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Type of the Paper (Article)

SiO_2-SnO_2:Er^{3+} glass-ceramic monoliths

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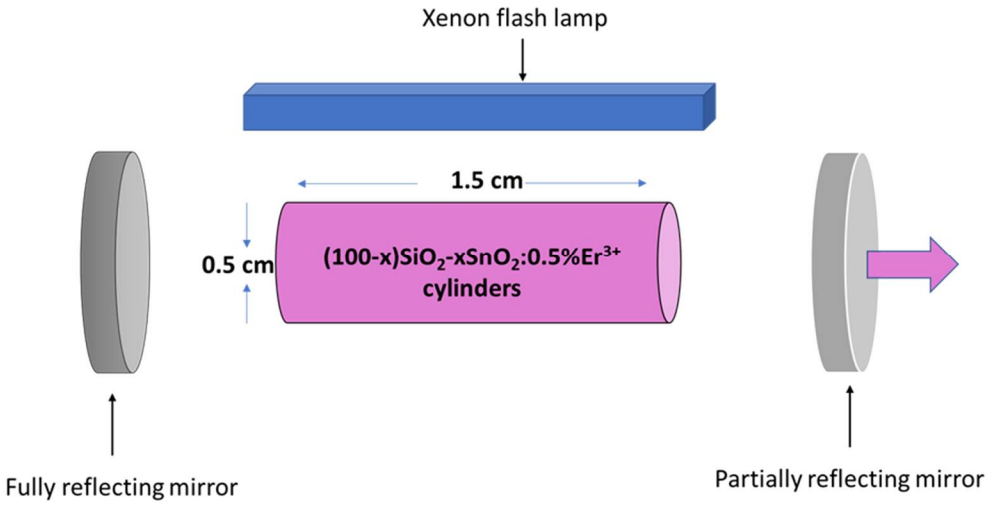
Featured Application: The goal of this work is to demonstrate: 1) a reliable fabrication protocol of monolithic SiO_2-SnO_2:Er^{3+} glass-ceramics; 2) the luminescence efficiency of this system. Based on these fundamental results we are working on developing a proof of concept of a solid state laser with lateral pumping as drawn below.



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Composition (100-x)SiO_2-xSnO_2:0.5%Er^{3+} (x: mol%)	Concentration of Er^{3+} (ions/cm^3)	Density ρ ± 0.1 (g/cm^3)	Refractive index @1542nm-TE (n ± 0.001)	Refractive index @1542nm-TM (n ± 0.001)	Estimated threshold power (W)*	Dimension of the cylinder
x = 5	$1.01*10^{20}$	2.2	1.478	1.480	0.47	diameter: 0.5 cm length: 1.5 cm
x = 10	$1.12*10^{20}$	2.3	1.495	1.497	0.46	

[*]value obtained by resolving the rate equations of Er^{3+} in SnO_2 nanocrystals[1]

Abstract:

The development of efficient luminescent systems, such as microcavities, solid state lasers, integrated optical amplifiers, optical sensors is the main topic in glass photonics. The building blocks of these systems are glass-ceramics activated by rare earth ions because they exhibit specific morphologic, structural and spectroscopic properties. Among various materials that could be used as nanocrystals to be imbedded in silica matrix, tin dioxide presents some interesting peculiarities, e.g. the presence of tin dioxide nanocrystals allows increase in both solubility and emission of rare earth ions. Here, we focus our attention on Er^{3+} - doped silica – tin dioxide photonic glass-ceramics fabricated by sol-gel route. Although the SiO_2-SnO_2:Er^{3+} could be fabricated in different geometrical systems: thin films, monoliths and planar waveguides we herein limit ourselves to the monoliths. The effective role of tin dioxide as luminescence sensitizer for Er^{3+} ions is confirmed by spectroscopic measurements and detailed fabrication protocols are discussed.

Keywords: Transparent glass-ceramics; Luminescence sensitizer; SiO_2-SnO_2; Erbium; Sol-gel; Time-resolved Spectroscopy;

1. Introduction

Looking at the literature of the last years is evident that glass-based rare-earth-activated optical structures represent the technological pillar of a huge of photonic applications covering Health and Biology, Structural Engineering, Environment Monitoring Systems and Quantum Technologies. Among different glass-based systems, a strategic place is assigned to transparent glass-ceramics, nanocomposite materials, which offer specific characteristics of capital importance in photonics[2–4]. These two-phase materials are constituted by nanocrystals or nanoparticles dispersed in a glassy matrix. The respective composition and volume fractions of crystalline and amorphous phase determine the properties of the glass-ceramics. The key to make the spectroscopic properties of the glass-ceramics very attractive for photonic applications is to activate the nanocrystals by luminescent species as rare earth ions[5]. From a spectroscopic point of view the more appealing feature of glass-ceramic systems is that the presence of the crystalline environment for the rare earth ions allows high absorption and emission cross sections, reduction of the non-radiative relaxation thanks to the lower phonon cut-off energy and tailoring of the ion-ion interaction by the control of the rare earth ion partition[6]. Here we focus on glass-ceramic photonic systems based on rare earth activated SiO_2-SnO_2 monoliths produced by sol-gel route. Although the system has been investigated since several years, chemical and physical effects, mainly related to the synthesis and to the ions interactions, which are detrimental for the efficiency of active devices, are subject of several scientific and technological investigations[5–7]. Among the different materials that are successfully used as nanocrystals to be embedded in silica matrix, tin dioxide presents specific interesting characteristics. Rare-earth-activated SnO_2-based bulk glass ceramics have been extensively studied for improving luminescence efficiencies of several rare-earth ions by exciton mediated energy transfer from SnO_2 nanocrystals to the rare-earth ion[7–9]. SnO_2 is a wide-band gap semiconductor (E_g = 3.6 eV at 300 K)

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with a maximum phonon energy of 630 cm^{-1}, exhibiting a broad window of transparency from visible to infrared covering significant emission range of rare earth ions[10].

Here we will present recent results concerning sol-gel fabrication of SiO_2-SnO_2:Er^{3+} glass-ceramic monoliths and their spectroscopic assessment for the development of luminescent systems such as solid state laser and active fibers.

2. Materials and Methods

2.1.Sample preparation: sol-gel derived route

In this work, sol-gel derived route was employed to synthesize the tin dioxide based glass-ceramic monoliths. The monoliths were prepared following five consecutive stages: sol formation, gelation, aging, drying and heat-treatment. Since the final monoliths were obtained based on the phase transformation from gels to glasses, the first four stages played critical roles in assembling the gel skeletal and it in turn defined a specific strategy for the heat-treatment to obtain the glass-ceramics. The synthesis recipe used for sol formation was similar to the one reported elsewhere[11]. Briefly, the syntheses started by dissolving TEOS, $SnCl_2 \cdot 2H_2O$ and $Er(NO_3)_3 \cdot 5H_2O$ in ethanol separately and then the solutions were mixed together. The solution of water and hydrochloric acid was poured drop by drop to the mixture. After that, the mixture was stirred for 1h to form the resulting solution. This solution was transferred into the containers and sealed before being applied to any further treatment.

Table 1. Table of the detailed composition of (100-x)SiO_2-xSnO_2:yEr^{3+} monoliths

SnO_2 content x (mol%)	**Er^{3+} concentration** $y = \frac{n_{Er^{3+}}}{(n_{SiO_2} + n_{SnO_2})}$ **(mol%)**	**H_2O/TEOS**	**EtOH/TEOS**	**HCl/TEOS**
10	0.5	10	4	0.009

However, since our target was to increase the SnO_2 content higher than 5 mol% as in[11], it was necessary to modify the condition of the next stages, i.e. gelation, aging, drying and heat-treatment. This change helped avoiding any phase separation when the content of SnO_2 was increased up to 10 mol%. The schematic synthesis procedure of 90%SiO_2-10%SnO_2:0.5%Er^{3+} monoliths is shown in Figure 1.

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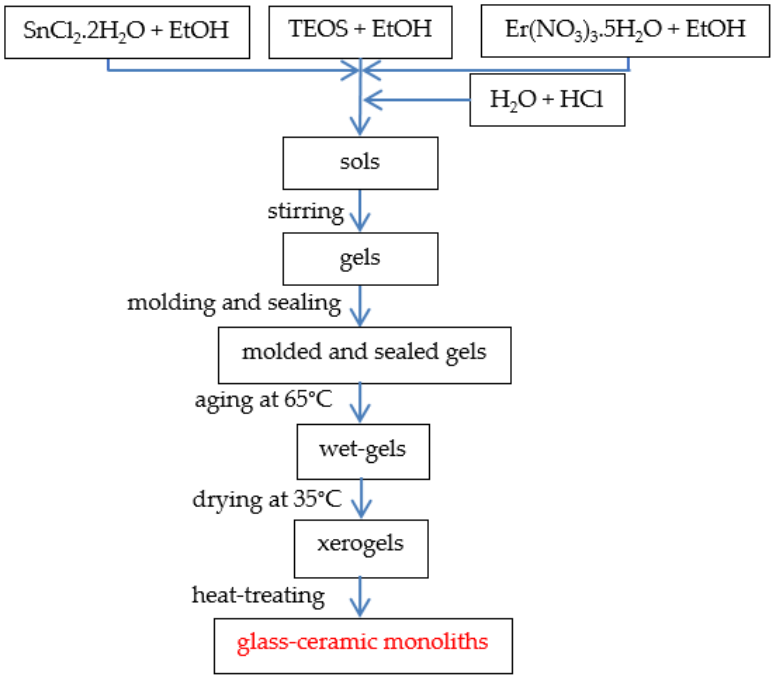

Figure 1. The flow-chart illustrated the synthesis procedure of 90%SiO_2-10%SnO_2:0.5%Er^{3+} monoliths

Figure 2 below shows the photos of two examples of the crack-free and transparent 90%SiO_2-10%SnO_2:0.5%Er^{3+} monolithic square and cylinder after the heat-treatment at 900 °C for 40 h.

Figure 2. Photo of the as-prepared 90%SiO_2-10%SnO_2:0.5%Er^{3+} monolithic square with size of 1×1 cm^2 and thickness of ~ 0.3 cm and the cylinder with diameter of 0.5 cm and length of 1.5 cm obtained after the heat-treatment at 900 °C for 40 h

2.2. Charaterization methods

To check the effective role of tin dioxide as luminescence sensitizer for Er^{3+} ions, the spectroscopic measurements based on different excitation sources were carried out on the 90%SiO_2-10%SnO_2:0.5%Er^{3+} monolith heat-treated at 900 °C for 40h. By the use of Xenon lamp 450 W coupled to monochromator Horiba mod. microHR, the 1500 nm emission spectra excited at different wavelengths and the excitation spectrum were performed. The excitation range was from 300nm to 750 nm with 1 nm scanning step and the spectral resolution of 0.25 nm. The results prove the energy transfer from SnO_2 to Er^{3+} and its effective role in this indirect excitation scheme in comparison with other direct ones. For the lifetime acquisition of the $^4I_{13/2}$-$^4I_{15/2}$ Er^{3+} transition, the 514.5 nm coherent laser beam from the Ar^+ laser Coherent mod. Innova-Sabre TSM 15 was obligatorily employed to perform the time-resolved 1500 nm florescence spectroscopy of the monolith. All the luminescence signal was dispersed by a 320 mm single-grating monochromator with a resolution of 0.5 nm and 2 nm for the emission and excitation spectra respectively, detected using a Hamamatsu photomultiplier tube and standard lock-in technique.

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3. Results

3.1. Emission spectra

Figure 1 shows the photoluminescence spectra of the 90%SiO_2-10%SnO_2:0.5%Er^{3+} monolith acquired at 1500 nm using Xenon lamp as an excitation source. Two different excitation schemes are presented in this figure. One is the indirect excitation, when the sample is excited at 330 nm corresponding to the maximum of the absorption band of SnO_2. The other at 514 nm is the direct excitation of Er^{3+} to its $^2H_{11/2}$ excited state. The Stark splitting shape and enhancement of the $^4I_{13/2}$ - $^4I_{15/2}$ emission of Er^{3+} ions from 330 nm indirect excitation are clearly evidenced. On the contrary, the 514 nm excitation leads to a broad and weaker emission band at 1500 nm.

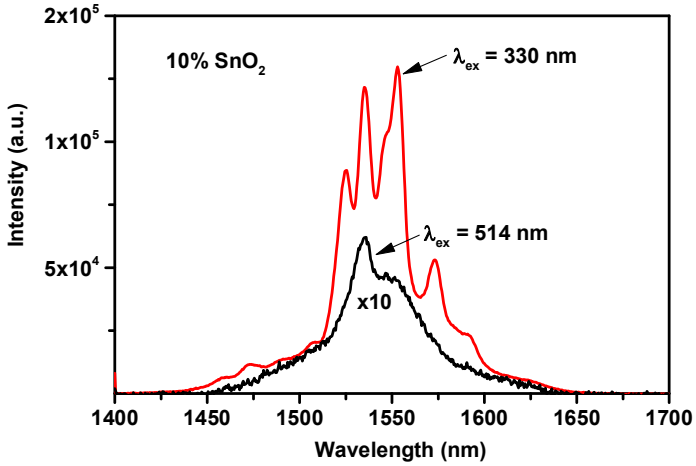

Figure 3. Emission spectra of 90%SiO_2-10%SnO_2:0.5%Er^{3+} monolith heat-treated at 900 °C for 40 h excited at 330nm and 514nm by using Xenon lamp as an excitation source

In Figure 4, the 1500 nm emission characteristics of the direct excitation is more evident under the coherent 514.5 nm laser beam excitation. The spectrum also reveals the Stark splitting, but it is less pronounced in comparison with the emission spectrum obtained upon 330 nm excitation (Figure 3).

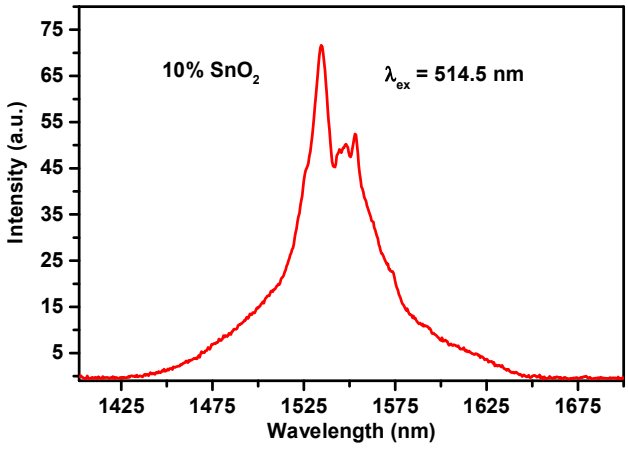

Figure 4. Emission spectra of 90%SiO_2-10%SnO_2:0.5%Er^{3+} monolith heat-treated at 900 °C for 40 h excited at 514.5 nm by using Ar^+ laser as an excitation source

3.2. Excitation spectra

Figure 5 shows the excitation spectra obtained by recording the luminescence signal at 1553.5 nm and 1535.5 nm. Both the observed wavelengths are corresponding to the $^4I_{13/2}$ - $^4I_{15/2}$ emission of Er^{3+}. However, the 1553.5 nm wavelength refers to a maximum peak of the emission spectrum which

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can be found only when Er^{3+} ions are embedded in a crystalline environment. On the other hand, the 1535.5 nm wavelength corresponds to the maximum peak of the emission spectrum of Er^{3+} in a disordered environment.

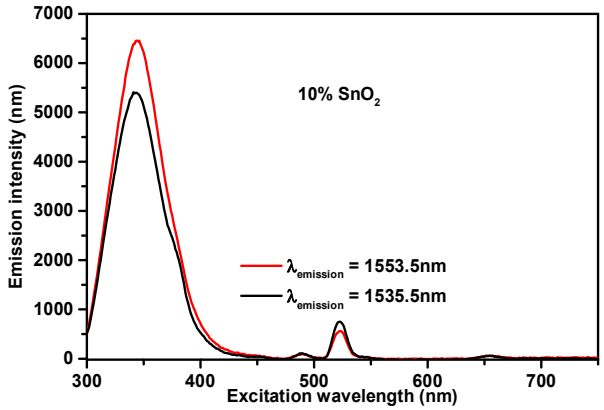

Figure 5. Excitation spectra detected at 1553.5 nm and 1535.5 nm of 90%SiO_2-10%SnO_2:0.5%Er^{3+} monoliths heat-treated at 900 °C for 40 h

3.3. Lifetime

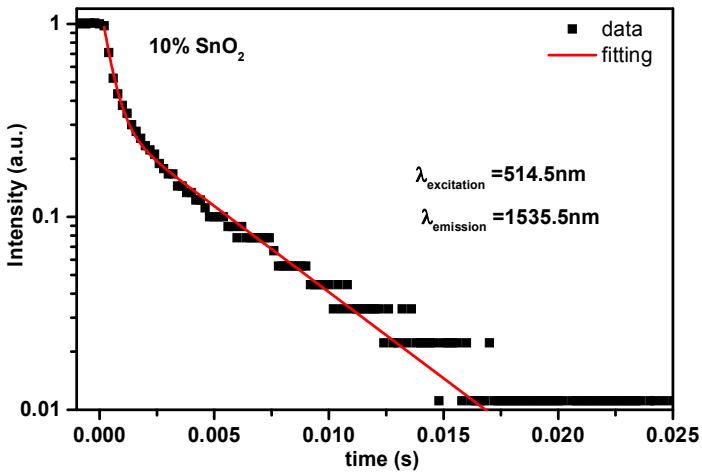

Figure 6. $^4I_{13/2}$-$^4I_{15/2}$ decay curve excited at 514.5 nm of Er^{3+} in 90%SiO_2-10%SnO_2:0.5%Er^{3+} monolith heat-treated at 900 °C for 40 h

Figure 6 shows the decay curve of the $^4I_{13/2}$ luminescence, acquired using the 514.5 nm Ar^+ laser beam. Considering the 1/e decay time, the obtained value is: $\tau_{1/e}$ = 1.2 ms. However, one can clearly see from the figure that the decay function is not single exponential. The fluorescence decay function is described as a sum of two exponentials as in[12]:

$$\phi(t) = A_1 \exp\left[-\frac{t}{\tau_1}\right] + A_2 \exp\left[-\frac{t}{\tau_2}\right] \quad (1)$$

Table 2 summarizes the obtained values of A_1, τ_1, A_2, τ_2. In addition, in this table, the ratio of the numbers N_1 and N_2 of the ions which decay with the lifetime τ_1 and τ_2 respectively are also listed following the approximation of the number of the total ions:

$$N = N_1 + N_2 = A_1\tau_1 + A_2\tau_2 \quad (2)$$

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Table 2. Table of the obtained values of A_1, τ_1, A_2, τ_1, N_1 and N_2

A_1	τ_1 (ms)	A_2	τ_1 (ms)	$\frac{N_1}{N_1+N_2}=\frac{A_1\tau_1}{A_1\tau_1+A_2\tau_2}$
0.32	4.9	1.03	0.5	75 %

4. Discussion

Under the indirect 330 nm excitation which is associated to SnO_2 band-gap, the $^4I_{13/2}$ - $^4I_{15/2}$ emission spectrum exhibits Stark splitting and narrowing peaks (see Figure 3). This aspect reveals two important points: (i) the location of Er^{3+} in the crystalline environment, i.e. SnO_2 nanocrystals; (ii) the energy transfer from SnO_2 to the RE ions. The 1500 nm broad band emission acquired by directly exciting Er^{3+} ions using 514 nm emission of the Xenon lamp discloses the location Er^{3+} in a disordered environment. Although the intensity-based analysis can suffer variations from the experimental factors, e.g. light sources, detectors and refractive indices, the difference in the integrated intensity of Er^{3+} emission band centered at 1500 nm in the case of the two excitation schemes is evident. The emission intensity is higher for 330 nm excitation in respect to 514 nm excitation. The more intense emission of Er^{3+} from the energy transfer process proves the efficient role of SnO_2 as luminescence sensitizer for the rare earth ions.

The emission characteristic of the direct excitation is confirmed by the spectrum shown in Figure 4 when the sample is excited by 514.5 nm laser beam. The large emission band, together with less pronounced Stark splittings are observed. This evidences the presence of Er^{3+} ions in a less ordered environment.

The excitation spectra in Figure 5 clearly show that the dominant contribution to both 1553.5 nm and 1535.5 nm emission is due to energy transfer from the SnO_2 nanocrystals to the imbedded Er^{3+} ions, i.e. the indirect excitation scheme. The weak bands observed at 489 nm, 520 nm and 655 nm are due to direct excitation of Er^{3+} electronic states. These results again confirm that SnO_2 are efficient sensitizers of Er^{3+} luminescence.

To assess some parameters that will be useful for the modelling of a possible laser, the lifetimes and the corresponding fractions of the ions in the $^4I_{13/2}$ metastable state were determined. The decay curve of Figure 6 is similar to the ones already observed in the (100-x)SiO_2-xTiO_2-1Er_2O_3 glass-ceramic system in[13]. The results listed in Table 2 show that about 75 % of the Er^{3+} ions in the $^4I_{13/2}$ state has an exponential decay of about 4 ms. Considering that the lifetime of the metastable state of Er^{3+} in SnO_2 crystals is in the order of 6 ms[14] is reasonable to assume that the majority of the Er^{3+} ions are imbedded in the SnO_2 crystals[15] The short decay component of 0.5 ms can be assigned to the ion-ions interaction energy transfer or Er-OH centers.

5. Conclusions

A viable sol-gel based fabrication protocol for the SiO_2-SnO_2:Er^{3+} glass-ceramic monoliths has been demonstrated. Based on different spectroscopic characterizations, the effective luminescence sensitizer role of SnO_2 for Er^{3+} has been assessed. The emission and excitation spectra show the luminescence effectiveness of the energy transfer from SnO_2 to Er^{3+} in comparison with the direct excitation of Er^{3+} ions. About 75 % of the Er^{3+} ions are imbedded in the SnO_2 nanocrystals.

Finally, SiO_2-SnO_2:Er^{3+} glass-ceramic is surely a fantastic host for rare earth ions and it appears that a pumping schema resonant with the SnO_2 energy gap absorption band could be of some interest in developing solid state laser.

Author Contributions: L.T.N.T., M.F., D.Z., A.L, conceived and designed the experiments; L.T.N.T., D.M, A.C, S.V, C.A performed the experiments; D.M, L.T.N.T., L.Z analyzed the data; L.T.N.T., M.F., D.Z., A.L, D.M, L.Z., G.C.R. wrote and revised the paper.

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Conflicts of Interest: The authors declare no conflicts of interest.

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