

Contents

Certificate	6
Acknowledgement	7
Abstract	8
1 Introduction	9
1.1 Radiation	9
1.2 Radiation Dosimetry	15
1.3 Thermoluminescence Dosimetry	18
1.4 Nanophosphors in Dosimetry	22
2 Synthesis and Characterization of Nanophosphors	24
2.1 Synthesis Of Nanophosphors	24
2.1.1 Co-Precipitation	24
2.1.2 Solid State Reaction	25
2.1.3 Combustion Method	26
2.2 Annealing	27
2.3 Characterization Of Nanophosphors	28
2.3.1 X-ray Diffraction	28
2.3.2 Scanning Electron Microscopy (SEM)	29
2.3.3 Transmission Electron Microscopy (TEM)	29
2.3.4 Fourier Transform Infrared Spectroscopy (FTIR)	30
3 Experiment	32
3.1 Lithium metasilicate (Li_2SiO_3)	32
3.1.1 Synthesis of Li_2SiO_3 (pure)	32
3.1.2 Dopant Optimization for Li_2SiO_3	32
3.2 Calcium Sodium Sulphate ($CaNa_2(SO_4)_2$)	33
3.2.1 Synthesis of $CaNa_2(SO_4)_2$ (pure)	33
3.2.2 Synthesis of $CaNa_2(SO_4)_2 : Eu$	34
3.3 Evaluation of Glow Curves	35
3.4 Ion Beam Irradiation	35
3.5 Deconvolution of TL Glow Curves	36
4 Result and Analysis	37
4.1 Lithium metasilicate (Li_2SiO_3)	37
4.1.1 Deconvolution of Glow Curves	39
4.2 Calcium Sodium Sulphate ($CaNa_2(SO_4)_2$)	40
4.2.1 Dose Response	41
Conclusion	42
Bibliography	43

List of Figures

1	Classification of radiation	9
2	Average Annual Radiation Dose[33]	10
3	Effective Radiation Dose [1]	11
4	Deterministic and Stochastic Effects of Radiation [26]	12
5	Principle of ALARA [25]	13
6	A thermoluminescent dosimeter[19]	18
7	Energy band model showing the electronic transitions in a TL material according to a simple two-level model.[6]	19
8	Theory of operation behind thermoluminescent dosimeters[29]	20
9	Nanophosphors being used in TLD badges[9]	22
10	Synthesis using Co-Precipitation Method[37]	24
11	Schematic diagram for Co-Precipitation Method[18]	25
12	Synthesis using Solid State Reaction Method[17]	25
13	Synthesis using Combustion Method[36]	26
14	Various stages of annealing[14]	27
15	A X-ray diffraction machine[23]	28
16	Schematic diagram of Scanning Electron Microscope[21]	29
17	Schematic diagram of Transmission Electron Microscope[27]	30
18	Fourier Transform Infrared Spectroscopy (FTIR) instrumentation[31]	30
19	Precursor materials being weighed and ground into a fine paste with acetone	32
20	Sample being ground after removing acetone by heating with heating mantle	32
21	Sample being annealed in a furnace at 900°C for 4 hours	33
22	Final sample after preparation	33
23	Formation of precipitate due to drop-wise addition of (NH ₄) ₂ SO ₄ to the solution	34
24	Centrifuging the sample	34
25	Sample ground to a fine powder	34
26	Final sample	34
27	Gamma ray irradiation chamber at Inter University Accelerator Centre (IUAC), New Delhi using ⁶⁰ Co source	35
28	Harshaw TLD Reader to for measurement of dose response of the samples	35
29	Hydraulic Pellet press to turn the samples into pellets	35
30	Pellets kept in a boat to be annealed in a muffle furnace	35
31	Samples being loaded in a ladder	36
32	Ion beam irradiation chamber at IUAC	36
33	Code snippet of R program for deconvolution of glow curves	36
34	Dose response for Lithium metasilicate when irradiated with Gamma rays at 50 Gy and 100 Gy	37
35	Dose response for Lithium metasilicate doped with 0.1mol% Europium when irradiated with Gamma rays at 50 Gy and 100 Gy	37
36	Dose response for Lithium metasilicate doped with 0.1mol% Dysprosium when irradiated with Gamma rays at 50 Gy and 100 Gy	37
37	Dose response for Lithium metasilicate doped with 0.1mol% Cerium when irradiated with Gamma rays at 50 Gy and 100 Gy	37
38	Dose response for Lithium metasilicate doped with 0.1mol% Erbium when irradiated with Gamma rays at 50 Gy and 100 Gy	37
39	Dose response for Lithium metasilicate doped with 0.1mol% Terbium when irradiated with Gamma rays at 50 Gy and 100 Gy	37
40	Dose response for various dopants when irradiated with Gamma rays at 50 Gy	38
41	Dose response for various dopants when irradiated with Gamma rays at 100 Gy	38
42	Comparision of dose response of <i>Li₂SiO₃</i> without doping and after doping with Terbium and Erbium when irradiated with Gamma rays at 50 Gy	38
43	Comparision of dose response of <i>Li₂SiO₃</i> without doping and after doping with Terbium and Erbium when irradiated with Gamma rays at 100 Gy	38
44	Comparision of peak intensity for various dopants when irradiated with Gamma rays at 50 Gy	38
45	Comparision of peak intensity for various dopants when irradiated with Gamma rays at 100 Gy	38
46	Deconvolution of glow curve of pure <i>Li₂SiO₃</i> at 50 Gy	39
47	Deconvolution of glow curve of pure <i>Li₂SiO₃</i> at 100 Gy	39
48	Deconvolution of glow curve of <i>Li₂SiO₃</i> doped with Erbium at 50 Gy	39
49	Deconvolution of glow curve of <i>Li₂SiO₃</i> doped with Erbium at 100 Gy	39
50	TL Glow Curve for Calcium Sodium Sulphate when irradiated with Gamma ray at 50 Gy	40

51	TL Glow Curve for Calcium Sodium Sulphate when irradiated with Gamma ray at 100 Gy	40
52	Dose response for Calcium Sodium Sulphate after doping with Europium when irradiated with Gamma ray	40
53	TL Glow curves for Calcium Sodium Sulphate doped with Europium at multiple doses	41
54	Dose response for Calcium Sodium Sulphate doped with Europium	41

Certificate

This is to certify that the dissertation project titled "**To investigate the thermoluminescent properties of synthesized nanophosphors for use in radiation dosimetry**" submitted to the **University of Delhi** for the degree of **Bachelors of Science (Honours) Physics**, is an authentic work of our bonafide student, **Meemik Roy**, carried out under my supervision and guidance. I also certify that neither a part of this work nor the whole of it has been published anywhere else or has formed the basis for the award of any other degree or diploma or other similar title to any candidate of this or any other university.

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Abstract

Radiation is a form of energy that can have both beneficial and harmful effects on living organisms. Accurate measurement and monitoring of radiation levels are crucial to prevent the harmful effects of radiation exposure. Thermoluminescence dosimetry (TLD) is a well-established technique that provides reliable and precise measurements of ionizing radiation doses. TLDs use a variety of materials that emit light when exposed to ionizing radiation. It has wide range of applications in medical, archaeology, geology, meteorology and also in space dosimetry or in industries to maintain the quality of products. The basic principle of thermoluminescence dosimetry involves the use of thermoluminescent materials, which are crystalline or amorphous substances capable of storing energy when exposed to ionizing radiation. Nanophosphors, also known as nanocrystalline phosphors or nanoparticles, have emerged as promising materials for dosimetry applications. These nanoscale phosphors exhibit unique properties that make them highly suitable for radiation dosimetry.

Ideally, a TL dosimeter should exhibit the following properties: linearity in dose response, high sensitivity, low fading, simple glow curve, reproducibility, tissue equivalence and stability against environmental factors (the material must not undergo physical changes in high humidity, react chemically with corrosive agents or show spurious thermoluminescence). Practically not all TL materials possess all of the above features. And thus, researchers throughout the world constantly search for materials that could possess most of these features. In the present work we have tried to prepare a TL material that possesses good number of essential features suitable for radiation dosimetry. Moreover, it is also interesting to understand the physics behind electronic excitation and relaxation phenomena in TL materials that are doped with dopants.

This work explores the advancements made in thermoluminescence dosimetry, its current applications, and the potential future prospects for this field. The research encompasses an in-depth analysis of TLD principles as well as various techniques required for synthesis and characterization of thermoluminescent materials, especially nanophosphors. In this work, two nanophosphors have been synthesized using two different techniques. *Calcium Sodium Sulphate ($CaNa_2(SO_4)_2$)* was synthesized using chemical co-precipitation method and *Lithium metasilicate (Li_2SiO_3)* was synthesized using solid-state reaction method their dose response was investigated. Variation in dose response due to doping was also one of the major points of focus in this research.

TL Dose Response measurements indicated increase in TL intensity due to doping in both the samples. Lithium metasilicate (Li_2SiO_3) in particular, shows two prominent peaks at distinct temperatures with increased intensity on doping. This on top of being near tissue equivalent shows promise to be used as an effective material to be used in dosimetry.

1 Introduction

Radiation is used in various fields, including medical treatment, industry, and research. Radiation is a form of energy that can have both beneficial and harmful effects on living organisms. Therefore, it is important to accurately measure and monitor the levels of radiation to which individuals are exposed. Radiation exposure is a major concern in various fields, including medical treatment, industry, and research. Accurate measurement and monitoring of radiation levels are crucial to prevent the harmful effects of radiation exposure. It is crucial to accurately measure and monitor radiation doses to ensure the safety of individuals working with radiation sources and to evaluate the potential risks associated with radiation exposure. **Thermoluminescence dosimetry (TLD)** is a well-established technique that provides reliable and precise measurements of ionizing radiation doses. TLDs use a variety of materials that emit light when exposed to ionizing radiation. Among these materials, nanophosphors show promising properties that make them a potential candidate for use in TLDs.

1.1 Radiation

Radiation is an integral part of our natural environment and encompasses various forms of energy emitted by atoms and subatomic particles. It is both a natural phenomenon and a byproduct of human activities, playing significant roles in numerous scientific, medical, and technological fields. While radiation has beneficial applications in areas such as medicine, energy production, and communication, it can also pose risks to human health and the environment. Understanding the nature, types, sources, and effects of radiation is essential for ensuring its safe and responsible use. Radiation is the transfer of energy through electromagnetic waves or subatomic particles. It occurs in various forms, including electromagnetic radiation (such as gamma rays, X-rays, ultraviolet, visible light, and radio waves) and particulate radiation (such as alpha particles, beta particles, and neutrons). Each type of radiation possesses unique properties and interacts differently with matter. Radiation can therefore be classified based on the way it interacts with matter as shown in Figure 1

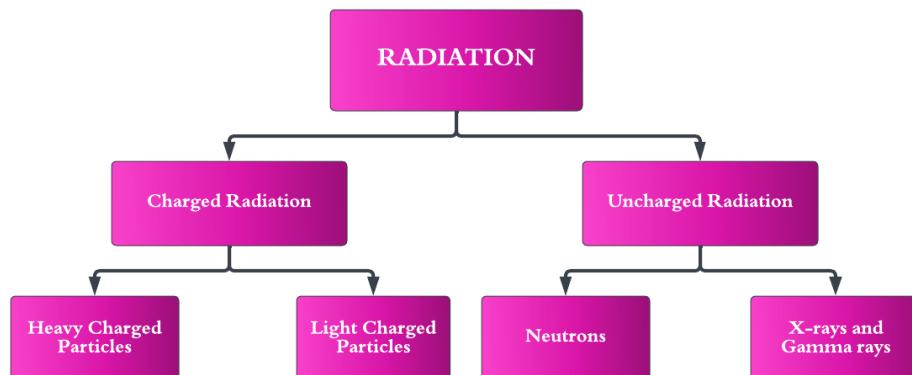


Figure 1: Classification of radiation

- **Uncharged Radiation**

A.) **X-Ray and Gamma Rays:** Gamma Rays and X-rays interact with matter through 3 main processes :-

- (a) **Photoelectric Effect:** In this process, a photon undergoes an interaction with an absorber atom in which the photon completely disappears. In its place, an energetic *photo-electron* is ejected by the atom from one of its bound shells. The energy of the photo-electron is given by:

$$E_{e^-} = h\nu - E_b$$

where E_b is the binding energy of photo-electron in its original shell.

- (b) **Compton Scattering:** In this process, the incoming γ -ray photon strikes an electron in the absorber medium and the γ -ray is deflected through an angle θ with respect to its original position. The photon transfers a portion of its kinetic energy to the electron, which is known as recoil electron. The amount of energy transferred depends on the angle of scattering θ . The energy of the scattered photon is given by:

$$h\nu' = \frac{h\nu}{1 + \frac{h\nu}{m_0 c^2} (1 - \cos \theta)}$$

where m_0c^2 is the rest mass energy of electron.

- (c) **Pair Production:** If the γ -ray energy exceeds twice the rest-mass energy of electron (1.02 MeV), the process of pair production is energetically possible. In this interaction, the γ -ray photon disappears and is replaced by an electron-positron pair.
- B.) **Neutrons:** Neutrons are generally difficult to detect as their interaction with matter is very less and can travel large distances without causing any interactions and hence can be detected only through indirect methods depending on its speed. **Slow Neutrons** do not directly interact with detector atoms, however they have a high probability of causing neutron induced nuclear reactions which in turn produce secondary particles/radiation which can be detected. While **Fast Neutrons** with high Kinetic Energies are mainly detected through scattering. For example, for reactions with moderators like hydrogen, the recoil neutrons become secondary radiation. If Kinetic Energy of neutrons is sufficiently high, inelastic collisions occur with detector atoms causing nucleus to excite to higher states, the excited nucleus quickly de-excite to release radiation, which can then be detected.

- **Charged Radiation** Charged particles interact with matter through *Coulomb Forces* between the radiation and the detector. These particles interact simultaneously with many electrons and in one such encounter, the electron receives sufficient impulse from Coulomb force of the passing particle to excite the electron to higher state (*excitation*) or completely remove the electron from the atom (*ionization*). The average energy loss is given by the **Bethe-Bloch**[32] formula:

$$\frac{dE}{dx} = -K \frac{Z}{A} \frac{\rho}{\beta^2} \left\{ \ln \frac{2mc^2\beta^2 E_M}{I^2(1-\beta^2)} \right\}$$

where β is the velocity (in units of c) of particle, $K = \frac{2\pi N z^2 e^4}{mc^2}$ and $E_M = \frac{2mc^2\beta^2}{1-\beta^2}$ is the maximum energy transfer allowed.

Sources of Radiation

Sources of radiation can be categorized into two main groups [2]: natural sources and artificial (or man-made) sources. The average annual radiation dose is shown in figure 2

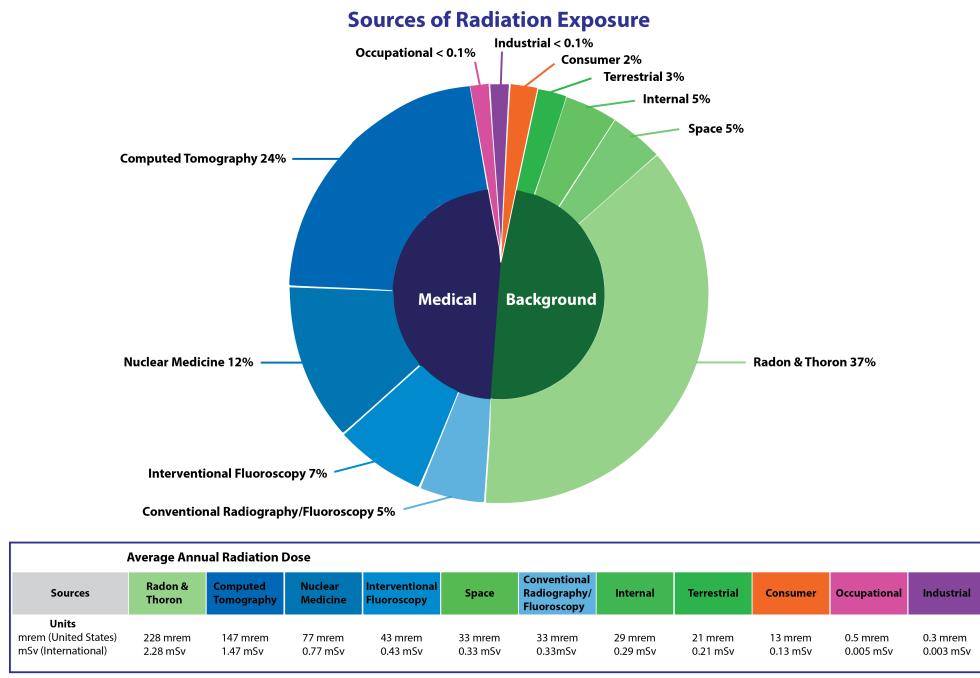


Figure 2: Average Annual Radiation Dose[33]

A.) Natural Sources of Radiation:

- **Cosmic Radiation:** High-energy particles, such as protons and atomic nuclei, that originate from outer space and reach the Earth's atmosphere.

- **Terrestrial Radiation:** Naturally occurring radioactive elements present in the Earth's crust, such as uranium, thorium, and radon gas.
- **Radon Gas:** A radioactive gas that is released from the decay of uranium and thorium in rocks, soil, and water. It can accumulate in buildings, particularly in basements and poorly ventilated areas.
- **Radioactive Isotopes in the Environment:** Small amounts of radioactive isotopes, such as potassium-40 and carbon-14, are naturally present in the environment and contribute to background radiation.

B.) Artificial (Man-Made) Sources of Radiation:

- **Medical and Dental Procedures:** Diagnostic imaging techniques like X-rays, computed tomography (CT) scans, and nuclear medicine procedures involve the use of ionizing radiation.
- **Radiation Therapy:** High-energy radiation, such as X-rays or gamma rays, used to treat cancer and other medical conditions.
- **Nuclear Power Plants:** Nuclear fission in reactors generates heat and electricity but also produces low-level radiation during normal operation and potential radioactive releases during accidents.
- **Industrial Applications:** Industrial radiography, gauging, and research activities that involve the use of radiation for inspection, measurement, and scientific experiments.

A chart depicting the levels of ionizing radiation is given below figure 3

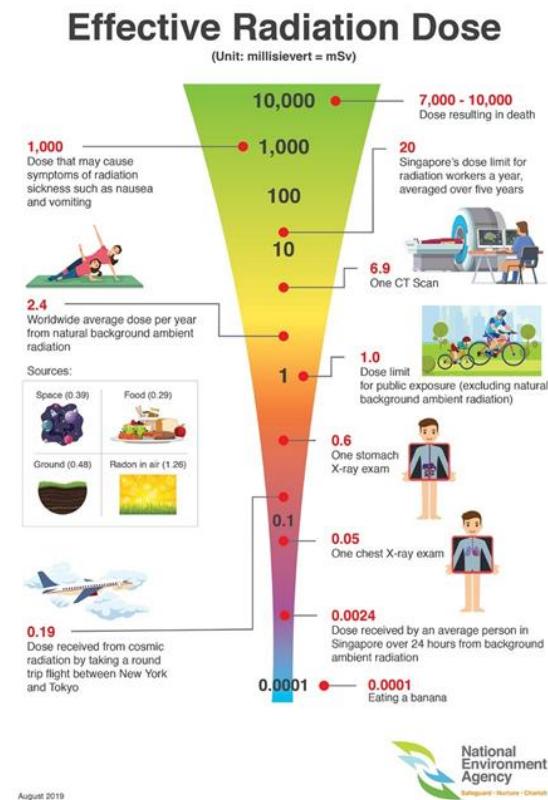


Figure 3: Effective Radiation Dose [1]

Effects of Radiation

The effects of radiation on living organisms can be divided into two main categories: deterministic effects and stochastic effects. These effects depend on various factors, including the type of radiation, the dose received, and the duration of exposure. Here is an overview of the different effects of radiation:

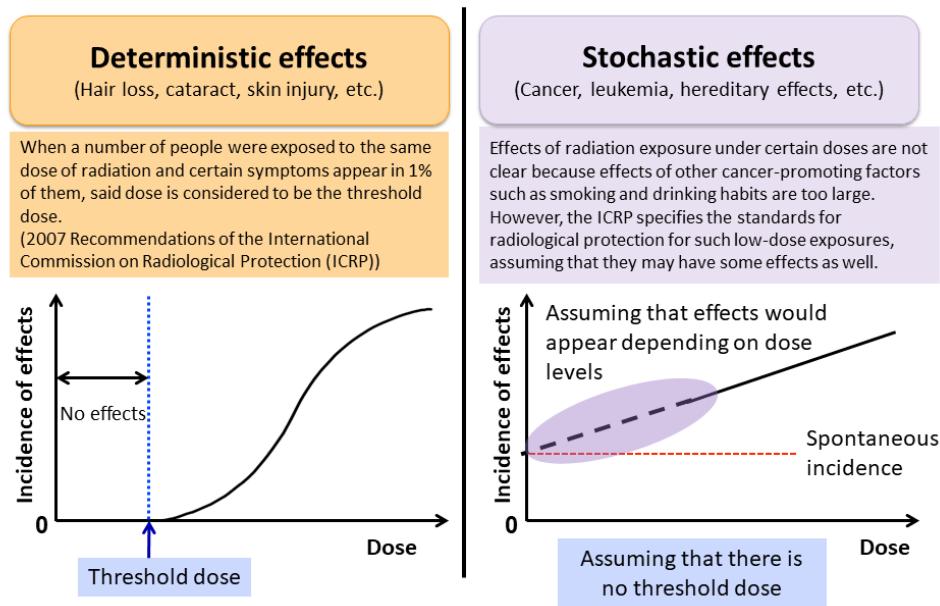


Figure 4: Deterministic and Stochastic Effects of Radiation [26]

- A.) **Deterministic Effects:** Deterministic effects, also known as non-stochastic effects, occur when an individual is exposed to high levels of radiation. These effects have a clear threshold dose below which no adverse effects are observed. As the dose increases above the threshold, the severity of the effects increases. Key deterministic effects include:
- **Radiation Sickness:** At high doses, radiation can cause radiation sickness, also known as acute radiation syndrome. Symptoms may include nausea, vomiting, fatigue, diarrhea, loss of appetite, and decreased blood cell counts. The severity and onset of symptoms depend on the radiation dose received.
 - **Skin Burns:** High doses of radiation can cause severe burns on the skin, similar to thermal burns. These burns can be painful and may require medical attention.
 - **Organ Damage:** Radiation exposure can result in damage to specific organs, such as the gastrointestinal system, cardiovascular system, and reproductive organs. The severity of organ damage depends on the dose received and the sensitivity of the organ to radiation.
- B.) **Stochastic Effects:** Stochastic effects, also called probabilistic effects, are random in nature and occur without a clear dose threshold. They are associated with long-term exposure to low or moderate doses of radiation. The probability of these effects occurring increases with higher radiation doses, but their occurrence is not guaranteed. Key stochastic effects include:
- **Increased Risk of Cancer:** Prolonged exposure to radiation, even at low doses, can increase the risk of developing cancer. Ionizing radiation has the potential to damage DNA, leading to genetic mutations that can initiate cancerous growth. The types of cancer that may develop depend on the irradiated organs and tissues.
 - **Hereditary Effects:** Radiation exposure can also affect future generations. High doses of radiation to reproductive cells (sperm or eggs) can increase the risk of genetic mutations and hereditary disorders in offspring.

It is important to note that the likelihood and severity of both deterministic and stochastic effects depend on factors such as the type of radiation (e.g., gamma rays, alpha particles), the duration of exposure, and the sensitivity of the exposed individual or organism. To protect individuals and mitigate the effects of radiation, radiation protection guidelines and regulations are implemented in various fields, including medicine, industry, and nuclear power. These measures aim to minimize radiation exposure, optimize radiation practices, and ensure the safety of workers and the general public.

Applications Of Radiation

Radiation has numerous applications across various fields, including medicine, industry, agriculture, research, and energy production. Here are some notable applications of radiation:

- **Medical Imaging:** Radiation is widely used in medical imaging techniques to diagnose and monitor diseases. X-rays and computed tomography (CT) scans utilize ionizing radiation to create detailed images of the body's internal structures, aiding in the detection of fractures, tumors, and other abnormalities.
- **Radiation Therapy:** Radiation therapy, also known as radiotherapy, is a common treatment modality for cancer. High-energy radiation, such as X-rays or gamma rays, is precisely targeted to destroy cancer cells and shrink tumors. Techniques like external beam radiation therapy and brachytherapy are used to deliver radiation to specific areas of the body.
- **Nuclear Medicine:** In nuclear medicine, small amounts of radioactive substances called radiopharmaceuticals are administered to patients for diagnostic and therapeutic purposes. These substances emit gamma rays that can be detected by imaging devices to create functional images of organs and tissues, aiding in the diagnosis and treatment of various conditions.
- **Industrial Applications:** Radiation is utilized in various industrial applications, including non-destructive testing (NDT) and quality control. Radiography techniques, such as X-ray and gamma-ray imaging, are employed to inspect welds, detect flaws in structures, and assess the integrity of materials without damaging them. Radiation is also used for sterilization of medical equipment, food preservation, and insect control in agriculture.
- **Research and Scientific Studies:** Radiation plays a crucial role in scientific research, enabling scientists to study the properties and behavior of materials, molecules, and subatomic particles. Techniques such as X-ray crystallography, neutron scattering, and positron emission tomography (PET) are employed to investigate the structure, composition, and dynamics of various substances.
- **Energy Production:** Nuclear power plants generate electricity by harnessing the energy released from nuclear reactions. Controlled fission of radioactive isotopes, such as uranium-235, produces heat, which is then converted into electrical energy. Nuclear energy provides a reliable and low-carbon source of power, contributing to the global energy mix.
- **Radiation Therapy for Food Preservation:** Ionizing radiation is used to preserve food by inhibiting the growth of bacteria, molds, and insects. This technique, known as food irradiation, can extend the shelf life of perishable foods, improve food safety, and prevent the spread of foodborne illnesses.

It is worth noting that the application of radiation is carefully regulated to ensure safety and minimize potential risks. Strict guidelines, protocols, and safety measures are in place to protect individuals, the environment, and public health in all areas where radiation is used.

Radiation Protection and Safety

Radiation protection and safety encompass a range of measures and practices aimed at minimizing radiation exposure and ensuring the safety of individuals, the public, and the environment [35]. The principles of radiation protection are based on the **ALARA** principle, which stands for "*As Low As Reasonably Achievable.*" as shown in figure 5

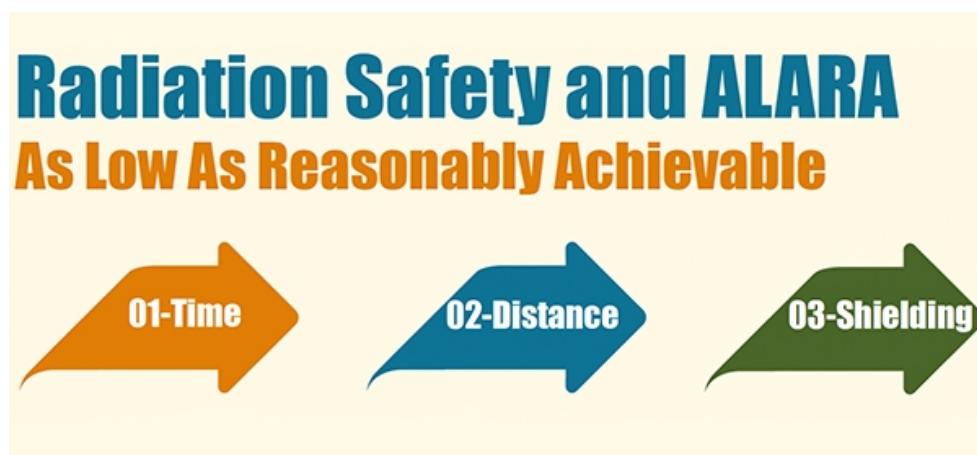


Figure 5: Principle of ALARA [25]

Here are some key aspects of radiation protection and safety:

- **Dose Limits:** Regulatory authorities and international organizations establish dose limits that specify the maximum allowable radiation doses for individuals in different contexts. These limits vary depending on factors such as occupation, public exposure, and medical procedures. Compliance with dose limits is essential to prevent excessive radiation exposure.
- **Risk Assessment and Management:** Radiation risks are assessed and managed by conducting thorough risk assessments. This involves evaluating the potential hazards associated with radiation sources, estimating the likelihood and magnitude of exposures, and implementing appropriate control measures to mitigate risks.
- **Shielding and Containment:** Shielding materials, such as lead, concrete, and steel, are used to attenuate radiation and protect individuals from exposure. Proper shielding design and construction are crucial in areas where radiation sources are used, such as medical facilities and nuclear power plants. Additionally, containment measures ensure that radioactive materials are safely stored and handled to prevent their release into the environment.
- **Personal Protective Equipment (PPE):** Personal protective equipment, such as lead aprons, gloves, and goggles, is utilized to protect individuals who work with or around radiation sources. PPE helps to reduce radiation exposure to specific body parts and ensures that workers adhere to safety protocols.
- **Training and Education:** Comprehensive training and education programs are essential for individuals who work with radiation sources. This includes radiation safety training, radiation protection protocols, proper handling and storage of radioactive materials, and emergency response procedures. Continuous education and awareness campaigns help maintain a strong safety culture.
- **Monitoring and Dosimetry:** Regular monitoring of radiation levels and individual doses is conducted to ensure compliance with safety standards and detect any potential overexposures. Dosimeters, such as **Thermoluminescent dosimeters (TLDs)** and electronic personal dosimeters (EPDs), are used to measure and record radiation doses received by individuals.
- **Regulatory Compliance and Inspections:** Regulatory bodies oversee and enforce radiation safety regulations. They conduct inspections, audits, and assessments to ensure that radiation practices comply with safety guidelines. Non-compliance can result in penalties, fines, or the suspension of operations.
- **Emergency Preparedness and Response:** Preparedness for radiation emergencies, such as accidents or incidents involving radiation sources, is crucial. Emergency response plans, evacuation procedures, and communication protocols are established to protect the public and mitigate the consequences of such incidents.

It is important to note that radiation protection and safety require a multidisciplinary approach involving collaboration among regulatory bodies, radiation safety officers, medical professionals, researchers, and workers handling radiation sources. Continuous improvement, adherence to best practices, and staying up-to-date with advancements in radiation protection are vital to ensuring the safe and responsible use of radiation.

1.2 Radiation Dosimetry

Radiation Dosimetry[12] is the science and practice of measuring and assessing the dose of ionizing radiation received by an object or an individual. It plays a crucial role in radiation protection, medical diagnostics, and therapeutic applications. By accurately quantifying radiation doses, dosimetry enables the evaluation of potential health risks, optimization of radiation procedures, and adherence to safety standards.

Measurement Techniques

Dosimetry measurement techniques[3] are used to directly or indirectly quantify the dose of ionizing radiation received by an object or an individual. These techniques vary depending on the type of radiation, the purpose of measurement, and the specific application. Here are some commonly used dosimetry measurement techniques:

- **Ionization Chambers:** Ionization chambers are widely employed in radiation dosimetry. They consist of a gas-filled chamber where ionization occurs when radiation interacts with the gas molecules. The resulting ion pairs are collected, and the electrical current generated is proportional to the radiation dose. Ionization chambers are versatile and can measure both high and low radiation doses accurately.
- **Thermoluminescent Dosimeters (TLDs):** TLDs are solid-state dosimeters that utilize the phenomenon of thermoluminescence. When exposed to ionizing radiation, certain crystals or materials trap energy within their lattice structure. Heating the TLD causes the trapped energy to be released as light, which is measured and correlated to the radiation dose. TLDs offer high sensitivity and can be used for personal and environmental dosimetry.
- **Film Dosimeters:** Film dosimeters use radiation-sensitive films, such as radiographic films or photographic emulsions, to measure radiation doses. The films darken when exposed to radiation, and the degree of darkening is related to the absorbed dose. Film dosimeters are commonly used in medical imaging, industrial radiography, and radiation therapy verification.
- **Solid-State Detectors:** Solid-state detectors employ semiconductor materials, such as silicon or germanium, to measure radiation doses. These detectors produce an electrical signal in response to ionizing radiation. Examples of solid-state detectors include diode detectors, silicon detectors, and metal oxide semiconductor field-effect transistors (MOSFETs). Solid-state detectors offer high precision, small size, and real-time dose measurement capabilities.
- **Scintillation Detectors:** Scintillation detectors utilize scintillating materials that emit light when exposed to radiation. The scintillation light is converted into electrical signals using photomultiplier tubes or photodiodes. Scintillation detectors are commonly used in nuclear medicine, environmental monitoring, and high-energy physics research.
- **Optically Stimulated Luminescence (OSL) Dosimeters:** OSL dosimeters use similar principles to TLDs but employ different materials. OSL dosimeters contain optically sensitive materials that store radiation energy. When exposed to light, the stored energy is released as luminescence, which is proportional to the radiation dose. OSL dosimeters are widely used in personal and environmental dosimetry.
- **Biological Dosimeters:** Biological dosimeters measure the biological effects of radiation on living cells or tissues. These dosimeters include techniques such as chromosomal aberration analysis, cytogenetic biodosimetry, and biological marker assays. Biological dosimeters are particularly useful in radiation accidents or emergencies to assess radiation exposure and estimate potential health risks.
- **Environmental Monitoring Instruments:** Environmental monitoring instruments, such as Geiger-Muller counters and scintillation detectors, are used to measure ambient radiation levels in the environment. These instruments provide real-time or continuous monitoring of radiation doses in occupational settings, nuclear facilities, or areas with potential radioactive contamination.

It is worth noting that dosimetry measurement techniques are often complemented by data analysis, calibration, and quality assurance procedures to ensure accurate and reliable results. The choice of dosimetry technique depends on factors such as the radiation type, dose range, required sensitivity, portability, and specific dosimetry application.

Types of Radiation Dosimetry

Radiation dosimetry encompasses different types of radiation and corresponding dosimetry techniques:

- **External Beam Dosimetry:** This type of dosimetry is employed in external beam radiation therapy, diagnostic imaging (e.g., X-rays, CT scans), and radiation protection. It focuses on measuring radiation doses delivered from an external radiation source to a specific target or region of interest.
- **Internal Dosimetry:** Internal dosimetry involves the assessment of radiation doses from internal radiation sources, such as radioactive materials ingested, inhaled, or injected into the body. It requires the estimation of radionuclide uptake, distribution, and elimination to calculate the radiation dose received by specific organs or tissues.
- **Environmental Dosimetry:** Environmental dosimetry aims to evaluate radiation doses in the environment, particularly in occupational settings or areas with potential radioactive contamination. It includes monitoring radiation levels in air, water, soil, and other environmental media to assess potential exposure risks.

Properties of Ideal dosimeter

An ideal dosimeter is a device or material that possesses certain key properties to accurately and reliably measure radiation doses. Here are some properties of an ideal dosimeter[30]:

- **Accuracy:** An ideal dosimeter should provide accurate measurements of radiation doses. It should have a linear response, meaning the measured dose should be directly proportional to the actual radiation dose received. The dosimeter should minimize uncertainties and errors in dose measurements.
- **Sensitivity:** The dosimeter should be highly sensitive to radiation, capable of detecting even low levels of radiation exposure. It should be able to measure doses accurately within the desired range, allowing for precise dose quantification.
- **Stability:** An ideal dosimeter should exhibit stability over time, meaning its response to radiation doses remains consistent throughout its operational lifespan. It should not show significant changes in sensitivity or response due to environmental factors or aging.
- **Energy Dependence:** The dosimeter should have minimal energy dependence, meaning it should provide consistent dose measurements across a broad range of radiation energies and types. This property ensures that the dosimeter can accurately measure doses from various radiation sources.
- **Linearity:** The dosimeter's response should be linear across a wide range of dose levels. This linearity ensures that the measured dose is directly proportional to the actual dose received, simplifying dose calculations and interpretations.
- **Tissue-Equivalent:** An ideal dosimeter should mimic the radiation absorption characteristics of human tissue. It should have similar energy deposition properties to accurately represent the dose received by biological tissues. Tissue-equivalent dosimeters provide more accurate measurements of the dose absorbed by human tissues in medical applications.
- **Readout Convenience:** The dosimeter should allow for easy and convenient readout of the accumulated dose. This could involve simple readout techniques, such as visual or electronic methods, allowing for efficient dose retrieval and interpretation.
- **Durability:** The dosimeter should be robust and durable, capable of withstanding environmental conditions and handling during usage. It should maintain its properties and performance even under challenging operational conditions.
- **Cost-effectiveness:** An ideal dosimeter should be cost-effective, considering factors such as the manufacturing cost, ease of use, and maintenance requirements. It should provide accurate dose measurements at a reasonable cost, making it accessible for various applications and users.
- **Radiation Type Independence:** The dosimeter should exhibit consistent response across different types of ionizing radiation, such as gamma rays, X-rays, and beta particles. This property ensures that the dosimeter can be used in diverse radiation environments without significant variations in its performance.

While it is challenging to achieve a dosimeter that meets all these ideal properties, ongoing research and development aim to improve dosimetry technologies and materials to enhance accuracy, sensitivity, and reliability in radiation dose measurements.

Applications of Radiation Dosimetry:

Radiation dosimetry has numerous practical applications:

- **Radiation Therapy:** In cancer treatment, radiation dosimetry is crucial for accurately delivering therapeutic radiation doses to tumor sites while minimizing radiation exposure to healthy tissues. Precise dosimetry measurements ensure effective treatment planning and monitoring of radiation therapy procedures.
- **Radiation Protection:** Dosimetry is essential for ensuring the safety of workers in radiation-related occupations. Occupational dosimetry involves monitoring radiation doses received by individuals to ensure compliance with safety regulations and implement necessary protective measures.
- **Radiological Accidents and Emergency Response:** Dosimetry plays a vital role in assessing radiation doses received by individuals involved in radiological accidents or emergencies. It aids in triaging patients, determining appropriate medical treatments, and evaluating potential long-term health effects.
- **Radiological Research:** Dosimetry is instrumental in research involving radiation, such as studying the effects of radiation on biological systems, evaluating radiation shielding materials, and optimizing radiation therapy techniques.

In summary, radiation dosimetry is an indispensable field that enables the measurement and assessment of radiation doses. By employing various measurement techniques, dosimetry contributes to radiation protection, medical treatments, emergency response, and scientific research. Accurate dosimetry practices are vital in ensuring the safe and effective use of ionizing radiation in various applications while minimizing potential health risks.

1.3 Thermoluminescence Dosimetry

Thermoluminescence Dosimetry (TLD) is a technique used to measure and assess radiation doses by exploiting the thermoluminescent properties of certain materials. It is widely employed in various fields, including medical radiation therapy, industrial radiation safety, environmental monitoring, and personal dosimetry.

The basic principle of thermoluminescence dosimetry involves the use of thermoluminescent materials, which are crystalline or amorphous substances capable of storing energy when exposed to ionizing radiation. These materials can be in the form of powders, pellets, or chips. Common thermoluminescent materials include lithium fluoride (LiF), calcium fluoride (CaF_2), and aluminum oxide ($Al_2O_3 : C$). A Thermoluminescence Dosimeter is shown in figure6

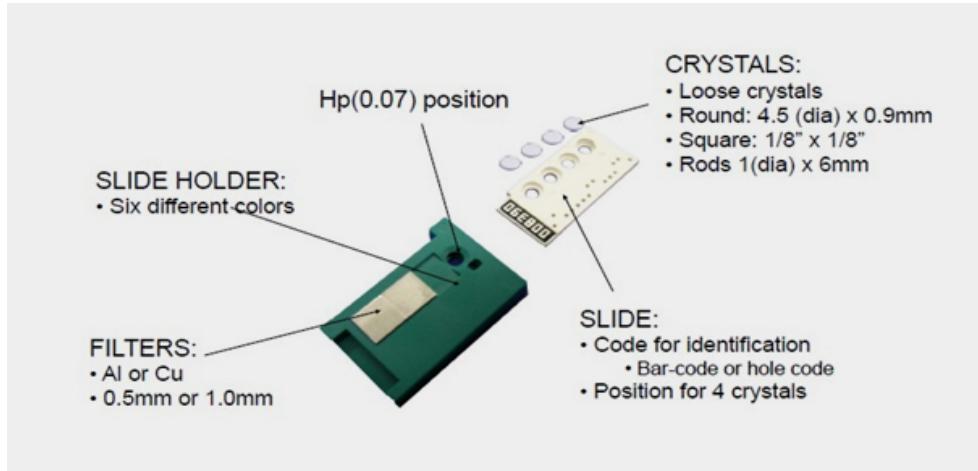


Figure 6: A thermoluminescent dosimeter[19]

Thermoluminescence

Thermoluminescence (TL) is a luminescence phenomenon[6] of an insulator or semiconductor which can be observed when the solid is thermally stimulated. In order for a material to be thermoluminous, it must first be an insulator or a semiconductor because metals do not show thermoluminescence. Secondly, energy must have been absorbed by the substance at some point while it was exposed to ionising radiation. Thirdly, heating the material causes the luminescence emission to occur. Thus, a thermoluminescent material is one that, when subjected to ionising radiation, absorbs some energy that is then stored. When the substance is later heated, the accumulated energy is released in the form of luminescence.[7]

There are many models that explain the phenomenon of thermoluminescence[13], one such model is the one trapping—one recombination centre model[24] that uses the energy band theory of solids. The majority of the electrons in an ideal crystalline semiconductor or insulator are found in the valence band. The conduction band, which is above the valence band and is separated from it by the so-called forbidden band gap (separated by energy E_g), is the highest band that electrons can inhabit. However, when a crystal has structural flaws or the lattice contains impurities, it is possible for electrons to have energies that are not permitted in a perfect crystal. In a simple TL model two levels are assumed, one situated below the bottom of the conduction band and the other situated above the top of the valence band as shown in figure7.

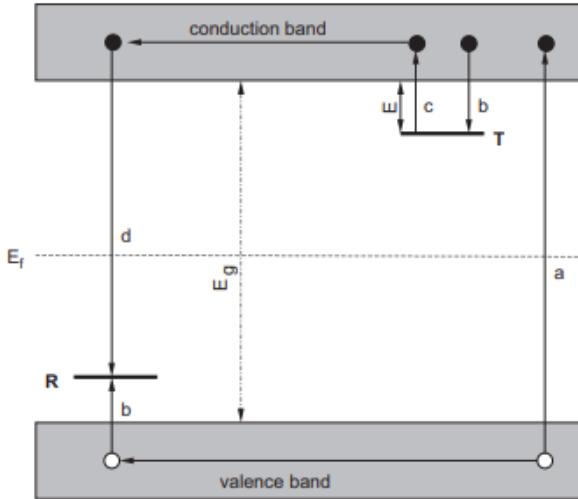


Figure 7: Energy band model showing the electronic transitions in a TL material according to a simple two-level model.[6]

The highest level is situated above the equilibrium Fermi level (E_f) and thus empty in the equilibrium state, i.e before the exposure to radiation and the creation of electrons and holes. It is therefore a potential electron trap. The other level is a potential hole trap and can function as a recombination centre. The absorption of radiant energy with hE_g results in ionisation of valence electrons, producing energetic electrons and holes which will, after thermalisation, produce free electrons in the conduction band and free holes in the valence band. The free charge carriers recombine with each other or become trapped.

An amount of energy will be generated in the case of direct recombination, which may stimulate a luminescent centre (which might coincide with the recombination centre). Under the influence of light emission, the luminescent centre relaxes (returns to the ground state). However, in semiconductors and insulators a certain percentage of the charge carriers is trapped. As the free electrons and holes are created and annihilated in pairs, there must be an equal population of trapped holes. Because the normal equilibrium Fermi level E_f is situated below level T and above level R (as shown in figure 7), these populations of trapped electrons and holes represent a non-equilibrium state.

By increasing the temperature of the TL material above T_0 , the return to equilibrium can be accelerated. The likelihood of detrapping will rise as a result, and the electrons will then be liberated from the trap and enter the conduction band. Until it undergoes recombination at the recombination centre R , the charge carrier migrates through the crystal's conduction band. In the simple model this recombination centre is a luminescent centre where the recombination of the electron and hole leaves the centre in one of the higher excited states. Return to the ground state is coupled with the emission of light quanta, which give rise to the phenomena of **Thermoluminescence**.

Process of Thermoluminescence Dosimetry

The TLD typically involves the following steps:

1. **Pre-Irradiation:** The thermoluminescent material is first subjected to a controlled pre-irradiation process. This step ensures that any residual luminescence from previous radiation exposures is removed, allowing the material to return to its baseline state.
2. **Irradiation:** The pre-irradiated thermoluminescent material is then exposed to ionizing radiation. The type of radiation and its energy will depend on the specific application and purpose of the dosimetry measurement.
3. **Post-Irradiation:** After irradiation, the thermoluminescent material retains energy in the form of trapped electrons within defects or lattice imperfections. The sample is carefully protected from any light exposure to prevent the release of the stored energy prematurely.
4. **Heating Process:** When the thermoluminescent material is heated, the trapped electrons are released from their energy states and recombine with the trapped holes, resulting in the emission of light in the form of thermoluminescence. The emitted light is proportional to the radiation dose received by the material.

- Light Detection:** The emitted thermoluminescent light is detected using specialized photomultiplier tubes, photodiodes, or other light-detecting devices. The detected light signal is then converted into an electrical signal for further analysis.
- Analysis and Dose Calculation:** The electrical signal is analyzed to determine the intensity or luminescent response of the thermoluminescent material. This response is correlated with a calibration curve or conversion factors established through calibration with known radiation doses. By comparing the luminescent response to the calibration curve, the radiation dose received by the material can be determined.

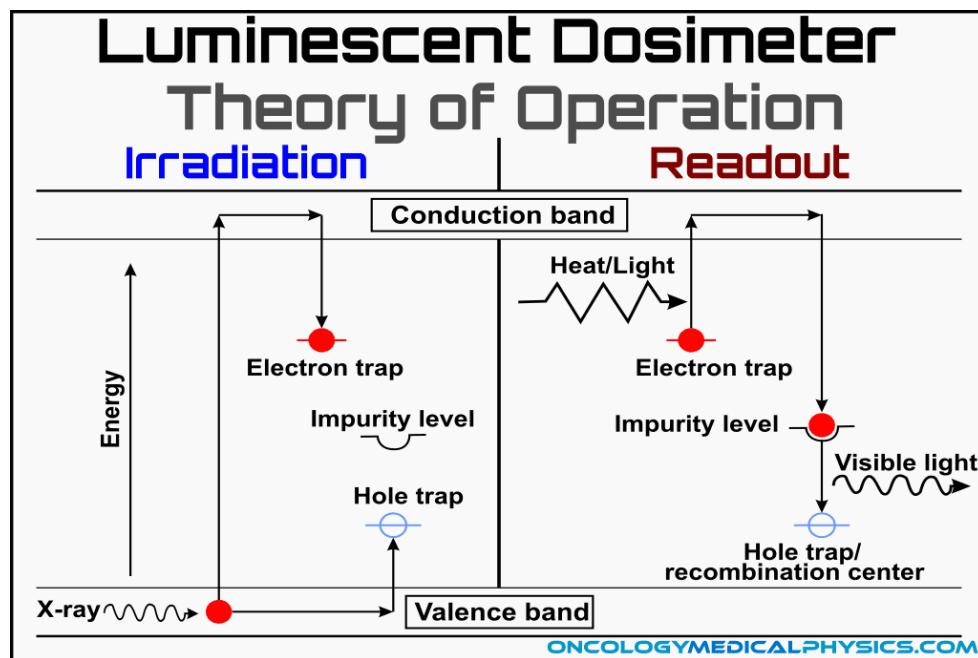


Figure 8: Theory of operation behind thermoluminescent dosimeters[29]

Applications of TLD

Thermoluminescence dosimetry (TLD) has numerous applications in various fields where accurate measurement of radiation doses is essential. Some of the key applications of thermoluminescence dosimetry are:

- Radiation Therapy:** In medical radiation therapy, TLD is used to measure the radiation dose delivered to patients during treatments such as external beam radiation therapy or brachytherapy. TLD allows for precise and reliable dose verification, ensuring that the prescribed radiation dose is accurately delivered to the target area while minimizing exposure to healthy tissues.
- Occupational Radiation Monitoring:** TLD is extensively used in industries involving radiation sources, such as nuclear power plants, radiography facilities, and research laboratories. Workers who are potentially exposed to ionizing radiation wear TLD badges or rings, which contain TLD materials. These dosimeters are then analyzed to assess the radiation dose received by the workers, ensuring compliance with safety regulations and maintaining a safe working environment.
- Environmental Radiation Monitoring:** TLD is employed in environmental radiation monitoring to assess radiation levels in the environment, including soil, air, and water. It helps identify potential sources of radiation and monitor any changes or increases in radiation levels that could pose risks to human health or the environment. TLD is particularly valuable in assessing long-term exposure and cumulative dose assessments in environmental studies.
- Personal Dosimetry:** TLD is used in personal dosimetry to measure individual radiation exposure in various settings. This includes personnel working with radiation sources in nuclear facilities, research laboratories, and industrial settings. TLD badges or rings worn by individuals capture their radiation exposure, which can be analyzed to assess dose levels and ensure compliance with safety regulations.

- **Radiation Protection:** TLD is employed in the design and evaluation of radiation shielding materials and techniques. It allows for the measurement of radiation doses behind shielding materials to ensure their effectiveness in reducing exposure. TLD is also used in quality assurance tests for radiation protection equipment, such as lead aprons and protective barriers, to verify their performance and ensure proper shielding.
- **Radiation Emergency Response:** In the event of a radiological incident or accident, TLD can be used to assess the radiation doses received by affected individuals. TLD badges or rings can be distributed to emergency responders and workers involved in recovery operations, providing valuable data on their radiation exposure and guiding appropriate medical interventions.
- **Archaeological Dating:** TLD is utilized in archaeological dating to determine the age of ancient artifacts and geological samples. By measuring the thermoluminescence emitted from the material, the accumulated radiation dose can be estimated, providing insights into the time since the material was last heated or exposed to sunlight.

Thermoluminescence dosimetry offers several advantages, including high sensitivity, wide dynamic range, and excellent tissue-equivalent properties. It is capable of measuring both high and low radiation doses, making it suitable for various applications. Additionally, TLD materials have good reproducibility and stability, allowing for reliable and accurate dose measurements over time.

1.4 Nanophosphors in Dosimetry

Nanophosphors, also known as nanocrystalline phosphors or nanoparticles, have emerged as promising materials for various applications, including lighting, displays, and imaging. These nanoscale phosphors possess unique properties that make them highly desirable in these fields. Here are some key aspects of nanophosphors[8]:

- **Size and Composition:** Nanophosphors are typically composed of crystalline materials, such as oxides, sulfides, or silicates, that emit light when excited by external energy sources. They are synthesized as nanoparticles with controlled sizes ranging from a few nanometers to several tens of nanometers. The composition of nanophosphors can be tailored to achieve specific desired properties, including emission wavelength and intensity.
- **Luminescent Properties:** Nanophosphors exhibit excellent luminescent properties, including high quantum efficiency and emission stability. When stimulated by an appropriate energy source, such as ultraviolet (UV) light or X-rays, they absorb energy and subsequently emit light in a process called luminescence. This emitted light can be tuned to different wavelengths depending on the composition and size of the nanophosphor.
- **Energy Transfer:** Nanophosphors can undergo energy transfer processes, where absorbed energy is efficiently transferred from one nanophosphor to another. This property is particularly useful in applications where multiple nanophosphors are combined to achieve desired emission colors or to enhance the overall emission efficiency.
- **Narrow Emission Bandwidth:** Nanophosphors typically exhibit a narrow emission bandwidth, which allows for sharper and more precise color generation. This property is beneficial for applications requiring high color purity, such as display technologies.
- **Stability and Longevity:** Nanophosphors are known for their stability and resistance to degradation over time. They have a long lifespan and can maintain their luminescent properties even under harsh environmental conditions, making them suitable for long-term applications.
- **Compatibility and Integration:** Nanophosphors can be integrated into various host matrices, including polymers, glasses, or ceramics, to create functional materials with tailored properties. This compatibility allows for their incorporation into a wide range of devices and systems, such as light-emitting diodes (LEDs), scintillators, and optical sensors.
- **Bioimaging and Biomedical Applications:** Nanophosphors have gained attention in the field of bioimaging and biomedical applications. Their small size, stability, and tunable emission properties make them attractive for fluorescent labeling, molecular imaging, and targeted drug delivery systems.

Nanophosphors hold significant potential in advancing various technological and biomedical applications due to their unique properties, including size control, luminescent efficiency, stability, and tunability. Nanophosphors have found significant uses in the field of radiation dosimetry due to their unique properties which make it extremely suitable for use in dosimeters.

Nanophosphors in Dosimetry

Nanophosphors, also known as nanocrystalline phosphors or nanoparticles, have emerged as promising materials for dosimetry applications. These nanoscale phosphors exhibit unique properties that make them highly suitable for radiation dosimetry. Here are some key aspects of nanophosphors in dosimetry:

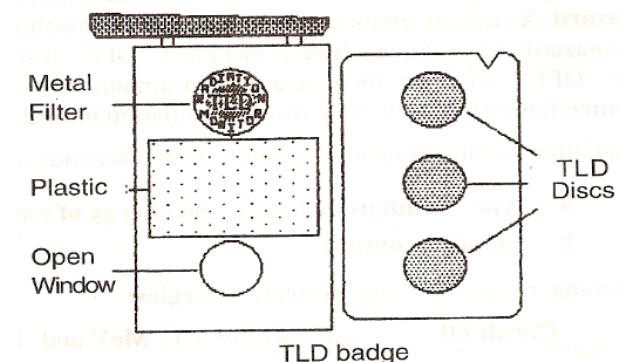


Figure 9: Nanophosphors being used in TLD badges[9]

- **Enhanced Sensitivity:** Nanophosphors possess a high surface-to-volume ratio due to their small size, resulting in increased sensitivity to radiation. This enhanced sensitivity allows for precise and accurate measurements of radiation doses, even at low levels.
- **Stability and Retention:** Nanophosphors are known for their long-term stability and low fading rates, which ensures the reliability of dose measurements over time. They can retain the stored energy and exhibit thermoluminescence or optically stimulated luminescence properties, allowing for subsequent readout and analysis.
- **Size-Tunable Properties:** The size of nanophosphors can be precisely controlled during synthesis, allowing tailoring of their optical and luminescent properties. By manipulating the nanoparticle size, the emission wavelength and intensity can be tuned to match specific dosimetry requirements.
- **Tissue-Equivalent Properties:** Some nanophosphors have tissue-equivalent characteristics, meaning they exhibit similar radiation response to human tissues. This property is crucial in medical dosimetry, where accurate measurement of radiation doses delivered to different tissues is essential.
- **Multifunctionality:** Nanophosphors can be functionalized with various coatings or surface modifications, enabling additional functionalities. For example, they can be embedded in polymeric matrices to form composite dosimeters with enhanced mechanical properties. Nanophosphors can also be conjugated with targeting molecules for specific applications, such as targeted radiation therapy.
- **Real-Time Monitoring:** The small size and compatibility of nanophosphors with different matrices facilitate their integration into real-time monitoring systems. They can be incorporated into sensors or wearable devices, providing continuous monitoring of radiation exposure in various environments.

2 Synthesis and Characterization of Nanophosphors

The synthesis and characterization of nanophosphors for thermoluminescence dosimetry involve several steps to prepare the nanocrystalline phosphor materials and evaluate their properties. Here is a general overview of the process:

2.1 Synthesis Of Nanophosphors

Synthesis techniques for nanophosphors involve the preparation of nanocrystalline materials with controlled size, composition, and luminescent properties. Various methods are used to achieve this, and here are some commonly employed synthesis techniques for nanophosphors:

2.1.1 Co-Precipitation

The **co-precipitation method**[16] is a commonly used technique for the synthesis of nanophosphors, including those intended for thermoluminescence dosimetry. This method involves the simultaneous precipitation of metal cations and anions from a solution, resulting in the formation of nanocrystalline particles. The co-precipitation method offers advantages such as simplicity, cost-effectiveness, and scalability for the synthesis of nanophosphors. However, careful control of reaction conditions and post-treatment steps is essential to achieve desired particle properties and optimize the dosimetric performance of the nanophosphors for thermoluminescence dosimetry applications. Here is an overview of the co-precipitation method shown in[10]:

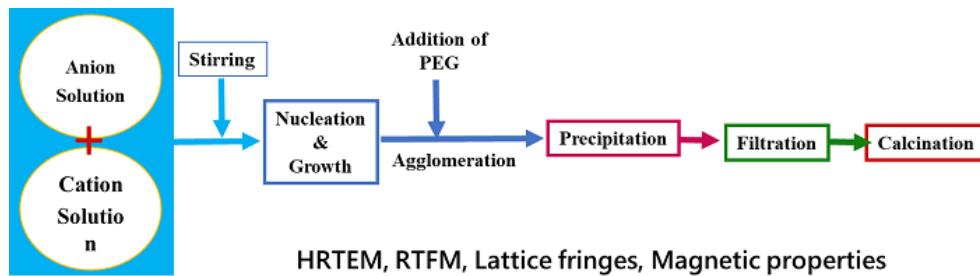


Figure 10: Synthesis using Co-Precipitation Method[37]

- **Selection of Precursor Salts:** Choose appropriate precursor salts that contain the desired metal cations and anions for the nanophosphor composition. The choice of precursors depends on the specific material being synthesized.
- **Preparation of Precursor Solution:** Dissolve the precursor salts in a suitable solvent to prepare a precursor solution. The solvent should facilitate the dissolution of the precursors and promote homogeneous mixing.
- **Control of Reaction Conditions:** Adjust the reaction conditions, including temperature, pH, and reaction time, to control the nucleation and growth of the nanophosphor particles. The reaction conditions play a crucial role in determining the size, composition, and crystallinity of the resulting nanophosphors.
- **Precipitating Agent Addition:** Add a precipitating agent to the precursor solution to induce the formation of the nanophosphor particles. The choice of precipitating agent depends on the specific reaction system and desired properties of the nanophosphors. Commonly used precipitating agents include ammonium hydroxide (NH_4OH), sodium hydroxide ($NaOH$), or carbonate solutions.
- **Precipitation and Aging:** Upon the addition of the precipitating agent, the metal cations and anions react to form insoluble salts, resulting in the precipitation of nanocrystalline particles. Allow the precipitates to age for a certain period, typically through continuous stirring or aging at a specific temperature, to promote particle growth and improve crystallinity.
- **Filtration and Washing:** Separate the formed nanophosphor precipitates from the solution by filtration. Wash the precipitates multiple times with a suitable solvent or deionized water to remove any residual impurities or unreacted ions.
- **Drying and Calcination:** Dry the filtered nanophosphor precipitates at a controlled temperature to remove the solvent and residual moisture. Depending on the specific material, the dried precipitates may undergo a subsequent calcination process to enhance crystallinity and remove any remaining organic species.

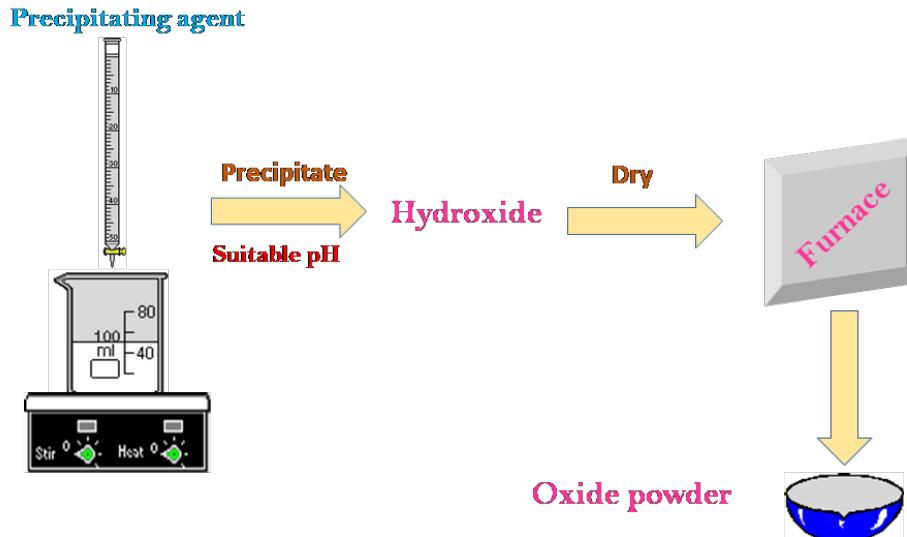


Figure 11: Schematic diagram for Co-Precipitation Method[18]

2.1.2 Solid State Reaction

The **solid-state reaction**[10] method is a commonly used technique for the synthesis of various materials, including nanophosphors. It involves the direct reaction between solid precursor materials to form the desired product. The solid-state reaction method offers simplicity, versatility, and the ability to control the reaction conditions for the synthesis of nanophosphors. However, it may require high temperatures and longer reaction times to ensure complete reaction and achieve the desired crystallinity. Optimization of the heat treatment parameters is crucial to obtain nanophosphors with the desired properties and luminescent behavior for thermoluminescence dosimetry applications. Here is an overview of the solid-state reaction method:

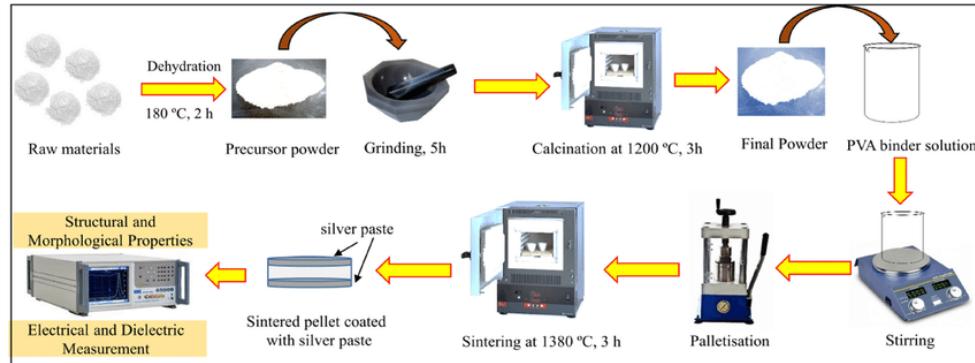


Figure 12: Synthesis using Solid State Reaction Method[17]

- **Selection of Precursor Materials:** Choose solid precursor materials that contain the necessary elements for the composition of the nanophosphor. These precursors may be in the form of powders or solid compounds.
- **Weighing and Mixing:** Accurately weigh and mix the precursor materials in the desired stoichiometric ratio. The mixing can be done using mortar and pestle, ball milling, or other mechanical methods to ensure a homogenous mixture.
- **Grinding and Milling:** If the precursor materials are not already in the form of fine powders, grinding and milling steps may be required to reduce their particle size and improve their reactivity. This step facilitates intimate contact and diffusion of the reactants during the subsequent heat treatment.
- **Pelletization or Pressing:** Depending on the application and desired final form of the nanophosphor, the mixture of precursor powders may be pressed into pellets or compacted using a hydraulic press to obtain a solid compact.

- **Heat Treatment (Calcination):** The compacted precursor mixture is subjected to heat treatment in a furnace. The temperature and duration of the heat treatment are critical to promote the desired chemical reactions and facilitate the formation of the nanophosphor phase. The heat treatment process is often referred to as calcination.
- **Cooling and Grinding:** After the heat treatment, the sample is allowed to cool to room temperature. It is then ground or milled again to break up any agglomerates and obtain a fine powder of the nanophosphor.

2.1.3 Combustion Method

The **combustion method**[20], also known as the combustion synthesis or self-sustained combustion method, is a versatile and rapid technique for the synthesis of various materials, including nanophosphors. It involves the exothermic reaction between fuel and oxidizer precursors, resulting in a self-sustained combustion process that generates high temperatures and promotes the formation of the desired product. The combustion method offers several advantages, including simplicity, rapid synthesis, and the ability to produce nanophosphors with fine particle sizes and high purity. The high temperatures generated during the combustion process promote the formation of crystalline nanophosphors with controlled properties. However, careful control of the stoichiometry and reaction conditions is crucial to ensure reproducibility and obtain the desired product. Here is an overview of the combustion method:

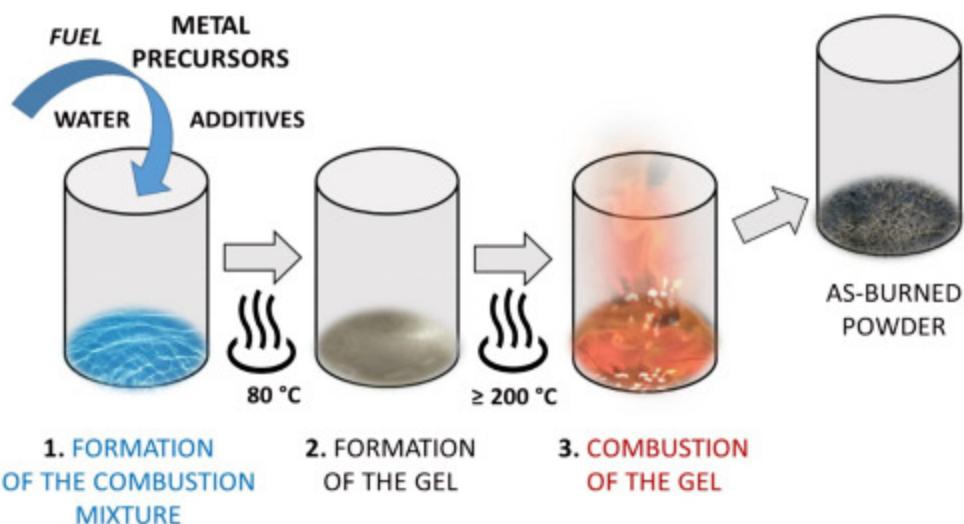


Figure 13: Synthesis using Combustion Method[36]

- **Selection of Precursor Materials:** Choose appropriate precursor materials that contain the necessary elements for the composition of the nanophosphor. These precursors typically include a fuel and an oxidizer.
- **Weighing and Mixing:** Accurately weigh and thoroughly mix the fuel and oxidizer precursors in the desired stoichiometric ratio. The mixing process is crucial to ensure a homogenous distribution of the reactants and promote a complete reaction.
- **Pelletization or Pressing:** Depending on the application and desired final form of the nanophosphor, the mixture of precursor powders may be pressed into pellets or compacted using a hydraulic press to obtain a solid compact.
- **Ignition:** Apply an external ignition source, such as a spark or a flame, to initiate the combustion reaction. The exothermic reaction between the fuel and oxidizer precursors releases a significant amount of heat, leading to self-sustained combustion.
- **Combustion Reaction:** The combustion reaction generates high temperatures, often exceeding $1000\text{ }^{\circ}\text{C}$, which facilitates rapid reaction kinetics and the formation of the desired nanophosphor. The reaction is typically completed within seconds to minutes.
- **Cooling and Grinding:** After the combustion reaction is complete, the sample is allowed to cool to room temperature. The resulting product is typically in the form of a porous solid or powder. Grinding or milling can be performed to break up any agglomerates and obtain a fine powder of the nanophosphor.

2.2 Annealing

Annealing is a heat treatment process that involves heating a material to a specific temperature and holding it at that temperature for a certain period, followed by controlled cooling. The purpose of annealing is to modify the material's properties, such as its microstructure, mechanical strength, electrical conductivity, and thermal stability. Annealing is widely used in various fields, including metallurgy, materials science, and semiconductor manufacturing. During the annealing process, several changes occur within the material:

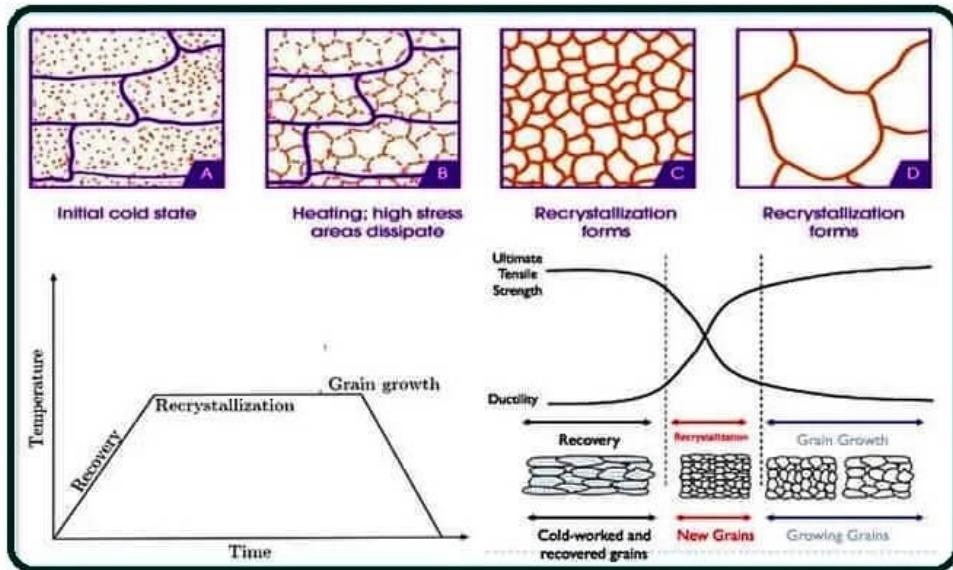


Figure 14: Various stages of annealing[14]

- **Recrystallization:** Annealing promotes the formation of new, defect-free crystals within the material. It helps eliminate dislocations and other crystal defects that may have occurred during processing or deformation, leading to the growth of larger, more regular grains.
- **Stress Relief:** Annealing helps relieve internal stresses within the material, which can result from processes like casting, forming, or rapid cooling. By heating the material to a specific temperature and holding it there, the internal stresses can be reduced, leading to improved dimensional stability and reduced risk of cracking or distortion.
- **Grain Growth:** Annealing allows for the controlled growth of grains within the material. This is particularly important in polycrystalline materials, where larger grains can enhance mechanical properties, such as strength and toughness.
- **Phase Transformation:** In some cases, annealing induces phase transformations within the material, leading to changes in its composition and crystal structure. This can result in the formation of desired phases with improved properties

2.3 Characterization Of Nanophosphors

Characterization of nanophosphors involves the use of various analytical techniques to evaluate their structural, morphological, optical, and luminescent properties. The characterization techniques provide valuable information about the composition, crystal structure, particle size, shape, surface properties, and luminescent behavior of the nanophosphors. Here are some commonly used characterization techniques for nanophosphors:

2.3.1 X-ray Diffraction

X-ray diffraction (XRD)[38] is a widely used technique for characterizing the crystal structure, composition, and phase identification of materials, including nanophosphors. It relies on the principle of X-ray scattering by the crystal lattice, providing information about the spacing and arrangement of atoms within the material. X-ray diffraction is a powerful technique for investigating the crystallographic properties of nanophosphors, including their crystal structure, phase composition, and crystallite size. It is a non-destructive and highly informative characterization tool used in materials science, chemistry, geology, and many other fields. Here is an overview of X-ray diffraction:



Figure 15: A X-ray diffraction machine[23]

- **Principle:** XRD involves exposing a sample to a beam of X-rays and measuring the resulting diffraction pattern. X-rays interact with the crystal lattice, causing constructive interference of scattered waves. The angles and intensities of the diffracted X-rays provide information about the crystal structure and spacing of atomic planes.
- **Bragg's Law:** The diffraction pattern is described by Bragg's law, which relates the angle of incidence (θ), the wavelength of the X-rays (λ), and the distance between crystal planes (d) in the material: $n\lambda = 2dsin(\theta)$, where n is an integer representing the order of diffraction.
- **Sample Preparation:** The nanophosphor sample is typically prepared as a powder or thin film to ensure a random orientation of the crystal planes. The sample should be finely ground to ensure a homogeneous scattering of X-rays.
- **X-ray Source:** XRD uses a monochromatic X-ray source, often a high-intensity source like a rotating anode X-ray tube or a synchrotron. The wavelength of the X-rays is selected depending on the material and the desired diffraction information.
- **Diffraction Pattern Measurement:** The X-rays diffracted by the sample are captured by a detector, typically a scintillation or solid-state detector. The detector records the intensity of the diffracted X-rays as a function of the diffraction angle (2θ). The diffraction pattern is a series of peaks representing the diffracted intensity as a function of the diffraction angle.
- **Data Analysis:** The diffraction pattern is analyzed using specialized software to determine the crystal structure and phase identification. The positions, intensities, and shapes of the diffraction peaks are compared with reference databases to identify the crystal structure and phase composition of the nanophosphor.
- **Quantitative Analysis:** XRD can also provide quantitative information about the crystalline phases present in the sample. By comparing the intensity of the diffracted peaks with known standards or through peak fitting analysis, the relative abundance of different phases can be determined.

2.3.2 Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM)[11] is a powerful imaging technique used to examine the surface morphology and composition of materials, including nanophosphors. It provides high-resolution images and allows for detailed analysis of the sample's topography, particle size, shape, and surface features. SEM is widely used in materials science, nanotechnology, biology, geology, and various other fields to investigate the surface morphology and composition of nanophosphors and other materials. It offers high-resolution imaging capabilities and provides valuable insights into the structural and surface properties of nanophosphors, aiding in their characterization and understanding for various applications.

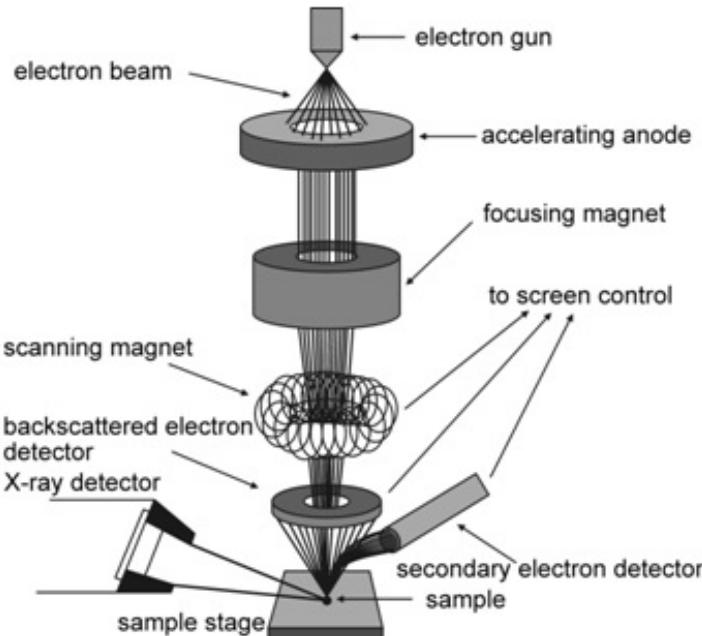


Figure 16: Schematic diagram of Scanning Electron Microscope[21]

SEM uses a focused beam of high-energy electrons instead of light to interact with the sample. The electron beam is generated from an electron source, typically a tungsten filament or field emission source. The nanophosphor sample is typically mounted on a conductive substrate or coated with a thin conductive layer, such as gold or carbon. This ensures that the sample can conduct electrons and avoids the accumulation of charge during imaging. The electron beam is scanned across the surface of the sample in a raster pattern. As the beam interacts with the sample, various signals are generated, including secondary electrons (SE), backscattered electrons (BSE), and characteristic X-rays. It primarily utilizes secondary electrons, which are low-energy electrons emitted from the surface of the sample due to the interaction with the primary electron beam. These electrons provide information about the sample's surface morphology and topography. Backscattered Electrons (BSE) are high-energy electrons that undergo elastic scattering after interacting with the atomic nuclei of the sample. BSE imaging provides information about the sample's composition, density variations, and atomic number contrast. The signals generated by the interaction of the electron beam with the sample are detected by specialized detectors. These detectors collect the signals and convert them into electrical signals, which are then processed to form images of the sample's surface. SEM images can be further analyzed using dedicated software to measure particle size, distribution, and perform quantitative analysis. Image processing techniques can enhance contrast, remove noise, and provide valuable information about the sample's features.

2.3.3 Transmission Electron Microscopy (TEM)

Transmission Electron Microscopy (TEM)[39] is an advanced imaging and analytical technique used to study the internal structure, crystallography, and composition of materials at the nanoscale. It provides high-resolution images and enables detailed analysis of the morphology, crystal structure, defects, and interfaces of nanophosphors.

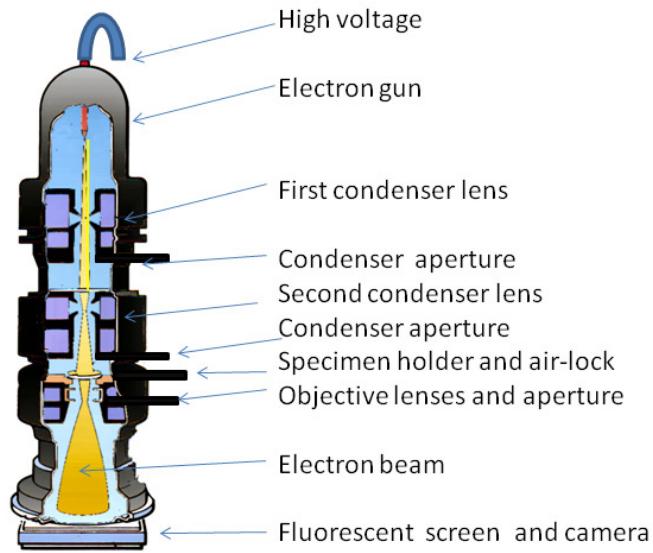


Figure 17: Schematic diagram of Transmission Electron Microscope[27]

TEM uses a focused beam of electrons that passes through an ultra-thin sample. The electron beam is generated from an electron source, such as a tungsten filament or field emission gun, and accelerated to high energy. To perform TEM analysis, the nanophosphor sample needs to be prepared as thin specimens with thicknesses typically ranging from tens to hundreds of nanometers. The most common technique for sample preparation is known as thinning by mechanical polishing or using specialized equipment like a focused ion beam (FIB) system. The electron beam passes through the sample, and the transmitted electrons are collected and focused onto a fluorescent screen or a charge-coupled device (CCD) camera. The resulting image represents the transmitted electron intensity, providing a projection of the sample's internal structure. It allows for high-resolution imaging of the sample, providing details at the atomic level. It enables the observation of lattice fringes, defects, grain boundaries, and other fine structural features of the nanophosphor.

TEM is a powerful tool for nanophosphor characterization, offering exceptional spatial resolution and the ability to study the nanoscale structure and properties of materials. It enables researchers to investigate the internal structure, defects, and interfaces of nanophosphors, contributing to a deeper understanding of their properties and guiding their development for various applications.

2.3.4 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) is a widely used analytical technique that provides information about the chemical bonds and molecular structure of materials, including nanophosphors. It is based on the principle of measuring the absorption, transmission, or reflection of infrared (IR) light by the sample over a range of frequencies. FTIR is a versatile and non-destructive technique used to characterize nanophosphors and understand their chemical composition, molecular structure, and functional groups. It is widely employed in materials science, chemistry, pharmaceuticals, and many other fields for quality control, material identification, and chemical analysis.

FTIR Instrumentation

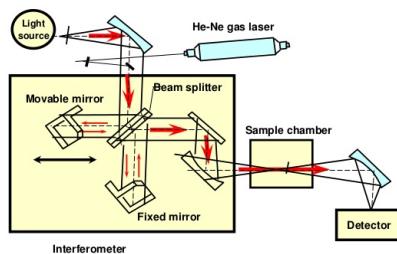


Figure 18: Fourier Transform Infrared Spectroscopy (FTIR) instrumentation[31]

FTIR utilizes infrared radiation, which falls in the region of the electromagnetic spectrum between visible

light and microwaves. This region corresponds to the vibrational and rotational energy levels of molecules, allowing for the analysis of chemical bonds and functional groups. For FTIR analysis, nanophosphor samples are typically ground into a fine powder and mixed with an IR-transparent matrix, such as potassium bromide (KBr), to form a solid pellet. The pellet is then placed in the IR spectrometer for analysis. When IR light interacts with the sample, it is absorbed by the molecular bonds, causing the atoms within the bonds to vibrate. Different chemical bonds and functional groups exhibit characteristic vibrational frequencies, which correspond to specific IR wavelengths. In FTIR, the IR light passing through the sample is split into two beams. One beam passes through the sample, and the other passes through a reference mirror. The two beams are then recombined, and their interference pattern is measured. This technique allows for the acquisition of the entire IR spectrum simultaneously. The interference pattern is transformed using a mathematical technique called Fourier transform, resulting in the acquisition of the sample's IR spectrum. The spectrum represents the absorption or transmission of IR light at different wavelengths/frequencies, providing information about the molecular vibrations and chemical groups present in the nanophosphor.

3 Experiment

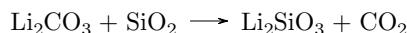
In this project, two different samples have been synthesized using two different methods and were investigated for potential use in thermoluminescence dosimetry. *Lithium metasilicate* (Li_2SiO_3) was synthesized as the first sample due to its relevance to dosimetry as a tissue equivalent material. The sample was synthesized using solid-state reaction method and dose response was taken. The sample was then doped with various rare-earth metals and investigated for optimum doping material. *Calcium Sodium Sulphate* ($CaNa_2(SO_4)_2$) was synthesized as our next sample using chemical co-precipitation method. The sample was then doped with Europium at various concentrations and its glow curves were taken. TL glow curves at multiple doses were taken to investigate its dose response.

3.1 Lithium metasilicate (Li_2SiO_3)

Lithium metasilicate is a compound with the chemical formula Li_2SiO_3 . It consists of lithium (Li) cations, silicate (SiO_3) anions. It is typically encountered as a white crystalline powder or granules. Its physical properties, such as density and melting point, can vary depending on factors such as crystal structure and the presence of impurities. These materials have physicochemical stability at high temperatures, compatibility with other types of structural materials, radiation stability and adequate heat transfer. The main feature is their high sensitivity to ionizing radiation for a wide energy range. Also, Li_2SiO_3 ($Z_{eff} = 10.5$) being low Z materials, are near tissue equivalent and may find application in the field of radiation dosimetry[4].

3.1.1 Synthesis of Li_2SiO_3 (pure)

Lithium metasilicate was prepared by solid state reaction method[5] using the following chemical reaction.



To synthesize Lithium metasilicate 5g of Li_2CO_3 and 4.065g of SiO_2 was measured and grinded using a mortar and pestle with the addition of acetone for 30 minutes to form a smooth white paste. This paste was then heated in a heating mantle for 30 minutes to let the acetone evaporate out. The residual solid was then grounded to a fine powder using mortar and pestle. The sample was then heated in a muffle furnace at $900^{\circ}C$ for 4 hours in air and then cooled down to room temperature. The resulting sample was grounded again for 30 minutes using mortar and pestle.



Figure 19: Precursor materials being weighed and grounded into a fine paste with acetone

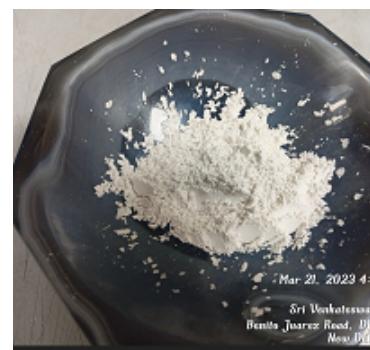
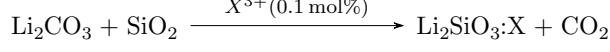


Figure 20: Sample being ground after removing acetone by heating with heating mantle

3.1.2 Dopant Optimization for Li_2SiO_3

To study the effect of doping on the thermoluminescence of the given sample, Li_2SiO_3 is prepared using solid state reaction method under the same conditions but the samples are being doped with 5 rare earth metals. Li_2SiO_3 was doped with Dysprosium, Europium, Terbium, Erbium and Cerium at 0.1 mol%. The chemical reaction for the process is given below:



where X is the dopant material, for example Dysprosium, Europium, Terbium, Erbium or Cerium To synthesize doped Lithium metasilicate 4.99g of Li_2CO_3 and 4.065g of SiO_2 along with calculated amount of rare earth metals was measured and grinded using a mortar and pestle with the addition of acetone for 30 minutes to

form a smooth white paste. This paste was then heated in a heating mantle for 30 minutes to let the acetone evaporate out. The residual solid was then grounded to a fine powder using mortar and pestle. The sample was then heated in a muffle furnace at 900°C for 4 hours in air and then cooled down to room temperature. The resulting sample was grounded again for 30 minutes using mortar and pestle.



Figure 21: Sample being annealed in a furnace at 900°C for 4 hours



Figure 22: Final sample after preparation

3.2 Calcium Sodium Sulphate ($\text{CaNa}_2(\text{SO}_4)_2$)

Calcium Sodium Sulphate ($\text{CaNa}_2(\text{SO}_4)_2$) consists of one calcium ion (Ca^{2+}) and two sodium ions (Na^+) combined with two sulfate ions (SO_4^{2-}). The calcium and sodium ions contribute to the cationic structure, while the sulfate ions form the anionic part. It typically exists as a white crystalline solid. It has a relatively high melting point and is sparingly soluble in water. The exact physical properties, such as density and hardness, may vary depending on the specific crystalline form and conditions. Calcium sodium sulfate is not commonly found in nature as a distinct mineral. However, it may form as a component in certain mineral deposits or as a result of industrial processes. Due to its limited natural occurrence, calcium sodium sulfate does not have significant industrial applications on its own. However, it may be used as a source of calcium and sodium ions in various chemical processes or as an ingredient in specific formulations. It can undergo various chemical reactions depending on the specific conditions. For example, it can react with acids to release sulfuric acid and form corresponding salts. It can also participate in precipitation reactions or form solid compounds with other ions.

3.2.1 Synthesis of $\text{CaNa}_2(\text{SO}_4)_2$ (pure)

Calcium Sodium Sulphate was prepared by chemical co-precipitation method[22][34] using the following chemical reaction.



To synthesize $\text{CaNa}_2(\text{SO}_4)_2$ 14.702g of CaCl_2 and 11.688g of NaCl was added to 100ml of double-distilled water in a beaker and stirred continuously with a magnetic stirrer. Simultaneously a solution of 26.426g of $(\text{NH}_4)_2\text{SO}_4$ in 100ml double distilled water was prepared and added drop wise to the previous solution using a burette while being continuously stirred using a magnetic stirrer. A milky white precipitate was observed to be forming at the bottom of the beaker.

The solution along with precipitate was then centrifuged 34 times using a centrifuge and the precipitate thus obtained was separated and placed on a heating mantle at 40°C for 2 hours and then the temperature was increased to 60°C for the next 5 hours while finally increasing temperature to 80°C and heating the sample for 1 hour. The sample was then let to cool down to room temperature and then grounded into a fine powder using a mortar and pestle. The sample was then stored in a cool and dark place to be tested for its thermoluminescence properties. The sample was then taken to Inter University Accelerator Centre (IUAC) where it was annealed at constant temp of 400°C in a muffle furnace for 2 hours and then brought back to room temperature to remove any previous radiation.



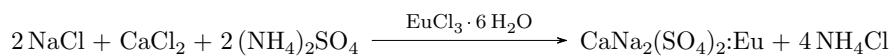
Figure 23: Formation of precipitate due to drop-wise addition of $(\text{NH}_4)_2\text{SO}_4$ to the solution



Figure 24: Centrifuging the sample

3.2.2 Synthesis of $\text{CaNa}_2(\text{SO}_4)_2 : \text{Eu}$

Calcium Sodium Sulphate doped with Europium was prepared by chemical co-precipitation method using the following chemical reaction.



In a beaker with 100ml double distilled water 14.702g of CaCl_2 and 11.676g of NaCl was added and stirred continuously with a magnetic stirrer. 0.1104g of $\text{EuCl}_3 \cdot 6 \text{H}_2\text{O}$ was then added to the above solution and stirred continuously for 15 minutes. A solution of 26.426g of $(\text{NH}_4)_2\text{SO}_4$ in 100ml double distilled water was prepared and added drop wise to the above solution using a burette while being continuously stirred using a magnetic stirrer over a period of 3 hours. A milky white precipitate was observed to be forming at the bottom of the beaker. The solution along with precipitate was then centrifuged 34 times using a centrifuge.

The precipitate thus obtained was separated and placed on a heating mantle at 40°C for 2 hours and then the temperature was increased to 60°C for the next 5 hours while finally increasing temperature to 80°C and heating the sample for 1 hour. The sample was then let to cool down to room temperature and then grounded into a fine powder using a mortar and pestle. Approximately 22g of powdered sample was obtained using this method. The sample was then stored in a cool and dark place to be tested for its thermoluminescence properties. The sample was then taken to Inter University Accelerator Centre (IUAC) where it was annealed at constant temp of 400°C in a muffle furnace for 2 hours and then brought back to room temperature to remove any previous radiation.



Figure 25: Sample grounded to a fine powder

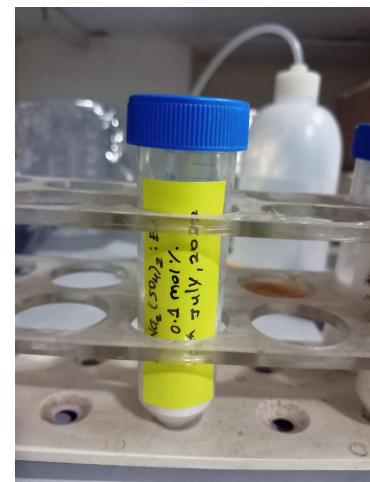


Figure 26: Final sample

3.3 Evaluation of Glow Curves

All The samples were then irradiated with Gamma rays using ^{60}Co source first with a dose of 5Gy and then 100Gy. After irradiation, 5mg from each sample was then measured and dose response of the samples were recorded utilizing Harshaw TLD reader by heating the sample from $50^{\circ}C$ to $400^{\circ}C$ at a continuous heating rate of $5^{\circ}C/s$. The raw data from Harshaw TLD reader was then plotted using Origin software for futher analysis.



Figure 27: Gamma ray irradiation chamber at Inter University Accelerator Centre (IUAC), New Delhi using ^{60}Co source



Figure 28: Harshaw TLD Reader to for measurement of dose response of the samples

3.4 Ion Beam Irradiation

Calcium Sodium Sulphate ($CaNa_2(SO_4)_2$) was irradiated with carbon beams at Inter-University Accelerator Centre (IUAC) at two energies 65MeV and 85MeV at different fluences. The samples were first made into pellets using a hydraulic pellet press (shown in figure29). The pellets were then annealed at $400^{\circ}C$ (shown in figure33) for 2 hours. The samples were then loaded into a ladder (shown in figure30) and then irradiated using carbon beams from pelletron accelerator at IUAC (shown in figure32). Glow curves were then taken and further analysis was conducted.



Figure 29: Hydraulic Pellet press to turn the samples into pellets



Figure 30: Pellets kept in a boat to be annealed in a muffle furnace



Figure 31: Samples being loaded in a ladder



Figure 32: Ion beam irradiation chamber at IUAC

3.5 Deconvolution of TL Glow Curves

Deconvolution of a glow curve refers to the process of separating individual glow peaks from a thermoluminescence (TL)[15] or optically stimulated luminescence (OSL) signal. Glow curves are typically obtained by heating a dosimeter material or stimulating it with light and measuring the emitted luminescence as a function of temperature or time, respectively. Each peak in the glow curve corresponds to a specific trap level in the material, and deconvolution allows the identification and characterization of these traps.

R programming language was used to deconvolute the glow curves obtained using package tgcd[28]. A code snippet for the R programme used for deconvolution is given below:

```

RGui (64-bit) - [D:\Rick\SRI VIPRA\R\caf2.R - R Editor]
File Edit Packages Windows Help
require(readxl)
require(tgcd)
my_data <- read_excel(file.choose())
head(my_data)
plot(my_data$Temp, my_data$Intensity,type="p")
mat<-data.matrix(my_data)
ddl <- tgcd(mat, npeak=3, model="gl",
            nstart=10, edit.inis=TRUE)
head(ddl$comp.sig)
 ddl$pars
 ddl$sp
 ddl$FOM

```

Figure 33: Code snippet of R program for deconvolution of glow curves

4 Result and Analysis

4.1 Lithium metasilicate (Li_2SiO_3)

The Thermoluminescence Glow curves (TL Glow Curves) obtained using Harshaw TLD Reader 3500 by heating the samples from $50^\circ C$ to $400^\circ C$ at a heating rate of $5^\circ C/s$ for all samples of Li_2SiO_3 after being irradiated with gamma rays at 50 Gy and 100Gy are shown below:

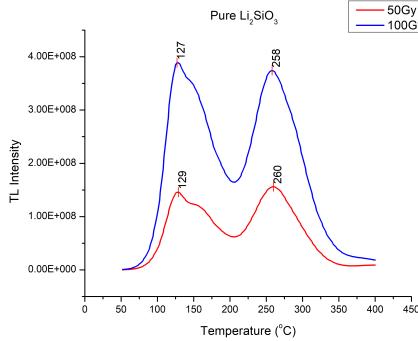


Figure 34: Dose response for Lithium metasilicate when irradiated with Gamma rays at 50 Gy and 100 Gy

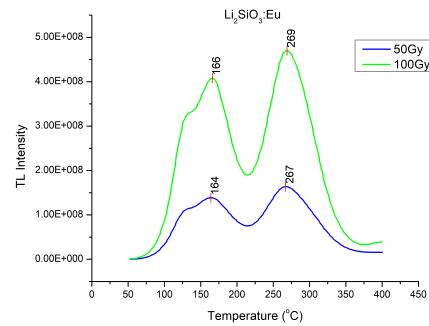


Figure 35: Dose response for Lithium metasilicate doped with 0.1mol% Europium when irradiated with Gamma rays at 50 Gy and 100 Gy

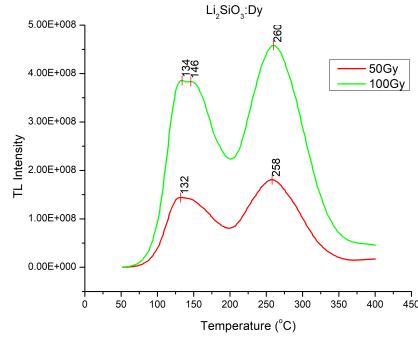


Figure 36: Dose response for Lithium metasilicate doped with 0.1mol% Dysprosium when irradiated with Gamma rays at 50 Gy and 100 Gy

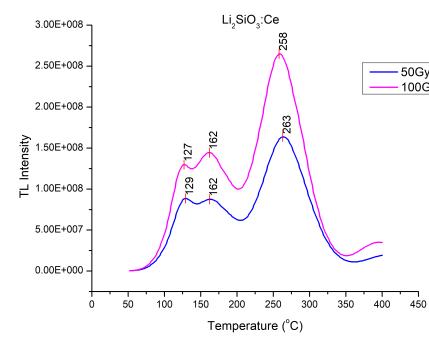


Figure 37: Dose response for Lithium metasilicate doped with 0.1mol% Cerium when irradiated with Gamma rays at 50 Gy and 100 Gy

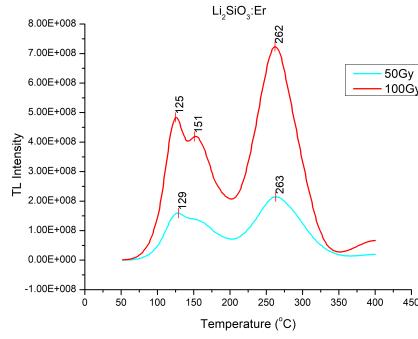


Figure 38: Dose response for Lithium metasilicate doped with 0.1mol% Erbium when irradiated with Gamma rays at 50 Gy and 100 Gy

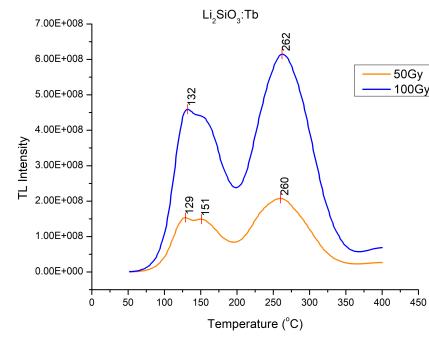


Figure 39: Dose response for Lithium metasilicate doped with 0.1mol% Terbium when irradiated with Gamma rays at 50 Gy and 100 Gy

As can be seen from the plots Lithium metasilicate yields glow curves with two to three major peaks. Pure lithium metasilicate (no dopant) has peaks at $129^\circ C$ and $260^\circ C$ at 50 Gy while it peaks at $127^\circ C$ and $258^\circ C$ at 100 Gy. Lithium metasilicate doped with Europium has peaks at $164^\circ C$ and $267^\circ C$ at 50 Gy while at $166^\circ C$ and

269°C at 100 Gy. Lithium metasilicate doped with Dysprosium has peaks at 132°C and 258°C at 50 Gy while it has 3 peaks at 134°C , 146°C and 260°C at 100 Gy. Lithium metasilicate doped with Cerium has 3 peaks at 129°C , 162°C and 263°C at 50 Gy while and at 127°C , 162°C and 258°C at 100 Gy. Lithium metasilicate doped with Erbium has peaks at 129°C and 263°C at 50 Gy while it has 3 peaks at 125°C , 151°C and 262°C at 100 Gy. Lithium metasilicate doped with Terbium has 3 peaks at 129°C , 151°C and 260°C at 50 Gy while it has 2 peaks at 132°C and 262°C at 100 Gy.

The effect of dopant on the dose response can be clearly seen in the following figures:

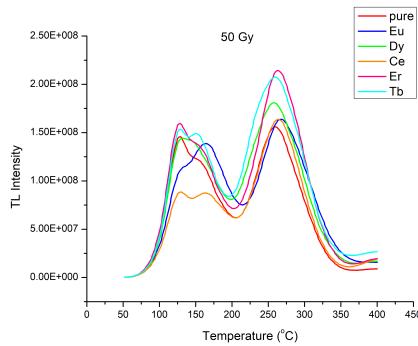


Figure 40: Dose response for various dopants when irradiated with Gamma rays at 50 Gy

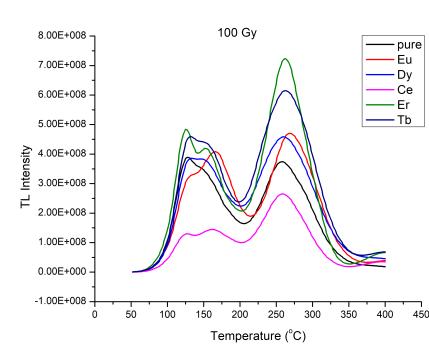


Figure 41: Dose response for various dopants when irradiated with Gamma rays at 100 Gy

As can be seen from the plots, doping lithium metasilicate with various dopants usually increases the thermoluminescence intensity, this can be attributed to increased number of traps and recombination centres due to doping. Maximum increase in intensity was observed after doping with Terbium and Erbium. A comparision plot is given below:

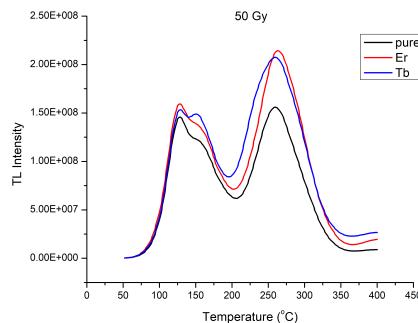


Figure 42: Comparision of dose response of Li_2SiO_3 without doping and after doping with Terbium and Erbium when irradiated with Gamma rays at 50 Gy

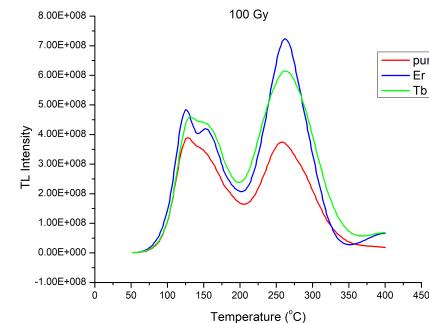


Figure 43: Comparision of dose response of Li_2SiO_3 without doping and after doping with Terbium and Erbium when irradiated with Gamma rays at 100 Gy

A comparision between peak intensities has been done for various dopants at both 50 Gy and 100 gy gamma ray irradiation. These plots indicate that the peak intensity for Terbium and Erbium is much higher as compared to other dopants.

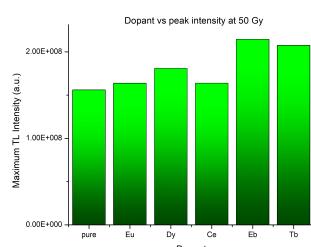


Figure 44: Comparision of peak intensity for various dopants when irradiated with Gamma rays at 50 Gy

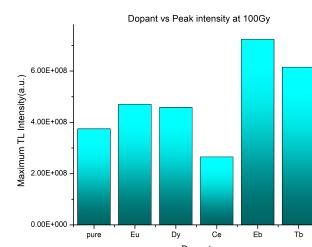


Figure 45: Comparision of peak intensity for various dopants when irradiated with Gamma rays at 100 Gy

Finally, it can be concluded that lithium metasilicate shows thermoluminescence and yields distinct peaks for definite temperatures with maximum intensity at higher temperatures. On addition of dopants, the thermoluminescence intensities in much higher than that of undoped sample. Doping with Terbium and Erbium results in maximum intensities for 50 Gy and 100 Gy gamma irradiation. More analysis is required in this field to provide a conclusive evidence of the use of lithium metasilicate as a viable and effective material for thermoluminescence dosimetry.

4.1.1 Deconvolution of Glow Curves

The glow curves obtained for Lithium metasilicate has been obtained for both undoped and after doping with Erbium All of these curves can be deconvoluted to 3 independent glow curves as shown in figure:

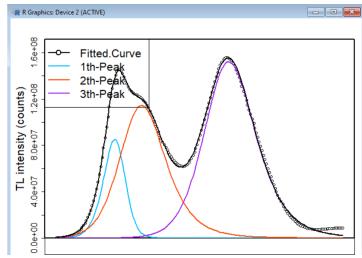


Figure 46: Deconvolution of glow curve of pure Li_2SiO_3 at 50 Gy

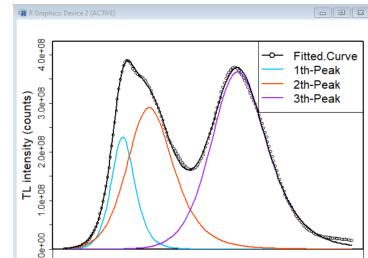


Figure 47: Deconvolution of glow curve of pure Li_2SiO_3 at 100 Gy

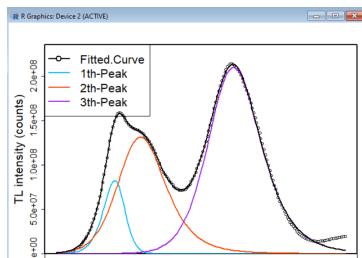


Figure 48: Deconvolution of glow curve of Li_2SiO_3 doped with Erbium at 50 Gy

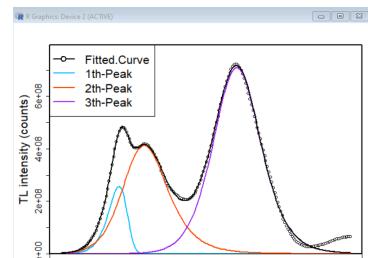
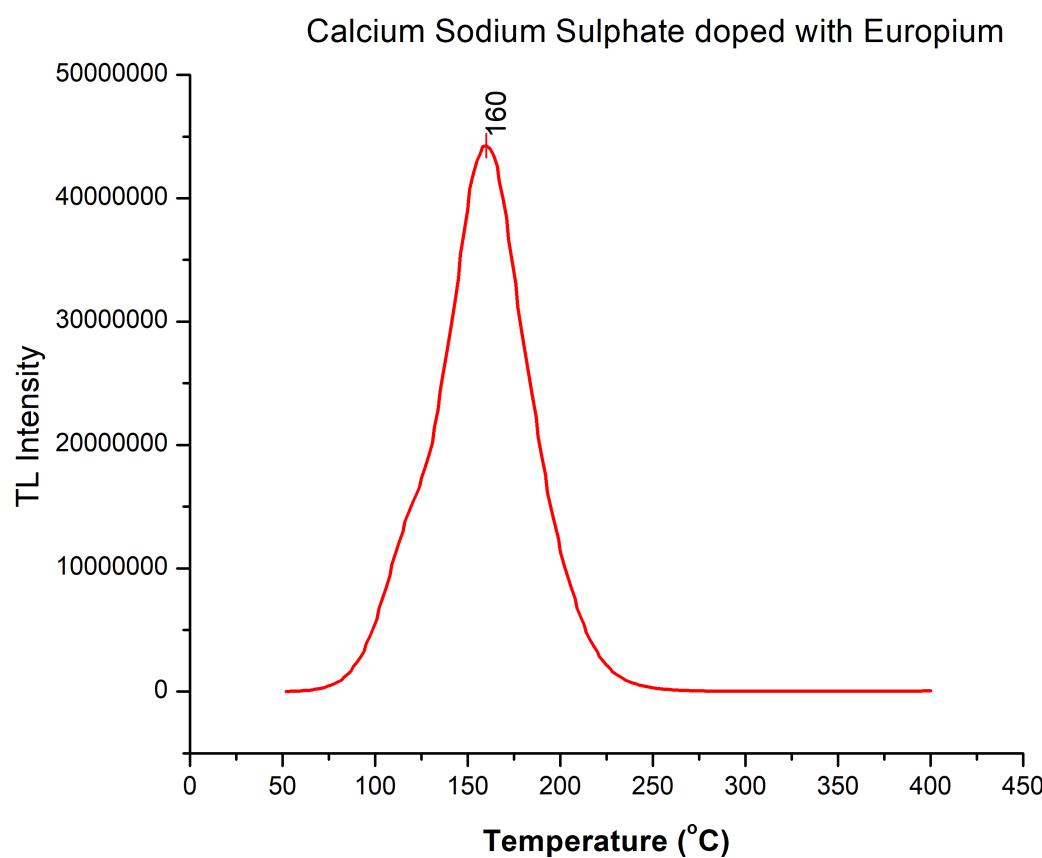
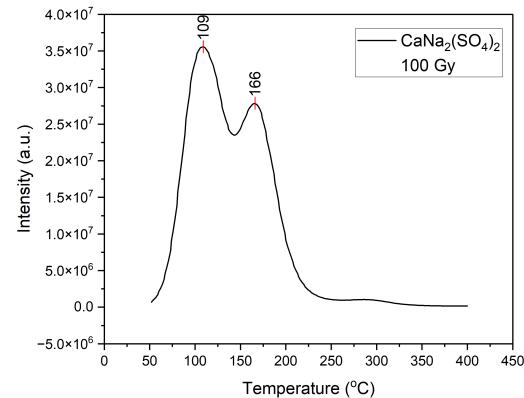
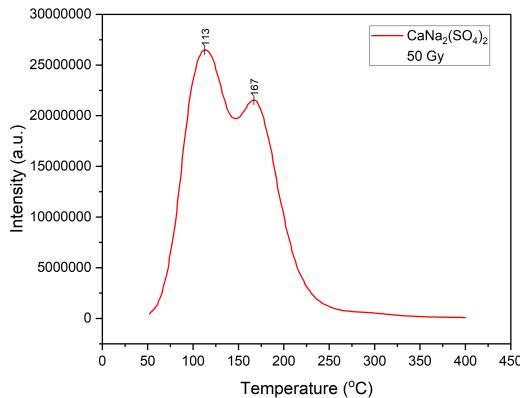


Figure 49: Deconvolution of glow curve of Li_2SiO_3 doped with Erbium at 100 Gy

4.2 Calcium Sodium Sulphate ($CaNa_2(SO_4)_2$)

The Thermoluminescence Glow curves (TL Glow Curves) obtained using Harshaw TLD Reader 3500 by heating the samples from $50^{\circ}C$ to $400^{\circ}C$ at a heating rate of $5^{\circ}C/s$ after being irradiated with gamma rays at 50 Gy and 100Gy are shown here:



Another study that we undertook is comparison of intensity of TL with the concentration of dopant. As can be seen, with addition of dopant, the glow curves come close to having a single peak as compared to pure

sample. Also the thermoluminescence intensity for doped sample is more compared to that of the pure sample. However, conclusive evidence cannot be provided as only one concentration of doped sample was synthesized and further analysis is required.

4.2.1 Dose Response

Dose response for $\text{CaNa}_2(\text{SO}_4)_2 : \text{Eu}$ was investigated by irradiating the sample with gamma rays at different intensities (10,50,100,400 and 1kGy) and glow curves were obtained. The dose response of $\text{CaNa}_2(\text{SO}_4)_2 : \text{Eu}$ was then evaluated as shown below.

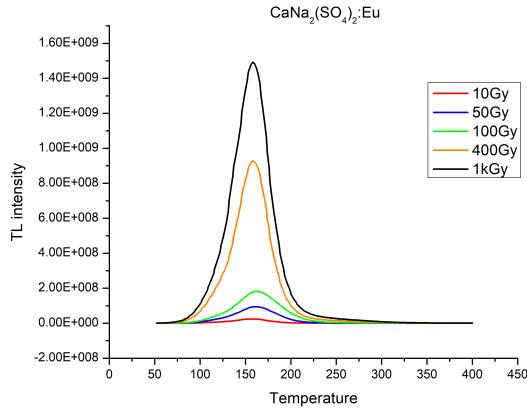


Figure 53: TL Glow curves for Calcium Sodium Sulphate doped with Europium at multiple doses

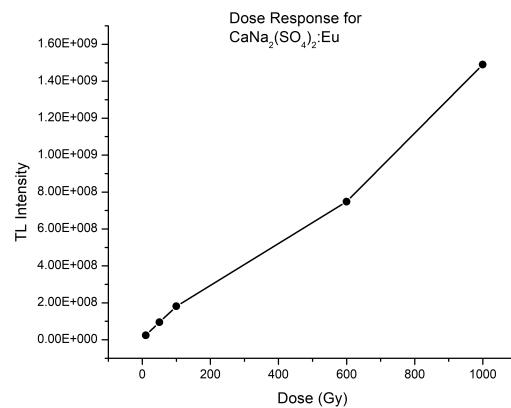


Figure 54: Dose response for Calcium Sodium Sulphate doped with Europium

Conclusion

Thermoluminescence dosimetry (TLD) is a well-established technique that provides reliable and precise measurements of ionizing radiation doses. TLDs use a variety of materials that emit light when exposed to ionizing radiation. Nanophosphors, also known as nanocrystalline phosphors or nanoparticles, have emerged as promising materials for dosimetry applications. These nanoscale phosphors exhibit unique properties that make them highly suitable for radiation dosimetry.

An in-depth analysis of TLD principles as well as various techniques required for synthesis and characterization of thermoluminescent materials, especially nanophosphors has been conducted. Two nanophosphors have been synthesized using two different techniques. *Calcium Sodium Sulphate ($CaNa_2(SO_4)_2$)* was synthesized using chemical co-precipitation method and *Lithium metasilicate (Li_2SiO_3)* was synthesized using solid-state reaction method their dose response has been investigated. Variation in dose response due to doping has also been investigated.

The thermoluminescent properties of $CaNa_2(SO_4)_2$ sample was investigated by irradiating it with ^{60}Co gamma rays at 50Gy and 100Gy. The TL glow curve of $CaNa_2(SO_4)_2$ shows 2 peaks at around $113^{\circ}C$ and $167^{\circ}C$ for 50 Gy dose and at around $109^{\circ}C$ and $166^{\circ}C$ for 100Gy dose. The sample of $CaNa_2(SO_4)_2 : Eu$ shows a very high intensity single TL glow peak at around $160^{\circ}C$. This shows that the addition of dopant enhances the thermoluminescent properties of $CaNa_2(SO_4)_2$. Further analysis with different dopant concentrations is required to study the application of $CaNa_2(SO_4)_2$ as an efficient thermoluminescent dosimeter.

The thermoluminescent properties of Li_2SiO_3 sample was investigated by irradiating it with ^{60}Co gamma rays at 50Gy and 100Gy. The main feature of lithium metasilicate is its high sensitivity to ionizing radiation for a wide energy range. Also, Li_2SiO_3 ($Z_{eff} = 10.5$) being low Z materials, are near tissue equivalent and may find application in the field of radiation dosimetry. Lithium metasilicate yields glow curves with two to three major peaks. Pure lithium metasilicate (no dopant) has peaks at $129^{\circ}C$ and $260^{\circ}C$ at 50 Gy while it peaks at $127^{\circ}C$ and $258^{\circ}C$ at 100 Gy. Lithium metasilicate doped with Europium has peaks at $164^{\circ}C$ and $267^{\circ}C$ at 50 Gy while at $166^{\circ}C$ and $269^{\circ}C$ at 100 Gy. Lithium metasilicate doped with Dysprosium has peaks at $132^{\circ}C$ and $258^{\circ}C$ at 50 Gy while it has 3 peaks at $134^{\circ}C, 146^{\circ}C$ and $260^{\circ}C$ at 100 Gy. Lithium metasilicate doped with Cerium has 3 peaks at $129^{\circ}C, 162^{\circ}C$ and $263^{\circ}C$ at 50 Gy while and at $127^{\circ}C, 162^{\circ}C$ and $258^{\circ}C$ at 100 Gy. Lithium metasilicate doped with Erbium has peaks at $129^{\circ}C$ and $263^{\circ}C$ at 50 Gy while it has 3 peaks at $125^{\circ}C, 151^{\circ}C$ and $262^{\circ}C$ at 100 Gy. Lithium metasilicate doped with Terbium has 3 peaks at $129^{\circ}C, 151^{\circ}C$ and $260^{\circ}C$ at 50 Gy while it has 2 peaks at $132^{\circ}C$ and $262^{\circ}C$ at 100 Gy. On addition of dopants, the thermoluminescence intensities are much higher than that of undoped sample. Doping with Terbium and Erbium results in maximum intensities for 50 Gy and 100 Gy gamma irradiation. More analysis is required in this field to provide a conclusive evidence for the use of lithium metasilicate as a viable and effective material for thermoluminescence dosimetry.

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