CHAPTER - 3

INSTRUMENTATION

3.1 THERMOLUMINESCENCE GLOW CURVE READER

TL glow curve reader system used in the present investigations consisted of two photomultiplier (PM) tubes (EMI 9514 S), a high voltage unit, a dc amplifier, temperature programmer and a double pen recorder. Based on the principle of double beam system with one PM tube described by Kolberg and Prydz (1973), in the present arrangement (Figure 3.1) two similar PM tubes were used. This eliminates the necessity to use reflectors and rotating sector mirror used by them. The noise and gain conditions of the PM tubes were verified by taking runs with a standard light source. To ensure minimum errors within experimental limits, TL runs were taken with a standard TL source (CaSO₄ dosimetry pellets irradiated to gamma ray dosage of 10 mR) both as a reference and the sample. Further checking was done by once interchanging the reference and the sample and second time by physically interchanging the position of PM tubes. The parameters viz. HV to PM tubes and the gain of pre-amplifiers, were adjusted to get an absolute straight line on the recorder for two identical samples, one used as the reference and the other as the sample.

The heating unit consists of a kanthal metal plate, secured electrically at its ends, with two identical grooves to hold the sample in one and the reference in the other.
FIG. 3.1 BLOCK DIAGRAM SHOWING THE DOUBLE PM DETECTION TECHNIQUE
Plate showing the thermoluminescence collector and recorder in operation.

Plate showing the thermoluminescence collector while setting the sample under study.
A cr-Al thermocouple junction is spot-welded to the centre of the heating plate. Power to the heater strip was fed from the temperature programmer designed to heat the sample at any required rate ranging from 0.5°C/Sec to 50°C/Sec. The TL glow intensity was simultaneously recorded along with the temperature variation by a double pen recorder (type 153 Electronik Duplex, Honeywell).

3.1.1 Recording the Initial Part of the Glow Peaks

Initial portion of the glow peaks was recorded to determine the activation energy by initial rise method as suggested by Garlick and Gibson (1948). As required for such a study there should be no contribution from the neighbouring peaks and population of charge carriers in a trap should remain unaffected. Since only the initial portion of the glow peak is needed for this purpose, therefore, it must be assured that the lower temperature peaks are completely removed.

To achieve this the sample was first heated at the rate of 25°C/min up to the temperature 15°C below the peak temperature so that even if a small part of any lower temperature peak was present, that would be removed completely. The other condition is taken care of by recording the glow peak up to the temperature when the emitted TL light is 2% of the total peak intensity. Such a technique produces negligible affect on the trap population.

3.2 TL EMISSION SPECTRA RECORDING SYSTEM

The emission spectra of fluorites studied in the present investigations were made by monochromator (Model 82-410).
The monochromator in this model has two gratings blazed at 300 mm and 600 mm mounted back to back and either can be selected by turning a knob. The gratings are ruled with 1180 lines/mm over an area of 64 x 64 mm² and give a dispersion of 3.3 nm/mm, while 2 mm wide slits are used at both entrance and exit ends of the monochromator. A 300 nm blazed grating is used for recording the spectrum in the wavelength range 200 nm - 500 nm and for wavelength higher than 500 nm a 600 nm blazed grating is used. About 20 mg of the sample under study is spread uniformly on kanthal strip placed in front of the entrance slit of the monochromator. The kanthal strip used in the present set up had the dimensions 40 x 6 x 0.25 mm³ with a 15 x 5 mm² of central area embossed to 0.50 mm depth and was secured tightly between the brass electrodes. The sample was heated by passing a constant DC current through the kanthal strip.

For detecting the TL light, EMI 9558 QB photomultiplier tube was mounted at the exit slit of the monochromator. Output of the photomultiplier tube was fed through a DC amplifier to a General Electric 5 mv strip chart recorder. The grating of the monochromator was rotated using a small motor of speed 4 RPM. Wavelength of the light at the exit slit varied at the rate of 100 nm/min and the chart speed used for recording the spectra was 5 cm/min. A block-diagram of the experimental set-up is shown in Figures 3.2.

3.3 RADIATION SOURCE

In the present investigations the samples under study were irradiated using Co-60 gamma cel AECL-220 having uniform
FIG. 3.2 - BLOCK DIAGRAM OF TL EMISSION SPECTRA RECORDER
Plate showing the thermoluminescence spectra recording set-up.
irradiation rate of \(8.5 \times 10^4\) rads per minute at the irradiation position.

The source has a sliding arrangement with a vertical drawer, which has a sample cavity on it. The cavity in the moving aluminium drawer is used for keeping the sample. The drawer can be operated by a motor and pulley system. The whole set-up is shown in Figure 3.3.

3.4 FURNACE OF ANNUSALING

Thermal annealing of the sample was done using a 'Therelek' muffle furnace with sample holding space of \((17 \times 8 \times 8)\) cm\(^3\). The temperature is adjusted using a temperature controller which supplies the required current to the furnace. This furnace was used to study the thermal effect on zircon samples.

3.5 ALPHA COUNTING SET UP

Alpha counting is found very useful in confirming the radioactivity and also in estimating uranium and thorium concentration of the samples. In the present investigations alpha activity was measured by using an alpha probe - SP 647 A manufactured by Electronics Corporation of India, utilizing a scintillation detector. It consists of a photomultiplier attached to an EHT unit and a count rate meter (Figure 3.4). The sample was placed over a ZnS screen which is mounted on a perspex ring. The sample with the scintillating screen facing the PM is placed at a distance of 0.2 mm from the PM tube face. All the samples were separately read for 168 hours at a stretch and the counting rate for each calculated.
FIG 3-3 Co-60 GAMMA IRRADIATION FACILITY (gamma cell)

1) Co-60 GAMMA SOURCE  2) Pb-CASTLE  3) MOVING SAMPLE-CARRIER  4) PROVISION FOR ELECTRIC LEADS  5) PULLEY ARRANGEMENTS
FIG 3.4 ALPHA COUNTER
PM - PHOTOMULTIPLIER
PA - PRE-AMPLIFIER
H - PM HOUSING
S - SAMPLE
Plates showing various aspects of alpha counting set-ups in operation.
3.6 Differential Thermal Analysis (DTA) Setup

The DTA apparatus consisted of three thermocouples, one to measure the temperature of the furnace, and the other two connected in opposition, to measure the difference between furnace and sample temperatures by the use of a millivoltmeter calibrated for temperature as well as a galvanometer of high sensitivity (Figure 3.5). Sample was placed on one of the holes of a triple sample holder and the function point of the first thermocouple was embedded in it. The function points of thermocouples 2 and 3 measuring the temperature of the furnace were surrounded by inert material (Alumina) that did not undergo any change under the effect of heat, but of a thermal conductivity similar to that of the sample. The sample was heated in an electric oven with a heating rate of 10°C per minute.

3.7 Density Measurements

The values of the density for different zircon samples used was determined by using two quartz pycnometers of 50 ml capacity. Radistilled water free from air bubbles was used and precautions were taken to reduce the evaporation to a minimum. Air bubbles adhering to the samples were completely removed by heating the pycnometer, containing the sample and 3/4 of its volume of water, to about 50°C under diminished pressure. Weighing was done with the help of an electrical balance, (E.Mettler, Zurich, Switzerland) which measures correctly up to four places of decimal. Corrections were made for the
FIG 3.5 INSTRUMENT FOR RECORDING DTA CURVES
differences in the displacement of air by the volume of the contents of the pycnometers. Three different determinations were made on different portions of each sample and the difference did not exceed 0.0009. The results tabulated may be considered correct to the nearest unit in the third decimal place.

3.8 **X-RAY DIFRACTOMETRY**

For every sand studied under the diffractometer, a representative sample is taken and ground in an agate mortar. The material is ground and sieved through a B.S.300 mesh sieve (opening 53 u), until all the sample has passed through the sieve.

The sample holder is a rectangular sheet of aluminium 1½ in. square and 1/16 in. thick. It has rectangular opening 7/12 in. x 19/24 in. Over this opening on one side of the plate, a clean glass slide is placed and bound firmly over the opening with cellotape. On its reverse, into the cavity formed, an excess of the sample powder is placed. The powder is tamped and compressed with another glass slide and levelled off. The surface of the powder is absolutely plain and the holder is then attached to the machine.

A Philips Diffractometer was used. The alignment of the instrument was checked by a silicon standard. The instrumental conditions are:

Cu Kα radiation, Ni filter, 30 KV, 24 mA. The goniometer was run at 4° per minute (2θ drive).
3.9 PREPARATION OF POLISHED SECTIONS FOR MINERAGRAPHY

A steel plate 1 in. thick, with a number of mould cavities 1\frac{1}{4} in. diameter and open at both ends, is given an application of a parting agent e.g. grease on the walls and laid out flat on a glass plate. A small quantity of epoxy resin is prepared and mixed thoroughly with the sand sample and poured into a mould to about one fourth full. The grains tend to settle down and after a few minutes the rest of the mould is topped up with only epoxy resin and allowed to cure. This was left overnight prior to stripping. This procedure is adopted for all the samples.

The mounted samples after stripping are ground flat prior to polishing. This is done by grinding with a 320 grit silicon carbide paper. These were then put over a 600 grit silicon carbide paper followed with 2/0 and 3/0 emery papers. The samples are then worked with 6 \mu and 3 \mu diamond pastes over a polishing wheel.

After every grinding or polishing step, the specimens were thoroughly cleaned with soap and water. Finally the samples were polished in a vibratory polisher with metallurgical media (0.05 - 0.1 Al2O3). All the samples were then observed under the ore microscope.

3.10 SCANNING ELECTRON MICROSCOPY

The Scanning Electron microscope studies were conducted with a J.S.M. - 2 microscope manufactured by the Japan Electron Optics Laboratory Co. Ltd.
In the scanning electron microscope, an electron beam emitted from an electron gun is focussed into a fine probe on a specimen surface by a condenser lens and an objective lens. The appropriate signal is converted by the detector into an electrical signal which is amplified and used to modulate the brightness of the display cathode ray tubes. The scanning circuit through the magnification unit operates the scanning coils so that the probe scans the specimen surface in a raster like a television. The scanning circuit also produces a synchronised scanning of the display tubes so that the signal from the specimen is transferred to the display giving a picture on the cathode ray tubes.

The high energy electron probe produces a number of effects, each of which can be measured and used to produce an image revealing different information about the specimen. Because of the relatively short wavelength of electrons and the very small angular apertures the resolution and depth of field is much better than that from conventional light microscopy. Resolutions down to 100 - 200 Å can be obtained compared with about 5000 Å for the light microscope and the depth of field is atleast 300 times that of the light microscope for all magnifications.

Low energy secondary electrons (less than 50V) are liberated from a thin layer on the specimen surface and these fly off in all directions. These electrons are collected and accelerated into a scintillator by applying a voltage of 10 kv to a metal ring around the scintillator. The electrons leave the
specimen surface and follow curved trajectories to the detector, the high voltage ensuring that each electron produces light when it strikes the scintillator. A photomultiplier is used to convert the light into an electrical signal.

For probe spot sizes greater than about 100 Å the area over which secondary electrons are liberated is only slightly greater than this value so that ultimate resolutions of better than 200 Å are obtainable.

The number of secondary electrons liberated depends on the angle between the primary beam and the specimen surface so that the resultant image shows the shape of the specimen surface. Back scattered electrons have a small effect since these are either detected or produce secondary electrons which are collected. A contrast in terms of atomic number is built up but this effect represents only a small fraction of the signal. If potential differences are present in the specimen, then these will be detected as an added contrast in the picture.

In specimen preparation for scanning electron microscopy, the specimen blocks (of aluminium) are coated with an adhesive tape on the top surface and the edges trimmed. Over this surface, the sand to be studied is gently sprinkled on. Bulk samples like mould pieces are similarly stuck on over larger sample blocks. These specimens are then gold coated in a vacuum chamber. The gold attached to a filament is melted and vapourises in the vacuum chamber. The specimens are coated whilst rotating and tilting the specimen holder for uniform coating. This is because in order to look at a specimen in the scanning electron microscope.
It is necessary to ensure that the specimen is electrically conductive. For non-conducting specimens, this is achieved, in the experiments conducted, by coating the specimen with a conducting layer of gold, approximately 100 Å thick.

The specimen is inserted into the microscope and an image is obtained on the TV Screen for the purpose of viewing and selection of appropriate area. The accelerating voltage used was 25 kv. A suitable point on the specimen is selected. Then the image is transferred to a high resolution cathode ray tube where by appropriate modulation of the signal suitable contrast of the image is obtained. This is then photographed.

3.11 DETERMINATION OF SINTERING POINT

A small amount of loose sand sample was taken in a porcelain boat and introduced into a tubular furnace. The temperature of the tubular furnace was gradually raised. Beyond 1000°C, the boat was withdrawn at intervals of 25°C and the sample seen through a magnifying glass. The temperature at which the first indications of fusion is noticed is taken as the sintering point of the sand.