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Yellowish green-emitting $KSrPO_4:Tb^{3+}$ phosphors with various doping concentrations prepared by using microwave assisted sintering

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Abstract

$KSr_{1-x}PO_4:xTb^{3+}$ phosphors with various concentrations ($x = 0.05$, 0.06, 0.07, 0.08) of Tb^{3+} ions were synthesized in succession by using microwave assisted sintering. The sintering condition was set at 1200 °C for 1 h in air. The microstructural and luminescent characteristics of $KSrPO_4:Tb^{3+}$ phosphors were investigated and are discussed here. The XRD result shows that the prepared $KSr_{1-x}PO_4:xTb^{3+}$ phosphors would have an impure phase as the Tb^{3+} ion increases to more than $x = 0.06$. The photoluminescence measurement shows that the series of the emission-state $^5D_4 \rightarrow {}^7F_6$, $^5D_4 \rightarrow {}^7F_4$, and $^5D_4 \rightarrow {}^7F_3$, corresponding to the typical $4f \rightarrow 4f$ intra-configuration forbidden transitions of Tb^{3+}, are appeared and the major emission peak is around at 542 nm. Moreover, the maximum photoluminescence intensity is appeared when the molar concentration of Tb^{3+} is 0.06. The decay time value of the $KSr_{1-x}PO_4:xTb^{3+}$ phosphors with $x = 0.06$ is about 0.27 ms.

Keywords: Phosphors; $KSrPO_4$; Microwave assisted sintering; Luminescence

1. Introduction

In recent years, white light-emitting diodes (LEDs) have been widely developed due to their advantages of low energy consumption, long lifetime, lack of pollutants and more [1]. The typical method to generate white light is to use a blue LED chip (460 nm) with a yellow-emitting phosphor, $Y_3Al_5O_{12}:Ce^{3+}$ (YAG). However, because of the lack of a red light component, such white light generated by this method usually obtained poor color rendering. Another way to provide white light is to use an ultraviolet (UV) LED (300–410 nm) to convert phosphors which have red, blue, and green luminescence [2]. Thus, in order to develop this technology, the phosphors with desirable color emission and high efficiency under UV excitation must be explored and developed. Recently, due to excellent thermal stability, phosphate compounds of the $ABPO_4$ (A = alkaline metals, B = alkaline earth metals) were reported and subsequently became an important family of luminescent host materials [3–7]. Moreover, phosphate compounds having the tetrahedral rigid three dimensional matrix of phosphate are desired for charge stabilization and excited under UV excitation. In the past, $ABPO_4$ based phosphors have been prepared by solid-state reaction using a conventional sintering furnace. $KSrPO_4$ phosphors having orthorhombic structures with space group Pnma and lattice constants $a \sim 7.35$ Å, $b \sim 5.56$ Å, $c \sim 9.64$ Å and $\alpha = \beta = \gamma = 90°$ were reported as good candidates for LED use The phosphors of $KSrPO_4$ doped with Eu^{2+}, Tb^{3+}, and Sm^{3+} were synthesized by solid-state reaction, and their luminescence properties were investigated [8–11]. However, the conventional sintering of phosphors usually needs high temperature to achieve the solid-state reaction and is difficult to produce fine powders, which could easily change the structural characteristics and photoluminescence properties [12]. It was known that the sintering treatment is an important factor for controlling size and crystalline [13]. Microwave assisted sintering is a novel synthesis method in the rapidly developed research field [13]. It has been reported that microwave assisted sintering consumes less energy and reduces the activation energy, thus lowering the sintering temperatures

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of the phosphors, as compared with conventional sintering treatments [14,15]. Even some phosphors prepared by microwave assisted sintering have been reported in recent years [16,17]; however, $KSrPO_4$ phosphors doped with Tb^{3+} prepared using microwave assisted sintering have not yet been reported. Therefore, an investigation of the microstructural properties and luminescent characteristics of $KSrPO_4$:Tb^{3+} phosphors prepared using microwave assisted sintering is needed.

In this paper, the $KSrPO_4$:Tb^{3+} phosphors were synthesized successively via the microwave assisted sintering. The effects of different concentrations of Tb^{3+} ions on microstructural and luminescent characteristics of $KSrPO_4$:Tb^{3+} phosphors were investigated by using X-ray diffraction (XRD), scanning electron microscopy (SEM) and photoluminescence measurement.

2. Experimental procedure

2.1. Samples preparation

KH_2PO_4, $SrCO_3$, and Tb_4O_7 powders all with a purity of 99.9% were used as the starting materials for the $KSr_{1-x}PO_4$:xTb^{3+} phosphors with different concentrations of Tb^{3+} ions ($x = 0.05, 0.06, 0.07, 0.08$). The powders were mixed with alcohol for the solvent and ball-milled for 1 h with zirconia balls. After drying, the mixed powders were sintered by using microwave assisted sintering in a microwave furnace to form the $KSr_{1-x}PO_4$:xTb^{3+} phosphors. The microwave furnace (Therm Wave Mod. III) with controllable microwave powers at 2.45 GHz up to 1.3 kW was used in this study. Due to a very strong heating response to 2.45 GHz microwaves, silicon carbide (SiC) was used as a susceptor to provide the indirect heating of the powders [18]. The material sample was placed on an Al_2O_3 crucible in a microwave cavity surrounded by four silicon carbide susceptors and encapsulated by a ceramic fiber insulating material. The mixed powders were sintered at 1200 °C for 1 h under an air atmosphere in microwave furnace with a power of 900 ± 20 W, which produced an average heating rate greater than 100 °C/min. After sintering, the samples were cooled to the room temperature and then characterized.

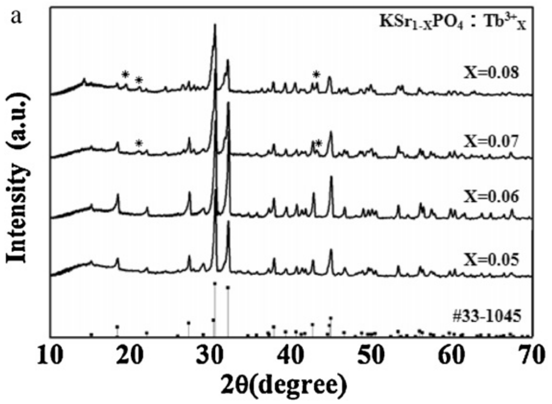
2.2. Characterization

The crystalline phases of the phosphors were identified by X-ray diffraction (XRD, Bruker D8 Advance) analysis with Cu Kα radiation of $\lambda = 1.54$ Å using a Ni filter, and with a secondary graphite monochromator so as to realize the effect of sintering process on the crystallization of the phosphors. A scan range of $2\theta = 10$–70° with a step of 0.03° and 0.4 s as a count time per-step were used. The scanning electron microscopy (SEM; HORIBA EX-200) was used to observe particle morphology of phosphors. The constituent elements of the phosphors were detected by energy dispersive spectrometer (EDS, JSM-6400F. JEOL, Japan) analysis. The excitation, emission spectra and fluorescence decay time were obtained using photoluminescence measurement (PL, JASCO FP-6000), which was equipped with a 150 W xenon lamp as the light source. In this study, in order to obtain the data accurately, specimens were measured within the same sample holder to ensure the consistent amount of phosphor materials in all samples.

3. Results and discussion

3.1. Structure

Fig. 1(a) shows the X-ray diffraction patterns of $KSr_{1-x}PO_4$:xTb^{3+} phosphors with different concentrations of Tb^{3+} ions prepared by microwave assisted sintering. The XRD result indicates that the diffraction patterns of the $KSr_{1-x}PO_4$:xTb^{3+} ($x = 0.05$ and 0.06) phosphors are consistent with the data reported for $KSrPO_4$ powder in JCPDS #33-1045. When Tb^{3+} ion concentration was further increased (x more than 0.06), the impure phase of $SrTbO_3$ was appeared and signed as symbol '*'. It is also found that all the 2θ angles of diffraction peak shifts slightly toward to the higher angle direction with increasing the Tb^{3+} ion concentration. Fig. 1(b) further shows the enlargement of the strongest diffraction peak of $KSr_{1-x}PO_4$:xTb^{3+} phosphors with different Tb^{3+} ion concentration. The result indicates the diffraction peak positions for various Tb^{3+} concentrations ($x = 0.05$, 0.06,



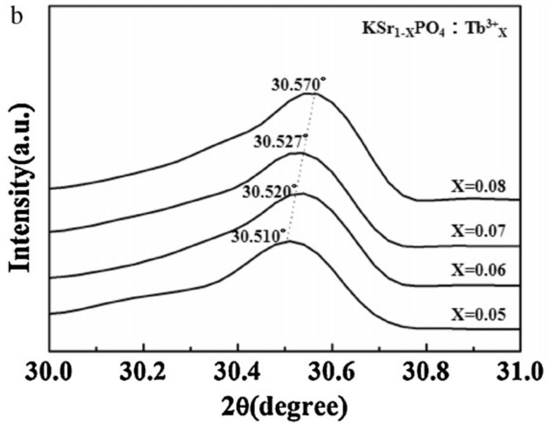


Fig. 1. (a) XRD pattern and (b) an enlargement of the strongest XRD peak diffraction peak of $KSr_{1-x}PO_4$:xTb^{3+} phosphors with different Tb^{3+} ion concentration prepared by using microwave assisted sintering.

0.07, 0.08) were appeared at 30.510°, 30.520°, 30.527° and 30.570°, respectively. From the Bragg's equation relation of interplanar spacing, miller index and cell parameters can be expressed as follows:

$$2d\sin\theta = \lambda \tag{1}$$

$$\frac{1}{d_{h\,k\,l}^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \tag{2}$$

where h, k and l are Miller indices, λ is the wavelength of Cu radiation (1.54 Å) in XRD measurement, while 2θ is the diffraction angle, determined from the XRD results. The higher angle shift of the diffraction peaks indicates the decrease in $d_{h\,k\,l}$ and lattice parameters (a, b, and c), since the ion radius of Tb^{3+} (0.92 Å) is smaller than that of Sr^{2+} (1.12 Å). Moreover, the result also indicates that Sr^{2+} ion in $KSrPO_4$ phosphate can be replaced by Tb^{3+} ion to form the solid solution when prepared by using microwave assisted sintering.

To further know the crystalline size (D) of $KSr_{1-x}PO_4{:}xTb^{3+}$ phosphors prepared by using microwave assisted sintering, the average crystalline size was determined by applying the Scherrer equation, as given by:

$$D = \frac{0.9\lambda}{\omega\cos\theta} \tag{3}$$

ω is the calibrated full width at half maximum (FWHM) of the strongest diffraction peak in radians, θ is the Bragg angle and λ is the X-ray wavelength (1.54 Å). Even the strongest diffraction peaks of $KSr_{1-x}PO_4{:}xTb^{3+}$ phosphors were shifted slightly, the result of which were crystalline sizes of all the phosphors being about 12.2 nm due to small shift variation.

4. Morphology and element analysis

Fig. 2 shows the SEM image and the EDS data of $KSr_{1-x}PO_4{:}xTb^{3+}$ phosphors with different concentrations of Tb^{3+} ion prepared by microwave assisted sintering. It was found that there are agglomerates and clusters of several particles. The particle sizes of $KSr_{1-x}PO_4{:}xTb^{3+}$ phosphors are in the range of 2–10 μm, which is much larger than the crystalline sizes around 12.2 nm of the phosphors. Namely, in a clustered particle there are many crystalline particles. In this study, it was observed that the particle sizes of the microwave-assisted sintered powders were fine and uniform, which was similar with those reported by Ishak and Daud [19]. The explanation of such is that the heat energy is generated within the material itself as materials interact with microwave power, then dispersed uniformly within the material [16]. Moreover, Lo et al. pointed out that the particle size and shape were influenced by the process parameters used for the synthesis of phosphors [20]. The particle morphology of the $KSr_{1-x}PO_4{:}xTb^{3+}$ phosphors was not much different from each other, but the particle size decreased obviously from 10 μm to 2 μm as the concentration of Tb^{3+} ion increases. The results may be that the doping ions act as nucleation, and the nucleation density of the micro crystallites increased in $KSrPO_4$ powders as Tb^{3+} ions increase. It was reported that the existence of doping impurities might affect the nucleation and growth rates, since the doping impurities usually lower the potential barrier for the formation of nuclei and reduce the growth velocities, which were possibly responsible for decrease in the particle size [14]. The composition of the $KSr_{1-x}PO_4{:}xTb^{3+}$ phosphors with different Tb^{3+} ions concentrations ($x = 0.05$, 0.06, 0.07, 0.08) was measured by EDS. The results, combined with the XRD results, confirm that Tb elements existed in $KSr_{1-x}PO_4{:}xTb^{3+}$ powder and that $KSr_{1-x}PO_4{:}xTb^{3+}$ phosphors could be synthesized in successive order by using the microwave-assisted sintering according to the existence of K, Sr, Tb, P, and O elements.

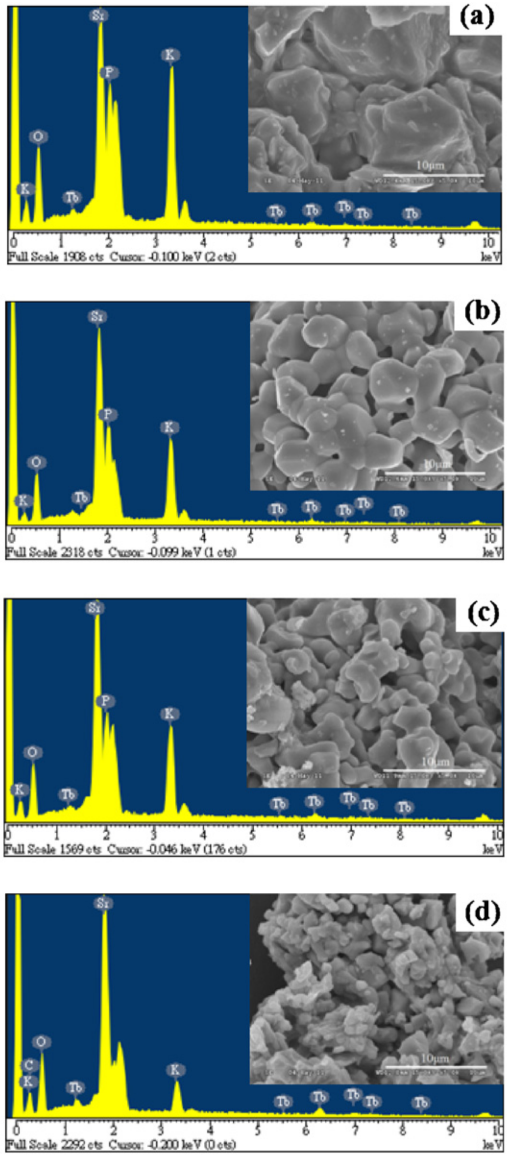


Fig. 2. SEM image and EDS data of $KSr_{1-x}PO_4{:}xTb^3$ phosphors with (a) $x = 0.05$, (b) $x = 0.06$, (c) $x = 0.07$, and (d) $x = 0.08$ prepared by using microwave assisted sintering.

5. Emission spectrum and decay time analysis

Fig. 3 shows the emission spectras of $KSr_{1-x}PO_4{:}xTb^{3+}$ phosphors with different concentrations of Tb^{3+} ions prepared by microwave assisted sintering. $KSr_{1-x}PO_4{:}xTb^{3+}$ phosphors

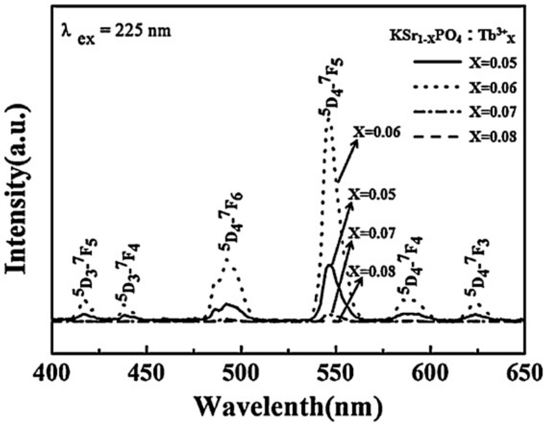


Fig. 3. Photoluminescence emission spectras of $KSr_{1-x}PO_4$:$x$$Tb^3$ phosphors with different Tb^{3+} molar concentrations prepared by microwave assisted sintering ($\lambda_{ex} = 225$ nm).

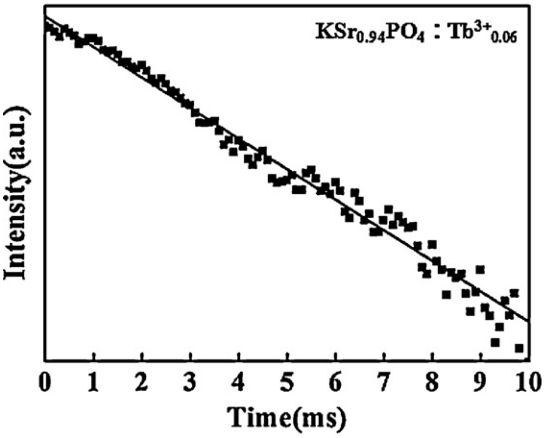


Fig. 4. Fluorescence decay time of 542 nm emission for $KSr_{1-x}PO_4$:$x$$Tb^{3+}$ phosphor with $x = 0.06$.

were excited with 225 nm wavelength from a xenon lamp. As shown in Fig. 3, all the $KSr_{1-x}PO_4$:$x$$Tb^{3+}$ phosphors emitted the light wavelength near yellowish green light, approximating a green luminescence with a peak wavelength of 542 nm. In the emission spectra, the series of the emission-state $^5D_4 \rightarrow {}^7F_6$, $^5D_4 \rightarrow {}^7F_4$, and $^5D_4 \rightarrow {}^7F_3$ corresponded to the typical 4f → 4f intra-configuration forbidden transitions of Tb^{3+} ion [21]. The major emission peak centered at 542 nm corresponds to $^5D_4 \rightarrow {}^7F_5$ transition, while $^5D_3 \rightarrow {}^7F_5$, $^5D_3 \rightarrow {}^7F_4$, $^5D_4 \rightarrow {}^7F_6$, $^5D_4 \rightarrow {}^7F_4$, and $^5D_4 \rightarrow {}^7F_3$ transitions were assigned to the emission peaks at 417, 438, 489, 588, 621 nm, respectively[9]. It was also found that the emission spectras of $KSr_{1-x}PO_4$:$x$$Tb^{3+}$ phosphors prepared by microwave assisted sintering were also comprised of two groups: the blue emissions below 450 nm from $^5D_3 \rightarrow {}^7F_5$, $^5D_3 \rightarrow {}^7F_4$ transitions of Tb^{3+} ion, and the yellowish green and red emissions above 450 nm from $^5D_4 \rightarrow {}^7F_6$, $^5D_4 \rightarrow {}^7F_5$, $^5D_4 \rightarrow {}^7F_4$, and $^5D_4 \rightarrow {}^7F_3$ transitions. Moreover, it is known that the luminescence intensity of phosphors is always dependent on the doping concentrations. In this study, the concentration quenching effect was obtained under the excitation at 225 nm. As the concentration of Tb^{3+} ion increased, the probability of the energy transfer among Tb^{3+} ions also increased. The luminescence intensity reached a maximum when the concentration of Tb^{3+} ion was $x = 0.06$, and then decreased with the increases of the Tb^{3+} concentration. Moreover, as discussed in XRD results, when the concentration of Tb^{3+} ion was further increased (x more than 0.06), the impure phase appeared, which resulted in a lower level of luminescence intensity.

Decay time measurement was also examined for enhanced luminescence spectra of $KSr_{1-x}PO_4$:$x$$Tb^{3+}$ phosphor with $x = 0.06$ prepared by microwave assisted sintering. Fig. 4 shows fluorescence decay time of 542 nm emission for $KSr_{1-x}PO_4$:$x$$Tb^{3+}$ with $x = 0.06$. The decay behavior can be expressed as [10]:

$$I = I_0 \exp\left(\frac{-t}{\tau}\right) \quad (4)$$

where I and I_0 are the luminescence intensities at time 0 and t, respectively, and τ is the decay time for exponential component. According to this equation, the decay time value calculated from the fitted curve is about 0.27 ms for the $KSr_{1-x}PO_4$:$x$$Tb^{3+}$ phosphor with $x = 0.06$. The decay time of electric-dipole allowed transition of f–d is usually not longer than 1 μs [22]. On the contrary, the decay time of Tb^{3+} ($^5D_4$) in this study is in the range of milliseconds due to the forbidden nature of the f–f transition [23]. Moreover, it is found that the decay time for the $KSr_{1-x}PO_4$:$x$$Tb^{3+}$ phosphor with $x = 0.06$ is relatively shorter as compared to the studies made from Park et al. [24]. In their research, they found that the decay time value of $Y_3Al_5O_{12}$: Tb phosphor was 4.2 ms. We also found that the optimum doping concentration of Tb^{3+} in $KSr_{1-x}PO_4$:$x$$Tb^{3+}$ phosphor prepared by using microwave-assisted sintering is inconsistent with that of $x = 0.07$ in $KSr_{1-x}PO_4$:$x$$Tb^{3+}$ phosphor prepared by using conventional sintering reported by Lin et al. [9]. The result indicates that different heating mechanism of phosphors would affect the optimum doping concentration in phosphor.

6. Conclusion

In summary, we have synthesized $KSr_{1-x}PO_4$:$x$$Tb^{3+}$ phosphors with various concentrations of Tb^{3+} ions ($x = 0.05$, 0.06, 0.07, 0.08) in successive order by using microwave assisted sintering at 1200 °C for 1 h. The relations between the microstructure and luminescent characteristics of $KSrPO_4$:Tb^{3+} phosphors were investigated and are discussed below. The XRD results showed that the prepared $KSr_{1-x}PO_4$:$x$$Tb^{3+}$ phosphors formed a solid solution and had an appearance of impure phase as Tb^{3+} ions increased to more than $x = 0.06$ by using the proposed microwave assisted sintering. The SEM images showed that the particle sizes of the prepared $KSr_{1-x}PO_4$:$x$$Tb^{3+}$ phosphors obviously decreased as the concentrations of Tb^{3+} ions increased. The major emission peak centering at 542 nm corresponds to $^5D_4 \rightarrow {}^7F_5$ transition, while $^5D_3 \rightarrow {}^7F_5$, $^5D_3 \rightarrow {}^7F_4$, $^5D_4 \rightarrow {}^7F_6$, $^5D_4 \rightarrow {}^7F_4$, and $^5D_4 \rightarrow {}^7F_3$ transitions can be assigned to the emission peaks at 417, 438, 489, 588, 621 nm, respectively. Moreover, it is found that the maximum photoluminescence intensity of $KSr_{1-x}PO_4{:}xTb^{3+}$ phosphors prepared by using microwave assisted sintering appeared when the molar concentration of Tb^{3+} was 0.06, and the decay time was about 0.27 ms since the decay time of Tb^{3+} ($^5D_4$) was in the range of milliseconds due to the forbidden nature of the f–f transition.

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