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(\text{Y}_{0.968}\text{Er}_{0.002}\text{Yb}_{0.030})_2\text{O}_3 \text{ Upconverting Particles as Optical Heater}

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Abstract

The optical heating of upconverting particles and its surrounding volume has a vital role in biomedical and therapeutic applications. The internal self-heating in \((\text{Y}_{0.968}\text{Er}_{0.002}\text{Yb}_{0.030})_2\text{O}_3\) upconverting particles synthesized by urea assisted combustion route has been detected under 980 nm diode laser excitation. A temperature rise up to 348 K has been computed using fluorescence intensity ratio (FIR) method of the thermalized upconversion emission bands due to \(\text{H}_{11/2} \rightarrow \text{S}_{3/2}\) and \(\text{S}_{3/2} \rightarrow \text{I}_{15/2}\) transitions of \(\text{Er}^{3+}\). The results indicate that the material is an interesting candidate for optical heater in biomedical applications.

Keywords: Upconversion, Optical heating, Fluorescence intensity ratio (FIR), Photoluminescence, Phosphors, Biomedical applications, Thermographic phosphors, Temperature sensors, Optical thermometry, Nanoheater, Temperature, Measurement, Applications, Laser, Fluorescence, Luminescence, Nonradiative relaxation, Thermal population, Optical material, Rare earth, Erbium, Thermal probe, Heating effect, Combustion, Laser diagnostics, Yttrium oxide, Optical sensors, Laser material

Introduction

The upconversion (UC) emission based materials have been a subject of interest because of their utility in extensive field of applications such as biological imaging, biological sensor, photodynamic therapy, temperature sensors, display devices, security printing, solar cell, etc. [1–5]. Among these applications, optical temperature sensors are one of the most demanded applications. Optical temperature sensors are considered to be attractive alternatives to the conventional temperature sensors due to their ability to sense temperature remotely where physical access is not possible. The temperature sensing performance of some rare earth doped upconverting materials have been studied recently [6–8]. The temperature dependent fluorescence intensity ratio (FIR) of two closely lying energy levels (thermally coupled) of a rare earth ion is studied at different temperatures [7–11]. The technique also includes the variation of FIR of the Stark sublevels in single rare earth ion and two closely lying levels of different rare earth ions [12,13]. As the laser light irradiates the material, some heat is generated due to laser excitation. The laser induced heating in the upconverting materials has been reported rarely [14–17]. The internal heating in \(\text{Yb}^{3+}/\text{Er}^{3+}\) doped fluoride nanoparticles up to 1073 K has been detected by Tikhomirov et al. [14], whereas Singh et al. [15] have investigated the laser induced optical heating in \(\text{Er}^{3+}/\text{Yb}^{3+}\) codoped \(\text{Gd}_2\text{O}_3\) phosphor by monitoring the \(\text{S}_{11/2} \rightarrow \text{I}_{15/2}\) and \(\text{S}_{3/2} \rightarrow \text{I}_{15/2}\) transitions of \(\text{Er}^{3+}\). Hayakawa et al. [16] have observed the optical heating in the \(\text{Er}^{3+}\) doped \(\text{TeO}_2-\text{ZnO-Nb}_2\text{O}_5\) glass. Suo et al. [17] have also studied the laser induced optical heating in \(\text{Tm}^{3+}/\text{Yb}^{3+}\) codoped \(\text{Ba}_2\text{Gd}_2\text{Zn}_2\text{O}_7\) up-conversion (UC) phosphors using Stark levels (\(\text{G}_{411} - \text{G}_{412}\)) of \(\text{Tm}^{3+}\) and observed rising of sample temperature from 278.8 to 321.8 K with increasing pump power from 638 to 1802 mW. Laser induced heating was also studied using \(\text{Ho}^{3+}/\text{Yb}^{3+}\) ions in \(\text{Ca}_3\text{Al}_2\text{O}_7\) phosphor by monitoring the green emissions coming from the \(\text{Ho}^{3+}\) ion [18].

In this work we prepared \((\text{Y}_{0.968}\text{Er}_{0.002}\text{Yb}_{0.030})_2\text{O}_3\) particles by solution combustion route and the laser induced heating in \((\text{Y}_{0.968}\text{Er}_{0.002}\text{Yb}_{0.030})_2\text{O}_3\) upconverting particles by monitoring the intensity ratio of two green emission bands of \(\text{Er}^{3+}\) ions upon near infrared (NIR) excitation is reported. The self-heating of the particles have been calculated employing temperature dependent FIR study of the synthesized material and based on the observed results the utilization of the material for probable applications is concluded herein.

Experimental

The \((\text{Y}_{0.968}\text{Er}_{0.002}\text{Yb}_{0.030})_2\text{O}_3\) particles have been synthesized through urea assisted combustion process [19]. This process has advantages over other usual techniques, e.g. low synthesis temperature, uniform mixing, high purity, less energy consumption, etc. Since, nitrate forms of the precursors are used in combustion synthesis; the staring materials namely, the \(\text{Er}_2\text{O}_3\), \(\text{Yb}_2\text{O}_3\), and \(\text{Y}_2\text{O}_3\) were dissolved in concentrated nitric acid to prepare their nitrates. The optimized composition for the upconversion emission of the sample was taken according to the given formula.
96.8mol% Y₂O₃+0.2mol% Er³⁺+3.0mol% Yb³⁺

The nitrates were mixed with urea solution and the whole mixture was stirred for 3 h at 343 K on a magnetic stirrer with 800 rpm rotation speed. Due to this vigorous stirring, the material became a transparent gel. This gel was then transferred to an alumina crucible and placed inside a preheated furnace at 773 K. Combustion with auto-ignition takes place and a fluffy white mass is produced. The as-obtained powder is further annealed at 1123 K for 2 h and used for further characterization purposes.

The X-ray diffraction (XRD) were recorded over the angular range 10° ≤ 2θ ≤ 80° using Cu-ka(1.5405 Å) radiation by a Bruker D8 Advance X-ray diffractometer with a scanning rate of 4 degree per minute. A continuous wave NIR diode laser of 980 nm wavelength was used as excitation source for the sample. The sample was kept inside a homemade heat chamber. The laser beam was 0.70 mm in radius and was focused on the sample by adjusting the collimating optics. A thermocouple was placed at very close (~2 mm) to the focal spot of laser on the sample. The temperature dependent and pump power dependent upconversion spectra recorded. The laser beam was set at 10 W cm⁻² and the spectra were recorded at different temperatures. For temperature dependent measurements the laser was chopped with an optical chopper.

**Results and discussion**

The X-ray diffraction pattern of the annealed sample is shown in Figure 1 and the pattern was matched well with JCPDS card no. 25-1200 that shows cubic phase structure with lattice constant 10.60 Å. The main peaks observed at 20.77°, 29.47°, 34.09°, 48.79°, 57.76°, and 79.15° correspond to the (211), (222), (400), (440), (622), and (662) planes, respectively.

The upconversion emission spectra of the sample have been recorded in 500–600 nm range upon 980 nm diode laser excitation and are shown in Figure 2a. The green emission bands observed at ~524 nm and ~550 nm corresponding to the ²H₁₁/₂ → ⁴I₁₅/₂ and ⁴S₃/₂ → ⁴I₁₅/₂ transitions of Er³⁺ ion respectively.

It is seen from the figure that on increasing excitation power the emission intensity of two bands are not varying equally rather the ratio of the two emission bands (I₅₂₄/I₅₅₀) varies in a systematic way with excitation power. It is well known that the variation in ratio of emission intensity of two close lying levels of rare earth (RE) ions is because of the change in populations in the corresponding levels[10,18,20]. The energy separation between ²H₁₁/₂ and ⁴S₃/₂ levels is ~770 cm⁻¹ and the variation in population of these two levels obey Boltzmann’s distribution law[11]. So, the observed variation in intensity ratio is due to change in population and this observation gives rise the concept of laser induced optical heating. For experimental verification of the idea of optical heating, the fluorescence intensity ratio as a function of laser pump power has been calculated and is shown in Figure 3a. The variation of upconversion emission spectra at different excitation powers are shown in Figure 2b. It is observed that the
The laser induced heating in (Y_{0.968}Er_{0.002}Yb_{0.030})_2O_3 upconverting particles has been detected successfully upon NIR laser excitation. The cubic phase structured (Y_{0.968}Er_{0.002}Yb_{0.030})_2O_3 particles were synthesized by urea assisted solution combustion route. The pump power dependent upconversion emission and temperature dependent upconversion emission were correlated to measure the laser induced heating of the material. Though the generated heat in the material is not too high, still the temperature gain in this material is in the physiological range. So, the present material may be useful in biomedical applications (viz. hyperthermia treatment).

**Conclusions**

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**References**

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