Mechanoluminescence behaviour on Eu$^{2+}$/Dy$^{3+}$ activated SrAl$_2$O$_4$ phosphor

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Abstract. Synthesis of Eu/Dy rare earth doped strontium aluminate phosphor or material with varying concentration of co-dopant (Dy$^{3+}$) and fixed concentration of dopant (Eu$^{2+}$) is reported inside the manuscript. Phosphor are prepared and synthesized by using the technique of solid-state reaction in reducing atmosphere and activated charcoal was used to create the atmosphere. Mechanoluminescence pattern were recorded for variable concentration of doping / co-doping ions and it is found with the increment in the concentration of co-dopant up-to 1 mol%, the ML intensity increases and after that the ML intensity decrement occurs due to the phenomenon of concentration quenching. Similarly, for the optimized concentration the ML pattern obtained for gamma – irradiated phosphor and it shows linear response with dose so our study supports the ML dosimeter application to detect the radiation via mechanoluminescence study. The suitable models are compared herein, which are already reported by so many authors.

1. Introduction
The concept of occurrence of mechanoluminescence behaviour, in which the light is emitted by phosphors when they undergo with mechanical deformation like rubbing, friction, cutting, collision, grinding and striking is reported by so many authors [1-3]. Various reports say that on around 50% of organic molecular solids and inorganic type of substances possesses the mechanoluminescence behaviour when deformed mechanically. Some researchers [4, 5] have got focus on the application of this mechanoluminescence phenomenon for the deformation detection or for finding the mechanical stresses generated from a far or remote location in which they use to convert the mechanical energy developed in to the visible source of lights. Earlier the detection of the developed mechanical energy is carried out with the use of piezoelectric substances, but this piezoelectric kind detection technique involves the use of electrodes to be in physical contacts with the material required testing and analysis for conversion of the mechanical energy in to the electrical pulses or signals.

The sensing of developed mechanical stresses can be carried out from a far or remote location only when the emission induced by mechanoluminescence is very strong, but the drawback associated with
this is that, the intensity produced in these occasions are normally very low due to which the real-life practical employability of this phenomenon becomes too much burdensome.

Till date, only a very few numbers of studies were reported for the non-destructive kind of mechanoluminescence development in substances. The phosphors based on SrAl$_2$O$_4$ when they are produced by codoping with the rare earth substances like Eu$^{2+}$ and Dy$^{3+}$ exhibits greater qualities like either no radiation or very less radiation, long lasting time for light emitting, higher side brightness and a very good quality of photo resistance. These newly developed phosphors have got a tremendous usages or applications in variety of fields of sensing and actuating [6]. A lot of studies are reflecting that the mechanoluminescence behaviour is observed when the holes are got released due to the occurrence of deformation/stresses on the crystalline phosphors and then these holes are got associated with the metastable Eu$^{+1}$ [6]

The study of luminescent phosphors like SrAl$_2$O$_4$:Ce, SAO:Ce and SrAl$_2$O$_4$:Ce,Ho, SAOHoCe, which are emitting the ultraviolet mechanoluminescence were reported by Zhang et al. [7]. The possibility behind the use of phosphors SrAl$_2$O$_4$:Eu$^{2+}$, Dy$^{3+}$ (SAO) is of great interest as these materials can be utilized for formation of the non-destructive and reproducible kind of load sensors or stress sensors has been reported by Sohn et al. [8].

The concept of impulsive excitation in the mechanoluminescent materials like SrAl$_2$O$_4$ have been reported by Jha et. al [9]. The authors have also reported that with the successive and consecutive impacts & collisions provided by loads on to the prepared phosphors the mechanoluminescence intensity have got decreased and the declined & reduced intensity of mechanoluminescence can roughly be recovered by using the UV irradiation [9].

The researchers Jha and Chandra [10] concluded about the presence of impulsive excitation of the mechanoluminescence phenomenon, in the prepared phosphors SrAl$_2$O$_4$:Eu$^{2+}$, Dy$^{3+}$ (SAO) when developed in the reducing environment defined by 95% Ar & 5% H$_2$ with using the solid-state reaction technique.

In this manuscript time resolved spectra shows the decay kinetics and it has persistence luminescence behaviour of prepared phosphor. The mechanoluminescence intensity of the prepared phosphor will increase with time because of the impulsive excitation produced due to the impact of load, gradually it increases and the it gets decreased after attaining the maximum value. When a graph has been plotted between the time and mechanoluminescence intensity, it has been observed that the peaks go on increasing and afterward they shift towards the smaller time values with the provision of increment in the impact velocities. The mechanoluminescence intensity which is represented by $I_m$, related and calculated on the basis of a graph plotted between the time and the mechanoluminescence intensity, has a tendency to increase linearly with the impact velocity. Also, the variation of the time $t_m$ is found linear and related with the factor 1000/V$_0$. The study of formation of mechanoluminescence, due to the impulsive excitation in the synthesized phosphors, have got a tremendous application on crack sensing as it is a very useful phenomenon in grasping the crack formation.

Sik kim et al. [11] monitored about the mechanoluminescence behaviour of SrAl$_2$O$_4$:Eu$^{2+}$, Dy$^{3+}$ materials & phosphors and they have reported that mechanoluminescence of SrAl$_2$O$_4$:Eu$^{2+}$, Dy$^{3+}$ phosphors are strongly dependent and are a functional factor of their expeditious loading rate.

They had reported about the systematic investigation of ML of SrAl$_2$O$_4$:Eu$^{2+}$, Dy$^{3+}$ phosphors using rate equations, which are almost same as experimental data. They have claimed that the ML of SrAl$_2$O$_4$:Eu$^{2+}$, Dy$^{3+}$ materials/phosphors is developed with the variation in load instead of static load, hence the rate of loading gives the profile of ML versus time curve.

Figure 1 represents the line diagram of the mechanism of the mechanoluminescence phenomenon in the prepared or synthesized SrAl$_2$O$_4$:Eu$^{2+}$ crystals. Here the step 1 represents the excitation of rare earth Eu$^{2+}$, step 2 represents the movement of electron in Conduction band, step 3 shows about the trapping of the electron, step 4 reveals about the release of the electrons, step 5 explained about the movement of the electron inside of the Conduction Band, step 6 shows the electron-capture at 5d of Eu$^{2+}$, and the step 7 shows the emission of the light. By using this representation, the locations of the
stages or band or levels of rare earth Eu$^{2+}$ and the traps in the band-gap were explained on the basis of data reported by Clabau et al. [12].

![Figure 1](image1.png)
**Figure 1.** Representation diagram of the ML emission in Eu$^{2+}$ doped phosphor [13].

![Figure 2](image2.png)
**Figure 2.** The technique of phosphorescence mechanism projected by Matsuzawa et al. for SrAl$_2$O$_4$:Eu$^{2+}$, Dy$^{3+}$ phosphors [12].

![Figure 3](image3.png)
**Figure 3.** The technique of phosphorescence mechanism projected by Beauger for SrAl$_2$O$_4$:Eu$^{2+}$ phosphors [12].

![Figure 4](image4.png)
**Figure 4.** The technique of phosphorescence mechanism projected by Clabau for SrAl$_2$O$_4$:Eu$^{2+}$, Dy$^{3+}$, B$^{3+}$ [12].

Figure 2, 3 and 4 shows the schematic diagram for the Phosphorescence mechanism proposed by Matsuzawa et al. for SrAl$_2$O$_4$:Eu$^{2+}$, Dy$^{3+}$ phosphors, Phosphorescence mechanism proposed by Beauger for SrAl$_2$O$_4$:Eu$^{2+}$ phosphors and Phosphorescence mechanism proposed by Clabau for SrAl$_2$O$_4$:Eu$^{2+}$, Dy$^{3+}$, B$^{3+}$ [12].

2. The constituent materials and the methods involved

2.1. Ingredient Materials utilized

Below mentioned table 1 contains the list of materials or chemicals utilized for making or synthesis of desired phosphors including their chemical formula, molecular weight in gm/mol and density in gm/cm$^3$. 

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Table 1. List of Chemicals with their molecular weight used for the synthesis of phosphors.

| S. No. | Chemical Formula | Name of Chemical       | Molecular Weight (gm/mol) | Density (gm/cm³) |
|--------|-----------------|------------------------|---------------------------|-----------------|
| 1.     | SrCO₃           | Strontium Carbonate    | 147.63                    | 3.5             |
| 2.     | Al₂O₃           | Aluminium Oxide        | 101.96                    | 3.95            |
| 3.     | H₂BO₃           | Boric Acid             | 61.83                     | 1.435           |
| 4.     | ZrO₂            | Zirconium Dioxide      | 123.22                    | 5.8             |
| 5.     | TiO₂            | Titanium Dioxide       | 79.87                     | 4.23            |
| 6.     | Eu₂O₃           | Europium Oxide         | 351.926                   | 7.4             |
| 7.     | Dy₂O₃           | Dysprosium Oxide       | 372.998                   | 7.8             |
| 8.     | (CH₃)₂CO        | Acetone                | 58.08                     | 0.7845          |
| 9.     | C               | Activated charcoal     | 12.011                    | 2.1             |

2.2. Preparation of samples

Many researchers have reported and concluded about the processes of synthesis and characterization of rare earth doped luminescent materials and phosphors [14-17]. The reports included about the combustion technique and solid-state reaction technique for synthesis. The several characterization techniques are XRD, SEM, TEM, EDS and FTIR, which provides the details of the synthesized phosphors.

To prepare the rare earth (europium and dysprosium) doped SrAl₂O₄ phosphor of different concentration, the stoichiometrically calculated (in mol% criteria of total weight of chemicals required for preparation of samples) quantity of ingredient chemicals are mixed to prepare a 5-gram sample and it is kept in alumina crucible and allowed for calcination (heated in muffle furnace for a time period of 2 hours) for a temperature of around 1000°C. After the calcination is done for 2 hours the prepared samples are allowed to cool inside the furnace and allowed to reach the room temperature. The samples were then taken out and successive intermediate grinding of calcinated samples is carried out using the mortar and pestle manually. The technique adopted for the preparation of rare earth Dy³⁺ and Eu²⁺ activated phosphor samples is a solid-state reaction/diffusion method. The constituent chemicals utilized for the preparation of desired phosphor are Strontium Carbonate (SrCO₃), Aluminium Oxide (Al₂O₃), Europium Oxide (Eu₂O₃), Dysprosium Oxide (Dy₂O₃) and Boric Acid (H₂BO₃) as flux.

For making the homogeneous powder mixture of samples, the mixture of reagent ingredients are grinded firmly using the agate mortar and pestle. The time required for making the firmly homogenous mixture is around 45 minutes by grinding process. After the intermediate grinding of samples, they were again transferred in to the alumina crucible for sintering process. These properly mixed and grinded samples are again kept in alumina crucible and heated in the muffle furnace up to a temperature of 1250°C for a time period of 3 hours, while keeping the reducing environment inside the furnace, with placement of a couple of activated charcoal filled crucibles along with the crucible of phosphor samples [18]. Then after the desired and prepared samples are allowed to cool naturally inside the furnace up to the room temperature.

The chemical reaction take places is:

\[
SrCO_{3} + Al_{2}O_{3} \rightarrow SrAl_{2}O_{4} + CO_{2}
\]  

(1)

Below mentioned table 2 and table 3 contains the weight of ingredient chemicals required for the preparation and synthesis of phosphors when the concentration of rare earth Eu²⁺ and Dy³⁺ in varying in mol% from 0.1, 0.2, 0.5, 1.0, 1.5, 2.0 and 2.5.
Table 2. Calculation of weight for ingredients in 5 grams of mixture keeping 1 mol% of Eu₂O₃ and variation of Dy₂O₃ from 1 mol% to 2.5 mol%.

| Dy₂O₃   | SrCO₃  | Al₂O₃  | Eu₂O₃ | Dy₂O₃ |
|---------|--------|--------|--------|--------|
| Mol %   | Weight (in grams) | Weight (in grams) | Weight (in grams) | Weight (in grams) |
| 0.1     | 2.9120 | 2.0112 | 0.0694 | 0.0074 |
| 0.2     | 2.9078 | 2.0082 | 0.0693 | 0.0147 |
| 0.5     | 2.8950 | 1.9994 | 0.0690 | 0.0366 |
| 1.0     | 2.8740 | 1.9849 | 0.0685 | 0.0726 |
| 1.5     | 2.8533 | 1.9706 | 0.0680 | 0.1081 |
| 2.0     | 2.8328 | 1.9565 | 0.0675 | 0.1431 |
| 2.5     | 2.8127 | 1.9426 | 0.0671 | 0.1777 |

Table 3. Calculation of weight for ingredients in 5 grams of mixture keeping 1 mol% of Dy₂O₃ and variation of Eu₂O₃ from 1 mol% to 2.5 mol%.

| Eu₂O₃   | SrCO₃  | Al₂O₃  | Dy₂O₃ | Eu₂O₃ |
|---------|--------|--------|--------|--------|
| Mol %   | Weight (in grams) | Weight (in grams) | Weight (in grams) | Weight (in grams) |
| 0.1     | 2.9099 | 2.0097 | 0.0735 | 0.0069 |
| 0.2     | 2.9058 | 2.0069 | 0.0734 | 0.0139 |
| 0.5     | 2.8938 | 1.9986 | 0.0731 | 0.0345 |
| 1.0     | 2.8740 | 1.9849 | 0.0726 | 0.0685 |
| 1.5     | 2.8533 | 1.9706 | 0.0721 | 0.1021 |
| 2.0     | 2.8328 | 1.9565 | 0.0716 | 0.1352 |
| 2.5     | 2.8127 | 1.9426 | 0.0712 | 0.1678 |

Figure 5 and 6 shows the alumina crucible contains the 5 gram of phosphors in varying concentration of Eu²⁺ and Dy³⁺ rare earth and the sequence and pattern followed for keeping of samples in the muffle furnace as shown in figure 7, for the calcination and sintering process. After the calcination and sintering the samples were kept in LDPE bottles to keep them protected from any impurity and moisture. The coding was adopted and mentioned on bottles for each of the sample with a specific concentration of Eu²⁺ and Dy³⁺ rare earth.
2.3. Theoretical approach related to mechanism of the mechanoluminescence in the prepared phosphor SAO doped with RE (Eu$^{2+}$, Dy$^{3+}$)

Some of the researchers have reported that for providing a good explanation of mechanoluminescence property of prepared samples one of the finest ways is electron detrapping model with a piezo-electric induction. As Jha and Chandra [10] have presented that, as in presence of Mn, the ZnS:Mn crystalline phosphor will emits the Elastico-mechanoluminescence and in presence of Ti, the ZrO$_2$:Ti crystals are possessing the Elastico-mechanoluminescence, the prepared desired samples of SAO doped with RE (Eu$^{2+}$, Dy$^{3+}$) will show the Elastico-mechanoluminescence phenomenon. With the above study it is clear that the property of piezoelectric constant is in more amount in the Elastico-mechanoluminescence phosphors near to the region of distortion at the activator ions. Following are some of the basic stages involved in the Elastico-mechanoluminescence emission from the persistent luminescent crystals:

- The characteristic of light emission from the excited ions may gets increased due to the de-excitation of the excited activator ions.
- The capturing of electrons, in excited state while moving in the conduction band, by the activator ions, which are placed at the bottom of the conduction band may occur where there is possibility of emission or release of excited Eu$^{2+}$ ions [19-22].
- Their may be chances of occurrence of thermal detrapping of the carriers due to the decrease of trap depth of carriers.

2.4. Experimental work

The solid-state diffusion technique, involved in the preparation of phosphors and samples, is one of the commonly practiced technique for the development of multicomponent solid ingredient chemical materials carried out at the higher temperatures. Due to the barrier faced by solids, that they can not interact with each other at the common room temperature, even when the thermodynamics favours, a high temperature involvement with a controlled environment is required to get the desired reaction between the reagent chemicals. The better feature associated with the solid-state reaction/diffusion technique is that it is a low-cost technique at the commercial level and there will be availability of ample of precursors [23]. The below figure 7 shows the muffle furnace utilized in this work for the calcination and the synthesis of desired phosphors doped with rare earth materials.
Before going to record the ML pattern of prepared phosphor samples, they were irradiated by Co$_{60}^+$
gamma rays. The magnitude of load applied or dropped is 500gm in each attempt and the quantity of
sample used is 2mg for the recording of ML pattern. This process is carried out at RTM university Nagpur.

![Muffle Furnace](image1.png)

**Figure 7.** Photograph of Muffle Furnace utilized for synthesis of phosphors

2.5. **Procedural analysis for Mechanoluminescence and Discussions**

The below Figures 8, 9 and 10 shows the experimental setup of mechanoluminescence device utilized
for the measurement of ML in the synthesized crystalline phosphors.

![Experimental setup](image2.png)

**Figure 8.** Experimental setup ML Device with stake height of 1 meter used for the measurement of ML Property in synthesized phosphors on time dependent basis.

![Wooden block](image3.png)

**Figure 9.** The wooden block, arrangement with transparent lucite plate and connections.

![Photomultiplier tube](image4.png)

**Figure 10.** The photomultiplier tube arrangement with connections to oscilloscope of ML device.
It has been found that the developed phosphor is possessing only one curve of glow with only one peak for the mechanoluminescence. Below Figure 11 depicts the time vulnerability of mechanoluminescence strength of γ- ray irradiated (Eu$^{2+}$, Dy$^{3+}$) doped SAO sample phosphor for the varying concentrations of different impurity Dy$^{3+}$.

![Figure 11](image1.png)

**Figure 11.** Time dependence of ML intensity of SAO:Eu, Dy (0.1 to 2 mol%) samples for the different concentrations of impurity.

![Figure 12](image2.png)

**Figure 12.** Dopant concentrations versus intensity plot for SAO phosphor doped with Dy$^{3+}$.

It has been observed that there will be an increment in mechanoluminescence intensity with the increment in the concentration of different impurity of dopants in the prepared desired phosphors. There will be an indication of single emission centre by the occurrence of only one single peak and hence the first observed peak can be compared with the time required for detrapping of the trapped electrons from the traps. Optimized concentration for intense ML is 1mol% for Eu and Dy doped SAO phosphor. Figure 12 shows the dopant concentrations versus intensity plot for SAO phosphor doped with Dy$^{3+}$, in which it has been observed that with the increase in concentration the intensity increases for up to 1mol% and after that the intensity decreases, for concentration of 1 mol % of Dy$^{3+}$, the maximum intensity is achieved.

Figure 13 depicts the glow curves for the mechanoluminescence of γ- ray irradiated (Eu$^{2+}$, Dy$^{3+}$) doped SAO samples, involving the varying impact velocities of the moving piston. The effect of increment in impact velocity governs the increment in the ML intensity. Also, it has been observed that there is a shifting of amount of time required related to the ML peak is towards the lower side while there will be an increase in the impact velocity.

When the experimental work of, dropping of a small mass from a particular height has been carried out, it has been observed that for the samples based on aluminium ingredients, the intensity of mechanoluminescence is increased for initial period, it increases and goes up to the peak value and then time dependent decrement is there. While comparing the appearance of the mechanoluminescence and its decay, it has found the quantities are lower in above case as compared to the amount developed in case of pressure application. The piezoelectric induces electron detrapping model is capable of explaining the mechanoluminescence behaviour in aluminium based phosphors. In these samples the trap depth has been reduced by piezoelectric field near the impurities of Eu$^{2+}$ & Dy$^{3+}$. Hence there will be occurrence of detrapping of the electron traps with the release of energy in non-radiative mode and obviously the mechanoluminescence has been observed.
Figure 13. Mechanoluminescence curve of glow for γ-ray irradiated SAO:Eu/Dy phosphors for various velocities of impact of the piston (for the 1 mol% concentration of Dy³⁺).

Figure 14. Variation of peak obtained of the ML intensity $I_m$ of SAO phosphors doped with RE (Eu²⁺, Dy³⁺) on different impact velocity.

Figure 15. Variation of peak time ($t_m$) with respect to the impact velocity ($V_0$) for the SAO doped with (Eu²⁺, Dy³⁺) phosphor.

The curve plot between the impact velocity and ML intensity is shown in Figure 14, which shows that the dependence of peak ML intensity $I_m$ of SAO phosphors doped with RE (Eu²⁺, Dy³⁺) on different impact velocity. For the lower impact velocity, the ML intensity is lower and on increasing the impact velocity the ML intensity is increased. Figure 15 represents the dependence of peak time ($t_m$) on impact velocity ($V_0$) for the SAO doped with (Eu²⁺, Dy³⁺) phosphor which concludes that on increasing the impact velocity the peak time is decreasing.
Chandra et. al. [24] have proposed the equation for time $t_m$ as below

$$
t_m = H \left[ \delta - \delta^2 V_0 \right] \ln 2
$$

(2)

As $\delta$ is a minute quantity for microcrystalline phosphors, it is clear that there will be very minor change in $t_m$ for increment in impact velocity $V_0$, as observed in Figure 16. Sahu et. al. [25, 26, 27] have reported about the variation of time with respect to the increasing impact velocity, for the powder phosphors.

2.5.2 Peak ML intensity $I_m$

Chandra et. al. [24] have also presented that the intensity of mechanoluminescence is having proportionality with the quantity of multiplication of rate of change of pressure and the pressure. The relation between impact pressure and impact velocity is studied and presented in various works [28-32].

2.5.3 Absorption Spectra and behaviour on load application

Choubey et. al. [33] reported about the Luminescence behaviour of SrAl$_2$O$_4$ on load application from height that for a time of 10 ms the maximum intensity of ML has been observed and they have also presented the absorption spectra of SrAl$_2$O$_4$ having absorption edge at 240nm.

2.5.4 XRD Analysis of Prepared phosphor

In order to determine the phase structure, powder XRD analysis has been carried out. The typical XRD patterns of SrAl$_2$O$_4$:Eu$^{2+}$, Dy$^{3+}$ nanophosphor with the standard XRD pattern is shown in Fig. The diffraction pattern depicted much broader and less intense peaks, thereby, giving a strong indication that the prepared nanophosphor has smaller grain size and lower crystallinity.
3. Results
The work done in the present study shows that, the mechanoluminescence behaviour is owned by Eu$^{2+}$ and Dy$^{3+}$ activated SrAl$2$O$4$ phosphor. The prepared phosphor is having a single mechanoluminescence peak with a single glow curve for every sample with variation of mol% in weight. The mol% concentration is varying as 0.1, 0.2, 0.5, 1.0, 1.5, 2.0 and 2.5.

The time dependent ML intensity study of irradiated rare earth (Eu$^{2+}$, Dy$^{3+}$) doped SAO phosphor shows that, the ML intensity id maximum for concentration of 1 mol%. The analysis of dopant concentrations versus intensity plot for SAO phosphor doped with Dy$^{3+}$, indicates that the increase in concentration the intensity increases for up to 1mol% and after that the intensity decreases, for concentration of 1 mol % of Dy$^{3+}$, the maximum intensity is achieved.

The ML glow curves of Y- ray irradiated (Eu$^{2+}$, Dy$^{3+}$) doped SAO samples for different impact velocities of the piston indicates the ML intensity increases with increasing impact velocity. For 30cm/s velocity the maximum intensity of ML in arbitrary unit is achieved. Also, as the impact velocity increases the peak time goes on decreasing.

4. Conclusion
The developed phosphors can be employed to a wide range of applications, in a non-invasive mode. The synthesized phosphors are capable of providing signals in quite well characterized pattern. They are also able to depict the ability of discrimination of impact energy produced beyond the binary sensing. The recent and trending application of these phosphors may include, the work out for finding of dependency of mechanoluminescence development on the impact energy. By utilizing the mechanoluminescence phenomenon, study about the trapping and detrapping of charge carriers can be carried out. The mechanoluminescence excitation can be achieved by dropping a mass from a prescribed height on to the phosphors, the produced ML intensity gradually increases with the time then reaches to its maximum value and the goes on decreasing.

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