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Novel Molten Salts Synthesis and Photoluminescence Properties of Eu (III) Doped Y_2O_3 Phosphor

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ABSTRACT

A novel molten salt method used for the synthesis of Eu^{3+} doped yttriumbased phosphor. It is well known that $Y_2O_3:Eu^{3+}$ is highly efficient red phosphors used for Lamp phosphor. The $Y_2O_3:Eu^{3+}$ phosphor was synthesized by reactions in molten salts method.The red emitting phosphor characterized through powder X-ray diffraction (XRD), and PL spectra. A novel molten salt is one step methodand decrease calcining temperature.

Keywords : $Y_2O_3:Eu^{3+}$,Molten salts method, PL spectra.

I. INTRODUCTION

The production of reliable and reproducible ceramic materials for high technology applications require strict control over their powder characteristics, which includes chemical homogeneity, low impurity levels, small particle size, narrow size distribution and freedom from agglomeration. A variety of methods e.g. sol-gel, chemical precipitation of precursors in aqueous or organic solutions, thermal decomposition of solutions by spraying technique, high alkaline and hydrothermal precipitation have been proposed for obtaining small, uniform un-agglomerated powders. These methods so-called wet chemical method, have been found to be successful for number of systems. Also self-sustaining combustion synthesis is a simple, inexpensive and quick way of synthesizing various oxide materials in comparison to the wet chemical techniques [1].

Compounds containing rare earths have long been used as phosphors and laser materials because of their sharp, intensely luminescent f-f electronic transitions.In particular, Eu^{3+} has five narrow emission bands corresponding to the $^5D_0 \rightarrow {}^7F_j$ transitions where, j = 0,1,2,3,4. The strongest transition, $^5D_0 \rightarrow {}^7F_2$ occurs at 613 nm, which is a characteristic of red fluorescence of Eu^{3+}. This transition has also been shown to exhibit laser emission under appropriate conditions in Eu^{3+} doped crystals [2, 3]. It is well known that the $Y_2O_3:Eu^{3+}$ is highly efficient red phosphor and has its own importance in scintillation, lamp and color TV picture tubes [4].

The preparation of these red emitting phosphors is critical and requires special methods such as wet chemical methods. Recently preparation of Eu^{3+} doped yttria was carried out by the alkoxide route and combustion process [5, 6]. Though these processes are efficient, requires expensive chemicals and special

equipment. Here we report the preparation of Y_2O_3: Eu^{3+} by the novel method of reactions in molten salts.

The term molten salt refersto the liquid state of compound, which melts to give liquids displaying a degree of ionic properties [7, 8]. Alkali metal nitrates have relatively low melting points (Table:1) whereas even lower melting points are obtained in their eutectic mixtures. A molten salt can behave as a solvent or as a reactant. Thus in a nitrate melt acid-base reactions can occur according to the Lux -flood formalism, whereby an acid is an oxide ion acceptor and a base is an oxide ion donor; nitrate ions are bases in this formalism [9]. Nitrite melts are more basic than nitrate melts whereas addition of Lux-flood bases such as Na_2O_2, Na_2O and NaOH to a nitrate melt, which its basicity.

The precursors are the inorganic compounds, in particular sulphates and chlorides that are blended with the alkali metal nitrates or nitrites as a powder mixture before heating to the reaction temperature. Table:1 shows the various eutectic mixtures and corresponding melting points.

Table-1 Melting points for alkali metal nitrates and eutectic mixtures.

Metal Nitrate	Melting point (^{0}C)
$NaNO_3$	307
KNO_3	334
50 mol% $NaNO_3$-50 mol% KNO_3	220
43 mol% $LiNO_3$- 57 mol% KNO_3	132

II. METHODS AND MATERIAL

2.1 Synthesis of $Y_2O_3:Eu^{3+}$

The precursors used were Y_2O_3 (AR) and Eu_2O_3 (AR). Both were mixed together in a china basin. A small quantity of double distilled (DD) water was added and paste was formed. Then HCl was added drop by drop and mixture was heated slowly under observation at 50^{0}C till the paste dissolved completely. The solution was further heated till the excess of acid was boiled off. Little quantity of double distilled (DD)water was again added and slowly evaporated to dryness. The resulting powder was YCl_3: Eu. The chemical reaction is $0.97\ Y_2O_3 + 0.03\ Eu_2O_3 + HCl \rightarrow 2\ Y_{0.97}Cl_3 : Eu_{0.03} + 3H_2O$ ---------- (1)

The eutectic mixture of nitrates $LiNO_3.3H_2O$ (43 mole %) and KNO_3 (57 mole %) were taken in a china basin and mixture was dried at 50^{0}C.The dry chloride YCl_3:Eu was added to this mixture of dried nitrates and thoroughly ground in a china basin. This mixture was then heated in a resistive furnace first at 100^{0}C for 1h and then with the gradual rise of temperature it was further heated at 425^{0}C for12 hours. The chemical reaction is $2Y_{0.97}Cl_3 :Eu_{0.03} + 0.43\ LiNO_3.3H_2O + 0.57\ KNO_3 \rightarrow Y_{1.94}O_3 : Eu_{0.06}$ --------- (2)

The mixture was then cooled slowly. A semi convex white solid was formed. With the help of sufficient lukewarm DD water, the solid was transferred to a glass beaker. Keeping the beaker in an oven at 60^{0}C for 10 minutes, the white solid was partly dissolved in water and the fine particles of Y_2O_3:Eu started settling down at the bottom. The precipitate was washed repeatedly by DD water and then dried. The dried precipitate was then calcinated at 800°C for 2h and quenched suddenly at room temperature.The calcinated and quenched powder was $Y_2O_3:Eu^{3+}$ phosphor.

2.2 Material characterization

The phase purities of $Y_2O_3:Eu^{3+}$ phosphor was studied using Rigaku miniflex II X-ray Diffractometer with scan speed of 2.000°/min and Cu Kα (λ = 1.5406 Å) radiation in the range 10° to 90°. PL and PL excitation (PLE) spectra were measured on (Hitachi F-7000) fluorescence spectrophotometer at room temperature. The parameters such as spectral resolution, width of the monochromatic slits (1.0 nm), photomultiplier

tube (PMT) detector voltage and scan speed were kept constant throughout the analysis of samples

III. RESULTS AND DISCUSSION

3.1 Powder XRD pattern of $Y_2O_3:Eu^{3+}$

The formation of the phase purity and crystal structure of Y_2O_3 synthesized by using molten salts at 800°C was confirmed by XRD pattern as shown in Figure. 1. All the peaks in XRD pattern very well agree with the standard data from ICDD file no. 01-071-0049. Also the XRD shows that the formed material was completely crystalline and was in single phase with cubic structure where a=b=c=10.5957 Å. The space group for Y_2O_3 is Ia-3 (206).The average crystallite size of $Y_2O_3:Eu^{3+}$determined from XRD pattern using Scherrer formula and it was found to be 965.52 nm [10]

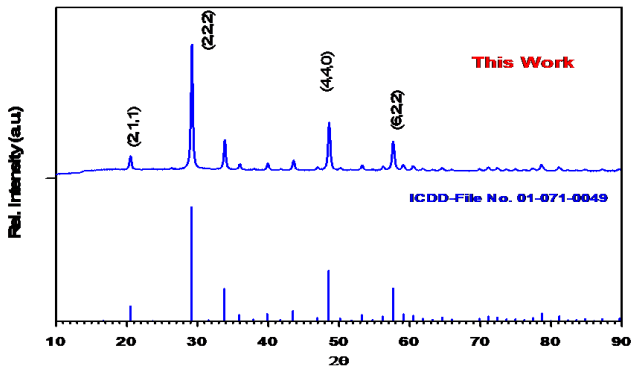

Figure:2 XRD pattern of $Y_2O_3:Eu^{3+}$.

Figure 2 shows PL spectra of $Y_2O_3:Eu^{3+}$ phosphor. It consists of a broadband excitation spectrum peaking at 246 nm, monitored at emission wavelength 613 nm. The emission spectrum is sharp peaking at 613 nm at excitation wavelength 254 nm, corresponding to the transition $^5D_0 \rightarrow {}^7F_2$ of Eu^{3+}. The intensity of emission is found to be comparable and excitation, emission wavelengths matches well with those of reported in literature.

The rare earth compounds such as $Y_2O_3:Eu^{3+}$ mainly belong to luminescent materials with individual luminescent center. Luminescence of these materials is due to the transition between 4f energy levels. Because of spin-orbit interaction, the degenerate 4f configuration is split into several energy levels such as ^5Dj and ^7Fj. The crystal field of host lattice affects the electronic transitions in Eu^{3+} [11]. For $^5D_0$ term, j = 0, so it cannot split (only one energy level). For the term $^7F_2$, j = 2 and $2_{j+1}=5$, so it can split into five energy levels (Γ_1, Γ_2,Γ_3, Γ_4, Γ_5). The strongest peak at 613 nm in $Y_2O_3:Eu^{3+}$ phosphor corresponds to the transition $^5D_0 \rightarrow {}^7F_2$ of Eu^{3+}. This method of reactions in molten salts is a low temperature and single step synthesis. This method is easy to synthesis and precursors used are readily available. The sample synthesized is a snow-white powder and particle size varies from sub-micron to nano. Frequent washing and calcinations enhances the PL intensity.

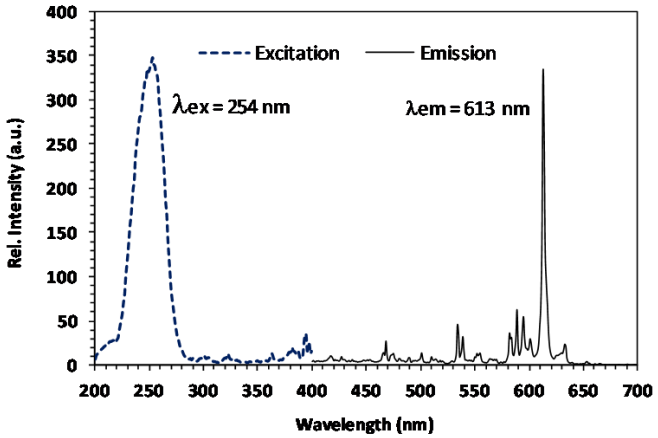

Figure:1 Photoluminescence emission and excitation Spectrum of $Y_2O_3:Eu^{3+}$

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IV. CONCLUSION

The excitation and emission wavelengths in PL spectra of $Y_2O_3:Eu^{3+}$ synthesized by molten salts method confirms the formation of desired phase and crystal structure in the compounds. As phosphors

with particles of required size and colour are needed for their use in the display devices and other applications. This method has new horizons in the lighting industries.

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