



Tb³⁺→Eu³⁺ energy transfer processes in crystals of TbAl₃(BO₃)₄ doped with Eu³⁺

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ABSTRACT

In this contribution, the dynamics of the energy transfer process involving Tb³⁺ ion in samples of trigonal huntite-type Tb_{1-x}Eu_xAl₃(BO₃)₄ (with x = 0, 0.01, 0.05, 0.1, 0.15 and 0.2) single crystals has been investigated upon fitting the Tb³⁺ ⁵D₄ luminescence decay curves with suitable mathematical equations. In the undoped TbAl₃(BO₃)₄ compound, the Inokuti-Hirayama model properly fits the decay, so that the dominant ⁵D₄ relaxation channel is the direct transfer of the excitation energy to the quenching impurity (Mo³⁺ or other crystal defects) at short times after the excitation pulse. On the other hand, the transfer of the excitation energy to this quenching impurity through energy migration is negligible. As for the Eu³⁺ doped samples, a generalization of the Parent et al. model has been employed. In this case, in the fitting equation the contribution of four different terms has been considered and calculated, one for each possible relaxation channel, that is: i) the intrinsic Tb³⁺ decay, ii) the direct (not assisted by energy migration) Tb³⁺→Mo³⁺ (or other crystal defects) energy transfer, iii) the direct (not assisted by energy migration) Tb³⁺→Eu³⁺ energy transfer and iv) the Tb³⁺→Eu³⁺ energy transfer assisted by energy migration in the Tb³⁺ sublattice. From a semi-quantitative point of view, our investigation aligns with what has been qualitatively previously concluded on Tb³⁺→Eu³⁺ energy transfer dynamics in TbAl₃(BO₃)₄, which would be ruled by an intermediate regime where the rate of the energy transfer among donors (Tb³⁺) is comparable to the rate of the Tb³⁺→Eu³⁺ energy transfer. More in detail, our study underlines the increasing impact of the term *W* on the ⁵D₄ decay with the increase of the Eu³⁺ concentration. This term is directly connected with the energy migration phenomenon involving the Tb³⁺ subarray.

1. Introduction

Non radiative Tb³⁺→Eu³⁺ energy transfer processes have been broadly investigated in inorganic-based materials [1–3] as, apart from the intrinsic interest related to the basic science, it is crucial to design efficient luminescent materials emitting in the visible spectral regions for the development of many optical devices (e.g. LEDs, phosphors, optical thermometers, and many others). In fact, the sensitization of the visible Eu³⁺ luminescence (located in the red spectral region) by Tb³⁺ ions can significantly increase the luminescence output of the former ion and efficient phosphors for red emitting LEDs can be obtained. Another very intriguing aspect of the Tb³⁺→Eu³⁺ energy transfer process is related to the possible tuning of the color of the light emitted. In inorganic crystalline solids, while a dominant red emission can be observed upon excitation into the Tb³⁺ sublattice when the efficiency of the Tb³⁺→Eu³⁺ energy transfer (η_{ET}) is close to 100 %, yellow or orange emissions are observed when η_{ET} is lower. Representative compounds belonging to the first class are samples doped with a small molar percentage of Eu³⁺ (typically 1 %) of the cubic Sr₃Tb(PO₄)₃ [4], tetragonal TbPO₄ [5] and orthorhombic Ca₃Tb₂Si₃O₁₂ [6]. At similar Eu³⁺ doping

level, much lower η_{ET} are observed for rhombohedral Ca₉Tb(PO₄)₇ [7] and trigonal TbAl₃(BO₃)₄ [8] hosts and as mentioned before the color of the emitted light is due to a weighted combination of green (from Tb³⁺) and red (from Eu³⁺) light. Undoped and doped TbAl₃(BO₃)₄ compound has been extensively studied, as for its spectroscopic properties [9–14], as well as for the magnetic features [15–17]. In a recent contribution by some of us [8], in agreement with the study performed by Kellendonk and Blasse on the neat TbAl₃(BO₃)₄ [18], it has been proposed that, upon excitation of the Tb³⁺ ions, a diffusion limited migration of the excitation energy in the ⁵D₄ level of the Tb³⁺ subset of cations occurs, followed by the energy transfer process to quenching impurities (*i.e.* the unintentionally introduced Mo³⁺ killer centers). In this contribution, thanks to a close inspection of the decay dynamics of the Tb³⁺ ⁵D₄ excited state, we propose a new description of the energy transfer processes to acceptor species (Mo³⁺ or other crystal defects and Eu³⁺) occurring in Tb_{1-x}Eu_xAl₃(BO₃)₄ single crystals (with x = 0, 0.01, 0.05, 0.1, 0.15 and 0.2) on the basis of a generalization of the Inokuti-Hirayama model [19] based on the Parent et al. model [20] adapted for different acceptors (unintentional and intentional impurities).

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2. Experimental

$Tb_{1-x}Eu_xAl_3(BO_3)_4$ (with $x = 0, 0.01, 0.05, 0.1, 0.15$ and 0.2), hereafter $TbAl_3(BO_3)_4$, $TbAl_3(BO_3)_4:x\%Eu$, with $x = 1, 5, 10, 15$ and 20), have been synthesized as single crystals as reported in the literature [8].

Room temperature decay curves were measured with a Fluorolog 3 (HoribaJobin Yvon) spectrofluorometer, equipped with a Xe lamp, a double excitation monochromator, a single emission monochromator (mod. HR320) and a photomultiplier in photon counting mode for the detection of the emitted signal.

3. Results and discussion

As described in details in ref.8, the $Tb^{3+} \rightarrow Eu^{3+}$ energy transfer efficiency (η_{ET}) is strongly dependent on the Eu^{3+} concentration and it ranges from 17 % for $TbAl_3(BO_3)_4:1\%Eu$ to 80 % in the case of $TbAl_3(BO_3)_4:20\%Eu$. Accordingly, upon normalization of the emission spectra at the area of the ${}^5D_4 \rightarrow {}^7F_5$ peak of Tb^{3+} (centered at 545 nm), we observed a significantly stronger Eu^{3+} emission for the latter sample (Fig. 1).

The modulation of the emission color, discussed in the introduction, can be found in the best way upon inspection of the International Commission on Illumination (CIE) diagram of the whole family of the investigated borates [$TbAl_3(BO_3)_4$ and $TbAl_3(BO_3)_4:x\%Eu$ ($x = 1, 5, 10, 15$ and 20); Fig. 2].

The red/orange emission of $TbAl_3(BO_3)_4:20\%Eu$ turns to orange in $TbAl_3(BO_3)_4:5\%Eu$, yellow in $TbAl_3(BO_3)_4:1\%Eu$ and finally green in the undoped $TbAl_3(BO_3)_4$.

3.1. Energy transfer in $TbAl_3(BO_3)_4$

The energy transfer mechanism occurring in $TbAl_3(BO_3)_4$ in the 1.4–300 K temperature range has been investigated in the past by Kellendonk and Blasse [18]. At room temperature, they observed a diffusion-limited migration of the Tb^{3+} excitation energy to the impurity killer centers (Mo^{3+}). The rate of transfer among the donors is lower than the rate of transfer from donors to acceptors, so that the decay of the donor system is governed by two processes. The decay of the donors is non-exponential for relatively short times after the excitation pulse due to direct transfer to nearby acceptor ions. For long times after the pulse the decay becomes exponential in the case of diffusion due to the influence of migration within the donor system. The decay time derived

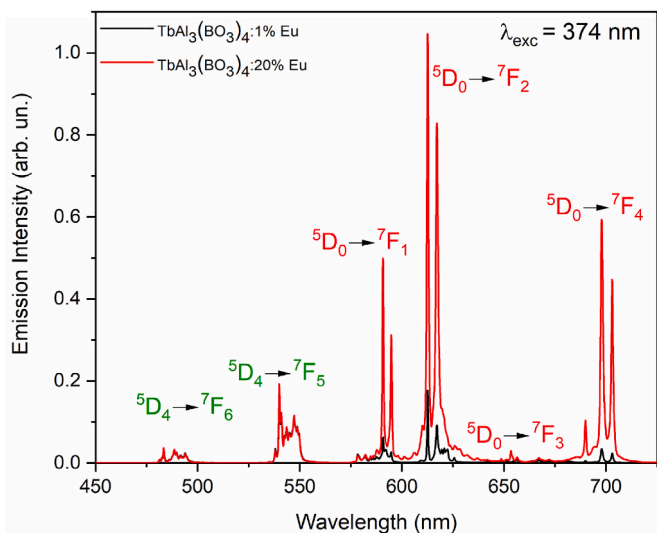


Fig. 1. Evolution of the emission spectra of $TbAl_3(BO_3)_4:x\%Eu$ ($x = 1$ and 20) upon excitation at 374 nm, into the 5D_3 level of Tb^{3+} . All the spectra are normalized at the area of the ${}^5D_4 \rightarrow {}^7F_5$ peak (centered at 545 nm).

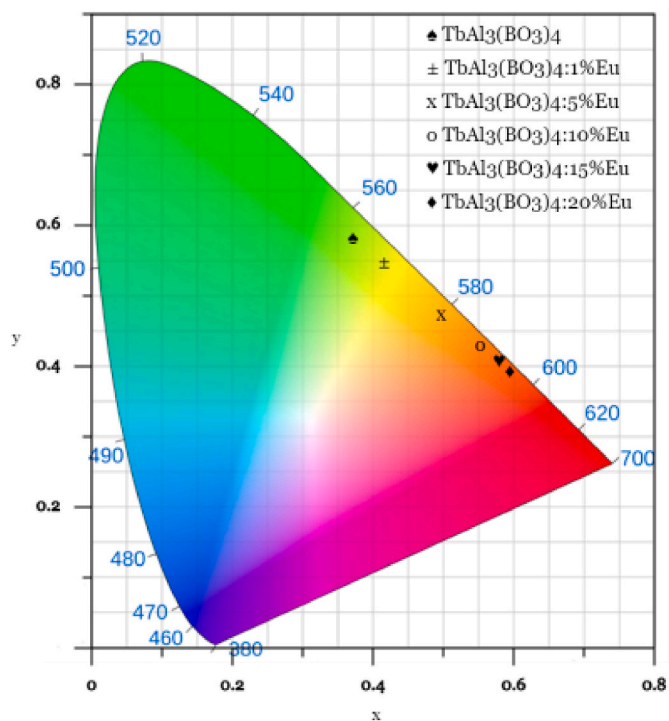


Fig. 2. CIE color space chromaticity diagram of the $TbAl_3(BO_3)_4$ and $TbAl_3(BO_3)_4:x\%Eu$ ($x = 1, 5, 10, 15$ and 20) family of compounds. $\lambda_{exc} = 374$ nm. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

from this exponential part is shorter than the radiative rate. Concentration quenching phenomenon is working since this decay time (1.42 ms) is significantly shorter than the lifetime recorded in $YAl_3(BO_3)_4:1\%Tb$ (1.9 ms), where the energy migration among Tb^{3+} ions and consequently the concentration quenching process can be neglected. Surprisingly, in our case the decay time related to the tail of the decay curve is 1.70 ms, which is close to the 1.9 ms value, that can be considered in first approximation as the radiative decay time of Tb^{3+} in this host. This allows us to conclude that in our crystals, the concentration quenching is less important, maybe thanks to a high quality of the crystals grown. In this condition, the probability with which the exciton migrating energy can be quenched by impurities is lower. Therefore in first approximation, we can neglect the transfer of the excitation energy to quenching impurities through energy migration and fit the decay curve of $TbAl_3(BO_3)_4$ by means of the Inokuti-Hirayama model [19]. The best fitting curve (see Fig. 3) is the one represented by the following equation:

$$I(t) = I(0) \exp \left[-\frac{t}{\tau_{5D4}} - Q_{5D4} \left(\frac{t}{\tau_{5D4}} \right)^{3/5} \right] \quad (1)$$

Considering a dipole-dipole interaction ($S = 6$) and a lifetime of $\tau_{5D4} = 1.70$ ms. The low value of $Q_{5D4} = 0.27$ supports the occurrence, for relatively short times after the excitation pulse, of a direct transfer of the excitation energy to nearby quenching impurity (Mo^{3+} or other crystal defects). It is important to remind that:

$$Q = \frac{4\pi}{3} \Gamma \left(1 - \frac{3}{S} \right) (C_{DA} \tau_{5D4})^{3/5} \quad (2)$$

where Γ is the gamma function and C_{DA} [19] represents the micro-parameter related to the direct energy transfer from Tb^{3+} to the impurities (acceptors).

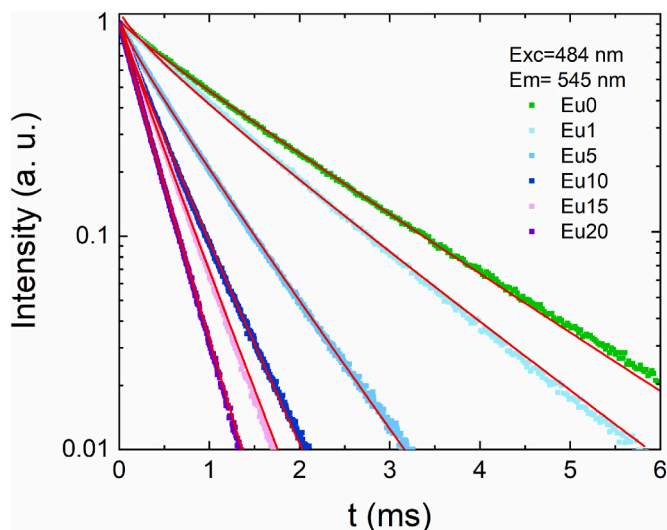


Fig. 3. Luminescence decays of the 5D_4 level of Tb^{3+} ions, upon excitation in the same level for the samples $TbAl_3(BO_3)_4$ and $TbAl_3(BO_3)_4:x\%Eu$, with $x = 1, 5, 10, 15$ and 20 . The red lines represent the fitting model described by equation (1) or (5). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

3.2. Energy transfer in $TbAl_3(BO_3)_4:x\%Eu$, with $x = 1, 5, 10, 15$ and 20

When the samples are doped with Eu^{3+} ions now we can generalize the previous equation. In particular, in the paper of Blumen and Manz [21] they generalized eq. (1) considering the energy transfer mechanism to different acceptors or traps. Therefore,

$$I(t) = I(0) \exp \left[-\frac{t}{\tau_{5D4}} - Q \left(\frac{t}{\tau_{5D4}} \right)^{3/5} \right] \quad (3)$$

Where:

$$Q = \frac{4\pi}{3} \Gamma \left(1 - \frac{3}{5} \right) \sum_{a=1}^A C_{Aa} (C_{DAa} \tau)^{3/5} \quad (4)$$

and the summation is extended to all number of acceptors (A). In this direction, we decide to generalize the Parent et al. model [20] applied in the case of $Nd^{3+} \rightarrow Yb^{3+}$ energy transfer in $LiLnP_4O_{12}$ glasses ($Ln = La, Nd$ and Yb), considering the different possible acceptors. Therefore, we proposed the following equation to model the decay curves of the $TbAl_3(BO_3)_4:x\%Eu$, with $x = 1, 5, 10, 15$ and 20 samples:

$$I(t) = I(0) \exp \left[-\frac{t}{\tau_{5D4}} - Q_{5D4} \left(\frac{t}{\tau_{5D4}} \right)^{3/5} - Q_{TbEu} \left(\frac{t}{\tau_{5D4}} \right)^{3/5} - Wt \right] \quad (5)$$

Where Q_{5D4} and Q_{TbEu} characterized the direct $Tb^{3+} \rightarrow Mo^{3+}$ (or other crystal defects) and $Tb^{3+} \rightarrow Eu^{3+}$ energy transfer without energy migration within Tb^{3+} population, respectively, while W represents the influence of the energy migration function over of the Tb^{3+} donor subarray. W is proportional to the product of the donor (Tb^{3+}) and acceptor (Eu^{3+}) concentrations and it is related to $C_{DD(Tb-Tb)}$ and $C_{DA(Tb-Eu)}$, the microparameters for $Tb^{3+} \rightarrow Tb^{3+}$ energy migration and $Tb^{3+} \rightarrow Eu^{3+}$ energy transfer, respectively.

As can be seen in Fig. 3, good fittings using Eq. (5) are obtained for the samples doped with Eu^{3+} with $S = 6$, and with the W values reported in Fig. 4. In all the fittings has been used a $\tau_{5D4} = 1.70$ ms and $Q_{5D4} = 0.27$ which are the values previously obtained in the neat $TbAl_3(BO_3)_4$. In the codoped samples the calculated Q_{TbEu} values are negligible. However, as can be seen from Fig. 4, the W values are proportional to the Eu^{3+} concentration as it is predicted in the Parent et al. model [20]. This result indicates that the transfer to Eu^{3+} , at higher concentrations of this

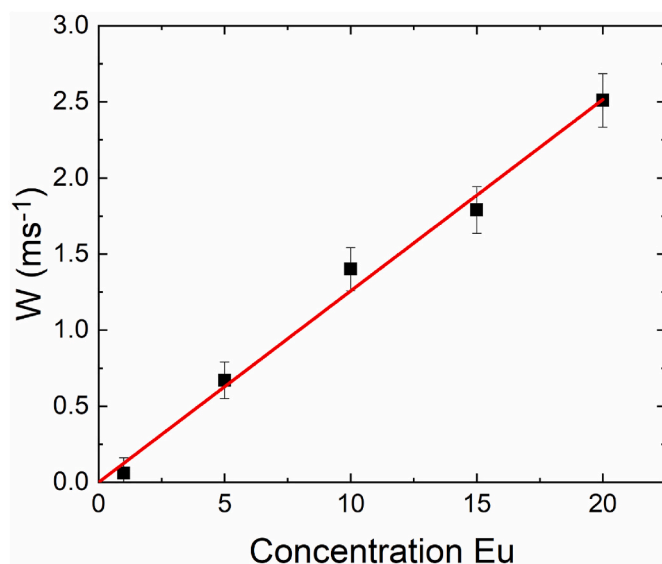


Fig. 4. Values obtained for the W parameter as a function of the Eu^{3+} concentration.

ion, is dominated by the migration processes among Tb^{3+} ions due the W parameter.

Upon excitation in the 5D_3 level at 374 nm, we recorded at 545 nm the same decay curves presented in Fig. 3. This is due to the fact that the 5D_4 level is fed by 5D_3 very quickly and no rise of the luminescence intensity is detected at short time in the luminescence decay curve. The very fast depopulation of 5D_3 level (and concomitant population of 5D_4) is caused by two efficient non-radiative relaxation processes, namely multiphonon relaxation and cross-relaxation [22]. The former process is mainly responsible for a very short decay of the 5D_3 level in $YAl_3(BO_3)_4:1\%Tb$ (lifetime around 25 μs ; data not shown) through the coupling with B-O stretching vibrations with $\hbar\omega \approx 1300 - 1500$ cm^{-1} [23].

4. Conclusions

In this contribution, we present a description of the dynamics of the energy transfer from Tb^{3+} to all possible acceptor species (impurities and Eu^{3+} ion) in $TbAl_3(BO_3)_4:x\%Eu$, with $x = 0, 1, 5, 10, 15$ and 20 samples, by fitting the 5D_4 luminescence decay curve with two main models: i) the Inokuti-Hirayama model [19] for the $TbAl_3(BO_3)_4$ sample and ii) an adapted Parent et al. model for the Eu^{3+} doped samples. The latter model includes the mathematical description of each energy transfer channel involving Tb^{3+} [i.e. $Tb^{3+} \rightarrow$ impurities as Mo^{3+} (or other crystal defects) and $Tb^{3+} \rightarrow Eu^{3+}$ that can be direct or assisted by energy migration in the Tb^{3+} sublattice]. In the neat sample, apart from the intrinsic decay, the dominant mechanism depopulating 5D_4 level of Tb^{3+} is the direct (not assisted by energy migration) energy transfer to quenching impurities. As previously hypothesized in the literature [8] in the Eu^{3+} doped samples, the energy transfer to Eu^{3+} ions should occur in an intermediate regime between two limiting cases in which the respective fastest processes are: i) the $Tb^{3+} \rightarrow Eu^{3+}$ energy transfer and ii) the migration of the energy among Tb^{3+} ions. According to our results, the energy transfer process to Eu^{3+} ions assisted by energy migration in the Tb^{3+} sublattice becomes more and more important as the concentration of Eu^{3+} increases. Finally, at short time the decay curve is dominated by the fast and direct $Tb^{3+} \rightarrow Mo^{3+}$ (or other crystal defects) energy transfer.

CRedit authorship contribution statement

Leonardo Ceccon: Methodology. **Silvia Ruggieri:** Validation.

Marco Bettinelli: Conceptualization. **Inocencio R. Martin:** Formal analysis, Data curation, Conceptualization. **Fabio Piccinelli:** Writing – review & editing, Conceptualization.

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