Green upconversion in $\text{Y}_2\text{O}_3:\text{Yb}$ nanopowder

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Abstract. Green emission lines, in addition to the blue and the red, were observed upon 980 nm excitation in yttrium oxide (Y$_2$O$_3$) nanopowder codoped with Yb$^{3+}$ and Tm$^{3+}$, synthesized by the chemical combustion method. Upconversion emission studies suggest that the number and characteristics of the green lines are influenced by the annealing temperature as well as by the Yb$^{3+}$/Tm$^{3+}$ concentration ratio, opening possibilities for new customized applications. The chromaticity properties of the upconversion spectra were quantified by the Commission Internationale de l’éclairage coordinate analysis. © 2015 Society of Photo-Optical Instrumentation Engineers (SPIE) [DOI: 10.1117/1.JNP.9.093054]

Keywords: upconversion luminescence; optical properties; nanophosphor; fluorescence spectroscopy; Commission Internationale de l’éclairage coordinate.

Paper 15043 received Jun. 4, 2015; accepted for publication Aug. 3, 2015; published online Sep. 11, 2015.

1 Introduction

In recent years, rare earth (RE) doped nanocrystalline materials have received considerable interest due to their unique properties for the potential applications in many fields of optoelectronics, photonics, and nanobiotechnology.1–5 The reason for this lies in the fact that such materials are able to convert near-infrared (NIR) light into light with a shorter wavelength: ultraviolet (UV), visible, and shorter NIR.

In the upconversion (UC) processes, the characteristics of host materials play an important role. Many host materials have been developed, such as Y$_3$Al$_5$O$_{12}$,6 Al$_2$O$_3$,7 MgAl$_2$O$_3$,7 Gd$_2$O$_3$,8 yttrium oxide (Y$_2$O$_3$),9,10 and NaYF$_4$,11,12 with high UC emission efficiency. Among these materials, Y$_2$O$_3$ is a suitable host material for doping with RE ions like ytterbium (Yb$^{3+}$), holmium (Ho$^{3+}$), erbium (Er$^{3+}$), europium (Eu$^{3+}$), praseodymium (Pr$^{3+}$), and thulium (Tm$^{3+}$).

RE doped Y$_2$O$_3$ materials are promising for numerous photonics applications because of their excellent physical properties and chemical and photochemical stability, as well as high luminescence efficiency. Y$_2$O$_3$ has a high melting point (2450°C), high thermal conductivity, broad transparency range (0.2 to 8 μm), and high refractive index (~2).13 Beside this, the non-radiative relaxation rate is relatively small for RE ions in Y$_2$O$_3$ host material, which can greatly enhance the luminescence emission.12,14,15

Tm$^{3+}$ ions exhibit strong luminescence in the UV, blue, and red regions, and do not absorb 980 nm radiation. The population of the upper levels of Tm$^{3+}$ ions can be achieved through energy transfer from a sensitizer. The studies show a significant increase in the UC yield when the host material is codoped with Yb$^{3+}$ as a sensitizer and Tm$^{3+}$ as an activator.16–18

The absorption cross-section of Yb$^{3+}$ ion near 980 nm is quite large, so Yb$^{3+}$ is well equipped to actively transfer the absorbed energy to other RE codopants. Because its energy levels are resonant with the f–f transitions of many lanthanide activators (Pr$^{3+}$, Ho$^{3+}$, Er$^{3+}$, and Tm$^{3+}$), it is favorable to codope RE ions with Yb$^{3+}$ to increase the UC efficiency. Depending on the activators, different UC emissions can be obtained with NIR excitation.

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Several works have examined the UC emission of Yb$^{3+}$ and Tm$^{3+}$ doped Y$_2$O$_3$. They are mainly focused on strong blue and weak red emission of Tm$^{3+}$ ions. Up until now, the green UC region has been mentioned (535 to 560 nm) only in a few studies. Here, we report efforts to enhance UC emission in the green region in Y$_2$O$_3$:Yb$^{3+}$, Tm$^{3+}$ nanopowders and to improve UC luminescence characterization of these nanopowders. We analyze the influence of the annealing temperature on the existence of UC emission in the green region, for nanopowders prepared via solution combustion synthesis. The influence of the Yb$^{3+}$/Tm$^{3+}$ concentration ratio on green UC lines is also discussed.

2 Experimental

Yb- and Tm-doped Y$_2$O$_3$ nanopowders were prepared by solution combustion synthesis. The solution of 18.38 g (192 mol %) of Y(NO$_3$)$_3$·6H$_2$O, 0.674 g (6 mol %) of Yb(NO$_3$)$_3$·5H$_2$O and 0.045 g (0.4 mol %) Tm(NO$_3$)$_3$·5H$_2$O was used as the reagent grade. These compounds were dissolved in a minimum amount of demineralized water. Then 7.508 g (500 mol %) of urea was added to the solution as the organic fuel. After preparation, the starting reagents were combusted with the flame burner at 500°C. The intensive exothermic reaction yielded a voluminous foamy powder. The synthesized products were annealed at two different temperatures, 1200 and 1400°C, for 2 h. All chemicals were commercially available and used in synthesis procedures without further purification.

The x-ray powder diffraction (XRD) patterns of the title nanocrystalline powder annealed at 1200 and 1400°C were collected using automatic two-circle powder diffractometer Seifert MZ IV using CuK$_\alpha$ radiation ($\lambda = 0.15406$ nm) monochromatized by the Ni $\beta$-filter. The x-ray tube was operated at 40 kV and 30 mA. Data were obtained within 2$\theta$ in the range of 10 to 120 deg, with a step of 0.02 deg/s.

Optical properties of Y$_2$O$_3$:Yb$^{3+}$, Tm$^{3+}$ were studied by upconversion photoluminescence (UPL) spectroscopy. UPL spectra were obtained upon exciting the sample with a 980 nm laser diode with an incident radiation power of 1500 mW. All upconverted signals passed through an optical bandpass filter (400 to 700 nm) and were collected by an Ocean Optics USB4000 spectrometer.

3 Results and Discussion

3.1 Characterization

The recorded diffractograms of codoped Y$_2$O$_3$ samples were compared with the JCPDS file no. 41-1105 and 83-0927 for pure Y$_2$O$_3$, as shown in Fig. 1, for the sample annealed at 1200°C. The sharp single peaks of the XRD patterns unambiguously confirm the formation of single phase compounds, which is attributable to the fact that the annealing process took place uniformly throughout the volume of the samples. All peaks match well with those of pure cubic Y$_2$O$_3$ (no peaks were observed due to dopants), thereby suggesting a highly crystalline body-cubic structure ($a = 1.06$ nm) belonging to the space group Ia$\bar{3}$ (SG206) and no structural phase transition during annealing.

The average size of the nanopowders was estimated from the linewidth of the XRD peaks using the well-known Scherrer’s equation $d = \frac{-K\lambda}{\beta\cos\theta}$, where $K = 0.9$ is a constant related to the nanopowder shape, $\lambda = 0.154$ nm is the wavelength of the x-ray radiation used, $\beta$ is the full width at half maximum (FWHM) of the diffraction peak in the XRD patterns in radians, and $\theta$ is the Bragg diffraction angle. The nine peaks [(211), (222), (400), (411), (332), (341), (431), (440), (622 nm)] have been selected for this determination. The average sizes of the nanopowders annealed at 1200 and 1400°C are 70.6 (23) nm and 74.8 (23) nm, respectively.

As the correction of the linewidths for the instrumental broadening was not included, we assume that the average nanopowder sizes are, in fact, somewhat greater than what is stated above. As the profiles of the peaks in the diffraction patterns of the samples are predominantly Gaussian, both size, $D$, and microstrain, $\varepsilon$, in the nanopowder were estimated by the Williamson-Hall (W-H) analysis. For a Gaussian line, the W-H relation is
\[ (\Gamma^2)_{G} \cos^2 \theta = \left( \frac{K\lambda}{D} \right)^2 + 16\epsilon^2 \sin^2 \theta, \] (1)

where \( \Gamma \) is the FWHM, and \( \epsilon \) represents the average microstrain present in the nanopowders. The plot \( (\Gamma^2)_{G} \cos^2 \theta \) versus \( 16\epsilon^2 \sin^2 \theta \) yields a straight line. The size was determined from the intercept of the linear fit with the axis, and the microstrain from the slope. The sizes are \( D_{W,H(1200)} = 70.5(3) \) nm and \( D_{W,H(1400)} = 74.7(4) \) nm, while the microstrain is the same for both samples, i.e., \( \epsilon = 1.96 \cdot 10^{-3} \), considering the uncertainty.

The grain size of the sample, which was annealed at 1400°C, was estimated based on the ratio of the half-width of the diffraction lines to those of the sample annealed at 1200°C. It was found that \( D_{eff}^{1400} = \left( \frac{H_{w}^{1200}}{H_{w}^{1400}} \right) D_{eff}^{1200} = 1.066(3) \cdot D_{eff}^{1200} \), where \( H_w \) is the FWHM. Hence, the grain is greater by 7% relative to the grain of the sample annealed at 1200°C.

### 3.2 Upconversion

The UC spectra of \( \text{Y}_2\text{O}_3:\text{Yb}^{3+}, \text{Tm}^{3+} \) nanopowders upon 980 nm excitation were obtained in the wavelength range of 400 to 750 nm at room temperature. The spectra exhibit two major bands centered at around 485 nm (blue emission) and 660 nm (red emission), and a minor band around 550 nm (green emission), as seen in Fig. 2. As the bands are separated, we performed the Gaussian–Lorentzian deconvolution analysis dividing the UC spectra into three ranges: 400 to 510 nm, 510 to 580 nm, and 580 to 750 nm. The decomposed individual Gaussian–Lorentzian peaks for these ranges are shown in Fig. 3. The schematic diagram of the upconversion processes is shown in Fig. 4.

In the first region (400 to 510 nm), five evident UC emissions are resolved, centered at 465 nm, (transition \(^1\text{D}_2 \rightarrow ^3\text{F}_4\) of \( \text{Tm}^{3+} \) ion), 477 nm \((^1\text{G}_4 \rightarrow ^3\text{H}_6)\), and 488 nm \((^1\text{G}_4 \rightarrow ^3\text{H}_6)\). The emission observed at 488 nm has the highest amplitude. For both samples, the first three peaks have the same peak positions within experimental errors.

The situation in the third region is quite different, where the wavelength ranges 580 to 750 nm and the difference in the overall emission intensity can be observed as well as a difference in the number of emission lines (Table 1). This is especially evident in the range 612 to 640 nm, where for the sample annealed at 1200°C, two emission peaks appear at 612 and 632 nm, both due to transitions \(^1\text{G}_4 \rightarrow ^3\text{F}_4\) of \( \text{Tm}^{3+} \) ion. On the other hand, the sample annealed...
at 1400°C has an emission peak at 632 nm, also due to the transition $^1G_4 \rightarrow ^3F_4$. Interestingly, the next positions of emission peaks 653, 665, 673, 686, and 697 nm for both samples are identical, taking into account the experimental errors.

Compared to the emissions in the blue and the red regions, the intensity peaks in the green region (510 to 580 nm) are considerably smaller. These weak UC emissions are the results of the energy transfer $^1D_2 \rightarrow ^3H_5$ of Tm$^{3+}$ ions. The reason for the low intensity is the very small cross-energy transfer rate of $^1D_2 \rightarrow ^3H_5$ transitions.\textsuperscript{27} For the sample annealed at 1200°C, we found two green UC lines (525 and 546 nm). These results are in good agreement with data reported by Mishra et al.\textsuperscript{24} for Y$_2$O$_3$:Yb$^{3+}$, Tm$^{3+}$, synthesized by the combustion technique and annealed at 1200°C (Table 1). They observed two UC emissions (530 and 555 nm), for ytterbium versus thulium concentration ratio (Yb$^{3+}$/Tm$^{3+}$) of 5. The difference in the position of the green lines between our data and the data from Mishra et al.\textsuperscript{24} can be attributed to the different Yb$^{3+}$/Tm$^{3+}$ ratio. For the sample annealed at 1400°C, four UC emission lines were found (524, 538, 550, and 564 nm). The green emission band for the sample annealed at 1200°C is significantly weaker. The weak emissions in the green region were also reported in different host codoping with Yb$^{3+}$ and Tm$^{3+}$ ions (in Ref. 28 at 574 nm, in Ref. 12 at 514 nm, and in Ref. 29 at 511 nm).

In the literature, several ways are described explaining how the $^1D_2$ level of Tm$^{3+}$ ions in different host materials is populated.\textsuperscript{30-32} Some controversy exists about the possibility of the population of the $^1D_2$ level via a fourth nonresonant transfer of energy from Yb$^{3+}$ ion to Tm$^{3+}$ ion in the $^1G_4$ level. The phonon-assisted energy transfer probability is governed by the energy-gap law $W = W_0 \exp(-k\Delta E)$, where $W_0$ is the energy transfer probability when the zero phonon lines overlap, $k$ is the material constant determined by the characteristics of the host material that surrounds the optically active ions, and $\Delta E$ is the energy gap between the electronic levels of the donor and acceptor ions.\textsuperscript{32} The population of the $^1D_2$ level is a result of the energy transfer $^2F_{5/2}(\text{Yb}^{3+}) + ^1G_4(\text{Tm}^{3+}) \rightarrow ^1D_2(\text{Tm}^{3+}) + ^2F_{7/2}(\text{Yb}^{3+})$. These transitions occur with little probability due to large energy mismatch ($\sim 3500 \text{ cm}^{-1}$) between Yb$^{3+}$ ($^2F_{5/2} \rightarrow ^2F_{7/2}$) and Tm$^{3+}$ ($^1G_4 \rightarrow ^1D_2$).\textsuperscript{24,30,32} Due to this, the intensities of the lines arising from $^1D_2$ should be very small. Contrary to this, it was reported for fluorescence transitions $^1D_2 \rightarrow ^3H_6$, the $^1D_2$ level of Tm$^{3+}$ is not populated via the fourth photon from Yb$^{3+}$ via energy transfer to $^1G_4$.\textsuperscript{23,33}

On the other hand, a cross-relaxation process may alternatively play an important role in population of the $^1D_2$ level of Tm$^{3+}$ ions. According to data from the literature, the energy transfer between Tm$^{3+}$ ions can take several possible routes.\textsuperscript{3,19,23,24,26,30}

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**Fig. 2 Measured upconversion spectra of Y$_2$O$_3$:Yb$^{3+}$, Tm$^{3+}$ nanopowders annealed at 1200 and at 1400°C. The sample was excited at 980 nm.**
Fig. 3 Deconvolution of upconversion spectra for $\text{Y}_2\text{O}_3: \text{Yb}^{3+}, \text{Tm}^{3+}$ annealed at 1400°C: (a) blue region, wavelength 400 to 520 nm, (b) green region, wavelength 510 to 580 nm, and (c) red region, wavelength range 600 to 750 nm.
The population of the $^{1}\text{D}_2$ increases with increasing Tm$^{3+}$ concentration. This causes a decrease in $^{1}\text{G}_4$ and other levels’ populations. In this work, the ratio of Yb$^{3+}$/Tm$^{3+}$
concentration was 15, while in the work of Mishra et al., it was 5 and the annealing temperature was 1200°C.

To better understand the UC mechanisms of Tm³⁺ and Yb³⁺ ions which lead to green emission lines in the Y₂O₃ matrix, we compare the emission UC intensity of different emission bands (Fig. 5). The data were integrated numerically. The integrated area, \( A \), of blue transitions (\( 1D_2 \rightarrow 3F_4 \) and \( 1G_4 \rightarrow 3H_6 \)) is somewhat higher (~6%) for the sample annealed at 1400°C compared with the sample annealed at 1200°C. Unlike the blue emission band, a significantly higher increase in ratio for emission intensity, \( A_{1400}/A_{1200} \), is observed for the red (~80%) and the green (~60%) emission lines.

With higher annealing temperature, the integrated intensity ratio of red to green emission bands \( A'/A'\theta \) increases from 9.8 to 11. The red and the green emission intensities are proportional to the population of the \( 1G_4/3F_{2,3} \) and \( 1D_2 \) states, respectively. An increase in ratio indicates that...
\( ^1D_2 \) and \(^3F_{2,3} \) states were populated more efficiently than the \( ^1D_2 \) state for the sample annealed at a higher temperature.

In contrast to \( A'/A'' \), the ratio of blue to green emission bands, \( A''/A' \), significantly decreases with increasing annealing temperature from 38 to 25. Both the blue and the green emission intensities depend on the population \( ^1D_2 \) (line 465 nm). Moreover, the blue emission depends on the population of the \( ^1G_4 \) state. On the other hand, the contribution of the line at 465 nm to total emission from transition \( ^1D_2 \rightarrow ^1F_4 \) in the blue bands is small and slightly changed (from 1 to 1.24%) with a rise in the annealing temperature. This indicates that the \( ^1D_2 \) state became more populated than the \( ^1G_4 \) state for the sample annealed at a higher temperature. Due to this, with higher annealing temperature, the intensity of the green emission from transition \( ^1D_2 \rightarrow ^3H_5 \) increases more and the blue emission from \( ^1G_4 \rightarrow ^3H_6 \) increases only slightly.

To identify the number of photons \( n \), which are required to populate the upper emitting state for the green emission process under unsaturated conditions, the dependence of UC fluorescence intensity on incident pump power is studied. The dependence of UC fluorescence intensity, \( I_{up} \), on incident pump power, \( P \), can be obtained according to the relation

\[
I_{up} \propto P^n.
\]

Hence, the number of photons, \( n \), can be estimated from the slope of the line \( \ln(I_{up}) \) versus \( \ln(P) \). The linear least squares fit approach was used to obtain these slopes. The results for selected emission bands for the two samples are presented in Fig. 6.

The slopes for the sample prepared at a higher temperature (1400°C) are somewhat greater than the slopes for the sample prepared at the lower temperature (1200°C). This difference is most pronounced (\( \sim 20\% \)) for the band at 654 nm. The values for this line are 3.8 ± 0.3 (1200°C) and 4.57 ± 0.13 (1400°C). Taking into account the uncertainty, the obtained data for the slope indicate that four (1200°C) and five (1400°C) photons are necessary to populate the \( ^1G_4 \) state. The blue line at 488 nm shows less dependence on the annealing temperature. The calculated values are 4.12 ± 0.29 (1200°C) and 4.43 ± 0.25 (1400°C), indicating that four (possibly five) photons are involved to generate this UC line. To our knowledge, such values for slope have not been reported in the literature for \( Y_2O_3:Yb, Tm \), or for any other host doped with \( Yb^{3+} \) and \( Tm^{3+} \) ions. Usually, the blue and the red UC emission lines are reported to be realized via two to three photons.\(^9,24,29\)

Interestingly, the green band also shows a deviation from the data in the literature. A fourth energy transfer has been proposed to populate the \( ^1D_2 \) level.\(^24\) For the weak green UC emission line at 511 nm detected in a different host (NaYF\(_4\); with \( Yb/Tm = 60 \)), it was found that four photons are involved in its generation.\(^34\) Here, the power dependence gives smaller values:
3.0 ± 0.8 (1200°C) and 3.14 ± 0.27 (1400°C) (Fig. 6). There is evidently a higher uncertainty in the slope for the sample annealed at 1200°C, arising from the nonlinear fitting/deconvolution process to identify the green lines, which are less intense compared with the intensity of green lines in the sample annealed at 1400°C. This indicates that three photons are involved in the pump process for the $^1D_2$ state of the Tm$^{3+}$ ion. Stecher et al.34 observed a smaller number of photons (two) for the 511 nm line in (Na)LaF$_3$:Yb, Tm (with Yb/Tm = 40). We believe that the $^1D_2$ level of Tm$^{3+}$ is populated by the four photon process, but the rapid nonradiative relaxation to the level $^1G_4$ appears to reduce the number of photons involved in the green emission. At the same time, this process contributes to the increased population of the level $^1G_4$. Hence, four/five photons participate in generating the red and the blue bands. The described behavior can be attributed to the combined effects of the improved nanopowder crystallinity (effect of annealing temperature), Yb/Tm ratio (i.e., concentration of dopant ions), crystal structure, and host materials.

Mechanisms responsible for populating the $^1D_2$ state are the cross-relaxation processes in the Y$_2$O$_3$:Yb$^{3+},$ Tm$^{3+}$ nanopowder. The results show that these processes become more pronounced for the higher annealing temperature and higher Yb$^{3+}$/Tm$^{3+}$ concentration ratios leading to increased green UC emission ($^1D_2$ → $^3H_5$).

Annealing of the Y$_2$O$_3$:Yb, Tm nanopowder at 1400°C was reported only in the work Giri et al.,35 but no green band was mentioned, although the ratio Yb$^{3+}$/Tm$^{3+} = 5$ is the same as in the work of Mishra et al.24 One of the possible reasons may be that with increasing annealing temperature, the average nanopowder size is increased.

### 3.3 Chromaticity Properties of Upconversion Emission

The chromaticity properties of UC emission have been quantified in terms of the The Commission Internationale de l’éclairage (CIE) 1931 chromaticity diagram.36,37 The CIE coordinates depending on the annealing temperature (1200 and 1400°C), as well as on the pump excitation powers from 500 to 1000 mW were determined. The variation in CIE color on the annealing temperature is evident and is shown on the CIE chromaticity diagram (Fig. 7).

![Fig. 7](image-url)
This behavior suggests that nanopowder size has a significant influence on its UC emission color.

The chromaticity coordinate for the nanopowder sample annealed at 1200°C lies in the pink and the orange-pink region of the CIE diagram. On the other hand, the sample annealed at 1400°C lies closer to white light and partly in the yellow region, although with a decrease in pump excitation power, the UC intensity decreases and there is no change in the peak positions of all emission bands. The obtained results show that the UC emission color of Y₂O₃:Yb, Tm can be tuned by the introduction of the green UC line, which can be achieved by changing the annealing temperature (nanocrystal size), the Yb³⁺/Tm³⁺ concentration ratio, and the incident laser power.

4 Conclusion

The analysis of green UC emission lines in Y₂O₃:Yb³⁺, Tm³⁺ nanopowders prepared by solution combustion synthesis showed that the annealing temperature has an important influence on the UC emission spectra. Increasing the annealing temperature for 200°C resulted in ~7% greater nanopowder average size, while the number of emitting green lines doubled and the integrated emission intensity increased by 60%. Another factor influencing the shape and intensity of the green lines is the Yb³⁺/Tm³⁺ concentration ratio.

The impact of different influencing factors on the UC spectra is quantified and characterized by the chromaticity coordinate in the CIE diagram.

With this knowledge, custom design of the UC emission spectra is possible and the use of Y₂O₃:Yb³⁺, Tm³⁺ nanopowder shows a promising potential for different but related applications, such as biolabeling, white light sources, back lighting, three-dimensional displays, etc.

Acknowledgments

This work has been done within the projects MESTD RS OI 171020, Bilateral Project Serbia–Slovenia #651-03-1251/2012-09/03, project MESTD RS III 45008 as part of the development of new materials in nanotechnology, and project BI-RS/12-13-003 (SLO Research Agency).

References


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