



Energy transfer and enhanced near-infrared emission in Er³⁺ ions doped composite containing In₂O₃ QDs



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ABSTRACT

Er³⁺ ions doped transparent composite containing In₂O₃ QDs was successfully prepared by a melt-quenching route. Intense Er³⁺-related near-infrared (NIR) emission around 1534 nm, important for optic telecommunication systems, was observed in the obtained composite. Impressively, the Er³⁺-related NIR emission was enhanced by 9 times with respect to the one without In₂O₃ content by optimal QDs size. Quantitative studies of steady-state and transient emission spectral data demonstrated that In₂O₃ QDs acted as sensitizers for the Er³⁺ emission. Furthermore, the influence of QDs size and Er³⁺ concentration on the sensitization efficiency was investigated in detailed. These results indicated that the obtained Er³⁺ ions doped composite containing In₂O₃ QDs could be a promising material for high-performance fiber amplifiers using broadband UV pumping.

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1. Introduction

Er³⁺ doped silica materials have attracted great interest because of their potential applications in Si-based optoelectronic devices [1–4]. Indeed, the ⁴I_{13/2} → ⁴I_{15/2} transition of Er³⁺ ions exhibits an emission peak around 1.54 μm, which corresponds to the absorption minimum of silica-based optical fibers. However, due to the forbidden intra-4f transition, the excitation cross-section of Er³⁺ ions is as low as 10⁻²¹ cm², resulting in low emission efficiency [5]. In recent decades, it is reported that Si nanocrystals may act as sensitizers for Er³⁺ ions, boosting the enhancement of the Er³⁺-related NIR emission [5,6]. Unfortunately, the reversed energy transfer (RET) from Er³⁺ ions to Si nanocrystals hampers further the improvement of the Er³⁺ emission efficiency because of the narrow band gap of Si nanocrystals [7,8].

In order to solve this problem, wide band gap semiconductor, such as In₂O₃ [1], SnO₂ [6], and ZnO [9,10], have been utilized to replace Si nanocrystals to avoid the detrimental RET. In this work, In₂O₃ QDs were selected as the sensitizer because the favourable spectral overlap between In₂O₃ QDs defect-related emission and Er³⁺ ions characteristic absorption should contribute to achieve efficient energy transfer [1]. NIR photoluminescence (PL) of Er³⁺

doped In₂O₃ QDs has been occasionally reported before [11,12]. However, as far as we known, Er³⁺ doped composite containing wide band gap semiconductor nanocrystals were prepared mainly by a sol-gel method [1,6,9–11]. Nevertheless, residual OH⁻ groups in sol-gel composite significantly quench the NIR Er³⁺ emission [13,14]. In addition, the mechanical performance of the sol-gel monolithic material is unable to meet the requirements of practical applications. Herein, efficient NIR emission from Er³⁺ ions doped composite remains a challenging target for their utilization in optoelectronics.

In this work, Er³⁺ ions doped composite containing In₂O₃ QDs was successfully fabricated by a melt-quenching route. The composite may display not only large excitation cross section from the efficient sensitization of In₂O₃ QDs, but also high mechanical stability and scarce OH⁻ groups of melt-quenched oxide glass. Based on the steady-state and transient PL spectra, the influence of In₂O₃ QDs size and the Er³⁺ concentration on the enhanced NIR Er³⁺ emission was discussed in detailed.

2. Experimental

Precursor glass with compositions (mol%) of 18Na₂O-10CaO-12Al₂O₃-(60-x-y)SiO₂-xIn₂O₃-yEr₂O₃ (x = 0 and 3; y = 0.1, 0.5, 1, and 2) was fabricated by a conventional melt-quenching route. About 15 g of a mixture of reagent grade chemicals were melted in a covered alumina crucible at 1520 °C for 30 min. Then the melt was

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poured into a 300 °C preheated copper mold to cool down to room temperature. The precursor glass was cut into the size of $5 \times 5 \text{ mm}^2$ square coupons. Finally, the precursor glass was annealed at 660 °C for different times (0, 4, 8, and 12 h; denoted as NC0, NC4, NC8 and NC12, respectively) to form transparent bulk composite.

The X-ray diffraction (XRD) data were measured by a powder diffractometer (D8 Advance) using $\text{Cu K}\alpha_1$ radiation ($\lambda = 0.1546 \text{ nm}$). The microstructure of the sample was carried out with a transmission electron microscope (TEM, JEM-2010). The excitation and emission spectra as well as the PL decay curves were measured by using an Edinburgh Instruments FLS920 spectrofluorimeter equipped with a 450 W Xe lamp. For the temperature-dependent PL measurement, the sample was placed in a closed-cycle helium cryostat equipped with four optical windows.

3. Results and discussion

The high resolution TEM (HRTEM) images of the precursor glass annealed at 660 °C for different times are shown in Fig. 1. Near spherical nanoparticles can be observed discretely embedded in the amorphous matrix. Insets in Fig. 1a–d exhibit their size distribution in the corresponding sample. The average sizes of In_2O_3 QDs in the NC0, NC4, NC8, and NC12 samples are listed in Table 1. Obviously, the average size of the QDs increases with

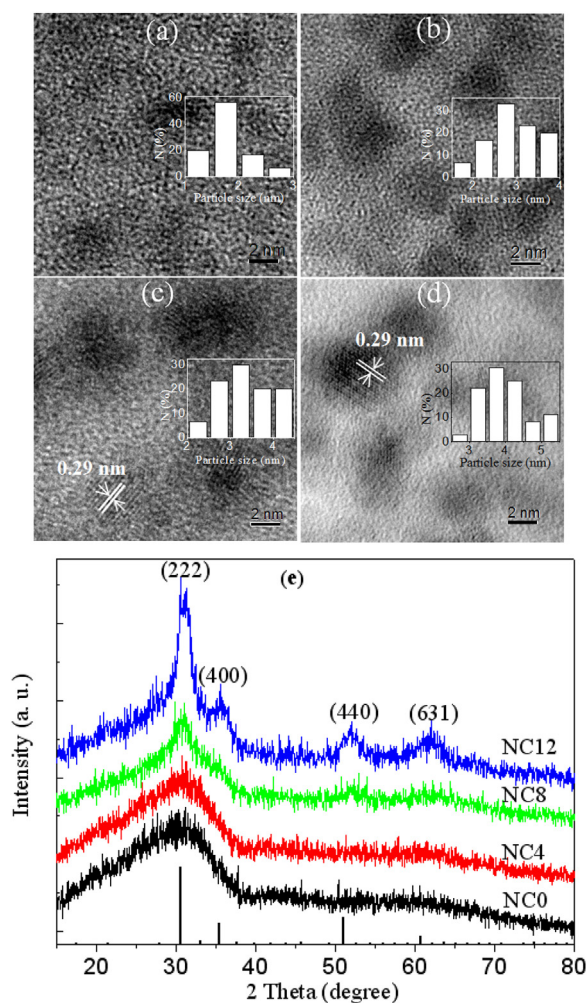


Fig. 1. HRTEM images for the precursor glass annealed at 660 °C for 0 h (a), 4 h (b), 8 h (c) and 12 h (d), respectively. Insets exhibit the size distribution of In_2O_3 QDs in the corresponding sample. (e) XRD patterns of the composite annealed for different times.

Table 1

The average size of In_2O_3 QDs in NC0, NC4, NC8 and NC12 samples; the standard deviations (SD) are also presented.

Sample	Size (nm)	SD (nm)
NC0	1.8	0.4
NC4	2.9	0.6
NC8	3.3	0.7
NC12	4	0.6

annealing time, suggesting that the QDs size can be tunable by precisely controlling the annealing time. As annealing time reaches 8 h, most nanoparticles are larger than the critical size of a nucleus and hence crystallize [15]. The well-distinguished lattice fringes in the HRTEM images (Fig. 1c and d) can be assigned to the (222) plane of cubic In_2O_3 with a d spacing of 0.29 nm. As shown in Fig. 1e, XRD patterns for the composite annealed for 8 or 12 h exhibit several diffraction peaks corresponding to cubic In_2O_3 nanocrystals (JCPDS No. 06-0416). Increasing annealing time results in the narrowing of these peaks, further confirming the growth of the In_2O_3 QDs.

Under 290 nm excitation, a characteristic 1534 nm NIR emission arisen from the $\text{Er}^{3+}4I_{13/2} \rightarrow 4I_{15/2}$ transition is observed in the 0.5 mol% Er^{3+} doped NC0 sample, along with a visible broad emission band centered at 490 nm originated from the carrier recombination between the valence band (VB) and the oxygen vacancies-induced defects states of In_2O_3 [15,16], as shown in Fig. 2a. Moreover, a sharp emission peak at 546 nm due to the $\text{Er}^{3+}2H_{11/2} \rightarrow 4I_{15/2}$ transition is also presented in the visible PL spectrum. To further explore the origin of the Er^{3+} -related and the In_2O_3 -related emissions, the PL excitation spectra of 0.5 mol% Er^{3+} doped NC0 sample were measured and shown in Fig. 2b. The spectrum monitored at 1534 nm consists of a broad band centered at 290 nm and four sharp peaks at 379 nm, 407 nm, 452 nm, and

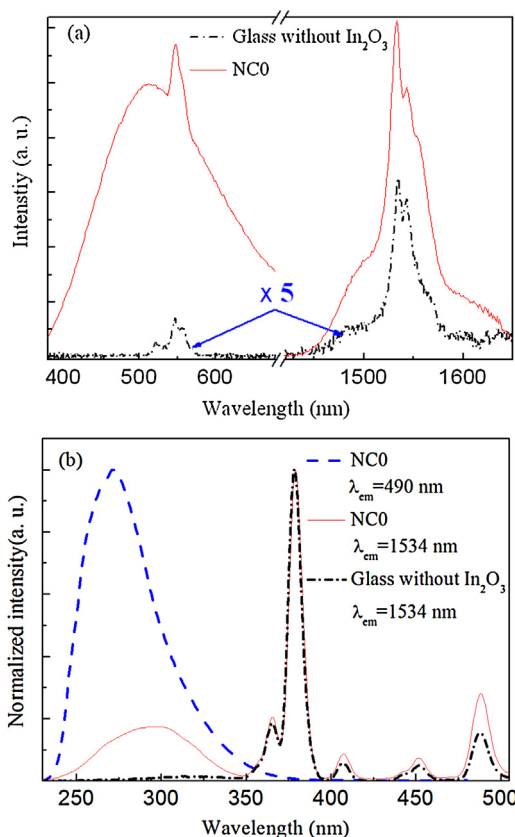


Fig. 2. PL spectra of 0.5 mol% Er^{3+} doped NC0 and glass without In_2O_3 content (a), and the corresponding PL excitation spectra (b).

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