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## Er<sup>3+</sup>/Yb<sup>3+</sup>-activated silica-hafnia planar waveguides for photonics fabricated by rf-sputtering

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### ABSTRACT

95.8SiO<sub>2</sub>-4.2HfO<sub>2</sub> planar waveguide activated by 0.2 mol% Er and 0.2 mol% Yb was fabricated by multi-target rf-sputtering technique. The optical parameters were measured by an m-line apparatus operating at 543.5, 632.8, 1319 and 1542 nm. The waveguide compositions were investigated by energy dispersive spectroscopy. The waveguide exhibits a single propagation mode at 1.3 and 1.5 μm with an attenuation coefficient of 0.2 dB/cm at 1.5 μm. The emission of <sup>4</sup>I<sub>13/2</sub> → <sup>4</sup>I<sub>15/2</sub> transition of Er<sup>3+</sup> ion, with a 42 nm bandwidth was observed upon TE<sub>0</sub> mode excitation at 980 and 514.5 nm. Photoluminescence excitation spectroscopy was used to obtain information about the effective excitation efficiency of Er<sup>3+</sup> ions by co-doping with Yb<sup>3+</sup> ions. Channel waveguide in rib configuration were fabricated by wet etching process in the active film.

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### 1. Introduction

Er-doped waveguide amplifiers EDWAs are promising candidates for integrated optical (IO) circuits operating in metropolitan area networks [1,2]. Lately, laser action [3] and internal gain at 1540 nm larger than 2.5 dB/cm has been achieved in silica-based waveguides doped with erbium and ytterbium [4]. Silicate-based glasses have a solubility for rare-earth ions of about  $6 \times 10^{20} \text{ cm}^{-3}$ , are transparent in the NIR-visible region and are compatible with IO technology [2]. Various technologies have been

employed for the fabrication of silica-based IO components [5], including ion-exchange [3], sol-gel [1,6], flame hydrolysis [7], chemical vapor deposition [8], pulsed laser deposition [9] and, recently, we have shown that rf-sputtering (RFS) is a suitable technique for fabrication of silica-titania planar waveguides activated by rare-earth ions [10,11]. Hafnium, like Ti, belongs to group 4 in the periodic table. Its oxide is transparent over a wide range of wavelengths and exhibits high refractive index [12] and sol-gel Er-doped SiO<sub>2</sub>-HfO<sub>2</sub> planar waveguides were demonstrated to be a viable system for 1.5 μm applications [6,13–15].

In this work, we present fabrication by RFS technique and spectroscopic assessment of Er<sup>3+</sup>/Yb<sup>3+</sup>-activated SiO<sub>2</sub>-HfO<sub>2</sub> planar waveguide. Moreover, preliminary result concerning the patterning of channel waveguides in these films is presented.

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## 2. Experimental

A  $\text{SiO}_2\text{-HfO}_2\text{:Er}^{3+}/\text{Yb}^{3+}$  waveguide was prepared by multi-target RFS technique. The waveguiding film was deposited on silica substrate. In order to improve the adhesion of the films, the substrate was cleaned inside the deposition chamber by removing some atomic layers just before the deposition procedure. Sputtering deposition of the film was performed by using a 4" silica target on which discs of  $\text{HfO}_2$ , metallic ytterbium and metallic erbium pieces were placed. The residual pressure, before deposition, was about  $2 \times 10^{-7}$  mbar. During the deposition process, the substrates were not heated. The sputtering was carried out with an Ar gas at a pressure of  $7 \times 10^{-3}$  mbar and the applied rf power was 150 W, with a reflected power of 18 W [10,11,15]. The deposition time, necessary to reach the appropriate thickness for one propagating mode at 1.5  $\mu\text{m}$ , was 4 h 15 min. The annealing of the as-deposited film was carried out in air for 6 h at 600 °C to achieve light propagation. The compositional analysis was performed using energy dispersive spectroscopy (EDS), by using a Noran Instruments mod. Voyager apparatus. Scanning electron microscopy (SEM) was used to analyze the morphology of the guiding films. The surface of the films was analyzed by a JEOL-JSM 6300 apparatus at 15 kV by covering the films with a 20 nm gold layer. The thickness of the waveguide and the refractive index at 543.5, 632.8, 1319 and 1542 nm were measured in TE and TM polarizations, by an m-line apparatus based on the prism coupling technique [16]. The losses at 632.8, 1319 and 1542 nm were evaluated by collecting the light intensity scattered out of the waveguide plane for the  $\text{TE}_0$  mode using a fiber scanner [17]. The  $\text{TE}_0$  mode excitation was used for photoluminescence (PL) measurements, detecting the scattered light from the front face of the waveguide. The PL spectroscopy, in the region of the  ${}^4\text{I}_{13/2} \rightarrow {}^4\text{I}_{15/2}$  transition of  $\text{Er}^{3+}$  ions, was performed using the 514.5 nm line of  $\text{Ar}^+$  ion laser and 980.6 nm line of a Ti:Sapphire laser with an excitation power of 380 mW and 100 mW, respectively. The luminescence was dispersed by a 320 mm single-grating monochromator with a resolution of 2 nm. The light was detected using a InGaAs photodiode and a standard lock-in technique. Decay curves were obtained by recording the signal with a digital oscilloscope. The details about the experimental setup were previously reported [10,11,15].

Photoluminescence excitation spectra were measured using a Ti:Sapphire laser tuned over the 860–1010 nm wavelength ranges, spanning the energy region of the  $\text{Yb}^{3+} {}^2\text{F}_{7/2} \rightarrow {}^2\text{F}_{5/2}$  and  $\text{Er}^{3+} {}^4\text{I}_{15/2} \rightarrow {}^4\text{I}_{11/2}$  absorption bands. The excitation beam was incident onto the sample surface under an angle of 30°. The observed wavelength was set to 1533 nm and the signal was recorded with the same apparatus used for the PL spectroscopy.

## 3. Results

The measured optical and spectroscopic parameters of the planar waveguide are reported in Table 1. The molar concentrations of the components in the active film, obtained by EDS are reported in Table 2.

The room temperature PL spectra in the region of the  ${}^4\text{I}_{13/2} \rightarrow {}^4\text{I}_{15/2}$  transition of  $\text{Er}^{3+}$  ions and in the region of the  ${}^2\text{F}_{5/2} \rightarrow {}^2\text{F}_{7/2}$  transition of  $\text{Yb}^{3+}$ , obtained upon excitations at 514.5 nm are shown in Fig. 1.

**Table 1**

Molar concentrations of the components in the  $\text{SiO}_2\text{-HfO}_2\text{:Er}^{3+}/\text{Yb}^{3+}$  waveguide obtained by energy dispersive spectroscopy. The estimated error is 10%.

$\text{SiO}_2/\text{SiO}_2 + \text{HfO}_2$ (mol%)	$\text{HfO}_2/\text{SiO}_2 + \text{HfO}_2$ (mol%)	$\text{ErO}_{1.5}/\text{SiO}_2 + \text{HfO}_2$ (mol%)	$\text{YbO}_{1.5}/\text{SiO}_2 + \text{HfO}_2$ (mol%)
95.8	4.2	0.2	0.2

**Table 2**

Optical and spectroscopic parameters of the  $\text{SiO}_2\text{-HfO}_2\text{:Er}^{3+}/\text{Yb}^{3+}$  planar waveguide.

Thickness ( $\pm 0.05 \mu\text{m}$ )	3.17 $\mu\text{m}$
Number of modes @ 543.5 nm	3
Number of modes @ 632.8 nm	2
Number of modes @ 1319 nm	1
Number of modes @ 1542 nm	1
<i>Refractive index</i>	
@ 543.5 nm ( $\pm 0.001$ )	
(TE)	1.478
(TM)	1.479
@ 632.8 nm ( $\pm 0.001$ )	
(TE)	1.475
(TM)	1.476
@ 1319 nm ( $\pm 0.001$ )	
(TE)	1.465
(TM)	1.465
@ 1542 nm ( $\pm 0.001$ )	
(TE)	1.462
(TM)	1.463
<i>Attenuation coefficient</i>	
@ 632.8 nm ( $\pm 0.1 \text{ dB/cm}$ )	0.7
@ 1319 nm ( $\pm 0.1 \text{ dB/cm}$ )	0.4
@ 1542 nm ( $\pm 0.1 \text{ dB/cm}$ )	0.2
Bandwidth ( $\pm 2 \text{ nm}$ )	42
Lifetime ( $\pm 0.1 \text{ ns}$ )	4.6

Photoluminescence excitation spectrum of the  $\text{SiO}_2\text{-HfO}_2\text{:Er}^{3+}/\text{Yb}^{3+}$  waveguide obtained detecting the emission at 1533 nm is reported in Fig. 2.

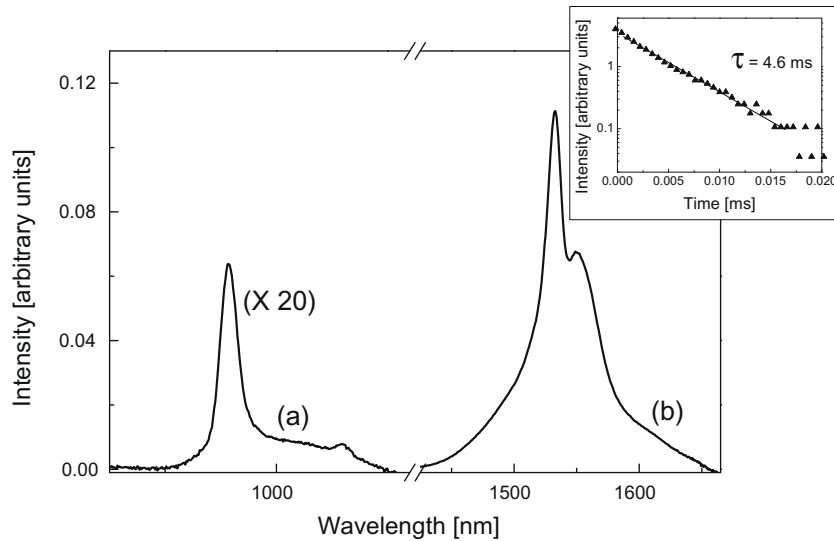
Rib waveguides were prepared starting from the  $\text{SiO}_2\text{-HfO}_2\text{:Er}^{3+}/\text{Yb}^{3+}$  planar waveguide. A thin chromium film, 100 nm thick, was sputtered on the top of the planar waveguide, and then covered by a spun Shipley 1811 film. After exposing and developing the resist and removing the unprotected chromium, the etching of the active film was carried out by a wet chemical process with a buffered HF etch. The rib widths were ranging from 3 to 10  $\mu\text{m}$ , while the etching depth was kept fixed at 2.0  $\mu\text{m}$ . A computer code based on the Wave-Matching Method [18] was used to simulate the behavior of the channels waveguides. A SEM micro-photo of one of the channels etched on the planar waveguide deposited on silica is shown in Fig. 3.

The normalized room temperature PL spectra for the planar and channel waveguides in the region of the  ${}^4\text{I}_{13/2} \rightarrow {}^4\text{I}_{15/2}$  transition of  $\text{Er}^{3+}$  ions, obtained upon excitations at 514.5 nm are shown in Fig. 4. PL measurements was performed using the  $\text{TE}_0$  mode excitation on the planar system (curve a), and injecting the laser light by a 20 $\times$  microscope objective in the channel on the etched system (curve b). The scattered light was recorded from the front surface of the waveguides in both the planar and channel systems.

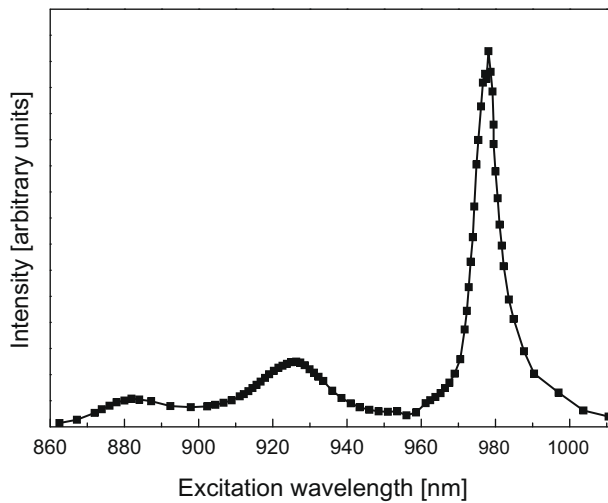
## 4. Discussion

M-line measurements indicate that the sample supports three TE and TM modes at 543.5 nm, two at 632.8 nm and one mode at 1319 and 1542 nm. The refractive indices measured in TE and TM polarizations are equal within the experimental uncertainty, so that film birefringence can be considered negligible. The waveguide exhibits reasonably low loss ( $0.4 \pm 0.1 \text{ dB/cm}$  at 1300 nm and  $0.2 \pm 0.1 \text{ dB/cm}$  at 1542 nm).

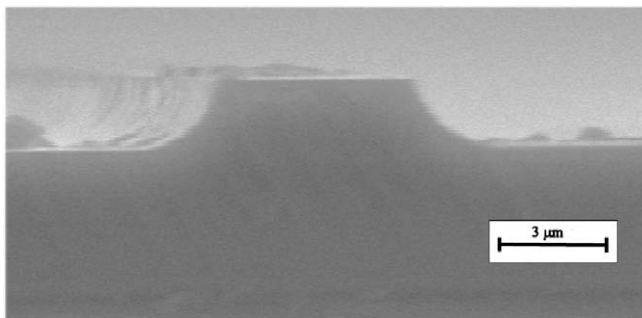
The shape of the emission spectra reported in Fig. 1 in the 1.5  $\mu\text{m}$  region is characteristic of the  ${}^4\text{I}_{13/2} \rightarrow {}^4\text{I}_{15/2}$  transition of  $\text{Er}^{3+}$  ions in silicate glasses [1,6,13–15]. The spectrum exhibits a main emission peak at 1533 nm with a spectral width of about 42 nm, measured at 3 dB from the maximum of the intensity. The bandwidth which is due to inhomogeneous and homogeneous broadening, plus additional Stark splitting of the excited and



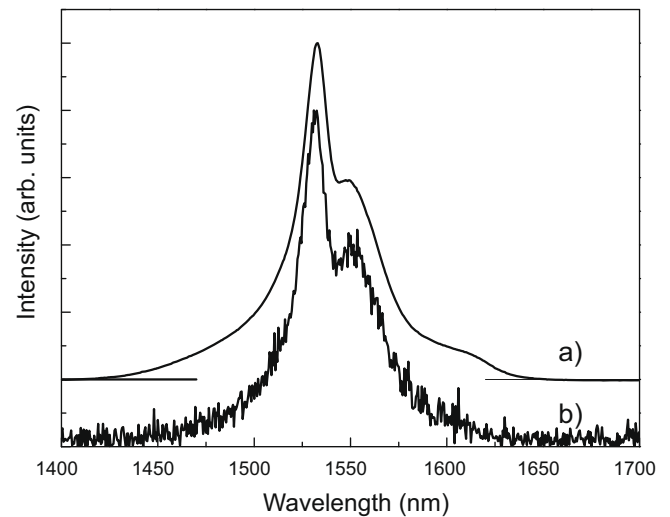
**Fig. 1.** Normalized room temperature photoluminescence spectrum of, (a) the  ${}^4I_{13/2} \rightarrow {}^4I_{15/2}$  transition of  $\text{Er}^{3+}$  ion and, (b) the  ${}^2F_{5/2} \rightarrow {}^2F_{7/2}$  transition of  $\text{Yb}^{3+}$  ion for the  $\text{SiO}_2\text{-HfO}_2\text{:Er}^{3+}/\text{Yb}^{3+}$  planar waveguide, obtained by exciting the  $\text{TE}_0$  mode at 514.5 nm. Inset reports the room temperature luminescence decay curve of the  ${}^4I_{13/2}$  state of  $\text{Er}^{3+}$  ion obtained upon excitation at 514.5 nm with a power of 380 mW.



**Fig. 2.** Photoluminescence excitation spectrum of the  $\text{SiO}_2\text{-HfO}_2\text{:Er}^{3+}/\text{Yb}^{3+}$  waveguide. The detection wavelength was set to 1533 nm.



**Fig. 3.** SEM microphoto of a rib waveguide etched in the  $\text{SiO}_2\text{-HfO}_2\text{:Er}^{3+}/\text{Yb}^{3+}$  planar waveguide.



**Fig. 4.** The normalized room temperature photoluminescence spectra of the  ${}^4I_{13/2} \rightarrow {}^4I_{15/2}$  transition of  $\text{Er}^{3+}$  ion obtained by exciting at 514 nm the waveguide  $\text{SiO}_2\text{-HfO}_2\text{:Er}^{3+}/\text{Yb}^{3+}$  before (a) and after (b) the etching process.

power of 380 mW. The decay curve exhibits a single-exponential behavior with a lifetime of 4.6 ms. The same decay profile was measured upon 980.6 nm excitation. We can compare the values of bandwidth and lifetime obtained in the present work with those reported by Zampedri et al. for  $\text{Er}^{3+}$ -activated silica-hafnia waveguides produced by sol-gel route [14]. Zampedri et al. reported a measured lifetime of about 7.1 ms and a bandwidth of 48 nm for a planar waveguide of composition  $90\text{SiO}_2\text{-}10\text{HfO}_2\text{-}0.3\text{ErO}_3/2$  [14]. The differences both in lifetime and bandwidth can be assigned to  $\text{Er}^{3+}$  local environments, which can be different for different fabrication techniques. In fact Zampedri et al. [14] and Gonçalves et al. [13] demonstrated that  $\text{Er}^{3+}$  spectroscopic properties are strongly affected by the local distortion induced by  $\text{Hf}^{4+}$ . It is possible that the waveguide prepared by RFS exhibits a more distorted local environment for the  $\text{Er}^{3+}$  ion, enhancing the electric dipole character of the  ${}^4I_{13/2} \rightarrow {}^4I_{15/2}$  transition, which results in a faster relaxation rate. Work is in progress to deeper compare the spectroscopic and structural properties of silica-hafnia waveguides prepared by sol-gel and RFS techniques.

ground states [19,20], is enough large for application in wavelength division multiplexed signal amplifiers [21].

The inset of Fig. 1 reports the luminescence decay curve of the  ${}^4I_{13/2}$  state of  $\text{Er}^{3+}$  ion obtained upon excitation at 514.5 nm with a

The spectrum of Fig. 1 also shows an emission band, with a peak at 978 nm and a smaller peak at 1010 nm, typical of the ytterbium ion emissions in glasses [22,23]. This pattern is an indication of the presence of the back energy transfer from  $\text{Er}^{3+}$  to  $\text{Yb}^{3+}$  ions [18]. In fact, using the 514.5 nm laser line we excite directly only the  $\text{Er}^{3+}$  ions but an emission relative to the  ${}^2\text{F}_{5/2} \rightarrow {}^2\text{F}_{7/2}$  transition of  $\text{Yb}^{3+}$  is observed. Nevertheless, the PL excitation spectrum indicates that an effective energy transfer from ytterbium to erbium ions is present. In fact, as reported in Fig. 2, the spectral shape of the excitation spectrum corresponds to the typical ytterbium absorption [23]. Work is in progress to study the kinetics of the energy transfer and back energy transfer processes.

The luminescence measurements performed on the planar and on the channel system indicate that there are no noticeable differences between the shape of the spectra obtained in the planar and channel systems. The lower signal to noise ratio in the spectrum obtained from the channel system in respect to that obtained from the planar system, is only due to the not perfect cleaving of the input facet of the channel.

## 5. Conclusions

$\text{Er}^{3+}/\text{Yb}^{3+}$ -activated silica-hafnia planar waveguides with valuable optical and spectroscopic properties were prepared by multi-target rf-sputtering technique. After thermal annealing in air for 6 h at 600 °C, light propagation occurs in the waveguide and an attenuation coefficient of 0.2 dB/cm was measured at 1542 nm. Luminescence in the third telecom region, with a spectral width of 42 nm and a lifetime of 4.6 ms, was observed upon excitation at 514.5 and 980.6 nm. There was no remarkable change in the spectral width and in the lifetime with the excitation wavelength. Emission assigned to the  ${}^2\text{F}_{5/2} \rightarrow {}^2\text{F}_{7/2}$  transition of  $\text{Yb}^{3+}$  is observed upon excitation at 514.5 nm, indicating the presence of the back energy transfer from  $\text{Er}^{3+}$  to  $\text{Yb}^{3+}$  ions. Nevertheless, photoluminescence excitation spectroscopy indicates that an effective energy transfer from ytterbium to erbium ions is present. Additional work is currently under way to investigate the affect of the  $\text{Er}^{3+}/\text{Yb}^{3+}$  concentration and excitation power on the  $\text{Er}^{3+}$  fluorescence intensity at 1.53  $\mu\text{m}$ . Rib waveguide was also produced by photolithography and wet etching process. Luminescence measurements were performed on the channel waveguide. The spectral shape of the erbium emission at 1.5  $\mu\text{m}$  obtained on the channel system is equal to that obtained on the planar system. The

measurements of signal enhancement when pumping erbium ions at 980 nm are in progress.

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