STUDIES ON THERMOLUMINESCENCE OF STRONTIUM PYROPHOSPHATE

Shekhar Chandra Manna, Dr. Ritesh Yadav , Dr. Santosh Pawar

Department of Physics, Dr. A.P.J. Abdul Kalam University, Indore (M.P.), India

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ABSTRACT:

The fundamentals and theoretical background of thermoluminescence and TL mechanism, methods for TL parameter and instrumentation of thermoluminescence, applications of thermoluminescence were evaluated. Thermoluminescence properties of rare earth doped strontium pyrophosphate after beta irradiation were discussed and presented. On the basis of theory and application, the outcome of TL has been framed. In this article, studies on thermoluminescence of strontium pyrophosphate has been discussed.

Keywords: Thermoluminescence, Strontium Pyrophosphate, Instrumentation

INTRODUCTION:

Thermoluminescence (TL) is a very old discovery which can initiated by scientists Robert Boyle. The concept of thermoluminescence was first observed by Robert Boyle in 1663. He reported to the Royal Society about observation ‘glimmering light’ emitted from a diamond upon a warming it by his part of “Naked Body’ that reveals the phenomenon of thermoluminescence. He observed similar light emission from the diamond by using more conventional sources of heat like a hot iron, friction and candle.

THEORETICAL BACKGROUND OF THERMO-LUMINESCENCE (TL):

The conventional explanation of Thermoluminescence (TL) also called as thermally stimulated luminescence (TSL) is the phenomenon of emission of light occurring from materials having large energy gap like the insulators or semiconductors invoked the absorption of energy from an ionizing radiation source. The absorption of radiation energy results into the excitation of free

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electrons and free holes. The successive trapping of these electrons and holes occur at defects or trapping states within the energy gap of the material. The emission of light occurs when the material is heated subsequently after removal of the excitation. The thermal energy is used to liberate the charge carriers of negative sign i.e., electrons which are then recombine with charge carriers of the positive sign i.e., holes. If the emission occurs due to recombination process of electron is radiative, the emission is termed as thermoluminescence (TL).

The absorption of energy from ionising radiation is generally the perturbation process in the definite case of TL, followed by the thermally stimulated relaxation (TSR) back to equilibrium, which is followed by monitoring the radiative emission of luminescence from the system during the transitions of the freed charges back to the ground state. The rate at which the system returns to equilibrium is proportional to the TL intensity of the emitted luminescence.

The TL phenomenon can be found in a wide range of synthetically produced solid state materials, including semiconductors, metallo-organic compounds, organic solids, and even complex biological systems like photosynthetic materials. The storage of radiation energy in metastable trap states formed in the energy gap is a well-known feature of TL phenomena, and this energy can then be released via thermally stimulated radiative detrapment.

THERMOLUMINESCENCE(TL) MECHANISM:

The basic theoretical concept of pure crystalline luminescence materials is usually established based on Bloch's "collective electron" model, which has been widely accepted. The allowed energy levels formed for electrons in such phosphors are made up of bands structure of energy states separated by forbidden energy bands, in which the atoms' outermost electrons are responsible for luminescence processes. Lattice defects, impurities, and other perturbations in an ideal crystal lattice can result in discrete energy levels similar to those of an isolated atom, which can be found in the forbidden energy region. Impurity ions or larger complexes agents can act as luminescence emission centres at discrete energy levels in other types of phosphors by providing electron trapping energy levels just below the semiconducting or insulating system's conduction band. Depending on the nature of the defect and the host lattice, the energy levels introduced in the forbidden gap may be discrete or distributed. The phenomenon of phosphorescence and thermoluminescence occurring inside phosphors has been attributed to such types of electron traps within the forbidden gap.

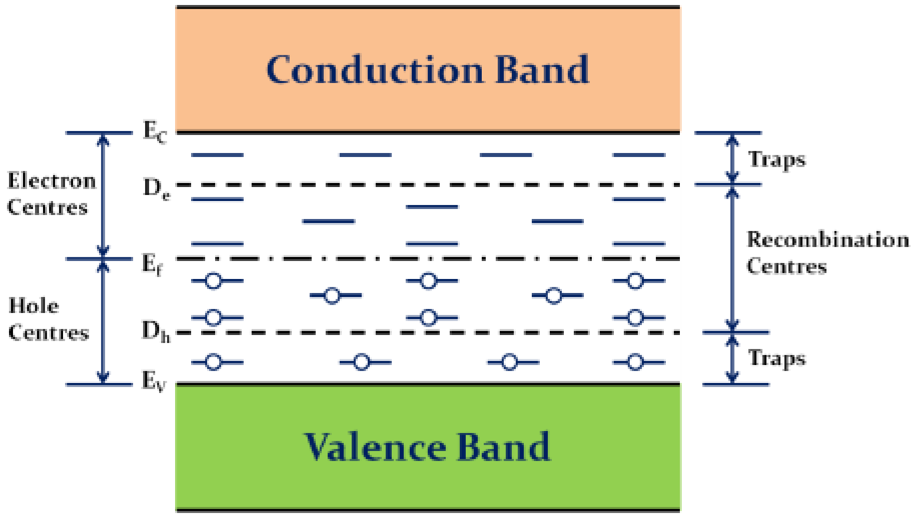

Figure 1. Energy levels diagram of an insulator and semiconductor at absolute zero temperature.

The mechanism of TL model in large band gap material is based band theory. According to this theory the ionizing radiation energy is absorbed which generate free electrons, holes and defects in the material. Following processes are take place in the TL mechanism:

- Trapping by luminescent centres (LCs): Different type of impurity ion can form electron or hole traps in energy gap with a free charge carrier (i.e., electron or hole). The emission of a photon (radiative recombination) can take place if the opposite charge can reaches to the filled the empty trap and recombines with the previously trapped charge. The emission of photons mainly depends on the characteristics of the impurity ion.

- Trapping by non-radiative centres: The free charge carriers (cation/anion) can form the bound states not only with luminescence centres, but also with other various ions/molecular ions. The basic characteristic nature of band states cannot remains same, it could be frequently change. When the charge carrier of the opposite sign arrives to fill the trap, it recombines with the previously trapped charge, but the released energy dissipates without emission of photons (non-radiative recombination).

- Mutual recombination: Some of the free charge carriers of opposite signs can recombine in the matrix without trapping by luminescence centres and non- radiative centres.

- Free charges can be recombined with defects of opposite charge at trapping centres via both radiative and non-radiative transition.

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- ❖ Thermal activation ejection of electrons and holes from traps: The trapped electrons and holes form a bound state with a trapping centres at different energy level. The binding energy of this trapping centres can determine the lifetime or the disintegration probability of the bound state. The disintegration probability increases with increasing temperature.

- ❖ Redistribution of electrons and holes between traps and luminescence centres due to thermal excitation into conduction and valence bands: The charge carriers can produce due to thermal activation from filled traps in the system can be retrapped by all other empty traps, i.e. both luminescence centres and non-radiative centres.

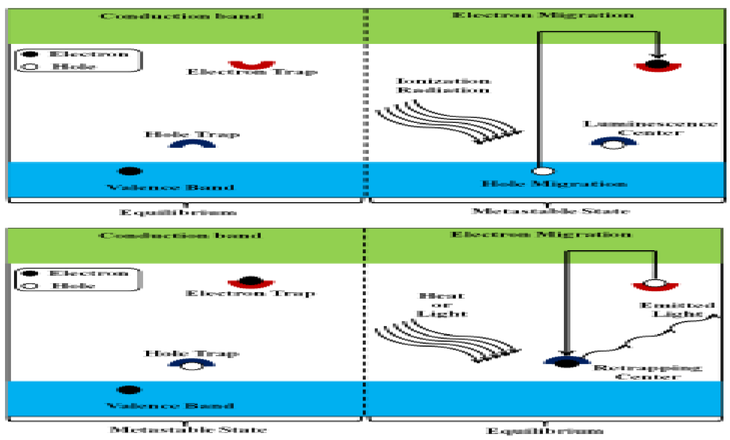

Figure 2 (a) Energy-level diagram of the energy storage stage for TL processes; (b) Energy-level diagram of the energy release stage for TL processes.

METHODS FOR THERMOLUMINESCENCE (TL) PARAMETER:

The primary goal of the thermoluminescence experiment is to extract important information from an experimental TL glow-curve. Using these data, various informative TL parameters can be found, which is very helpful in understanding the mechanism associated with the charge transfer process in the material under study. The TL parameters, such as the trap depths (activation energy) "E," the frequency factors "s," and the densities of the various traps, recombine at the thermoluminescence emission ion centres.

Several methods for evaluating the TL parameters have been used to evaluate the TL glow-curve, and the results from the various methods have been compared to estimate the outcome of the glow curve. To determine TL parameters, researchers used the initial rise (IR) method, peak shape methods (PSM), Kitis et al. equation for curve fitting (GCD fitting), and the whole glow curve-peak method.

- **Initial Rise (IR) Method**

Initial rise method is been recognized by Garlick & Gibson (1969), where they considered the intensity of initial rise part of a TL glow-curve or the rate of change of the trapped carrier concentration is exponentially dependent on temperature according to the relation,

$$I(t) = -\frac{dn}{dt} = (constant) \times exp\left(-\frac{E_a}{k_B T}\right) \dots\dots\dots\dots\dots\dots\dots\dots (1)$$

This equation termed as if the heating temperature of the sample is low enough, the trapped carrier concentration ‘n’ to be remains approximately constant because of very small amount of detrapping have taken place. This relation is independent of the order of kinetics of TL mechanism. The essential condition for initial rise method is that “n’ remains approximately constant if the increase in temperature of sample beyond a critical temperature “Tc’ than this assumption becomes invalid. The critical temperature ‘Tc’ corresponds to the TL intensity ‘Ic’ less than 10-15% of the maximum TL intensity. Initially occupancy of traps and recombination centres are assumed to be remains constant only up to a limiting temperature range under ‘Tc’. If a plot of In versus 1/kgT) is made for the initial rise region, a linear function with a slope of (E./kp) from which the activation energy Es is simply determined, while the intercept on the In(I) axis of the plot gives In(s/B) which the frequency factor ‘s’ is found. Here, Ea is the activation energy, kg is the Boltzmann’s constant and T is the temperature, B is heating rate. The activation energy obtained from this method cannot be the true valued.

- **Whole Glow-Peak Method**

In initial rise method, TL parameter has been calculated using only the limited data of the initial rising region which is severely limited condition for analysis. This difficulty can be overcome by using the whole curve data for evaluate TL parameters. Chen and McKeever proposed a simple model to give explanation of the basic TL phenomenon. According to this model, the number of electrons released per second is proportional to the total concentration of trapped electrons. This means that there is no retrapping of electrons from the conduction band. From these assumptions, the rate of production of emitted photons or intensity I (T) of TL for second order kinetic is given by

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$$I(T) = -\frac{dn}{dt} = n^2 \frac{s}{N} exp\left(-\frac{E_a}{kT}\right) \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots (2)$$

The whole glow-peak method yields information about the activation energy FE, and the effective frequency factor s'=s/N from bellow expression.

$$I(T) = n_0^b \frac{s}{N} exp\left(-\frac{E_a}{kT}\right)\left[1 + \frac{n_0 s}{\beta N}\int_{T_0}^{T} exp\left(-\frac{E_a}{kT'}\right) dT'\right]^{-b} \ldots \ldots (3)$$

Where, no is the area under active curve, b is the order of kinetics, (s/N) = s' is the frequency factor, kg is the Boltzmann's constant, 2 is the heating rate, and E, is the activation energy. In whole curve method, a plot of In[*I(T)/nb*] versus 1/*T* gives a straight line having slope (-E,/k_B), and an intercept of In (s/β). The straight line can be obtained by putting the proper value of b in above equation. The major difficulties came about to use whole curve method is that it requires merely isolation glow peaks and a clean descending part of the last peak in the glow-curve.

- **Peak Shape Method**

Peak shape method (PSM) used for analysis of the glow-curve can just utilize three points of the TL glow-curve rather than the partial or whole peak analysis methods, because of the shape of glow-peak is mainly affected by the order of kinetics as a result the method strongly dependent on the order of kinetics.

TL parameters have been easily calculated using the Chen's peak shape method. Chen (1969) proposed three equations for both first-order kinetics, second-order kinetics and general-order kinetics. The TL parameters of the glow-curves have been calculated using three parameters τ, δ and ω. $\tau = T_M - T_1$ $\delta = T_2 - T_M$ $\omega = T_2 - T_1$

Where,'t' is the low temperature half width of a peak, "δ' is the half width at the high temperature and 'ω' is the total half-intensity width. T1 and T2 are the low and high temperatures corresponding to half-maximum intensity, respectively. Tm is the temperature at maximum intensity. Three equations of activation energy for t, ω and δ are given by:

For first-order kinetics:

$$E_\tau = \frac{1.51kT_M^2}{\tau} - 1.58(2k_BT_M) \ldots\ldots\ldots\ldots\ldots\ldots (4)$$

$$E_\delta = \frac{0.976k_BT_M^2}{\delta} \ldots\ldots\ldots\ldots\ldots\ldots (5)$$

$$E_\omega = \frac{2.52k_BT_M^2}{\omega} - 2kT_M \ldots\ldots\ldots\ldots\ldots\ldots (6)$$

For second-order kinetics:

$$E_\tau = \frac{1.81k_BT_M^2}{\tau} - 2(2kT_M) \ldots\ldots\ldots\ldots\ldots\ldots (7)$$

$$E_\delta = \frac{1.71k_BT_M^2}{\delta} \ldots\ldots\ldots\ldots\ldots\ldots (8)$$

$$E_\omega = \frac{3.54k_BT_M^2}{\omega} - 2kT_M \ldots\ldots\ldots\ldots\ldots\ldots (9)$$

For general-order kinetics:

$$E_\tau = \frac{1.72k_BT_M^2}{\tau} - 1.874(2kT_M) \ldots\ldots\ldots\ldots\ldots\ldots (10)$$

$$E_\delta = \frac{1.487k_BT_M^2}{\delta} \ldots\ldots\ldots\ldots\ldots\ldots (11)$$

$$E_\omega = \frac{3.234k_BT_M^2}{\omega} - 2kT_M \ldots\ldots\ldots\ldots\ldots\ldots (12)$$

Where Eτ, Eω and Eδ are the activation energy of TL glow curve for the three temperature co-efficient τ, ω and δ.

- **Glow Curve Deconvolution (GCD) Method**

The analysis of composite TL glow curve have been done by the glow-curve deconvolution (GCD) technique, in which the whole curve is divided into the individual glow peaks [78]. The TL glow curves are analysed by using the glow curve deconvolution (GCD) method developed by Kitis et al. This method can used for first-order kinetics, second-order kinetics and general-order kinetics TL glow curves. The mathematical expression for different kinetics of TL glow curve are mainly depends on two experimentally measured parameters, one of them is the maximum TL intensity 'Im' and another is the temperature corresponding to the maximum TL intensity "Tm'.

Kitis, et al equation for first-order kinetics:

$$I(T) = I_M \exp\left[1 + \frac{E_a}{k_B T}\cdot\frac{T-T_M}{T_M} - \frac{T^2}{T_M^2}\times\left(1-\frac{2k_B T}{E_a}\right) exp\left(\frac{E_a}{k_B T}\cdot\frac{T-T_M}{T_M}\right) - \frac{2k_B T_M}{E_a}\right] \ldots\ldots (13)$$

Kitis, et al equation for second-order kinetics:

$$I(T) = 4I_M exp\left(\frac{E_a}{k_B T}\cdot\frac{T-T_M}{T_M}\right) \times\left[\frac{T^2}{T_M^2}\cdot\left(1-\frac{2k_B T}{E_a}\right) exp\left(\frac{E_a}{k_B T}\cdot\frac{T-T_M}{T_M}\right) + 1 + \frac{2k_B T_M}{E_a}\right]^{-2} \ldots\ldots (14)$$

Kitis, et al equation for general-order kinetics:

$$I(T) = I_M b^{b-1} exp\left(\frac{E_a}{k_B T}\cdot\frac{T-T_M}{T_M}\right) \times\left[(b-1)\frac{T^2}{T_M^2}\cdot\left(1-\frac{2k_B T}{E_a}\right) exp\left(\frac{E_a}{k_B T}\cdot\frac{T-T_M}{T_M}\right) + 1 + -1)\frac{2k_B T_M}{E_a}\right]^{-\frac{b}{b-1}} \ldots\ldots (15)$$

Where, T_M is the maximum temperature corresponding to I_M, T is the temperature TL data, k_B is the Boltzmann's constant and E_a is the activation energy. The TL intensity obtained theoretically for different activation energy from the experimental data has been compared and the excellent fitting have been done in GCD method. The precise fitting of experimental TL glow curve and theoretical TL glow curve is been determined by calculating the figure of merit (FOM).

The FOM is obtained by the bellow the relation:

$$FOM = \frac{\sum_p |I_{Exp} - I_{Th}|}{\sum_p |I_{Th}|} \ldots\ldots (16)$$

The frequency factor (s) of TL glow curve has been calculated by using the proper value of activation energy E, of that glow peak for which the curve fitting is the most precise.

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Frequency factor of first order kinetics is obtained by the bellow the relation:

$$s = \frac{\beta E_a}{k_B T_M^2} exp\left(\frac{E_a}{k_B T_M}\right) \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (17)$$

Frequency factor of second order kinetics is obtained by the bellow the relation:

$$s = \frac{\beta E_a}{k_B T_M^2 \left(1 + \frac{2kT_M}{E_a}\right)} exp\left(\frac{E_a}{k_B T_M}\right) \ldots\ldots\ldots\ldots\ldots\ldots (18)$$

Frequency factor of general order kinetics is obtained by the bellow the relation:

$$s = \frac{\beta E_a}{k_B T_M^2 \left[1 + \frac{2kT_M(b-1)}{E_a}\right]} exp\left(\frac{E_a}{k_B T_M}\right) \ldots\ldots\ldots\ldots\ldots (19)$$

where T_M is the maximum temperature forI_M, $\beta$is the heating rate (K/s), k is the Boltzmann's constant and E_a is the activation energy.

INSTRUMENTATION OF THERMOLUMINESCENCE:

The electronic functional block diagram of the TL reader conjugated with PC system and TL reader software shown in Figure PC controlled TL reader type TL1009 manufactured by Nucleonix used for TL measurements of the phosphor is shown in Figure.

The TL reader consists of following segments of electronic circuits and other components.

Low Voltage Supply: It is a DC power supply circuit that provide low voltage of the order of +5V @ 1A, +/- 12V @ 0.5A, +24V @O0.5A. It provides power required for functionality of all the circuits. It consists an input line filter circuit, step down transformer with four secondary voltages, a three terminal regulator (bridge rectifier) and filter capacitors.

High Voltage Module: High voltage power supply provides desire biasing voltage require to the photo multiplier tube because photo multiplier tube is negative biased to DC mode of operations in the voltage range of 0 to -1500V. The output of high voltage supply is a highly stabilized regulated voltage supply which generates (0-1500V) @ 1.0mA with less than 30mV rippled & noise (peak to peak).

PMT bleeder circuit: The main work function of the photo multiplier tube (PMT) is to detect the light photon emitted during the heating process of phosphor that incident onto it, and to covert the light due to TL emission into D.C. current. PMT is biased by bleeder resistor network that operate it in low dark current mode. DC output of the PMT from the anode is applied to the I-F converter. For the DC current mode operation, the photo cathode of the PMT being connected to the negative bias and anode is connected with virtual zero potential.

I/F (current to frequency) converter: A charge balancing type of current to frequency (I-F) converter circuit is used in TL reader. It has a linearity better than 1% over a frequency range from | Hz to 100 KHz. The work function of I-F circuit is to convert anode current from the PMT into a continuous pulse of frequency proportional to the current.

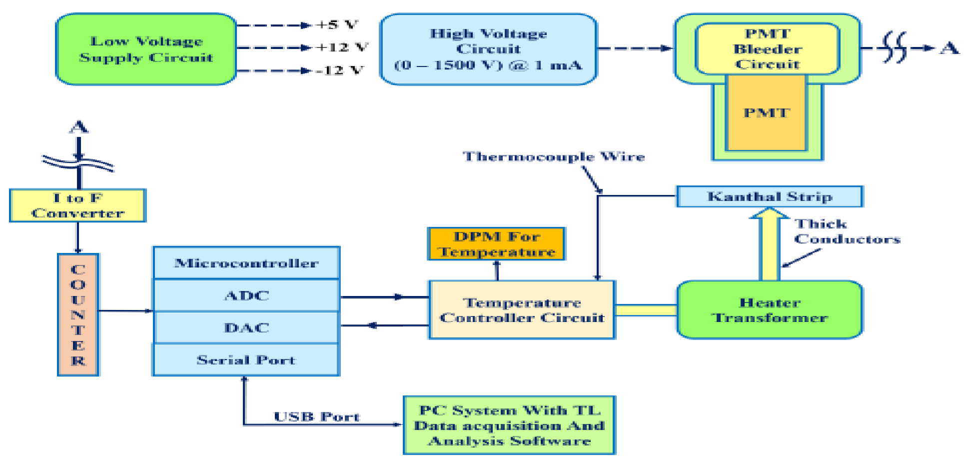

Figure 3 Block diagram of the TL Reader.

Microcontroller with BCD counter, ADC, DAC, serial port: The work-functions of this section of the TL instrument are the temperature calibration data and interpret in terms of actual temperature, generate various heating profiles & heating rates, command instructions from the PC, to receive TTL pulses from I to F converter which corresponds to TL intensity data and storing them in the memory.

Temperature controller: The heater transformer, heater element with thermocouple, millivolt amplifier, and display modules make up the temperature controller system (DPM). In this system, the millivolt thermocouple output from the kanthal strip is fed to a monolithic thermocouple amplifier, which then feeds the output to an op-amp amplifier to condition the signal level appropriately. This signal is then fed to a 3.5 digital DPM, which can be calibrated to show the kanthal strip's temperature. As a result, the signal is sent to an ADC, where the value is read by a microcontroller, which then interprets it in terms of temperature using a PC programme. The sample can be heated at a rate of 1°C/sec to 40°C/sec, with a maximum set temperature of 500°C for different heating profiles such as single or multiple plateaus.

A computer system that includes the following software is used to collect and analyse data. The required graphical TL glow curve data acquisition and analysis software is provided by a personal computer (PC) system with TL instrument function software. The following functions are carried out by the software: Temperature Calibration, Light Stimulation Profile, Acquisition, and Linear, Single, and Multi-Plateau Heating Profiles Background spectrum / sample data can be obtained, and Background Subtraction can be performed. Spectrum overlap, Spectrum subtraction, and Spectrum export to Excel.

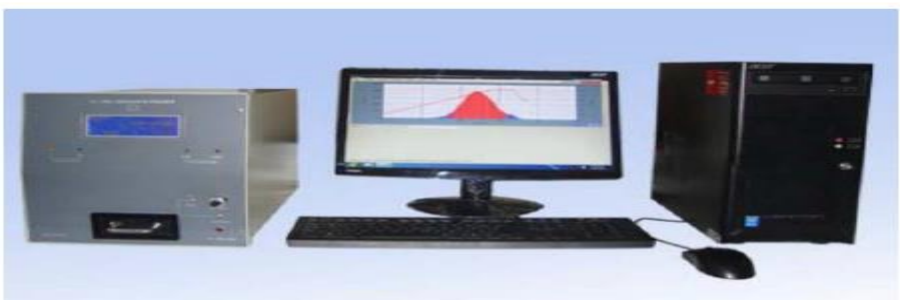

Figure 4 PC controlled TL reader type TL1009 manufactured by Nucleonix.

APPLICATIONS OF THERMOLUMINESCENCE(TL):

The TL phenomenon has been vastly studied by many researchers to understand the mechanism involved in thermally stimulated emission and it application in present. However, due to the deep knowledge of TL mechanism, the innovation and improvement in the TL instrumentation helped the researchers in various fields to solve their problems, out of large area of application some of the applications are explained briefly. Figureshows some important applications of the TL phenomenon in present world are summarized in the chart.

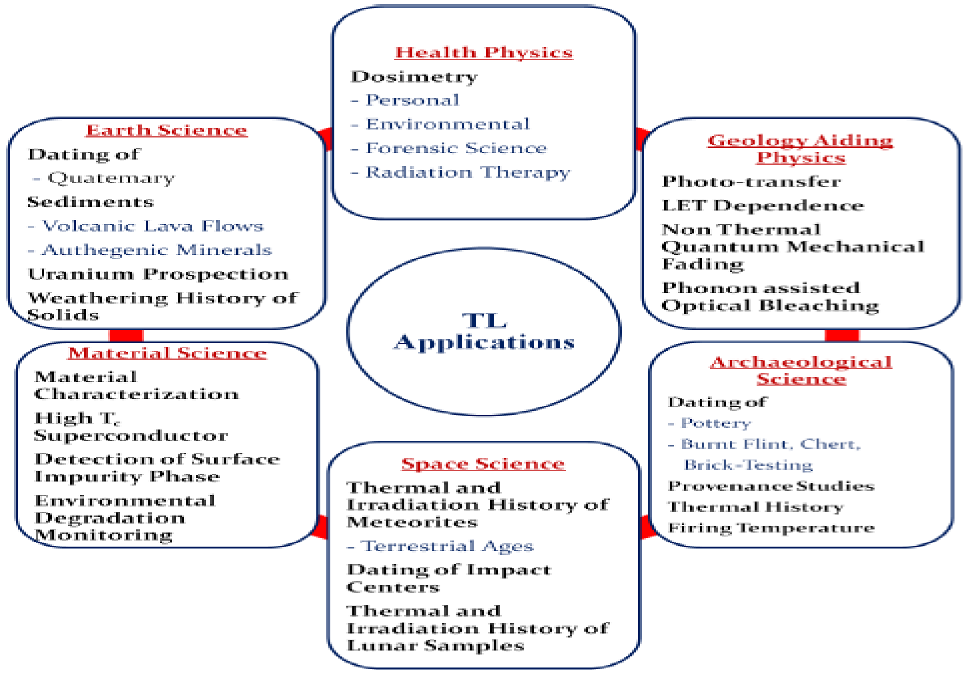

Figure. 5 Applications of TL in various research area

➢ TL Dosimetry (TLD)

In Medical Physics and Science, radiation dosimetry applications are a necessary and fundamental requirement for measuring ionization radiations and radioisotopes. Ionizing radiations are most commonly found in places where X-rays, gamma radiation, and beta particles are commonly used for diagnosis and radiotherapy, X-rays are used in airport security systems, and radiations are emitted during experimental studies in Physics and other Science branches. In the major medical centres, high-energy electrons, heavy particles, and neutrons are used.

Personnel Dosimetry

In personal dosimetry, the radiation dose measurement has been performed to measure the radiation absorbed by the personnel for the duration of their repetitive occupational exposure at working place. There are several working fields like nuclear industry, workers deal with X-ray units, hospital medical physicists, radiotherapy technicians, workers in industrial radiography, high intensity gamma irradiators and personnel on nuclear power plants, etc. On the basis of monitoring such measurements of exposure, it is recognized that the personnel absorbed

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radiation under certain safety limits, which are prescribed by the International Commission of Radiological Protection (ICRP) for different field of application of radiation sources.

Environmental Dosimetry

Pollution from nuclear power has posed a serious threat to the health of living organisms in recent years. The majority of pollution is caused by gamma and UV radiation, both of which are extremely harmful to humans. As a result, environmental regulatory authorities in both developed and developing countries are more concerned about environmental health. TLD systems have been installed near nuclear power stations, low-level waste disposal, nuclear fuels reprocessing units, incidents of nuclear power station accidents places, nuclear power industry, and other sources of nuclear radiation and ionization radiation in many countries to monitor the environment. The tissue equivalence size is not necessary for the phosphor used in environmental dosimetry. However, the time spent monitoring the environment is much longer, allowing for long periods of material exposure. As a result, for environmental dosimetry, the TLD phosphor should be more stable and highly sensitive to low radiation exposure.

Medicinal Dosimetry

TLD materials are widely used in medical science for determining the appropriate ionising radiation dose administered to patients during diagnostic or therapeutic procedures. TLD exposed during treatment is retrieved for radiation dose analysis, allowing doctors to determine the actual doses delivered to critical internal organs during these procedures and prescribe necessary additional treatments based on this information. X-ray exposure in mammography, dentistry, general health screening, and radiotherapy are just a few of the areas where clinical radiation is exposed to humans. Radiation doses used in radiology range from 10° to 107 Gy, with radiotherapy doses ranging from 20-60 Gy. The tissue equivalency of the TLD phosphors used in medical dosimetry, as well as the phosphors' low radiation absorbance capacity, is a more important factor for good TLD.

Archaeological Science

Thermoluminescence (TL) technique is most powerful and efficient technique for the calculation of age of the ancient pottery since firing. The age of ancient pottery is determined as the time period for which it has been exposed to the irradiation following to its previous heating process.

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Using a TL dating, it is possible to estimate the age of ancient pottery or archaeological object, if the rate of radiation energy stored in this materials can be measured and the formulation between the absorbed radiation and the emitted light is established. The relation for calculating the age of a specimen of pottery using TL is given bellow.

$$Age = \frac{Natural\ TL}{(TL\ per\ unit\ dose) \times (Natural\ dose\ rate)}$$

Geology

The geological age of rocks can be determined using TL, which is a versatile technique. In nature, geological activities take place over a long period of time, and several radioactive elements can be found in small amounts within the rock structure. Natural radiations emitted by radioactive elements were used to expose these types of rocks. When heated, these rocks produce natural thermoluminescence. The thermoluminescence of rocks provides information about the radiation accumulated in the rocks, allowing researchers to determine how long the rocks were exposed to these radiations and thus the age of the rocks. Thermoluminescence was first used in geology for a variety of purposes, including mineralization dating, igneous activity dating, sedimentation dating, and determining the rate of growth of beaches and sand dunes. Thermoluminescence is a technique for dating geologically recent specimens that is very useful.

CONCLUSION:

The application of thermoluminescence has recently been found to be extremely valuable in a variety of scientific disciplines. Personal dosimetry, environmental and retrospective dosimetry, geological and archaeological dating, and a variety of medical applications like radiation therapy, diagnostic radiology, and radiotherapy dosimetry are just a few of the applications. A dosimeter with a flat energy response to low-energy photons is preferable for diagnostic radiology.The basic description of TL phenomenon can be given in the following statement which is very similar to the thermally stimulated luminescence (TSL) phenomena: “TL requires the perturbation of the system from a state of thermodynamic equilibrium, via the absorption of external energy, into a metastable state. This is then followed by the thermally stimulated relaxation of the system back to its equilibrium condition”.

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