CHAPTER - IV

EXPERIMENTAL DETAILS

Modern thermoluminescence recording equipment can vary from the very simple to the extremely sophisticated. The nucleus of all the various designs is a light detection system, a sample heater and a temperature control unit but the designs of each of these components are many and varied. However, the requirements for an experimental study of a phosphor can be divided mainly into four steps: (i) Excitation, (ii) Heating, (iii) Detection, and (iv) Recording and display. The TL intensity of an irradiated phosphor material can be recorded in two ways: (i) TL intensity as a function of wavelength and (ii) Intensity vs temperature. In the first case the emission spectrum of the TL dosimetric peak can be obtained by maintaining the temperature of the TLD material a little below the reference peak-temperature and scanning the emission by means of a monochromator.

The study of TL intensity vs temperature is important for radiation dose measurements. A simple experimental arrangement to measure TL will consist of a light-tight box in which a small sample of the TLD material, facing the window of photomultiplier (PM) tube is heated. The output signal of the PM tube, which is 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 about other TL kinetics.

A Sample Preparation

The NaF:K samples used in the present investigation were prepared by using NaF powder as a host material, in which potassium was introduced in the form of its salt (KF). The base material used was "Guaranteed Reagent" sodium fluoride supplied by LOBA-Chemic Indoaustranal Co., Bombay, India was certified to have a purity of 99%. The manufacturer's data indicates the major fraction of the impurities unavoidably present in the base material to be insoluble matter 0.005%, free acid (HF) 0.04%, chloride (Cl) 0.002%, fluorosilicate (SiF_6) 0.12%, phosphate (PO4) 0.0005%, sulphate (SO_4) 0.005%, iron (Fe) 0.002%, Heavy metals (pb) 0.002%, potassium (K) 0.01%.

Extreme care was taken regarding cleanliness and purity while preparing and handling the samples. All surfaces, which were to come in contact with the phosphor were cleaned with hydrochloric and nitric acid. After cleaning, the surfaces were kept in contact with boiling water for two hours, and then dried thoroughly in an oven. The impurity used in the present work is potassium in the form of its salt vis:potassium fluoride. The introduction of this impurity into the base material is accomplished by the usual method of recrystallization from aqueous solution. The exact weight of the impurity, as determined by the part per million (ppm) calculation, is dissolved in double distilled demineralized water. A weighted quantity of sodium fluoride 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 specimen collected in this way are designated as "as-obtained from solution". Specimens with four different impurity concentrations (namely 200, 500, 1000 and 2000 ppm) have been prepared by this method.

B Thermal Annealing Treatments

Thermal annealing of "as-obtained" pure and potassium doped sodium fluoride specimens was done in muffle furnace in batches at 200, 400 and 600°C. A silica boats containing the "as-obtained" samples were kept in the furnace fixed at 200°C for two hours. On completion of the annealing time, the specimens were rapidly quenched to room temperature by withdrawing the boats on to a block of aluminium, exposing them to a blast of air. The second and third batch of specimens were annealed at 400 and 600°C respectively for two hours and rapidly quenched to room temperature in a similar manner. Such specimens are designated as annealed and quenched or pre-heat treated or thermally treated specimens. The NaF:K (1000 ppm) specimens quenched from 400°C are designated as NaF:K(T) and the NaF specimens quenched from 400°C are designated as NaF(T).

C Radiation Source

The source used in the present work for beta irradiation of the specimen was ^{90}Sr 50 milli-curie source having the dose rate of 700 rad min^{-1}.

D TL Glow Curve Reader

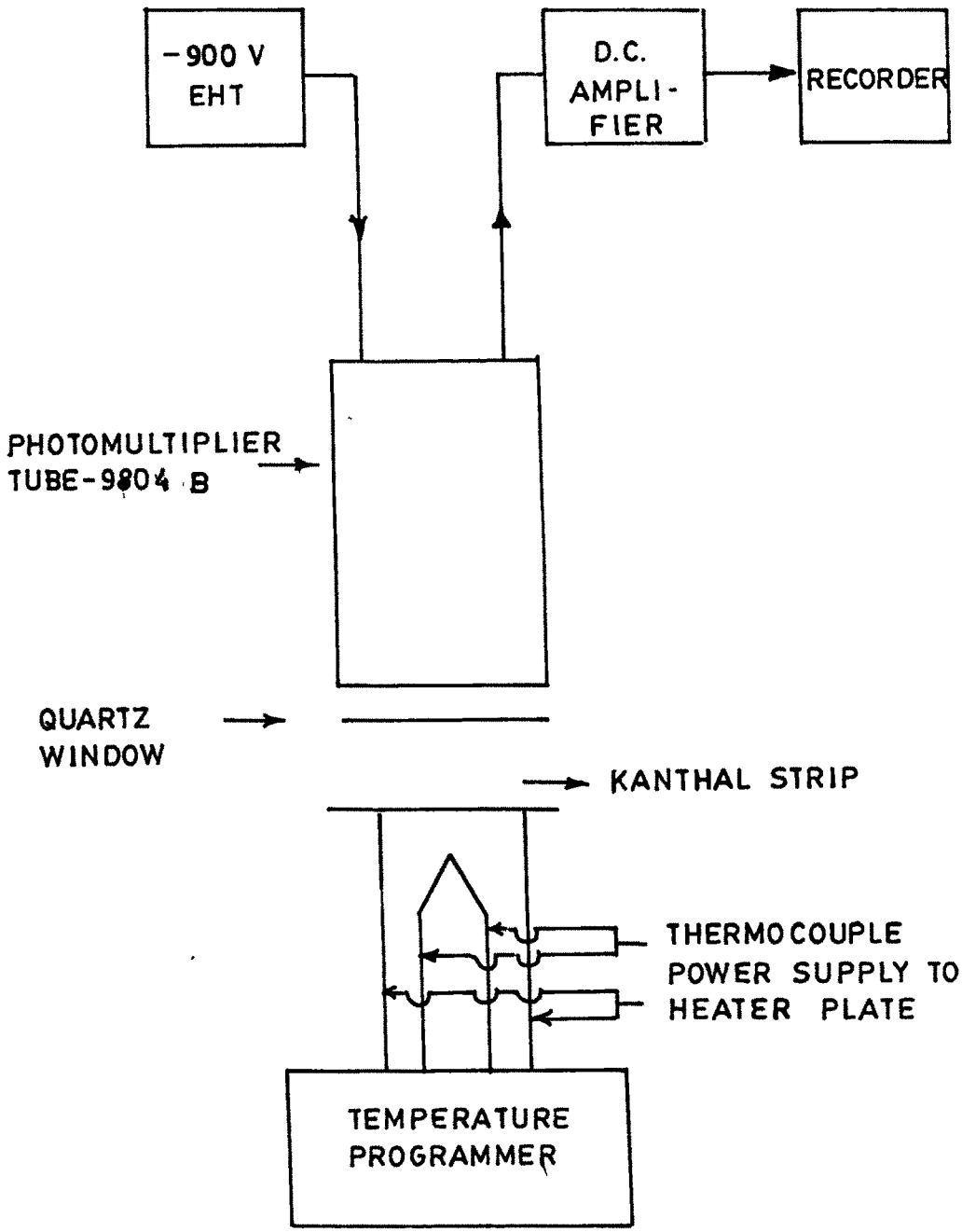

FIG. IV-1 BLOCK DIAGRAM OF TL SET-UP

The TL glow curve recorder system consists of a photomultiplier tube, a high voltage DC supply unit, a DC amplifier, a temperature programmer and X-Y strip chart recorder. Block diagram of the unit is shown in Fig.IV-1.

The sample is spread uniformly on a metallic (kanthal) strip which is an alloy of Fe 72%, Cr 23%, Al 3%, Co 2%, which serves as a heater, the size of the kanthal strip is 30 x 15 x 0.25 mm^3 with a circular depression of 5 mm diameter and 1 mm depth at its centre. A cromel-alumel thermocouple is spot welded at the centre on the lower side of the kanthal strip. Because of the small size of the heater, high temperature can be attained with low power input and cooling is very fast after the power is switched off. A linear relationship between the rise in temperature and time is maintained by the uniform heating rate, for this purpose, model 487 temperature programmer provides all the necessary options for TL studies. Heating rate can be continuously varied from 10 to 1000°C min^{-1}.

In the present work, all the glow curves are recorded with specimen 5 mg in weight using a linear heating rate of 400°C min^{-1}. A photomultiplier tube EMI 9804B is housed in a light-tight brass cylinder. A high voltage of 900 volts is supplied to it. When the kanthal strip with irradiated specimen is placed in front of PMT window, the light emitted by the specimen during heating is recorded through the PMT and a DC amplifier by a strip chart recorder with speed of 4 inch $min.^{-1}$