE : IV-2

slit of the monochromator. Specimen is heated by passing a constant dc current through the kanthal strip, for obtaining the constant temperature of 80, 130 and 200°C. For detecting the TL-light, EMI 9558 QB photomultiplier tube having S20 response (fig. IV-2) is mounted at the exit slit of the monochromator. The output of the photomultiplier is fed to the recorder through a dc amplifier, to record the TL-emission spectra. The recorder and heater supply are operated simultaneously by a master switch.