



Preparation and photoluminescence properties of YAG:Ce³⁺ phosphors by a series of amines assisted co-precipitation method



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ABSTRACT

In this work, YAG:Ce³⁺ phosphor powders were firstly prepared by a co-precipitation method, in which a mixed solution according to stoichiometric molar amounts of aluminum, yttrium and cerium nitrates were co-precipitated using different precipitants. Pure YAG phase were obtained at 800 °C, which is lower than those applied for preparing YAG:Ce³⁺ phosphors by co-precipitation method using ammonia water (1000 °C) and ammonium hydrogen carbonate (900 °C) as precipitants. The structural, morphological and optical properties of YAG:Ce³⁺ were investigated by X-ray diffraction (XRD), FT-IR spectroscopy, the thermogravimetry-differential scanning calorimetry (TG-DSC), scanning electron microscopy (SEM) and photoluminescent spectra measurements at ambient temperature. The results show the sintering temperature, the solvent volume ratio of ethanol/water and dosage of triethylenetetramine have strongly effects on the structural, morphology, and optical properties of YAG:Ce³⁺ phosphor. Meanwhile, diethylenetriamine (DETA) and tetraethylenepentamine (TEPA) have been also used as precipitants, the properties of these samples are comparable to that of the sample by using triethylenetetramine as precipitant. Meanwhile, 1 mol% of Ce³⁺ has been also proved the optimal doped concentration for the PL emission intensity of samples. Thus diethylenetriamine, triethylenetetramine and tetraethylenepentamine might be valuable precipitants for the preparation of yttrium aluminum garnet based materials.

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1. Introduction

Recently, phosphor-converted white light-emitting diodes (pc-WLEDs) have been extensively investigated for applications in solid-state lightings and display systems. Among them, trivalent cerium-doped yttrium aluminum garnet (Y₃Al₅O₁₂:Ce³⁺, YAG:Ce) has been regarded as the most commonly available yellow phosphor in commercially white LEDs due to its high luminescent efficiency and chemical stability. YAG:Ce phosphor can convert the blue light from blue InGaN LEDs in the range of 440–460 nm into a broad band yellow emission, then the combination of the yellow emission of YAG:Ce and the residual blue light that escapes through

the phosphor generates white light [1–3].

Conventionally, YAG:Ce phosphor is mainly prepared by solid-state reaction, in which Y₂O₃ and Al₂O₃ were used as starting materials with repeated extensive milling and high sintering temperature (about 1600 °C) to eliminate several intermediate phases, such as YAM and YAP, then the desired optical properties can be obtained [4,5]. In view of the disadvantage of the solid-state method, lots of wet chemical methods including co-precipitation method [6], sol–gel method [7], spray-pyrolysis [8–11] and solvothermal method [12–15], have been widely developed for low temperature production of pure YAG phase. YAG based materials have been prepared by precipitation method using different precipitants such as ammonium hydrogen carbonate, ammonia water [6], urea [16,17], oxalate [18] etc.

In this paper, we produced YAG:Ce phosphor via co-precipitation method using triethylenetetramine as a novel

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Table 1
All experimental conditions.

Sample	X	Temperature/°C	The volume of Precipitator/mL	Ethanol/water	Precipitator
S1	0.03	750	2.4	pure ethanol	triethylenetetramine
S2	0.03	800	2.4	pure ethanol	triethylenetetramine
S3	0.03	850	2.4	pure ethanol	triethylenetetramine
S4	0.03	900	2.4	pure ethanol	triethylenetetramine
S5	0.03	1000	2.4	pure ethanol	triethylenetetramine
S6	0.03	1100	2.4	pure ethanol	triethylenetetramine
S7	0.03	1200	2.4	pure ethanol	triethylenetetramine
S8	0.03	1300	2.4	pure ethanol	triethylenetetramine
S9	0.03	1400	2.4	pure ethanol	triethylenetetramine
S10	0.03	1100	0.6	pure ethanol	triethylenetetramine
S11	0.03	1100	1.0	pure ethanol	triethylenetetramine
S12	0.03	1100	1.8	pure ethanol	triethylenetetramine
S13	0.03	1100	2.4	pure ethanol	triethylenetetramine
S14	0.03	1100	2.4	10/5	triethylenetetramine
S15	0.03	1100	2.4	7.5/7.5	triethylenetetramine
S16	0.03	1100	2.4	5/10	triethylenetetramine
S17	0.03	1100	2.4	pure water	triethylenetetramine
S17	0.03	1300	2.4	pure water	diethylenetriamine
S17	0.03	1300	2.4	pure water	tetraethylenepentamine

precipitant. The effect of different sintering temperatures, ethanol/water ratio, dosage of triethylenetetramine on structural, morphological and optical properties of YAG:Ce phosphors were comparatively investigated. Meanwhile, diethylenetriamine and tetraethylenepentamine have been also used as precipitants, the properties of these samples are comparable to that of sample where triethylenetetramine were used as a precipitant. In summary, diethylenetriamine, triethylenetetramine and tetraethylenepentamine might be valuable precipitants for the preparation of yttrium aluminum garnet based materials.

2. Experimental section

2.1. Preparation of samples

The starting materials include yttrium (III) nitrate hexahydrate ($\text{Y}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, 99.5%), aluminum nitrate nonahydrate ($\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, 99%), cerium nitrate hexahydrate ($\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, 99%), diethylenetriamine (95%), triethylenetetramine (95%), tetraethylenepentamine triethylenetetramine (95%) and absolute ethanol. These chemicals were used without further purification. Aqueous

solutions were prepared using distilled water.

First of all, stoichiometric molar amounts of $\text{Y}_3\text{Al}_5\text{O}_{12}:\text{Ce}$ were added into 15 mL solvent (different ethanol/water ratio). The Y^{3+} ion concentration was about 0.1 mol/L, and the Al^{3+} ion concentration was about 0.06 mol/L. After magnetic stirring for 30 min at room temperature, different volumes of triethylenetetramine between 0.3 and 2.4 mL were added into the above mother salt solution under magnetic stirring for 8 h at room temperature to form the precursor solution perfectly. Then the precursor precipitate was filtered and washed repeatedly with deionized water and pure ethanol for three times and dried for 24 h at 80 °C. After finely milled in an agate mortar, the precursor powders were calcined at 750–1400 °C with a heating rate of 10 °C/min for 3 h in a muffle furnace in air. Finally, the furnace was cooled to room temperature naturally, and yellow YAG:Ce³⁺ phosphor powders were obtained.

For comparison, the sample where diethylenetriamine and tetraethylenepentamine were used as precipitants had also been prepared via the same procedure Table 1.

2.2. Characterizations

The crystalline phases were characterized by powder X-ray diffractometer (XRD, Rigaku, D/max 2200) using Cu K α radiation from 10° to 70° at a speed of 8°/min. Fourier transform infrared (FT-

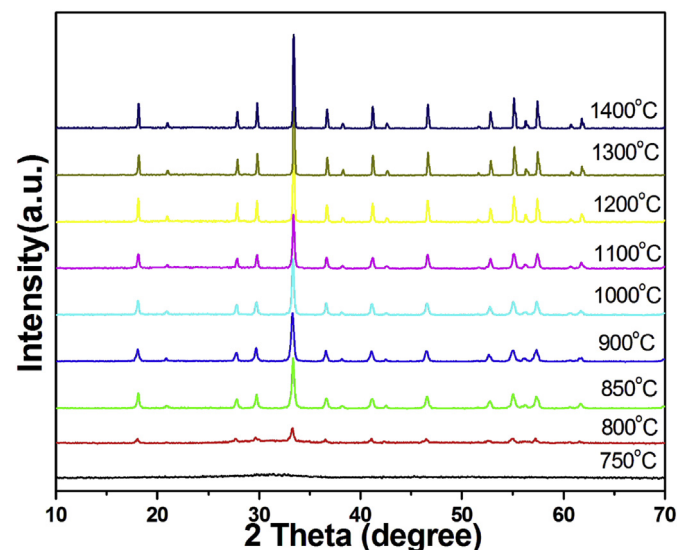


Fig. 1. XRD patterns of YAG:Ce³⁺ phosphors synthesized by using triethylenetetramine as precipitant at different temperatures for 3 h.

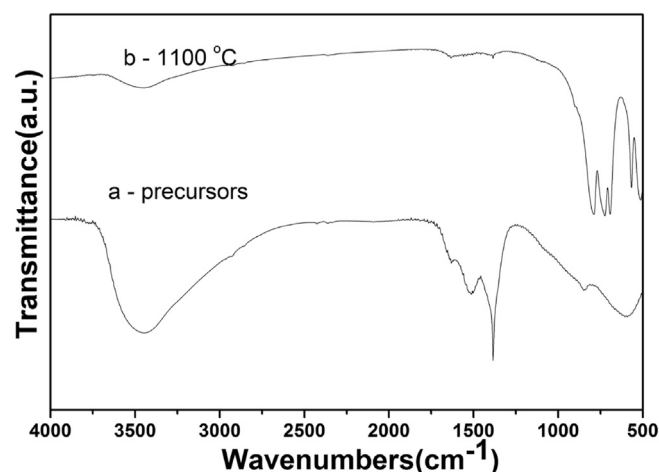


Fig. 2. FT-IR spectra of precursors (a) and YAG:Ce³⁺ NPs prepared at 1100 °C for 3 h (b).

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