Microstructural Investigation of Nanostructured Er-Doped Yttrium Oxide Powders

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Abstract

In this study, 5 mole% erbium doped Y₂O₃ powders were synthesized via sol-gel method. The powders were heat-treated at different temperatures for various time and the effects of the heat treatment conditions on the lattice parameters, crystallite size and lattice strain were investigated. The heat-treated powders were analyzed by X-ray diffraction technique (XRD) using Cu Kα radiation. The lattice parameter of as-prepared powders was calculated by Cohen-Wagner (C-W) method. Moreover, crystallite size and lattice strain of the samples were calculated using X-ray peak broadening by Williamson-Hall (W-H) combined with uniform deformation model. The produced powders were also characterized by Fourier-transform infrared spectroscopy (FT-IR).

1. Introduction

In recent years, rare element doped sesquioxides (Y₂O₃, Lu₂O₃, Sc₂O₃, La₂O₃, In₂O₃) have been extensively studied and are well known as excellent laser host materials (1, 2). Yttrium oxide (Y₂O₃) is one of the promising hosts among the oxide materials for rare earth ions such as erbium, samarium, gadolinium and europium [3]. And also, it has high refractive index, high melting point (2430 °C), high chemical and thermal stability, low phonon energy (380 cm⁻¹), broad transmittance (230-8000 nm) [4, 5]. Er doped Y₂O₃ materials can be synthesized by several methods which are sol-gel [6], solution combustion reaction [7], laser ablation [8], atomic layer deposition [9], pulsed laser deposition [10], metal-organic and chemical vapour deposition [11] etc. The sol-gel method is more suitable and cheaper than the other methods. It is carried out at low processing temperature and requires simple equipments. Although there are many studies on optical characterization and synthesis of Er doped Y₂O₃ in the literature, there are not much studies for the crystallite size effect on the properties of these powders.

In the present study, various sol-gel heat-treatment conditions were examined to produce 5% Er doped Y₂O₃ powders. A detailed structural investigation was carried out on crystallite size and lattice strain values by altering the calcination temperature and time.

2. Experimental studies

2.1 Powder preparation

Y(NO₃)₃.6H₂O and Er(NO₃)₃.5H₂O were used as starting chemicals to synthesize 5 mole % erbium doped Y₂O₃ powders in the sol-gel reactions. The starting chemicals were dissolved in deionized water. Subsequently, chelating and precipitating agents were added to the solution. The solution was stirred by using a magnetic stirrer for 2h at room temperature and then, the solution was filtered. The powders were dried at ambient condition. After drying, powders were calcined at different temperatures and times. The sample codes and the calcination parameters were given in Table 1.

<table>
<thead>
<tr>
<th>Calcination parameters</th>
<th>Sample codes</th>
</tr>
</thead>
<tbody>
<tr>
<td>temperature (°C)</td>
<td>time (hour)</td>
</tr>
<tr>
<td>800</td>
<td>3</td>
</tr>
<tr>
<td>5</td>
<td>ErY8003</td>
</tr>
<tr>
<td>1000</td>
<td>3</td>
</tr>
<tr>
<td>ErY10003</td>
<td></td>
</tr>
<tr>
<td>1100</td>
<td>3</td>
</tr>
<tr>
<td>ErY11003</td>
<td></td>
</tr>
</tbody>
</table>

2.2 Characterization of powders

XRD analysis was conducted using Cu Kα radiation to determine the phases. The X-ray peak broadenings were used to calculate the crystallite size and lattice parameter.
values of the powders by the W-H and C-W methods, respectively. FT-IR spectra of the samples were recorded in the range of 4000–400 cm⁻¹.

3. Results and Discussion

3.1. X-ray Diffraction Analyses

3.1.1 Effect of calcination temperature

X-ray diffraction patterns of 5% Er doped Y₂O₃ calcined at different calcination temperatures were shown in Figure 1.

![Figure 1. X-ray diffraction patterns of ErY8003, ErY10003 and ErY11003 samples.](image)

The XRD pattern in Figure 1 showed that samples were the standard cubic phase of Er doped Y₂O₃. The diffraction peaks of ErY8003 at 2θ=28.78, 33.40, 48.15, 57.23 were attributed to (222), (400), (440), (622) planes (Figure 1). Also, the diffraction peak width of the samples decreased with increasing calcination temperature.

The effect of calcination temperature and calcination time on the crystallite size were investigated by using Williamson Hall method integrated with uniform deformation model (UDM). The crystallite size of powders calcined at various temperatures was evaluated using the X-ray peak broadening.

The crystallite size and lattice strain were determined by Williamson Hall (W-H) method. First, instrumental peak broadening was eliminated to calculate the exact crystallite size value. The individual contribution of crystallite size (β₀) and lattice strain (βₛ) to total peak broadening are described in given Equation (1);

\[
\beta_{hkl} = \beta_D + \beta_s
\]  

(1)

The strain-caused broadening originating from crystal distortion was given in Equation (2)

\[
\varepsilon \approx \beta_s / \tan \theta
\]  

(2)

Debye-Scherrer equation and Equation (2) are combined together to yield following relations;

\[
D = \frac{k\lambda}{\beta_D \cos \theta}
\]  

(3)

\[
\beta_{hkl} = \left( \frac{k\lambda}{D \cos \theta} \right) + (4\varepsilon \tan \theta)
\]  

(4)

\[
\beta_{hkl} \cos \theta = \left( \frac{k\lambda}{D} \right) + (4\varepsilon \sin \theta)
\]  

(5)

Where \(\beta_{hkl}\) is the total 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 powders, ε is the lattice strain.

Results of the W-H analyses were given in Figure 2.

![Figure 2. Williamson-Hall analysis integrated with UDM of (a) ErY8003, (b) ErY10003, (c) ErY11003 samples.](image)
Wagner analysis of powders calcined at 800°C for 3 hours was given in Figure 3.

![Figure 3. C-W plot of ErY8003 sample.](image1)

When the temperature increased from 800 °C to 1100 °C, the crystallite size of 5% Er doped Y₂O₃ increased gradually due to the lattice expansion. Moreover, the lattice parameter of these samples also increased as shown in Table 2.

### Table 2. Crystallite size, lattice strain and lattice parameter of ErY8003, ErY10003 and ErY11003 samples.

<table>
<thead>
<tr>
<th>Sample codes</th>
<th>Crystallite size (nm)</th>
<th>Lattice strain (°A)</th>
<th>Lattice parameter (°A)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ErY8003</td>
<td>15.25</td>
<td>-0.0006256</td>
<td>10.616</td>
</tr>
<tr>
<td>ErY10003</td>
<td>27.90</td>
<td>-0.0002650</td>
<td>10.701</td>
</tr>
<tr>
<td>ErY11003</td>
<td>38.29</td>
<td>-0.0002676</td>
<td>10.754</td>
</tr>
</tbody>
</table>

#### 3.1.2 Effect of calcination time

In order to further study the structures of the samples calcined different time was also characterized by XRD as given in Figure 4.

![Figure 4. X-ray diffraction analyses of ErY8003, ErY8005 and ErY8007 samples.](image2)

The diffraction peaks at 2θ = 28.78, 33.40, 48.15, 57.23 were associated to (222), (400), (440), (622) planes of body centered cubic phase of yttrium oxide (JCPDS card no: 01-086-1107). As expected, the peak broadening was found to be decreases with increasing calcination time. In order to investigate the effect of calcination time, the crystallite sizes and lattice parameters of the samples were calculated by W-H and C-W, respectively. The W-H analyses of the samples calcined at different calcination time were exhibited in Figure 5.

![Figure 5. Williamson-Hall analysis integrated with UDM of (a) ErY8005, (b) ErY8007 samples.](image3)

### Table 3. Crystallite size, lattice strain and lattice parameter of ErY8003, ErY8005 and ErY8007 samples.

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<tr>
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</thead>
<tbody>
<tr>
<td>ErY8003</td>
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<td>-0.0006256</td>
<td>10.616</td>
</tr>
<tr>
<td>ErY8005</td>
<td>21.52</td>
<td>0.0007487</td>
<td>10.656</td>
</tr>
<tr>
<td>ErY8007</td>
<td>23.72</td>
<td>0.0007702</td>
<td>10.715</td>
</tr>
</tbody>
</table>
3.2 Fourier-Transform Infrared Spectroscopy Analysis

FT-IR spectrum of the ErY8003 sample was shown in Figure 6.

![Figure 6. FT-IR spectra of ErY8003 sample.](image)

It was observed that the characteristic vibrational modes of the Y-O bonds were at 463 and 557 cm\(^{-1}\) and broad band at around 1404 cm\(^{-1}\) was assigned to N-O bonds. Additionally, the broad band at around 1507 cm\(^{-1}\) indicated hydroxyl group (O-H) [12].

5. Conclusions

In this study, 5 moles % erbium doped Y\(_2\)O\(_3\) powders were synthesized by a cost-effective sol-gel method. The powders were heat-treated at varied temperatures for the different times. The influence of heat treatment conditions on lattice parameter, crystallite size and lattice strain were investigated by Williamson-Hall analysis integrated with uniform deformation model and Cohen Wagner method. Along with microstructural analysis, FT-IR analysis was also carried out. Crystallite size increased as the calcination temperature rose. Prolonged calcination time caused the crystallite size and lattice parameters to increase.

References.