Intense red-emitting $Y_4Al_2O_9$:Eu^{3+} phosphor with short decay time and high color purity for advanced plasma display panel

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Abstract: A new phosphor $Y_4Al_2O_9$:Eu^{3+} (YAM:Eu^{3+}) emitting intense monochromatic red at 612 nm under vacuum ultraviolet (VUV) and ultraviolet (UV) excitations has been developed for application in next generation plasma display panels (PDPs). The developed phosphor has better luminescence efficiency, colour purity and shorter decay time than commercial (Y,Gd)BO$_3$:Eu^{3+} red emitting PDP phosphor. High color purity (x = 0.67, y = 0.32) under VUV excitation with short decay time (1.03 msec) and excellent stability against degradation during PDP panel preparation suggest that YAM:Eu^{3+} is a potential candidate for present and future PDPs. Surface coating by SiO$_2$ further improved phosphor characteristics.

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1. Introduction

Plasma display panels (PDPs) dominate the segment of the next generation flat panel displays which uses VUV excitation of Red, Green and Blue (RGB) phosphors for image display. For full coloured large area flat display, PDP is a very promising technique due to the progress made in technology by way of improvements in cost, resolution, lifetime, power consumption, high performance and luminescence efficiency which results in the reductions of thickness and weight [1,2] Motivated by the advances in PDP technique, the demand of highly efficient vacuum ultraviolet (VUV) phosphors has increased tremendously in the past decade [3]. The demand became crucial as phosphors play a very important role in PDPs in terms of augmented performance such as higher efficiency for lower power consumption and higher reliability for longer lifetime. Luminescence characteristics of phosphors and their behaviour under panel making process, energetic discharge ions, electrons and solarization from VUV generated by the Xe-Ne gas plasma are important factors for PDP. In PDP's plasma resonance, vacuum ultraviolet (VUV) radiation lines of Xe atoms at 147 nm and a molecular excimer Xe^2 at 172 nm are used for excitation of phosphors to emit visible luminescence in red, green and blue. All the three red, blue and green phosphors have shown degradation in luminescence intensity due to the thermal treatment of PDP manufacturing process and this remains a major problem for PDP phosphors [4–7]. Another challenge in PDP technology today is to improve the purity of red phosphor so as to obtain monochromatic red emission with good color coordinates [8,9]. Commercially used $(Y,Gd)BO_3:Eu^{3+}$ ($YGB:Eu^{3+}$) red phosphor has poor color purity with a dominant orange component and the CIE chromaticity x value (0.65) [10] falls short of declared standard value of 0.67 by National Television

Standard Committee (NTSC). For viewing high speed motor sports, video gaming and other high definition programs, decay time of the phosphor should be as low as possible to avoid image overlap. Decay time of YGB:Eu^{3+} phosphor is long (12 msec) which makes this phosphor unsuitable for next generation PDP sets. It is, therefore, necessary to develop highly efficient, baking resistant, high color purity red-emitting phosphor with shorter decay time. As an alternative, the search for new efficient red-emitting phosphor other than YGB:Eu^{3+} is being conducted and reviewed by many researchers [3,11] Europium-doped Yttrium Aluminium Garnet (YAG) has attracted substantial attention in recent years because of its high resistance to electron irradiation which makes Yttria – Alumina system promising candidates in Cathode Ray Tubes, Field Emission Display, Vacuum Fluorescent Displays [12] etc. The Yttria – Alumina system has several phases, including $YAlO_3$ (YAP: Yttrium Aluminium Polymorphs), $Y_4Al_2O_9$ (YAM: Yttrium Aluminium Monoclinic), and $Y_3Al_5O_{12}$ (YAG: Yttrium Aluminium Garnet). Commonly, YAP and YAM form as an intermediate product of YAG phase in solid state reactions. Even if YAG is synthesized with a stochiometric mixture of Y_2O_3 and Al_2O_3, two detrimental phases, YAP and YAM, often co-exist as by-products [12,13]. We have deliberately synthesised single phase YAM:Eu^{3+} by conventional solid state reaction technique and investigated the luminescence properties in detail to see the effect of changed crystal field in Eu^{3+} emission characteristics. The luminescence properties of YAM:Eu^{3+} under UV excitation was reported earlier by many researchers for different applications [14–16]. However, very few reports [17,18] are available on the VUV excitation properties of YAM:Eu^{3+} for PDP application so far. To the best of our knowledge, we report for the first time Eu^{3+} doped YAM that is highly efficient, degradation controlled, pure red-emitting phosphor with additional feature of short decay time suitable for advanced PDPs.

2. Experimental

To synthesize YAM:Eu^{3+} phosphor by solid state reaction technique [19], stoichiometric amounts of starting material Y_2O_3 (99.99%), α-Al_2O_3 (99.9%), and Eu_2O_3 (99.99%) were weighed according to the molar ratio of $(Y_{1-x}Eu_x)_4Al_2O_9$ (x = 0.01 ≤ x ≤0.50). The powders were mechanically ground with 10 mole% H_3BO_3 (99.9%) as flux in a mortar pestle, packed in an alumina crucible and subjected to two stage calcinations and sintering process between 700 and 1300°C in air atmosphere for 2-10 hours. Initial calcination was done at 700°C for 2 hours, at this temperature melting of boric acid (H_3BO_3) starts which enables proper mixing of the precursor materials. Addition of H_3BO_3 drastically reduces the synthesis temperature from 1500°C to ~1300°C and also promotes the crystallization by acting as a high temperature solvent. Therefore second stage sintering was done at 1300°C for 10 hrs to improve the crystallinity.The sintered mass was ground thoroughly to obtain fine powder phosphor. Mechanical grinding and rigorous ultrasonication in alcohol leads to de-agglomeration of particles. The YAM:Eu^{3+} powder grains were then subjected to surface treatment for silica coating from a precursor solution of tetraethylorthosilicate (TEOS) by Stöber process [20] followed by high temperature annealing for uniform coating of the grains. Surface coating of phosphor particles was basically done for the purpose of preventing the degradation of the phosphors due to panel baking process in air at temperature ~500°C. For simulation of thermal baking process in PDP panel, the YAM:Eu^{3+} powder was mixed with organic vehicle used for screen printing process and subsequently fired in air atmosphere at 500°C for 30 min to remove the organic volatiles from the phosphor.

3. Results and discussion

X-Ray diffraction (XRD) was employed to identify the phase by Rigaku Miniflex II X-ray powder diffractometer using Cu Kα radiation.
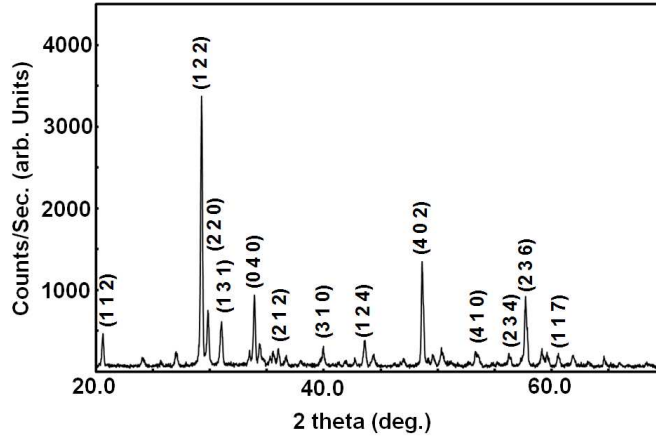
Fig. 1. XRD pattern of prepared YAM:Eu³⁺ PDP phosphor

The XRD (Fig. 1) revealed monoclinic monophasic YAM [21] without any precipitated phase implying effectiveness of the synthesis process in single phase crystallization. The dopant Eu^{3+} ions effectively substitutes Y^{3+} ions. Figure 2 shows the scanning electron microscope image composed of near rounded particles of ~1μm size of as synthesized YAM:Eu^{3+}.
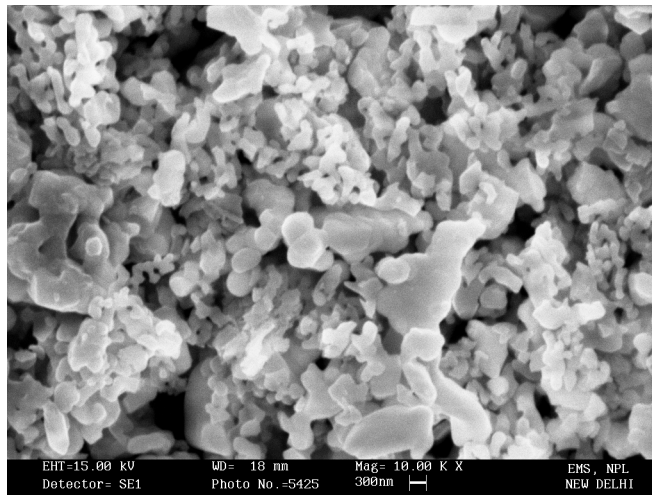

Fig. 2. SEM micrograph of as synthesized YAM:Eu³⁺ phosphor sample.

Low sintering temperature controls the growth of individual phosphor particles. Silica coating process provided a near uniform distribution of separated grains of approximate 1μm size. TEM image (Fig. 3) clearly shows the individual silica coated phosphor grains of size below 1μm with nearly spherical morphology. As theoretical considerations based on Mie scattering theory have shown that best phosphor efficiency can be achieved in 1-2 μm grain size [22], the particle size of developed phosphor seems ideally suitable.

The prepared YAM:Eu^{3+} phosphor possesses an intense monochromatic red-emission at 612 nm under UV excitation (225-275 nm) (Fig. 4) and under VUV excitation at 147 nm, 172 nm (Fig. 5). The photoluminescence (PL) spectra of Eu^{3+} in synthesized powder YAM and commercial YGB under UV excitation are shown in Fig. 4.
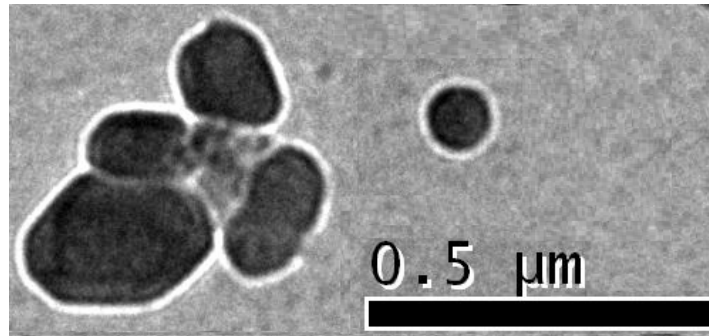
Fig. 3. TEM image of silica coated YAM:Eu^{3+} particles.

The PL emission from the YAM:Eu^{3+} phosphor exhibits a strong emission peak at 612 nm which can be compared to the three emission peaks of commercial YGB: Eu^{3+} phosphor under identical conditions. The strongest emission peak observed at 612 nm is due to the electric dipole transition of $^5D_0$-$^7F_2$. It is clearly seen from the figure that emission intensity of YGB:Eu^{3+} phosphor is much lower with an undesirable 590 nm ($^5D_0$-$^7F_1$) peak that impedes the colour purity. The integrated PL emission intensity (inset in Fig. 4) of Y$_{3.8}$Eu$_{0.2}$Al$_2$O$_9$ is 23% more than that of standard red emitting YGB:Eu^{3+} red phosphor which is represented as blue line. PL emission spectra of YAM:Eu^{3+} phosphor under 147 and 172 nm VUV excitation (Fig. 5) show a strong peak at 612 nm showing the monochromaticity of the developed phosphor in both the VUV excitation wavelengths employed in a conventional PDP panel. The spectrum under 147 nm excitation [Fig. 5(a)], however, was measured under vacuum in a different setup which may be the reason for slight change in the emission line width.
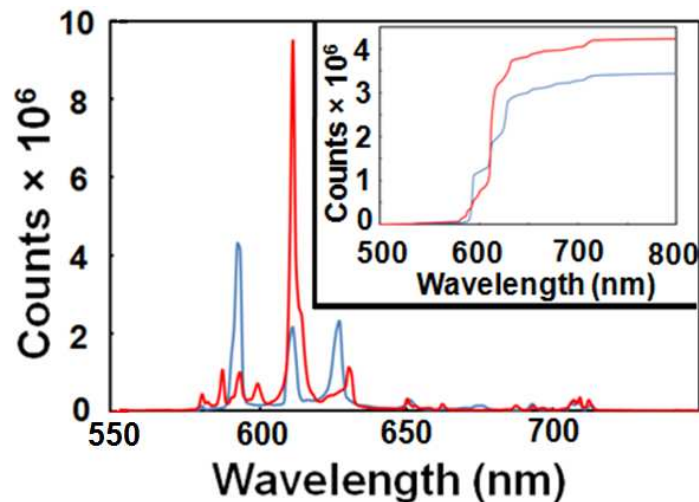

Fig. 4. (color online) Room-temperature emission of YAM: 0.2Eu^{3+} (Red line) phosphor and commercial YGB:Eu^{3+} (Blue line) phosphor (λ_{ex} = 250 nm). The inset shows integrated emission intensity of the same phosphors.

In order to optimize the Eu^{3+} doping concentration for maximum luminescence intensity, Eu^{3+} concentration was varied according to (Y$_{1-x}$Eu$_x$)$_4$Al$_2$O$_9$ with x = 0.01, 0.03, 0.07, 0.10, 0.20, 0.30 and 0.50. It was observed that the integrated emission intensities from Eu^{3+} initially increased with an increase of the concentration x, reaching a maximum value at x = 0.20. The PL intensity decreased with further increase of Eu^{3+} concentration in the phosphor sample due the concentration quenching effect. Thus, the optimum concentration for Eu^{3+} is 0.20 moles of Y^{3+} in Y$_4$Al$_2$O$_9$ host. In general, the concentration quenching of luminescence at higher

concentration is due to the energy migration among the activator ions as the excitation energy can be lost by ion-ion non-radiative energy transfer resulting in the decrease of the PL intensity.
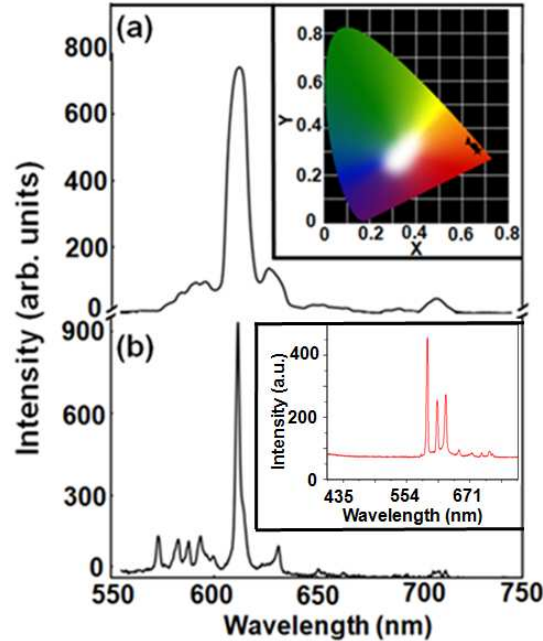

Fig. 5. (color online) Room-temperature PL spectra of prepared YAM:Eu^{3+} (a) at 147 nm excitation and (b) at 172 nm excitation. Inset in (a) shows the CIE chromaticity diagram where triangle and star denotes CIE coordinates (λ_{ex} = 147 nm) of commercially used YGB:Eu^{3+} and prepared YAM:Eu^{3+} phosphor respectively, circle denotes CIE coordinates (λ_{ex} = 172 nm) of YAM:Eu^{3+}. Inset in (b) shows YGB:Eu^{3+} emission at 172 nm excitation.

It is known that the PDP panel baking process induces a decrease in luminescence efficiency of phosphors which is dependent on the stability of host and activator. Therefore, it is required that the phosphors are stable during the heat treatment during PDP panel processing. Surface coating of phosphor grains using silica was done to arrest degradation during baking step. Increase in integrated PL intensity upto 30% due to silica coating was observed in our red phosphor. The SiO$_2$ coating passivates the surface charges that increase the transmission of exciting radiation into the phosphor by eliminating diffuse reflection from grain i.e. the effective UV/VUV absorption of the phosphor by SiO$_2$ layer increases [23]. Due to silica coating reflectivity in the visible region is also increased which aids in increase in luminous efficacy [24]. It has been evidenced that higher luminescence with SiO$_2$ coated phosphor has a strong relation with VUV, which is produced in the gas discharge and is absorbed more effectively into the phosphor via SiO$_2$ layer. In other words, less reflection at interfaces between phosphor and the gas due to the small refractive index difference via SiO$_2$ coating is responsible for retention of high PL intensity even after baking [25]. Moreover, silica coating passivates the surface and prevents inactivation of available luminescence centres. The phosphor baking process with organic vehicle had negligible effect on the PL intensity which remained same within experimental error implying arresting of degradation. Hence, the phosphor prepared is well suited for PDP application. One added advantage of the silica coated smaller size rounded phosphor grain would be a highly compact phosphor layer with high reflectivity which would enhance the extraction of emitted visible photons [26] and hence luminous efficacy.

The strong orange emission is dominant in the commercial red-emitting YGB:Eu^{3+} phosphor, which is undesired for colour reproduction and color purity. However, total luminescence intensity of YGB:Eu^{3+} becomes much inferior to the present YAM:Eu^{3+} phosphor if the orange peak is filtered for improving the color purity. It is known that the PDP phosphors show strong broad excitation band in VUV region with absorption band edge at 200 nm, monitored at 612 nm emission [3]. The broad band in the VUV region may be attributed to the absorption by the host lattice. The emission at 590 nm originates from $^5D_0$-$^7F_1$ magnetic dipole transition of the Eu^{3+} ions and this transition is insensitive to site symmetry. The red emission transition $^5D_0$-$^7F_2$ is electric dipole transition induced by lack of inversion symmetry at the Eu^{3+} site and can be much stronger than transition to the $^7F_1$ level. The space group of the crystal structure of $Y_4Al_2O_9$ is P2$_{1/c}$ has been reported to be monoclinic in which each Al atom is linked to four oxygen atoms and Y atoms are co-ordinated to either six or seven oxygen atoms with C_1 site symmetry is. When the Eu^{3+} ions are located at low local symmetry site, the hypersensitive transition $^5D_0$-$^7F_2$ is dominated in its emission spectrum. In view of the valence state and the ionic radius, the doped Eu^{3+} ions occupy the Y^{3+} sites in $Y_4Al_2O_9$, resulting in the $^5D_0$-$^7F_2$ emission of Eu^{3+} being the most prominent peak in the emission spectrum. It is known that the intensity (I_1) of a magnetic dipole transition ($^5D_0$-$^7F_1$) is relatively insensitive to the ligand field around the Eu^{3+} ion. The intensity (I_2) of electric dipole transition $^5D_0$-$^7F_2$ is strongly hypersensitive to the occupancy of Eu^{3+} ion in a site without inversion symmetry and the nature of its ligand surroundings [27,28]. A ratio between the intensity of these two transitions, I_2/I_1, is used in lanthanide based system as a probe of the cation local surroundings [29]. The ratio of I_2/I_1 in YAM:Eu^{3+} is 7.80 and 6.0 for 147 and 172 nm VUV excitation respectively, suggesting that the Eu^{3+} ions are located in a distorted (or asymmetric) cation environment. Eu^{3+} replaces Y^{3+} in YAM in which four Y^{3+} sites have C_1 point symmetry without inversion centre [18]. As a result, the transition $^5D_0$-$^7F_2$ is much stronger than the transition $^5D_0$-$^7F_1$ [Fig. 5(a) and 5(b)] due to mixing of opposite parity states that makes the $^5D_0$-$^7F_2$ transition partially allowed. This is favourable for improving the illuminating intensity and color purity of the red-emitting YAM:Eu^{3+} phosphor. Commercially used YGB:Eu^{3+} phosphor exhibits orange-red emission with ratio of I_2/I_1 = 0.60 calculated from spectra (blue line) of Fig. 4 showing a poor color purity and CIE coordinates than that of the YAM:Eu^{3+} phosphor. The computation of chromaticity co-ordinates is based on the procedures made available by Commission of International de L'Eclairage (CIE), France. The CIE chromaticity (x = 0.65, y = 0.34) of YAM:Eu^{3+} at 147 nm and (x = 0.67, y = 0.32) at 172 nm is shown in inset of Fig. 5, compared with YGB:Eu^{3+} (x = 0.64, y = 0.35). This information provides an evidence that the Eu^{3+} doped YAM phosphor exhibit vivid red emission with CIE color coordinates and color purity that are comparable to or better than the commercially used YGB:Eu^{3+} phosphor. The prepared YAM:Eu^{3+} phosphor has CIE values much closer to the standard NTSC (x = 0.67, y = 0.33) values. All these results supplement that YAM:Eu^{3+} is potentially a good candidate for PDP application as there is an effective energy transfer from host to the Eu^{3+} ions giving rise to efficient emission at 612 nm due to radiative recombination between $^5D_0$-$^7F_2$ levels.

It is well known that the phosphors used for PDP should have fast response so that there is no lag in image transformation. The ideal decay time of phosphor should be less than 2 msec (1/10 decay time) to prevent cross-talk or ghost image formation [30]. A conventional PDP phosphor YGB:Eu^{3+} has 1/10 decay time of 12 msec (manufacturer's data sheet) and 1/e decay time of 8.5 msec [31]. This decay time is far too long for realization of a recently conceptualized concept of 3D PDPs and also for High Definition (HD) PDPs [30]. Therefore, this phosphor should be precluded from usage for PDPs and it is essential to develop fast-decaying red-emitting phosphors with satisfactory PL properties for the next generation 3D PDP. As a best candidate for a fast-decaying red-emitting phosphor, YAM:Eu^{3+} was prepared with a decay time of 2.88 msec (1/10 decay time) and 1.03 msec (1/e decay time).

The Eu^{3+} ($^5D_0$-$^7F_2$) PL decay in YAM and YGB hosts at 250 nm excitation by time correlated single photon counting technique is shown in Fig. 6. The PL decay curve was measured with a microsecond pulsed xenon flash lamp as the source of excitation. The lifetime data was very well fitted single exponentially and the parameters generated from the fitting are listed in the inset of Fig. 6. Due to mixing of spin and parity states of Eu^{3+} occupying a non centro symmetric site, the otherwise parity forbidden $^5D_0$-$^7F_2$ transition becomes partially allowed leading to faster decay to the ground state.
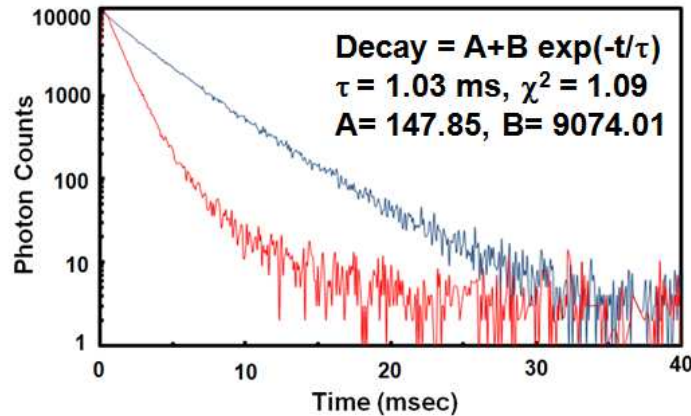

Fig. 6. (color online) Time-resolved PL decay spectrum of 612 nm $^5D_0$-$^7F_2$ luminescence of Eu^{3+} in (a) as prepared YAM host (red line) and (b) commercially used YGB host (blue line) at 300 K.

4. Conclusion

In summary, monophasic and monochromatic YAM:Eu^{3+} phosphor were prepared by solid-state reaction method. Results reveal that prepared YAM:Eu^{3+} phosphor is well crystallized and has uniform morphology with a controlled size ~1 μm which meet the requirement of advanced PDP and HDTVs. Post synthesis SiO_2 coating of YAM:Eu^{3+} phosphor grains followed by baking gives a degradation arrested highly intense phosphor. When compared with commercially available YGB:Eu^{3+}, the CIE colour coordinates of prepared YAM:Eu^{3+} are closer to that of standard NTSC values under 147 nm and 172 nm VUV excitation. Also the prepared YAM:Eu^{3+} phosphor shows 23% better integrated intensity than commercial one. The measured quantum efficiency of the commercial red phosphor is 74% at 250 nm. Hence developed YAM:Eu^{3+} phosphor has better quantum efficiency that commercial YGB:Eu^{3+}phosphor. To make possible the advancement of PDP TVs, fast-decaying YAM:Eu^{3+} phosphor system was found suitable with 1/e decay time 1.03 msec, which is substantially lower than the commercially used PDP red phosphor. Finally, Due to their excellent short decay time, PL and chromaticity the present YAM:Eu^{3+} phosphor have potential application in the field of advanced PDPs.

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