Cathodoluminescence Studies and Characterization of Yb Doped Y₂O₃ Synthesized by Wet Chemical Method

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Abstract

Yb:Y₂O₃ (8 % Yb doped) powders were synthesized by sol-gel method is a wet chemical technique. Produced powders were characterized by scanning electron microscopy (SEM). Structural investigation of powders was conducted by X-ray diffractometer, employing Cu-Kα radiation and crystallite size of powders was evaluated from X-ray diffraction peaks broadening by modified Debye Scherer (MDS) method. Moreover, Raman analysis of powder was recorded at room temperature. Cathodoluminescence (CL) studies of ytterbium (Yb) doped yttrium oxide (Y₂O₃) powders at varied excitation voltage were performed by using single photon counters. Yb doped Y₂O₃ powders exhibit cathodoluminescence properties at higher than 10 kV excitation voltage. As expected, it was observed that the increment in the initial excitation voltage leads to the increase of luminescence efficiency.

1. Introduction

Yttrium oxide and derived compounds have taken attention, especially in optoelectronic applications. Yttrium oxide is a very promising candidate in optical application due to large energy band gap (~ 5.5 eV), low phonon energy (~ 600 cm⁻¹) and broad transmittance (230-8000 nm) of Y₂O₃ [1–5]. Luminescent materials are attracted to many fields such as displays, bioanalysis and telecommunication in various forms such as nanorods, nanowires, nanotubes, nanoparticles and bulk [2,6–11]. There are various types of luminescence such as stimulation with electrons (called cathodoluminescence), stimulation with a photon (called photoluminescence), stimulation with an electric field (called electroluminescence). Cathodoluminescence studies are confronted in a variety of areas such as biology, solid state physics, ceramic etc. Recently, ytterbium doped yttrium oxide is used as a laser source and in cathodoluminescence applications. In Y₂O₃ cubic elementary cell, Y³⁺ ion situates in 24 Noncentrosymmetric (C2) and 8 centrosymmetric (C3i) sites. Noncentrosymmetric (C2) sites are of import for luminescence properties because of having an inversion center. The Yb³⁺ ion gets into Y₂O₃ substitute for C2 or C3i sites of Y³⁺. Charge transfer bands are changed depending on Yb³⁺ ion situated C2 or C3i site. As ground state ⁵F₇/₂ and excited state ⁷F₅/₂, Yb³⁺ ion has two manifolds. Moreover, charge transfer is occupied in ultraviolet 340–350 nm (CT→⁵F₇/₂) and visible regions 480–500 nm (CT→⁷F₅/₂) [12]. Various methods have been used to synthesize rare earth (Yb, Er, Eu, Tb, Tm) doped Y₂O₃ powders such as combustion, spray pyrolysis, sol-gel, hydrothermal precipitation [6,13–17]. However, size and morphology of particles can be adjusted by sol-gel process is very cheap and low temperature technique. Furthermore, it is possible to synthesize various morphology particles. Luminescence behavior is affected by properties of doped materials, surface area, particle size and morphology. Moreover, it is worth to emphasize that microstructure has an impact upon luminescence behavior of materials.

In our present study, it was reported that 8 % Yb doped Y₂O₃ powders were synthesized by sol-gel process. Synthesized powders characterized by X-ray diffraction analysis, Raman and cathodoluminescence analysis. Based on the X-ray peak broadening, the crystallite size of powders was calculated by modified Debye Scherer method.

2. Experimental studies

2.1 Synthesis of particles

Yb:Y₂O₃ (8 % Yb doped) powders were prepared by wet chemical method. Y(NO₃)₃.6H₂O and Yb(NO₃)₃.xH₂O were dissolved in deionized water. Subsequently, citric acid and ammonia solution (NH₄OH) were added to the solution in determined amounts. The prepared solution was stirred by using a magnetic stirrer for 2 hours at room temperature. After precipitation, the solution was filtered and dried. After drying, powders were calcined at
800°C for 3 hours and finally cooled to room temperature. Schematic drawing of the process was given in Figure 1.

![Schematic drawing of wet chemical process for producing Yb doped Y₂O₃](image)

**Figure 1.** Schematic drawing of wet chemical process for producing Yb doped Y₂O₃

### 2.2 Characterization of particles

X-ray diffraction patterns were obtained for determination of crystal structure and crystallite size. Chemical compositions of particles were analyzed by energy dispersive spectroscopy (EDS). Morphology of the samples was investigated by field emission scanning electron microscopy (FE-SEM, JEOL JSM 700F). The cathodoluminescence properties of Yb doped Y₂O₃ particles were studies by using single photon counters. Using X-ray peak broadening, crystallite size was calculated by modified Debby Scherer (MDS) method.

### 3. Results and Discussion

X-ray diffraction pattern of Yb doped Y₂O₃ was given in Figure 2. The peaks appearing at 2θ range of 20.55°, 29.22°, 33.87°, 43.59°, 48.65°, 57.77° can be attributed to the (211), (222), (400), (134), (440) and (622) planes and crystalline structures corresponding to pure Yb doped Y₂O₃ powders. All diffraction peaks of Yb doped Y₂O₃ are attributed to C-type bixbyite structure space group la-3 (206) compatible with standard JCPDS data with card number (#01-087-2369). The narrow and sharp peaks indicate that synthesized powders are highly crystalline.

![X-Ray diffraction analysis of Yb doped Y₂O₃](image)

**Figure 2.** X-Ray diffraction analysis of Yb doped Y₂O₃

In Debye-Scherrer method, crystallite sizes, which is calculated from each diffraction peak in the pattern, should have the same value. However, crystallite size values cannot be obtained same value from each diffraction peak due to systematic error. The least square method was applied to eliminate the error and Debye-Scherrer Equation (1) can be reformulated by this way. Debye Scherer Equation was given in the following relation (1)

\[
\lambda = \frac{k \lambda D}{\theta} \tag{1}
\]

When both sides logarithm is taken, Equation (2) can be achieved:

\[
\ln \beta = \ln k \lambda D + \ln(1/\cos \theta) \tag{2}
\]

Where β is the peak broadening, k is the shape factor, λ is the wavelength of Cu-Kα radiation (λ=0.154051 nm), D is the crystallite size of synthesized particles and θ is the Bragg angle. Graphs of ln(β) versus ln(1/cosθ) of Yb doped Y₂O₃ particles were drawn in Figure 3. Crystallite size was estimated from the y-intercept of the fitted line in the Figure 3.
Crystallite size of Yb doped \( \text{Y}_2\text{O}_3 \) was calculated as 19.90 nm by modified Debye Scherrer method. Chemical compositions of powders were analyzed by energy dispersive spectroscopy (EDS). Morphology of the samples was investigated by field emission scanning electron microscopy. SEM and EDS analyses were exhibited in Figure 4.

The synthesized powders had sponge morphology and ytterbium was detected in spectrum by EDS analysis. Figure 5. illustrates the room temperature Raman spectra of Yb doped \( \text{Y}_2\text{O}_3 \) particles. Several raman peaks are observed for both samples. These peaks have been attributed to the fundamental vibrational modes corresponding to the cubic \( \text{Y}_2\text{O}_3 \). The most intense band located at 378 cm\(^{-1}\) is characteristic of \( \text{Y}_2\text{O}_3 \) cubic structure. Graphs of cathodoluminescence measurements of Yb doped \( \text{Y}_2\text{O}_3 \) powders at various excitation voltage were given in Figure 6.

Although no change was observed when the Yb doped \( \text{Y}_2\text{O}_3 \) powders were excited with 10 kV. After the 20 kV excitation value, the change in the CL values was started to be observed and as the kV value increased, the count values continued to increase.

4. Conclusion

\% 8 Yb doped \( \text{Y}_2\text{O}_3 \) powders were synthesized by wet chemical method. The synthesized powders were characterized by scanning electron microscopy, X-ray diffraction analysis and Raman analysis. All the synthesized Yb doped \( \text{Y}_2\text{O}_3 \) particle have C-type bixbyite structure with polycrystalline behaviour. It was proved that Yb doped \( \text{Y}_2\text{O}_3 \) exhibits cathodoluminescence properties above 20 kV excitation voltage.

References


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