Appl Phys B (2011) 105:479–484
DOI 10.1007/s00340-011-4685-y

Applied Physics B
Lasers and Optics

Combustion synthesis of YAG:Ce and related phosphors

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Received: 13 January 2011 / Revised version: 25 March 2011 / Published online: 19 August 2011

Abstract YAG:Ce is an important phosphor having applications in various fields ranging from solid state lighting to scintillation detectors. YAG phosphors doped with activators are mainly synthesized by solid state reaction techniques that require high sintering temperatures (above 1500°C) to eliminate YAM and YAP phases. Though several soft chemical routes have been explored for synthesis of YAG, most of these methods are complex and phase pure materials are not obtained in one step, but prolonged annealing at temperatures around 1000°C or above become necessary. One step combustion synthesis of YAG:Ce^{3+} and related phosphors carried out at 500°C furnace temperature is reported here. Activation with Ce^{3+} could be achieved during the synthesis without taking recourse to any post-combustion thermal treatment. LEDs prepared from the combustion synthesized YAG:Ce^{3+}, exhibited properties comparable to those produced from the commercial phosphor.

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1 Introduction

The YAG:Ce phosphor developed in 1967 by Blasse and Brill [1] has been practically used as cathode ray tube phosphor (P 46 and P 48). Blasse and Brill [2] also showed that this phosphor emitted in yellow region when excited by blue light. Emission color could be tuned by substituting Gadolinium at Yttrium site for the red shift, and Gallium at the aluminum site for blue shift. Modifications brought in the PL characteristics by substitutions continue to interest researcher to date [3–5]. Use of the long wavelength excitation band centered round 460 nm was reported by Wyner and Daigneault [6], and Van Kemenade et al. [7]. They described the application of YAG:Ce in low-pressure mercury vapor discharge lamps to absorb the Hg-plasma lines in the blue/violet part of the spectrum, viz. 405 and 436 nm. The conversion of the violet and blue emission lines into yellow light adds to the white light emitted by the halophosphate phosphor (the commonly used phosphor in those days) to create a warmer white light. YAG:Ce is also a commercial phosphor (Sylvania 251) used for improving CRI in HPMV lamps. In the 1990s, YAG:Ce and the higher density analogue LuAG:Ce were proposed as fast and efficient scintillator materials, and interest in this topic is continuing to date [8, 9]. YAG:Ce scintillators are used in a variety of applications that include a calorimeter for a Proton Computed Radiography [10]. A recent application of Ce^{3+}-doped YAG luminescence is the use under single-crystalline disks to monitor and control the dose of vacuum/ultraviolet (VUV) radiation in wafer steppers. The dose applied for photoetching is measured by converting a small part of the VUV radiation from an excimer laser pulse into visible light [11]. Active-tip based, near-field optics are realized by coating a standard tip with Ce^{3+}-doped YAG particles [12]. YAG:Ce^{3+} nanoparticles conjugated with biomolecules work as biomarkers for fluorescent bioimaging [13, 14]. Recently, Ce^{3+}–Yb^{3+}-codoped YAG has been suggested as a downconversion material for converting the visible and near UV solar radiation, with quantum efficiencies exceeding 100%, to IR region where c-Si solar cells are most efficient [15, 16]. Ce^{3+}–Nd^{3+} energy transfer in YAG host is used to obtain efficient IR emitting phosphors [17]. Ce^{3+}-doped YAG is the most widely applied phosphor in white light LEDs [18–20]. Part of the blue light from the (In,Ga)N LED is absorbed by a thin layer of Ce^{3+}-doped YAG and then it is converted into yellow light. The combination of blue and yellow gives a bright white light source with an overall energy efficiency that is exceeding that of the compact fluorescent lamp.

Research on YAG:Ce has been conducted on single crystals, ceramics, single crystal films (SCF), bulk, and nanopowders. Due to high melting point, it is difficult to grow transparent single crystals needed for scintillation studies. Moreover, the single crystals contain lattice defects that lead to UV emission with longer decay times [21]. Transparent ceramics obtained by sintering powders are more suited. Alpha and beta particles have small range, and thus SCF are useful in their detection. At present, the fields of applications of SCF as luminescent detectors of ionizing radiation include scintillators for monitoring of alpha and beta and "phosvich" type scintillators/detectors for registration of the components of mixed ionizing fluxes, screens for visualization of X-ray images with high spatial resolution, and screens for electron-beam tubes [22]. Mechanical robustness facilitates the preparation of SCF of YAG:Ce down to about 1 micron thickness by liquid phase epitaxy (LPE) [23]. Nanoparticles can be easily attached to biomolecules due to comparable size, and hence they are important in bioimaging and similar applications. They also offer ease of coating needed in an application like phosphor sensitized solar cells. For white LED applications, it is reported that YAG:Ce in nanosized order performs better than it does in larger particles [24]. This is because nanosized YAG:Ce can reduce internal light scattering when coated onto a blue LED surface.

A good overview of the different synthesis methods for crystalline powders is provided by Pan et al. [25]. There are several methods to prepare Ce:YAG phosphor, but to achieve high emission efficiency, a fine nonagglomerated powder with good purity, narrow particle size distribution, and high crystallinity are prerequisites. YAG phosphors doped with activators are mainly synthesized by solid-state reaction techniques that require high sintering temperatures (above 1800°C) to eliminate YAM and YAP phases [26]. Fluxes such as BaF_2 or YF_3 can lower the sintering temperature down to 1500°C [27]. Several novel methods have been used to obtain phase pure YAG:Ce^{3+} at lower temperatures. Thus, YAG:Ce phosphor has been prepared using novel methods such as precipitation [28–30], heterogeneous precipitation [31], and spray pyrolysis from ethylenediaminetetraacetic acid solution [32], Spray pyrolysis of polymeric precursors [33], flame spray pyrolysis [34, 35], sol–gel pyrolysis [36], sol–gel [37–39], sol–gel combustion [40] glycothermal [41], combustion synthesis using urea [42, 43] or carbohydrazide [44] fuels, in situ esterification [45], microwave homogeneous synthesis, which is a combination of the traditional homogeneous coprecipitation with microwave irradiation [46], etc.

Though several soft chemical routes have been explored for synthesis of YAG, most of these methods are complex and high cost for the industrialization. Moreover, phase pure materials are not obtained in one step, but prolonged annealing at temperatures around 1000°C or above become necessary.

In this paper, we describe one-step combustion synthesis of YAG:Ce^{3+} and related phosphors carried out at 500°C furnace temperature. Activation with Ce^{3+} could be achieved during the synthesis without taking recourse to any post-combustion thermal treatment. This could be achieved by modifying the conventional solution combustion. Mixed fuel (glycine + urea) was used. Various components of the combustion mixture were mixed in the solid phase instead of solution. This route facilitated incorporation of Ce^{3+}. In solutions, cerium tends to be in partially tetravalent form. Advantages of the combustion synthesis over the other methods are well known. It is a fast, energy saving, and low cost method. Though the combustion reaction is initiated at furnace temperature of 500°C, the flame temperature attained due to exothermic nature of the reaction is as high as 1400°C.

2 Experimental

Instead of the conventional solution combustion synthesis, we used the modified procedure which led to the formation of the desired compounds in a single step. Reagent grade (Indian Rare Earths, Ltd.) rare earth oxides/carbonates were converted to the corresponding nitrates by dissolving in nitric acid. The nitrates were dried by prolonged, gentle warming. Stoichiometric amounts of hydrated nitrates of yttrium, aluminum, and cerium were thoroughly mixed with urea/glycine. The nitrate to fuel ratios were calculated by the method described earlier [47, 48]. Table 1 gives the details of the ingredients used in syntheses of various phosphors. Due to the presence of large water of crystallization in aluminum nitrate, a thick paste was formed. A china dish containing the paste was inserted in a furnace preheated to 500°C. Within minutes, the paste foamed and a flame was produced which lasted for several seconds. The china dish was immediately removed from the furnace. X-ray diffraction patterns were recorded on Philips PANalytical X'pert

Table 1 Combustion mixtures used in preparation of various phosphors

Sr. No.	Composition	Ingredients mol ratios YN:CeN:GN:AlN:glycine:urea	Remarks
1	$Y_{2.985}Ce_{0.015}Al_5O_{12}$	2.985:0.015:0:5:0:20.0	No PL, no phase formation
2	$Y_3Al_5O_{12}$	3.0:0:0:5:5:12.5	Phase pure YAG
3	$Y_{2.985}Ce_{0.015}Al_5O_{12}$	2.985:0.015:0:5:5:12.5	Phase pure YAG, strong PL
4	$Y_3Al_4GaO_{12}$	3.0:0:0:5:5:12.5	Compound formed with small amount of YAP,
5	$Y_{2.985}Ce_{0.015}Al_4GaO_{12}$	2.985:0.15:1:4:6.64:10.0	Compound formed with small amount of YAP, strong PL
6	$Y_3Al_3Ga_2O_{12}$	3.0:0:0:5:5:12.5	Compound formed with small amount of YAP
7	$Y_{2.985}Ce_{0.015}Al_3Ga_2O_{12}$	2.985:0.15:2:3:10.0:7.5	Compound formed with small amount of YAP, strong PL

YN → $Y(NO_3)_3{\cdot}6H_2O$, CeN → $Ce(NO_3)_3{\cdot}6H_2O$, AlN → $Al(NO_3)_3{\cdot}9H_2O$ GN → $Ga(NO_3)_3$

Pro diffractometer. PL characteristics in the range of 200–700 nm, at room temperature were studied using a Hitachi F-4000 spectrofluorimeter, with 1.5 nm spectral slit width.

The phosphor (3 wt%) was dispersed in a transparent silicone resin (Wells Electronic Materials Company, 5012-2A and 5012-2B), and LED was then fabricated by coating the blue LED chip (CREE 458 nm, 300 micron) with the epoxy resin. The electroluminescence (EL) spectra, color temperature, CIE chromaticity coordinates, CRI, and lumen output at room temperature were measured using a 300 mm integrating sphere and lumen meter (Hangzhou Zhongwei Photoelectricity Company ZVision ZW 3900). The measurements were carried out at 3.2 V, 20 mA.

3 Results and discussions

Yang et al. [49] prepared YAG using combustion synthesis with urea as a fuel. However, the combustion product was admixture of YAG and YAP. Repeated sintering for 5 h at 1000°C was necessary to obtain YAG. More or less similar results were obtained by Pan et al. [42]. Fu et al. [44] used carbohydrazide fuel, but annealing at 1150°C for 6 hours was required to achieve good crystallinity and PL intensity. In our experiments, when urea was used as a fuel, the combustion products were poorly crystallized. Yttrium nitrate does not have exothermic reaction with urea. Hence, we tried mixed (glycine + urea) fuel. Glycine has exothermic reaction with yttrium nitrate and urea with aluminum nitrate. XRD pattern of the product obtained with this fuel is shown in Fig. 1. An excellent match is obtained with ICDD file 88-2048 corresponding to YAG. No lines corresponding to YAP (2 theta = 33.36), YAM (2 theta = 29.289) or YAH (2 theta = 32.832) could be seen. Phase pure YAG is thus obtained in a single step combustion process without any additional thermal treatment when mixed fuel (Glycine + urea) was used.

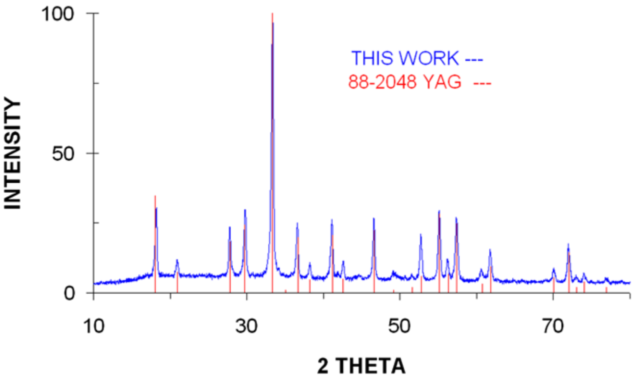

Fig. 1 XRD results for YAG XRD data for YAG is compared with the ICDD data file 88-2048

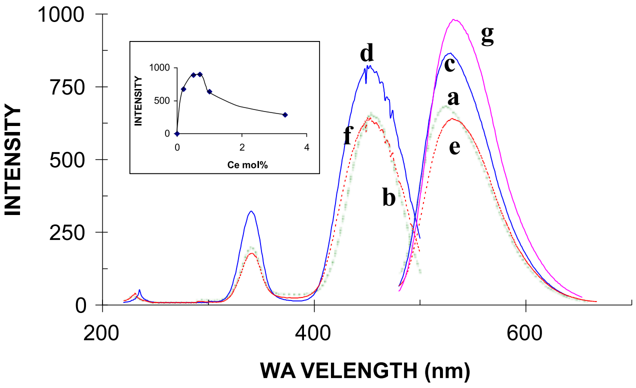

Fig. 2 PL spectra for Ce^{3+} activated samples: *a*, *c* and *e* > YAG:Ce^{3+} emission for 465 nm excitation, Ce^{3+} concentration *a* > 0.5, *c* > 0.7 and *e* > 3.33 mol%, *b*, *d*, *f* > excitation for 528 nm emission of YAG:$Ce^{3+}Ce^{3+}$ concentration, *b* > 0.5, *d* > 0.7 and *f* > 3.33 mol%. *g* > emission of commercial YAG:Ce^{3+} (Lumitech LM460) for 465 nm excitation

Activation with Ce^{3+} was also attempted by adding serous nitrate to the combustion mixture. Figure 2 shows PL results for the phosphor so prepared. For 465 nm excitation, intense emission was observed with a broad maximum

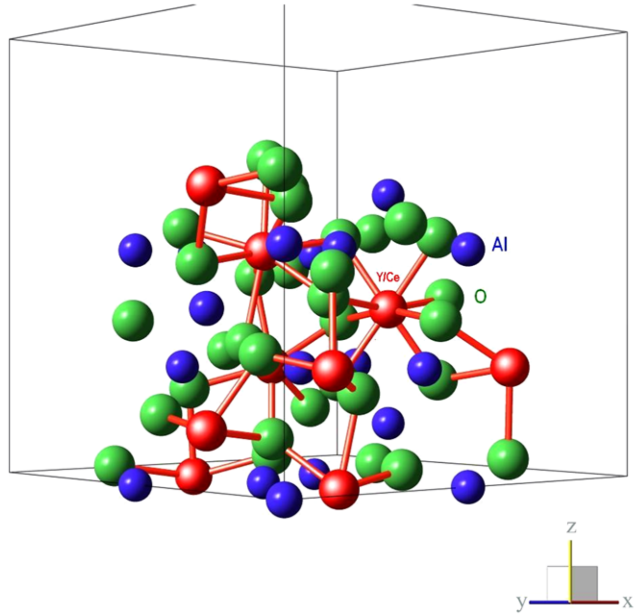

Fig. 3 Part of YAG unit cell showing 8 coordination of Y^{3+} with neighboring oxygen ions

at 528 nm and a shoulder around 540 nm (Fig. 2, curve a). This is in excellent agreement with the recent literature results [40]. The excitation spectrum (Fig. 2, curve b), consists of several bands; those around 460 and 340 nm being prominently seen. These positions are also in close agreement with the most of the literature results on YAG:Ce phosphors. Moreover, no emission attributable to YAP or YAM phases was observed. For YAM:Ce, Blasse and Brill [2] mention two emission bands at 320 and 357 nm. Excitation spectrum consisted of bands at 241, 294, and 306 nm. In case of YAP:Ce, the emission spectrum for 303 nm excitation consists of an unresolved doublet having maximum at 368 nm with a shoulder on short wavelength side around 340 nm. No such emission was observed in YAG:Ce prepared in this work. Inset of Fig. 2 shows concentration dependence of PL intensity. Maximum PL was observed for Ce concentration of 0.7 mol%. PL intensity is about 85% of that for the commercial sample (Lumitech LM460).

The PL results are compatible with the known energy level structure of Ce^{3+} in YAG host. The YAG crystal has a bcc structure (space group I a $\bar{3}$d) [50]. The unit cell consists of four formula units with a single type of Y-site. Eight oxygen atoms in a distorted dodecahedron coordinate the Y atom, giving it D_2 symmetry. The large Ce^{3+} ion occupies the 24(c) sites of Y^{3+} with D_2 point group symmetry, and each of the ions is in an almost dodecahedral coordination of eight oxygens, four at 230.3 pm, and four at 243.2 pm (Fig. 3). The second coordination shell is made of two Al^{3+} at 300.2 pm, with coordination four quasitetrahedral, four Al^{3+} at 335.6 pm, with coordination six quasioctahedral, and four Y^{3+} at 367.7 pm, with coordination eight quasi-dodecahedral, all of them sharing two coordination oxygens with the central Y^{3+} (or Ce^{3+}) [51]. Due to tetragonal symmetry of the dodecahedral yttrium site in YAG, the 5d state splits into lower-lying doublet E_g and upper-lying triplet T_{2g} level which are further split due to given symmetry and spin-orbit interaction into five levels. This results into five well-known absorption bands around 460 and 340 nm, corresponding to transitions to split levels originated from $2E_g$ and 261, 224, and 205 nm related to transitions to split levels originated from T_{2g} [52]. The origin of 261 nm band is under discussion in the literature and it is not clear whether it is a transition to the third 5d level or a transition related to a defect or impurity in the YAG host lattice. For the present work, the origin of this band is not important. Bands around 460 and 340 nm are commonly observed at RT. Actual positions depend upon Ce^{3+} concentration and the method of preparation as well [53]. The two remaining 4f–5d absorptions from the ground state are hidden by host absorptions. The emission of YAG:Ce^{3+} is made of seven 5d → 4f transitions which overlap to produce a two-peak band.

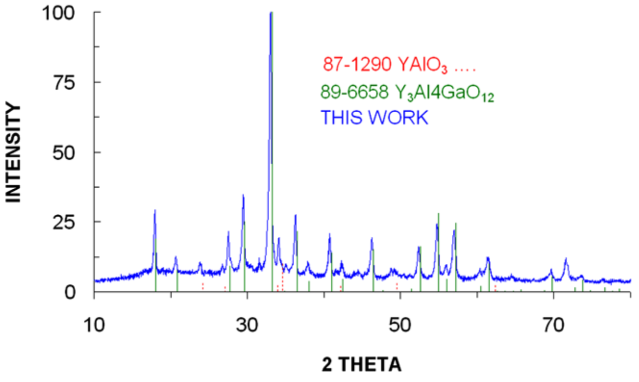

Fig. 4 XRD Results for $Y_3Al_4GaO_{12}$. XRD data for $Y_3Al_4GaO_{12}$ is compared with the ICDD data file 89-6658

After being successful, one step synthesis of YAG:Ce^{3+}, the next logical step is to attempt preparation of the related phosphors by a similar procedure. Substitution of Ga at Al site shifts the emission to shorter wavelengths. Synthesis of $Y_{2.985}Ce_{0.15}Al_4GaO_{12}$ and $Y_{2.985}Ce_{0.15}Al_3Ga_2O_{12}$ was attempted. Figures 4 and 5 show results of the XRD measurements. The desired phases are formed. However, there are some extra weak lines which are close to the positions of YAP phase [54]. Other possible phases like YAM [55] or YAH [56] were not indicated. No lines corresponding to YAM (29.289) or YAH (32.832) could be seen.

PL spectra are presented in Fig. 6. Emission and the strongest excitation band around 465 nm show blue shift with incorporation of Ga. The excitation band around 340 nm, on the other hand, shows a red shift.

LEDs were fabricated using YAG:Ce phosphors prepared in this work and the commercial phosphor as described in the experimental. Figure 7 shows the emission spectra

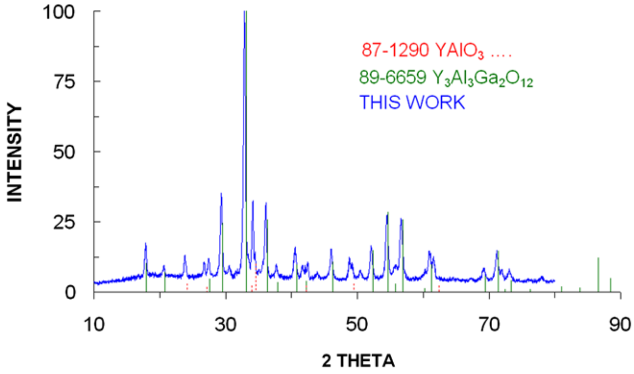

Fig. 5 XRD results for $Y_3Al_3Ga_2O_{12}$. XRD data for $Y_3Al_3Ga_2O_{12}$ is compared with the ICDD data file 89-6659

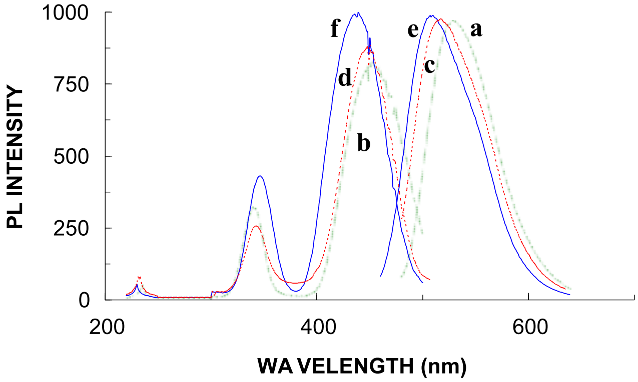

Fig. 6 PL spectra for Ce^{3+} activated samples: $a >$ YAG:Ce^{3+} emission for 465 nm excitation, $b >$ excitation for 528 nm emission of YAG:Ce^{3+}, $c >$ $Y_3Al_4GaO_{12}$:Ce^{3+} emission for 450 nm excitation, $d >$ excitation for 517 nm emission of $Y_3Al_4GaO_{12}$:Ce^{3+}, $e >$ $Y_3Al_3Ga_2O_{12}$:Ce^{3+} emission for 440 nm excitation and $f >$ excitation for 510 nm emission of $Y_3Al_3Ga_2O_{12}$:Ce^{3+}

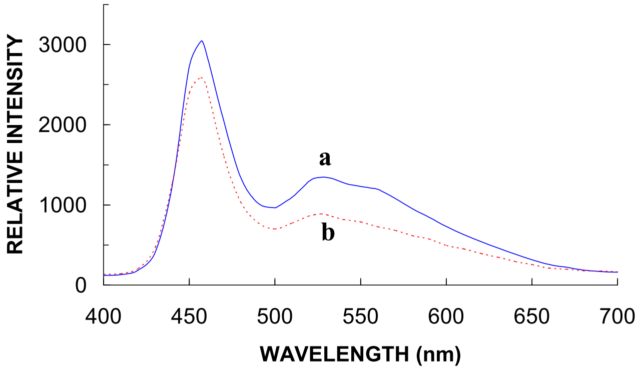

Fig. 7 Emission spectra of LED: $a >$ LED fabricated from commercial YAG:Ce phosphor, $b >$ LED fabricated from YAG:Ce phosphor prepared in this work

for these LEDs. For the LED fabricated with the commercial phosphor, the color coordinates are 0.322, 0.337; color temperature 6000 K, lumen output 87.5 lumen/Watt, and CRI 70. For the LED fabricated with our phosphor, the corresponding figures are color coordinates 0.300, 0.317; color temperature 6700 K, lumen output 72.5 lumen/Watt, and CRI 67. Ratio of blue to yellow is higher for the phosphor prepared in this work. By varying the concentration of the phosphor in the resin, all these parameters could be changed. Figure 8 shows the variation of LED emission with the phosphor concentration.

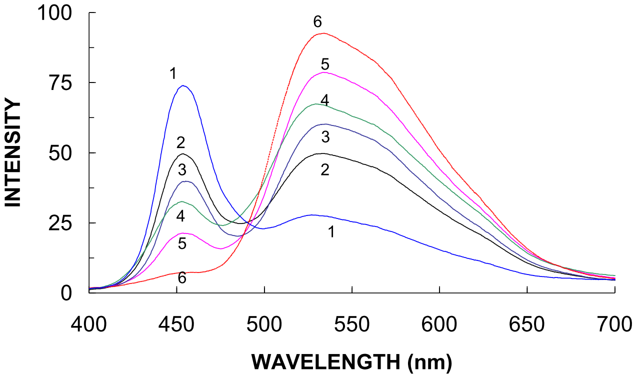

Fig. 8 Emission spectra of LED for various phosphor concentrations. Emission spectra of LEDs fabricated from YAG:Ce phosphor prepared in this work are shown. Phosphor concentration (wt% of epoxy resin)—1 > 3, 2 > 6, 3 > 9, 4 > 12, 5 > 15 and 6 > 18. LED characteristics were measured at 3.2 V, 20 mA forward current

Almost all of the entire blue light is converted to yellow for phosphor concentration of 18%. The corresponding color temperature varies in the range of 4500–6700 K, the color coordinates from 0.372, 0.441 to 0.300, 0.317, and CRI from 55 to 70. The lumen output is of the order of 75 lumen/Watt.

4 Conclusions

Phase pure YAG could be prepared by a one-step combustion synthesis employing (Glycine + urea) mixed fuel at the furnace temperature of 500°C. No further heat treatment was needed. Replacing Al by gallium, however, resulted in contamination of YAG by YAP phase. Preparation of Ce^{3+} activated phosphors was also attempted. Ce^{3+} was successfully doped into YAG and other related phases. PL and LED spectra of the YAG:Ce phosphor prepared and the LED fabricated in this work was comparable to those for the commercial phosphor.

Acknowledgements This work received financial support from the Department of Science and Technology, New Delhi.

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