Research Article

Improving Upconversion Photoluminescence of $\text{GdAlO}_3\text{:Er}^{3+}$, Yb^{3+} Phosphors via Ga^{3+} , Lu^{3+} Doping

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Received 5 April 2019; Accepted 29 May 2019; Published 1 July 2019

Academic Editor: Sulaiman W. Harun

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$\text{GdAlO}_3\text{:Er}^{3+}$, Yb^{3+} phosphors doped with Lu^{3+} , Ga^{3+} ions were prepared via coprecipitation. The influences of substituting Ga^{3+} for Al^{3+} and substituting Lu^{3+} for Gd^{3+} on the structure and upconversion photoluminescence (UCPL) of the phosphors were studied. The experimental results show that the crystal textures did not change but the lattice parameters changed slightly via Lu^{3+} , Ga^{3+} doping. This results in a decrease in the host phonon energy and a marked improvement in the green emission spectrum at 546nm. Moreover, the amounts of surface CO_2 , CO_3^{2-} , and OH^- species gradually decreased with Lu^{3+} , Ga^{3+} doping. The combined effects led to an improvement in the UCPL efficiency with Ga^{3+} , Lu^{3+} doping.

1. Introduction

Upconversion (UC) materials can absorb two or three energy photons to convert infrared light to visible light [1, 2]. In recent years, Ln^{3+} -doped UC materials have attracted much attention because of their potential applicability in many attractive research areas, such as solid-state lasers, biological imaging, solar cells, and the quantum dots [3–9]. It is well known that rare earth metals have a particular electronic structure with different 4f electronic numbers, and these are suitable for the UC process. Among the rare earth metals, Er^{3+} has a rich 4f level structure, and its long intermediate state energy level lifetime can absorb two or more photons to achieve UC luminescence under excitation of 650, 808, 980, and 1500 nm wavelengths. However, Yb^{3+} which has a large absorption cross-section and an energy level that matches with that of Er^{3+} is often codoped as the sensitizer. Yb^{3+} can absorb energy at 980 nm and can transfer the energy to Er^{3+} to improve the UC photoluminescence (UCPL) efficiency of phosphors [10–12].

Currently, phosphors with oxides as the host material exhibit high UC luminescence efficiency because of their low phonon energy, good chemical stability, mild synthesis conditions, and ecofriendliness. A lot of oxides, such as

Sr_2CeO_4 , Bi_2WO_6 , NaGdTlO_4 , and $\text{KBaLu}(\text{MoO}_4)_3$, exhibit excellent UC luminescence properties as have been successively reported [13–16]. To the best of our knowledge, GdAlO_3 belongs to the perovskite structure, orthogonal crystal system, and Pbnm space group. The density of GdAlO_3 is $7.437\text{g}\cdot\text{cm}^{-3}$, and the phonon energy is 670 cm^{-1} , which is good for UCPL [17]. In particular, the phonon energy and symmetry of the matrix have a great influence on UCPL efficiency. The phonon energy of the host material is mainly related to the relative atomic mass and bond length of the matrix elements. In this work, the lattice parameters of perovskite GdAlO_3 are substituted with other ions, such as Ga^{3+} , Lu^{3+} , to adjust the phonon energy of the host material and then to improve the $\text{GdAlO}_3\text{:Er}^{3+}$, Yb^{3+} phosphor UCPL.

2. Experimental Section

2.1. Phosphors Preparation. The precursors of $\text{GdAlO}_3\text{:Er}^{3+}, \text{Yb}^{3+}$ phosphors doped with $\text{Ga}^{3+}/\text{Lu}^{3+}$ were prepared using the coprecipitation method [18]. Gd_2O_3 (99.99%), Yb_2O_3 (99.99%), Er_2O_3 (99.99%), Lu_2O_3 (99.99%), and Ga_2O_3 (99.99%) were prepared as the starting materials without further purification. These were dissolved in dilute nitric acid under heating, and then a required amount of

$\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ and ethanol aqueous solution were added sequentially under vigorous stirring until a homogenous solution formed in a 45°C water bath. Then $1 \text{ mol} \cdot \text{L}^{-1}$ NH_4HCO_3 aqueous solution was slowly added to obtain a white precipitate with magnetic stirring at a rate of $2 \text{ mL} \cdot \text{min}^{-1}$. After complete precipitation, the final pH of the slurry was around 7.5. The agitator was then turned off, and the precipitate was ripened at room temperature for 12 h. The precipitate was filtered and washed several times with deionized water. The precipitate was dried in an oven at 120°C for 12 h. In the end, the precipitate was ground and annealed in air at 1200°C for 6 h to obtain the white phosphor sample.

The samples were characterized using powder X-ray diffraction (XRD) performed on a Bruker D8 Advance diffractometer with $\text{Cu K}\alpha$ radiation ($\lambda=0.154056 \text{ nm}$) operated at 30 mA and 40 kV. The UCPL spectra of the phosphors were recorded using an Ocean Optics PlasCalc-2000-UV-VIS-NIR plasma monitor and control system. The phosphors were excited with an MDL-H-980 model 980 nm infrared laser made by Changchun New Industries Optoelectronics Tech. Co., Ltd. Fourier-transform infrared (FT-IR) absorption spectra were recorded using a Nicolet 360 FTIR spectrometer. All of the measurements were carried out at room temperature. The energy level diagram was reproduced from Taoli Deng et al. (2018) [under the Creative Commons Attribution License/public domain] [17].

3. Results and Discussion

3.1. $\text{GdAlO}_3:\text{Er}^{3+}, \text{Yb}^{3+}$ Phosphors Doped with Ga^{3+} . Figure 1 presents the XRD patterns of the $\text{Gd}(\text{Al}_{1-x}\text{Ga}_x)\text{O}_3:\text{Er}^{3+}_{0.01}, \text{Yb}^{3+}_{0.08}$ ($x=0.00, 0.01, 0.03, 0.05$) phosphors. All of the phosphor spectra match the standard data for perovskite GdAlO_3 (PDF#46-0395) [19], and no other impurities were detected. Compared to the pattern of the $\text{Gd}_{0.91}\text{AlO}_3:\text{Er}^{3+}_{0.01}, \text{Yb}^{3+}_{0.08}$ phosphor, the spectral peaks of the phosphors are shifted to smaller diffraction following Ga^{3+} doping. Because the ionic radii of Ga^{3+} (0.0620 nm, CN=8) are smaller than that of Al^{3+} (0.0535 nm, CN=8) [20], the host lattice expands, and this indicates that Lu^{3+} ions were successfully incorporated into the GdAlO_3 host lattices. The average sizes of the crystallites are estimated using Scherrer's equation [21]:

$$D = \frac{0.89\lambda}{\beta \cos \theta} \quad (1)$$

where D is the average crystallite size, λ is the wavelength of the $\text{Cu K}\alpha$ line, β is the full width at half maximum in radians, and θ is the Bragg angle.

The strongest peak of the phosphor is at 28.6° ($\beta=0.064$) and was used to estimate the crystallite size using Scherrer's equation. Using this procedure, the prepared phosphor particles had an average crystallite size of 2.44 nm by this procedure.

To optimize the UCPL of the phosphor, a series of samples with different doping concentrations of Ga^{3+} were synthesized, and their UCPL spectra were investigated. Figure 2 shows that all of the $\text{Gd}(\text{Al}_{1-x}\text{Ga}_x)\text{O}_3:\text{Er}^{3+}_{0.01}, \text{Yb}^{3+}_{0.08}$

FIGURE 1: XRD patterns of $\text{Gd}(\text{Al}_{1-x}\text{Ga}_x)\text{O}_3:\text{Er}^{3+}_{0.01}, \text{Yb}^{3+}_{0.08}$ phosphors.

FIGURE 2: UCPL spectra of $\text{Gd}(\text{Al}_{1-x}\text{Ga}_x)\text{O}_3:\text{Er}^{3+}_{0.01}, \text{Yb}^{3+}_{0.08}$ phosphors.

($x=0.00, 0.01, 0.03, 0.05$) phosphors have three main emission bands with maxima at 524 nm (green), 546 nm (green), and 659 nm (red) excited by 980 nm radiation. The green emission peaks are assigned to $^2\text{H}_{11/2} \rightarrow ^4\text{I}_{15/2}$ and $^4\text{S}_{3/2} \rightarrow ^4\text{I}_{15/2}$ transitions of Er^{3+} , and the red emission peak is ascribed to the $^4\text{F}_{9/2} \rightarrow ^4\text{I}_{15/2}$ transition of Er^{3+} [22]. Moreover, the position of the emission peak did not shift significantly with the substitution of Ga^{3+} . Clearly, a marked improvement in the emission spectra at 546 nm is observed with Ga^{3+} doping. As seen in Figure 3, when the substitution concentration of Ga^{3+} was increased to 5%, the intensity ratio of the phosphor was 2.36 times greater than that of the phosphor without any ion doping, and when the Ga^{3+} doping concentration was increased, the intensity ratio of red/green emission decreased significantly.

To determine the reasons for changes in the UCPL properties with the substitution of different concentrations Ga^{3+} for Al^{3+} , FT-IR spectra of $\text{Gd}(\text{Al}_{1-x}\text{Ga}_x)\text{O}_3:\text{Er}^{3+}_{0.01}, \text{Yb}^{3+}_{0.08}$ phosphors were tested in Figure 4. All of the phosphors appear to have absorption peaks at 3460 cm^{-1} and 1635 cm^{-1} and in the region less than 1000 cm^{-1} . On one hand, absorption below 1000 cm^{-1} is ascribed to the vibration of the $\text{Gd}(\text{Al}_{1-x}\text{Ga}_x)\text{O}_3$ host material, and the maximum vibration absorption peak of the phosphor matrix material shifts slightly in the low wavenumber direction with the substitution of Ga^{3+} for Al^{3+} . Specifically, the matrix vibration

FIGURE 3: UCPL intensity of $\text{Gd}(\text{Al}_{1-x}\text{Ga}_x)\text{O}_3: \text{Er}^{3+}_{0.01}, \text{Yb}^{3+}_{0.08}$ phosphors at 546nm (inset is the intensity ratio of red/green emission on Ga^{3+} concentration).

FIGURE 4: FT-IR spectra of $\text{Gd}(\text{Al}_{1-x}\text{Ga}_x)\text{O}_3: \text{Er}^{3+}_{0.01}, \text{Yb}^{3+}_{0.08}$ phosphors.

is 670 cm^{-1} without substitution, and the matrix vibration is 665 cm^{-1} with 5% substitution. The results show that the phonon energy of the matrix decreases with Ga^{3+} doping, and this is helpful for improving the UCPL of the phosphors. On the other hand, the absorption at 3460 cm^{-1} and 1635 cm^{-1} is caused by the stretching vibration of OH^- groups and the deformation vibration of HOH [23]. Figure 4 shows that the concentration of the surface OH^- groups decreased with Ga^{3+} doping, enhances the UC emission intensity, and decreases the red/green relative intensity.

3.2. $\text{GdAlO}_3: \text{Er}^{3+}, \text{Yb}^{3+}$ Phosphors Doped with Lu^{3+} . XRD patterns for all of the samples ($\text{Gd}_{0.91-y}\text{Lu}_y\text{AlO}_3: \text{Er}^{3+}_{0.01}, \text{Yb}^{3+}_{0.08}$ ($y=0.00, 0.01, 0.03, 0.05$)) with various doping concentrations of Lu^{3+} were measured as showed in Figure 5. All of the diffraction peaks can be assigned to GdAlO_3 (PDF#46-0395), and no diffraction peak is detected. However, when the substitution concentration of Lu^{3+} was increased to 10% or even 25%, the XRD spectra of the phosphors no longer match the standard

FIGURE 5: XRD patterns of $(\text{Gd}_{0.91-y}\text{Lu}_y)\text{AlO}_3: \text{Er}^{3+}_{0.01}, \text{Yb}^{3+}_{0.08}$ phosphors.

FIGURE 6: UCPL spectra of $(\text{Gd}_{0.91-y}\text{Lu}_y)\text{AlO}_3: \text{Er}^{3+}_{0.01}, \text{Yb}^{3+}_{0.08}$ phosphors.

spectra of GdAlO_3 because the radii of the ions ($r_{\text{Lu}^{3+}}=0.0861 \text{ nm}$ and $r_{\text{Gd}^{3+}}=0.1053 \text{ nm}$) are quite different. It is difficult to replace Gd^{3+} with a large amount of Lu^{3+} in the same crystal because of the 15% difference in radius according to Vegard's law [24].

Figure 6 shows the UC emission spectra of the samples $(\text{Gd}_{0.91-y}\text{Lu}_y)\text{AlO}_3: \text{Er}^{3+}_{0.01}, \text{Yb}^{3+}_{0.08}$ ($y=0.00, 0.01, 0.03, 0.05$) excited at 980 nm. In the spectra, there are three emission bands that peak at around 524, 546, and 656 nm, and the position of the emission peak did not shift significantly with the substitution of Lu^{3+} , and this same observation is made with Ga^{3+} doping. From Figure 7 it can be seen that the strength of the green emission at 546 nm of the phosphor doped with 5% Lu^{3+} is 2.73 times greater than that of the phosphor $\text{Gd}_{0.91}\text{AlO}_3: \text{Er}^{3+}_{0.01}, \text{Yb}^{3+}_{0.08}$. Also, the intensity ratio of the red/green emission decreased with Lu^{3+} doping.

Figure 8 shows the FT-IR spectra of the $(\text{Gd}_{0.91-y}\text{Lu}_y)\text{AlO}_3: \text{Er}^{3+}_{0.01}, \text{Yb}^{3+}_{0.08}$ phosphors. All of the phosphors have absorption peaks at 3460 cm^{-1} (corresponding to the stretching vibration of OH^- groups) and 1635 cm^{-1} (corresponding to the deformation vibration of HOH) and in the region less than 1000 cm^{-1} (corresponding to the vibration of the host materials). First,

FIGURE 7: UCPL intensity of $(\text{Gd}_{0.91-y}\text{Lu}_y)\text{AlO}_3: \text{Er}^{3+}_{0.01}, \text{Yb}^{3+}_{0.08}$ phosphors at 546nm (inset is the intensity ratio of red/green emission on Lu^{3+} concentration).

FIGURE 8: FT-IR spectra of $(\text{Gd}_{0.91-y}\text{Lu}_y)\text{AlO}_3: \text{Er}^{3+}_{0.01}, \text{Yb}^{3+}_{0.08}$ phosphors.

the vibration of the host material gradually shifts from 670 cm^{-1} to 663 cm^{-1} with substitution of Lu^{3+} for Al^{3+} . At the same time, the concentration of the surface OH^- groups also decreased with Lu^{3+} doping; this is because the rare earth metals Lu^{3+} , Gd^{3+} have an abundant electronic structure with their 4f electronic number being different, so that they have relatively high coordination number which can possibly obtain the coordination bonds with OH^- . This can enhance the green emission with a decrease in the red/green relative intensity.

3.3. UCPL Mechanism. The UC luminescence mechanisms for $\text{Er}^{3+}/\text{Yb}^{3+}$ -codoped systems are well understood [25, 26] and can be described using the energy level diagram illustrated in Figure 9 reproduced from Taoli Deng et al. (2018) [under the Creative Commons Attribution License/public domain]. Under excitation at 980 nm, Yb^{3+} is excited to the $^2\text{F}_{5/2}$ excited state. Meanwhile, Er^{3+} is excited from the $^4\text{I}_{15/2}$ ground state to the excited state $^4\text{I}_{11/2}$ via ET from Yb^{3+} in the $^2\text{F}_{5/2}$ state. Because the lifetime of the $^4\text{I}_{11/2}$ state of

Er^{3+} is very long, it is expected that the population of this excited state can be further excited to the upper state of $^4\text{F}_{7/2}$ through a second ET from another Yb^{3+} ion that is also in its excited state. After the fast $^4\text{F}_{7/2} \rightarrow ^2\text{H}_{11/2}$ or $^2\text{H}_{11/2} \rightarrow ^4\text{S}_{3/2}$ nonradiative relaxations, the excited Er^{3+} returns to the $^4\text{I}_{15/2}$ ground state, producing the green emissions at 524 and 546 nm that radiate from the $^2\text{H}_{11/2} \rightarrow ^4\text{I}_{15/2}$ and $^4\text{S}_{3/2} \rightarrow ^4\text{I}_{15/2}$ transitions, respectively. At the same time, some of the populations of the $^4\text{F}_{7/2}$ state relax nonradiatively to the $^4\text{F}_{9/2}$ state, from which the red emission at 659 nm is observed for the $^4\text{F}_{9/2} \rightarrow ^4\text{I}_{15/2}$ transition. Alternatively, Er^{3+} in the $^4\text{I}_{11/2}$ excited state is pumped by the first ET from Yb^{3+} and may relax to the $^4\text{I}_{13/2}$ level via nonradiative relaxations. It may then be promoted to the red-emitting $^4\text{F}_{9/2}$ level after a second ET from another Yb^{3+} , which also produces red emission at 659 nm. First, when the phonon energy of a phosphor is high, the excited states of the activated ion can be coupled with the host lattice vibrational phonon, and this leads to an increased probability of nonradiative transitions for the activated ion in excited states to return to the ground state. Thus, the UC efficiency decreases significantly. Otherwise, from the schematic diagram, it is seen that the $^4\text{I}_{11/2} \rightarrow ^4\text{I}_{13/2}$ and $^4\text{S}_{3/2} \rightarrow ^4\text{F}_{9/2}$ nonradiative transitions of Er^{3+} play a key role in determining the green emission intensity and the ratio of red-to-green emission of UCPL spectra. In our previous work [17, 19], we showed that the energy gaps of $^4\text{I}_{11/2} \rightarrow ^4\text{I}_{13/2}$ and $^4\text{S}_{3/2} \rightarrow ^4\text{F}_{9/2}$ of Er^{3+} are about 3600 cm^{-1} and 3140 cm^{-1} ; these values are close to the vibrational frequency of OH^- (3460 cm^{-1}). The decrease in the OH^- concentration in the phosphor had a marked influence on reducing the $^4\text{I}_{11/2} \rightarrow ^4\text{I}_{13/2}$ and $^4\text{S}_{3/2} \rightarrow ^4\text{F}_{9/2}$ nonradiative transitions. Consequently, the red emission at 659 nm from the $^4\text{F}_{9/2} \rightarrow ^4\text{I}_{15/2}$ transition is prevented, and the main green emission is enhanced. Therefore, the red/green emission ratio decreases with the decrease in the concentration of OH^- in the phosphors.

4. Conclusions

The $\text{GdAlO}_3:\text{Er}^{3+}, \text{Yb}^{3+}$ phosphors that have Al^{3+} and Gd^{3+} substituted by Ga^{3+} and Lu^{3+} , respectively, were prepared via coprecipitation. All of the samples had three emission band peaks at around 524, 545, and 656 nm, and the positions of the emission peaks do not shift significantly with the substitution of Ga^{3+} and Lu^{3+} . A marked improvement in the emission spectra at 546nm is observed with Ga^{3+} and Lu^{3+} doping. For the phosphors doped with 5% Ga^{3+} and Lu^{3+} , the strengths are, respectively, 2.36 and 2.73 times greater than the strength of the $\text{Gd}_{0.91}\text{AlO}_3:\text{Er}^{3+}_{0.01}, \text{Yb}^{3+}_{0.08}$ phosphor, and the intensity ratio of the red/green emission decreased with an increase in the Ga^{3+} and Lu^{3+} doping concentration. The factors that influence the improvement of the UCPL phosphors are discussed. On one hand, the crystal textures did not change but the lattice parameters changed slightly via Lu^{3+} and Ga^{3+} doping, and this resulted in a decrease in the phonon energy of the host. On the other hand, the

FIGURE 9: Schematic diagram of energy levels of Er^{3+} , Yb^{3+} ions and possible transitions under 980nm excitation. EBT: energy back transfer; ET: energy transfer; NR: nonradiative transfer.

concentration of the surface OH^- groups decreased with Lu^{3+} and Ga^{3+} doping, and this enhanced the main emission peak intensity and decreased the red/green relative intensity.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

This work was financially supported by the Youth Growth S&T Personnel Foundation of Guizhou Education Department (KY [2016]273 and KY [2017]283), the Joint Science and Technology Funds of Guizhou S&T Department, Anshun City People's Government, and Anshun University (LH [2016]7271).

Supplementary Materials

All of the $\text{GdAlO}_3:\text{Er}^{3+}, \text{Yb}^{3+}$ phosphors that have Al^{3+} substituted by Ga^{3+} have three main emission bands with maxima at 524 nm (green), 546 nm (green), 659nm (red) excited by 980 nm radiation. The green emission peaks are assigned to $^2\text{H}_{11/2} \rightarrow ^4\text{I}_{15/2}$ and $^4\text{S}_{3/2} \rightarrow ^4\text{I}_{15/2}$ transitions of Er^{3+} , and the red emission peak is ascribed to the $^4\text{F}_{9/2} \rightarrow ^4\text{I}_{15/2}$ transition of Er^{3+} . Moreover, the position of the emission peak did not shift significantly with the substitution of Ga^{3+} . Clearly, a marked improvement in the

emission spectra at 546nm is observed with Ga^{3+} doping. (*Supplementary Materials*)

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