Luminescence intensity ratio by three thermalized levels in YAG:Er^{3+}/Yb^{3+} nanoparticles

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Abstract

Luminescence thermometry is a remote temperature sensing method by observing temperature dependent spectral changes for temperature readout. Chase for increasing temperature readout sensitivity motivated research of employing 3rd thermalized level of Er^{3+} emission in Yb^{3+}/Er^{3+} upconversion photoluminescence. For this purpose, highly stable and efficient yttrium aluminium garnet (YAG): Yb^{3+}/Er^{3+} nanoparticles were prepared by a modified Pechini method. The emission spectra were recorded from 300 to 800 K, and two luminescence intensity ratios between emissions of $^4S_{3/2}$, $^2H_{11/2}$, and $^4F_{7/2}$ were obtained. Apart from excellent matching theoretical predictions, the readout by using the $^4F_{7/2}$ method provided a 3.5-fold increased relative sensitivity over the luminescence intensity ratio by $^2H_{11/2}$ level, which is limited by being usable only above 600 K. The method by emission from $^2H_{11/2}$ is to be used from 300 to 600 K, while emission from $^4F_{7/2}$ provides the best luminescence intensity ratio at temperatures from 600 K to 800 K. YAG:Yb^{3+}/Er^{3+} nanoparticles proved to be an excellent sensor material for the luminescence intensity ratio method by employing multiple thermalized levels.

Keywords: Luminescence thermometry; upconversion nanoparticles; fluorescence intensity ratio; Pechini method.

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1. INTRODUCTION

Luminescence is the emission of light that occurs when a substance absorbs energy, such as photons or electrons, and then releases that energy in the form of light emission. This phenomenon is commonly observed in various contexts, including fluorescence, phosphorescence, and bioluminescence, and it is essential in fields like lighting, materials science, and biological imaging.

Luminescence thermometry is a remote temperature measurement technique that relies on the temperature induced spectral changes of luminescence from a material to determine its temperature. By analysing the emitted luminescence, it is possible to accurately and remotely measure the temperature of the material, making the method valuable for various applications, including in industrial processes and scientific research [1]. The read-out method and its probe are unperturbed even by high electromagnetic fields, radiation, and are often resistant to corrosive environments. Luminescence thermometry has an advantage over infrared (IR) cameras and pyrometers in its ability to provide highly accurate and non-contact temperature measurements even for materials with low or variable emissivity, as it relies on the intrinsic properties of luminescent materials rather than surface characteristics like infrared radiation [2]. Additionally, it can be employed from the nanoscale up to measuring the temperature of a large surface. Thus, luminescent thermometry is used in niche applications where other contact methods, like thermocouples, or remote methods, like IR cameras, fail.

The most frequently used method for luminescence thermometry is called luminescence intensity ratio (LIR), which uses the ratio of emission intensities (I) for the read-out. LIR is ratiometric, *i.e.* it does not depend on the fluctuations in excitation. Usually, LIR is taken as a ratio of emissions from two thermally coupled levels. Thermally coupled levels are

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those in which the energy difference is sufficient for the thermal energy to produce excitation from the lower (L) to the higher (H) thermally coupled energy level. LIR then follows Boltzmann distribution according to Equation (1) [3]:

$$\mathrm{LIR} = \frac{I_\mathrm{H}}{I_L} = Be^{-\frac{\Delta E}{kT}} \quad (1)$$

where B is the temperature invariant constant that depends on the material properties, I_H is the intensity from H level, I_L is the intensity from the L level, and ΔE is the energy difference between the thermally coupled levels, and k = 0.695 cm^{-1} K^{-1} is the Boltzmann constant. The performance of a temperature probe material is estimated by the figure of merit called relative sensitivity [4], Equation (2):

$$S_\mathrm{r} = \frac{100}{\mathrm{LIR}}\left|\frac{\partial \mathrm{LIR}}{\partial T}\right| = 100\frac{\Delta E}{kT^2} \quad (2)$$

The relative sensitivity (% K^{-1}) is the normalized rate of change of LIR and can be used for comparison with other sensor probes or even other readout methods.

Upconversion luminescence is a process in which a material absorbs multiple lower-energy photons and then emits a single higher-energy photon. This phenomenon is used in various applications, such as upconversion nanoparticles, where it enables the conversion of low-energy near-infrared light into visible light for purposes like biological imaging and energy-efficient photovoltaic devices [5]. It also has advantages in luminescence thermometry as it eliminates background radiation and autofluorescence that could appear under UV excitation of the probe material. The most famous upconverting pair of lanthanide ions are erbium and ytterbium, as they have excited energy levels with similar energies, thus there is an efficient energy transfer from Yb^{3+} to Er^{3+}. Yb^{3+} has an order of magnitude larger absorption cross-section than Er^{3+}, thus the upconversion efficiency by Yb^{3+} co-doping is increased multiple times [6]. After two energy transfers from Yb^{3+} (which is excited by near-infrared (NIR) light) to Er^{3+}, Er^{3+} exhibits visible, green emission. Er^{3+} is also the most frequently used ion in luminescence thermometry as there is an excellent thermal coupling between its thermalized levels $^4S_{3/2}$ and $^2H_{11/2}$ and the latter is unusually very intense due to its favourable radiative properties [7]. However, the energy separation of about 700 cm^{-1} between those two levels limits the relative sensitivity according to Eq. 2. The current trend in luminescence thermometry is either finding sensor materials that exhibit higher relative sensitivities or finding methods or their improvements that enable the same [8]. The recent improvement in the LIR readout method, presented in literature [9,10], enable the use of energetically higher thermalized levels at elevated temperatures for reaching larger relative sensitivities. As the relative sensitivity, according to Eq. 2, linearly depends on the energy gap, using this 3rd excited level in LIR produces higher sensitivity than conventional LIR [9].

Ultimately, high stability of the host matrix is of utmost importance for luminescence thermometry. Oxide crystals are considered the most thermally and chemically stable hosts, among which yttrium aluminium garnet (YAG, $Y_3Al_5O_{12}$) is well known for both its stability and its optical properties, as it is used in the most prominent of solid-state lasers, Nd:YAG, and as a phosphor in commercial white LED chips. The Pechini method offers several advantages for synthesizing YAG, including excellent homogeneity, precise compositional control, and the production of fine powders with enhanced properties such as mechanical strength. Additionally, it allows for easy integration of dopant ions, making it ideal for tailoring YAG's luminescence and other properties for specific applications [11]. The only previous research employing the 3rd thermalized level in Er^{3+} is in a less stable fluoride matrix [10]. Here we present the investigation of using the 3 thermalized levels for luminescence thermometry by upconverting pair of Yb^{3+}/Er^{3+} in the YAG matrix, report on its performance, and demonstrate the increase in sensitivity by employing the 3rd thermalized emissive level.

2. EXPERIMENTAL

Herein, the sample with the following formula: $Y_{2.64}Yb_{0.3}Er_{0.06}Al_5O_{12}$ (YAG:10Yb2Er) was synthesized *via* a modified Pechini method [12,13]. The doping ions concentration of 10 (Yb^{3+}), and 2 mol % (Er^{3+}) were chosen arbitrary based on our previous extensive experience in working with lanthanide activated garnet type of materials, based on the well known fact that the optimal ratios of Yb/Er for upconversion process are 4:1 or 5:1 [14,15]. The concentrations do not affect the ratios of thermalized emissions employed for thermometry. Metal nitrates (yttrium(III) nitrate hexahydrate, $Y(NO_3)_3{\times}6H_2O$;

ytterbium(III) nitrate pentahydrate, $Yb(NO_3)_3{\times}5H_2O$; erbium (III)-nitrate pentahydrate, $Er(NO_3)_3{\times}5H_2O$; aluminium(III) nitrate nonahydrate, $Al(NO_3)_3{\times}9H_2O$; Alfa Aesar, USA, purity 99.9, 99.9, 99.99+ and 98+ %, respectively); citric acid - CA ($HOC(COOH)(CH_2COOH)_2$, Sigma Aldrich, USA, ACS reagent, ≥99.5 %) and ethylene glycol - EG ($HOCH_2CH_2OH$, Sigma Aldrich, country, anhydrous, 99.8 %) were used as starting materials without further purification. In a typical procedure, calculated amounts of metal nitrate precursors were added to the solution of citric acid in ethylene glycol (M: CA: EG = 1: 5: 25) and stirred for 30 min at 80 °C. After rising temperature to 120 °C, stirring was continued until a yellow brownish gel was obtained, which was then transferred into a alumina crucible and placed into a furnace at 600 °C for 2 h, followed by annealing at 1100 °C for 2 h. The powder sample obtained after annealing was cooled to room temperature and ground in a mortar.

Crystal structure and phase purity of the material were assessed by X-ray diffraction (XRD) measurements conducted by using a Rigaku SmartLab instrument (Rigaku, Japan), utilizing Cu-Ka radiation with a wavelength of 15.40 nm. The sample morphology was analysed by transmission electron microscopy (TEM) using Philips CM-20 SuperTwin TEM (Philips, Holland). After placing the sample powder inside the holder of the heating stage the emission spectra were recorded by, *via* Andor Kymera 193i spectrometer (Oxford Instruments, UK) with a CCD detector. Single emission/excitation spectrum was obtained at each temperature, from 300 to 800 K, with step 25 K. Excitation was performed by Ekspla NT342B OPO laser (producer, country). The sample was heated by the Linkam THMS600 stage (Linkam, UK).

3. RESULTS AND DISCUSSION

XRD measurements demonstrated that the YAG:10Yb2Er crystallizes in a body-centred cubic structure with $Ia\bar{3}d$ (230) space group (ICDD card no. 01-073-3184), as presented in Figure 1a. A TEM image shows that the YAG:Er^{3+}/Yb^{3+} sample consists of agglomerated nanoparticles of size between 30 nm and 40 nm (Figure 1b).

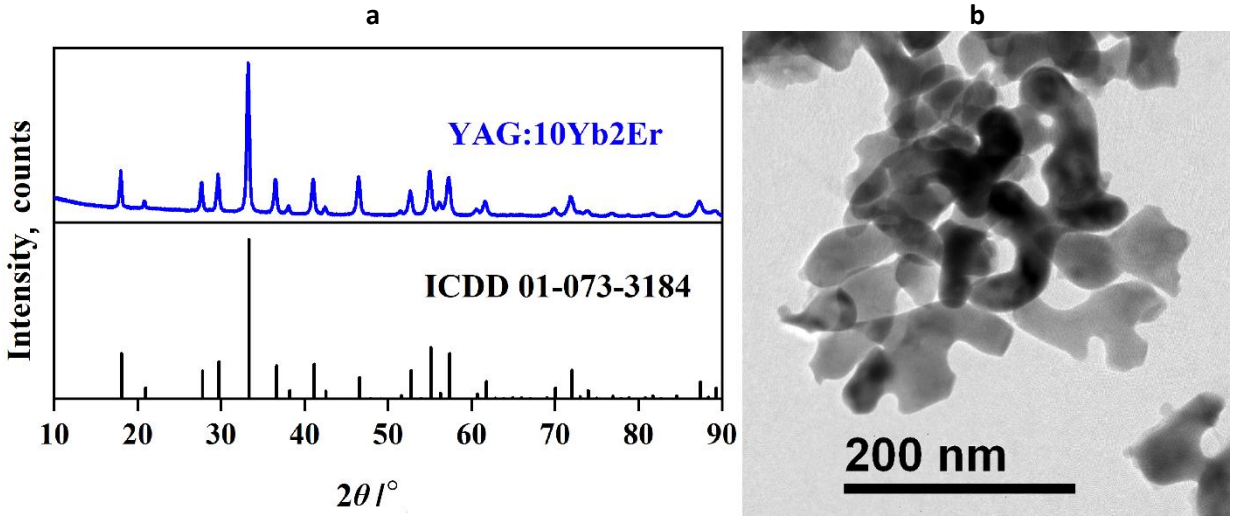

Figure 1 Characterization of YAG:Yb^{3+}/Er^{3+} powder: a) XRD, b) TEM micrograph (scale bar: 200 nm)

The excitation spectrum obtained by monitoring the most intense emission at room temperature, $^4S_{3/2}{\rightarrow}{}^4I_{15/2}$, is presented in Figure 2a. The spectra are featured by the excitations from the ground multiplet of Yb^{3+} and a small peak originating from direct excitation of Er^{3+} to the $^4I_{11/2}$ level. The Stark splitting of Yb^{3+} $^2F_{7/2}$ and $^2F_{5/2}$ levels in the YAG matrix is well known [13]: the ground level is split into 4 sublevels, and the excited level into 3 sublevels. Due to the thermalization of the Stark sublevels of the ground level of Yb^{3+}, there is also a possible excitation from the 2nd and 3rd Stark sublevels into the $^2F_{5/2}$ [16]. After excitation into $^2F_{5/2}$, the non-radiative energy transfer from Yb^{3+} to Er^{3+} occurs, populating the $^4I_{11/2}$ level, with a much higher probability than excitation directly from the Er^{3+} ground level [17]. After the second energy transfer from the Yb^{3+} ion, Er^{3+} gets excited to the $^4F_{7/2}$ level. From $^4F_{7/2}$ the electron can de-excite radiatively or non-radiatively to the lower levels. Due to the small energy gap between $^4F_{7/2}$ and its neighbouring level $^2H_{11/2}$, and somewhat energetically lower $^4S_{3/2}$, multiphonon de-excitation is the most probable mechanism [18]. As the

energy difference between those 3 levels, $^4F_{7/2}$, $^2H_{11/2}$, and $^4S_{3/2}$ are small enough, the relative optical centre population between them is given by the Boltzmann distribution, and those 3 levels are said to be thermalized (see Figure 2b). Due to the larger energy gap between $^4S_{3/2}$ and its next energetically lower level the probability for multiphonon de-excitation significantly drops, and the $^4S_{3/2}$ level is highly efficient for radiative emission.

The emission spectra under 916 nm excitation (1-7 Stark levels of Yb^{3+}) from 300 K to 800 K, with a step of 25 K, are presented in Figure 2c. $^4S_{3/2}\rightarrow{}^4I_{15/2}$ emission uniformly decreases with increasing temperature. The $^2H_{11/2}\rightarrow{}^4I_{15/2}$ emission first increases with temperature because of obtaining optical centres from the $^4S_{3/2}$ level according to the Boltzmann distribution, and then decreases due to the temperature quenching and increased share of electrons to the even higher level, $^4F_{7/2}$. $^4F_{7/2}\rightarrow{}^4I_{15/2}$ emission uniformly increases with increasing temperature.

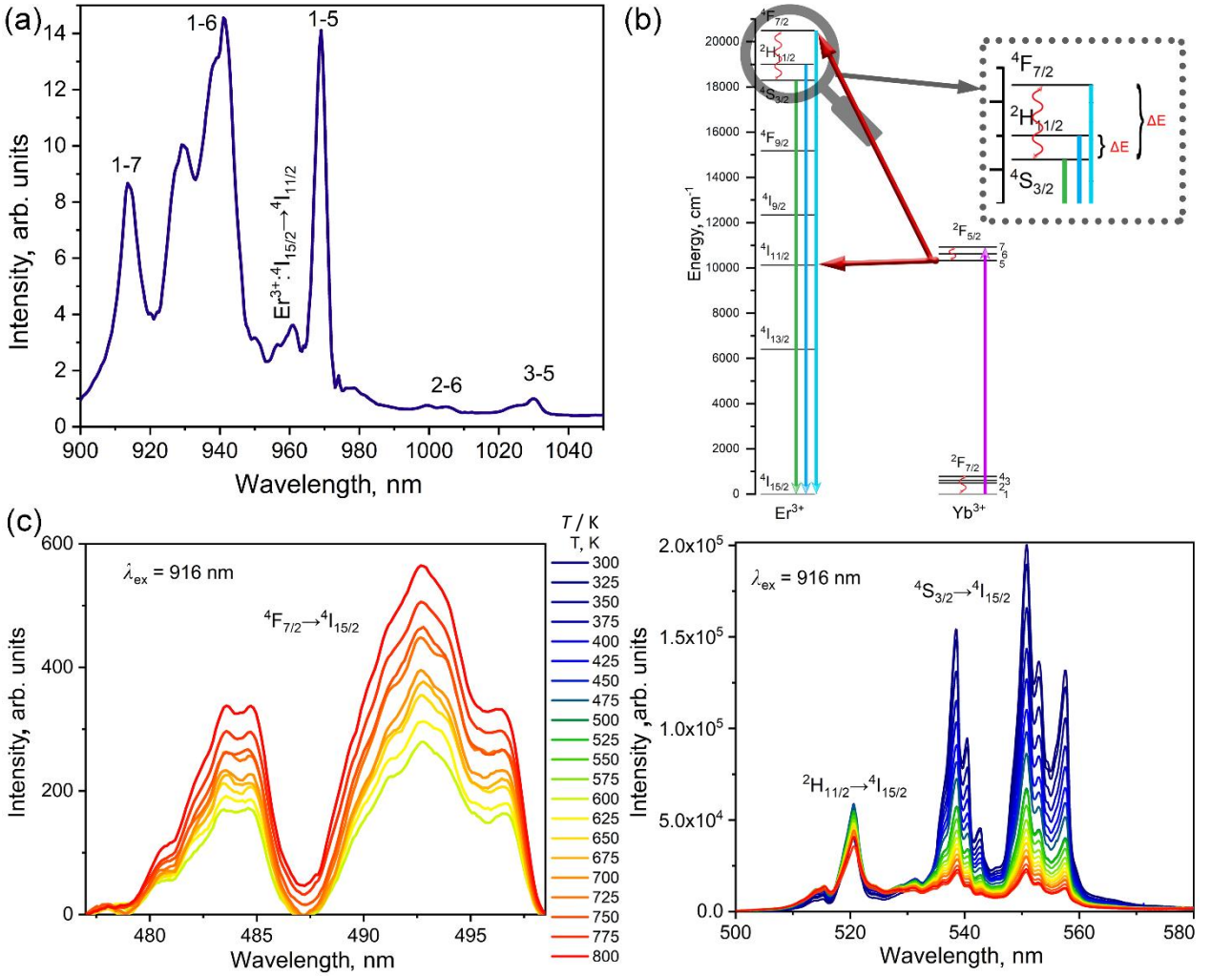

Figure 2. Luminescence of YAG:Er^{3+}/Yb^{3+} (a) NIR excitation spectrum obtained by monitoring $^4S_{3/2}\rightarrow{}^4I_{15/2}$ emission. (b) Energy level diagram of excitation, energy transfers, thermalizations, and emissions in Yb^{3+}/Er^{3+} upconversion. (c) Upconversion emission spectra of YAG:Er^{3+}/Yb^{3+} at various temperatures.

The integrated emission intensities of $^4S_{3/2}\rightarrow{}^4I_{15/2}$, $^2H_{11/2}\rightarrow{}^4I_{15/2}$, and $^4F_{7/2}\rightarrow{}^4I_{15/2}$ are given in Figure 3a. From the integrated intensities and Eq. 1 two LIRs are obtained: between the 1st and 2nd thermalized levels (Figure 3b), and between the 1st and 3rd thermalized levels (Figure 3c). Conventional LIR between the 1st and 2nd thermalized levels, obtained by fitting to the Boltzmann distribution, provided for an energy gap of 633 cm^{-1} between the $^2H_{11/2}$ and $^4S_{3/2}$, which is a value close to the one given in the literature [19]. The relatively high value of the *B* parameter is an indication of a high intensity of emission from the $^2H_{11/2}$ level. By using the emissions from $^4F_{7/2}$ and $^4S_{3/2}$ levels the fitted energy gap is equal to 2197 cm^{-1}. Due to the low intensity of $^4F_{7/2}$ emissions LIR using the 3rd thermalized level is impractical at

temperatures below 600 K. An excellent fit qualities and fitting to the Boltzmann relation without added offset are proof of good thermalization and adequate experimental conditions without stray light [3].

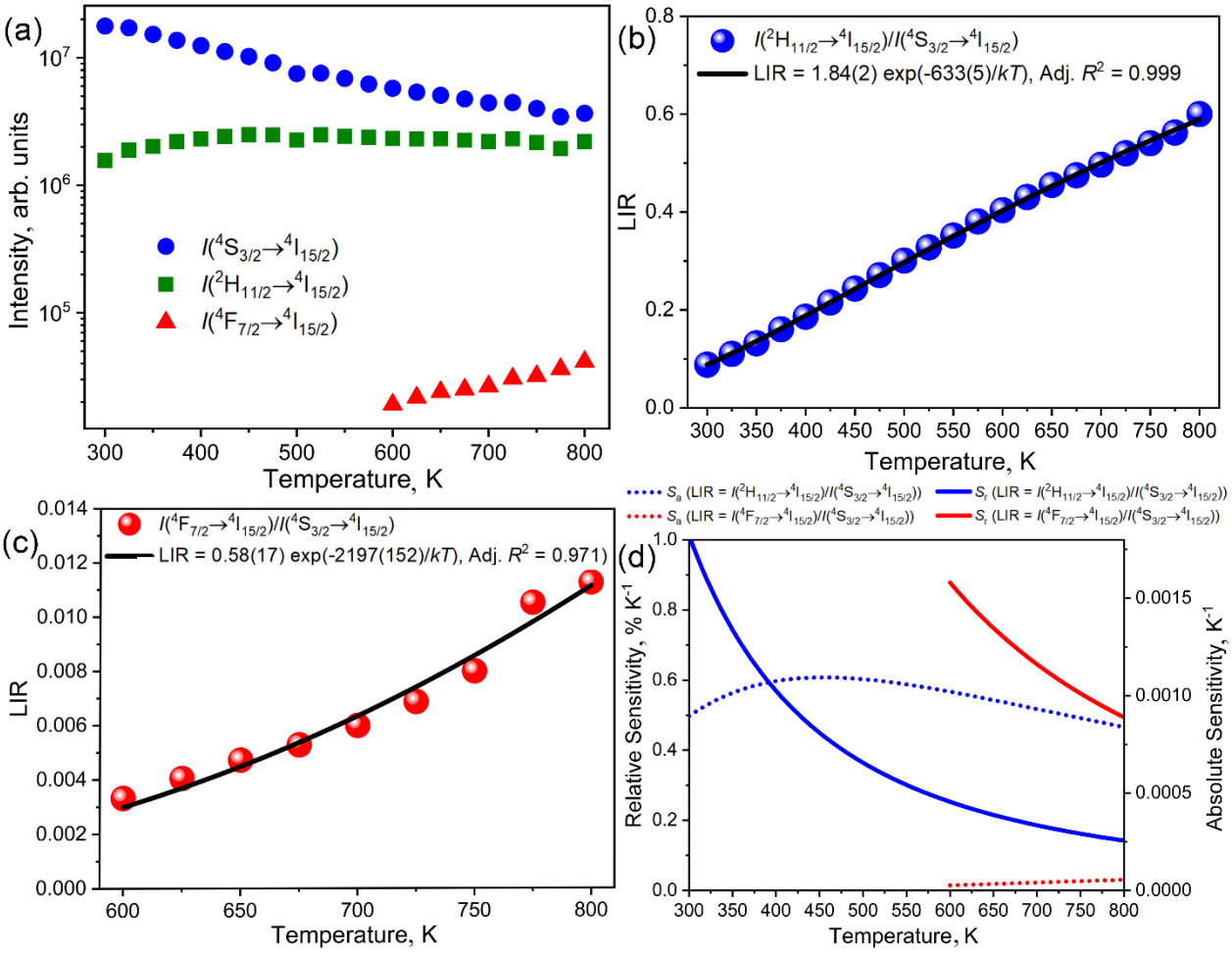

Figure 3. Thermometric analysis of YAG:Er^{3+}/Yb^{3+} by LIR methods. (a) Integrated intensities of $^4S_{3/2}\rightarrow{}^4I_{15/2}$, $^2H_{11/2}\rightarrow{}^4I_{15/2}$, and $^4F_{7/2}\rightarrow{}^4I_{15/2}$ emissions; (b) LIR of $^2H_{11/2}\rightarrow{}^4I_{15/2}$ and $^4S_{3/2}\rightarrow{}^4I_{15/2}$ emissions; (c) LIR of $^4F_{7/2}\rightarrow{}^4I_{15/2}$ and $^4S_{3/2}\rightarrow{}^4I_{15/2}$ emissions; (d) Comparison of absolute sensitivities (S_a) and relative sensitivities (S_r) of LIRs given under (b) and (c)

Relative sensitivities of those two LIRs are calculated from the fitted relations and according to Eq. 2. The results are shown in Figure 3d. Note that this figure also features the absolute sensitivity (S_a), which is a non-normalized rate of change of LIR, given by Equation (3):

$$S_a = \left|\frac{\mathrm{d\ LIR}}{\mathrm{d}T}\right| = \mathrm{LIR}\ S_r \tag{3}$$

Figure 3d demonstrates that LIR by using 1st and 3rd thermalized levels is significantly more sensitive than the conventional LIR at temperatures above 600 K where this method is practical. The improvement of sensitivity is directly proportional to the rate of energy gaps, Equation (4):

$$\frac{\Delta E\left(3^{rd}-1^{st}\right)}{\Delta E\left(2^{nd}-1^{st}\right)} = \frac{S_r\left(3^{rd}-1^{st}\right)}{S_r\left(2^{nd}-1^{st}\right)} \approx 3.5 \tag{4}$$

meaning that the relative sensitivity of the YAG:Er^{3+}/Yb^{3+} sensor by using the 3rd thermalized level is 3.5-fold greater than the conventional LIR by using only the 2nd level. At temperatures below 600 K, conventional LIR thermometry has to be used. Thus, this broad-temperature range sensor should employ two LIR variants for temperature readouts with the best performance, depending on the temperature range.

4. CONCLUSION

Luminescence thermometry by upconversion nanoparticles is a novel, prospective, and industrially relevant remote temperature sensing technique for niche applications. Due to the advantages over other methods, it is important to perfect the signal (spectrum) information processing to obtain the highest possible sensitivity. This problem was addressed by employing emission from the 3rd thermalized level in a highly stable YAG matrix doped by the most recognized upconverting pair Yb^{3+}/Er^{3+}. YAG:Yb^{3+}/Er^{3+} nanoparticles were prepared by a modified Pechini method after which their pure phase was confirmed, while the average nanoparticle diameter was determined as around 35 nm.

Under NIR excitation the Er^{3+} exhibited efficient green upconversion emission consisting of emissions from $^4S_{3/2}$, $^2H_{11/2}$, and $^4F_{7/2}$ levels. Spectra were recorded in a wide temperature range, from room temperature up to 800 K. From the integrated intensities two LIR relations were obtained, by the ratio of emissions from $^2H_{11/2}$ and $^4S_{3/2}$, and by $^4F_{7/2}$ and $^4S_{3/2}$ levels. Fitting to the Boltzmann relation by both LIRs the obtained energy gaps are similar to those reported in the literature. The energy gap in the LIR by using $^4F_{7/2}$ and $^4S_{3/2}$ levels is 3.5-fold greater than that obtained by the conventional LIR by using the first two thermalized levels, providing several times greater relative sensitivity. However, low intensity from the 3rd thermalized level prevents this method from being used at relatively lower temperatures. Thus, for the best utilization of YAG:Yb^{3+}/Er^{3+} nanoparticles, it is recommended to combine both LIRs depending on the measured temperature range.

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Odnos intenziteta luminescencije tri termalizovana nivoa u YAG:Er^{3+}/Yb^{3+}nanočesticama

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(Naučni rad)

Izvod

Luminiscentna termometrija je metoda za očitavanje temperature pomoću daljinskog senzora temperature posmatranjem temperaturno zavisnih spektralnih promena. Potraga za povećanom osetljivosti očitavanja temperature motivisala je istraživanje korišćenja trećeg termalizovanog nivoa Er^{3+} emisije u fotoluminiscenciji Yb^{3+}/Er^{3+}. Za ovu svrhu pripremljene su veoma stabilne i efikasne nanočestice YAG:Yb^{3+}/Er^{3+} modifikovanom Pećinijevom metodom. Emisioni spektri su snimljeni u temperaturnom opsegu od 300 K do 800 K i dobijena su dva odnosa intenziteta luminiscencije između emisija $^4S_{3/2}$, $^2H_{11/2}$ i $^4F_{7/2}$. Pored odličnog poklapanja sa teorijskim predviđanjima, očitavanje korišćenjem $^4F_{7/2}$ metode je omogućilo 3,5 puta povećanje relativne osetljivosti u odnosu na odnos intenziteta luminiscencije za nivo $^2H_{11/2}$, uz ograničenje upotrebljivosti samo iznad 600 K. Metoda emisije iz $^2H_{11/2}$ treba da se koristi od 300 K do 600 K, dok emisija od $^4F_{7/2}$ obezbeđuje najbolji odnos intenziteta luminiscencije od 600 K do 800 K. YAG:Yb^{3+}/Er^{3+} nanočestice su se pokazale kao odličan senzorski materijal za luminescentnu metodu odnosa intenziteta korišćenjem više termalizovanih nivoa.

Ključne reči: luminiscentna termometrija; apkonvertujuće nanočestice; odnos intenziteta fluorescencije; Pećini metod