

Article

Excellent Color Purity and Luminescence Thermometry Performance in Germanate Tellurite Glass Doped with Eu^{3+} and Tb^{3+}

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Abstract: Germanate tellurite glasses doped with Eu^{3+} and Tb^{3+} were synthesized by the conventional melt-quenching method. There is no indication of the energy transfer between dopant ions in this host, but the co-dopants exhibit excellent color purity of 100% for Eu^{3+} and 80% for Tb^{3+} . The co-doped glass exhibits yellow luminescence. The quantum yield of the Eu^{3+} emission is equal to 23% under 395 nm excitation. The thermal quenching of Eu^{3+} and Tb^{3+} luminescence occurs at different temperature ranges, which enables the thermal sensing properties of the material. The relative fluorescence intensity ratio (FIR) sensitivity of $0.16\% \text{ K}^{-1}$ was recorded in the wide range of temperatures spanning from -193°C up to 0°C . The temperature dependence of the decay times was also studied. The lifetime-based temperature sensitivity was determined to be $0.95\% \text{ K}^{-1}$ at 250°C for $\text{Tb}^{3+} {}^5\text{D}_3$ level emission and $0.3\% \text{ K}^{-1}$ at 225°C for $\text{Eu}^{3+} {}^5\text{D}_1$ level emission.

Keywords: glass; phosphor; luminescence; thermometry



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1. Introduction

Germanate and tellurite glasses possess several fundamental properties that make them highly suitable for applications in optical techniques. They have a high refractive index [1], which is essential for the development of optical fibers, waveguides and lenses. Secondly, both types of glasses exhibit broad infrared transparency, making them ideal candidates for infrared optics and telecommunications [2,3]. Germanate glasses possess excellent thermal stability, allowing them to withstand high temperatures without significant structural changes, which makes them suitable for applications in high-power lasers and other optical devices operating under extreme conditions [4]. Tellurite glasses exhibit low phonon energy, which promotes the incorporation of rare-earth ions and facilitates the realization of highly efficient luminescent materials for lasers and amplifiers. In addition to favorable optical properties, tellurite glasses reveal other beneficial qualities, such as excellent chemical durability, mechanical strength and low processing temperature [5]. Compared with germanate glass, tellurite glass is characterized by lower phonon energy ($\sim 750 \text{ cm}^{-1}$) but less advantageous thermal stability [6]. The integration of tellurite and germanate components imparts unique properties to these glasses, making them highly suitable for versatile optical applications. The motivation behind choosing the germanate tellurite glass system was to take advantage of the superior qualities of both types of glass. Regarding germanate–tellurite glass, it combines the advantages of both germanate and tellurite glasses, corresponding to relatively low phonon energy, suitable thermal stability and high luminescent ion solubility [7]. The combination of numerous structural units such as TeO_4 bi-pyramid, TeO_3 trigonal pyramid and GeO_4 tetrahedron or GeO_3 octahedron gives rise to considerable structural diversity. Accordingly, the location of rare earth ions in these variable surroundings may result in intense and broad band optical spectra [8]. Therefore, this

amorphous system is a promising optical and luminescent material that is useful for various photonic applications.

Optical materials doped with Tb^{3+} and Eu^{3+} ions have gained significant attention for their applications as luminescent materials [9–11]. Tb^{3+} ions exhibit strong green emission, while Eu^{3+} ions display characteristic red emission when excited by suitable wavelengths of light. By incorporating these ions into host materials such as glasses, crystals or nanoparticles, luminescent materials with tunable emission properties can be achieved [12]. Furthermore, substantial and effective coupling between terbium and europium can be observed and studied in some optical materials. For instance, Solarz et al. recently documented the phenomena of excitation, emission and energy transfer between Tb^{3+} and Eu^{3+} at low and room temperatures. An effective 60% yield of Tb-Eu energy transfer in GAB (20 at.%Tb, 20 at.%Eu) was achieved, and the efficient impact of terbium on the relaxation dynamic of europium luminescent level was also recognized [13]. These different materials find applications in various fields, including lighting, displays and sensors, especially in luminescence thermometry—a technique for measuring temperature based on the temperature-dependent emission characteristics of dopant ions [14]. Tb^{3+} and Eu^{3+} ions exhibit characteristic emissions that can be sensitive to temperature change, making them suitable for this purpose [15]. The current hosts for Eu^{3+} and Tb^{3+} used in the mentioned fields include polycrystalline oxides [16–18].

Glass materials offer the advantage of tailoring the glass composition to achieve specific temperature sensitivity and response ranges [19,20]. These materials are valuable in various applications, including temperature monitoring in harsh environments, biological systems and optoelectronic devices, where conventional thermometers may not be practical or suitable. Some examples of tellurite glasses studied as luminescent temperature sensors include Nd-doped germanate–tellurite glasses [21].

In this paper, novel GTS glasses (50 GeO_2 –35 TeO_2 –15 SrF_2 doped with Eu^{3+} and Tb^{3+}) are studied by means of UV/VIS spectroscopy. The absorption, excitation, emission spectra and luminescence decay times are determined in order to evaluate their potential applications. Judd–Ofelt (J-O) parameters [22,23] are calculated based on Eu^{3+} emission spectra.

2. Materials and Methods

The glass samples of general composition, (50 – x – y) GeO_2 –35 TeO_2 –15 SrF_2 –x Eu_2O_3 –y Tb_4O_7 , where x = 0, 0.2, 1, 2, 4 and y = 0, 0.2, 1, 4, were prepared by a conventional melt-quenching method. The starting materials, GeO_2 (Sigma Aldrich, Burlington, MA, USA, 99.998%), TeO_2 (Alfa Aesar, Ward Hill, MA, USA, 99.99%), SrF_2 (Alfa Aesar, 99.99%), Eu_2O_3 (Alfa Aesar, 99.99%) and Tb_4O_7 (Alfa Aesar, 99.998%), were weighed and mixed in a porcelain crucible. Then, the mix was placed in a corundum crucible and put into a preheated furnace at the temperature of 1000 °C for 45 min to melt the starting materials. Next, the melt was taken out of the furnace and poured onto a brass plate at room temperature. The resulting glass was annealed at 350 °C for 12 h to remove residual stress. Parts of the samples were ground into powder before measurements to ensure uniformity of the light scattering and produce comparable luminescence intensity output. The powder X-ray diffraction patterns in the 2θ range 10–80° were measured in a linear PIXcel detector (PANalytical, Almelo, The Netherlands) using Cu K α radiation ($\lambda = 1.54056 \text{ \AA}$).

Raman scattering was measured using a Renishaw (Wotton-under-Edge, UK) InVia Raman spectrometer equipped with a confocal DM 2500 Leica optical microscope, a thermoelectrically cooled CCD as a detector and an argon laser operating at 488 nm. IR spectra were measured with a Nicolet (Madison, WI, USA) iS50 FT-IR spectrometer using pellets of powdered glasses mixed with KBr. The spectral resolution of Raman and IR spectra was set to 2 cm^{-1} .

The excitation and emission spectra, as well as the luminescence decay times, were recorded using the Edinburgh Instruments (Edinburgh, UK) FLS 1000 equipped with a

Xenon lamp. The emission as a function of temperature was measured using Linkam (Surrey, UK) THMS 600 Heating/Freezing Stage (The McCrone group, Westmont, IL, USA), a 375 nm laser diode as an excitation source and the Hamamatsu Photonic multichannel analyzer PMA-12 equipped with a BT-CCD linear image sensor (Hamamatsu Photonics K.K, Shizuoka, Japan). The quantum yield (QY) was measured using the Hamamatsu PMA-12 spectrophotometer equipped with an integrating sphere. The accuracy of measurement is estimated to be $\pm 2.5\%$.

The J-O parameters were calculated using refractive index values $n = 1.835$ (taken from [24]) and integrated intensities of ${}^5D_0 \rightarrow {}^7F_J$ ($J = 1, 2, 4$ and 6) lines denoted as I_{0J} , using the following Equations (1) and (2) [25]:

$$A_{0-J} = A_{0-1} \frac{I_{0J} \nu_{01}}{I_{01} \nu_{0J}} \quad (1)$$

where ν_{0J} are the wavenumbers, and A_{0-J} are the transition rates of $4f-4f$ Eu^{3+} transitions. A_{0-0} can be obtained from Equation (2)

$$A_{0-1} = n^3 (A_{0-1})_{vac} \quad (2)$$

where $(A_{0-1})_{vac}$ is equal to 14.64 s^{-1} [26].

Then, the Ω_J parameters were calculated from Equation (3)

$$A_{J-J'} = \frac{64\pi^4 \nu^3 e^2}{3h} \cdot \frac{1}{4\pi\epsilon_0} \chi \Omega_J \cdot \left| \langle 4f^N SLJ \| U^\lambda \| 4f^N S' L' J' \rangle \right|^2 \quad (3)$$

where the nonzero reduced matrix parameters $\langle U^\lambda \rangle$ equal to 0.0032, 0.0023 and 0.0002 for $\lambda = 2, 4$ and 6 , respectively [27]; ν is transition frequency; e is the electron charge; h is a Planck constant; ϵ_0 is a vacuum permittivity; and χ is equal $n(n^2 + 2)^2/9$. Based on the radiative transition rates A_r and experimental transition rates A_{tot} (and its inverse, the experimental decay time τ_{exp}), the internal quantum efficiency η was calculated using Equation (4)

$$\eta = \frac{A_r}{A_{tot}} = \frac{\tau_{exp}}{\tau_r} \quad (4)$$

The radiative decay time (τ_r) can be calculated from Equation (5) [28]:

$$\tau_r = \frac{1}{A_r} \quad (5)$$

where A_r is given by Equation (6):

$$A_r = \sum_{J=1,2,4,6} A_{0-J} \quad (6)$$

3. Results and Discussion

The GTS glass is transparent, and the Eu^{3+} and Tb^{3+} dopants do not cause sample coloration (Figure 1a top). Under UV light (Figure 1a bottom), the single-doped samples doped with Eu^{3+} and Tb^{3+} exhibit orange, red-orange and blue-green emission, respectively. The co-doped sample exhibits purple-pink emission. All the perceived colors are a result of the mixing of original Eu^{3+} and Tb^{3+} emissions with the scattered blue light introduced by the excitation source. The amorphous structure of the samples was confirmed using XRD (Figure 1b). No Eu -, Tb -, and Eu , Tb co-doped samples exhibit sharp diffraction peaks, indicating no crystalline phase in the volume of the sample.

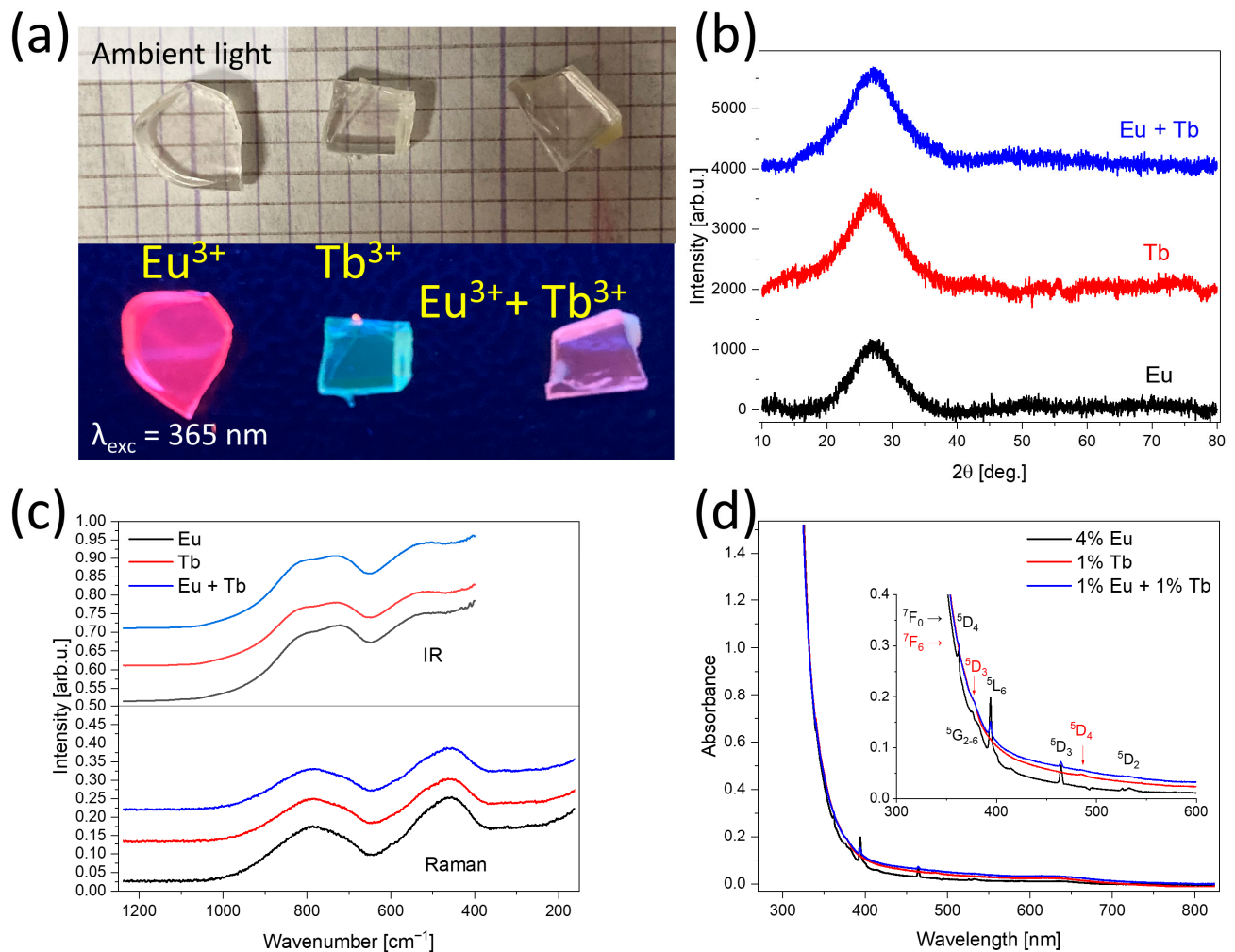


Figure 1. (a) Photographs of the single- and co-doped glass samples in ambient light and under UV excitation; (b) the XRD results; (c) IR and Raman spectra; (d) absorption spectra of the Eu³⁺-, Tb³⁺- and co-doped glasses.

The Raman, IR and absorption spectra were aimed to investigate whether the doping with Eu³⁺, Tb³⁺ or both has an impact on the structural properties of the studied GTS glasses. The IR and Raman spectra measured for Eu-doped, Tb-doped and Eu/Tb-co-doped samples (Figure 1c) reveal no significant differences between each other and are very similar to the ones reported for Nd³⁺-doped germanate–tellurite glasses [21]. The bands on the Raman spectrum can be attributed to the stretching vibrations of Ge–O–Ge bridges in GeO₄ tetrahedra (788 cm⁻¹), the stretching vibrations of TeO₃ trigonal pyramid and TeO₄/TeO₃₊₁ trigonal bipyramids (735 cm⁻¹), and the bending vibrations of Ge–O–Ge and Te–O–Te bridges (446 cm⁻¹) [8,29,30]. The IR spectra (Figure 1c) comprise mainly the broad band with two components located at 722 and 822 cm⁻¹, which can be assigned to the Te–O–Te vibrations and Ge–O–Ge vibrations, respectively. The other broad band at 525 cm⁻¹ is characteristic of GeO₆ octahedra [31].

The absorption spectra (Figure 1d) revealed that besides the appearance of weak, narrow lines originating from the 4f–4f transition of lanthanides, the dopants do not alter the absorption edge of the GTS glass.

The excitation and emission spectra of Eu³⁺-doped glass samples are characteristic of Eu³⁺ in amorphous material [32], with the maximum excitation at 394 nm and the maximum Eu³⁺ emission at 612 nm (Figure 2a). The wide band at the excitation spectrum can be a result of the host–Eu³⁺ energy transfer. The large bandgap and low phonon energies

allow for observing the emission from the higher-lying $^5D_{1,2,3}$ levels [33] (Figure 2a inset, Figure S1). The integral intensity was the highest for the sample doped with 2% Eu_2O_3 (Figure 2b). Above that concentration, the quenching mechanism caused the intensity to decrease [34].

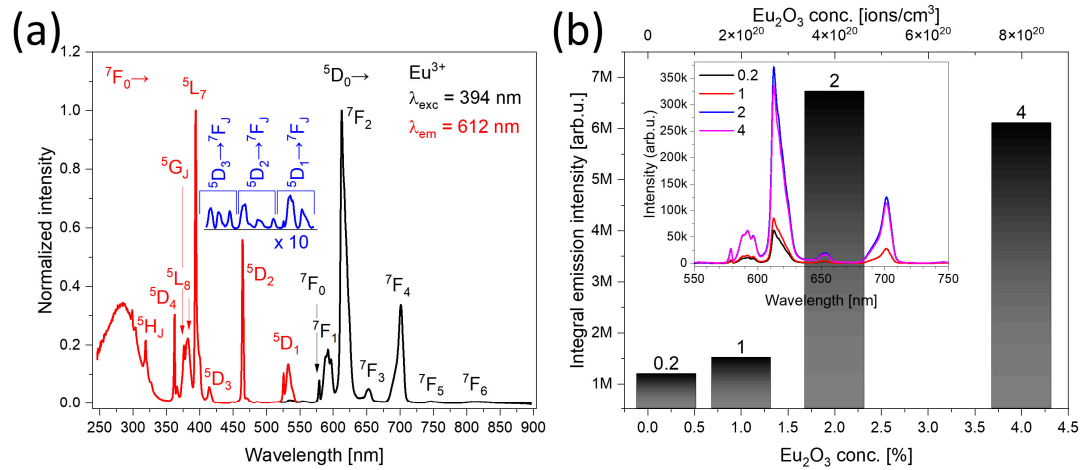


Figure 2. (a) Excitation and emission spectra of Eu^{3+} -doped samples; (b) emission intensity as a function of dopant concentration; inset: emission spectra of samples with different Eu^{3+} concentrations.

The Tb^{3+} -doped samples exhibit efficient excitation bands in the 340–380 nm region, and predominantly green emission is a result of four narrow emission lines (Figure 3a). The maxima of excitation and emission are located at 378 nm and 542.2 nm, respectively. As in the case of Eu^{3+} , it is possible to observe the weak emission from the higher-lying 5D_3 level [35] (Figure 3a inset). The sample doped with 4% Tb_4O_7 exhibits the strongest luminescence (Figure 3b); however, to ensure the stability of the glass, for the co-doped samples, the concentration of 1% was chosen for both Eu^{3+} and Tb^{3+} .

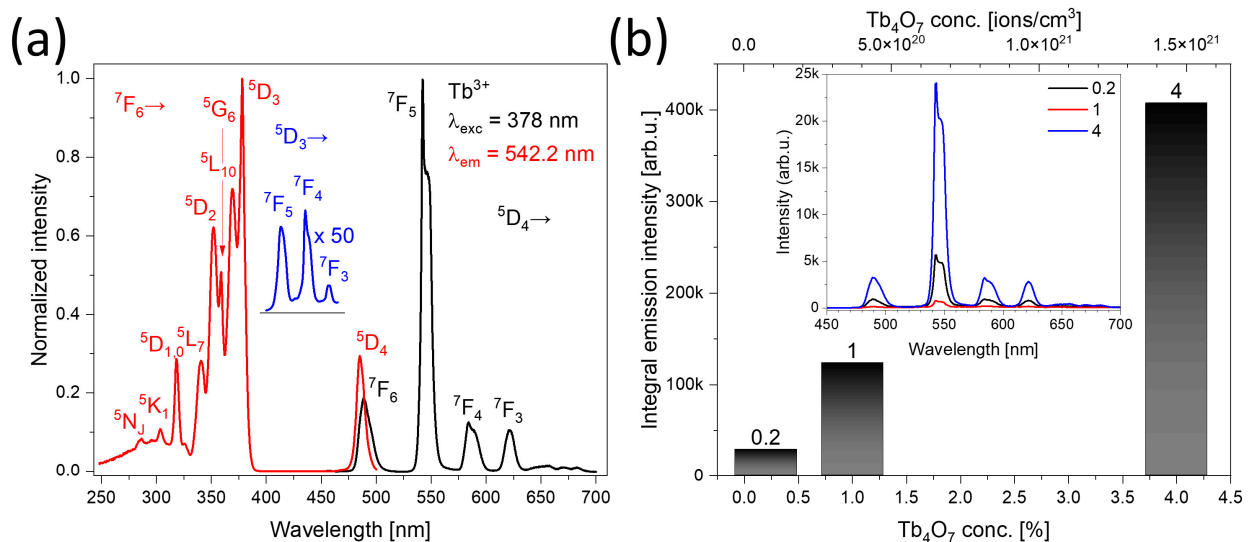


Figure 3. (a) Excitation and emission spectra of Tb^{3+} -doped samples; (b) emission intensity as a function of dopant concentration; inset: emission spectra of samples with different Tb^{3+} concentrations.

The co-doped sample exhibits both Eu^{3+} and Tb^{3+} emission when excited at 375 nm (Figure 4a). Despite the excitation wavelength being a better fit for Tb^{3+} , the Eu^{3+} emission is more intense than that of Tb^{3+} due to the aforementioned higher concentration quenching rate for Tb^{3+} ions. The Commission internationale de l'éclairage (CIE) coordinates were calculated for both single-doped and co-doped samples, and the luminescence is located at

the red (Eu^{3+}), green (Tb^{3+}) or yellow (co-doped Eu^{3+} and Tb^{3+}) regions of the chromaticity scale (Figure 4b).

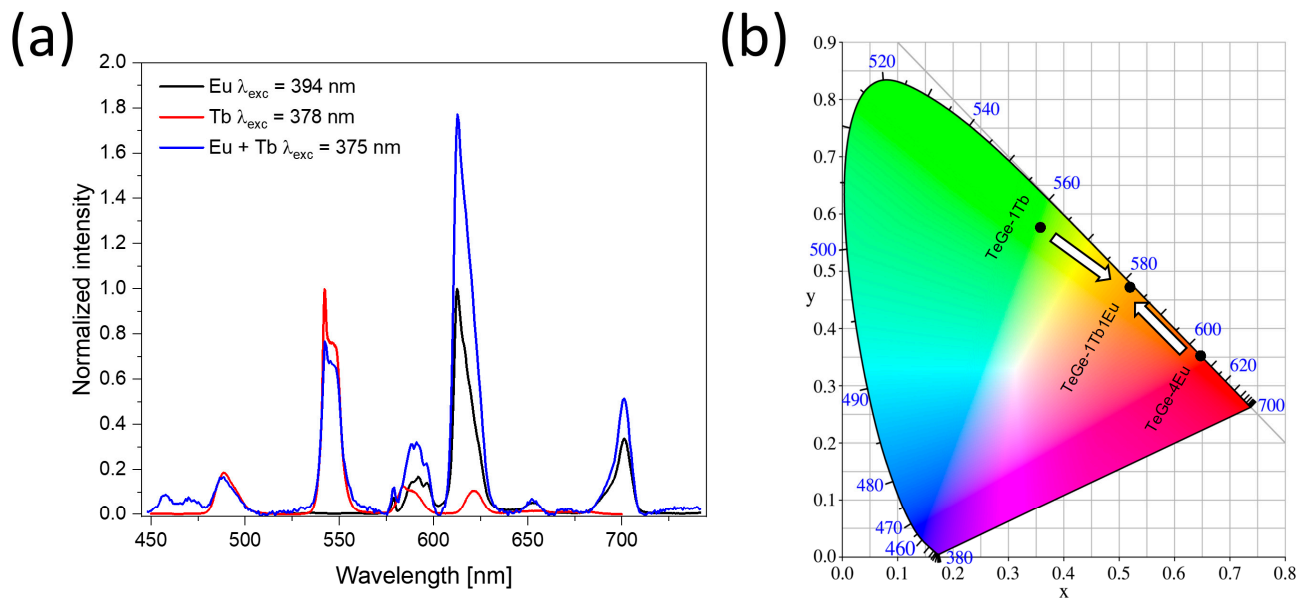


Figure 4. (a) Emission spectra of Eu^{3+} -doped, Tb^{3+} -doped and Eu^{3+} , Tb^{3+} -co-doped glass; (b) CIE diagram of the Eu^{3+} -doped, Tb^{3+} -doped and Eu^{3+} , Tb^{3+} -co-doped glass.

The color purity was calculated based on the Equation (7) [36]:

$$CP = \frac{\sqrt{(x - x_i)^2 + (y - y_i)^2}}{\sqrt{(x_d - x_i)^2 + (y_d - y_i)^2}} \quad (7)$$

where (x, y) is the CIE coordinates of the sample, $(x_i, y_i) = (0.3333, 0.3333)$ is the coordinates of white light, and (x_d, y_d) is the coordinates of the dominant wavelength of the emission. The color purity was calculated to be equal to 100% for Eu^{3+} -doped glass and 80% for Tb^{3+} -doped glass. The Judd–Ofelt parameters were calculated for the Eu^{3+} -doped sample (Table 1). The Ω_λ parameters follow the $\Omega_2 > \Omega_6 > \Omega_4$ relation, which has been reported for other germanate glass systems [19,37]. Based on the Judd–Ofelt theory, the radiative luminescence decay time of the $^5\text{D}_0$ excited state was calculated to be equal to 1.6 ms, which, compared to experimentally derived mean decay time, yields quantum efficiency equal to 86%.

Table 1. The Judd–Ofelt parameters (Ω_λ), radiative (τ_r) and experimental (τ_{exp}) decay time and internal quantum efficiency (η) for the Eu^{3+} -doped sample.

Ω_2 (10^{-20} cm^2)	Ω_4 (10^{-20} cm^2)	Ω_6 (10^{-20} cm^2)	τ_r (ms)	τ_{exp} (ms)	η (%)
6.7	4.3	6.4	1.6	1.4	86

The measured external quantum yield (Figure 5a) of the Eu^{3+} -doped glass has an expected maximum at 395 nm excitation and is equal to $23.0 \pm 2.5\%$. The external quantum yield for a Tb^{3+} -doped sample is lower, with a maximum at 375 nm excitation and equal $6.9 \pm 2.5\%$.

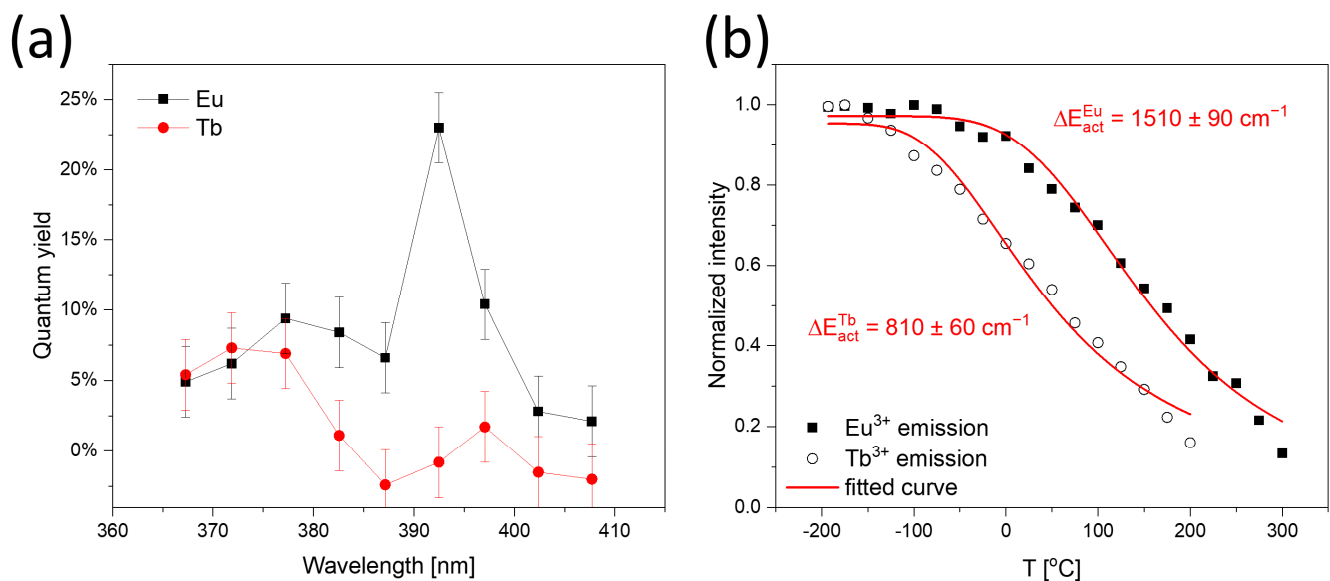


Figure 5. (a) External quantum yield in the function of excitation wavelength for Eu- and Tb-doped samples; (b) thermal quenching curves for Eu and Tb emission in studied glass system; red curves were fitted function (8), and ΔE_{act} is an activation energy for thermal quenching. R^2 (COD) of fitted curves are equal to 0.98887 and 0.98518 for Eu and Tb, respectively.

The thermal quenching curves (Figure 5b) reveal the discrepancy between the Eu³⁺ and Tb³⁺ luminescence in function of temperature. The Eu³⁺ emission is more stable, as the activation energy ΔE_{act} derived from the fitting Equation (8) [38] is equal to $1510 \pm 90 \text{ cm}^{-1}$, which is twice as high as for Tb³⁺ ($810 \pm 60 \text{ cm}^{-1}$). The difference in activation energy stems from the different energetic structures of the excited states in Eu³⁺ and Tb³⁺, as well as the different locations of these levels with respect to the conduction band of the host material [39], which causes different nonradiative relaxation mechanisms.

$$I = \frac{I_0}{1 + \frac{\Gamma_0 - \Gamma_v}{\Gamma_v} \frac{\Delta E_{act}}{kT}} \quad (8)$$

where I is the intensity, I_0 is the initial intensity for the lowest temperature, ΔE_{act} is the activation energy for thermal quenching, T is the temperature, and k is the Boltzmann constant. The expression $\frac{\Gamma_0}{\Gamma_v}$ can be omitted as a constant value.

Due to the different activation energies and thermal quenching profiles of the co-dopants, it is possible to use the co-doped material as a temperature sensor. To estimate and compare its performance, the emission spectra of the co-doped samples were measured in the temperature range of -193 – 200 °C (Figure 6a). The increase in the temperature induces a change in the emission spectrum due to a change in the Tb³⁺ to Eu³⁺ emission ratio, which causes the chromaticity shift (Figure 6b). The resulting chromaticity shift (ΔE_C) caused by the heating of the sample from 0 up to 100 °C was calculated using Equation (9) and is equal to $80 \cdot 10^{-3}$, which is twice as much as found for the commercial phosphors [40], yet still within the order of magnitude for LED phosphors [41,42].

$$\Delta E_C = \sqrt{(u_f - u_i)^2 + (v_f - v_i)^2 + (w_f - w_i)^2} \quad (9)$$

where $u = 4x/(3 - 2x + 12y)$, $v = 9y/(3 - 2x + 12y)$ and $w = 1 - u - v$. The x and y are the chromaticity in CIE 1931 color space, and the i and f indices indicate the chromaticity points before and after heating, respectively.

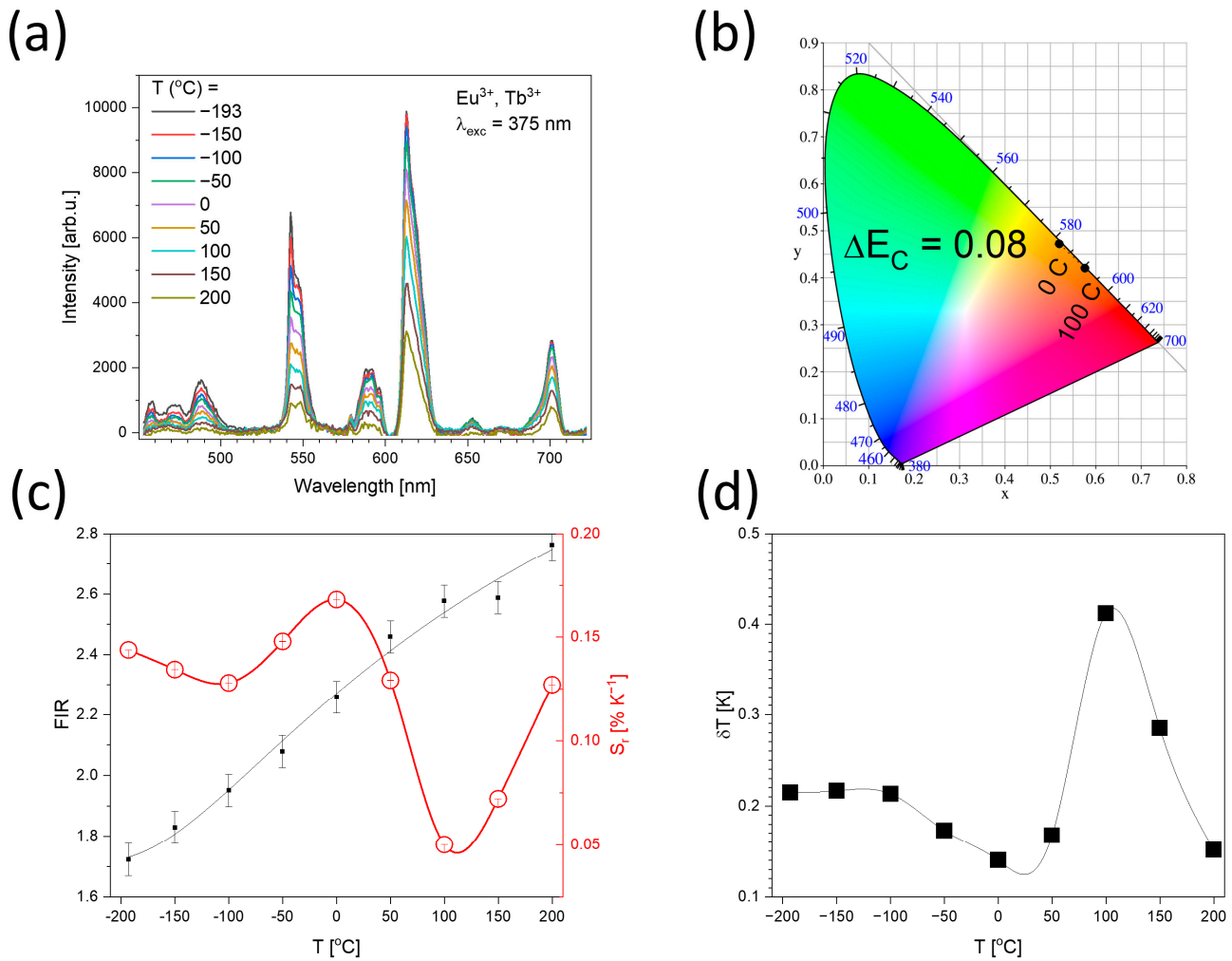


Figure 6. (a) Temperature-dependent emission spectra of Eu^{3+} , Tb^{3+} -co-doped sample; (b) CIE diagram of the Eu^{3+} , Tb^{3+} -co-doped glass chromaticity shift; (c) fluorescence intensity ratio and relative sensitivity. The red line serves only to guide the eyes. (d) Thermal resolution of the temperature sensor based on FIR. The black line serves only to guide the eyes.

The fluorescence intensity ratio (FIR) (Figure 6c) was calculated between the most intense emission lines for each co-dopant: $^5\text{D}_0 \rightarrow ^7\text{F}_2$ for Eu^{3+} (at 612 nm) and $^5\text{D}_4 \rightarrow ^7\text{F}_5$ for Tb^{3+} (at 542.2 nm). The relative sensitivity (Figure 6c) was then calculated from Equation (10) [14]:

$$S_r(T) = \frac{1}{\text{FIR}(T)} \left| \frac{\partial \text{FIR}(T)}{\partial T} \right| \quad (10)$$

The FIR profile is stably increasing in the full range of temperature, and the relative sensitivity has a maximum at 0 °C equal to 0.16% K^{−1}, which is similar to that reported for $\text{Tb}^{3+}/\text{Eu}^{3+}$ -doped $\text{Sr}_3\text{GdNa}(\text{PO}_4)_3\text{F}$ phosphor [43], but generally lower than that reported for $\text{Eu}^{3+}/\text{Tb}^{3+}$ -co-doped crystalline (ranging from 0.37 to 5.04% K^{−1}) [18,43–47] and glass materials (ranging from 0.36 to 1.1% K^{−1}) [48,49]. The thermal resolution of the temperature sensor based on FIR was calculated using Equation (11),

$$\delta T = \frac{1}{S_r} \cdot \frac{\Delta \text{FIR}}{\text{FIR}} \quad (11)$$

where ΔFIR is the uncertainty of the determination of FIR with regard to the exponential function $\text{FIR} = B + C \exp(-\Delta E'/kT)$ [50]. The thermal resolution is around or below 0.2 K for

low-temperature regions, with an increase up to 0.4 K correlating with a drop of sensitivity at 100 °C (Figure 6d).

In fact, a relaxation of terbium and europium luminescent excited states in the studied glasses is governed by competing radiative and nonradiative processes. The nonradiative phenomena are attributed to intra-ion multiphonon relaxation and the energy transfer mechanisms involved in inter-ionic interactions promoted by the increase in rare earth concentration. The related energy levels scheme of Tb^{3+} and Eu^{3+} in the studied glasses is presented in Figure 7. The $^5\text{D}_4$ (Tb) and $^5\text{D}_0$ (Eu) excited states are characterized by a significantly larger energy gap to the lower-lying levels in relation to $^5\text{D}_3$ (Tb) and $^5\text{D}_1$ (Eu) manifolds. Moreover, in view of the various routes of the possible energy transfer phenomena, the substantially different effects of temperature and nonradiative processes on the Tb^{3+} and Eu^{3+} luminescent levels decay can be explored. To verify this assumption, the experimental lifetimes of the aforementioned terbium and europium excited states were examined at room temperature and as a function of temperature up to 400 °C.

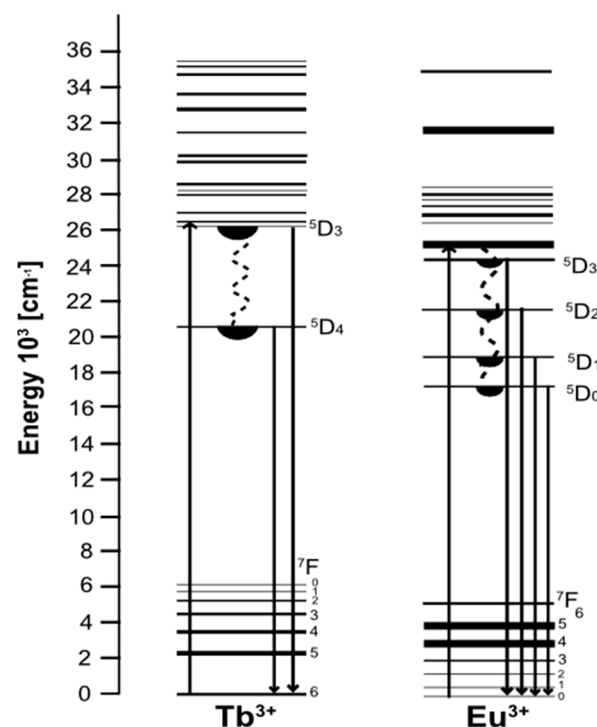


Figure 7. Scheme of Tb^{3+} and Eu^{3+} levels in germanate–tellurite glass.

The luminescence decay curves are single-exponential for both Eu^{3+} - and Tb^{3+} -doped samples (Figures 8a,b and 9a,b). The decay curves of the lowest emitting levels ($^5\text{D}_0$ for Eu^{3+} and $^5\text{D}_4$ for Tb^{3+}) of both rare earth dopants are similar and have time constants $\tau = 1.4$ ms for Eu^{3+} and $\tau = 1.7$ ms for Tb^{3+} . The energy transfer from Tb to Eu was investigated by comparing the Tb^{3+} emission decay in single-doped and co-doped samples (Figure S2). No difference was observed, and consequently, it was concluded that no energy $\text{Tb}^{3+} \rightarrow \text{Eu}^{3+}$ transfer occurs in this system. The decay curve of the $^5\text{D}_1$ level of Eu^{3+} in the Eu-doped sample was recorded as single-exponential with a decay time of 35 μs (Figure 8b). This observation is consistent with other Eu-doped materials, in which the decay time of $^5\text{D}_1$ level is significantly shorter than that of $^5\text{D}_0$ [51]. At elevated temperatures, the decay times become shorter. The decay time of the $^5\text{D}_0$ level remains stable up to 250 °C; in higher temperatures, it becomes gradually shorter down to 0.95 ms at 400 °C (Figure 8c). The decay time of the $^5\text{D}_1$ level is much less thermally stable due to multiphonon relaxation [32]

and decreases to 12 μs at 400 $^{\circ}\text{C}$ (Figure 8d). The values of the decay times were fitted with Equation (12) [52].

$$\tau(T) = \frac{\tau_R \cdot \tanh\left(\frac{h\nu}{2kT}\right)}{1 + \left(\frac{\tau_R \cdot \tanh\left(\frac{h\nu}{kT}\right)}{\tau_{NR}} \cdot \exp\left(\frac{-\Delta E_q}{kT}\right)\right)} \quad (12)$$

where τ_R is the radiative lifetime at 0 K, $h\nu$ is an average phonon energy for this host, τ_{NR} is the nonradiative relaxation lifetime, k is the Boltzmann constant, and ΔE_q is the activation energy of the process. The parameters of the fit are listed in Table S1.

As expected, the decay time of the $^5\text{D}_4$ level of Tb^{3+} (Figure 9a,c) is much longer than that of the $^5\text{D}_3$ level (Figure 9b,d), which is equal to 38 μs . This is in line with other reported decay times of this level [53]. Both $^5\text{D}_4$ and $^5\text{D}_3$ levels in Tb-doped samples are significantly less stable than $^5\text{D}_0$ and $^5\text{D}_1$ of Eu^{3+} in the same glass system, which confirms the findings of the thermal stability of emission intensity and calculated activation energies of thermal quenching.

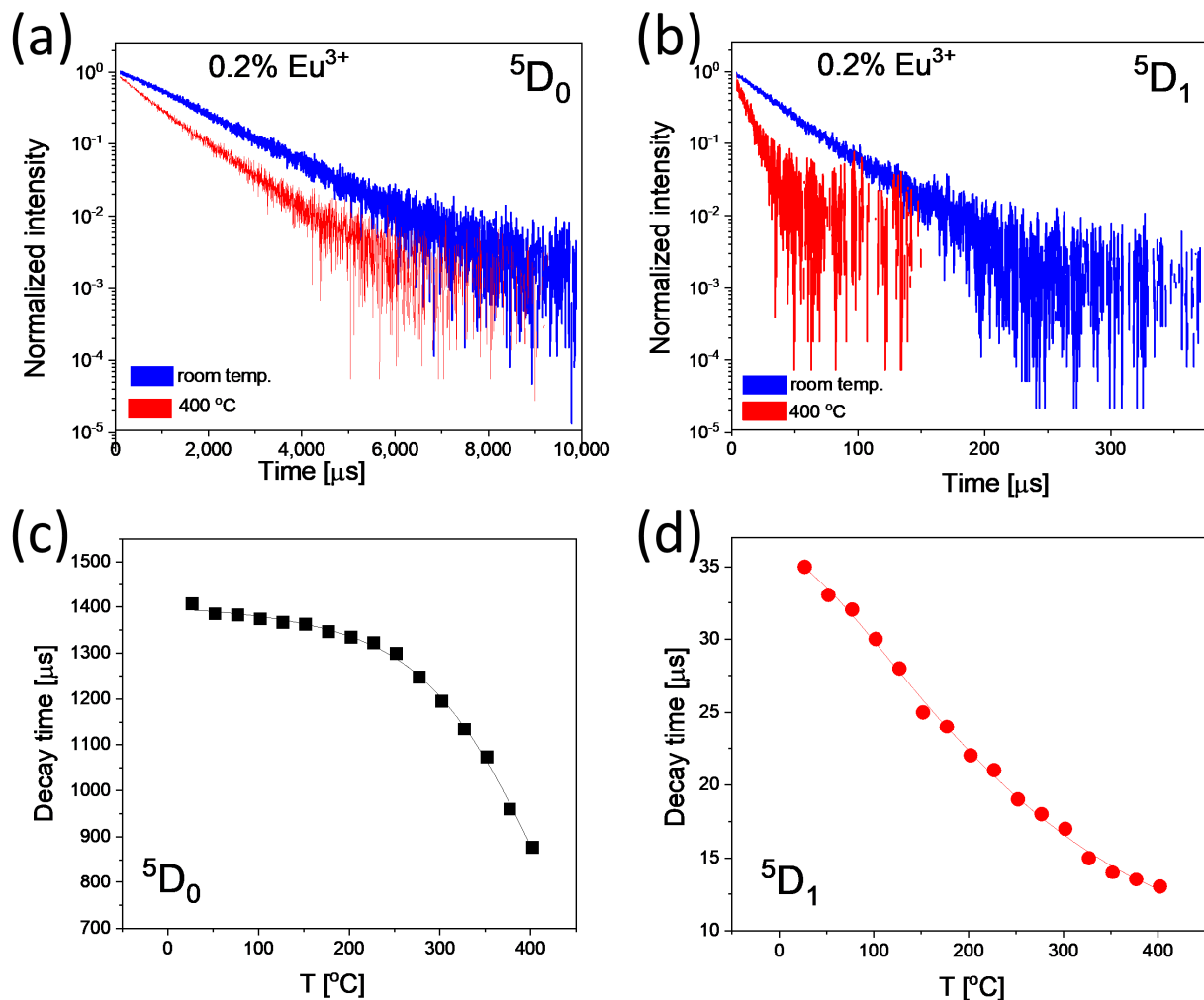


Figure 8. Decay curves of 0.2% Eu-doped sample's emission from $^5\text{D}_0$ (a) and $^5\text{D}_1$ (b) levels recorded at room temperature and 400 $^{\circ}\text{C}$. Decay time values of emission from $^5\text{D}_0$ (c) and $^5\text{D}_1$ (d) levels fitted with function (12).

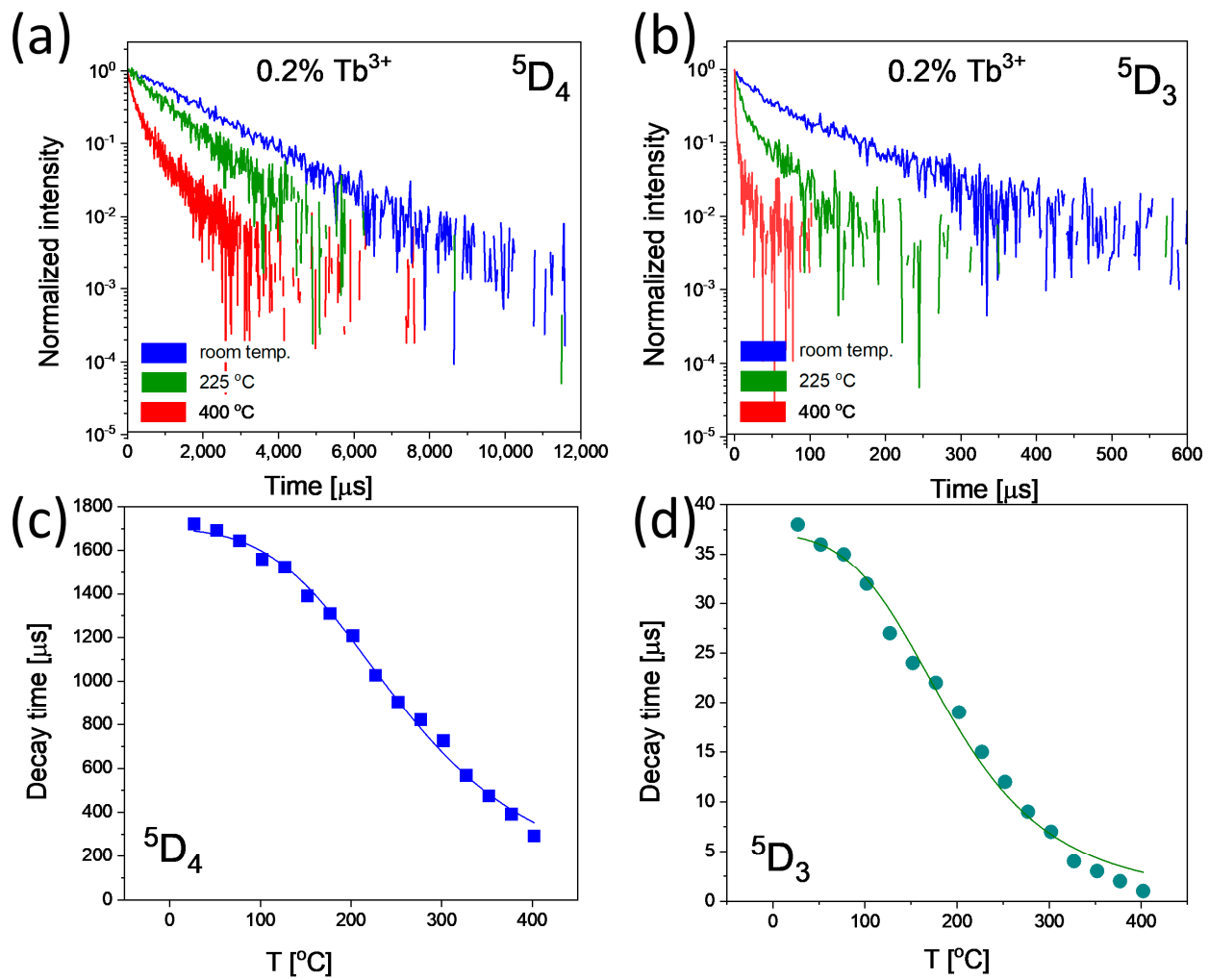


Figure 9. Decay curves of 0.2% Tb-doped sample's emission from 5D_4 (a) and 5D_3 (b) levels recorded at room temperature, 225 °C and 400 °C. Decay time values of emission from 5D_4 (c) and 5D_3 (d) levels fitted with function (12).

The impact of temperature on the values of the decay times allows for lifetime-based thermometry to be estimated. The relative sensitivity S_r^τ of the lifetime-based thermometer (Figure 10a,b) and the thermal resolution δT^τ are calculated analogically to the ratiometric sensitivity using the following Equations (13) and (14) [54]:

$$S_r^\tau(T) = \frac{1}{\tau(T)} \left| \frac{\partial \tau(T)}{\partial T} \right| \quad (13)$$

$$\delta T^\tau = \frac{1}{S_r} \cdot \frac{\Delta \tau}{\tau} \quad (14)$$

where $\Delta \tau$ is the uncertainty of the decay time, analogically to ΔFIR .

The sensitivities of the Tb^{3+} emission decay time (Figure 10b) are higher than those of Eu^{3+} emission (Figure 10a), especially at elevated temperatures, where the sensitivity of the 5D_3 level decay reaches $0.95\% \text{ K}^{-1}$ at 250 °C, which is comparable with reported $1\% \text{ K}^{-1}$ obtained by Ryadun et al. in crystalline $\text{CsGd}(\text{MoO}_4)_2:\text{Yb}^{3+}\text{-Tb}^{3+}$ [55]. But the sensitivity of the Eu^{3+} emission decay of the 5D_1 level is moderately stable in the wide temperature range and is equal to $0.3\% \text{ K}^{-1}$ at 225 °C, which is higher than the values reported for Eu^{3+} emission in tantalate powders by Hua et al. [56] equal $0.18\% \text{ K}^{-1}$. The Eu^{3+} emission decay of the 5D_1 level also exhibits the consistently best thermal resolution around 5 K in the

300–500 K range (Figure 10c). However, the thermal resolution of the lifetime thermometry is worse than that of FIR.

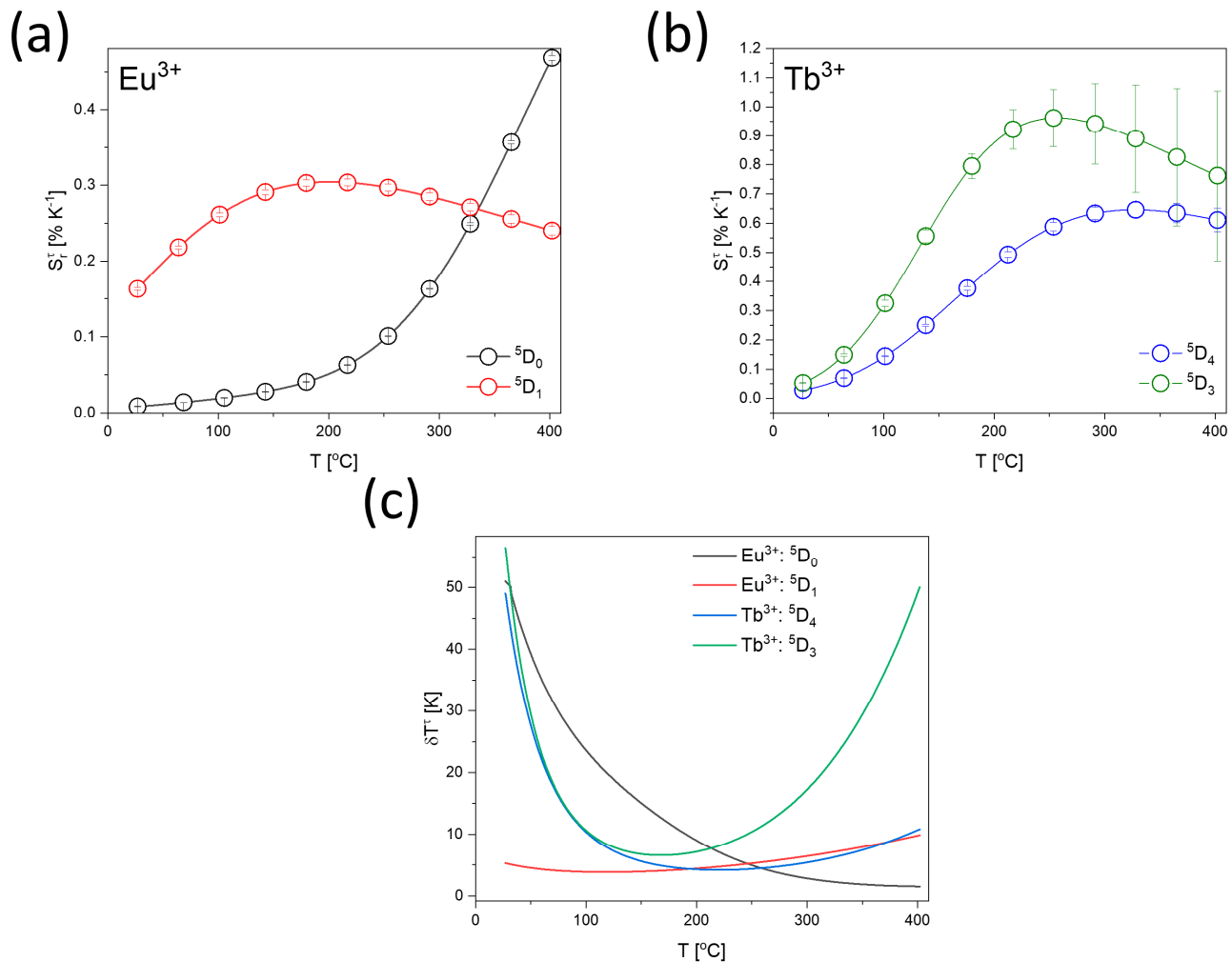


Figure 10. (a) Relative temperature sensitivities of Eu³⁺ ⁵D₀ and ⁵D₁ lifetime; (b) relative temperature sensitivities of Tb³⁺ ⁵D₄ and ⁵D₃ lifetime; (c) thermal resolutions of the temperature sensors based on lifetime.

4. Conclusions

The GTS glasses are an excellent host for Eu³⁺ and Tb³⁺ dopants. The luminescence thermometry can be realized in both ratiometric and lifetime-based modes. In both cases, the relative sensitivities are comparable with Eu³⁺- and Tb³⁺-doped glasses and crystalline materials. The ratiometric thermometry is more sensitive at low temperatures (below 0 °C) and contrary to lifetime-based thermometry at room temperature and high temperature.

The color purity of the emission is very good for both—100% for Eu³⁺ and 80% for Tb³⁺—and the mixing results in efficient yellow luminescence, which has the potential to be useful for wLED sources. The Eu³⁺ is especially very efficient, with a quantum yield equal to 23% under 390 nm excitation.

No energy transfer between Eu³⁺ and Tb³⁺ dopants was detected.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/app14104198/s1>, Figure S1: The wide-scale emission spectrum of GTS: Eu glass; Figure S2: Decay curves of Tb³⁺ emission in Tb- and Eu, Tb-co-doped samples.; Table S1: Fitting parameters of decay time thermal quenching.

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