



Effects of temperature on luminescent properties of Gd₂O₃:Er, Yb nanophosphor

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Received: 8 October 2019 / Accepted: 7 April 2020 / Published online: 18 April 2020
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Abstract

In this study we analyze nano powder Gd₂O₃ doped with Er³⁺ and Yb³⁺. Material was synthesized by simple and efficient solution combustion method. The nano phosphor was optically excited at 980 nm by using pulsed laser diode. We have obtained time resolved upconversion luminescence spectra of nano powder samples of Gd₂O₃:Er,Yb using a streak camera and analyzed temperature dependence of erbium optical line emission intensities. The possibilities of using Gd₂O₃:Er,Yb for temperature sensing were analyzed by means of luminescence intensity ratio method. We show that synthesized Gd₂O₃:Er,Yb material is useful for temperature measurements up to 550 K.

Keywords Photoluminescence · Thermographic phosphor · Laser spectroscopy · Combustion synthesis

1 Introduction

There is a growing interest in recent years for luminescence upconverting (UC) materials (Zhao et al. 2019; Gavrilovic et al. 2016; Ang et al. 2011). Using of upconversion nanoparticles provides many advantages compared to using of conventional phosphors. The effects of autofluorescence and of stray excitation light are inherently removed. Moreover, signal-to-noise ratio is notably improved.

The optical thermometry is widely used to remotely measure the temperature. There are several techniques of temperature sensing by using the thermographic phosphors, analyzed by us in (Sevic et al. 2020); they are based on intensity ratio, lifetime or rise time of luminescence. However, the luminescence intensity ratio (LIR) based temperature sensing is

This article is part of the Topical Collection on Advanced Photonics Meets Machine Learning.

Guest edited by Goran Gligoric, Jelena Radovanovic and Aleksandra Maluckov.

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most commonly used and will be analyzed here. The technique is based on calculating the optical emission intensity variations of thermally coupled transitions, measured at different temperatures. Literature survey shows that the Er^{3+} ion is a prevalently used for the LIR based temperature sensing with UC phosphors (Li X. et al. 2016; Li L. et al. 2016; Avram et al. 2018; Du et al. 2017; Du et al. 2018; Dong et al. 2007; Xu et al. 2012; Liu et al. 2016; Santos et al. 1999; Wang et al. 2015; Manzani et al. 2017; Singh et al. 2009; Krizan et al. 2014).

The luminescence temperature dependence of downconverting materials is exhaustively analyzed in literature (Goss et al. 1989; Feist et al. 1999; Heyes 2009; Cates et al. 2002; Eldridge et al. 2012). The detailed analyses of thermographic phosphors are provided in review papers (Allison et al. 1997; Alden et al. 2011; Brites et al. 2012; Jaque and Vetrone 2012; Khalid and Kontis 2008). Studies of sensing of temperature using UC materials doped with Er, Yb could be found in (Li X. et al. 2016; Li L. et al. 2016; Avram et al. 2018; Du et al. 2017; Du et al. 2018; Dong et al. 2007; Xu et al. 2012; Liu et al. 2016; Santos et al. 1999; Wang et al. 2015; Manzani et al. 2017; Singh et al. 2009; Krizan et al. 2014).

The aim of this study is analysis of potentials of using the $\text{Gd}_2\text{O}_3:\text{Er}^{3+}, \text{Yb}^{3+}$ for temperature measurements. Gd_2O_3 host doped with Er and Yb was prepared by simple and cost effective process. There are various improvements of nano phosphor materials in regard the luminescent efficiency (Li et al. 2017; May et al. 2018; Ou et al. 2018; Su et al. 2019; Xing et al. 2017; Zhang et al. 2019). However, due to appropriately positioned erbium transition lines, $\text{Gd}_2\text{O}_3:\text{Er}^{3+}, \text{Yb}^{3+}$ is a very good candidate for luminescence intensity ratio based remote temperature sensing.

This is not our first study about Gd_2O_3 host. Analysis of temperature effects on $\text{Gd}_2\text{O}_3:\text{Er}^{3+}, \text{Yb}^{3+}$, using continuous excitation, was presented in (Krizan et al. 2014). The pulsed laser excitation, used in this study, has some advantages compared to continuous excitation in regard of local heating of material. The luminescence temperature dependence of the Gd_2O_3 host doped with Eu was also analyzed before (Nikolic et al. 2013).

2 Experimental procedures

The Solution Combustion Synthesis (SCS) method, explained in detail in (Krizan et al. 2014), was used to prepare the studied nano phosphor.

Stoichiometric quantities of starting chemicals $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, $\text{Er}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ and $\text{Yb}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ with the high purity were chosen to obtain the Er concentration of 2.0 at.% and Yb concentration of 0.5 at.% in $\text{Gd}_2\text{O}_3:\text{Er}, \text{Yb}$ ($\text{Gd}_{1.95}\text{Er}_{0.04}\text{Yb}_{0.01}\text{O}_3$).

For solution combustion synthesis we have used the dry mixture of 14.67 g (32.5 mmol = 195/6 mmol) of $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, 0.296 g (0.667 mmol = 4/6 mmol) of $\text{Er}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ and 0.075 g (0.167 mmol = 1/6 mmol) of $\text{Yb}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$, combined with the mixture of 1 g of β -alanine and 5 g of urea which were used as organic fuels.

Pulsed laser diode excitation at 980 nm was used to acquire the photoluminescence emission spectra of $\text{Gd}_2\text{O}_3:\text{Er}^{3+}, \text{Yb}^{3+}$ nano phosphor. The optical emission of analyzed samples was detected by using the streak camera system, used and explained in (Sevic et al. 2020; Vlastic et al. 2018). The detailed explanation of the setup for temperature measurements is provided in (Rabasovic et al. 2016).

A high resolution scanning electron microscope (SEM) equipped with a high brightness Schottky Field Emission gun (FEGSEM, TESCAN) operating at 4 kV was used to check the structure of studied nano phosphor.

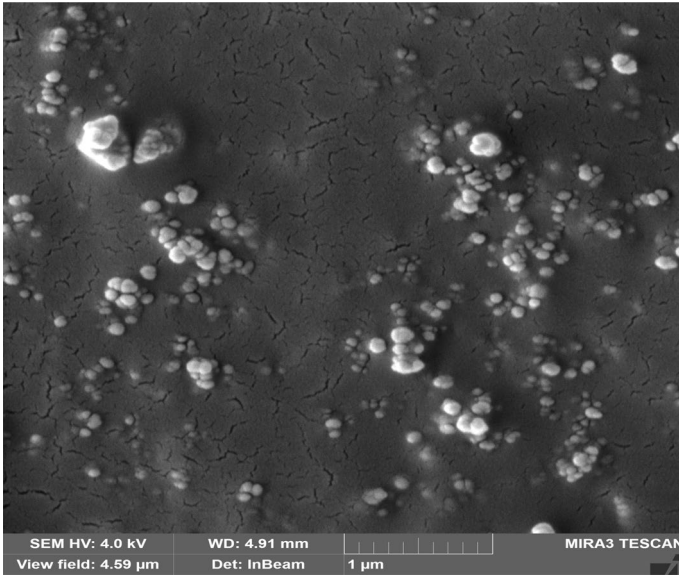


Fig. 1 SEM image of $\text{Gd}_2\text{O}_3:\text{Er}^{3+}, \text{Yb}^{3+}$ nano phosphor prepared by SCS method

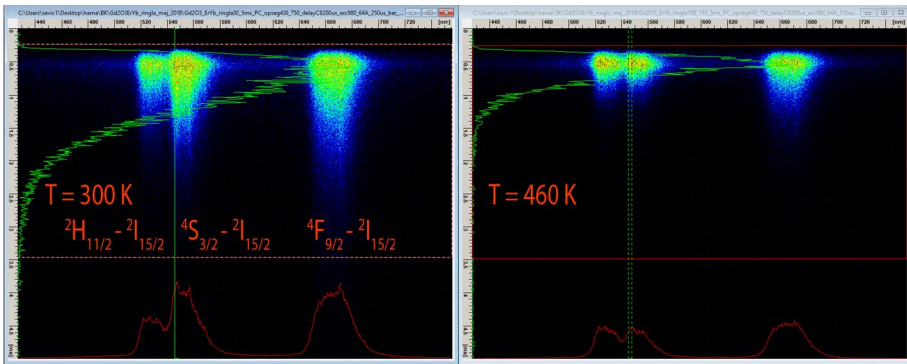


Fig. 2 Time-resolved luminescence spectra of $\text{Gd}_2\text{O}_3:\text{Er}^{3+}, \text{Yb}^{3+}$ at two temperatures. Pulsed laser diode excitation is at 980 nm

3 Results and discussion

The representative SEM image of the synthesized $\text{Gd}_2\text{O}_3:\text{Er}^{3+}, \text{Yb}^{3+}$ nano material, see Fig. 1, shows the agglomerated grains and particles with dimensions from 50 nm to over 100 nm.

The photoluminescence emission of $\text{Gd}_2\text{O}_3:\text{Er}^{3+}, \text{Yb}^{3+}$ was measured over the temperature range 300–660 K. To get the general impression of luminescence temperature dependence, time-resolved luminescence spectra of $\text{Gd}_2\text{O}_3:\text{Er}^{3+}, \text{Yb}^{3+}$ at two temperatures are displayed in Fig. 2. The emission band centered at 528 nm originates from ${}^2\text{H}_{11/2} \rightarrow {}^2\text{I}_{15/2}$ transition, the emission band at 553 nm originates from ${}^4\text{S}_{3/2} \rightarrow {}^2\text{I}_{15/2}$

transition, and the emission bands at 670 nm originates from ${}^4F_{9/2} \rightarrow {}^2I_{15/2}$ transition of Er^{3+} ion.

Luminescence quenching, i.e. decreasing of luminescence quantum yield at increasing concentrations of dopants in phosphors is a well known phenomenon. Literature survey (Lojpur et al. 2013; Li X. et al. 2016; Li L. et al. 2016; Avram et al. 2018; Du et al. 2017; Du et al. 2018; Dong et al. 2007; Xu et al. 2012; Liu et al. 2016; Santos et al. 1999; Wang et al. 2015; Manzani et al. 2017; Singh et al. 2009) shows that concentrations of dopants in our work are too small in regard the concentration quenching phenomenon, so it was neglected.

Concentration effects on luminescence intensities of Er transitions were analyzed in (Lojpur et al. 2013). It was shown that by varying the concentration of Yb, it is possible to vary the intensity and lifetime ratio of green and red component of luminescence (color tuning). By decreasing the Yb concentration the ratio between the green and red component increases in favor of the green component, emitted at two Er transitions, namely ${}^2H_{11/2} \rightarrow {}^2I_{15/2}$ and ${}^4S_{3/2} \rightarrow {}^2I_{15/2}$ transitions.

At the room temperature, the sample of $\text{Gd}_2\text{O}_3:\text{Er},\text{Yb}$ analyzed in this work has similar intensities and lifetimes (in order of about 0.7 ms, measured from data obtained by streak image shown in Fig. 2) of green and red component of luminescence. So, in regard of using of this material for remote temperature sensing, the dopant concentrations could be considered as optimal.

Integrated-in-time luminescence spectra of $\text{Gd}_2\text{O}_3:\text{Er}^{3+},\text{Yb}^{3+}$ at several temperatures are shown in Fig. 3. We see that for temperatures above 550 K the measured signal gets weak and impractical for reliable analysis.

Based on our estimations of shape of spectral response and time durations of luminescence of erbium transitions shown in Fig. 2, we propose the schematic view of $\text{Gd}_2\text{O}_3:\text{Er}^{3+},\text{Yb}^{3+}$ energy transfer processes presented in Fig. 4.

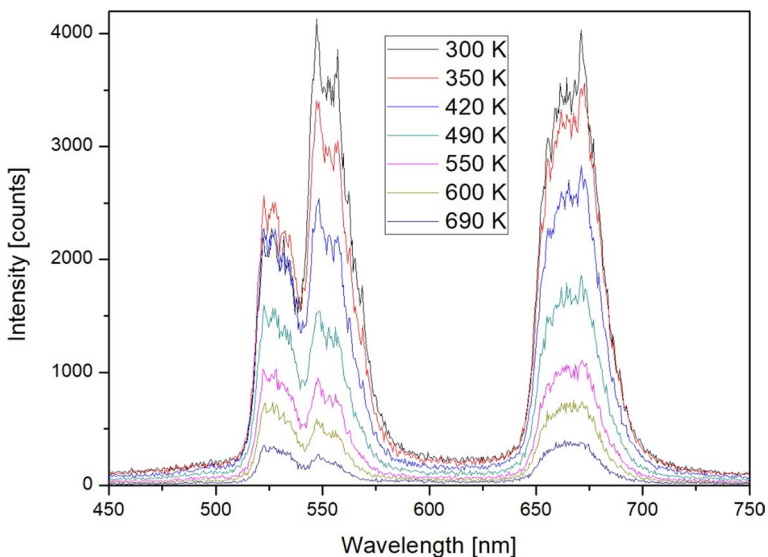
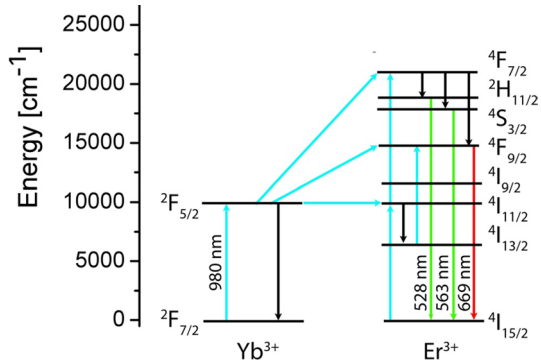


Fig. 3 Luminescence spectra of $\text{Gd}_2\text{O}_3:\text{Er}^{3+},\text{Yb}^{3+}$ at several temperatures. Pulsed laser diode excitation is at 980 nm

Fig. 4 Schematic view of $Gd_2O_3:Er^{3+}, Yb^{3+}$ energy transfer processes



Looking at Figs. 2 and 3 it is easily noticed that luminescence intensity ratio between lines at 528 nm and 553 nm is temperature dependent. It is well known that the luminescence intensity ratio is independent on small variations of laser excitation energy or optical gain of acquisition system (Brites et al. 2012; Jaque and Vetrone 2012). Two emission lines, with a small energy gap between them, are mostly chosen to implement the temperature sensing. The higher energy level becomes more populated with increasing of temperature by thermalization effects from the lower energy level. In this way, relative intensity of higher level, in regard the lower level, gradually increases by temperature increase.

Ratio of intensities of two levels, IR , is determined by Boltzmann-type population distribution given by (Brites et al. 2012; Jaque and Vetrone 2012; Đačanin et al. 2014; Nikolic et al. 2018):

$$IR = \frac{I_{31}}{I_{21}} = C \exp\left(-\frac{\Delta E}{kT}\right) \tag{1}$$

where k is the Boltzmann constant, $k=0.695\ 034\ 76(63)\ \text{cm}^{-1}\ \text{K}^{-1}$ and ΔE is the energy gap between two excited levels.

However, as explained in (Lojpur et al. 2016; Sevic et al. 2020) another constant, A , should be introduced to Eq. (1), to account for the finite value of IR at low temperatures:

$$IR = \frac{I_{31}}{I_{21}} = A + C \exp\left(-\frac{\Delta E}{kT}\right) \tag{2}$$

Figure 5 shows temperature sensing calibration curve based on luminescence intensity ratio of two Er emission lines, at 528 nm and 553 nm. Being of strong intensity and closely located, these two lines satisfy all criteria for choosing them for LIR method. Estimated value of the energy gap is $\Delta E=728\ \text{cm}^{-1}$. It is obtained by fitting the sensing calibration curve by Eq. (2). Our estimation of the energy gap is similar to results presented in (Li X. et al. 2016; Li et al. 2016; Avram et al. 2018; Du et al. 2017; Du et al. 2018; Dong et al. 2007; Xu et al. 2012; Liu et al. 2016; Santos et al. 1999; Wang et al. 2015; Manzani et al. 2017; Singh et al. 2009). We have discarded the measured points above 550 K to avoid large fitting errors.

The rate at which IR changes with the temperature defines the absolute thermal sensitivity, S :

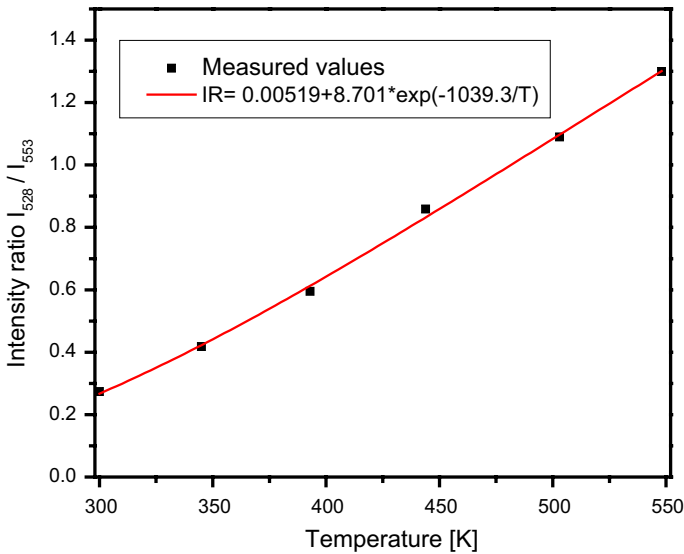


Fig. 5 Temperature sensing calibration curve of $\text{Gd}_2\text{O}_3:\text{Er},\text{Yb}$, using intensity ratio of two Er emission lines, at 553 nm and 529 nm. Experimental points are denoted by black squares and fitted temperature calibration curve is denoted by red line

$$S = \left| \frac{dIR}{dT} \right| \quad (3)$$

The absolute thermal sensitivity is of a small practical use for comparison purposes, so, we use the relative thermal sensitivity, S_r , defined by Eq. (3):

$$S_r = \left| \frac{1}{IR} \frac{dIR}{dT} \right| \quad (4)$$

Figure 6 shows the relative sensitivity curve, calculated by Eq. (4). Looking at the Fig. 6 we see that the relative sensitivity is highest at room temperature, its value is $1.18\% \text{ K}^{-1}$, and its lowest value is about $0.35\% \text{ K}^{-1}$ at 550 K.

Comparing the results presented here and in (Krizan et al. 2014), we see that the temperature sensing calibration curves look very similar, only slightly in favor of pulsed excitation because of somewhat better intensity ratio. However, the pulsed excitation offers possibility for using the luminescence lifetime for temperature sensing, providing grounds for multi-mode temperature sensing described in (Sevic et al. 2020).

4 Conclusion

We have studied the possibilities of using $\text{Gd}_2\text{O}_3:\text{Er}^{3+},\text{Yb}^{3+}$ for remote temperature sensing. Analyzed samples were fabricated by simple and low-cost Solution Combustion Synthesis (SCS) method. We have used the pulsed laser excitation because it has some advantages compared to continuous excitation in regard of local heating of material. Erbium spectral lines at 528 nm (${}^2\text{H}_{11/2} \rightarrow {}^2\text{I}_{15/2}$ transition) and 553 nm (${}^4\text{S}_{3/2} \rightarrow {}^2\text{I}_{15/2}$ transition)

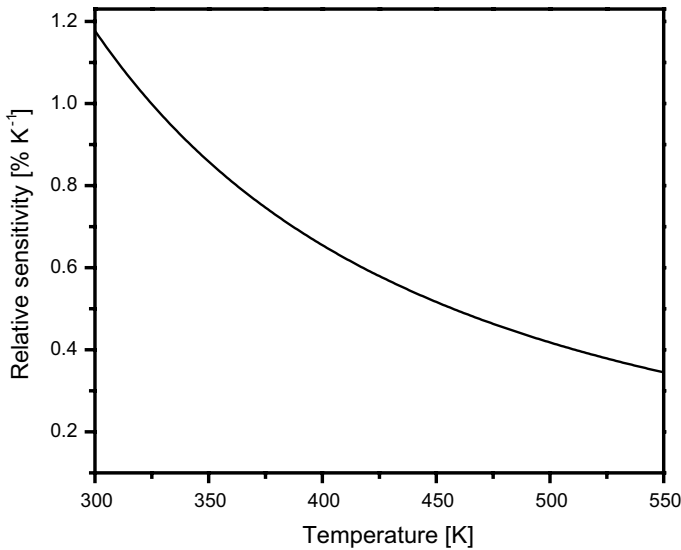


Fig. 6 Relative sensitivity curve as the function of temperature for $\text{Gd}_2\text{O}_3:\text{Er,Yb}$

were selected for implementation of luminescence intensity ratio method for temperature measurements. Our analysis shows that synthesized $\text{Gd}_2\text{O}_3:\text{Er,Yb}$ material is useful for temperature measurements up to 550 K, with relative sensitivity of $1.18\% \text{ K}^{-1}$ at room temperature. With increasing the temperature the relative sensitivity decreases up to $0.35\% \text{ K}^{-1}$ at 550 K. In our future work we will analyze prospects of using the erbium optical emission lifetime for temperature sensing.

Acknowledgements This work was financially supported within the Projects of Ministry of Education, Science and Technological Development of the Republic of Serbia OI171020.

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