Synthesis, Measurement of TL Intensity And TL Glow Curve Analysis of $LiMgALF_6$:Eu And $LiNa_2ALF_6$:Eu Phosphors

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Abstract- *The present paper reports the synthesis of $LiMgAlF_6$:Eu and $LiNa_2AlF_6$:Eu phosphor by wet chemical method and study their TL intensity and TL glow curve analysis. For the optimization of intensity of the samples, different combinations of variations in the procedure were made. The slight variation in respect of starting materials and synthesis conditions adopted for optimizing the process include changing the precursors, changing the reaction temperatures, methods of insertion of dopant, drying techniques, medium of reaction and quenching temperature. For the improvements in the luminescence intensity, the wet chemical method is modified number of times for systematic synthesis of trial samples by changing the starting materials such as LiOH, LiCl, H_3PO_4 and $(NH_4)H_2PO_4$ and synthesis conditions like quenching temperatures (650˚C to 1100˚C), methods of making solutions and method of precipitation. This study demonstrated the Synthesis of $LiMgAlF_6$:Eu and $LiNa_2AlF_6$:Eu Phosphors by wet chemical method, measurement of their TL intensity and study of their TL glow curve analysis.*

Co-precipitation technique is the simplest wet chemical method of synthesis of Phosphors. It is becoming increasingly important in order to distribute the activator in host in an atomic scale. This process is used to prepare multi-component ceramic oxides through formation of intermediate precipitates, usually hydrous oxides or oxalates, so that an intimate mixture of components is formed during precipitation and chemical homogeneity is maintained on calcinations and the constituents are dissolved in suitable solvent

Keywords- $LiMgAlF_6$; $LiNa_2AlF_6$, wet chemical method; TL intensity; TL glow curve

I. INTRODUCTION

Fluorides are susceptible to hydrolysis during preparing fluoride-based phosphors. Use of freshly synthesized powders from oxides with dry HF gas as fluorinating agent in an inert atmosphere is most effective in preparing OH^- free phosphors. However, the method then becomes tedious and expensive. Modifications of wet chemical processes could be successful to prevent hydrolysis. Fresh fluoride powders may be prepared by neutralization of HF with metal carbonates. Precipitation in the presence of chlorine ions is also known to prevent hydrolysis. Hence various procedures were tried for preparation of lithium fluoride powders. The low Z phosphors synthesized by using this wet chemical method are LiMgAlF6 and LiNa2AlF6 doped each with Eu. For the optimization of intensity of the samples, different combinations of variations in the procedure were made (1-5).

II. SYNTHESIS OF $LiMgAlF_6$:Eu AND $LiNa_2AlF_6$:Eu PHOSPHORS

$LiMgAlF_6$ and $LiNa_2AlF_6$, Eu doped phosphors were synthesized by using this wet chemical method. For the optimization of intensity of the samples, different combinations of variations in the procedure were made. For the synthesis of $LiMgAlF_6$ and $LiNa_2AlF_6$, Eu doped phosphors we mixed metal carbonated and metal carbonates $Al(NO_3)_3{\cdot}9H_2O$ to get slurry, HF is mixed with it to get precipitation, it is then dried by blowing air we obtained host material. Now dopant was mixed obtained materials and dried. The obtained product was heated in reactive atmosphere for 1 hour at 450^0C. The obtained product melted and quenched to room temperature, final product is obtained (6-10)

Chemical reactions for synthesis of $LiMgAlF_6$:Eu:

$0.5Li_2CO_3 + MgCO_3 + Al(NO_3)_3{\cdot}9H_2O + 6HF + 0.001EuCl_3 \rightarrow LiMgAlF_6{:}Eu + 1.5CO_2 \uparrow + 3NH_3 \uparrow + 7.5H_2O + 6O_2 \uparrow$

$LiCl + MgCl_2{\cdot}6H_2O + AlCl_3{\cdot}6H_2O + 0.001EuCl_3 + 6HF \rightarrow LiMgAlF_6{:}Eu + 6HCl + 12H_2O$

Chemical reactions for synthesis of $LiMgAlF_6$:Eu:

$0.5Li_2CO_3 + Na_2CO_3 + Al(NO_3)_3{\cdot}9H_2O + 6HF + 0.001EuCl_3 \rightarrow LiNa_2AlF_6{:}Eu + 1.5CO_2 \uparrow + 3NH_3 \uparrow + 7.5H_2O + 6O_2 \uparrow$

$LiCl + 2NaCl + AlCl_3 \cdot 6H_2O + 0.001EuCl_3 + 6HF \rightarrow LiNa_2AlF_6:Eu + 6HCl + 6H_2O$

III. IRRADIATION OF PHOSPHORS

γ Exposures: A Theratron 780E therapy machine employing a ^{60}Co source at Regional Cancer Hospital, Nagpur, was used for γ ray exposures to various samples. The exposure rate was calculated for the field size of irradiation of 10cm x 10cm at 80 cm distance. It was 909.2 mGy/minute on 09/06/2006. The rate was calculated each time, when samples irradiated. Accordingly the time required for various dose was calculated. The exposure to phosphors was given from 0.1 Joule/Kg to 10 Joules/Kg.

β Exposure: Exposure of β was obtained from $^{90}Sr \rightarrow ^{90}Y$ source at Radiation Safety System Division, BARC, Mumbai. The exposure rate was 20 mGy/minute. The time required for various dose was calculated and accordingly the exposure was given to the phosphors from 0.01 Joule/Kg to 1 Joule/Kg.

X-ray Exposure: Different energy photons of effective energies 17.6 KeV, 22.7 KeV, 24.85 KeV, 65.90 KeV, 83.10 KeV,117.6 KeV, 125 KeV and 223.9 KeV of X-rays were obtained from YXLON International X-ray machine at BARC, Mumbai. The source was first calibrated by using ionization chamber for each time by putting it at a distance of 2 metres in air. The samples were put at the same distance. The time required for 0.01 Joule/Kg of dose was calculated for various energy photons and accordingly the exposure was given to the phosphors.

IV. RESULTS AND DISCUSSION

TL intensities of $LiMgAlF_6$ and $LiNa_2AlF_6$ phosphors doped with Eu reported in table-1 which represents the most suitable method for synthesis of phosphors. In this work we have synthesized the phosphor by various methods most predicted method represented by using balance chemical reactions and reported in the following table. The values of TL intensities predict the TL glow curves for selected procedures with respect to each host and the activator (11-15).

S.N.	Phosphor		Case 1	Case 2	Case 3	Case 4	Highest Intensity	% with respect to CaSO4:Dy
	Host	Dopant						
1	$LiMgAlF_6$:	Eu	40	8	117	**157**	157	10.51%
2		Dy	21	6	27	**95**	95	6.36%
3		Mn	**182**	102	92	76	**182**	12.18%
4	$LiNa_2AlF_6$:	Eu	188	**250**	43	34	**250**	16.73%
5		Dy	2	1	**16**	5	16	1.07%
6		Mn	1	4	4	**9**	9	0.60%

TL glow curves of $LiMgAlF_6$ for Eu activator

Figure 1 shows the glow curves of $LiMgAlF_6$ samples with various activators of concentration 1000 ppm. The glow curves for all the three activators are symmetric. It is observed that Mn shows good TL than Eu and Dy. The TL peak for Mn is at about 170°C while for Eu and Dy it is around 150°C. As compared to Standard $CaSO_4$:Dy (RchenTech TT88) the sensitivity of $LiMgAlF_6$:Eu is around 11% which is not promising. Mg may not be allowing Eu to be at its site and not creating Li^+ vacancy therefore the TL sensitivity is not promising in this case (16-18).

TL glow curves of $LiNa_2AlF_6$ for Eu, Dy and Mn activators:

Figure 2 shows the glow curves of $LiNa_2AlF_6$ samples with various activators of concentration 1000 ppm. It is observed that only Eu shows considerable performance. The TL peak intensity for Mn and Dy are very poor. The TL peak for Eu is at about 170°C. As compare to Standard $CaSO_4$:Dy (RchenTech TT88), the sensitivity of $LiNa_2AlF_6$:Eu is found out as 17%.

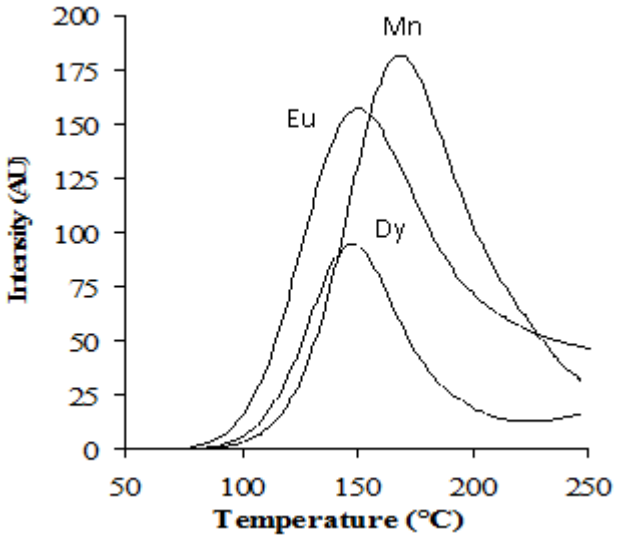

Fig.1 Typical glow curves for $LiMgAlF_6$ doped with 1000 ppm of various impurities (Mn, Eu and Dy), exposed to 1 Joule/kg

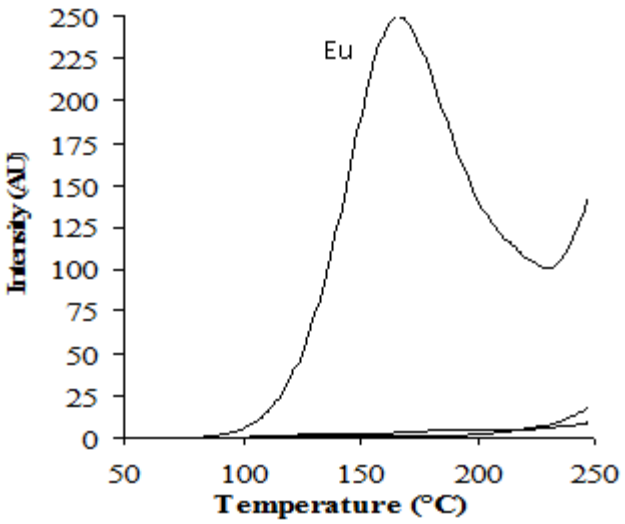

Fig. 2 Typical glow curves for $LiNa_2AlF_6$ doped with 1000 ppm of various impurities (Eu, Mn and Dy), exposed to 1 Joule/kg

Comparison of TL glow curves of $LiMAlF_6$:Eu

Table 2: Comparison of TL parameters of Lithium Fluorides, prepared by wet Chemical methods with Standard $CaSO_4$:Dy (RchenTech TT88)

Sr. No	Selected TL material	Z_{eff}	Peak Temperature	TL intensity related to $CaSO_4$:Dy	TL mg^{-1} Gy^{-1}
1	$LiMgAlF_6$:Eu	10.3	170°C	11%	33
2	$LiNa_2AlF_6$:Eu	10.2	167°C	17%	51

V. CONCLUSIONS

1. Eu is the most suitable activator than others studied
2. 1000ppm is the optimum concentration for Eu as a dopant in $LiMgAlF_6$ and $LiNa_2AlF_6$
3. Quenching temperatures for the best product should be more than 700°C
4. Typical glow curves for $LiNa_2AlF_6$ doped with 1000 ppm for Eu shows most ideal performance

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