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Photonic glass ceramics based on SnO_2 nanocrystals: advances and perspectives

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ABSTRACT

SnO_2-based glass-ceramics activated by rare earth ions have been extensively investigated because of the need to develop reliable fabrication protocols and clarify some interesting optical, structural, and spectroscopic features of the system. There is one important weakness in glass photonics when the rare earth ions are employed as luminescent sources. This is the low absorption cross section of the electronic states of the rare earth ions. A sensitizer is therefore requested. In the last years, we demonstrated that SiO_2-SnO_2 glass ceramics, presenting a strong absorption cross section in the UV range due to the SnO_2 nanocrystal, are effective rare earth ions sensitizers. Another interesting property of the SiO_2-SnO_2 system is its photorefractivity. The high photorefractivity of sol-gel-derived SnO_2-SiO_2 glass-ceramic waveguides has been demonstrated in several papers published by our consortium. It has been shown that the UV irradiation induces refractive index change allowing the direct writing of both channel waveguides and Bragg gratings.

The results presented in this communication not only demonstrate the viability and outstanding properties of the SiO_2-SnO_2 glass-ceramics for photonic applications but also put the basis for the fabrication of solid state and integrated lasers. The next steps of the research are the fabrication of the channels and mirrors exploiting the photorefractivity as well as to draw glass ceramic fiber, checking the lasing action and corresponding functional characteristics. Finally, it is worth noting that the dynamic of the energy transfer from the nanocrystals to the rare earth ions is still an exciting open question.

Keywords: SiO_2-SnO_2, photonic glass-ceramics, rare earth ions, luminescence sensitizers, photorefractivity, sol-gel technology, glass photonics, nanocrystals.

1. INTRODUCTION

In the current Age of Light, Photonics is a key enabling technology[1] for different segments: life science and health, industrial manufacturing and quality, information and communication, emerging light, electronics and displays. And, for the development of optical devices, i.e. the heart elements of photonic applications, glass-based rare-earth-activated optical structures play an essential role[2]. In this area of glass photonics, photonic glass-ceramics have become a strategic choice because they combine the optical and glass-manufacturing processing properties of the amorphous phase and the single-crystal-like optical and spectroscopic properties of the crystalline phases, which is important in luminescent materials activated species, e.g. rare-earths[3-6]. Following this strategy, photonic glass-ceramics based on SnO_2 nanocrystals have drawn much special attention since they exhibit two innovative and unique characteristics: (i) luminescence sensitizing and (ii) photorefractivity. Furthermore, SnO_2 based glass-ceramics gives a solution to two crucial points which are decisive in the development of an optically pumped rare-earth-based laser: (i) the low absorption cross section of the rare-earth ions; (ii) the writing of channels and mirrors in the case of waveguide integrated laser. The role of SnO_2 nanocrystals as rare-earth ion luminescence sensitizers allows to overcome the low absorption cross section of the Er^{3+} ion[7,8]. The photorefractivity of sol-gel derived SnO_2 glass-ceramics allows applying the robust direct laser photoinscription technique on the systems to fabricate Bragg gratings and channel waveguides for waveguide integrated laser[9,10,11]. The results presented in this communication not only demonstrate the viability and outstanding properties of the SiO_2-SnO_2 glass-ceramics for photonic applications but also put the basis for the fabrication of solid state and integrated lasers.

Considering the limitation of low SnO_2 content or the presence of nonstoichiometric SnO_x encountered by several fabrication methods (MCVD, melt-quenching and ion-implantation), sol-gel has been a profitable alternative to produce SiO_2-SnO_2 photonic glass-ceramics with higher SnO_2 content thanks to its low temperature and melt-free synthesis[2,5]. High content of SnO_2 nanocrystals in the sol-gel derived SiO_2-SnO_2 glass-ceramic systems gives advantages not only to obtain effective rare earth luminescence sensitization but also high photorefractivity as indicated in our recent works[8,11,12]. However, to achieve this, the first research demand is to develop reliable fabrication protocols and controlling the ion-ion interaction[7,13]. In this work, we show the viability of sol-gel technology for fabricating transparent

SiO_2-SnO_2 glass-ceramics in different forms of monoliths and planar waveguides containing high SnO_2 nanocrystal contents in silica matrix: 10 mol% for the monoliths and 30 mol% for the planar waveguides. Afterwards, by the spectroscopic properties of the $90SiO_2$-$10SnO_2$:$0.5Er^{3+}$monoliths, we confirm the effective role of SnO_2 nanocrystal as Er^{3+} luminescence sensitizer. Furthermore, our recent results on the photorefractivity of the sol-gel derived SiO_2-SnO_2 glass-ceramics are presented in comparison with other substitutional tin-doped silica glasses showing the viability of the development of not only Bragg gratings and channel waveguides but also active optical integrated devices, e.g. light sources and monolithic optical integrated circuits based on REs-doped SiO_2-SnO_2 glass-ceramics.

2. FABRICATION PROCESSES

2.1 Sol-gel derived fabrication processes for SiO_2-SnO_2 glass-ceramics

In this session, the fabrication of SiO_2-SnO_2:Er^{3+} planar waveguides and monoliths using sol-gel method is described. In general, both sol-gel routes used for the planar waveguides and monoliths fabrication started from the solution syntheses in which all hydrolysis and condensation reactions could occur. Concerning the chemical reactants, tetraethyl orthosilicate (TEOS), tin(II) chloride ($SnCl_2.2H_2O$) and erbium(III) nitrate pentahydrate ($Er(NO_3)_3.5H_2O$) were used as the precursors of SiO_2, SnO_2 and Er^{3+} respectively and HCl was used as a catalyst to promote the reactions.

At the beginning, all these precursors were dissolved in Ethanol (EtOH) and mixed together. Then, a proper amount of de-ionized water containing HCl catalyst was poured into the mixture to proceed the hydrolysis and condensation reaction to obtain the final solution. The quantities of all the chemicals used for the SiO_2-SnO_2:Er^{3+} syntheses were calculated based on the fundamental ratios of TEOS/H_2O/HCl which were 1/10/0.009 for the monoliths[14,15] and 1/2/0.0037 for the planar waveguides[8,11,12].

Afterwards, depending on the structural geometry of each system, the viscosity, reactant ratios and treatment for the solutions were established specifically. In monoliths, to obtain 3D structure xerogel, the synthesized solutions were transferred into molds and left to finish aging, being dried and eventually heat-treated. In planar waveguides, i.e. 2D-structures, for the dip-coating deposition, low viscosity of the solutions for the waveguides was required[11,16] which was much lower than the ones for the monoliths and thus, the aging time was also limited in several hours. The solutions then are used for dip-coating to deposit the planar waveguides. The chemicals and synthesized solutions played principal role in defining texture and consequent properties of the final sol-gel derived products. Therefore, the development of synthesis protocols, with appropriate thermal treatment was the first demand to obtain homogeneous and transparent SiO_2-SnO_2 glass-ceramics.

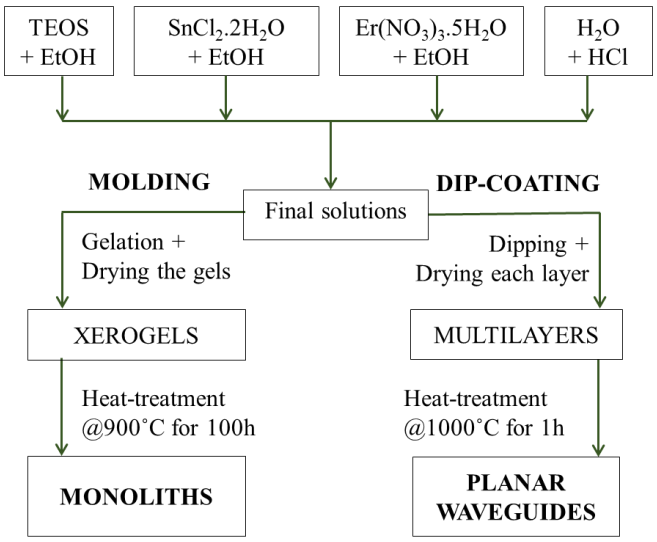

Figure 1. Flow chart of sol-gel derived fabrication processes of SiO_2-SnO_2:Er^{3+} glass-ceramic monoliths and planar waveguides

Concerning the heat-treatment, after the continuously consolidated works[14,15,11,12,8], optimal heat-treatment processes for obtaining optically qualified SiO_2-SnO_2:Er^{3+} glass-ceramic systems were defined. Based on the study on thermal analyses[14,15], a multi-step heat-treatment up to 900ºC for a prolonged annealing time, e.g. 100h[12,11,8] has recently shown the best performance in terms of water removal and densification for the monoliths. Referring to the planar waveguides, the optimal heat-treatment was 1000ºC for 1h[11,12]. Briefly, the fabrication protocols for both SiO_2-SnO_2:Er^{3+} glass ceramic monoliths and planar waveguides are described in Figure 1.

2.2 Obtained SiO_2-SnO_2 glass-ceramics

Sol-gel technology provides versatility of fabricating multicomponent materials with controlled composition, shape, morphological and textual properties of the final products[8,11,12]. One can see that from Table 1 which summarizes all the details concerning the composition (SnO_2 content and Er^{3+} dopant concentration), and geometry of the SiO_2-SnO_2:Er^{3+} glass-ceramic monoliths and planar waveguides obtained by sol-gel fabrication protocols described in Figure 1.

Table 1. Composition and geometrical details of the fabricated transparent SiO_2-SnO_2:Er^{3+} glass-ceramic systems including monoliths and planar waveguides

System	Composition $(100-x)SiO_2$-$xSnO_2$:yEr^{3+}		Geometrical details of fabricated transparent SiO_2-SnO_2 glass-ceramics
	SnO_2 content x (mol%)	Er^{3+} concentration $y = \frac{n_{Er^{3+}}}{n_{SiO_2} + n_{SnO_2}}$ (mol%)	
Monoliths	0, 5, 7.5 and 10	0.25, 0.50, 0.75 and 1.00	• Monolithic squares: 1×1 cm^2 cross section and 0.3 cm height • Cylinders: 0.4 cm diameter and 1.5 cm height • Big pillars: 1 cm diameter and 3.3 cm height
Planar waveguides	0, 5, 10, 15, 20, 25 and 30	0.5, 1.0 and 1.5	Thickness: ~1.2 µm

From Table 1, we have demonstrated that sol-gel is a profitable route for obtaining transparent SiO_2-SnO_2 glass-ceramics containing high SnO_2 nanocrystal contents: up to 10 mol% for the monoliths and 30 mol% for the planar waveguides. After the final heat-treatment, i.e. 900°C for 100h in case of the monoliths and 1000°C for 1h in case of the planar waveguides, all the glass-ceramics are crack-free and densified. Furthermore, these sol-gel protocols allow controlling the size of SnO_2 nanocrystals of less than 10 nm as demonstrated in the structural characterization in our recent publication[12]. All obtained compositional planar waveguides have thickness of around 1.2 µm and with this thickness, the SiO_2-SnO_2 glass-ceramic planar waveguides containing higher than 10 mol% SnO_2 nanocrystals supports single TE and TM mode propagation at 1.5 µm. Different geometries of the monoliths were fabricated with the attention to the oriented applications. The monolithic squares (cross section of 1×1 cm^2 and height of 0.3 cm) were produced for writing channel waveguides.

Figure 2. Photo of the fabricated 95SiO_2-5SnO_2:0.5Er^{3+} big pillar (1 cm diameter and 3.3 cm length) obtained after heat-treatment at 900 °C for 100h[11].

3. SnO_2 NANOCRYSTAL AS AN EFFECTIVE Er^{3+} LUMINESCENCE SENSITIZER

With the strong absorption cross section in the UV, SnO_2 nanocrystals has been demonstrated to be effective sensitizers for the rare-earth luminescence[7,8,11]. The strategy of exploiting SnO_2 nanocrystals as rare earth luminescence sensitizers provides the solution for the low absorption cross section issue of the electronic states of the rare earth ions arising from the parity-forbidden 4f–4f transitions with naturally weak intensity[2,8]. In Figure 3a, the absorption spectrum in the UV-Vis region of the $90SiO_2$-$10SnO_2$:$0.5Er^{3+}$ monoliths showed an optical absorption band edge centered at around 3.5 eV (~ 340 nm) corresponding to the UV absorption of the interband transition of SnO_2 nanocrystals[7,17,18]. Several absorption bands corresponding to the transitions of Er^{3+} from the ground state $^4I_{15/2}$ to other excited states such as $^4G_{11/2}$, $^4F_{5/2}$, $^4F_{7/2}$, $^2H_{11/2}$, $^4S_{2/3}$ and $^4F_{9/2}$ are also assigned. From this figure, one can see that the UV interband absorption of SnO_2 nanocrystals is much stronger than the absorption bands corresponding to the transitions of Er^{3+} from the ground state $^4I_{15/2}$ to the other excited states. Therefore, from Figure 3b, i.e. the excitation spectrum of the $90SiO_2$-$10SnO_2$:$0.5Er^{3+}$ monoliths obtained by recording the luminescence signal at 1530 nm, the fingerprint of the $^4I_{13/2} \rightarrow {}^4I_{15/2}$ transition of Er^{3+}, the more intense 1530 nm emission from the Er^{3+} metastable state $^4I_{13/2}$ is evidently achieved by exciting in the SnO_2 band gap at around 340 nm in comparison with directly exciting Er^{3+} to its electronic states such as $^4F_{5/2}$, $^4F_{7/2}$, $^2H_{11/2}$, $^2F_{9/2}$ and $^4I_{11/2}$. These results demonstrate the role of SnO_2 as an effective Er^{3+} luminescence sensitizer as well as the promising of an efficient laser exploiting this luminescence sensitization as a pumping scheme.

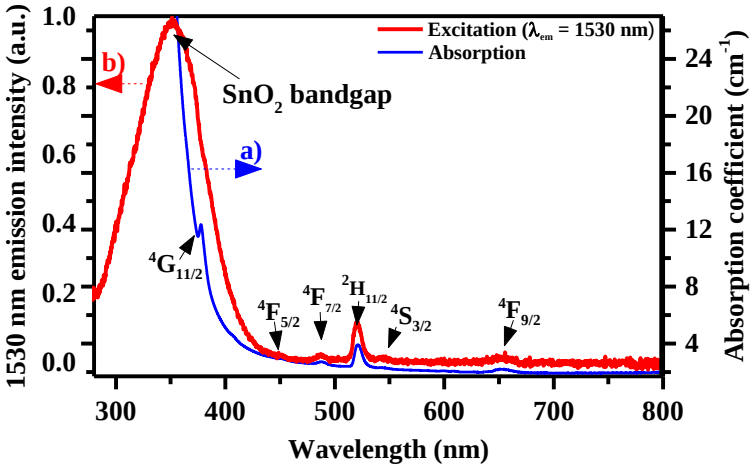

Figure 3. a) Room temperature absorption spectrum (blue line) and b) room temperature excitation spectrum (red line) obtained by detecting at 1530 nm emission of the $90SiO_2$-$10SnO_2$:$0.5Er^{3+}$ glass-ceramic monolith. The SnO_2 nanocrystal bandgap absorption and the electronic transitions from the ground state $^4I_{15/2}$ of the Er^{3+} ion are indicated.

The 1.5 μm emission spectrum of the $90SiO_2$-$10SnO_2$:$0.5Er^{3+}$ glass-ceramic monolith under the excitation at 340 nm in the SnO_2 bandgap shown in Figure 4 which exhibits Stark splitting and narrow peaks confirms (i) the energy transfer from SnO_2 nanocrystals to Er^{3+} ions and (ii) the evidence of Er^{3+} ions locating in the crystalline environment, i.e. SnO_2 nanocrystals[7,11,12,15]. Although the mechanism of energy transfer from SnO_2 to Er^{3+} can be proposed to be mediated by exciton and defects as in the works[13,19,8], the dynamic of the energy transfer from the nanocrystal to the rare earth ion is still an exciting open question which needs to be clarified by detailed sets of experiments.

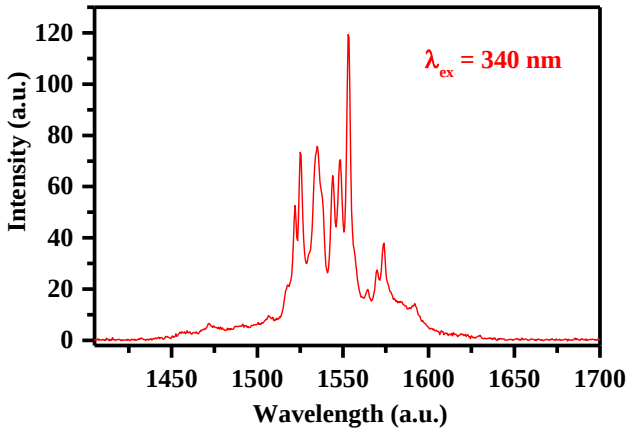

Figure 4. Room temperature $^4I_{13/2} \rightarrow {}^4I_{15/2}$ emission spectrum of Er^{3+} ions of the $90SiO_2$-$10SnO_2$:$0.5Er^{3+}$ glass-ceramic monolith obtained upon excitation at 340 nm in the SnO_2 nanocrystal bandgap.

4. HIGH PHOTOREFRACTIVITY OF SiO_2-SnO_2 GLASS-CERAMICS

The photorefractivity potential of tin dioxide-based transparent materials was first realized in tin-codoped germanosilicate[20,21] and phosphosilicate[21,22] optical fibres. By introducing tin in such silicate glasses, strong photorefractive gratings with enhanced refractive index change up to ~$1.2\text{-}1.4\times10^{-3}$ were obtained. Thanks to Sn presence, the refractive index change was even 3 times larger than in the case of pure germanosilicate[20,21]. Therefore, the photorefractivity of the binary tin dioxide-silica materials has attracted numerous of researches[8,9,11].

In substitutional tin-doped silica glasses, in which a very small content of tin (less than 0.5 mol%[23]) was introduced into silica, the refractive index change is positive and in the order of 10^{-4}. The photorefractivity was demonstrated to relate to laser induced bleaching of the Sn-oxygen deficient centres (ODC) absorption[24], and Sn-$(SiO_4)_n$ rings structural units of which the dimensions were reduced during UV exposure[25-28].

Table 2. Photorefractivity of SiO_2-SnO_2 binary materials

SnO_2 content (mol%)	Fabrication method	Irradiation process		Induced refractive index change (Δn)	The work
		UV laser	Cumulative fluence		
0.15	MCVD	248 nm KrF excimer lasers	20 kJ/cm^2	$\Delta n = +3.0\times10^{-4}$	Brambilla et.al.[24]
0.4	Sol-gel	266 nm pulsed the 4 harmonic Nd-YAG laser	0.17 kJ/cm^2	$\Delta n = +4.0\times10^{-4}$	Chiodini et.al.[26]
5	Sol-gel	266 nm pulsed the 4 harmonic Nd-YAG laser	0.15 kJ/cm^2	$\Delta n = -6.0\times10^{-4}$	Chiodini et.al.[23,26]
25	Sol-gel	248 nm KrF excimer lasers	7.6 kJ/cm^2	$\Delta n = -1.6\times10^{-3}$	Berneschi et.al.[10] Lukowiak et.al.[9]
30	Sol-gel	248 nm KrF excimer lasers	0.3 kJ/cm^2	$\Delta n = -2.8\times10^{-3}$	Tran[11]

If the content of SnO_2 in SiO_2 matrix is high (more than 0.5 mol%), after proper heat-treatments, a segregation of SnO_2 nanocrystals occurred and formed the tin dioxide-silicate glass-ceramics[11,23]. In this case, the photorefractivity of SiO_2-SnO_2 glass-ceramics is demonstrated to be negative[8-11,23]. In the works[8-10], the mechanism was suggested to involve two factors: (i) the volume expansion which is compensated by (ii) the material polarizability. However, due to the complexity and diversity of photorefractive manifestation depending on glass composition, fabrication history and irradiation condition, defining the origin of the photorefractivity of such glass-ceramics needs further experimental investigation to be completely understood. Nevertheless, we have demonstrated that the values of refractive index change of the sol-gel derived tin dioxide-based glass-ceramics are one order of magnitude higher than the ones of tin-doped silica glasses, and they increase with the increasing SnO_2 content in SiO_2 matrix[8,11] as evidenced from Table 2. In other words, the high SnO_2-containing glass-ceramics that have been achieved using sol-gel technology give the advantage of greater UV induced refractive index changes. Moreover, with the high refractive index change in the order of 10^{-3}, the sol-gel derived SiO_2-SnO_2 glass-ceramics are potential for the UV direct writing of channel waveguides and gratings. Therefore, it is compelling for the development of active optical integrated devices, e.g. light sources and monolithic optical integrated circuits based on REs-doped SiO_2-SnO_2 glass-ceramics[8-11].

5. CONCLUSION

In summary, this work shows the consolidated results and recent advances in Er^{3+}-activated SnO_2-SiO_2 transparent glass-ceramics, putting basis for the fabrication of solid state and integrated lasers. Concerning the fabrication, reliable sol-gel synthesis protocols with defined thermal processes have been developed to prepare both the compositional photonic monoliths and planar waveguides. The homogeneous and transparent glass-ceramic monoliths were obtained with SnO_2 content up to 10 mol%. Three different bulk forms were produced for the desired applications: the cylinders for solid state laser construction, the monolithic squares for writing channel waveguides and big pillars for the usages for fiber preform preparation. Referring to the planar waveguides, high SnO_2 content up to 30 mol% was successfully introduced in SiO_2 matrix. The spectroscopic measurements demonstrate: (i) energy transfer from SnO_2 to Er^{3+} and (ii) the role of SnO_2 nanocrystal as an effective Er^{3+} luminescence sensitizer as well as the promising of an efficient laser exploiting this luminescence sensitization as a pumping scheme. The photorefractivity investigation shows that the sol-gel derived SiO_2-SnO_2 glass-ceramics with high negatively induced refractive index change (in the order of 10^{-3}) is viable for the UV direct writing of Bragg gratings and channel waveguides. With the two demonstrated unique characteristics, REs-doped SiO_2-SnO_2 glass-ceramics are compelling candidates for the development of active optical integrated devices, e.g. light sources and monolithic optical integrated circuits.

The consolidated steps of the research are fabricating the channels and mirrors exploiting the obtained photorefractivity with a proper pumping scheme and checking the lasing action and corresponding functional characteristics. Referring to the SiO_2-SnO_2:0.5Er^{3+} monoliths, the current results are reasonably qualified for the checking of lasing action with the designed lateral pumping scheme as a proof of concept. An elaboration for the big pillars preparation for fiber preforms and consequently, drawing the SiO_2-SnO_2:Er^{3+} glass-ceramic fibers is demanded. The dynamic of the energy transfer from the nanocrystals to the rare earth ions deserves further experiments to be completely explored.

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