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Crystal growth and photoluminescence of europium-doped strontium titanate prepared by a microwave hydrothermal method

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Abstract

This article reports that europium-doped strontium titanate ($\text{SrTiO}_3:\text{Eu}^{3+}$) was successfully synthesized using a co-precipitation method at room temperature with processing in a microwave-hydrothermal system at 140 °C for 30 min. Phase composition and structure were examined using X-Ray Diffraction, and Fourier-Transform Raman spectroscopy, revealing a cubic structure with a $Pm\bar{3}m$ space group. The optical properties were investigated by ultraviolet-visible absorption and photoluminescence, which showed that red emissions originate from Eu^{3+} transitions. Field emission scanning electron microscopy revealed spherical-like Eu^{3+} doped SrTiO_3 nanoparticles.

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1. Introduction

SrTiO_3 compounds are members of an important inorganic materials family with a distinctive perovskite-type structure and have been investigated because of their unique structure, good chemical and physical stability, strong visible luminescence, and excellent optical properties [1,2]. Such characteristics can be used in applications, such as optoelectronic devices, gas sensors, photocatalysts, or photoelectrodes, to improve efficiency [3,4]. Moreover, structural changes can result in SrTiO_3 having diverse physical and chemical properties. Some new optical properties of this material can be obtained by doping it with rare earth (RE) ions [5,6]. Nanoparticles of inorganic compounds activated by RE ions have received much attention due to their broad applicability and potential use in technology [7,8]. New matrices are

desperately needed for doping to enrich the categories of products and develop a more common and comprehensive theory [9]. Eu^{3+} ions are important because of their potential application as red phosphors, in optical amplifiers, in electro-luminescent devices, and in lasers [10,11]. Among all the RE ions, Eu^{3+} is usually employed as a red emitting center because of its unique $4f^6$ configuration that can be effectively activated by ultraviolet rays or cathode rays and emits high purity red light [12].

Preparation techniques that have been proposed to fabricate SrTiO_3 include solid-state reaction procedures [13] and polymeric precursor methods [14]. However, these synthesis routes result in agglomeration of particles and require a high synthesis temperature and large amounts of energy. In addition, they produce a poor morphology of phosphor particles and an uneven distribution of particle size at a low production rate with long preparation cycles. Consequently, it is very important to develop economic and simple synthesis methods for titanate materials. The microwave hydrothermal (MH) method has drawn tremendous attention owing to its

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advantages, such as low reaction temperature, energy economy, well-defined product morphology, and energy efficiency, because power is only applied within the reactive mixture. The microwave-assisted processing is fast, clean, simple, and often more energetically efficient than conventional heating [15,16].

This article reports a successful synthesis of SrTiO₃ doped with 1 mol% of Eu³⁺ crystal phosphors by the MH method. SrTiO₃ was chosen as the host, because it has attractive chemical and thermal stability properties.

2. Experimental details

2.1. Synthesis and MH processing of SrTiO₃:Eu crystals

SrTiO₃ doped with 1 mol% of Eu³⁺ powders were synthesized by co-precipitation without surfactants in aqueous solutions. Ti(OC₃H₇)₄ (99.99%, Aldrich), SrCl₂·2H₂O (99.9%, Merck), and KOH (99%, Merck) were used as starting materials. First, 0.01 mol of Ti(OC₃H₇)₄ was slowly added to 25 mL of deionized water while stirring. Similarly, 0.01 mol of SrCl₂·2H₂O was dissolved in 25 mL of deionized water, separately, with constant stirring. Eu(NO₃)₃·5H₂O (1 mol% Eu³⁺ with respect to Sr²⁺, 99.99%, Aldrich) was added to this reaction mixture. KOH was used as a mineralizer agent. The mixture containing all the ions was transferred to a Teflon autoclave with 100 mL capacity (80% filled), sealed, and placed in the MH system using 2.45-GHz microwave radiation with a maximum power of 800 W. The reaction mixture was heated at 140 °C for 30 min. This was followed by natural cooling of the autoclave to room temperature. The product was washed several times until a neutral pH was obtained and dried at 70 °C for 6 h.

2.2. Characterization of SrTiO₃:Eu³⁺ crystals

The obtained crystals were structurally characterized from X-ray powder diffraction (XRD) patterns using a Shimadzu-XRD-6000 (Japan) with Cu-K α radiation ($\lambda=1.5406$ Å) in the 2θ range from 10° to 70° with a scanning velocity of 2°/min in normal routine scanning. FT-Raman spectroscopy was performed with a Bruker-RFS 100 (Germany). The Raman spectra were obtained using a 1064-nm line with a Nd:YAG laser, while keeping its maximum output power at 100 mW, in the range from 50 to 1000 cm⁻¹. The morphologies of SrTiO₃:Eu³⁺ crystals were observed by field emission scanning electron microscopy (FE-SEM) through a Carl Zeiss, model Supra 35-VP (Germany) operated at 6 kV. Ultraviolet-visible (UV-vis) diffuse reflectance spectra were produced using a Varian spectrophotometer model Cary 5G (USA) in diffuse reflectance mode. Photoluminescence (PL) measurements were performed using a Jobin Yvon-Fluorolog spectrofluorometer under continuous Xe lamp (450 W) excitation at room temperature ($\lambda=393$ nm).

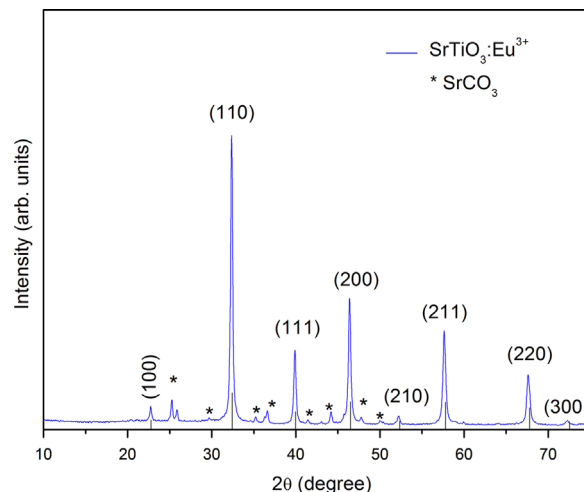


Fig. 1. XRD patterns of SrTiO₃:Eu³⁺ processed in a microwave-assisted hydrothermal method at 140 °C for 30 min.

3. Results and discussion

3.1. XRD patterns

The XRD patterns of the SrTiO₃ obtained by the co-precipitation method and processed in an MH system are shown in Fig. 1. The diffraction peaks match the standard data of a cubic phase with a *Pm3m* space group, according to the Joint Committee for Powder Diffractions Standards, JCPDS Card N° 35-0734 as indicated. Some traces of additional peaks correspond to impurity phases (marked by *), which are attributed to the SrCO₃ phase and are probably due the existence of strontium vacancies in the SrTiO₃ structure. The SrCO₃ phase corresponds to the *Pmcn* orthorhombic structure indexed by JCPDS card N° 05-0418.

3.2. FT-Raman spectroscopy analysis

Raman spectroscopy has been used to study the structure and symmetry in solids as well as phase transitions in different perovskites [17]. The phenomenon of inelastic light scattering is generally used to investigate the behavioral changes in the local symmetry of ceramics. Fig. 2 illustrates the Raman spectrum for the sample of SrTiO₃:Eu prepared by co-precipitation and processed using an MH method. Six Raman-active modes were observed in the range of 150–1100 cm⁻¹ that were assigned to the cubic structure. Assignments of Raman active modes for the europium-doped- SrTiO₃ structure are shown in Table 1. The information in the table shows an excellent match between the Raman shift of peaks at 177, 264, 545, 736, 800, and 1069 cm⁻¹ and the frequencies of TO₂, TO₃, TO₄, TO, LO₄, and SrCO₃ phonons, respectively. These results agree with those of Moreira et al. [18] who prepared pure strontium titanate nanospheres. The purpose was to perform a joint experimental analysis and first-principle calculations on MH synthesis of ST nanospheres. According to the XRD analysis, the Raman spectrum (Fig. 2) displays a peak at 1069 cm⁻¹ that corresponds to SrCO₃. Similar

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