Photoluminescence and cathodoluminescence properties of a novel $\text{CaLaGa}_3\text{O}_7:\text{Dy}^{3+}$ phosphor

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Received July 14, 2011; accepted October 19, 2011; published online December 27, 2011

A single host white emitting phosphor, $\text{CaLaGa}_3\text{O}_7:\text{Dy}^{3+}$, was synthesized by chemical co-precipitation. Field emission scanning electron microscopy, X-ray diffraction, laser particle size analysis, and photoluminescence and cathodoluminescence spectra were used to investigate the structural and optical properties of the phosphor. The phosphor particles were composed of microspheres with a slight tendency to agglomerate, and an average diameter was of about 1.0 μm . The Dy^{3+} ions acted as luminescent centers, and substituted La^{3+} ions in the single crystal lattice of $\text{CaLaGa}_3\text{O}_7$ where they were located in C_s sites. Under excitation with ultraviolet light and a low voltage electron beam, the $\text{CaLaGa}_3\text{O}_7:\text{Dy}^{3+}$ phosphor exhibited the characteristic emission of Dy^{3+} ($^4\text{F}_{9/2}-^6\text{H}_{15/2}$ and $^4\text{F}_{9/2}-^6\text{H}_{13/2}$ transitions) with intense yellow emission at about 573 nm. The chromaticity coordinates for the phosphor were in the white region. The relevant luminescence mechanisms of the phosphor are investigated. This phosphor may be applied in both field emission displays and white light-emitting diodes.

phosphor, cathodoluminescence, photoluminescence, field emission display

Citation: Zhao W Y, An S L, Fan B, et al. Photoluminescence and cathodoluminescence properties of a novel $\text{CaLaGa}_3\text{O}_7:\text{Dy}^{3+}$ phosphor. *Chin Sci Bull*, 2012, 57: 827–831, doi: 10.1007/s11434-011-4938-5

Recently, rare-earth-doped phosphors have been used widely in field emission displays (FEDs) and white light-emitting diodes (LEDs) because of advantages such as excellent luminescent characteristics, good stability, high luminescence efficiency, corrosion-free gas emission under electron bombardment and a strong excitation band in the blue or UV region [1,2]. Among these phosphors, much attention has been focused on sulfide, nitride, oxide and oxynitride phosphors. However, sulfide phosphors decompose readily and emit sulfide gases during continuous electron beam irradiation. This not only causes cathodes to deteriorate but also lowers the luminous efficiency of the phosphors [3,4]. Nitride and oxynitride phosphors require rigorous preparation conditions [5]. Therefore, oxide-based phosphors doped with rare-earth ions have been extensively studied because of their high stability and luminescence efficiency as well as being environ-

mentally friendly, relatively cheap and easy to prepare [6,7].

Calcium lanthanum gallium oxide $\text{CaLaGa}_3\text{O}_7$ forms tetragonal crystals that belong to the space group $\text{P}\bar{4}2_1\text{m}$. This crystal structure is composed of GaO_4 layers formed in the ab plane. Between the layers, divalent Ca and trivalent La cations are distributed randomly in eight coordinate sites with C_s symmetry [8]. However, this material has not been reported as a host material for phosphor applications. In addition, most phosphors are prepared by traditional high temperature solid-state reaction methods. However, because of insufficient mixing and the low reactivity of the raw materials, the prepared product usually possesses an irregular morphology or tendency to agglomerate with serious reunion and high hardness. In addition, luminescence efficiency of the product will be descended after milling. Use of chemical co-precipitation can overcome these disadvantages. Dy^{3+} ions, which luminesce in the 470–500 nm region because of the $^4\text{F}_{9/2}-^6\text{H}_{15/2}$ transition and in the 570–600 nm region be-

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cause of the $^4F_{9/2} \rightarrow ^6H_{13/2}$ transition, have attracted much attention because of their white light emission [9,10]. In this paper, a novel $\text{CaLaGa}_3\text{O}_7:\text{Dy}^{3+}$ phosphor was prepared by chemical co-precipitation. The photoluminescence (PL) and cathodoluminescence (CL) properties of this novel phosphor are investigated in detail.

1 Experimental

1.1 Synthesis of $\text{CaLaGa}_3\text{O}_7:\text{Dy}^{3+}$

The phosphor $\text{CaLaGa}_3\text{O}_7:\text{Dy}^{3+}$ was prepared by chemical co-precipitation. Oxalic acid was used as a precipitation agent. Based on single factor experiments, the optimum doping concentration for Dy^{3+} was found to be 5 mol%.

In a typical procedure, a nitrate solution was formed by adding $\text{Ca}(\text{NO}_3)_2$ (1 mol/L, 5 mL), $\text{La}(\text{NO}_3)_3$ (1 mol/L, 4.95 mL), $\text{Ga}(\text{NO}_3)_3$ (1 mol/L, 5 mL) and $\text{Dy}(\text{NO}_3)_3$ (0.1 mol/L, 0.5 mL) to a 100 mL beaker. Oxalic acid solution (1 mol/L, 25 mL) was added dropwise to the nitrate solution under magnetic stirring. The pH was adjusted to 10–12 by adding ammonia solution (25%, w/v) dropwise. After 4 h, the resulting white powder was filtered, washed with distilled water and absolute ethanol several times, and then dried under vacuum at 80°C for 4 h. Finally, the phosphor $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ was obtained by calcining the above powder at 1100°C for 3 h in air.

For comparison with $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$, the phosphor $\text{LaOCl}:2\%\text{Dy}^{3+}$ was prepared by the Pechini-type sol-gel method [11].

1.2 Characterization

The morphology of the sample was observed using a JEOL JSM-6700F field emission scanning electron microscope (FE-SEM). The particle size distribution of the sample was examined using a Mastersizer 2000 laser diffractometer. X-ray diffraction (XRD) measurements were performed on a Rigaku D/max-III B diffractometer using $\text{Cu K}\alpha$ radiation ($\lambda = 0.15405$ nm). An accelerating voltage and emission current of 35 kV and 60 mA, respectively, were used. PL measurements were performed on a Hitachi F-4500 spectrophotometer equipped with a 150 W Xenon lamp as the excitation source. CL measurements were performed in an ultrahigh-vacuum chamber ($<8.0 \times 10^{-4}$ Pa), where the phosphors were excited by an electron beam at a voltage range of 1.0–6.0 kV and different filament currents. CL spectra were recorded by a spectrometer (Hitachi F-4500) using a charge-coupled device camera through an optical fiber. All the measurements were performed at room temperature.

2 Results and discussion

Figures 1 and 2 show an FE-SEM micrograph and the particle

Figure 1 FE-SEM micrograph of the $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ phosphor.

Figure 2 Particle size distribution of the $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ phosphor.

size distribution of the $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ particles, respectively. The $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ sample consisted of microspheres exhibiting slight agglomeration. The particles had a rather narrow size distribution, with an average diameter of about 1.0 μm . According to our experience, these properties are suitable for making phosphor screens in FEDs.

Figure 3 shows the XRD pattern of the $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ phosphor and the Joint Committee for Powder Diffraction Standards (JCPDS) card 39-1127 of $\text{CaLaGa}_3\text{O}_7$ as a reference. All of the diffraction peak positions of the sample were consistent with those of the JCPDS card, with three primary peaks at 2θ of 30.283°, 35.655° and 50.069°. These peaks were ascribed to the (311), (420) and (422) diffraction planes of the $\text{CaLaGa}_3\text{O}_7$ phase, respectively. No additional peaks of other phases were detected, indicating the Dy^{3+} ions have been effectively doped into the $\text{CaLaGa}_3\text{O}_7$ host lattice. It is generally believed that impurity ions always take the place of the ions of similar radii and the same valence. In the crystal structure of $\text{CaLaGa}_3\text{O}_7$, La^{3+} cations are surrounded by eight anions in an octahedral fashion, and La^{3+} ions occupy sites with point symmetry of C_s . As a result, the Dy^{3+} ions substitute La^{3+} with a point symmetry C_s because of the small difference in ionic radii and identical valence state (+3) between Dy^{3+} (102.7 pm)

Figure 3 XRD pattern of $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$. The standard data for tetragonal phase $\text{CaLaGa}_3\text{O}_7$ (JCPDS No. 39-1127) is presented for comparison.

and La^{3+} (116 pm) ions in eightfold coordination [12,13]. According to the chekcell software (Collaborative Computational Project Number 14 (CCP14); <http://www.ccp14.ac.uk/ccp/web-mirrors/lmgp-laugier-bochu/>), the lattice constants of the $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ sample were $a=11.249$ Å, $c=5.251$ Å, $V=664.5$ Å³. So, compared with the lattice constants of tetragonal $\text{CaLaGa}_3\text{O}_7$ (JCPDS No. 39-1127, $a=11.252$ Å, $c=5.273$ Å, $V=667.2$ Å³), the sample presents slightly smaller lattice constants and unit cell volume because of the substitution of La^{3+} by Dy^{3+} with smaller ionic radius.

Figure 4 shows the excitation spectrum of the $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ sample ($\lambda_{\text{em}}=573$ nm). There are a number of peaks with the highest intensity peak at 349 nm. Clearly, these peaks are caused by the f-f transitions of Dy^{3+} in a $4f^9$ configuration, and can be assigned to the electronic transitions ($^6\text{H}_{15/2} \rightarrow ^5\text{P}_{3/2}$) at 324 nm, ($^6\text{H}_{15/2} \rightarrow ^6\text{M}^{2}_{1/2}$) at 349 nm, ($^6\text{H}_{15/2} \rightarrow ^6\text{P}_{7/2}$) at 362 nm, ($^6\text{H}_{15/2} \rightarrow ^4\text{I}_{13/2}$) at 386 nm, ($^6\text{H}_{15/2} \rightarrow ^4\text{G}_{11/2}$) at 426 nm, and ($^6\text{H}_{15/2} \rightarrow ^4\text{I}_{15/2}$) at 452 nm [14]. This large range of excitation peaks is appropriate for application

Figure 4 Excitation spectrum of $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$.

in UV- and blue chips to generate visible light.

Figure 5 shows the emission spectrum of $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ ($\lambda_{\text{ex}}=349$ nm). The emission spectrum only contains bands from f-f transitions from a $\text{Dy}^{3+} 4f^9$ electron configuration, i.e., $^4\text{F}_{9/2} \rightarrow ^6\text{H}_{15/2}$ (476, 487, 495 nm) in the blue region, and $^4\text{F}_{9/2} \rightarrow ^6\text{H}_{13/2}$ (573 nm) in the yellow region. It is well known that the $^4\text{F}_{9/2} \rightarrow ^6\text{H}_{15/2}$ transition is predominantly magnetically allowed and hardly varies with the crystal field strength around the Dy^{3+} ion. Moreover, the $^4\text{F}_{9/2} \rightarrow ^6\text{H}_{13/2}$ transition belongs to a hypersensitive transition with the selection rule $\Delta J=2$, which is a forced electric dipole transition that is only allowed at low symmetry with no inversion center [15,16]. So, according to Figure 5, Dy^{3+} is located at low-symmetry local sites or lacks an inversion center. As XRD analysis showed, the Dy^{3+} ions occupy sites with a point symmetry of C_s in the host $\text{CaLaGa}_3\text{O}_7$. Consequently, the symmetry of the Dy^{3+} point sites remains C_s . In addition, the octahedron is distorted because the radius of the Dy^{3+} ions is smaller than that of the La^{3+} ions, which has been shown by XRD analysis. The different radii of La^{3+} and Dy^{3+} reduce the symmetry of Dy^{3+} . Reduced symmetry of the coordination environment around the Dy^{3+} ions will enhance the yellow emission from the $^4\text{F}_{9/2} \rightarrow ^6\text{H}_{13/2}$ transition. This evidence confirms that the Dy^{3+} ions are mainly located in sites of the crystal lattice without inversion symmetry.

Figure 6 shows CL spectra of the $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ and $\text{LaOCl}:2\%\text{Dy}^{3+}$ phosphors. The CL spectrum obtained for $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ consists of a broad blue band with a maximum at 385 nm and two more intense emission bands in the blue (470–500 nm) and yellow regions (560–600 nm). The blue emission at 488 nm and the yellow emission at 576 nm are characteristic emissions of Dy^{3+} , and are caused by the transitions $^4\text{F}_{9/2} \rightarrow ^6\text{H}_{15/2}$ and $^4\text{F}_{9/2} \rightarrow ^6\text{H}_{13/2}$, respectively. The CL spectra are similar to the PL spectra of $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ except for the broad band at 385 nm and the different intensity ratio of the $^4\text{F}_{9/2} \rightarrow ^6\text{H}_{15/2,13/2}$ band. The

Figure 5 Emission spectrum of $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$.

Figure 6 CL spectra of the $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ phosphor under low voltage electron beam excitation (accelerating voltage: 1, 3, 5 and 6 kV) and the $\text{LaOCl}:2\%\text{Dy}^{3+}$ phosphor under low voltage electron beam excitation (accelerating voltage: 3 kV).

broad band at 385 nm can be attributed to the transition of a self-activated optical center related to octahedrally coordinated GaO_6 groups [3] and/or corresponds to an energy relaxation of electrons transiting from $^4\text{T}_{2\text{B}}$ to $^4\text{A}_2$ energy levels of the distorted Ga-octahedral structure [17]. The different branch ratio of the $^4\text{F}_{9/2} \rightarrow ^6\text{H}_{15/2,13/2}$ band may relate to differences in the responses of the apparatus and/or the excitation mechanisms involved in CL and PL.

In PL, UV and/or visible light is used to excite luminescent materials. The energy of these photons is only around 4–6 eV. Luminescent centers are directly excited by UV photons, such as light with a wavelength of 349 nm. After excitation, most of the energy transfers to the $^4\text{F}_{9/2}$ level of Dy^{3+} , which results in the characteristic transitions of Dy^{3+} , as shown in Figure 5. The influence of crystal field splitting and the level splitting of energy for Dy^{3+} cannot be neglected in this process. However, under electron-beam irradiation during CL, luminescent centers in phosphors are excited by either direct or indirect excitation [18]. The energy of electrons under acceleration from anode voltage can be tuned from a few thousand to thousands of eV. When a high-energy electron reaches the surface of a phosphor, it may penetrate into it. After penetrating into the host lattice, the fast primary electrons will cause ionization. This ionization creates many secondary electrons. These secondary electrons can also cause ionization and create further secondary electrons [19]. When lower energy secondary electrons reach the luminescent centers, they may directly excite the luminescent centers. Also, these secondary electrons generate many electron-hole pairs (EHs) by collision with lattice ions. Generated EHs do not directly recombine in the lattice. They act as mobile carriers in the particles, and recombine at luminescent centers or Ga^{3+} ions. These excitons, if they form, will decay nonradiatively through a resonant or quasi-resonant transfer to the 4f shell of Dy^{3+} ions and in-

duce its characteristic emission. Moreover, it causes electrons to transition from the $^4\text{A}_2$ to the $^4\text{T}_{2\text{B}}$ energy level of Ga^{3+} and causes a band at 385 nm. As a result, the influence of crystal field splitting and the level splitting of energy for the Dy^{3+} ions are not important in the CL process.

According to Figure 6, when the filament current is fixed at 50 mA, the CL intensity increases with raising the accelerating voltage from 1.0 to 6.0 kV. This can be attributed to the deeper penetration of the electrons into the phosphor. The electron penetration depth can be estimated using the empirical formula $L(\text{\AA}) = 250(A/\rho)(E/Z^{1/2})^n$, where $n = 1.2/(1 - 0.29 \log_{10} Z)$. A is the atomic or molecular weight of the material, ρ is its bulk density, Z is the atomic number or the number of electrons per molecule in the case of compounds, and E is the accelerating voltage (kV) [20]. For the $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ sample, $Z = 226$, $A = 500.15$, and $\rho = 4.974 \text{ g/cm}^3$, so the electron penetration depths at anode voltages of 1.0, 3.0, 5.0 and 6.0 kV are estimated to be 0.9, 5.7, 39.1 and 78.0 nm, respectively. Hence, the increase in CL intensity with increasing applied voltage is caused by an increase in the number of excited luminescent centers resulting from an increase in electron penetration depth.

In addition, for CL, quantum efficiency (q) is irrelevant. The radiant efficiency (η) is defined as the emitted power to the power of the electron beam falling on the luminescent material. The luminous efficiency (L) is defined in a similar manner as for PL [21]. L is the ratio of the luminous flux ($\Phi(v)$) emitted by the material to the absorbed power. $\Phi(v)$ in the general case is given by the folding or weighting of the spectral power distribution $\Phi(\lambda)$ with the normalized photopic response curve $V(\lambda)$ and the constant factor K_m [22]. $\Phi(\lambda)$ is directly proportional to the CL intensity (in height). So, for the $\text{CaLaGa}_3\text{O}_7:\text{Dy}^{3+}$ and $\text{LaOCl}:\text{Dy}^{3+}$ phosphors, η can be also compared roughly by considering their CL emission peak areas [3,11]. L can be also compared roughly by the weighting of their CL intensity (in height) with $V(\lambda)$. It can be seen from Figure 6 that the emission peak area of $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ (3 kV, curve (b)) is higher than that of $\text{LaOCl}:2\%\text{Dy}^{3+}$ (3 kV, curve (c)), but the weighting of CL intensity (in height) with $V(\lambda)$ of $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ is smaller than that of $\text{LaOCl}:2\%\text{Dy}^{3+}$. So η of $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ might be higher than that of $\text{LaOCl}:2\%\text{Dy}^{3+}$, but L of $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ might be smaller than that of $\text{LaOCl}:2\%\text{Dy}^{3+}$.

Figure 7 (A–E) shows the chromaticity coordinates (X , Y) of the $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ phosphor excited at 349 nm, and 1.0, 3.0, 5.0 and 6.0 kV, respectively, in the Commission Internationale de L'Eclairage (CIE) 1931 chromaticity diagram. It is obvious that almost the whole emission of the $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ phosphor is in the white light region. Therefore, according to the PL spectra, the $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ phosphor is suited for use as a color converter of white LEDs by combining a UV- or blue LED chip with this phosphor. Compared with the yellow phosphor LaOCl :

Figure 7 (Color online) CIE chromaticity diagram for $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ and $\text{LaOCl}:2\%\text{Dy}^{3+}$.

$2\%\text{Dy}^{3+}$ [11], the chromaticity coordinates of the $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ phosphor are located in the white-light region under electron beam irradiation. Hence, $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ is a promising material for use as a white phosphor in FEDs.

3 Conclusions

The phosphor $\text{CaLaGa}_3\text{O}_7:\text{Dy}^{3+}$ was prepared by chemical co-precipitation. FE-SEM images and particle size analysis suggest that the particles are spherical or almost spherical and show a narrow size distribution of about $1.0\ \mu\text{m}$. XRD revealed that Dy^{3+} ions were effectively doped into the $\text{CaLaGa}_3\text{O}_7$ host lattice. PL and CL spectra showed that the Dy^{3+} ions were mainly located in a site lacking inversion symmetry of the crystal lattice with the maximum emission wavelength at about 573 nm. PL and CL of the phosphor differed slightly because of the excitation mechanism. The radiant efficiency of $\text{CaLaGa}_3\text{O}_7:\text{Dy}^{3+}$ may be higher than that of the yellow phosphor $\text{LaOCl}:2\%\text{Dy}^{3+}$. Under ultraviolet (349 nm) and low-voltage electron beam excitation (1.0–6.0 kV), the $\text{CaLaGa}_3\text{O}_7:5\%\text{Dy}^{3+}$ sample showed white light emission. This unique property could be applied in many fields, including displays and light sources, such as FEDs and white LEDs.

This work was supported by the Natural Science Foundation Committee of Inner Mongolia (20080404MS0204), the National High Technology Re-

search and Development Program of China (2008AAXX0310) and the Innovation Fund of Inner Mongolia University of Science and Technology (2010NC026).

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