

FINAL TECHNICAL REPORT

No. 1222/ CCOST/MRP dt 31/03/2010

“Synthesis and characterization of rare earth Doped SrAl_2O_4 nanophosphors and their Applications”

Dr. Mrs. Nameeta Brahme

(Professor)

Principal Investigator

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Raipur (C.G) - 492010

FINAL TECHNICAL REPORT

CHHATTISGARH COUNCIL OF SCIENCE AND TECHNOLOGY (CCOST)

MIG-25 Indravati Colony, Raipur

1. Project Report for the Period : July 2011 - June 2012

2. Name of the Project : "Synthesis and characterization of rare earth Doped SrAl_2O_4 nanophosphors and their Applications".

3. No. & Date of Sanction : No. 1222/ CCOST/MRP dt 31/03/2010

4. Name & Address of PI : Dr. Mrs. Nameeta Brahme (Professor)
S.O.S in Physics & Astro Physics, Pt.
R.S.S. University, Raipur (C.G)

5. (A) Amount Sanctioned for Three years (July2010 – July2013)

S. No.	Funds	I st Year(Rs)	II nd Year(Rs)	III rd Year(Rs)	Total
1.	Staff Salary(PF)@3500/-	42,000/-	42,000/-	42,000/-	1,26,000/-
2.	Contingency	8700/-	8700/-	6600/-	24000/-
3.	Minor equipment	50,000/-	-	-	50,000/-
	TOTAL	100,700/-	50,700/-	48,600/-	2,00,000/-

(B) Expenditure:

(i) Ist Year (July 2010 - June 2011): Audited Statement / UC Already Submitted (copy enclosed).

Sr.	Funds	Allocated Amount	Received Amount	Expenditure Amount	Balance Amount
1.	Staff	42,000/-	42,000/-	42,000/-	Nil
2.	Contingency	8700/-	8700/-	8737/-	(-) 37=00
3.	Minor Equip.	50,000/-	Nil	Nil	Nil
	Total =	100,700/-	50,700/-	50,737/-	(-) 37=00

(ii) IInd Year (July 2011 - June 2012): Audited Statement / (UC Already Submitted copy enclosed).

Sr.	Funds	Allocated Amount	Received Amount	Expenditure Amount	Balance Amount
1.	Staff	42,000/-	42,000/-	42,000/-	Nil
2.	Contingency	8700/-	8700/-	8751/-	(-)51=00
3.	Minor Equip.	50,000/-	Nil	Nil	Nil
	Total =	100,700/-	50,700/-	50,751/-	(-)51=00

(iii) IIIrd Year (July 2012 - June 2013): Audited Statement / UC (Enclosed in original)

Sr.	Funds	Allocation Amount	Received Amount	Expenditure Amount	Balance Amount
1.	Staff	42,000/-	42,000/-	42,000/-	Nil
2.	Contingency	6600/-	6600/-	6822/-	(-) 222=00
3.	Minor Equip.	50,000/-	50,000/-	51,912	(-) 1,912
	Total =	98,600/-	98,600/-	1,00734/-	(-) 2134=00

6. Name of the Project Fellow (PF) appointed in the project along with dates of joining and leaving.

- Miss Geetanjali Tiwari: Date of joining: 01/07/2010; Date of leaving: 30/06/2013.

7. Total work done in the Project (Duration: July 2010 – June 2013)

There are two outstanding processes by which a material can become a generator or origin of light (radiation) after absorbing suitable extraneous primary energy. In one process the absorbed energy is converted (degraded) into low-quantum-energy heat that diffuses through the material which then emits radiation called thermal radiation. In the other process an appreciable part of the absorbed energy is temporarily localized as relatively high-quantum-energy excitation of atoms or small groups of atoms which then emit radiation called luminescence radiation. The term "luminescence" was introduced into literature by Wiedemann in 1888. Luminescence is a general term for the emission of electromagnetic radiation from the substance during or following the absorption of energy from suitable sources such as U.V radiation, X-rays or high energy particle. The energy lifts the atoms of the material into an excited state, and then, because excited states are unstable, the material goes back to its ground state, and the absorbed energy is liberated in the form of either light or heat or both. Luminescence is a consequence of the radiative recombination of the excited electrons/holes. Luminescence process involves at least two steps – a) **Excitation of**

electronic system of the substance and b) The subsequent emission of photon. These two steps may or may not be separated by intermediate process. Depending upon types of excitation source there are different types of luminescence – Photoluminescence, Mechanoluminescence, Thermoluminescence, Cathodoluminescence, Electroluminescence, Chemiluminescence, Lyoluminescence, Sonoluminescence etc.

Mechanoluminescence (ML) is an important physical phenomenon where an emission of light is observed due to mechanical deformation of materials when they are subjected to some mechanical stress like rubbing, cleavage, compressing, impulsive crushing, grinding, shaking etc. [1-5]. Emission via ML has also been observed due to many other processes e.g. thermal shocking (immediate cooling or heating), phase transition and separation of two different materials in contact. ML can be due to both the elastic and plastic deformation of the solids. Depending upon the nature of mechanical stress, ML has also been termed as fractoluminescence, elasticoluminescence, plasticoluminescence and triboluminescence in the literature [6, 7]. Many organic and inorganic crystals, polymers, ceramic and glasses have shown to exhibit ML [8]. ML has found various important applications such as impact sensors in spacecrafts (the emission intensity can be used to determine the kinetic energy of impact), fracture sensor, damage sensor, sensor for stress and its distribution in the solids, crack propagation in solids and understanding of the basic mechanism of crack growth [9-11]. Chandra et al. have presented various theoretical studies on various kind of ML [4, 12-15]. Thermoluminescence (TL) in solids is the light emission (mainly visible) that takes place during heating of a solid following an earlier absorption of energy from radiation. The essential condition for TL to occur in an insulator or semiconductor is that the material must have been previously exposed to radiation. Once the TL emission has been observed, the material will not show it again after simply cooling the specimen and reheating it, but has been re exposed to radiation to obtain TL again. TL, although based upon the same fundamental principles as other luminescence processes, is conventionally is misnomer since the heat radiation is only a stimulant and not an exciting agent. Urbach (1930) is usually credited with suggesting TL as a potentially useful research tool for trap-level analysis. A large number of dielectric materials exhibit TL emission , including minerals, rocks, inorganic semiconductors and insulators, glasses and ceramics, organic compounds, biological materials and biochemicals. Materials such as LiF, CaSO₄, CaF₂, BeO, Al₂O₃ and Li₂B₄O₇ are most extensively studied TL materials because of their applications in dosimetry.

The rare earth-activated alkaline earth aluminates are an important class of phosphorescence materials because of their high quantum efficiency in visible region [16], long persistence of phosphorescence, good stability, color purity and good chemical, thermal and radiation resistance [17-21]. The potential benefit of rare earth ions as an activator has now well established in the field of luminescence. Different activators contribute significantly in tailoring the afterglow properties of phosphors from few seconds to many hours. In Eu the life time of 5d-4f transitions are about 3 orders of magnitude shorter than 4f-4f transition lifetime exhibited by other lanthanides and thus are of immense importance. In polycrystalline $\text{Sr}_3\text{Al}_2\text{O}_6\text{:Eu}$, Dy intense ML was reported [22]. For $\text{SrAl}_2\text{O}_4\text{:Eu}^{2+}$ it was demonstrated that ML is due to $4f^7\text{-}4f^65d$ transition of Eu^{2+} [23]. ML from monoclinic structure SrAl_2O_4 was reported and it was found that only the $\alpha\text{-SrAl}_2\text{O}_4$ phase produces strong ML [24]. These phenomenons are attracting considerable attention because they can be applied to sensing of structural damage and fracture. Although it was reported that SrAl_2O_4 doped with Eu^{2+} exhibits ML, the atomistic depiction about the effect of stress or strain on the rare-earth ion still remains unclear.

This research project proposed to discover some new mechanoluminescent and thermoluminescent phosphor and then to characterize the phosphor and finally optimized luminescent aluminate phosphors according to different parameters. These optimized phosphors will be suitable for development of pressure sensors and can also be used for dosimetry purposes. The phosphors were synthesized using Combustion Technique, which is novel, economic and takes less time to prepare. The total work carried out in the project has been on the lines proposed in the original research proposal. The overall progress of the work in the total project duration has been quite satisfying, as envisaged. The outcomes of the present investigations have been published in the form of papers in various National/International Journals. The total work done in the present project is briefly described in the next section and the list of research papers is given below along with the copy of paper attached

here

with.

List of Publications:

1. "Synthesis of $\text{SrAl}_2\text{O}_4\text{:Eu}$ phosphor by combustion method and its possible applications for mechanoluminescence dosimetry" A. K. Choubey, **Nameeta Brahme**, D. P. Bisen. **Indian Journal of Pure and Applied Physics** vol 50, Nov (2012) 851-854
Impact Factor 0.76 .
2. "Thermoluminescence of gamma-irradiated $\text{SrAl}_2\text{O}_4\text{:Dy}$ " Anil Kumar Choubey, **Nameeta Brahme**, D P Bisen, S J Dhoble.
Recent Research in Science and Technology. 2012; 4(8):49-51.
3. "Mechanoluminescence properties of $\text{SrAl}_2\text{O}_4\text{:Tb}^{3+}$ phosphor" Sanjay Kumar Sao, **Nameeta Brahme**, D. P. Bisen, Geetanjali Tiwari, Shalinta Tigga, Ishwar Prasad Sahu and Ugendra Kurrey.
Recent Research in Science and Technology. 2012; 4(8):119-120.
4. "Mechanoluminescence By Impulsive Deformation And Photoluminescence Of $\text{SrAl}_2\text{O}_4\text{:Eu}$ Phosphor Prepared By Combustion Synthesis" Anil Kumar Choubey, **Nameeta Brahme**, D. P. Bisen.
Physics procedia 2012; 29:104-108.
5. "Mechanoluminescence & Thermoluminescence of $\text{SrAl}_2\text{O}_4\text{:Eu}$ Nano-Phosphors" Anil Kumar Choubey, **Nameeta Brahme**, D P Bisen, Ravi Sharma.
The Open Nanoscience Journal 2011; 5 (Suppl 1-M3):41-44.

Papers at International Conferences:

1. "Thermoluminescence Study of γ -irradiated Dy-doped SrAl_4O_7 phosphor" Anil Kumar Choubey, **Nameeta Brahme**, S. J. Dhoble, D. P. Bisen.
Advanced Materials World Congress (AMWC 2013) to be organized at Çeşme, Turkey during 16-19 September, 2013. (Accepted for presentation).
2. "Mechanoluminescence and Thermoluminescence of γ -irradiated Europium doped Strontium Aluminate Phosphor" Anil Kumar Choubey, **Nameeta Brahme**, D. P. Bisen, Manisha Singh.
International Conference on Luminescence and Its Applications ICLA-2012, Organized by Rajiv Gandhi University of Knowledge Technologies, Hyderabad, India, Indian

Institute of Chemical Technology, Hyderabad, India, Society for Information Displays (India Chapter) And Luminescence Society of India, February 7-10, 2012.

3. "Mechanoluminescence By Impulsive Deformation And Photoluminescence Of $\text{SrAl}_2\text{O}_4\text{:Eu}$ Phosphor Prepared By Combustion Synthesis" Anil Kumar Choubey, **Nameeta Brahme**, D. P. Bisen.

16th International Conference on Luminescence & Optical Spectroscopy of Condensed Matter, Organized by Ann Arbor University, Michigan, USA, June 26 – July 1, 2011.

4. "Synthesis of $\text{SrAl}_2\text{O}_4\text{:Eu}$ Phosphor by Combustion Method and Its Possible Applications for Mechanoluminescence Dosimetry" **Nameeta Brahme**, Anil Kumar Choubey, D. P. Bisen.

International Conference on Accelerator radiation safety, Organized by Indian Society for particle accelerators at Bhabha Atomic Research Centre-TIFR, Mumbai, November 16-18, 2011

5. "Mechanoluminescence by impulsive deformation of $\text{SrAl}_2\text{O}_4\text{:Eu}$ Phosphor prepared by Combustion Synthesis" Anil Kumar Choubey, **Nameeta Brahme**, D. P. Bisen, , Ravi Sharma, Manisha Singh,

Indo-Russian Workshop on Nano-Technology and Laser Induced Plasma-2009 (IRNANO-2009) November 24-26, 2009 organised at University of Delhi. (Won 1st Prize for Poster Presentation).

Papers at National Conferences:

1. "Mechanoluminescence of SrAl_2O_4 nano-phosphors" A. K. Choubey, **Nameeta Brahme**, D. P. Bisen, Sanjay Sao, Geetanjali Tiwari, Shalinta Tigga.

National Conference on Emerging Trends in Science Technology and Management (NCETSTM) 9 February, 2012 organized by KITE, Raipur.

2. "Thermoluminescence Characteristics of $\text{SrAl}_2\text{O}_4\text{:Eu}$ Nanophosphor" A. K. Choubey, **Nameeta Brahme**, D. P. Bisen, S. J. Dhoble.

National Conference on Synthesis and characterization of advanced materials" (NCSCAM-2011) organized by Departments of Physics, Janata Mahavidyalaya, Chandrapur.

3. "Mechanoluminescence of pure- SrAl_2O_4 nano-phosphors prepared by combustion synthesis" Anil Kumar Choubey, **Nameeta Brahme**, D. P. Bisen, S. J. Dhoble, RAVI SHARMA, R. S. Kher.

National Conference on Luminescence and Its Applications (NCLA-2010) February 9-11, 2010 organized by Luminescence Society of India, Gandhigram Rural Institute, Kalasalingam University.

4. "Thermoluminescence of $\text{SrAl}_2\text{O}_4:\text{Eu}$ nano-phosphors prepared by combustion synthesis" Anil Kumar Choubey, **Nameeta Brahme**, D. P. Bisen, Ravi Sharma, Manisha Singh, National Conference on Luminescence and Its Applications (NCLA-2010) February 9-11, 2010 organized by Luminescence Society of India, Gandhigram Rural Institute, Kalasalingam University.

Date:

Signature of PI

Nameeta
12/09/2013

Dr. Mrs. Nameeta Brahme

Dr. Nameeta Brahme

Principal Investigator

Research Project C COST

S.O.S. in Physics & Astrophysics
Pt. R. S. University, RAIPUR

Signature of Co-PI

D. P. Bisen
12/09/2013

Dr. D.P. Bisen

Dr. D.P. Bisen

Professor

SOS in Physics & Astrophysics
Pt. R.S. University, Raipur (C.G.)

for
Nameeta
12/09/2013
Forwarded by
Professor & Head,
S.O.S. in Physics & Astrophysics,
Pt. Ravishankar Shukla University,
RAIPUR, (C.G.) 492010.

Summary of Total Work-done

Broad Objectives:

1. Synthesis of rare earth doped SrAl_2O_4 phosphors by combustion technique.
2. Characterization of Phosphors.
3. Mechanoluminescence & Thermoluminescence study of the phosphor after UV- / γ -irradiation.

1. Synthesis of rare earth doped SrAl_2O_4 phosphors by combustion technique:

Analytical grade strontium nitrate $\text{Sr}(\text{NO}_3)_2$, aluminum nitrate $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, Europium oxide Eu_2O_3 /Dysprosium oxide Dy_2O_3 /Terbium oxide Tb_2O_3 and urea $\text{CO}(\text{NH}_2)_2$ were used as the starting materials. To prepare the phosphors, stoichiometric composition of the metal nitrates (oxidizers) and urea (fuel) were calculated using the total oxidizing and reducing valences of the components, which serve as the numerical coefficients so that the equivalence ratio is unity and the heat liberated during combustion is at a maximum. Then Eu_2O_3 / Dy_2O_3 is converted into nitrate form by mixing into 2 ml of dil. HNO_3 . The weighed quantities of each nitrate and urea were mixed together and crushed into mortar for 1 hour to form a thick paste. The resulting paste is transferred to crucible and introduced into a vertical cylindrical muffle furnace maintained at different initiating temperatures. Initially the mixture boils and undergoes dehydration followed by decomposition with the evolution of large amount of gases (oxides of carbon, nitrogen and ammonia). The process being highly exothermic continues and the spontaneous ignition occurs. The solution underwent smoldering combustion with enormous swelling, producing white foamy and voluminous ash. The flame temperature, as high as 1400 - 1600 °C, converts the vapor phase oxides into mixed aluminates. The flame persists for ~30 seconds. The crucible is then taken out of the furnace and the foamy product can easily be milled to obtain the precursor powder.

The following phosphors were prepared –

$\text{SrAl}_2\text{O}_4\text{:Eu}$, $\text{SrAl}_2\text{O}_4\text{:Dy}$, $\text{SrAl}_2\text{O}_4\text{:Tb}$ at initiating temp. 500 °C and 600 °C with different dopant concentration.

2. Characterization of Phosphors:

The phosphors were characterized by different techniques viz. Absorption spectra / XRD / SEM / FTIR.

- Thermoluminescence Studies:** Thermoluminescence studies for UV-irradiated as well as γ -irradiated samples were done and some basic parameters such as trap depth, frequency factor, order of kinetics were calculated. The obtained results are listed in Table 3.
- Mechanoluminescence Studies:** The Mechanoluminescence studies of UV-irradiated samples were done which shows that the ML is recovered when the samples are irradiated with UV-radiation. Similarly the ML study of γ -irradiated samples were done; which shows that the ML intensity depends upon the γ -dose given, hence it was concluded that $\text{SrAl}_2\text{O}_4\text{:Eu}$ and $\text{SrAl}_2\text{O}_4\text{:Dy}$ could be used for ML-dosimetry.

SrAl_2O_4 Phosphors were synthesized with different dopants. The doping concentration for TL was optimized. Fig.1 shows the TL glow curve of SrAl_2O_4 with different concentration of Eu with γ - irradiation 1180Gy. It is clear that the TL intensity is maximum for 5 mole% concentration of Eu.

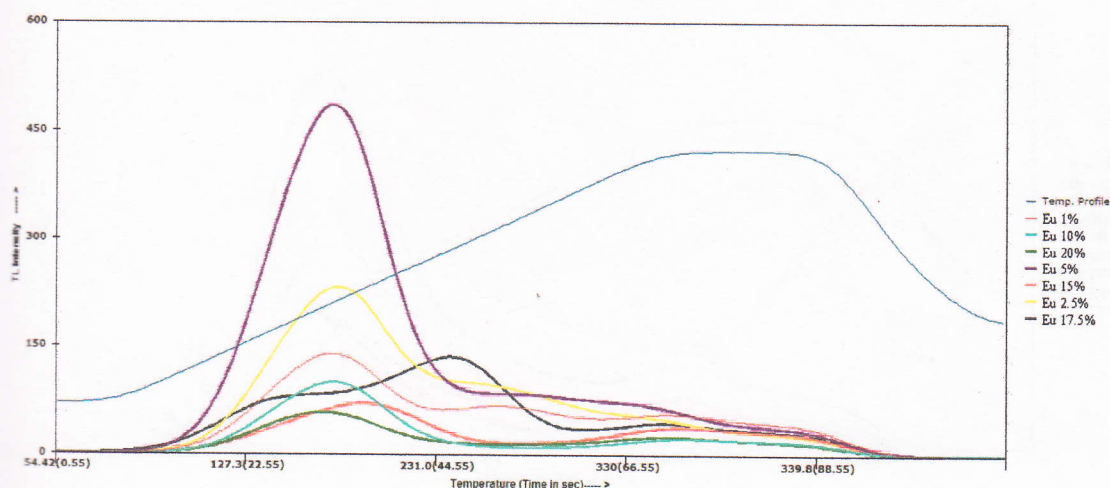


Fig. 1. TL glow curve of Eu doped SrAl_2O_4 with change in Eu concentration irradiated with 1180 Gy *1 dose*

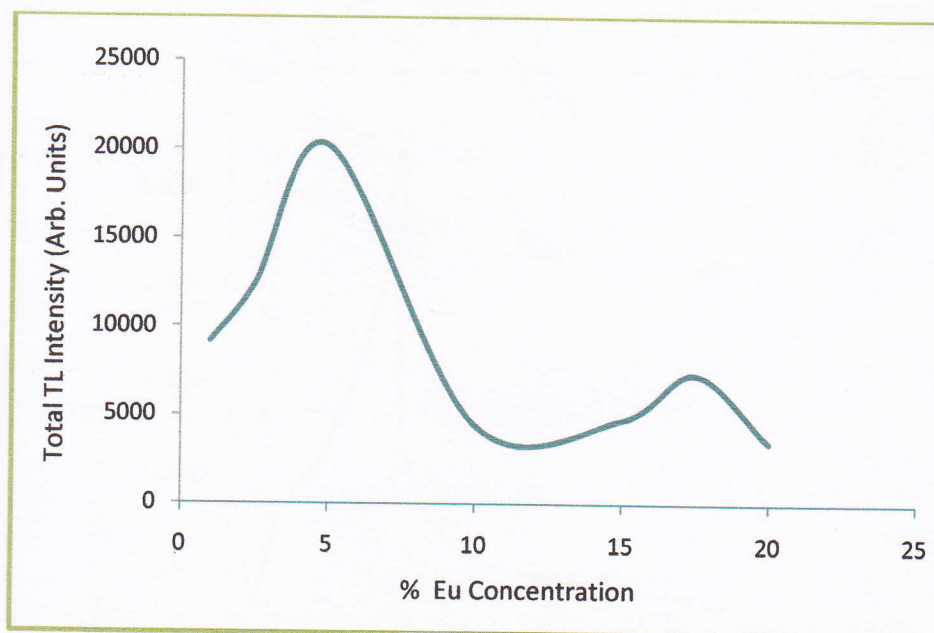


Fig. 2 Plot between % Eu concentration and Total TL Intensity with γ -dose of 1180 Gy.

Similarly the optimization for Dy concentration was done as shown in fig. (3). Which shows that TL intensity is maximum for 17.5% Dy concentration for 1180Gy of γ -dose. Fig.(4) shows the plot between %Dy concentration and Total TL Intensity with γ -irradiation dose of 1180 Gy.

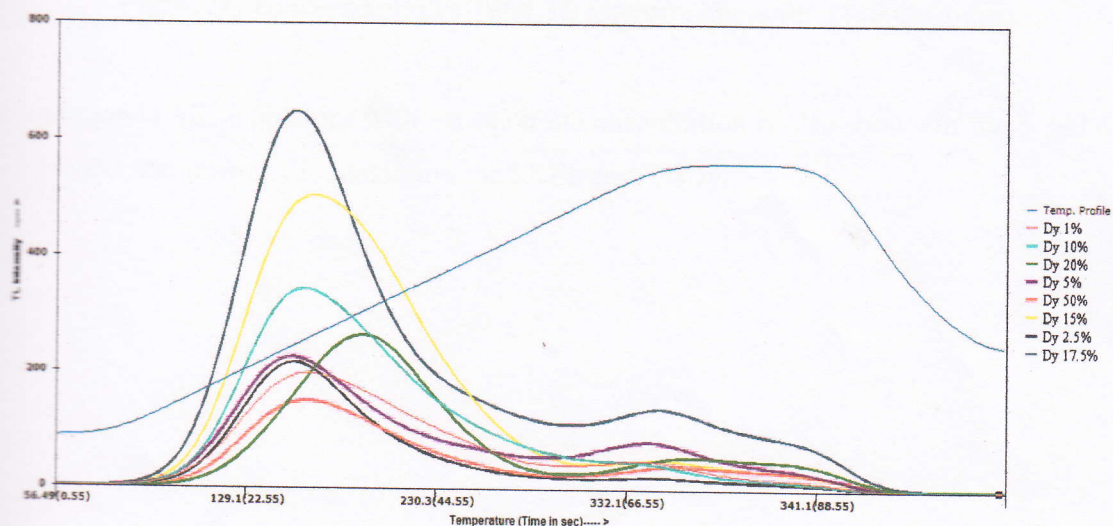


Fig 3 TL glow curve of Dy doped SrAl_2O_4 with change in Dy concentration irradiated with 1180 Gy.

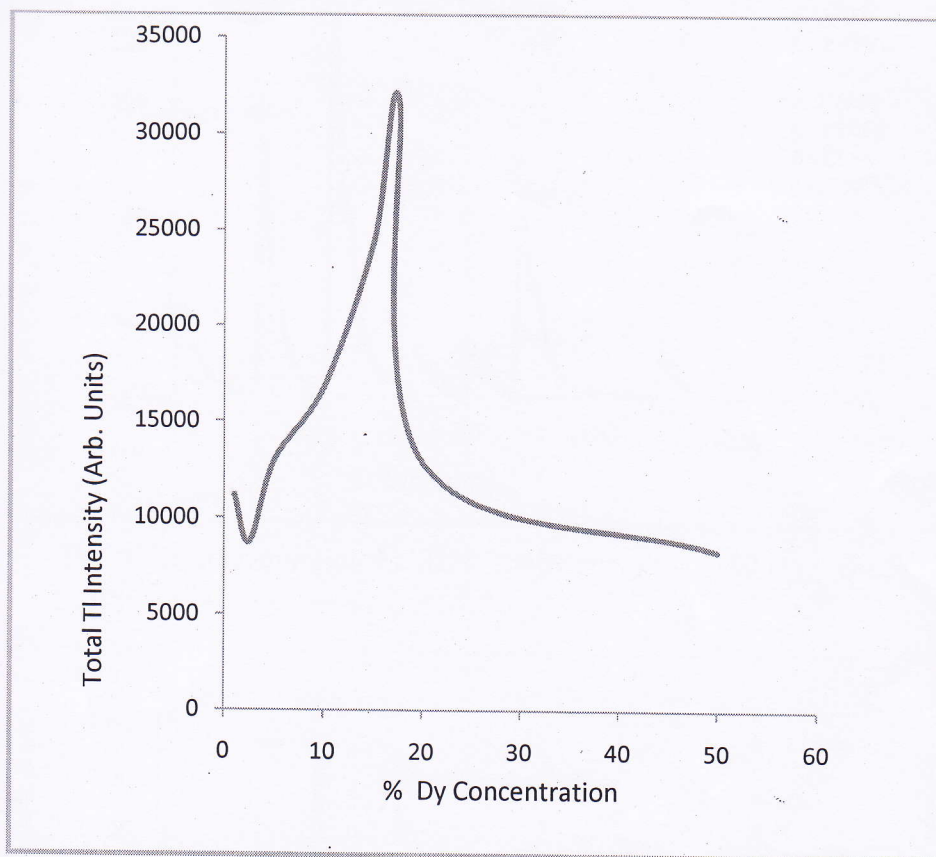


Fig. 4. Dy concentration vs Total TL intensity curve for 1180 Gy γ -dose

The variation in ML intensities with variation in concentration is also shown in fig. 5 and 6. It is clear that the ML intensity is maximum for 5%Eu and 5%Dy.

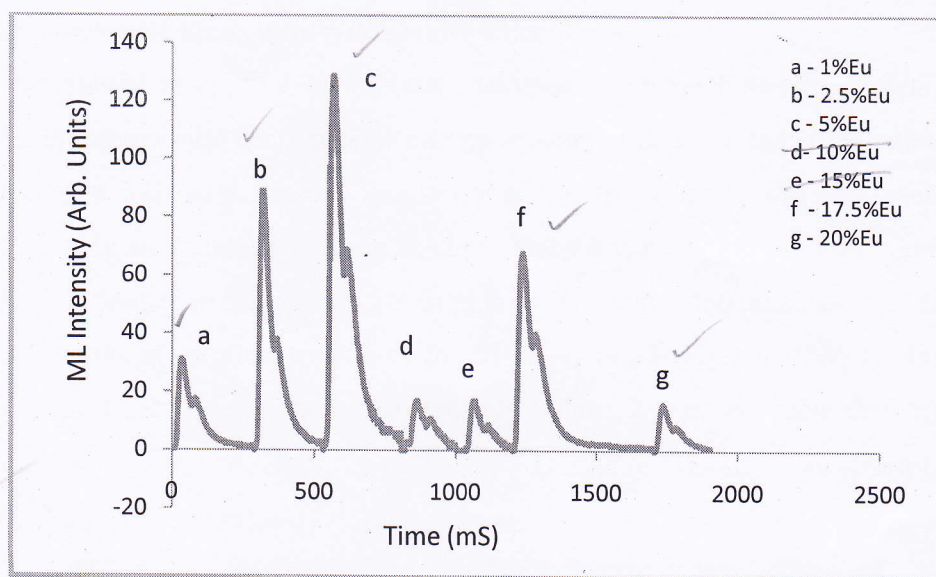


Fig. 5. Eu concentration vs ML intensity curve for 1180 Gy γ -dose

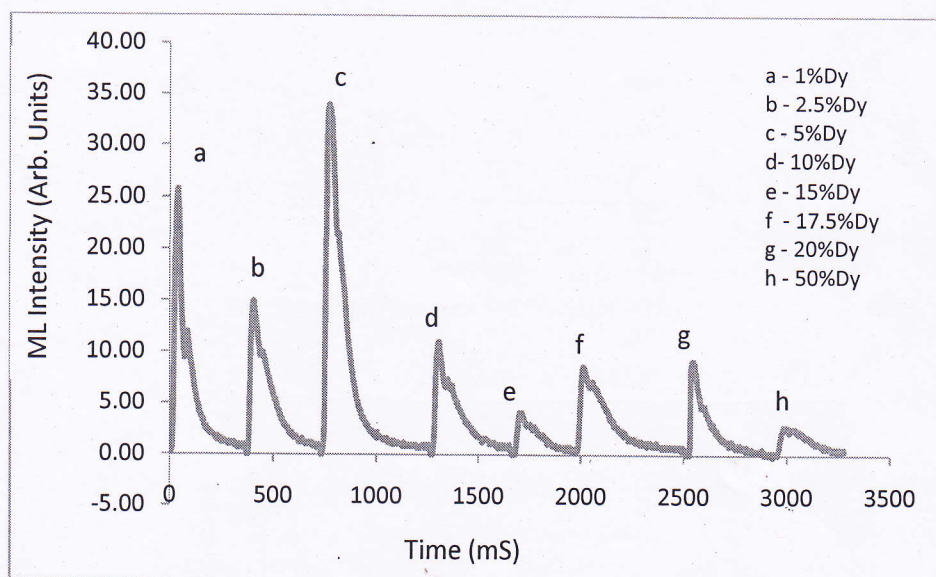


Fig. 6. Dy concentration vs ML intensity curve for 1180 Gy γ -dose

It was found that the TL properties were found best for 5% Eu, 17.5% Dy and 5% Tb. Hence the characterization, Thermoluminescence and Mechanoluminescence properties of these materials are discussed in brief in following sections.

Absorption Spectra of Strontium Aluminates Phosphors:

The study of optical absorption is important to understand the behavior of nano-crystals. A fundamental property is the band gap-the energy separation between the filled valence band and the empty conduction band. Optical excitation of electrons across the band gap is strongly allowed, producing an abrupt increase in absorption at the wavelength corresponding to the band gap energy. This feature in the optical spectrum is known as the optical absorption edge. Fig 7(a, b, and c) shows the absorption spectra of the 5% Eu, 17.5% Dy and 5% Tb doped SrAl_2O_4 Phosphors respectively in the range of 190nm-500nm. It can be seen that the spectra is featureless and no absorption occur for wavelength $\lambda > 390\text{nm}$ (visible). The absorption edge and the band gap E_g is given in table-1.

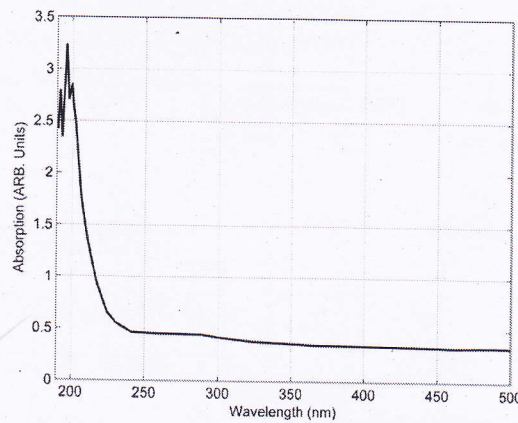


Fig. 7(a). Absorption spectra of $\text{SrAl}_2\text{O}_4:\text{Eu}(5\%)$

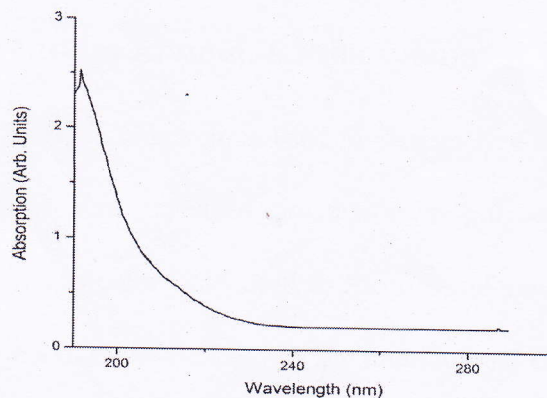


Fig. 7(b). Absorption spectra of $\text{SrAl}_2\text{O}_4:\text{Dy}(17.5\%)$

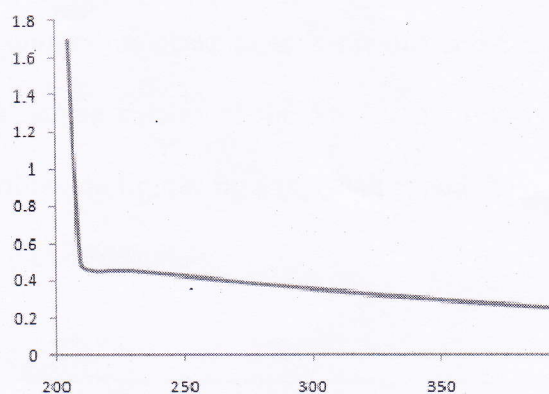


Fig.7(c) Absorption spectra of SrAl₂O₄:Tb(5%)

Table 1: Band Gap Calculation of doped SrAl₂O₄

	Absorption Edge (nm)	Band Gap E _g (eV)
SrAl ₂ O ₄ :Eu(5%)	240	5.16
SrAl ₂ O ₄ :Dy(17.5%)	236	5.24
SrAl ₂ O ₄ :Tb(5%)	210	5.89

XRD Spectra of Strontium Aluminates Phosphors:

Powder diffraction (XRD) is a technique used to characterize the crystallographic structure, crystallite size (grain size), and preferred orientation in polycrystalline or powdered solid samples. Powder diffraction is commonly used to identify unknown substances, by comparing diffraction data against a database maintained by the International Centre for Diffraction Data. It may also be used to characterize heterogeneous solid mixtures to determine relative abundance of crystalline compounds and, when coupled with lattice refinement techniques, such as Rietveld refinement, can provide structure information on unknown materials. Powder diffraction is also a

common method for determining strains in crystalline materials. An effect of the finite crystallite sizes is seen as a broadening of the peaks in an X-ray diffraction as is explained by the Scherrer Equation. The XRD diffraction pattern of the 5% Eu, 17.5% Dy and 5% Tb doped SrAl_2O_4 Phosphors is shown in following figures fig 8 (a, b and c) and the particle size with the planes are shown in Table 2(a, b and c) respectively.

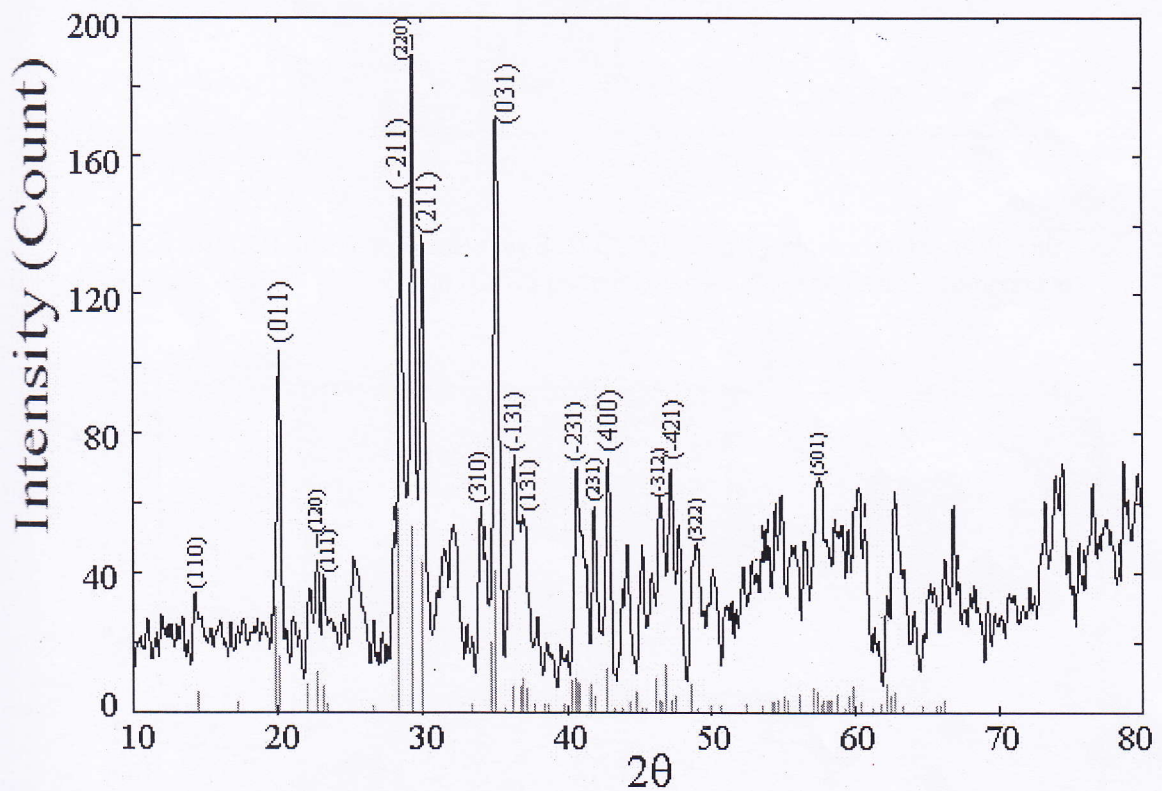


Fig.8 (a) XRD diffraction pattern of $\text{SrAl}_2\text{O}_4:\text{Eu}(5\%)$ which matches well with JCPDS 34-379. The JCPDS pattern is shown at the bottom for comparison.

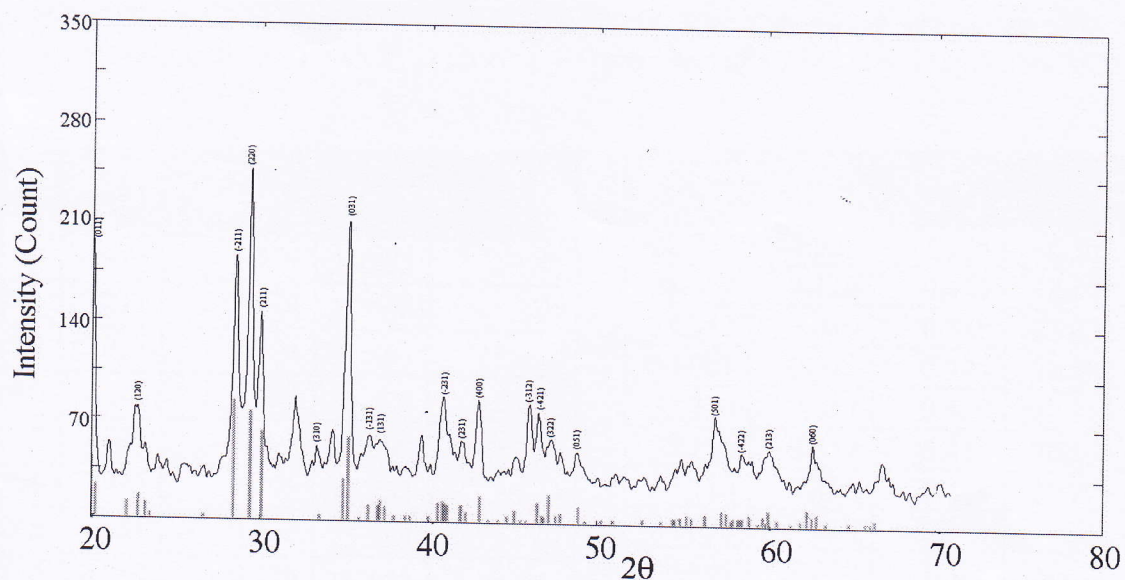


Fig.8 (b) XRD diffraction pattern of $\text{SrAl}_2\text{O}_4:\text{Dy}(17.5\%)$ which matches well with JCPDS 34-379. The JCPDS pattern is shown at the bottom for comparison.

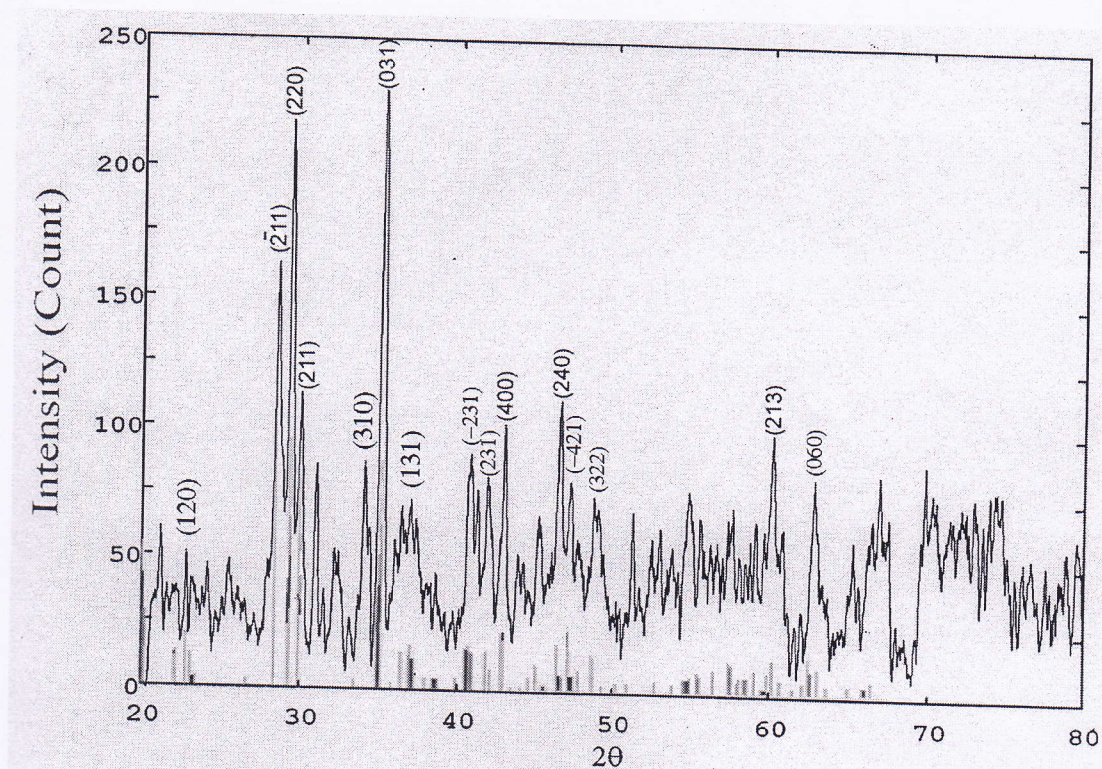


Fig.8(c) XRD diffraction pattern of $\text{SrAl}_2\text{O}_4:\text{Tb}(5\%)$ which matches well with JCPDS 34-379. The JCPDS pattern is shown at the bottom for comparison.

Table 2(a) Calculation of particle size and hkl parameters of $\text{SrAl}_2\text{O}_4:\text{Eu}(5\%)$ from XRD pattern.

2Theta	d (Å)	FWHM	Size (nm)	hkl
20.102	4.414	0.374	21.572	(0 1 1)
22.721	3.911	0.374	21.666	(1 2 0)
23.161	3.837	0.374	21.683	(1 1 1)
28.510	3.128	0.443	18.498	(-2 1 1)
29.353	3.040	0.391	21.009	(2 2 0)
30.008	2.975	0.461	17.853	(2 1 1)
34.052	2.631	0.533	15.588	(3 1 0)
35.124	2.553	0.423	19.690	(0 3 1)
36.359	2.469	0.820	10.197	(-1 3 1)
36.686	2.448	0.820	10.207	(1 3 1)
36.937	2.432	0.820	10.214	(-2 3 1)
40.706	2.215	0.400	21.183	(2 3 1)
41.937	2.153	0.466	18.273	(4 0 0)
45.927	1.974	0.594	14.523	(-3 1 2)
46.499	1.951	0.500	17.293	(-4 2 1)
47.826	1.900	0.435	19.959	(3 2 2)
56.520	1.627	0.435	20.715	(5 0 1)
Average Particle Size (nm)=			17.654	

Table 2(b) Calculation of particle size and hkl parameters of $\text{SrAl}_2\text{O}_4:\text{Dy}(17.5\%)$ from XRD pattern.

2Theta	d (Å)	FWHM	Size (nm)	hkl
22.747	3.906	1.274	6.360	(1 2 0)
28.509	3.128	0.445	18.407	(-2 1 1)
29.352	3.040	0.348	23.632	(2 2 0)
30.001	2.976	0.394	20.880	(2 1 1)
33.320	2.687	0.320	25.914	(3 1 0)
35.136	2.552	0.414	20.152	(0 3 1)
36.410	2.466	0.414	20.225	(-1 3 1)
37.038	2.425	0.414	20.261	(1 3 1)
40.804	2.210	1.225	6.920	(-2 3 1)
41.884	2.155	1.225	6.945	(2 3 1)
42.893	2.107	0.591	14.449	(4 0 0)
45.918	1.975	0.569	15.171	(-3 1 2)
46.463	1.953	1.721	5.024	(-4 2 1)
47.232	1.923	1.721	5.038	(3 2 2)
48.770	1.866	1.721	5.069	(0 5 1)
56.973	1.615	1.302	6.942	(5 0 1)
58.561	1.575	1.302	6.995	(-4 2 2)
60.182	1.536	2.732	3.361	(2 1 3)
62.762	1.479	1.798	5.177	(0 6 0)
Average Particle Size (nm)=			12.470	

Table 2(c) Calculation of particle size and hkl parameters of $\text{SrAl}_2\text{O}_4:\text{Tb}(5\%)$ from XRD pattern.

2Theta	d (Å)	FWHM	Size (nm)	(hkl)
28.512	3.128	0.360	22.769	(-2 11)
29.331	3.043	0.340	24.152	(2 2 0)
29.989	2.977	0.360	22.845	(2 1 1)
34.033	2.632	0.320	25.962	(3 1 0)
35.132	2.552	0.400	20.832	(0 3 1)
36.372	2.468	0.280	29.864	(1 3 1)
41.114	2.194	0.420	20.201	(-2 3 1)
41.857	2.156	0.520	16.357	(2 3 1)
42.934	2.105	0.360	23.712	(4 0 0)
46.507	1.951	0.360	24.019	(2 4 0)
47.199	1.924	0.300	28.898	(-4 2 1)
48.720	1.868	0.320	27.252	(3 2 2)
60.110	1.538	0.360	25.495	(2 1 3)
62.776	1.479	0.440	21.150	(0 6 0)
Average Particle Size (nm) =			23.822	

Thermoluminescence Studies of Strontium Aluminates Phosphors:

A) TL study of UV-irradiated $\text{SrAl}_2\text{O}_4:\text{Eu}(5\%)$: The TL glow curve for UV-irradiated $\text{SrAl}_2\text{O}_4:\text{Eu}(5\%)$ is shown in fig.9(a). The irradiation time was varied from 5 min to 25 min.

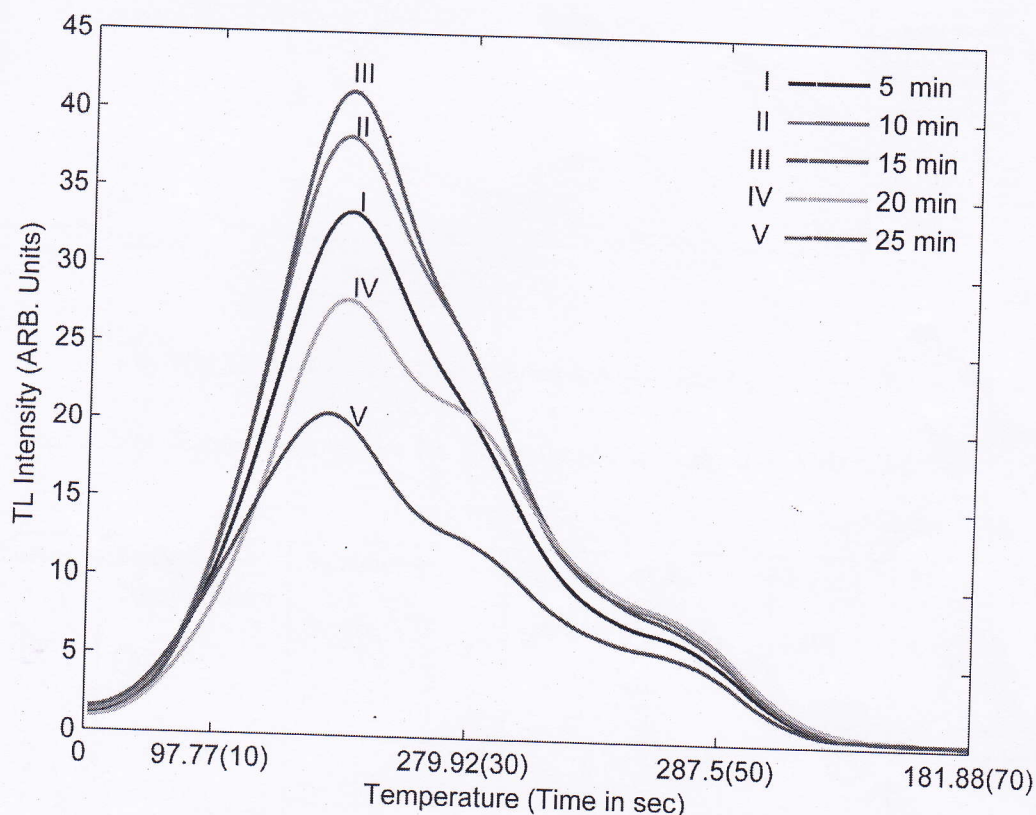


Fig. 9(a) TL glow curve of UV-irradiated $\text{SrAl}_2\text{O}_4:\text{Eu}(5\%)$.

The trap depth was calculated using Chen's peak method and initial rise method giving trap depth 0.25 eV and 0.24 eV respectively. The TL intensity is maximum for 15 minutes irradiation time. Since UV irradiation excites the atoms at the surface only hence initially the TL intensity increases with irradiation time and becomes maximum for 15 min. after that the bleaching of color centers takes place and TL intensity decreases on further increase of irradiation time.

B) TL study of γ -irradiated $\text{SrAl}_2\text{O}_4:\text{Eu}(5\%)$: TL glow curve of γ -irradiated irradiated $\text{SrAl}_2\text{O}_4:\text{Eu}(5\%)$ is shown in fig. 9(b). The γ -dose was varied from 49.16 Gy to 2360 Gy. Symmetry factor and Trap depth was calculated, which is listed in table- 3(a).

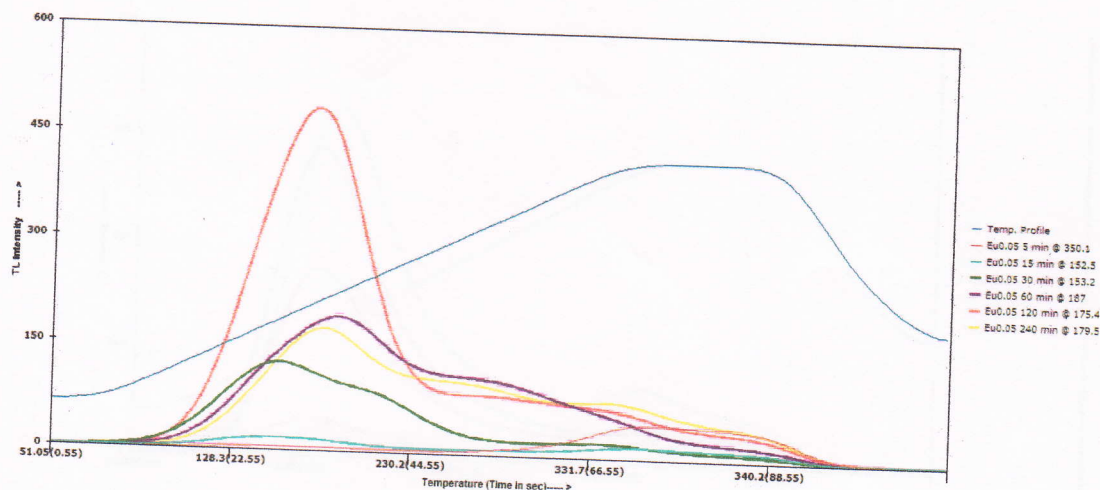


Fig. 9(b) TL glow curve of γ -irradiated $\text{SrAl}_2\text{O}_4:\text{Eu}(5\%)$

Table: 3(a)- Kinetics parameter for TL study of γ -irradiated $\text{SrAl}_2\text{O}_4:\text{Eu}(5\%)$

γ -Dose (Gy)	Peak Temperature ($^{\circ}\text{C}$)	Symmetry factor μ	E_{τ} (eV)	E_{δ} (eV)	E_{ω} (eV)	Mean E (eV)
147.5	152.52	0.57	0.58	0.43	0.36	0.47
295	153.72	0.65	0.71	0.37	0.32	0.47
590	186.97	0.67	0.58	0.26	0.18	0.34
1180	175.4	0.46	0.56	0.53	0.44	0.51
2360	179.47	0.73	0.83	0.26	0.21	0.43

C) TL study of γ -irradiated $\text{SrAl}_2\text{O}_4:\text{Dy}(17.5\%)$: TL glow curve of γ -irradiated $\text{SrAl}_2\text{O}_4:\text{Dy}(17.5\%)$ is shown in fig. 9(c). The γ -dose was varied from 49.16 Gy to 2360 Gy. Symmetry factor and Trap depth calculated is listed in table- 3(b).

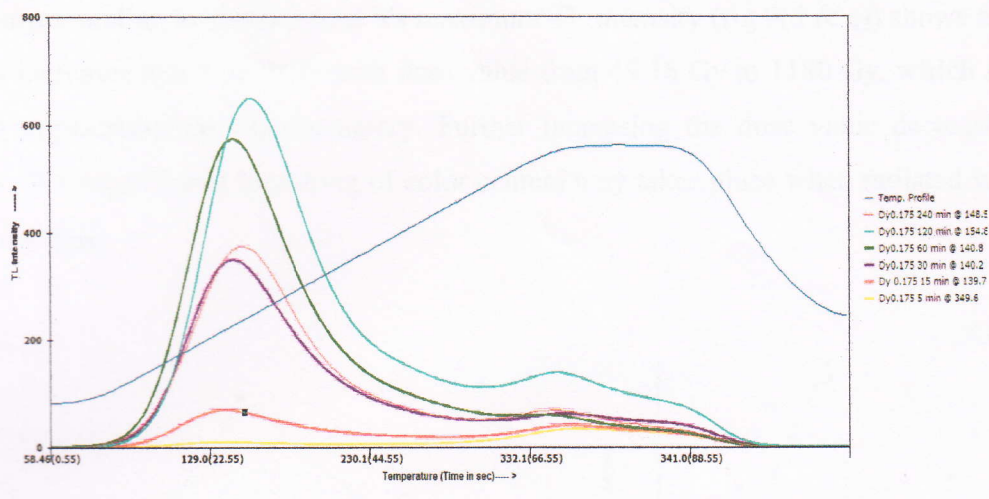


Fig. 9(c) TL glow curve of γ -irradiated $\text{SrAl}_2\text{O}_4:\text{Dy}(17.5\%)$.

Table:3(b)- Kinetics parameter for TL study of γ -irradiated $\text{SrAl}_2\text{O}_4:\text{Dy}(17.5\%)$

γ -Dose (Gy)	Peak Temperature ($^{\circ}\text{C}$)	Symmetry factor μ	E_{τ} (eV)	E_{δ} (eV)	E_{ω} (eV)	Mean E (eV)
147.5	139.69	0.63	0.77	0.47	0.43	0.56
295	140.17	0.61	0.75	0.51	0.46	0.57
590	140.8	0.61	0.84	0.56	0.32	0.64
1180	154.79	0.56	0.69	0.54	0.47	0.57
2360	148.5	0.56	0.68	0.53	0.46	0.55

A plot corresponding to gamma dose Vs maximum TL intensity (fig 9(d & e)) shows that the TL intensity increases nearly linearly with dose value from 49.16 Gy to 1180 Gy, **which shows the possible application in TL dosimetry**. Further increasing the dose value decreases the TL intensity. We suggest that bleaching of color centers may takes place when radiated with higher values of γ -dose.

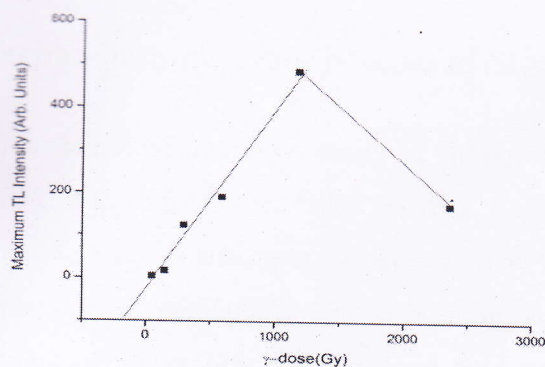


Fig. 9(d) γ -dose vs I_{\max} of SrAl₂O₄:Eu(5%)

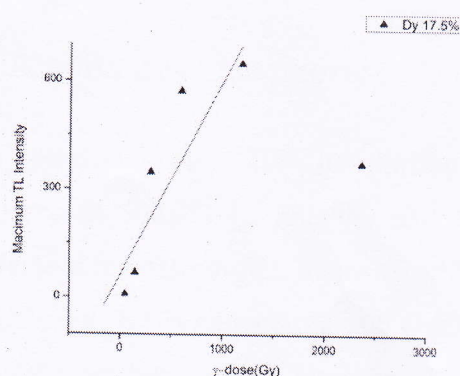


Fig. 9(e) γ -dose vs I_{\max} of SrAl₂O₄:Dy(17.5%)

D) TL study of UV-irradiated SrAl₂O₄:Tb(5%): TL glow curve of UV-irradiated SrAl₂O₄:Tb(5%) is shown in fig. 9(f). The irradiation time was varied from 1 min to 10 min.

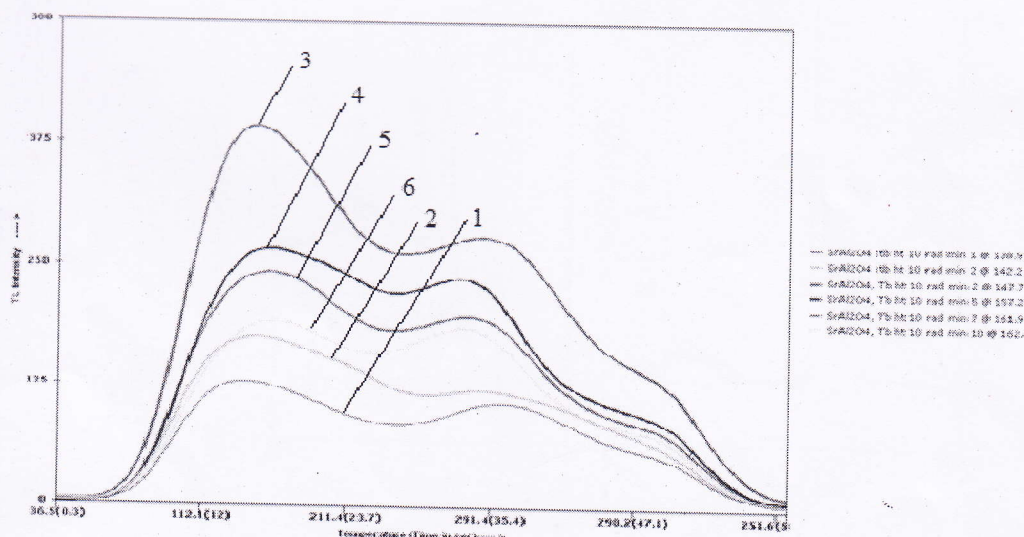


Fig. 9(f) UV-irradiated TL glow curve of SrAl₂O₄:Tb(5%).

Two peaks are observed at 136.2⁰C and 288.8⁰C suggesting the presence of two trap levels. The intensity of first peak is high as the concentration of trap center is high in first trap level. From fig it is clear that the intensity increases with increase in the UV exposure time and it is maximum for radiation time 2 minute for SrAl₂O₄: Tb. On further increase in exposure time, TL intensity decreases. We suggest that quenching of colour centers may take place when samples are exposed to UV light for longer period and TL intensity decreases.

Mechanoluminescence Studies of Strontium Aluminates Phosphors:

- A) **Mechanoluminescence study of SrAl₂O₄:Eu(5%) phosphor after UV- irradiation:** The ML study of UV-irradiated SrAl₂O₄:Eu(5%) prepared at 500 °C (sample A) and 600°C (Sample B) is shown in Fig 10(a). There is considerable increase in ML intensity when the phosphor is prepared at 600°C; because when SrAl₂O₄:Eu(5%) is prepared at 600°C it exists more in monoclinic form and only monoclinic phase contribute to ML intensity. The ML intensity depends upon the load applied it increases with the increase in load fig.10(b). After repetitive application of load the ML intensity ceases and disappear this is recovered when the sample is irradiated with UV-irradiation (fig. 10(c)).

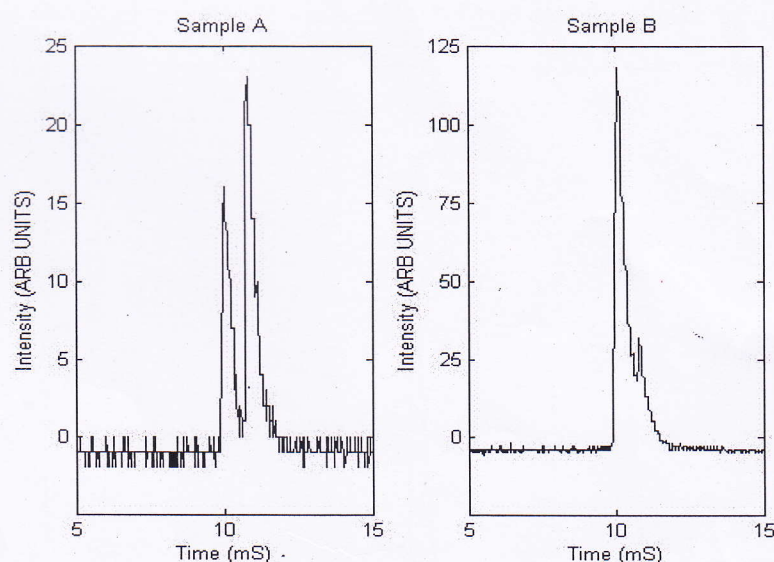


Fig. 10(a): ML Characteristics of SrAl₂O₄:Eu for 2.62 m/s velocity of 400 gm piston.

Variation in ML Intensity with Load Applied

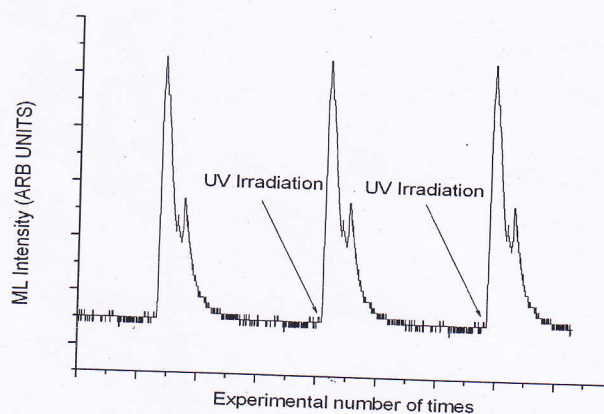
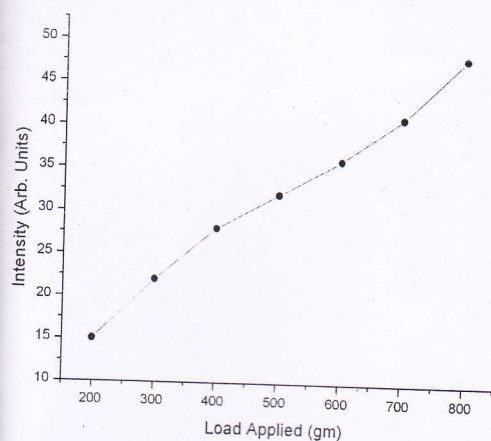


Fig. 10(b) Change in ML intensity with load applied Fig. 10(c) Recovery phenomenon of ML intensity with UV irradiation.

B) Mechanoluminescence study of $\text{SrAl}_2\text{O}_4:\text{Eu}(5\%)$ phosphor after γ - irradiation: When a constant load is dropped from a fixed height, the ML intensity increases with the γ -dose which is shown in fig 10(d). it shows the ML intensity with the γ -dose a) 295 Gy b) 590 Gy c) 1180 Gy d) 2360 Gy. When maximum ML intensity is plotted with the γ -dose we get a curve which is linear between 295 Gy to 1180 Gy after which it increases slightly fig 10(e). **We suggest that $\text{SrAl}_2\text{O}_4:\text{Eu}(5\%)$ can be used for ML dosimetry in the range 295 Gy to 1180 Gy.** Similar result is obtained in Total ML intensity vs Dose plot fig 10(f).

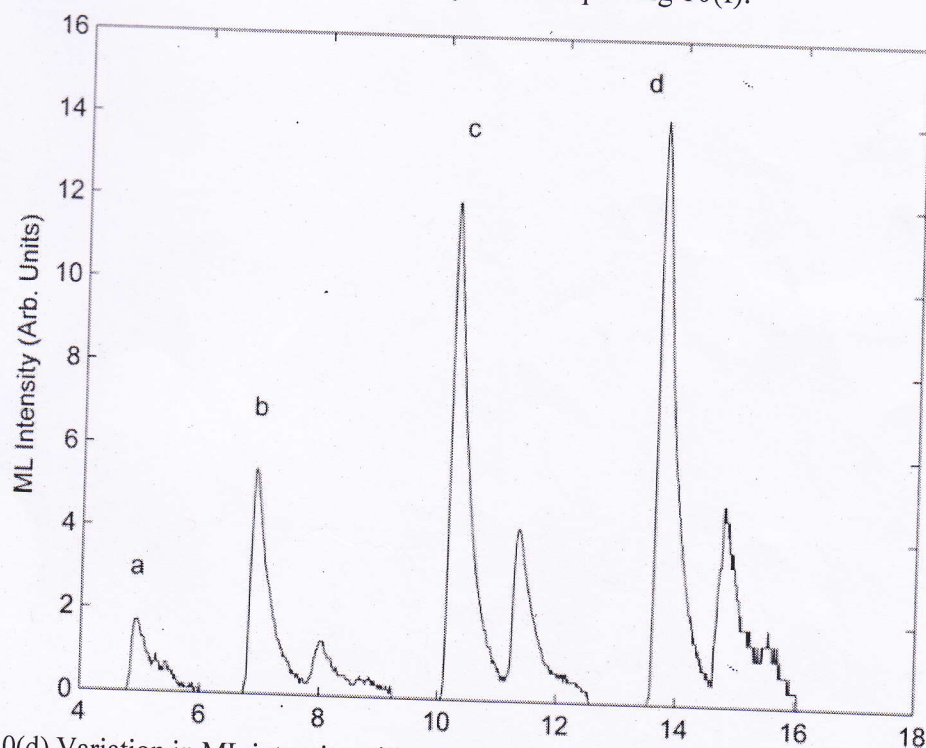


Fig. 10(d) Variation in ML intensity with γ -dose of $\text{SrAl}_2\text{O}_4:\text{Eu}(5\%)$. a) 295 Gy b) 590 Gy c) 1180 Gy d) 2360 Gy

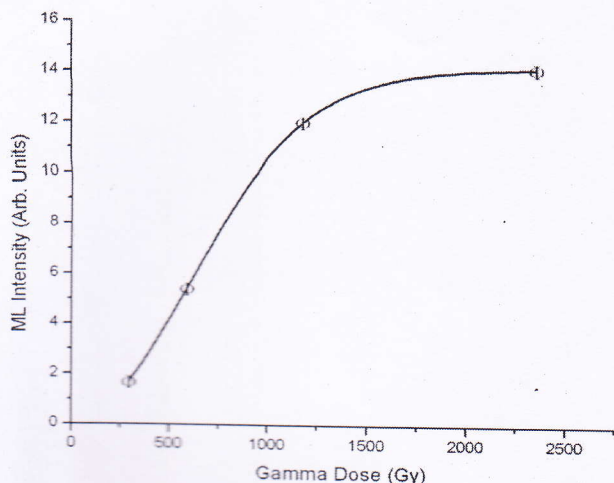


Fig. 10(e) γ -dose vs ML intensity of SrAl₂O₄:Eu(5%)

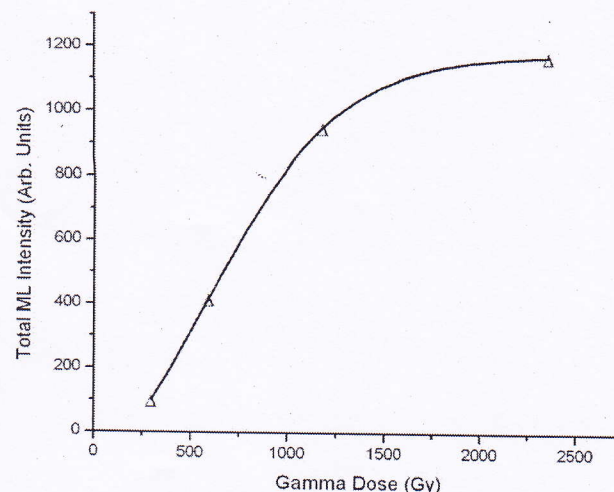


Fig. 10(f) γ -dose vs Total ML intensity of SrAl₂O₄:Eu(5%)

C) **Mechanoluminescence study of SrAl₂O₄:Dy(5%) phosphor after γ -irradiation:** Since the ML intensity was maximum for 5% Dy doped SrAl₂O₄, the γ -irradiated ML study for SrAl₂O₄:Dy(5%) is shown in fig. 10(g). It shows the ML intensity with the γ -dose a) 295 Gy b) 590 Gy c) 1180 Gy d) 2360 Gy. The ML intensity increases with the gamma-dose which is nearly linear between 295 Gy to 1180 Gy Fig. 10(h).

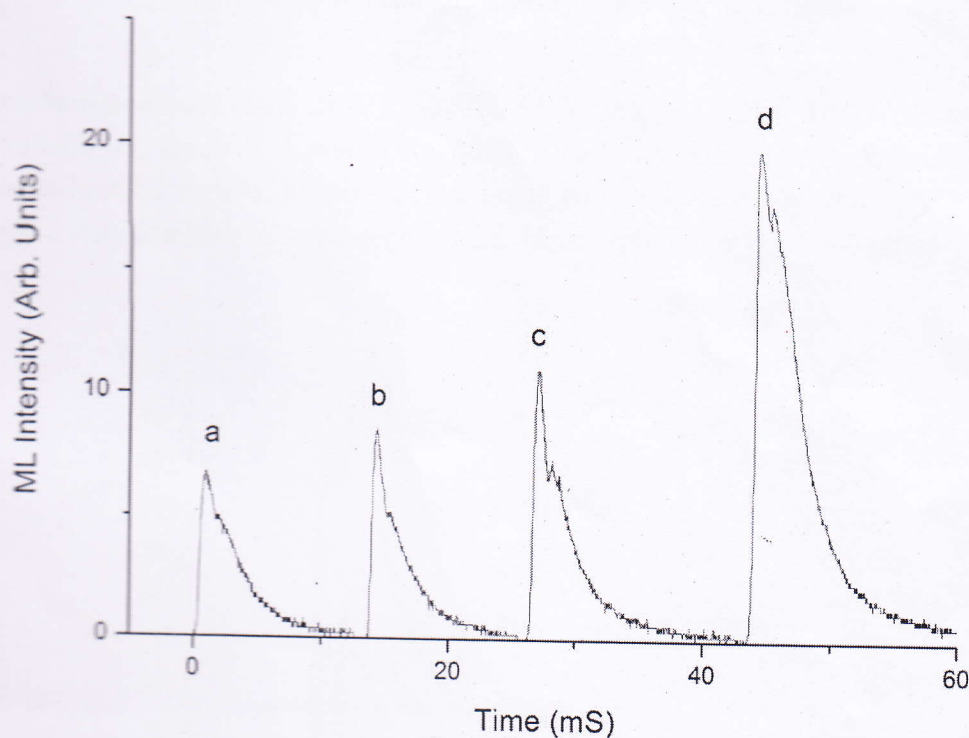


Fig. 10(g) Variation in ML intensity with γ -dose of SrAl₂O₄:Dy(5%). a) 295 Gy b) 590 Gy c) 1180 Gy d) 2360 Gy

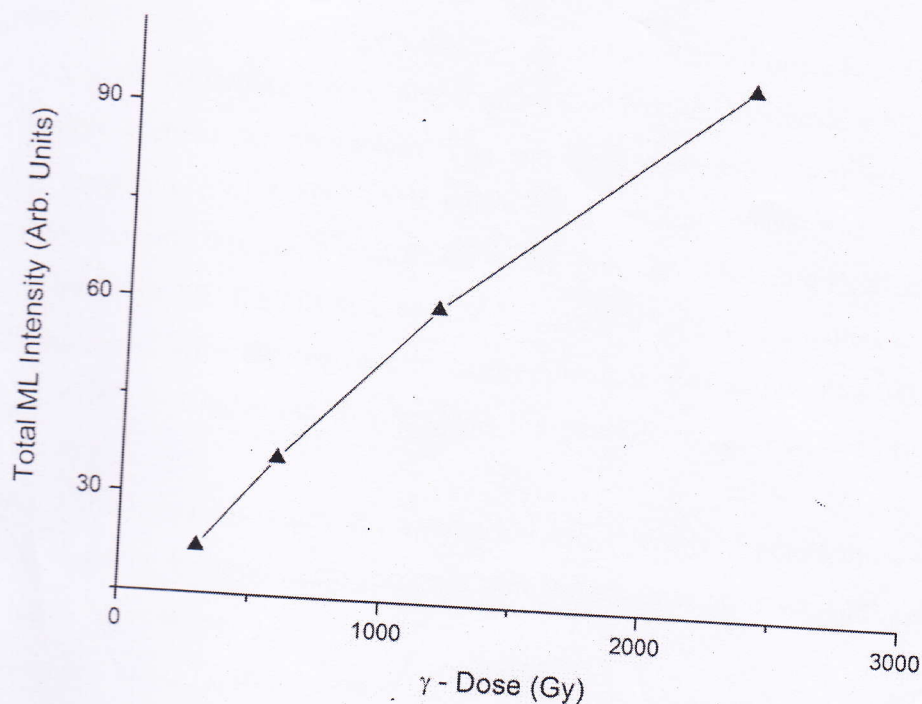


Fig. 10(h) γ -dose vs Total ML intensity of $\text{SrAl}_2\text{O}_4:\text{Dy}(5\%)$

D) Mechanoluminescence study of $\text{SrAl}_2\text{O}_4:\text{Tb}(5\%)$ phosphor after UV-irradiation: The UV-irradiated ML study is shown in fig. 10(i). Clearly the ML intensity increase with the increase in irradiation time, it becomes maximum and then decreases with irradiation time, we suggest that bleaching of color centers takes place with higher irradiation time.

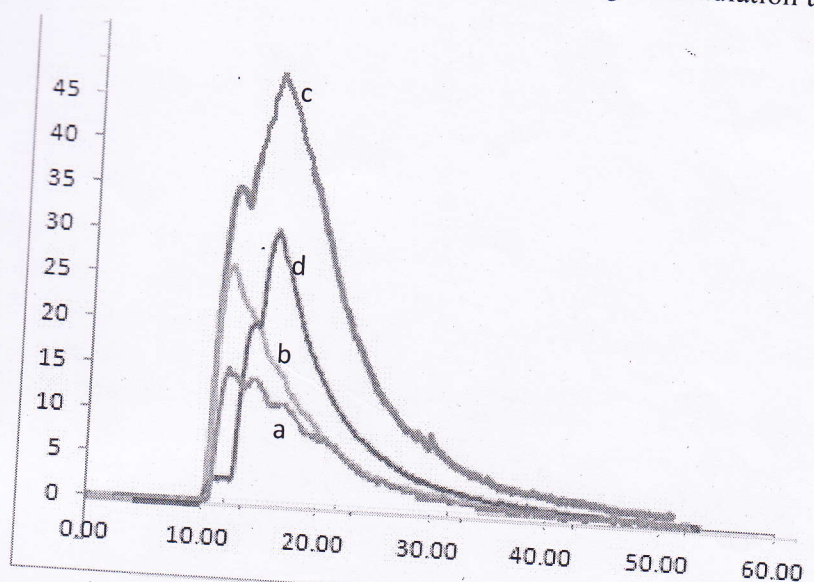


Fig. 10(i) ML study of $\text{SrAl}_2\text{O}_4:\text{Tb}(5\%)$ a) 5min b) 10 min c) 15 min and d) 20 min UV irradiation time

Conclusion and Significance of the present work:

The work done in the present project was carried out as planned originally. Some the rare-earth doped Strontium Aluminates were prepared by Combustion Synthesis which appears to be a more feasible method for production. The strontium aluminate phosphors were prepared at initiating temperature of 600°C as it exists in monoclinic phase when prepared at 600°C temperature and only this phase contributes to ML Intensity. The identification of the phosphors was done by XRD, SEM, FTIR studies. The ML study after UV-irradiation reveals that the ML intensity increases with the increase in load applied; it also shows that ML phenomenon is recovered with UV-irradiation; this suggests the possible application for pressure sensor and impact sensors.

The gamma irradiated ML study shows that the ML intensity depends upon the gamma dose given to the system, it shows linear response with gamma irradiation which suggests the possible application in ML dosimetry.

The gamma irradiated TL study shows that the TL intensity depends upon the gamma-dose given which suggests the possible application in TL dosimetry.

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References:

1. W. A. Hollerman, N. P. Bergeron, S. M. Goedeke, S. W. Allison, C. I. Muntele, D. Ila and R. J. Moore, Surf. Coating Technol. 210, 8382 (2007).
2. Nameeta Brahme, Manju Shukla, D.P. Bisen, U. Kurrey, Anil Choubey, R.S. Kher Manish Singh Journal of Luminescence 131 (2011) 965-969.
3. S. Aman and J. Tomas, Power Technol. 146, 147 (2004).
4. B. P. Chandra, C. N. Xu, H. Yamada and X. G. Zheng, J. Luminescence 130, 442 (2010).
5. Nathan C. Eddingsaas and Kenneth S. Suslick, J. Am. Chem. Soc. 129, 6718 (2007).
6. N. P. Bergeron, W. A. Hollerman, S. M. Goedeke and R. J. Moore, Int. J. Impact Eng. 35, 1587 (2008).

7. Y. Kawaguchi, Jpn. J. Appl. Phys. 37, 1892 (1998).
8. B. P. Chandra, "Mechanoluminescence in luminescent solids," edited by Vij, D.R., Plenum Press, New York, pp 361-389. (1998).
9. J. S. Kim, Y. N. Kwon, N. Shin and K. S. Sohn, Appl. Phys. Lett. 90, 241916 (2007).
10. C. N. Xu, X. G. Zheng, M. Akiyama, K. Nonaka and T. Watanabe, Appl. Phys. Lett. 76, 179 (2000).
11. K. S. Sohn, S. Y. Seo, Y. N. Kwon and H. D. Park, J. Am. Ceramic Soc. 85, 712 (2002).
12. B. P. Chandra, R. N. Baghel, A. K. Luka, T. R. Sanodiya, R. K. Kuraria and S. R. Kuriaria, J. Lum. 129, 760 (2009).
13. B. P. Chandra, J. Lum. 128, 1217 (2008).
14. B. P. Chandra, S. K. Mahobia, P. Jha, R. K. Kuraria S. R. Kuraria R. N. Baghel and S. Thaker, J. Lum. 128, 2038 (2008).
15. B. P. Chandra, A. K. Bagri and V. K. Chandra, J. Lum. 130, 309 (2010).
16. P. C. Palilla, A. K. Levine, M. R. Tomkus, Fluorescent properties of alkaline earth aluminates of the type MAI_2O_4 activated by divalent europium, *J. Electrochem. Soc.*, **115**, 642 (1968).
17. W. Y. Zia, H. B. Yuan, W. M. Yen, Phosphorescent dynamics in $SrAl_2O_4: Eu^{2+}, Dy^{3+}$ single crystal fibers, *J. Luminescence*, **76**, 424 (1998).
18. G. Blasse, W. L. Wanmaker, A. Bril, Fluorescence of Eu^{2+} activated alkaline earth aluminates, *Philip. Res. Rep.*, **23**, 201 (1968).
19. G. Groppi, C. Christiani, P. Forzatti, Phase composition and mechanism of formation of Ba- \square -alumina-type systems for catalytic combustion prepared by precipitation, *J. Mater. Sci.*, **29**, 3441 (1994).
20. M. Peng, Z. Pei, G. Hong, Q. Su, Study on the reduction of $Eu^{3+} - Eu^{2+}$ in $Sr_4Al_{14}O_{25}:Eu$ prepared in air atmosphere, *Chem. Phys. Lett.*, **371**, 1 (2003).
21. J. Holsa, J. Hogne, M. Lastusaari, J. Niittykoski, Persistent luminescence of Eu^{2+} doped alkaline earth aluminates $MAI_2O_4:Eu^{2+}$, *J. Alloys Compounds*, **324**, 326 (2001).
22. M. Akiyama, C. N. Xu, K. Nonaka, T. Watanabe, Intense visible light emission from $Sr_3Al_2O_6:Eu, Dy$, *Appl. Phys. Lett.*, **73**, 3046 (1998).
23. C. N. Xu, T. Watanabe, M. Akiyama, X. G. Zhang, Direct view of stress distribution in solid by Mechanoluminescence, *Appl. Phys. Lett.*, **74**, 2414 (1999).
24. C. N. Xu, Hiroshi Yamada, Xusheng Wang, Xu Guang Zheng, Strong elasticoluminescence from monoclinic structure $SrAl_2O_4$, *Appl. Phys. Lett.*, **84(16)**, 3040 (2004).

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