Radiation trapping in selected Er3+ doped chalcogenide glasses and the extraction of the nonradiative lifetime

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**Abstract:** Radiation trapping (RT) is a phenomenon wherein photons are emitted, absorbed and re-emitted many times before they leave the volume of the material. Trivalent Er3+ ions are particularly prone to RT because there is a whole set of strongly overlapping emission and absorption bands including 4I13/24I15/2 and 4I11/24I15/2 bands. The effect of RT on the PL decay time was investigated experimentally in this work in a variety of Er3+-doped GeGaS, GeGaSe, GaLaS(O) glasses. Sample geometry (powders, plates, disks, cylinders) and size were varied and the samples were also immersed in glycol, a liquid with high refractive index. PL decay times were measured and compared with the Judd-Ofelt results. A simple model of RT was developed and applied to the above mentioned bands. By comparing model conclusions with experimental data for different sample sizes, we were able to separate the direct relaxation of the 4I11/2 state to ground 4I15/2 state and relaxation via the intermediate 4I13/2 state; and hence obtain an approximate nonradiative lifetime.

1. Introduction

Radiation trapping (RT) is a phenomenon wherein photons are emitted, absorbed and re-emitted many times before they leave the volume of the material. RT has been observed and reported in variety of materials where there is a strong overlap of the emission and absorption bands. Historically, its discovery dates back to the mid-1920s [[[1]](#endnote-1),[[2]](#endnote-2)]. Indeed, Lucy Hayner's paper in 1925 included a term "radiation imprisonment" to describe the delay involved in the passage of luminescence radiation through an Hg vapor [1]. Over the last two decades it has been widely observed in materials doped with multivalent rare earth ions [[[3]](#endnote-3) −[[4]](#endnote-4),[[5]](#endnote-5),[[6]](#endnote-6),[[7]](#endnote-7),[[8]](#endnote-8),[[9]](#endnote-9),[[10]](#endnote-10),[[11]](#endnote-11),[[12]](#endnote-12),[[13]](#endnote-13)]. Among various rare earths ions, the trivalent Er3+ offers a unique opportunity for RT because there is a whole set of perfectly overlapping emission and absorption bands [[[14]](#endnote-14)]. Usually, RT is regarded a ‘nuisance’ that leads to the distortion of PL spectra and radiative lifetimes [3,11]. Researchers try to eliminate RT by experimenting on fine powders [7,11] or by using excitation and detection through spatially separated pinholes [[[15]](#endnote-15)[[16]](#endnote-16)-[[17]](#endnote-17)] or by using a confocal setup [[[18]](#endnote-18)].

In the present work, we use RT as a means of separating the direct relaxation of 4I11/2 state to ground 4I15/2 state and relaxation via intermediate 4I13/2 state. Based on our previous findings [[[19]](#endnote-19)], we build a simple model of RT. We apply this model to 4I13/24I15/2 and 4I11/24I15/2 bands of Er3+ embedded in several chalcogenide glasses with different compositions and offer a way to separate direct radiative transition and nonradiative relaxation of the 4I11/2 state.

1. Experimental procedure

Er3+ doped glasses were prepared by melt quenching of raw materials mixed in the proportions shown in Table 1. The lanthanum sulphide-oxide glasses (rows 1 and 2 in Table 1) were prepared at the University of Southampton. The detailed description of their preparation technique may be found elsewhere [19]. The germanium gallium sulphide (3) and germanium gallium selenide (4) glasses were synthesized at the University of Saskatchewan. The synthesis details have been described in references [[[20]](#endnote-20),[[21]](#endnote-21)].

**Table 1. Glass compositions; *Eg* - optical gap; **10(0) and **20(0) PL decay times in fine powders for 4I13/24I15/2 and 4I11/24I15/2 bands, respectively; *a* - slope of the straight lines in Fig. 3; **20and **21 relaxation times of 4I11/24I15/2 and 4I11/24I13/2, respectively (deduced in the present paper). Optical gap (*Eg*) is accepted to be equal to the photon energy at which absorption coefficient is equal to 103 cm-1.**

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| --- | --- | --- | --- | --- | --- | --- | --- |
|  | **Glass compositions** | ***Eg*, eV** | ****10(0)**  **ms** | ****20(0)**  **ms** | ***a*** | ****20 , ms** | ****21 , ms** |
| 1 | 73 Ga2S3 + 27 La2O3 + 0.5 Er2O3 | 3.05 | 3.35 | 0.89 | 0.22 | 3.98 | 1.15 |
| 2 | 66 Ga2S3 + 32 La2S3 + 3 La2O3 + 0.5 Er2O3 | 2.79 | 2.73 | 1.40 | 0.53 | 2.62 | 2.98 |
| 3 | 90 GeS2 + 10 Ga2S3 + 0.5 Er2S3 | 3.00 | 2.71 | 1.58 | 0.40 | 3.96 | 2.63 |
| 4 | 90 GeSe2 + 10 Ga2Se3 + 0.5 Er2S3 | 2.31 | 1.91 | 0.94 | 0.59 | 1.60 | 2.29 |

The effects of photon trapping depend on the sample geometry. Following a geometrically wise approach, we have used three types of samples (as an illustration see inset of Fig. 2).

(1) *Fine powders* with an average particle diameter around 30 m were prepared by crushing bulk materials and passing them through as a sequence of sieves with appropriate meshes. The powders were collected on a sticky scotch tape. A control experiment confirmed the absence of a PL response of the virgin scotch tape.

(2) The optical *plates* were cut off from a glass rod (diameter around 2 mm) and had typical thickness around 1 mm. They were polished on both sides for optical transmittance and PL measurements.

(3) *Cylinders* were cut off from the same glass rod and polished on both sides. The length of cylinders varied from 10 to 44 mm. As a result of polishing, the cylinders had all shiny surfaces favoring an effective "imprisonment" of light due to internal reflection. For some of the experiments, the samples were submerged in glycol, a liquid with high refractive index. More details on sample preparation and experiments involving glycol submersion may be found in reference [19].

PL corresponding to 4I13/24I15/2 and 4I11/24I15/2 transitions in Er3+ ions was dispersed by Cornerstone monochromator and detected using Peltier cooled InGaAs detector. The excitation corresponding to the 4I15/24I9/2 transition in Er3+ ions was performed by laser diode operating at 808 nm. A ORIEL mechanical chopper was used for transient PL measurements and the signal from the InGaAs detector built-in-amplifier was directly coupled to PicoScope oscilloscope for registration and further analysis.

It should be mentioned that GaLaS(O) glasses used in this work have negligible OH content as reported previously and confirmed by the absorption spectra around 3 m [[[22]](#endnote-22)]. In the case of GeGaS and GeGaSe glasses, the addition of Ga leads to a large increase in the solutibility of Er3+ in this glass system, especially if stoichiometric compositions of constituent compounds are alloyed as in this work [21]. Further, the Er3+ concentration used in this work is much less than that needed for concentration quenching [7] and the results and conclusions are not affected by the latter phenomenon.

1. Experimental results

Fig. 1 compares the PL decays from the steady-state after cessation of excitation at 808 nm in samples of Er3+ doped gallium lanthanum sulphide-oxide glasses with two different compositions marked as samples 1 and 2 in Table 1. The measurements were performed for two different emission bands 4I11/24I15/2 and 4I11/24I15/2. The fastest PL decays were observed in powdered materials with particle size *L* ≈ 30 m. As the geometrical size of samples becomes bigger, the PL decays become slower. This effect was observed in all investigated glasses in this work and for both emission bands.



Fig. 1. The influence of geometrical size on PL decays from steady-state after switching-off the excitation in two samples with compositions shown in Table 1 and for two emission bands. The meaning of sample size (*L*) is explained in the caption of Fig. 2. Solid lines are the best single exponential fits to experimental data. All experiments are performed in air. Excitation is by a laser diode at 808 nm.

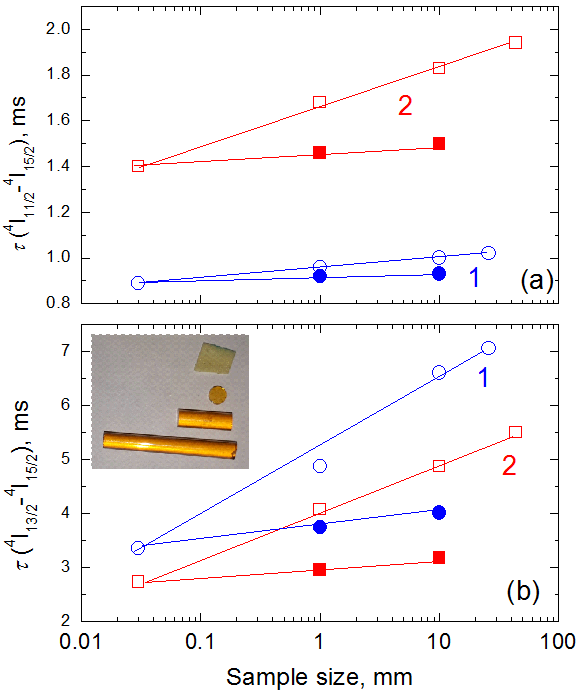


Fig. 2.The PL decay times vs. geometrical size of samples for 4I11/24I15/2 (upper pane (a)) and 4I13/24I15/2 (lower pane (b)) emission bands. Numbers 1 and 2 stand for two different glass compositions as shown in Table 1. Open symbols are results collected in air. Full symbols show the results for samples submersed in glycol. Powders in air and in glycol give coinciding results. The inset to figure (b) illustrates the geometry of samples (glass #1) used in present research. The samples of fine powders with particle size (*L≈* 0.03 mm) were collected on scotch tape. Round optical plates had the diameter close to 2 mm and thickness (*L* ≈ 1 mm (shown in the inset)). Cylinders with both polished ends had the diameter close to 2 mm and varying heights (*L* = 10, 26 and 44 mm. Cylinders with *L* = 10 and 26 mm are shown in the inset). Solid lines are guides to eye.

Fig. 2 depicts the dependence of PL decay times on the geometrical size of samples. This figure illustrates that the rate of increase depends on the glass composition and the emission band. Thus, changes of PL decay time are less pronounced for glass 1 than glass 2 in particular for 4I11/24I15/2 emission band. Fig. 2 shows also the acceleration of PL decay (reduction of decay time) when the sample is submersed in glycol.

Here and after we will refer to 4I15/2 manifold as ground or 0th level, to 4I13/2 as 1st level and to 4I11/2 as 2nd level. Accordingly, the measured PL decay times for 4I13/24I15/2 transitions in fine powders and bulk materials will be referred to as**1(0) and **1(*L*), respectively. Similar notations **2(0) and **2(*L*) will be used for PL decay times for 4I11/24I15/2 transitions in fine powders and bulk materials, respectively. Fig. 3 compares the ratios of PL decay times for 4I11/24I15/2 and 4I11/24I15/2 emission bands for different glasses. It shows linear dependence in a form

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| --- | --- |
|  | (1) |

where *a* is a slope with values listed in Table 1. The meaning of the graphs and the importance of linearity are explained and discussed in detail in the next section.



Fig. 3. Ratio of PL decay times **2(*L*)/**2(0) versus ratio **1(*L*)/**1(0). Here **2(*L*) is the PL decay time for Er3+ 4I11/24I15/2 emission band in bulk samples; **2(0) is the PL decay time for the same band in fine powders;**1(*L*) is the PL decay time for Er3+ 4I13/24I15/2 emission band in bulk samples and **1(0) is the PL decay time for the same band in fine powders. Numbers from 1 to 4 correspond to different glass compositions as listed in Table 1. Open symbols are results collected in air. Full symbols are for samples submersed in glycol. Solid lines are least square fits to experimental data using equation (1) with slopes *a* listed in Table 1.

1. Model and discussion

In this section, a simplistic model of radiation trapping is developed. Let *Ni* be the *total number* of excited ions in the *whole* sample. Suppose excited ions may relax through two independent processes with characteristic times **1 and **2. In this case, the decay of *Ni* after switching-off the excitation may be described by a simple rate equation

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| --- | --- |
|  | (2) |

The solution of Equation (2) will obviously be single exponential decay with a characteristic time

|  |  |
| --- | --- |
|  | (3) |

For the future let us assume that the first process (with characteristic time **1) is a radiative transition. Experimentally, **(0) may be observed in fine powders where the influence of RT has been proven to be negligible, as, for example, in references [7, 11]. However in a presence of RT some modifications of Equation (2) are required. First of all, we note that the radiative transition becomes “less efficient” because part of emitted PL photons is reabsorbed by Er3+ ions. Therefore, Equation (2) should be corrected as

|  |  |
| --- | --- |
|  | (4) |

here *p*esc(*L*) is the probability of photon to escape the sample with the size *L* without being reabsorbed. Obviously, the parameter *p*esc depends on the sample size. Thus, in fine powders, *p*esc(*L*)→1 due to small size of powder particles and the inefficiency of reabsorption. On the other hand, in large samples *p*esc(*L*)→ 0 meaning that nearly all photons are being reabsorbed within a huge volume of the sample. The solution of Equation (4) gives again a single exponential decay but with a different characteristic time

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|  | (5) |

which is observed in bulk samples with *effective geometrical size* *L* as PL decay time. Dividing Equation (3) by (5) we find a ratio of two characteristic PL decay times in powdered and bulk materials as

|  |  |
| --- | --- |
|  | (6) |

Equation (6) is a general one that can be applied to some particular cases. Let us start with the 4I13/24I15/2 relaxation of Er3+ ions. Usually, relaxation from 4I13/2 level (it is referred to as level 1 in this paper) to ground level 4I15/2 (level 0) occurs by radiative emission which is known to be the dominant relaxation process in many glasses [7, [[23]](#endnote-23)]. In other words, there is only one relaxation mechanism and hence equation (6) may be reduced to

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| --- | --- |
|  | (7) |

here the index (1) shows that Equation (7) refers to the first emission/absorption band, i.e. 4I13/2  4I15/2.

Er3+ in second exited state 4I11/2 may relax either directly to 4I15/2 ground state or through intermediate excited 4I13/2 state. The first 4I11/24I15/2 relaxation is purely radiative and may be strongly affected by RT due to the presence of matching 4I15/24I11/2 absorption band. The presence of RT is supported by Fig. 1 and reported earlier for some other glasses [3-47,14]. Let us assume that 4I11/24I15/2 radiative lifetime is **20.

The relaxation from 4I11/2 to 4I13/2 state may be radiative or non-radiative. Obviously, for non-radiative relaxation there is no RT. However, for 4I11/24I13/2 radiative relaxation the RT influence is also negligible because detectable 4I13/24I11/2 excitation stimulated absorption appears only at very high pumping levels far exceeding those used in present paper. Assuming that radiative lifetime is  and non-radiative relaxation time is we get an effective time of relaxation from 4I11/2 to 4I13/2 as **21 = (1/+1/)1 and in case of relaxation of 4I11/2 level equation (6) may be presented as

|  |  |
| --- | --- |
|  | (8) |

By combining experimental results presented by equation (1) with equations (7) and (8) it is easy to derive the relation between and as

|  |  |
| --- | --- |
|  | (9) |

In very big samples (*L*→∞) as we already mentioned earlier the escape becomes improbable, i.e. *p*esc→0 for both bands giving us a simple relation between **20 , **21 and experimental value of *a*

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| --- | --- | --- | --- |
|  | or |  | (10) |

Equation (10) is the final result of our simple model and it shows that the slope of the linear dependence (*a*) in Equation 1 may be used to evaluate **20 and **21 as

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| --- | --- | --- | --- |
|  | and |  | (11) |

The results of these calculations for four different glasses doped with Er3+ ions are summarized and presented in Table 1. The validity of these conclusions is presented in Table 2 which compares the predicted values of **20 with calculations using Judd-Ofelt model. In overall, Table 2 shows quite good agreement of 4I13/24I15/2 radiative recombination times  with experimental values of PL decay times in fine powders **10(0) confirming the applicability of JO model to our case. Table 2 also shows a reasonable agreement of values of 4I13/24I15/2 radiative recombination times  and **20 which are calculated in present paper.

**Table 2. Glass compositions; Judd-Ofelt parameters2, 4 and 6) ; ** - branching ratio for level 4I11/2 ; - electric dipole 4I13/2 4I15/2 transition rate; - magnetic dipole 4I13/2 4I15/2 transition rate; - calculated 4I13/2 4I15/2 transition radiative lifetime; - electric dipole 4I11/2 4I15/2 transition rate; - calculated 4I11/2 4I15/2 transition radiative lifetime. The values of **10(0) and **20 are reproduced from Table 1 to facilitate comparison with  and , respectively. The values for Judd-Ofelt parameters for glasses 3 and 4 were published previously in [[[24]](#endnote-24)] and [7], respectively.**

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| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
|  | Glass | 2 | 4 | 6 | ** |  |  |  | **10(0) |  |  | **20 |
|  | compositions | (10-20cm2) | (10-20cm2) | (10-20cm2) | (%) | (s-1) | (s-1) | (ms) | (ms) | (s-1) | (ms) | (ms) |
| 1 | 73 Ga2S3 + 27 La2O3 + 0.5 Er2O3 | 6.78 | 1.17 | 0.49 | 14 | 186 | 112 | 3.36 | 3.35 | 267 | 3.75 | 3.98 |
| 2 | 66 Ga2S3 + 32 La2S3 + 3 La2O3 + 0.5 Er2O3 | 6.22 | 1.92 | 0.49 | 14 | 236 | 127 | 2.75 | 2.73 | 402 | 2.49 | 2.62 |
| 3 | 90 GeS2 + 10 Ga2S3 + 0.5 Er2S3 | 11 | 2.9 | 1.6 | 11 | 314 | 70 | 2.60 | 2.71 | 349 | 2.87 | 3.96 |
| 4 | 90 GeSe2 + 10 Ga2Se3 + 0.5 Er2S3 | 8.6 | 2.1 | 1.1 | 12 | 417 | 117 | 1.87 | 1.91 | 699 | 1.43 | 1.6 |

1. Conclusions

We investigated radiation trapping in four different classes of Er3+ doped chalcogenide glasses. Radiation trapping appears in materials in which there is a strong overlap of emission and absorption spectra. From this point of view, Er3+ ions are ideal the manifestation of radiation trapping as they possess many overlapping absorption and emission bands including 4I13/2-4I15/2 and 4I11/2-4I15/2. We have developed a simple model for radiation trapping that incorporates the sample size effect into the observed PL decay time, and applied this model to the above mentioned bands in Er3+ ions in four difference chalcogenide glass hosts. By comparing model conclusions with experimental data for different sample sizes, we were able to separate the direct relaxation of 4I11/2 state to ground 4I15/2 state and relaxation via intermediate 4I13/2 state. This procedure was shown to be effective for four different glasses with very different compositions.

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