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Enhanced up-conversion luminescence and optical thermometry characteristics of Er^{3+}/Yb^{3+} co-doped transparent phosphate glass-ceramics

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ABSTRACT

Novel Er^{3+}/Yb^{3+} co-doped transparent glass-ceramics (GCs) containing orthorhombic $NaZnPO_4$ nanocrystals (NCs) were successfully prepared for the first time by a conventional melt-quenching and subsequent heating. Under 980 nm laser prompting, the GC samples produced intense red and green up-conversion emissions. The emission intensities varied with Yb^{3+} concentration and heat treatment conditions. Optimum emission intensities were obtained for the sample with 2 mol% of Yb^{3+} heat treated at 580 °C for 4 h. Furthermore, the temperature dependent fluorescence intensity ratio (FIR) of thermally coupled emitting states ($^4S_{3/2}$, $^2H_{11/2}$) in Er^{3+}/Yb^{3+} co-doped GCs was evaluated under 980 nm. A high relative temperature sensitivity of 1.329% K^{-1} was obtained at 303 K and the maximal absolute temperature sensitivity at 612 K was evaluated to be 5.732 × 10^{-3} K^{-1}. It is expected that the as-fabricated GCs containing $NaZnPO_4$ NCs are an efficient up-conversion material with potential application in optical temperature sensor.

1. Introduction

In recent years, trivalent rare-earth (RE) ions doped up-conversion (UC) GCs have emerged as a fascinating field of research due to their potential application in all-solid compact lasers, thermal imaging, fiber amplifiers, spectral conversion, photo-voltaic solar cells, drug delivery carriers cancer treatment, temperature sensors as well as optical heaters [1–15]. One of the especially interesting applications is as non-contact temperature sensors which are regarded as a promising tool for temperature detection because of their noninvasive operation mode, excellent accuracy, high spatial resolution and fast response [16,17]. UC is a non-linear optical phenomenon which involves the sequential absorption of two or more low energy (NIR) photons to emit a high energy (visible) photon. Nowadays, phosphate based materials have become an efficient luminescent productive hosts in the photoluminescence (PL) emission studies due to its peculiar structure, low phonon energy and excellent doping ability for RE ions [18]. Phosphates based GCs possess excellent physicochemical properties, thermal stability, optical stability, better color rendering index (CRI), and low phonon energy. This leads to produce the luminescent materials with high luminescence efficiency for practical applications. Among the phosphates based materials, the sodium zinc orthophosphate ($NaZnPO_4$) having excellent coordination flexibility and strong Zn-O-Zn linkages within the lattice is of particular interest, because the materials with such properties may improve the PL performance [19–21]. The host materials not only play the important role to improve the PL performance but also the interaction between the RE ions and the host lattice. Erbium ion (Er^{3+}) is a well studied RE ion as dopants in the GCs technology because under NIR excitation it gives efficient visible UC emission. Er^{3+} ion has low absorption for 980 nm corresponding to the $^4I_{15/2} \rightarrow {}^4I_{11/2}$ transition while Yb^{3+} ions show a broad absorption cross section corresponding to the $^2F_{7/2} \rightarrow {}^2F_{5/2}$ transition. Additionally, the energy levels of Yb^{3+} ($^2F_{5/2}$) and Er^{3+} ($^4I_{11/2}$) are almost resonant which enables the possible efficient energy transfer (ET) from the Yb^{3+} to Er^{3+} ions which significantly enhances the UC luminescence efficiency [22–25].

Among many reported RE ions doped materials for temperature sensing based on the change of fluorescence intensity ratio (FIR) of two thermally-coupled energy levels (TCELs) with temperature, Er^{3+} has two sets of TCELs, a pair ($^2H_{11/2}$, $^4S_{3/2}$) with agap of~780 cm^{-1} and the other pair ($^4G_{11/2}$, $^2H_{9/2}$) with a gap of ~1530 cm^{-1} [26]. By making use of both sets of levels, Er^{3+} could be suitable for optical thermometry from very low to very high temperature with excellent sensitivity [3]. The high measurement accuracy and wide measurement temperature range can be achieved with the GCs samples, which indicates the Er^{3+}/Yb^{3+} co-doped transparent GCs containing $NaZnPO_4$ NCs has potential application in optical temperature sensor. To our

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knowledge, there have been no reports on up-conversion luminescence and optical thermometry characteristics of Er^{3+}/Yb^{3+} co-doped transparent $NaZnPO_4$ glass- ceramics.

In the present work, a novel stable and environment friendly Er^{3+}/Yb^{3+} ions co-doped transparent phosphate glass ceramics have been prepared via conventional melt quenching technique and characterized by differential scanning calorimetry (DSC), XRD, TEM, transmittance spectrum, photoluminescence spectra, decay time, and energy level diagram. The effects of Yb^{3+} content and heat treatment temperature in Er^{3+}/Yb^{3+} co-doped $NaZnPO_4$ transparent GCs on enhancing the UC luminescence properties were investigated. Furthermore, the optical temperature sensing study in the Er^{3+}/Yb^{3+} co-doped $NaZnPO_4$ transparent GCs has been carried out based on FIR technique.

2. Experimental

2.1. Sample preparation

The samples were prepared by a melt-quenching method with the specially designed composition (in mol%): $20Na_2O$–$42ZnO$–$38(P_2O_5+B_2O_3)$–xYb_2O_3–$0.05Er_2O_3$ (x = 0, 0.5, 1, 1.5,). The analytical reagents comprising Na_2CO_3, ZnO, $NH_4H_2PO_4$, H_3BO_3, (≧99.5%) and high purity Yb_2O_3 and Er_2O_3 (≧99.99%) (Guo-Yao Co. Ltd, Shanghai, China) were used as starting materials. The chemicals were mixed thoroughly calcined at 600 °C for 60 min to remove vapor and melted in a covered alumina crucible at 1250 °C in air for 100 min in an electric furnace. The melt was poured into a 350 °C pre-heated copper mold to form the precursor glass (denoted as PG). The as-quenched glass was annealed in a muffle furnace at 420 °C for 10 h and then cooled down naturally to room temperature to release thermal stress. The glass samples were cut into 10 × 10 × 1 mm^3 and polished for spectral measurement. Afterwards, the PGs were then heat-treated at 560 °C, 580 °C and 600 °C for 4 h respectively to form GCs via glass crystallization, which were labeled as GC560, GC580 and GC600. Simultaneously, the PG was heat-treated at 580 °C for 2 h, 4 h and 6 h with a heating rate of 5 K/min to form GCs. All samples were polished optically for further characterization.

2.2. Characterization

The thermal properties of the PG were analyzed by differential scanning calorimetry (STA-449-F3-Jupiter, Netzsch). The crystallization phases of GCs were identified via X-ray powder diffractometer (D8-Advance, BRUKE) with CuKα radiation at room temperature. The microstructure of GCs was studied using a transmission electron microscopy(TEM, JEM-2010). The optical transmittance spectra were recorded on METASH UV–vis–NIR spectrophotometer (UV-6000) in the wavelength range from 350 to 1100 nm. The decay curve measurements were recorded on an Edinburgh Instruments FS5 spectrofluorometer. The photoluminescence spectra were recorded on a FuoroSENS9000A Fluorescence spectrometer with a 980 laser excitation with a very low power of 156 mW to avoid the possible laser induced heating. The temperature dependent emission spectra were recorded on an Edinburgh Instruments FS5 spectrofluorometer equipped with a homemade temperature controlling stage.

3. Results and discussion

3.1. Structure and morphology

The representative DSC curve of PG sample is shown in Fig. 1. It is observed that the glass transition temperature (T_g), the initial crystallization temperature (T_x) corresponding to the onset of crystallization and the temperature of crystallization peak (T_p) are 460 °C, 560 °C and 629 °C, respectively. In order to obtain transparent GCs, it is suitable for selecting heat treatment temperature between 560 °C and 629 °C.

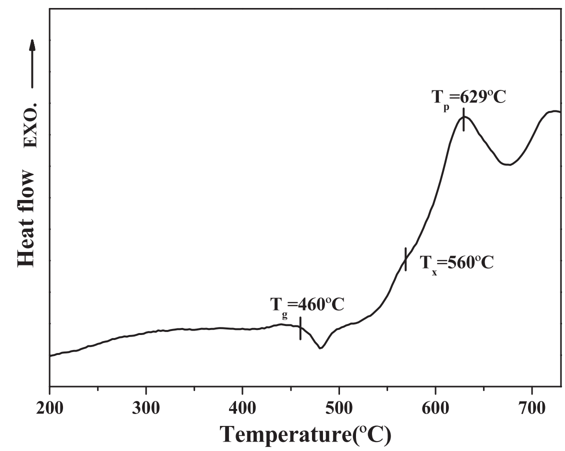

Fig. 1. DSC curve of Yb^{3+}/Er^{3+} (2.0/0.1 mol%) co-doped glass at a heating rate of 10 K/min.

To investigate the structure and the purity of PG and GCs, these samples were characterized by XRD measurements and the results are shown in Fig. 2(a)-(b). There are a broad diffuse humps without any sharp peaks in the XRD pattern of PG sample, indicating its amorphous structure. However, after heat treatment, some intense diffraction peaks are found to superimpose on the broad diffuse humps. All these diffraction peaks can be perfectly indexed to orthorhombic $NaZnPO_4$ phase (JCPDS No. 49-1185) and no any other diffraction peaks were found, indicating that pure GCs containing $NaZnPO_4$ have been successfully fabricated. It is observed that raising temperature or holding time indeed enhance the formation of $NaZnPO_4$ phase. The average crystallite size D of GC580 is estimated to be 21 nm according to the Scherrer equation [27].

$$D = \frac{k\lambda}{\beta \cos\theta} \tag{1}$$

where k = 0.89, λ (0.154056 nm) represents the wavelength of CuKa radiation, θ is the Bragg angle of the XRD peak and β represents the corrected half width of diffraction peak.

Transmittance, a vital parameter of transparent GCs, was recorded between 350 and 1100 nm at room temperature. As shown in Fig. 3, the transmittance obviously deceases with the increment in temperature or holding time. The GC580 remains relative high transparent with a transmittance of approximately 73–76% in the wavelength range of 500–600 nm, indicating that the size of the $NaZnPO_4$ NCs is much smaller than the wavelength of visible light and that the $NaZnPO_4$ NCs are distributed uniformly in the glass matrix. A battery of obvious absorption peaks located at 377 nm, 487 nm, 522 nm, and 658 nm, corresponding to the transitions of Er^{3+} from the ground state $^4I_{15/2}$ to $^4G_{11/2}$, $^4F_{7/2}$, $^2H_{11/2}$ and $^4F_{9/2}$ respectively, were observed, and the broad absorption band at around 975 nm can be mainly ascribed to the transition from $^2F_{7/2}$ to the $^2F_{5/2}$ multiplet state of Yb^{3+} ions with a wide absorption cross section around 980 nm, apart from weak contributions from the $^4I_{15/2}\rightarrow{}^4I_{11/2}$ transition of Er^{3+} [28]. Hence, the 980 nm can be chosen as the excitation source. It is found that the transmittance of all the samples tends to decrease at short wavelengths, which is predominately due to the degree of crystallization increasing. This phenomenon could be explained by Henry theory, in which the intensity of scattered light in GCs follows a $\lambda^{-8}R^7$ relationship, where λ is the wavelength of light and R is the average radius of NCs in GCs [29]. Therefore, it can be inferred that the transmittance of GCs with larger crystal size decreases more promptly with wave length. Typically, the average crystallite size of $NaZnPO_4$NCs is estimated to be about 21 nm for GC580.

Fig. 3(c) and (d) depicts TEM and HRTEM images of GC580 to provide further details of the microstructure morphology of the

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