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Up-conversion fluorescence dynamics in Er^{3+}/Yb^{3+} co-doped tellurite glasses

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The infrared to visible conversion in zinc tellurite glasses (TZG), activated by Er^{3+} ions and sensitized by Yb^{3+} ions, has been investigated under 980 nm pulsed laser excitation. The up-conversion mechanism was studied by means of time-resolved luminescence spectroscopy. A model for the dynamics of frequency up-conversion in Er^{3+}/Yb^{3+} co-doped TZG based on the rate equations was proposed. The dynamics of the up-converted emissions were studied to evaluate energy rates between Er^{3+} and the Yb^{3+} ions, and the rate excited state absorption (ESA) in Er^{3+} ions.

Keywords: up-conversion, energy transfer, rare earth doped materials (erbium and ytterbium).

1. Introduction

Up-conversion materials have attracted significant attention as the development of infrared and up-conversion lasers and optical amplifiers and so on [1–5]. Many trivalent rare earth ions such as Er^{3+}, Tm^{3+}, Pr^{3+} and Nd^{3+} were doped as luminescent ions earlier in certain hosts [6–8], Yb^{3+} and Er^{3+} ions as suitable laser emitters for several interesting applications and with the advantages of diode-pumping and up-conversion mechanisms [9–12]. Considerable attention has been devoted to the study of up-conversion luminescence in rare-earth doped glasses.

In addition, the up-conversion mechanism is usually involved at the following processes: ground state absorption (GSA), excited state absorption (ESA), and energy transfer (ET), multiphonon relaxation (MR), cross relaxation (CR) and so on. The different types of energy transfer up-conversion (ETU) processes were described very well in the literature [13–18].

The up-conversion fluorescence has already been studied in Er^{3+}/Yb^{3+}-doped glasses and fibers [19], phosphate glasses [20], germanate glasses [21], tellurite glasses [22], $LiNbO_3$ [23], fluoride phosphate glasses [24] and $PBO–Bi_2O_3–Ga_2O_3–GeO_2$ glasses [25].

In this paper, we present an analysis of Er^{3+}/Yb^{3+} optical transitions behavior in the visible range. We have focused our study details on the green emission centered on 550 nm. Finally, we have investigated the dynamics of the $^4S_{3/2}$ state and explained that it has been based on the relative efficiency of different mechanisms.

2. Experiment

Glasses were prepared from oxide powders of TeO_2, ZnO, Er_2O_3 and Yb_2O_3 as starting materials using the conventional melt-quenching method. The material used in our measurement has a composition of $70TeO_2-30ZnO$ and was either single doped with 9.9×10^{19} ions/cm^3 of Er_2O_3 or co-doped with 9.9×10^{19} ions/cm^3 of Er_2O_3 and 3.1×10^{20} ions/cm^3 of Yb_2O_3.

The intrinsic lifetimes of the levels were obtained by exciting the samples with a laser analytical system dye laser pumped by a pulsed frequency doubled Nd:YAG laser from BM Industries. The duration of pulses was 8 ns. The emitted light has been focused on a Jobin–Yvon HR S2 spectrophotometer. The detection has been performed by using an R 1767 Hammamatsu photomultiplier and a Lecroy 9410 averager oscilloscope. All experiments were performed at room temperature.

3. Up-conversion results and mechanisms

When Er^{3+} doped materials are sensitized by trivalent ytterbium, both the high absorption cross-section of the ytterbium sensitizer and the efficient energy-transfer mechanism between Yb^{3+} ions and the Er^{3+} (rare-earth acceptor) ions lead to a considerable enhancement in the up-conversion efficiency as demonstrated previously [26–28].

Figure 1 shows the up-conversion spectra of the Er^{3+} (9.9×10^{19} ions/cm^3) singly doped, and Er^{3+}/Yb^{3+} co-doped tellurite glasses as for Yb^{3+} (3.1×10^{20} ions/cm^3)

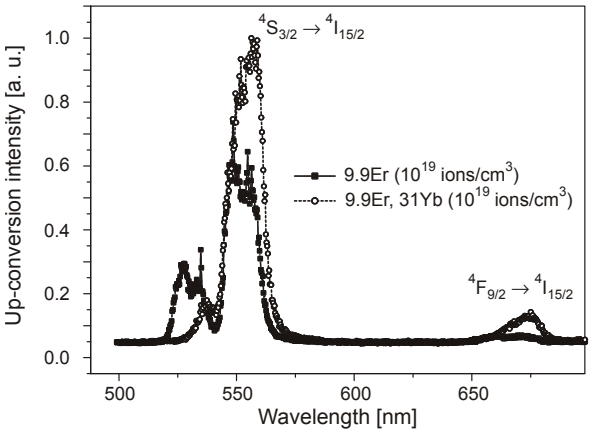

Fig. 1. Up-conversion spectra of the Er^{3+} (9.9×10^{19} ions/cm^3) doped, and Er^{3+}/Yb^{3+} co-doped (9.9×10^{19}/3.1×10^{20} ions/cm^3), in the $70TeO_2$–30ZnO tellurite glass at T = 300 K, of the green and red emissions.

content at room temperature, obtained under 980 nm pulsed laser excitation. We can see two emission bands peaked at 550 and 670 nm that correspond to the transitions of Er^{3+} ions from excited states to the ground state. Intense green and red emission bands at around 550 and 670 nm wavelength are attributed to the transitions from $({}^4S_{3/2} \rightarrow {}^4I_{15/2})$ and $({}^4F_{9/2} \rightarrow {}^4I_{15/2})$, respectively. All the up-conversion luminescences are enhanced strongly with Yb^{3+} contents increasing. It is due to the contribution of Yb^{3+} in energy transfer process.

The energy transfer efficiency mainly depends on the ratio of the backward transfer rate and the multiphonon relaxation rate of the Er^{3+}: ${}^4I_{11/2}$ level. Compared with silicate and germinate glasses, phosphate glasses have high efficiencies because of the small values of this ratio [29].

In our tellurite glasses, considerable observed transfer rates indicate that an important transfer efficiency and/or a weakness back transfer from Er^{3+} to Yb^{3+}, because of the fast decay of the ${}^4I_{11/2}$ level, provides an efficient sink for the excitation transferred from Yb^{3+} and the low ratio between Er^{3+} and Yb^{3+} ions.

In a frequency up-conversion process, the increase in up-conversion emission intensity I_{UC} is proportional to the *n*-th power of infrared excitation intensity.

We measured the pump power dependence of the emission intensities and we found, on a log–log plot of intensity versus power, a slope very close to two for both the green and red transitions. It is found that the red and the green emission in TZG results both from a two-photon excitation (see Figs. 2**a** with 2**b**).

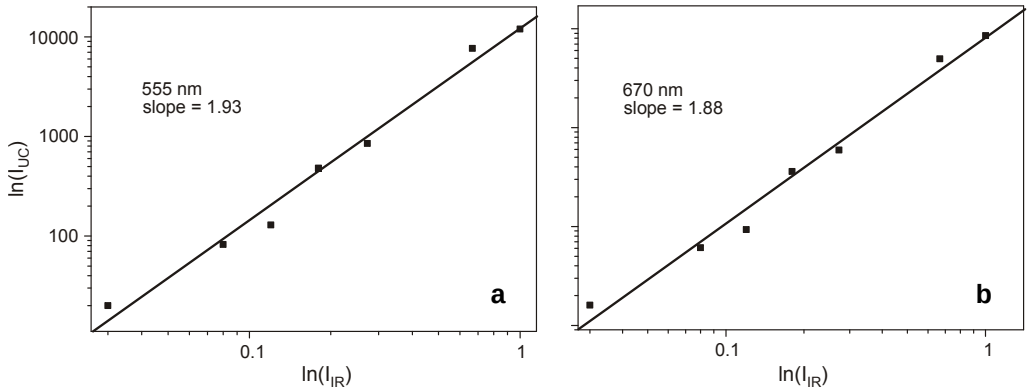

Fig. 2. Pump power dependence of green (**a**) and red (**b**) emissions.

This demonstrates that the energy transfer (ET) and the excited state absorption (ESA) processes can explain the up-converted luminescence.

For the green luminescence (at 550 nm), the possible up-conversion luminescence mechanisms of the Er^{3+} ions can be described by: *i*) excited state absorption (ESA), and *ii*) energy transfer up-conversion (ETU) concluding cross relaxation (CR) between two Er^{3+} ions and energy transfer (ET) between Er^{3+} ions and Yb^{3+} ions.

According to the energy matching conditions and the dependence of up-conversion emission intensity on excitation power, the possible up-conversion mechanism is

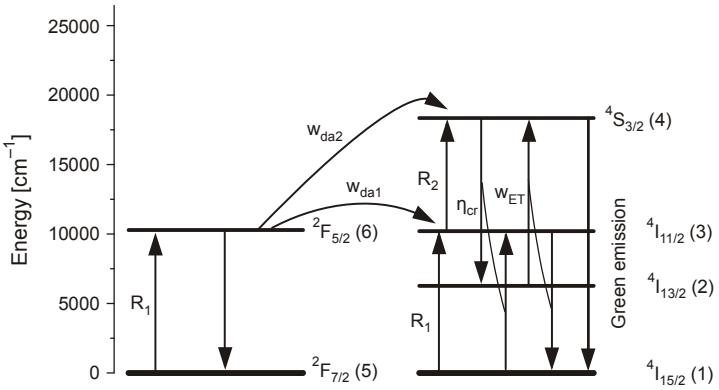

Fig. 3. Energy transfer and up-conversion processes considered in the model proposed in this work to explain the green dynamics in Er^{3+}/Yb^{3+} co-doped tellurite glasses.

analyzed on the basis of the simplified energy level diagrams of Yb^{3+} and Er^{3+} ions as illustrated in Fig. 3.

When the glass is excited by a 980 nm laser, the $^2F_{5/2}$ level of Yb^{3+} is populated by the ground state absorption (GSA) first. And then the energy is transferred from Yb^{3+} to Er^{3+} which is in $^4I_{11/2}$ state. Consequently, the $^4I_{11/2}$ level is populated via the energy transfer (ET) step (Yb^{3+}:$^2F_{5/2}$ + Er^{3+}:$^4I_{15/2}$ → Yb^{3+}:$^2F_{7/2}$ + Er^{3+}:$^4I_{11/2}$). It is important to point out that this ET process is necessary for Er^{3+}: $^4I_{11/2}$ level population.

In the Er^{3+}/Yb^{3+} co-doped system, the energy transfer process from Yb^{3+} to the metastable level of Er^{3+} ions is not instantaneous owing to the finite lifetime of level $^4I_{11/2}$.

First, the transition from $^4I_{11/2}$ state is excited to $^4F_{7/2}$ level by another energy transfer from the Yb^{3+} ions. The populated $^4F_{7/2}$ level Er^{3+} then relaxes rapidly and non-radiatively to the next lower levels $^2H_{11/2}$ and $^4S_{3/2}$ resulting from the small energy gap between the levels, and finally green light is emitted through the transition from $^4S_{3/2}$ to the manifold ground level $^4I_{15/2}$.

In the second process (ETU), we can explain the up-conversion of the green emitting $^4S_{3/2}$ state by three mechanisms:

– The first one is the energy transfer up-conversion ETU:

$$^4I_{13/2} + {}^4I_{11/2} \rightarrow {}^4S_{3/2} + {}^4I_{15/2}$$

– The second one is the energy transfer process from Yb^{3+} to Er^{3+} ions:

$$w_{da1}\text{: } Yb^{3+}\text{: } {}^2F_{5/2} + Er^{3+}\text{: } {}^4I_{15/2} \rightarrow Yb^{3+}\text{: } {}^2F_{7/2} + Er^{3+}\text{: } {}^4I_{11/2}$$

$$w_{da2}\text{: } Yb^{3+}\text{: } {}^2F_{5/2} + Er^{3+}\text{: } {}^4I_{11/2} \rightarrow Yb^{3+}\text{: } {}^2F_{7/2} + Er^{3+}\text{: } {}^4S_{3/2}$$

– The third one mechanism is the ESA:

$$^4I_{11/2} \rightarrow {}^4S_{3/2}$$

And of the cross-relaxation (CR):

$$^4I_{15/2} + {}^4S_{3/2} \rightarrow {}^4I_{11/2} + {}^4I_{13/2}$$

Based on the previous studies [22] in the Er^{3+}/Yb^{3+} co-doped tellurite glasses, a set of the rate equations was introduced to analyze the energy transfer and green up-conversion processes. Following approximations were made to establish the equations. The $^4I_{9/2}$ level is estimated to be almost empty because of the fast multiphonon decay from the $^4I_{9/2}$ level.

The energy back transfer from Er^{3+}: $^4I_{11/2}$ level to Yb^{3+}: $^2F_{5/2}$ level is so low that it can be neglected.

3.1. Decay times investigation

The time-resolved decay curves of up-conversion luminescence can be useful to distinguish ESA and ETU characteristics for the up-conversion mechanisms [30–33]. ESA process takes place during the excitation pulse, while ETU can persist after the pulse for a longer period related to the lifetime of the level providing energy transfer.

Therefore, the up-conversion decay based on ESA exhibits an exponential behavior similar to that by direct excitation, while the ETU decay exhibits a clear rise time and a non-exponential behavior. It can be seen that a clear rise time appears at 555, 670 and 1500 nm up-conversion decay.

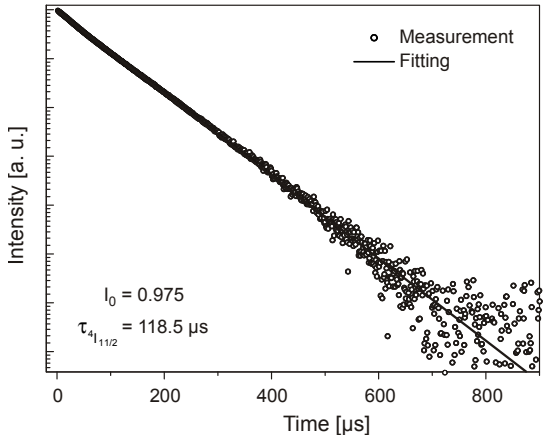

Fig. 4. The fluorescence decay curves of transition $^4I_{11/2} \rightarrow {}^4I_{15/2}$ under 980 nm excitation.

Experimental decay curve of $^4I_{11/2}$ level is fitted very well with exponential curve. Its fluorescence decay is presented in Fig. 4. The fluorescence intensity I versus the time t is approximated by the following function:

$$I = I_0 \exp\left(-\frac{t}{\tau}\right)$$

where I_0 is the initial intensity and τ is the fluorescence decay time.

Decay profiles of the $^4I_{13/2}$, $^4F_{9/2}$ and $^4S_{3/2}$ levels were non-exponential; therefore their average decay times can be given with the formula below:

$$\tau_m = \int_0^\infty \frac{I(t)}{I_0}\,\mathrm{d}t$$

The cross-relaxation energy transfer efficiency η_{cr} can be evaluated by the following equation:

$$\eta_{cr} = 1 - \frac{\tau_{Er-Yb}}{\tau_{Er}}$$

where τ_{Er-Yb} and τ_{Er} are the fluorescence lifetimes of the $^4S_{3/2}$ level of co-doped Yb^{3+}–Er^{3+} and Er^{3+} (9.9×10^{19} ions/cm^3) doped glasses in the case of green emission. The lifetimes τ_{Er-Yb} = 66.5 μs and τ_{Er} = 102 μs for the $^4S_{3/2}$ erbium level, correspond to the samples with ytterbium concentration 3.1×10^{20} and 4.9×10^{19} ions/cm^3, respectively, and were determined as the averages of decay times.

The energy transfer efficiency from Yb^{3+} to Er^{3+} was evaluated by using the expression [34]

$$\eta_{da} = 1 - \frac{\tau_{Yb-Er}}{\tau_{Yb}}$$

where τ_{Yb-Er} and τ_{Yb} are the fluorescence lifetimes of the $^2F_{5/2}$ level of co-doped Yb^{3+}–Er^{3+} and Yb^{3+} (3.1×10^{20} ions/cm^3) doped glasses. The lifetimes τ_{Yb-Er} = = 220 μs [34] and τ_{Yb} = 500 μs [34] for the $^2F_{5/2}$ ytterbium level correspond to the samples with erbium concentration 9.9×10^{19} and 0 ions/cm^3, respectively.

T a b l e 1. Values of measured lifetimes τ_{mes} of different levels, η_{cr} and η_{da} in tellurite glasses co-doped with Er^{3+}/Yb^{3+}.

Measured lifetimes	$\tau_{^4I_{13/2}}$ [μs]	$\tau_{^4I_{11/2}}$ [μs]	$\tau_{^4F_{9/2}}$ [μs]	$\tau_{^4S_{9/2}}$ [μs]	$\tau_{^2F_{7/2}}$ [μs]	Reference
	2546	118.5	94	66.5	220 [34]	This work
Er^{3+}/Yb^{3+}	3690	–	126	80	–	[22]
	4050	–	190	100	–	[35]
	η_{cr} ($^4S_{3/2}$)	η_{da}				
9.9Er 31Yb (10^{19} ions/cm^3)	0.39	0.56				

Table 1 summarizes the measured lifetimes and the cross-relaxation energy transfer efficiency and the energy transfer efficiency of the emitting.

The measured average decay time of the red emission is not close to that of the green one, suggesting that $^4F_{9/2}$ level has been reached by an ETU process not originating from $^4S_{3/2}$ level.

3.2. Up-conversion investigation

We consider a four-level system as depicted in Fig. 4 in the case of the Er^{3+}, with a ground state denoted by 1, a final state labeled 4, and a two-level in the case of the Yb^{3+}. The populations of the levels are labeled n_1, n_2, n_3, n_4, n_5 and n_6.

The ground state absorption from the $^4I_{15/2}$ level to the $^4I_{11/2}$ level is represented by the arrow R_1 $(1 \rightarrow 3)$ and the transition $^4I_{11/2} \rightarrow {}^4S_{3/2}$ is represented by the arrow R_2 $(3 \rightarrow 4)$.

The parameters of the model are gathered in Tab. 2.

T a b l e 2. Values of the parameters used in our model.

τ_2 [S^{-1}]	τ_3 [S^{-1}]	τ_4 [S^{-1}]	τ_6 [S^{-1}]	β_{32} [%]	β_{42} [%]	β_{43} [%]	η_{cr} [%]
2546	118.5	66.5	220	4.9	5	16	39

The evolution of the populations of the seven energy levels is written as:

$$\frac{dn_4}{dt} = -\frac{n_4}{\tau_4} + R_2 n_3 + w_{ET} n_3 n_2 - \frac{\eta_{cr} n_1 n_4}{(1-\eta_{cr})\tau_4} + w_{da2} n_3 n_6$$

$$\frac{dn_3}{dt} = -\frac{n_3}{\tau_3} + R_1 n_1 + \beta_{43}\frac{n_4}{\tau_4} - R_2 n_3 + w_{da1} n_1 n_6 + \\ + \frac{\eta_{cr} n_1 n_4}{(1-\eta_{cr})\tau_4} - w_{ET} n_3 n_2 - w_{da2} n_3 n_6$$

$$\frac{dn_2}{dt} = -\frac{n_2}{\tau_2} + \beta_{42}\frac{n_4}{\tau_4} + \beta_{32}\frac{n_3}{\tau_3} + \frac{\eta_{cr} n_1 n_4}{(1-\eta_{cr})\tau_4} - w_{ET} n_2 n_3$$

$$n_1 + n_2 + n_3 + n_4 = 1$$

$$\frac{dn_6}{dt} = R_1 n_5 - \frac{n_6}{\tau_6} - w_{da1} n_1 n_6 - w_{da2} n_3 n_6$$

$$n_5 + n_6 = 1$$

where R_1, R_2 are the pumps rates of Yb^{3+} and Er^{3+} ions; n_5 and n_1 are the ground state populations of donor and acceptor ions, respectively. The transfer rates between

T a b l e 3. Values of fitting parameters: R_1, R_2, w_{da1}, w_{da2} and w_{ET}.

Parameter	Value			
R_1 [S^{-1}]	755	–	–	–
R_2 [S^{-1}]	0	–	–	–
w_{da1} [cm^3S^{-1}]	7×10^{-18}	5×10^{-18} [36]	4×10^{-17} [37]	5×10^{-16} [38]
w_{da2} [cm^3S^{-1}]	1×10^{-18}	–	–	5×10^{-16} [38]
w_{ET} [cm^3S^{-1}]	8.5×10^{-17}	1×10^{-16} [36]	3.5×10^{-18} [37]	3×10^{-19} [38]

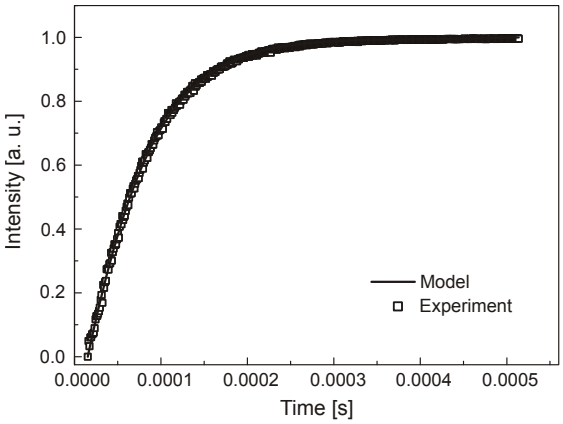

Fig. 5. Time evolution of the green fluorescence originating from the $^4S_{3/2}$ level at 980 nm excitation wavelength. The squares are the prediction of our model; the solid curve is the experimental data.

the donor ion and the $^4I_{11/2}$ and $^4S_{3/2}$ levels of the acceptor ion are denoted by w_{da1} and w_{da2}, respectively; β_{ij} is the radiative transition; w_{ET} and η_{cr} are the energy transfer up-conversion rate and the cross-relaxation coefficient.

The parameters of the model are gathered in Tab. 3.

Other authors have also reported fitting parameters of Er^{3+}/Yb^{3+} values for various up-conversion processes and materials [36–38]. These values are comparable to our results.

System (1) leads to the fit of the time evolution of the green fluorescence represented by the line in Fig. 5 (at 980 nm). The fit was performed with five fitting parameters: R_1, R_2, w_{da1}, w_{da2} and w_{ET}.

Table 3 compares the numerical values for Er^{3+}-doped tellurite glass, and it can be seen that ASE is insignificant.

4. Conclusions

Red and green up-conversion emissions have been obtained in tellurite glasses co-doped with Er^{3+}/Yb^{3+}. The intense green and red emissions around 550 and 670 nm

corresponding to transitions of Er^{3+} were observed under 980 nm excitation. It is possible to distinguish the contribution of ESA and ET processes.

The dynamics of the green emission is well explained with the proposed model. The energy transfer from Yb^{3+} to Er^{3+} is responsible for enhancement of the visible up-conversion emissions.

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