Enhanced Photoluminescence of Tb$^{3+}$ Ions Ce$^{3+}$ Ions and SnO$_2$ Nanocrystals Co-doped Silica Thin Films

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Abstract
Co-doping Ce$^{3+}$ ions and size-tunable SnO$_2$ nanocrystals into Tb$^{3+}$ ions embedded silica thin films produces a ninefold enhancement of Tb$^{3+}$ related emission. Firstly, by optimizing the doping ratio of Sn$^{4+}$ ions and annealing temperature, the size of SnO$_2$ nanoparticles was well tailored for achieving a greatly enhanced photoluminescence emission from Tb$^{3+}$ ions. In order to further increase the emission of Tb$^{3+}$ ions, a proper concentration of Ce$^{3+}$ ions were also added into material. The co-doping situation was researched in this work. The emission of Tb$^{3+}$ reaches the top when the concentration of Sn$^{4+}$ and Ce$^{3+}$ were recorded 15% and 2%. On the other side, companied with the increase of Ce$^{3+}$ ions concentration, the growth of CeO$_2$ nanoparticles were proved seriously attenuated the photoluminescence intensity of Tb$^{3+}$ ions. Therefore, the reducing gas phenomenon for annealing process could be very important. Finally, by analyzing the annealing temperature of co-doped sample, we conclude that a high annealing temperature such as 1000$^\circ$C can enhance the emission as well. These results indicated that an appropriate proportion of Ce$^{3+}$ ions and suitable size of SnO$_2$ nanocrystals can effectively sensitize the luminescence of Tb$^{3+}$ ions.

Keywords Photoluminescence enhancement · Rare earth co-doping · Silica thin film · Sol–gel method

1 Introduction

The design and manufacture of silicon-based light sources, are main challenges in modern optoelectronics (Zhang et al. 2015; Ng et al. 2001; Won 2018). Now researchers are trying to dope different light-emitting centers into a precursor, and then cover the silicon substrate in the form of thin film to obtain different silicon-based light sources. Sol–gel method is one of the most commonly used methods. As a wet chemical technology, the sol–gel method can produce high purity glass at temperatures below the melting point and allows incorporation of much higher concentration of dopants than the melt process (Hall,
et al. 2016; Akpan and Hameed 2010; Rahman and Padavettan 2012). Because of its good physical properties and chemical stability, sol–gel method has been widely used in the research of silicon-based light sources with rare earth ions as dopants, such as Trivalent terbium (Seed Ahmed et al. 2012). Therefore, the silicon-based luminescent film doped with Tb$^{3+}$ made by the sol–gel method has good potential as an effective green light emissive center. Despite Tb$^{3+}$ ions being good green light emissive center, its f-f electronic transitions are parity forbidden, so that direct 4f excitation with conventional light sources normally yields only weak luminescence (Faustino et al. 2018).

Many researchers are trying to improve the emission efficiency of terbium with other rare earth ions through various methods. O.M. Ntwaeaborwa et al. prepared Ce$^{3+}$ ions and Tb$^{3+}$ ions powder phosphors by the sol–gel method. Blue emission from the Ce$^{3+}$ ions was completely suppressed by Tb$^{3+}$ ions co-doping, in the meantime the luminous intensity of Tb$^{3+}$ ions was greatly enhanced (Ntwaeaborwa et al. 2006a). Yagoub M Y A et al. believe that the overlap occurred between emission band of Ce$^{3+}$ and several of the Tb$^{3+}$ 4f-4f excitation bands meets the requirement for the energy transfer between these two RE ions (Ntwaeaborwa et al. 2006a). Faustino B M et al. produced SnO$_2$ nanoparticles doped with a range of lanthanide ions (Ln$^{3+}$) by sol–gel method. It is also found that nanoparticles of SnO$_2$ can effectively sensitize the emission of some RE ions (Faustino et al. 2018). Zhang et al. (2015) proposed that the effective sensitization process can be realized because of the suitable spectral overlap between the defect state energy level of SnO$_2$ nanoparticles and the excitation level of RE ions.

Our experimental results illustrated that the size of SnO$_2$ nanoparticles directly affects the sensitization effect of SnO$_2$ nanoparticles on Tb$^{3+}$ ions (Zhang et al. 2014). The size of SnO$_2$ nanoparticles prepared by sol–gel method is determined by the concentration of Sn$^{4+}$ ions. However, a too large or too small SnO$_2$ nanoparticles will suppress the sensitization effect on the emission of Tb$^{3+}$ ions, thus the density of SnO$_2$ nanoparticles in silicon thin films should be carefully selected after the size of SnO$_2$ nanoparticles is determined. To further improve the luminous efficiency of Tb$^{3+}$ ions, we introduced another kind of RE ions Ce$^{3+}$, a good sensitizer for Tb$^{3+}$ into thin film with an appropriate doping concentration of Sn$^{4+}$ ions. Tb$^{3+}$ ions are simultaneously sensitized by Ce$^{3+}$ ions and nanoparticles of SnO$_2$. Spectral analysis shows that the co-sensitization effect of Ce$^{3+}$ ions and nanoparticles of SnO$_2$ is much better than that of Ce$^{3+}$ ions or nanoparticles of SnO$_2$ sensitized Tb$^{3+}$ ions alone.

### 2 Experimental

Tb$^{3+}$ ions, Ce$^{3+}$ ions and SnO$_2$ nanoparticles co-doped silica thin films were prepared by using the sol–gel method and spin-coating technique. Tetraethyl orthosilicate (TEOS,98%), de-ionized water and ethanol were mixed (6:1:6 in volume) as silica sol. Hydrochloric acid was then added to the mixture dropwise under rigorous stirring to adjust pH value to 2.0. Different amounts of terbium nitrate (Tb(NO$_3$)$_3$·6H$_2$O,99.9%) salt, cerium nitrate (Ce(NO$_3$)$_3$·6H$_2$O,99.95%) salts and tin tetrachloride (SnCl$_4$,98%) were dissolved in the solution to obtain desired concentration. Then the precursor was heated to 65 °C to complete hydrolysis and gelation was performed by leaving the sealed container for one day. The as-prepared gel was spin-coated onto Si substrates to form SiO$_2$ films followed by the post-annealing at 1000 °C for one hours to eliminate hydroxyl groups (-OH), which may
lead to the quenching of rare-earth-related emission as nonradiative carrier-recombination centers.

The excitation spectra, emission spectra and luminescence decay curves of the as-prepared Tb$^{3+}$ ions and Ce$^{3+}$ ions co-doped silica thin films were measured using an Edinburgh Instruments FLS980 fluorometer system equipped with a Xenon lamp as an excitation source. The crystal structure and orientation of the films were characterized by X-ray diffraction (XRD, Bruker D8 Advance). The XRD data were collected by using a scanning mode in the 2θ ranging from 10° to 80° with a scanning step size of 0.02°.

3 Results and discussion

In this part, the concentrations we used in legends are all refer to molar mass concentration. Particularly, after 1000°C annealing process, the Sn$^{4+}$ ions is oxidized into SnO$_2$ nanocrystals. A TEM graph of SnO$_2$ nanocrystals is shown in Fig. 1a, the diameter of SnO2 nanocrystals is about 5 nm and the lattice constant is observed about 0.34 nm.

3.1 Sensitization energy-transfer mechanism

We produced two silica thin film samples individually doped with Tb$^{3+}$ and Ce$^{3+}$ ions respectively and tested their PL and PLE spectrum, as shown in Fig. 1b. The dashes line refers to the PLE spectrum of 4% Tb$^{3+}$ doped sample and the solid line refers to the PL spectrum of 2% Ce$^{3+}$ samples. Obviously, there is an overlap area between these two curves, which is marked as shaded part.

Due to the existence of overlap area, the photon emitted from Ce$^{3+}$ 5d-4f states jump is going to be absorbed by f-f transition in Tb$^{3+}$ ions. This resonance energy transfer is believed the main factor to make the Ce$^{3+}$ a good sensitizer for Tb$^{3+}$ ions photoluminescence (Chung et al. 2010; Caldiño et al. 2011).

The sample of silica thin film individually doped with SnO$_2$ nanocrystals was tested and the PL and PLE spectrum are shown in Fig. 2a. The emission wavelength was 395 nm in the excitation spectrum and the excitation wavelength was 265 nm in the emission spectrum. Based on the energy state theory, we drew a band structure of SnO$_2$ nanocrystals.

Fig. 1 a The TEM image of SnO$_2$ nanocrystals after 1000 °C annealing in silica thin film. b Overlap region of the excitation spectrum of Tb$^{3+}$ and the emission spectrum of Ce$^{3+}$ for singly doped silica samples
to illustrate the energy transfer between oxide nanocrystals and rare earth ions, as shown in the Fig. 2b. The 395 nm peak in PL emission of SnO₂ is a consequence of luminescent centers in the band (~3.1 eV) gap originating from lattice defects in nanoparticles such as oxygen vacancies, Sn interstitials and surface defects, i.e. Sn–Cl or Sn–OH species (Fasutino et al. 2018; Lin et al. 2009). As shown in the Fig. 2b, the 5D₄ energy level of Tb³⁺ ions is lower than the defect states of SnO₂ nanocrystals (~3.1 eV), which provides the possibility for the occurrence of energy transfer from SnO₂ nanocrystals to Tb³⁺ ions.

As shown in Fig. 3, the PLE spectrum of Co-doped sample (solid line) has a strong peak near 270 nm, which perfectly matches the SnO₂ excitation spectrum in Fig. 2, and this peak cannot be found in Tb³⁺ doped spectrum (dashed line). We ascribe these peak to the band-to-band transition in size-confined SnO₂ nanocrystals due to the quantum confinement effect. This phenomenon indicates that an absorption happened between SnO₂ states and the photon energy is transferred from SnO₂ nanoparticle to Tb³⁺ ions. Another small peak is observed in Tb³⁺ single doped sample near 255 nm wavelength. This peak is from the 4f⁸-4f⁷5d transition of Tb³⁺ and it is overlapped by broad excitation band of SnO₂ nanocrystals(Guodong et al. 1997).

3.2 Analysis of Tb³⁺ ions Ce³⁺ ions and SnO₂ nanocrystals co-doped

We selected 4% Tb³⁺, 2% Ce³⁺ and 15% SnO₂ nanoparticles as dopants to discuss the influence of different doping strategy. As shown in Fig. 4, under the excitation of 320 nm, the four PL spectra of silica thin films co-doped with different dopants all consist of four stable characterized emission peaks at 488 nm, 542 nm, 586 nm, and 622 nm, corresponding to the typical Tb³⁺ atomic energy level transitions from 5D₄ to ⁷F₆, ⁷F₅, ⁷F₄, and ⁷F₃, respectively. Obviously, doping of other ions has basically no influence on the emission wavelength, which is because these four Tb³⁺ peaks are generated by f-f transitions happened on 4f orbit. As inner shell 4f orbitals are shielded by filled 5s and 5p value orbitals, these four peaks display very stable wavelength and hard to be influenced by the outside world(Seed Ahmed et al. 2012). Therefore, the doping of Ce³⁺ ions and SnO₂ nanocrystals does not change the wavelength of Tb³⁺ ions emission peak. On the contrary, the PL emission peak of Tb³⁺ ions at 542 nm was enhanced due to the sensitization of Ce³⁺ ions and SnO₂ nanocrystals. It can be seen from Fig. 4 that the PL emission intensity of the samples
Co-doped with Tb$^{3+}$ ions, Ce$^{3+}$ ions and SnO$_2$ nanocrystals at 542 nm is nine times higher than that of single doped Tb$^{3+}$ ions. Clearly, the Ce$^{3+}$ ions and SnO$_2$ nanocrystals both can enhance the emission of Tb$^{3+}$, and these two sensitizers can collaborate to further increase
the emission intensity of $\text{Tb}^{3+}$ ions. Also, it can be observed from Figs. 1b and 2b that the PLE peak of $\text{Ce}^{3+}$ and $\text{SnO}_2$ nanoparticles are located on 265 nm and 315 nm, respectively, while the emission peak of these two sensitizers are both on about 380 nm, which perfectly matches the excitation wavelength area of $\text{Tb}^{3+}$ ions. As a result, we believe there are few energy transfer happening between the states of $\text{Ce}^{3+}$ and $\text{SnO}_2$ nanocrystals, the majority part of energy absorbed by two sensitizers were transferred to the states of $\text{Tb}^{3+}$ ions. Despite the collaboration outcome, in order to clarify the ability as sensitizers and their influence on PL spectrum, we choose the concentration of one sensitizer ($\text{Ce}^{3+}$ or $\text{SnO}_2$ nanocrystals) as independent variable, while the other sensitizer has constant concentration as reference.

The PL emission spectra of 4% $\text{Tb}^{3+}$ and 2% $\text{Ce}^{3+}$ doped with $\text{SnO}_2$ at different concentrations are shown in Fig. 5. The graph shows a $\text{Tb}^{3+}$ characteristic peak at 542 nm under the excitation of 320 nm UV light. When increasing $\text{Sn}^{4+}$ concentration to 15%, the 542 nm emission intensity is gradually enhanced and reaches the peak, tripling the intensity of un-sensitized sample. If the $\text{Sn}$ concentration is furtherly increased, the result shows a sharp descent in emission intensity, and possible reason is considered luminescent quenching effect. As described in Ref (Zhang et al. 2014), the size and density of $\text{SnO}_2$ nanocrystals are strongly related to the concentration of $\text{Sn}^{4+}$ and annealing temperature. When the concentration of $\text{Sn}^{4+}$ surpassed 15%, the $\text{SnO}_2$ nanocrystals start to aggregate and impact the density of $\text{SnO}_2$, leading the decrease of emission intensity. Figure 6 shows the XRD patterns of samples in different $\text{SnO}_2$ concentrations after 1000 °C annealing. The wide peak that can be observed in all samples from 15°to 30°refers to amorphous silica, the matrix material. Comparing with the standard data of $\text{SnO}_2$ in JCPDS file 41–1445, the peaks are perfectly match the XRD pattern of $\text{Sn}^{4+}$, which suggest a remarkable amount of $\text{SnO}_2$ was generated during the annealing process. When doping 5% $\text{Sn}^{4+}$, there is only a very weak peak. In the case of 5% $\text{SnO}_2$, the $\text{SnO}_2$ diffract peak is overlapped by the peak.

![Fig. 5 PL spectra of different $\text{Sn}^{4+}$ and 4% $\text{Tb}^{3+}$,2% + $\text{Ce}^{3+}$ co-doped thin film samples](image)
of amorphous silica and too low to detect. As the doping concentration increases, the characteristic peak of SnO₂ becomes more and more obvious. From three XRD results with SnO₂ peaks, the average values of SnO₂ crystallite size can be evaluated by using Scherrer’s formula Eq. 1 (Arif et al. 2018; Venkateswarlu et al. 2010).

\[ D = \frac{k\lambda}{\beta \cos \theta} \] (1)

where \( D \) is the volume weighted crystallite size (nm); \( k \) is the shape factor (\( k = 0.89 \)), \( \lambda \) is the wave length of the X-rays (\( \lambda = 0.154056 \) nm for Cu Kα radiation); \( \theta \) is diffraction angle (°) and \( \beta \) is FWHM. The estimated value of SnO₂ crystal sizes of three samples doped with 10%, 15% and 20% Sn⁴⁺ are 2.4 nm, 3.7 nm, 4.7 nm, respectively.

Therefore, we can optimize the energy transfer efficiency by modulating the size and density of SnO₂ nanocrystals. Higher density of SnO₂ nanocrystals shortens the average distance between SnO₂ nanocrystals and Tb³⁺ ions, leading to a more effective energy transfer. When the concentration of Sn⁴⁺ reaches 20%, the formation size of nanocrystals increases greatly after annealing process, consequently the surface-to-volume ratio of SnO₂ nanocrystals decreases rapidly, and meantime the density of interface defects related to SnO₂ nanocrystals decreases as well, which results in the decline of photoluminescence intensity.

Here we change the independent variable to Ce³⁺ concentration, the PL emission spectra of 4% Tb³⁺ and 15% Sn⁴⁺ doped with Ce³⁺ at different concentrations under 320 nm excitation are shown in Fig. 7. Focus on the Tb³⁺ characteristic peak about 542 nm, the intensity reaches the top when Ce³⁺ ions is 2%, and then decrease gradually with the increase of Ce³⁺ concentration. When the Ce³⁺ concentration reaches 8%, the
According to Ref (Kroon et al. 2012), Ce³⁺ is sensitive to concentration quenching, and only a relatively low concentration of Ce³⁺ is able to work as a good activator for Tb³⁺ photoluminescence.

Figure 8 shows the XRD results of samples in different Ce³⁺ concentrations after 1000 °C annealing. As the concentration of Ce³⁺ increases, 4 peaks at 28°, 33°, 47° and 56° become more obvious. Comparing with the standard data of CeO₂ in JCPDS file 34–0394, the four peaks perfectly match the XRD pattern of CeO₂, which suggest a remarkable amount of CeO₂ particles were generated during the process of annealing. This process is considered can be effectively inhibited by using reducing gas protection during annealing process. In our experiment, the samples were annealed under air phenomenon and we studied the size of CeO₂ particles generated in different concentration of Ce³⁺ samples. Naturally, the Ce³⁺ ions have the electronic configuration of the noble gas Xe plus a single 4f electron that can effectively carry out photoluminescence and energy transfer from closed 5s and 5p shells. On contrary, the Ce⁴⁺ ions with oxidation state are hard to cause luminescent effect or absorb energy because the last outmost electron is captured and become a stable Xe-like configuration (Ntwaeaborwa et al. 2006b). As a result, the mass generation of CeO₂ effectively reduced the emission intensity of Ce³⁺ and consequently hindered the energy transfer from Ce³⁺ to Tb³⁺ ions.

Based on Scherrer’s formula, the size of CeO₂ particles can be calculated. The estimated results illustrate that the CeO₂ crystal sizes of the samples doped with 2%, 4%, 6%, 8% Ce³⁺ are 2.9 nm, 5.7 nm, 6.2 nm, 7.5 nm, respectively, monotonously increased with the rise of Ce³⁺ ions. Large CeO₂ nanoparticles directly destroy the energy transfer system among different particles in the sample. The most direct manifestation is that 8% Ce³⁺ ions are oxidized to CeO₂ particles about 7.5 nm during annealing. At this time, the luminescence intensity of the sample at 542 nm is weaker than that without Ce³⁺.
Fig. 8 The XRD results of samples in different Ce$^{4+}$ concentrations after 1000 °C annealing

Figure 9 shows the PL spectra of thin films with different temperature. With the increase of annealing temperature from 400 °C to 1000 °C, the characteristic emission intensity of Tb$^{3+}$ ions in the co-doping samples increases obviously. We think it is caused by the following two reasons. Firstly, this increased emission intensity is attributed to the elimination
of non-radiative recombination centers, -OH groups. Generally speaking, these -OH groups have relatively high characteristic phonon energy, which are common non-radiative recombination centers, which quench the luminescence of the luminescent center. Fortunately, the existing studies show that with the increase of annealing temperature, the intensity of each characteristic peak for -OH group decreases significantly, which will also decrease (Xiao et al. 2012). In Ref (Zhang et al. 2015), the existence of –OH bonds was confirmed by FTIR method and it gradually disappeared with the increase of annealing temperature. Secondly, the annealing temperature has a direct effect on the formation of SnO₂ nanocrystals. according to the TEM picture given by Ref (Zhang et al. 2015), the average size of SnO₂ nanocrystals increases from 2.5 nm to 5.2 nm when annealing temperature was changed from 800°C to 1000°C. Figure 10 shows the XRD results of samples containing 15% Sn⁴⁺ annealed at different temperatures. When annealed at 400 °C, there is no obvious SnO₂ characteristic peak(JCPDF NO.41–1445). The corresponding characteristic peaks of SnO₂ appear at 600 °C and 800 °C. However, the corresponding SnO₂ nanocrystals with a size of tens of nanometers are not suitable for energy transfer. When the annealing temperature reaches 1000 °C, the diameter of SnO₂ nanocrystals is about 3.7 nm. The energy level of quantum dots overlaps with the intrinsic excitation lines of rare earth (as shown in Fig. 2b), and energy transfer occurs.

4 Conclusions

In summary, amorphous silica thin films co-doped with Tb³⁺, Ce³⁺ ions and size-tunable SnO₂ nanocrystals ions have been fabricated by facile so-gel method and spin-coating technique. The sensitivity of Ce³⁺ to Tb³⁺ is limited by Ce³⁺ concentration. Highly doped Ce³⁺ will produce large-sized CeO₂ when annealing in air atmosphere, and sometimes inhibit the light intensity of Tb³⁺. The sensitivity of SnO₂ to Tb³⁺ is limited by Sn⁴⁺.
doping concentration and annealing temperature. The results show that when annealed at 1000 °C, the sensitization effect of Sn4+ (the diameter of SnO2 is about 3.7 nm) is the best. However, when annealing temperature is reduced, nano-sized SnO2 nanocrystals cannot be effectively generated, which is not conducive to Tb3+ sensitization. Finally, we hope that this can be effectively used in green light signal source for CMOS compatible sensing.

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Author’s contribution Xin Zhang, Feng Guo and Li zhuang performed the experiment, data collection and draft writing. Xin Zhang and Xiaowei Zhang design and developed the research, revised the manuscript, Qinghui Jin performed the data analysis, graph revision. All authors were involved in manuscript writing and revising.

Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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