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Low losses Er^{3+}-doped flexible planar waveguide: Toward an all-glass flexible planar photonic system

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ABSTRACT

Thin films have a crucial role in integrated optics. The development of passive and active devices such as splitters, waveguides, multiplexers and optical amplifiers is by now established on rigid substrates. Therefore, an important further step to improve this kind of technologies and open the route to new applications is to extend these functionalities to mechanically flexible devices. One fundamental brick to obtain this features extension is the fabrication of low loss inorganic active planar waveguides on flexible glass substrate. Here, we present the preliminary results of a novel *top-down* fabrication of an active SiO_2–HfO_2:Er^{3+} all-glass flexible planar waveguide, via radio frequency sputtering. The waveguiding system, deposited on ultrathin flexible glass substrate, showed an attenuation coefficient lower than 0.2 dB/cm at 1.53 μm and exhibits emission in the NIR region.

1. Introduction

While the remarkable advances in electronics have revolutionized the fields of computing and communications, the consequent ever-increasing demands in terms of bandwidth, signal propagation, and switching speeds paved the way for the development of photonics that often exceed the capabilities of electronics. The advantages of the systems based on photonics include, for example, low transmission losses and heat generation, a large information capacity, *i.e.* a broad bandwidth, absence of electromagnetic interference and crosstalk [1]. All these factors have resulted in photonics playing a crucial role in many different fields, from telecommunications to sensing [2–8]. At that point, the next step, to expand even more the spectrum of application of these systems, was the integration of photonic devices of unaltered functionalities on flexible substrates. Since the 2000s, significant progresses have been made in the flexible photonic field, but most of proposed solutions and developed systems relied on polymers, both in terms of substrates [8–10] and actual functional components [1,11–14]. Compared to polymers, inorganic oxide glasses are particularly suitable for photonics application thanks to have some general interesting features such as a wide transparency window (from UV to NIR), a better resistance to radiation and corrosion, better thermal stability and -generally- higher refractive indices [15]. Recently, thanks to the commercialization of ultrathin glass sheets to serve as possible substrates [16–18], the idea of an all-glass flexible photonics has recently taken hold. Ultrathin glasses are expected to perform under a variety of

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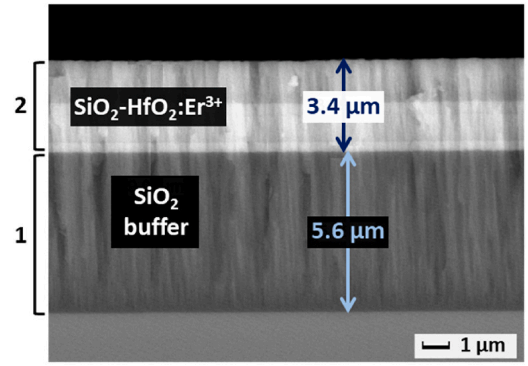

Elemental atomic percentage obtained by EDX analysis

	1	2
O K	66.2	68.3
Si K	33.8	29.2
Hf M	/	1.6
Er M	/	0.9

Fig. 1. Cross-sectional SEM micrograph of the waveguide, SiO_2–HfO_2:Er^{3+} active layer (*bright*) + SiO_2 buffer layer (*dark*), and the elemental atomic percentages of the main elements derived from the EDXS analysis: O (K line), Si (K line), Hf (M line) and Er (M line).

deformation conditions (bending, rolling, twisting, etc.) [19–21], representing potential components for emerging technologies including flexible photonics and integrated optoelectronics.

Hence, the idea to combine these new flexible glasses with an important building blocks in the modern photonic applications, *i.e.* waveguides, results to be very appealing [22]. In particular, waveguides are often employed in integrated optical devices, optical amplifiers, and sensors and in the recent augmented reality technologies [5,6,11,12,21,23–27]. Here, we present the fabrication and characterization of an active SiO_2–HfO_2:Er^{3+} flexible planar waveguides on ultrathin glass (AS 87 eco SCHOTT glass) substrate. This kind of waveguides, already prepared by sol-gel technique (*bottom-up* approach) on rigid conventional substrates [5,28,29] has shown valuable optical, spectroscopic, and structural properties for telecommunications application. Among the different fabrication methods Radio Frequency (RF) sputtering has demonstrated to be suitable for the fabrication of thin-film devices for flexible glass-based systems [8–10]. This method, in fact: *i*) allows the deposition of inorganic oxide layers of high optical-quality, which is essential to reduce losses and *ii*) does not necessarily require thermal-treatments, which may generally deteriorate the properties of the substrates, such as polymers [30] or glasses [31,32], reducing their flexibility. Thus, the fabrication of rare-earth activated optical films has been assured [32].

In this paper an effective *top-down* route is developed to fabricate SiO_2–HfO_2:Er^{3+} planar waveguides with mechanical flexibility and low optical losses via RF-sputtering.

2. Experimental

The Er^{3+}-doped planar waveguide was prepared by multi target non-reactive RF-sputtering technique, using ultrathin AS 87 eco SCHOTT glass (75mm × 25mm × 0.175mm) and silica-on-silicon (SoS) (10mm × 5mm × 1mm) as substrates.

In the case of SoS substrate, the developed fabrication protocol includes the deposition of a silica buffer layer (*ca.* 5.5 µm) between the silicon platform and the active layer SiO_2-HfO_2:Er^{3+} (*ca.* 3.5 µm), in order to have a suitable SoS substrate for integrated optics with maximum confinement of the light [33]. Before deposition, silicon was cleaned using an ultrasonic bath in deionized water, then both the substrates were cleaned with ethanol and finally dried in nitrogen. The residual chamber pressure before the deposition was $4.5 \cdot 10^{-7}$ mbar. During the deposition procedure, the substrates were not heated, and the sample holder temperature was kept at 30 °C. The sputtering was performed in an Ar atmosphere ($5.4 \cdot 10^{-3}$ mbar) and an RF power of 100 W was applied on both the targets (T1 and T2) where SiO_2 plates (15cm × 5cm) were placed. Above one (T2) of the two silica targets were also placed: 1 metallic erbium piece (10mm × 5mm) and 10 disks of HfO_2 (diameter 5 mm). After the deposition of the buffer layer by sputtering exclusively the SiO_2 target T1, the active layer SiO_2-HfO_2:Er^{3+} was prepared by sputtering the T2 target [34,35]. After the deposition, to achieve the correct stoichiometry, the sample was treated in air at 400 °C for 8 h [34]. The chosen heat-treatment temperature was below the glass transition temperature $T_g = 621$ °C of AS 87 eco SCHOTT glass, according to the datasheet [36].

Scanning Electron Microscopy (SEM) analyses were carried out by means of a JEOL JSM-7001F SEM-FEG instrument equipped with an Energy Dispersive X-ray Spectroscopy (EDXS) detector (INCA PentaFETx3, Oxford Instruments). Measurements were performed at 15.0 keV electron beam energy and at 10.0 mm of working distance.

The thickness of the single layers was estimated by processing the cross-sectional SEM micrographs, while the composition was obtained from EDXS analysis. The cross-section analysis was performed fixing the film on a 90° stub and using Backscattering Electrons (BSE) images to better separate the composite material (Si-O-Hf-Er) film, SiO_2 layer and Si substrate (Fig. 1). To have a conductive surface, a layer of about 20 nm of carbon was deposited on the sample.

The m-line spectroscopy was employed to measure the refractive indices and thickness of the planar waveguide at 633, 1319 and 1542 nm in both transverse electric (TE) and magnetic (TM) polarization by using a Metricon mod. 2010 prism coupler instrument, integrated with an optical fibre probe and photodetector for the losses measurements [34,37,38]. By scanning the optical fibre probe and photodetector along the propagating streaks to detect the light intensity scattered from the surface of the waveguide, the propagation losses of the planar waveguide were measured.

Photoluminescence spectroscopy was performed using the 514.5 nm line of a diode laser as excitation source. The planar waveguide was excited by prism coupling technique in the fundamental transverse electric (TE_0) mode. The luminescence was dispersed by a 320 mm single-grating monochromator with a resolution of 1 nm. The light was detected by using a Hamamatsu photomultiplier tube and standard lock-in technique. More details about the experimental setup can be found in Refs. [34,35,39].

3. Results and discussion

The active SiO_2–HfO_2:Er^{3+} flexible planar waveguide was fabricated via RF-sputtering technique on both ultrathin glass (AS 87 eco SCHOTT glass) to obtain a photonic structure with flexible behaviour, similarly to the recent paper [31], and on SoS in order to characterize the structure also from the morphological and compositional point of view. In Fig. 1, the cross-sectional SEM micrograph allows to distinguish the active waveguide (*bright part*) from the SiO_2 buffer layer (*dark part*) and to determine their thicknesses 3.4 µm and 5.6 µm, respectively.

Moreover, the Energy-Dispersive X-ray Spectroscopy (EDXS) allowed to estimate the composition of the sample, confirming the presence of a SiO_2 layer (*dark part*) near the substrate, followed by a composite material (*bright part*), which is composed by O, Si, Hf and Er. Considering

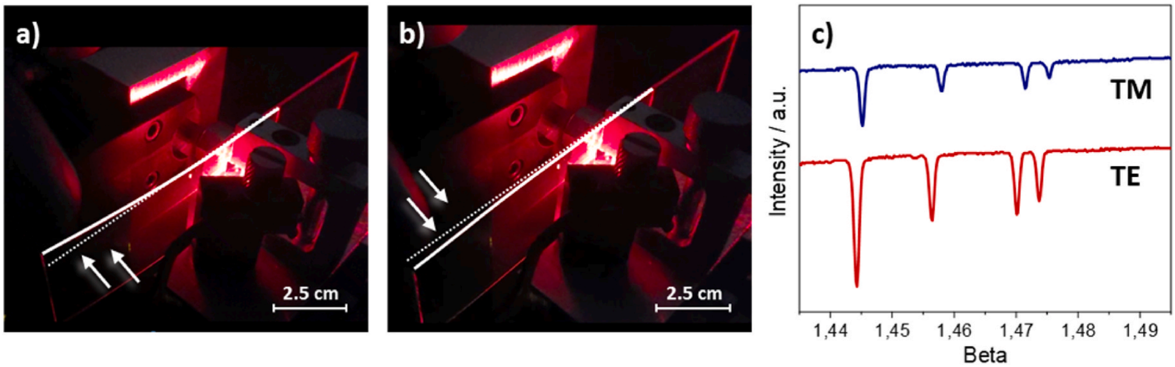

Fig. 2. a) and b) light coupling inside the waveguide in a m-line apparatus based on prism coupling technique, white continuous and dot lines are added as eye guideline to highlight the flexibility of the fabricated system and c) m-line spectra of the beta values obtained in TM (*blue*) and TE (*red*) polarization at 633 nm. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

Table 1
Refractive indexes of the planar waveguide before and after the heat-treatment (400 °C × 8 h) at 633, 1319 and 1542 nm in TE and TM mode and number of modes at the different wavelengths.

	Untreated sample		Heat-treated sample (400 °C × 8 h)		Number of modes
	n(TE)	n(TM)	n(TE)	n(TM)	
633 nm	1.475 ± 0.001	1.477 ± 0.001	1.472 ± 0.001	1.474 ± 0.001	4
1319 nm	1.473 ± 0.001	1.474 ± 0.001	1.470 ± 0.001	1.472 ± 0.001	3
1542 nm	1.470 ± 0.001	1.472 ± 0.001	1.467 ± 0.001	1.470 ± 0.001	2

the values of Si, Hf and Er atomic percentage (Fig. 1), we can estimate the SiO_2, HfO_2 and Er^{3+} molar percentages, which correspond to 92.1%, 5.1% and 2.8%, respectively.

The optical parameters of the planar waveguide at 633, 1319 and 1542 nm were measured in TE and TM polarization by an m-line apparatus based on prism coupling technique [38], as shown in Fig. 2a and b, where it is shown the coupling of the light inside the waveguide under bending conditions.

In Fig. 2c, the effective refractive indexes β obtained at 633 nm in TE and TM mode are reported. Each drop in intensity indicates the coupling of input light into one of the optical modes of the planar waveguide. The obtained refractive index values at different wavelengths before and after the heat-treatment (400 °C × 8 h) are listed in Table 1. The obtained values are in agreement with those expected for the composition corresponding to the relative amount of SiO_2 and HfO_2 in the waveguide active layer observed at EDXS analysis and applying the Lorentz-Lorenz model [27]. The waveguide supports at least 2 TE and 2 TM modes at each measured wavelength, also allowing the determination of its thickness, which resulted to be 3.4 μm in agreement with what has been observed by SEM (3.4 μm). It is possible to note non-significant differences between TE and TM refractive indexes indicating that birefringence is negligible in this kind of system.

SiO_2-HfO_2:Er^{3+} glass-ceramics and amorphous waveguides fabricated by *bottom-up* (sol-gel) technique were reported to have interesting propagation properties at 1.54 μm with an attenuation coefficient around 0.3 dB/cm [5]. Here, we present a significant improvement in terms of propagation properties, obtained for a SiO_2-HfO_2:Er^{3+} planar waveguide fabricated by *top-down* approach (RF-sputtering). The system, in fact, shows a very low attenuation coefficient at 1.54 μm, *i.e.* around 0.2 dB/cm or lower, resulting particularly suitable for integrated optics applications [5,23]. The higher attenuation coefficients observed at low wavelengths (0.7 dB/cm at 633 nm and 1.2 dB/cm at 1319 nm) could be attributed to the surface roughness and the increasing effect of the scattering losses at these wavelengths [5,40], highlighting that further optimizations, in terms of fabrication protocol, are needed in order to reduce as much as possible the propagation losses. Moreover, it is important to note that no variations of the attenuation coefficient are observed after the bending of the samples and the thermal treatment at 400 °C.

Finally, to confirm the fabrication of an active waveguide the photoluminescence (PL) properties of Er^{3+} were characterized. In Fig. 3, PL emission spectra and decay curves of the near infrared (NIR) luminescence of the Er^{3+} ions inside the waveguide are shown, for films before and after the thermal treatment at 400 °C. Direct excitation of Er^{3+} in the green region ($^2H_{11/2} \leftarrow {}^4I_{15/2}$, λ_{ex} = 514.5 nm), was exploited to observe the lanthanide emission in the NIR range. The shape of the $^4I_{13/2} \rightarrow {}^4I_{15/2}$ transition (1.53 μm) in the NIR PL spectra (Fig. 3a) is almost

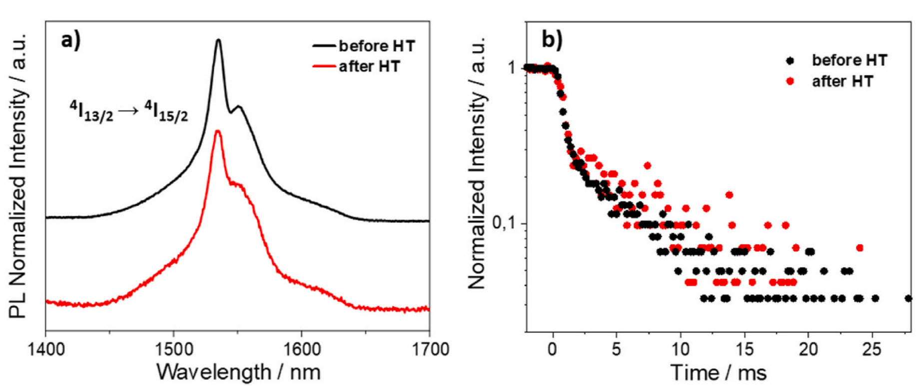

Fig. 3. Normalized Er^{3+} a) $^4I_{13/2} \rightarrow {}^4I_{15/2}$ photoluminescence spectra and b) $^4I_{13/2}$ decay curves before (*black*) and after (*red*) the heat-treatment at 400 °C for 8 h (λ_{ex} = 514.5 nm). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

perfectly superimposable, hence, the thermal treatment at a relatively low temperature (400 °C) does not significantly influence the Er^{3+} coordination environment, that results to be amorphous [28,29] before and after the heat-treatment (HT). This confirms what is expected, *i.e.* waveguides fabricated via RF-sputtering technique does not require subsequent thermal-treatment, because the system is already densified and Er^{3+} emitting ions behaviour is not influenced by the treatment. This is a crucial aspect to consider when working with flexible substrates, in this case AS 87 eco SCHOTT ultrathin glass that, as reported in previous studies [31,32], cannot be subjected to heat treatment at mild temperatures (≥400 °C) without showing a decrease in the mechanical flexibility.

The $^4I_{13/2}$ state decay curve (Fig. 3b) presents two components. A fast decay with a lifetime τ_e of *ca.* 0.8 ms and a tail with a decay time of about 5 ms. A lifetime around 5 ms for the 1.5 μm emission of Er^{3+} is typical of silica-hafnia RF-sputtering waveguides mainly due to the disruptive role on the local environment induced by the Hf^{4+} ions [34,41]. The fast decay, observed in the waveguides here investigated, is explained considering the quite high amount of erbium (2.8 mol%) present in the waveguide that can lead to non-radiative deactivation pathways, such as cross-relaxation, due to the physical clustering [42]. In fact, although the important role of HfO_2 and SiO_2 in the rare earth ions solubility [34], the matrix does not seem to be able to resolve the clustering when a so high quantity of Erbium is employed. To alleviate the clustering, it will be necessary to opportunely modify the deposition protocol in order to optimize the waveguide composition. Considering the deposition procedure here presented, it appears that the relative volume fraction of the targets should be carefully checked, requiring a not easy experimental work. Various alternatives could be tested to achieve this goal, for example: *i*) tailor the RF-power in order to reduce the possible defects generated during the deposition; *ii*) reduce the size of the erbium metallic target.

4. Conclusions

Glass-based flexible luminescent devices will gain a key role in flexible photonics field and here we have presented the fabrication protocol to obtain all-glass SiO_2–HfO_2:Er^{3+} flexible planar waveguides. The preliminary results, obtained for the system fabricated using this *top-down* approach, show an attenuation coefficient as low as 0.2 dB/cm at 1.53 μm, an interesting value especially in view of future fabrication optimizations. The $^4I_{13/2} \rightarrow {}^4I_{15/2}$ emission at 1.53 μm of the erbium ions in an amorphous environment was clearly observed and resulted to not be influenced by the heat-treatment at 400 °C, confirming the fact that with RF-sputtering deposition technique thermal treatments can be avoided. In fact, RF-sputtering techniques allow not only to obtain optical-quality inorganic oxide layers but also to avoid thermal treatments. The Er^{3+} active planar waveguide, fabricated on ultrathin flexible glass substrate, by combining its mechanical flexibility, a low attenuation coefficient and the emission in the NIR region, represents a successful step toward modern flexible photonic applications.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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