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Luminescence properties of Ba₄Yb₃F₁₇:Er³⁺ nanocrystals embedded in glass ceramics for optical thermometry

 Sixing Li,^a Liang Li,^a Wenming Wang,^a Hongmei Chen,^a ^a Yong Li,^a ^{*,a} Xianshan Huang^{*,a} and Yan Pan^{*ab}

Transparent glass ceramics (GCs) containing Ba₄Yb₃F₁₇:Er³⁺ nanocrystals were successfully fabricated by a traditional melt-quenching method. The formation of Ba₄Yb₃F₁₇ nanocrystals was confirmed by X-ray diffraction, transmission electron microscopy, and selected area electron diffraction. Compared with the precursor glass, the enhanced emission intensity and lifetime of GCs indicate that the Er³⁺ ions incorporate into the Ba₄Yb₃F₁₇ nanocrystals after crystallization. The color tuning properties with doping under 980 nm excitation have been systematically discussed. It was found that the red/green ratio increased with Er³⁺ ion doping and the corresponding color changed from greenish-yellow to yellow-green. Furthermore, the temperature-dependent luminescence properties were studied in detail by the fluorescence intensity ratio (FIR) technique. The monotonic change of FIR with temperature indicates that this material is suitable for temperature sensing. At a temperature of 450 K, the relative sensitivity of the prepared sample reached its maximal value of 0.20% K⁻¹. The results show that the GCs containing Ba₄Yb₃F₁₇:Er³⁺ nanocrystals are candidate materials for temperature sensing.

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1. Introduction

Currently, lanthanide (Ln) ion doped upconverting materials with temperature-dependent luminescence properties have attracted noticeable attention due to their potential applications in optical heaters, optical refrigeration, temperature sensors, cancer treatment, and thermal imaging.^{1–5} The upconverting materials are commonly used in non-contact temperature sensors considering their superior properties in temperature detection, such as fast response, high spatial resolution, excellent precision, and noninvasive operation mode.^{6,7} Jiang *et al.*⁸ reported the thermometry at the nanoscale, which has lifetime-based thermal sensitivities and remarkable fluorescence intensity in the physiological temperature range. Without contacting the measured object, the fluorescence intensity ratio (FIR) technique of optical materials based on non-contact temperature sensors can be used in several particular circumstances, such as biological imaging systems, corrosive circumstances, electrical transformer temperature in power stations, and fire detections. The FIR of two thermally coupled energy levels (TCELs) from Ln ions based on the optical temperature sensor is independent of the variations of test

conditions including external disturbance, fluctuations in signal losses, and excitation power.^{9,10} Thus, the optical thermometer possesses high resolution and accuracy compared to other optical methods, such as amplified spontaneous emission, raise time, and decay time.^{11,12}

The separation between TCELs should be in the 200–2000 cm⁻¹ range to avoid the strong overlapping of two emissions and ensure the existence of a sufficient population of the higher level of active optical ions in the interesting temperature range.^{13,14} A pair of adjacent levels with a narrow energy gap (ΔE) in the 200–2000 cm⁻¹ range can be found in some trivalent Ln ions, such as Dy³⁺ (⁴I_{15/2} and ⁴F_{9/2}), Nd³⁺ (⁴F_{5/2}/²H_{9/2} and ⁴F_{3/2}), Ho³⁺ (⁵F₁/⁵G₆ and ⁵F_{2,3}/³K₈), Tm³⁺ (³F_{2,3} and ³H₄), and Er³⁺ (²H_{11/2} and ⁴S_{3/2}).^{15–19} Among these Ln ions, Er³⁺ is considered an excellent ion for optical thermometry because of its applicable energy gap (~800 cm⁻¹) and two intense green emissions from ²H_{11/2} and ⁴S_{3/2} TCELs.¹ The demand for optical thermometers has greatly stimulated the research of Er³⁺ doped materials. Yb³⁺/Er³⁺ codoped NaYF₄ transparent glass ceramics (GCs) used as an optical-fiber temperature sensor was reported by Jiang *et al.*¹¹; Yang *et al.*²⁰ successfully synthesized Yb³⁺/Er³⁺ codoped La₂O₂S phosphor and revealed its promising application in optical thermometry; Zhang *et al.*²¹ have reported Er³⁺ doped SrYbInO₄ phosphor used as optical thermometry.

The selection of an appropriate host is essential to obtain excellent temperature detecting performance. The effective luminescence of Ln ions in a suitable host can make spectral acquisition more accurate.²² To improve the properties of an

^aSchool of Mathematics and Physics, Anhui University of Technology, Maanshan 243000, China. E-mail: yongli@ahut.edu.cn; huangxs@ahut.edu.cn; yanpan@ahut.edu.cn

^bAnalysis and Testing Central Facility, Anhui University of Technology, Maanshan 243000, China



optical thermometer, the temperature-dependent luminescence properties of many materials doped with Ln have been studied, such as glass, ceramics, fluoride nanocrystals (NCs), and organics.^{1,23–25} There is a big difference in temperature detecting performance among these different materials systems. It is hard to produce effective luminescence in glass, such as aluminosilicate and silica glass host, due to their large number of defects that act as quenching centers and high phonon energy, so they are not suitable for optical temperature measurement. Difficulties such as high preparation costs and complicated synthesis processes are the challenges of transparent ceramics. Fluoride with low phonon energy favors effective luminescence, which makes the emission spectra acquisition more easily, sensitively, and accurately. However, phase transition may occur at high temperatures due to its poor thermal stability, so the repeatability in heating-cooling cycles will be affected. Organics have the advantages of systematically adjusting their sensitivity and being compatible with biological systems, however, their stability at higher temperatures is insufficient, so its operating temperature is limited to the physiological temperature range. Therefore, it is necessary to find novel Er³⁺ ion doped materials with improved sensitivity, effective luminescence, and good stability to improve the performance of optical thermometers.

So far, many favorable results have been achieved in the research of fluoride and oxide hosts.^{7,26} Fluoride materials have been drawing widespread attention due to their low phonon energy, for example, Yb³⁺/Er³⁺ co-doped hexagonal phase NaYF₄ has shown the highest upconversion (UC) luminous efficiency so far.²⁷ In addition, the oxide materials have higher stability than fluoride, which can meet the thermal stability requirements of the temperature sensor for the host.²⁸ For practical applications, it is important to design materials with a wide operating temperature range and excellent luminous intensity. To obtain a host with high physical and chemical stability and meet the requirements of Ln ions for a low phonon energy environment, an ideal host of oxy-fluoride GCs (OXGCs) doped with Ln ions for optical thermometers have been developed. OXGCs are synthesized at suitable temperatures by controlling the crystallization of the fluoride embedded in oxide glass with a specially designed composition. OXGCs not only have the low phonon energy of fluoride but also have the high mechanical stability and durability of oxide. In addition, OXGCs have the advantages of crystal-like optical performance and low-cost synthesis.²⁹ And their ion radius and available valence are suitable for Ln ions doping.³⁰ Therefore, for temperature measurement, OXGCs have the advantages of improved sensitivity, excellent precision, and wide operating temperature range. To produce high-efficiency luminescence in OXGCs, it is necessary to distribute the Ln ions as an optical activator into the fluoride lattice and deposit the required fluoride NCs uniformly. In addition, the transparency of OXGCs needs to be considered. When the size of NCs dispersed in OXGCs is smaller than the wavelength of the light, scattering can be avoided so that the high transparency of OXGCs can be achieved. The optical temperature measurement behavior of OXGCs doped with Ln ions has been extensively studied. Hu *et al.*³¹ presented the Sm³⁺ doped NaGdF₄ GC with good stability

for optical temperature measurement; the GC containing NCs doped with Yb³⁺ and Er³⁺ ions as a temperature sensor was reported by Li *et al.*³²; Jiang *et al.*¹¹ reported the GC containing NaYF₄:Yb³⁺/Er³⁺ NCs as an optical thermometer and achieved a high sensitivity of 1.24% K⁻¹ at 300 K.

Yb³⁺ ions usually act as sensitizers in the UC process since they have a larger absorption cross-section at 980 nm and can effectively transfer energy to other Ln ions.³³ Ln ions doped OXGCs are considered as potential candidate probes for optical temperature measurement.³⁴ However, most materials including OXGCs have luminescent thermal quenching behavior, which is the luminescence decreasing phenomenon caused by nonradiative transitions of excited states at high temperatures.³⁵ Fortunately, the introduction of Yb³⁺ ions can enhance the UC luminous intensity at high temperature.³⁶ In addition, the introduction of Yb³⁺ ions can prolong the lifetime because the surface quenching mediated by energy transfer (ET) caused by heating is alleviated.³⁷ Therefore, fluoride NCs based on Yb³⁺ ions such as NaYbF₄, LiYbF₄, SrYbF₅, and BaYbF₅ have received widespread attention, and all results indicate that these UC materials are excellent host.^{18,38–40} For example, the luminous intensity of the Ln doped sensitizer-rich LiYbF₄ with intense UC luminescence reported by Zou *et al.*³⁸ increases with temperature because the ²F_{5/2} higher levels of Yb³⁺ ions are thermally excited, which is the cooperative ET process of Yb³⁺ ions. Moreover, the Ln doped NaYbF₄ with the same crystal structure as the LiYbF₄ reported by Du *et al.*¹⁸ also exhibited a negative thermal quenching effect and bright visible UC emissions. However, so far there is no research on Ba₄Yb₃F₁₇:Er³⁺ NCs embedded GCs used for optical temperature measurement. And the Ba₄Y₃F₁₇ reported by Grube *et al.*⁴¹ can produce effective luminescence after doping with Ln ions. Therefore, it seems feasible to study the optical thermometry behavior of Ba₄Yb₃F₁₇:Er³⁺ NCs embedded GCs with possible effective luminescence.

Here, the transparent aluminum silicate GCs containing Ba₄Yb₃F₁₇:Er³⁺ NCs were successfully synthesized by a traditional melt-quenching method and a detailed study on their UC emission behaviors has been performed. The optical temperature measurement performance based on the FIR of Er³⁺ ions green emissions was systematically explored at low-power laser 980 nm excitation to reduce the heating effect of Yb³⁺ ions. The results show that the transparent aluminum silicate GCs containing Ba₄Yb₃F₁₇:Er³⁺ NCs are promising candidate materials for optical temperature measurement.

2. Experimental

Er³⁺ doped Ba₄Yb₃F₁₇ GCs were elaborated through melt-quenching technique with the following constituent: 62SiO₂–15Na₂CO₃–7Al₂O₃–3NaF–6BaF₂–(7–x)YbF₃–xErF₃ (x = 0.2, 0.4, 0.8, 1.6 mol%). The raw materials of SiO₂ (AR), Na₂CO₃ (AR), Al₂O₃ (AR), NaF (AR), BaF₂ (AR), and high purity YbF₃ (4 N), ErF₃ (4 N) were completely mixed in an agate mortar and then fused in alumina cauldron at 1500 °C for 30 min. The melts were dumped onto a warmed (300 °C) copper plate and squeezed rapidly by another plate to produce precursor glass (PG). Afterward, PG was annealed at 450 °C for 3 h to release the



internal defect and was cut and polished to a ~ 2 mm thickness. The acquired samples were consequently cut into pieces and heat-treated at 600 °C and 650 °C for 2 h to produce GCs (labeled as GC600 and GC650 respectively).

The measurements of the X-ray diffraction (XRD) are performed on a diffractometer (Bruker D8 advance) with Cu $K\alpha$ radiation over the angular range $10^\circ \leq 2\theta \leq 80^\circ$. The optical transmittance of the samples is measured by an ultraviolet-visible (UV-vis) spectrophotometer (Hitachi U-3700). Microstructural characterization of the resultant GCs was conducted by a transmission electron microscope (TEM, JEM-2010) assembled with the selected area electron diffraction (SAED). The temperature-dependent UC luminescence was measured by an Omni- λ 5028i monochromator equipped with a temperature controller (Lake Shore Model 335). Decay curves were obtained by Tektronix TDS2024 digital storage oscilloscope equipped with a tunable 980 nm pulse laser as the excitation source (Model Opolette 355 LD OPO system).

3. Result and discussion

3.1 Structure and characterization

Fig. 1(a) is the XRD patterns of PG and GCs samples. PG exhibits a feature of the amorphous phase because it has only one hump peak. The diffraction peaks corresponding to the $\text{Ba}_4\text{Yb}_3\text{F}_{17}$ phase (PDF #44-0956) are observed after heat treatment, indicating that NCs are deposited in PG. Fig. 1(b) is the XRD patterns of different ErF_3 concentrations doped GCs. The doping of ErF_3 does not change the crystal phase because the diffraction peaks are almost unchanged.

Fig. 1(c) is the transmittance spectra of PG and GC samples with a ~ 2 mm thickness. In the visible range, the transmittance of PG and GC is higher than 80%. And the transmittance of GC is lower than that of PG, which is caused by the scattering and absorption of incident light by the crystal grains and the agglomeration of the crystal grains in the glass caused by the increase of the crystal grain size. Some absorption peaks at 376, 406, 450, 486, 519, 651 nm are observed, which correspond to the transition of Er^{3+} ion from $^4\text{I}_{15/2}$ ground state to $^4\text{G}_{11/2}$, $^2\text{H}_{9/2}$, $^4\text{F}_{5/2}$, $^4\text{F}_{7/2}$, $^2\text{H}_{11/2}$, $^4\text{F}_{9/2}$ excited states, respectively.

Fig. 2(a) and (b) are TEM images of GC600 and GC650 respectively. For GC600, spherical particles with an average size of 23.0 nm are observed. And the size of NCs increases as the heat treatment temperature increases to 650 °C. Fig. 2(c) is the HRTEM image of GC600. The clear lattice fringe with an interplanar plane distance of 0.34 nm corresponding to the (006) plane of $\text{Ba}_4\text{Yb}_3\text{F}_{17}$ is observed. The SAED pattern of GC600 is shown in Fig. 2(d). The diffraction rings from the inside to the outside corresponds to the plane (122), (214), (410), and (422) of $\text{Ba}_4\text{Yb}_3\text{F}_{17}$ respectively. It is proved that these NCs are $\text{Ba}_4\text{Yb}_3\text{F}_{17}$ phase, which is consistent with the XRD result.

3.2 Upconversion luminescence and excitation mechanism of upconversion

Fig. 3(a) shows the UC spectra of PG and GC600 excited by a 980 nm laser. The characteristic luminescence peaks at 523, 541, and 652 nm are caused by the transitions of Er^{3+} ions from $^2\text{H}_{11/2}$, $^4\text{S}_{3/2}$, and $^4\text{F}_{9/2}$ to $^4\text{I}_{15/2}$, respectively. The luminescence intensity is significantly enhanced after heat treatment, which is caused by the Er^{3+} ion doped into $\text{Ba}_4\text{Yb}_3\text{F}_{17}$ NCs with lower

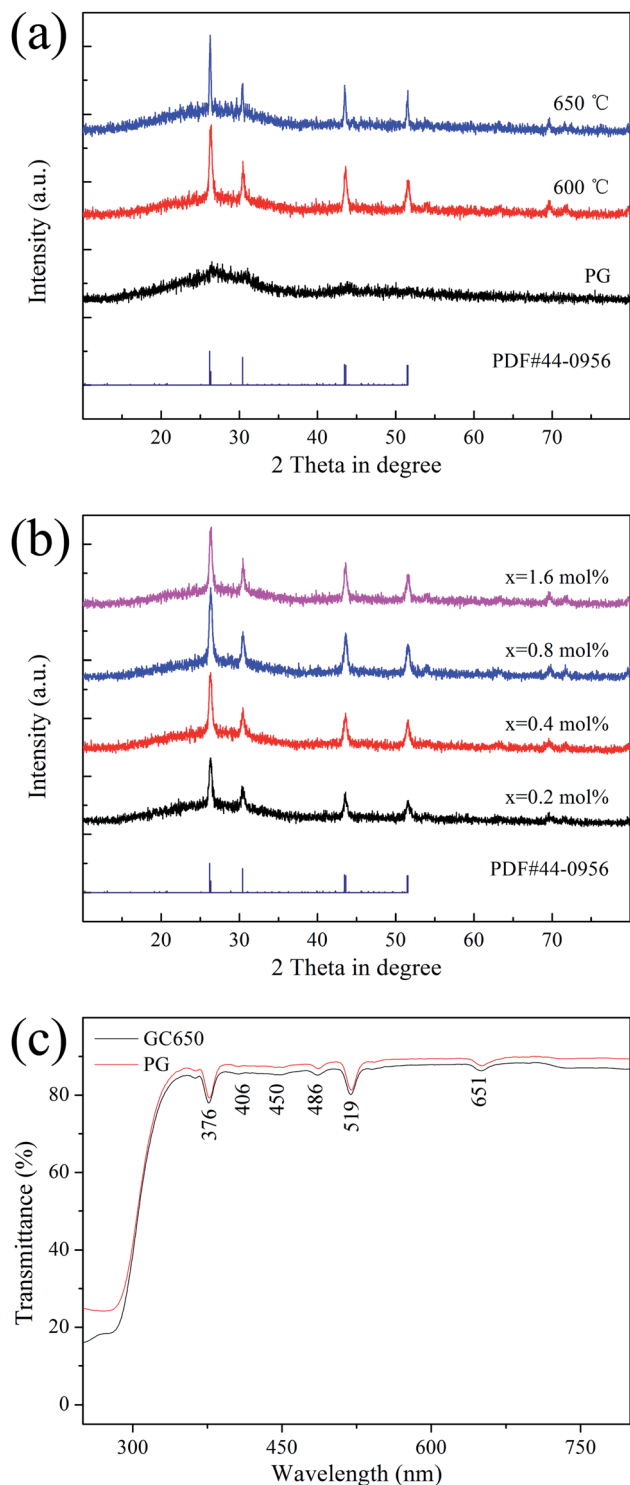


Fig. 1 XRD patterns of (a) PG, GC600, GC650 and (b) $x\text{ErF}_3$ doped GCs ($x = 0.2, 0.4, 0.8, 1.6$ mol%). (c) Transmittance spectra of PG and GC650.



Table 1 Chromaticity coordinates and lifetimes of PG, GC600 and xErF₃ doped GCs (x = 0.2, 0.4, 0.8, 1.6 mol%)

Samples	CIE	Fluorescence lifetime (μs)		
		² H _{11/2}	⁴ S _{3/2}	⁴ F _{9/2}
PG	—	82.85	86.85	214.77
GC600	—	125.07	128.58	376.65
x = 0.2 mol%	(0.47, 0.52)	125.07	128.58	376.65
x = 0.4 mol%	(0.46, 0.53)	113.19	128.56	409.50
x = 0.8 mol%	(0.45, 0.54)	95.17	106.78	357.68
x = 1.6 mol%	(0.43, 0.55)	68.22	79.85	236.08

decreases with the doping of Er³⁺ ions. The promotion of the CR is caused by the absorption of a photon with a wavelength of 980 nm by Er³⁺ ions or the ET from Yb³⁺ to Er³⁺ ions. For the first case, the ⁴I_{15/2} → ⁴I_{11/2} transition of Er³⁺ ions occurs by absorbing a 980 nm photon, followed by the ⁴I_{11/2} → ⁴F_{7/2} transition of Er³⁺ ions by absorbing the second photon. For the second case, the ²F_{7/2} → ²F_{5/2} transition of Yb³⁺ ions occurs through absorbing a 980 nm photon. The ⁴I_{15/2} → ⁴I_{11/2} and ⁴I_{11/2} → ⁴F_{7/2} transition of Er³⁺ ions occur sequentially by absorbing the energy from Yb³⁺ ions. Since the absorption of Er³⁺ ions at 980 nm is weaker than Yb³⁺ ions, the CR is mainly affected by the ET process described in the second case.⁴² In this Er³⁺ doped Yb³⁺ based system, when the Er³⁺ ions concentration increases, the Yb³⁺ ions concentration decreases, and the ET and CR decrease sequentially. Finally, the red (⁴F_{9/2} → ⁴I_{15/2}) emission produced by the CR decreases sharply. The output color changes from greenish-yellow (0.47, 0.52) to yellow-green (0.43, 0.55) with the doping of ErF₃ (Fig. 3(d)) and the

emitting light is obvious to the naked eyes (inset, Fig. 3(d)). The chromaticity coordinates at different doping concentrations are listed in Table 1.

The power-dependent UC emission intensity is studied to interpret the UC mechanism of the ErF₃ doped GCs, as Fig. 3(e) shows. Generally, the emission intensity (*I*) and pump power (*P*) follow the formula $I \propto P^n$, where *n* is the number of photons absorbed by each high-energy photon emitted. The slope of the linear fit is 1.69 for ²H_{11/2} → ⁴I_{15/2} (523 nm), 1.57 for ⁴S_{3/2} → ⁴I_{15/2} (541 nm), and 1.67 for ⁴F_{9/2} → ⁴I_{15/2} (652 nm), respectively. This means that these emissions are two-photon processes. The possible UC mechanism based on ET is shown in Fig. 3(f). By absorbing a 980 nm photon, the ²F_{7/2} → ²F_{5/2} transition of Yb³⁺ ions occurs. The ⁴I_{15/2} → ⁴I_{11/2} and ⁴I_{11/2} → ⁴F_{7/2} transition of Er³⁺ ions occur sequentially by absorbing the energy from Yb³⁺ ions. The transition from ⁴F_{7/2} to ²H_{11/2}/⁴S_{3/2} and ⁴F_{9/2} of Er³⁺ ions occurs sequentially through a nonradiative relaxation process. Meanwhile, the ⁴I_{13/2} → ⁴F_{9/2} transition of Er³⁺ ions occurs through the nonradiative ⁴I_{11/2} → ⁴I_{13/2} relaxation. Finally, the green emission (²H_{11/2} → ⁴I_{15/2}, ⁴S_{3/2} → ⁴I_{15/2}) and red (⁴F_{9/2} → ⁴I_{15/2}) emission of Er³⁺ ions take place.

3.3 Decay behaviors

Fig. 4 is the decay curves of all samples at different emissions. All curves can be well fitted with the following single-exponential function,

$$I(t) = A_1 \exp(-t/\tau_1) + A_2 \exp(-t/\tau_2) \quad (1)$$

where *I*(*t*) is the luminescence intensity at time *t*, *A*₁ and *A*₂ are pre-exponential constants, τ₁ and τ₂ are the fitted fluorescent lifetimes. The average lifetime (τ) is calculated by,

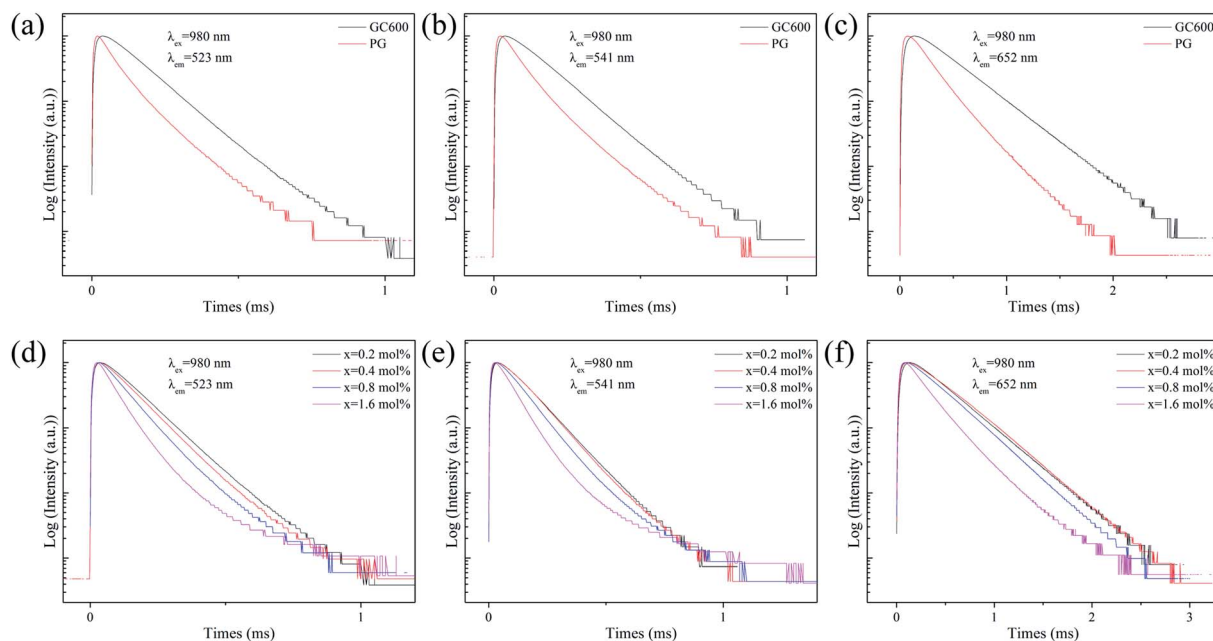


Fig. 4 Fluorescence decay curves of Er³⁺ ion at (a and d) 523 nm, (b and e) 541 nm and (c and f) 652 nm emission in PG, GC600 and xErF₃ doped GCs (x = 0.2, 0.4, 0.8, 1.6 mol%).



$$\tau = \frac{A_1\tau_1^2 + A_2\tau_2^2}{A_1\tau_1 + A_2\tau_2} \quad (2)$$

The fluorescence lifetimes of all samples are summarised in Table 1. For the lifetime of PG and GC600 at different emissions derived from the decay curves shown in Fig. 4(a–c), the lifetime of all emissions in GC600 is longer than that in PG. This phenomenon is caused by the decrease of the nonradiative relaxation rate of Er^{3+} ions in $\text{Ba}_4\text{Yb}_3\text{F}_{17}$ NCs with lower phonon energy.⁷ The increased lifetime also proves that Er^{3+} ions incorporate into the $\text{Ba}_4\text{Yb}_3\text{F}_{17}$ NCs after crystallization. For the lifetime of GCs at different doping concentrations derived from the decay curves shown in Fig. 4(d–f), almost all emission decreases with the doping of ErF_3 , which is caused by the concentration quenching effect.

3.4 Optical thermometry behavior

A detailed study on temperature-dependent luminescence behavior has been performed to evaluate the practical application of the thermometers. Fig. 5(a) is the UC luminescence spectra of ErF_3 doped GC in the range 300–600 K. Since thermal quenching can cause nonradiative relaxation, it is easy to observe the phenomenon that the luminous intensity decreases with temperature. This material can be used for temperature measurement because the population of the $^2\text{H}_{11/2}$ and $^4\text{S}_{3/2}$ of Er^{3+} ions follows the Boltzmann distribution. As shown in Fig. 5(b), the FIR of $^2\text{H}_{11/2}$

$\rightarrow ^4\text{I}_{15/2}$ and $^4\text{S}_{3/2} \rightarrow ^4\text{I}_{15/2}$ transition monotonically increases from 0.23 to 0.92 as the temperature increases from 300 to 600 K. The FIR from the TCEs is expressed by,⁴³

$$\text{FIR} = A \times \exp\left(-\frac{\Delta E}{kT}\right) + B \quad (3)$$

where T is the absolute temperature, k is the Boltzmann constant, ΔE is the energy gap between $^2\text{H}_{11/2}$ and $^4\text{S}_{3/2}$, A and B are constants. After fitting, the FIR is,

$$\text{FIR} = 6.23 \times \exp(-1248/T) + 0.13 \quad (4)$$

The energy gap ΔE obtained is 868 cm^{-1} , which is derived from the well fit of formula (3) to the experimental FIR data. The relative sensitivity (S_R) of optical thermometers is an important parameter to evaluate the optical thermometric performance of luminescent materials. The S_R is expressed by,

$$S_R = \frac{1}{\text{FIR}} \times \frac{d\text{FIR}}{dT} = \frac{\text{FIR} - B}{\text{FIR}} \times \frac{\Delta E}{kT^2} \quad (5)$$

Fig. 5(c) is the corresponding curve of S_R vs. T change. The S_R first increases and then decreases as the temperature increases from 300 to 600 K. At the temperature of 450 K, the S_R of the prepared sample reached its maximal value of $0.20\% \text{ K}^{-1}$. Multiple temperature-recycle experiments show good repeatability and reversibility, as shown in Fig. 5(d). All these results

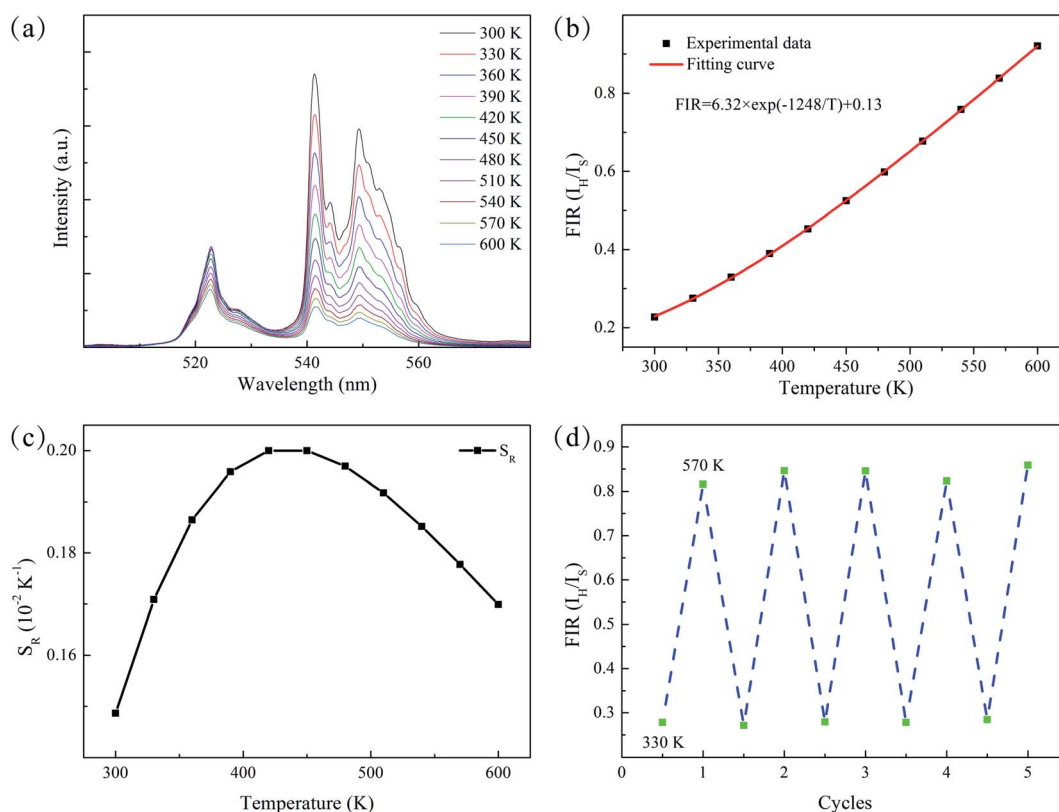


Fig. 5 (a) Temperature-dependent UC luminescence spectra of GC600. (b) A plot of FIR value versus absolute temperature as well as the fitting curves. (c) Relative temperature sensitivity S_R of GC600 at various temperatures. (d) Switching of the FIR value induced by alternating temperature between 330 K and 570 K.



show that the GCs containing Ba₄Yb₃F₁₇:Er³⁺ NCs are candidate materials for temperature sensing.

4. Conclusion

In summary, transparent GCs containing Ba₄Yb₃F₁₇:Er³⁺ NCs were successfully fabricated by a traditional melt-quenching method. Compared with PG, the enhanced emission intensity and lifetime of GC indicate the successful deposition of Ba₄Yb₃F₁₇ NCs in the glass host after heat treatment. The green emissions at 523 and 541 nm from the transitions of ²H_{11/2} and ⁴S_{3/2} TCEs to ⁴I_{15/2} were observed. The relationship between FIR and *T* has been explored to evaluate the optical thermometric performance. At the temperature of 450 K, the *S_R* of the prepared sample reached its maximal value of 0.20% K⁻¹, and Δ*E* is 868 cm⁻¹. This shows that the GCs containing Ba₄Yb₃F₁₇:Er³⁺ NCs are candidate materials for temperature sensing.

Conflicts of interest

There are no conflicts to declare.

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