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ABSTRACT

We present the radio frequency sputtering fabrication protocols for the fabrication on flexible polymeric substrates of glass-based 1D photonic crystals and erbium activated planar waveguides. Various characterization techniques, such as atomic force microscopy and optical microscopy, are employed to put in evidence the good adhesion of the glass coating on the polymeric substrates. Transmittance measurements are performed on the multilayer structure and indicate that there are no differences between the samples deposited on the polymeric and SiO\textsubscript{2} substrates, even after bending. Prism coupling technique is used to measure the optical parameter of the planar waveguide fabricated on flexible substrates. The \( ^{4}I_{132} \rightarrow ^{4}I_{5/2} \) emission band, detected upon TE\textsubscript{0} mode excitation at 514.5 nm, exhibits the spectral shape characteristic of Er\textsuperscript{3+} ions embedded in a crystalline environment.

**Keywords:** Flexible photonics; rf-sputtering; optical planar waveguide; 1D photonic crystal; glass photonics

1. INTRODUCTION

Glasses are a key material in photonics. Glasses, when activated by rare-earth ions, have demonstrated to be effective in the realization of planar waveguides, fibers and bulk devices operating as sensors, amplifiers, integrated lasers and luminescent systems [1, 2]. Oxide glasses are largely employed in several photonics applications because of their wide transparency window from the ultraviolet to the near-infrared and their good resistance to temperature, corrosion and radiation. [3–6]. These features make glasses very suitable for the fabrication of optically active confined structures [7]. A further crucial step in the development of photonic devices is the mechanical flexibility [8]. As already done in electronics, photonic devices require integration on flexible substrates for a broad spectrum of application ranging from optical interconnection to sensors for civil infrastructure and environments, to coherent and incoherent light sources and functionalized coatings for integration on biological tissue [8, 9]. While conventional

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photonic devices are fabricated on rigid substrates, integration on deformable polymer substrates has given birth to flexible photonics, a field which has rapidly emerged in recent years to the forefront of photonics. Adding mechanical flexibility to planar photonic structures, the spectrum of application acquires an incredible expansion. Planar integrated photonic structures such as gratings, and channel waveguides but also solar cells and protective coatings offer unique performance characteristics as compared to their rigid classical structures. [10] To date, flexible planar photonic devices were almost exclusively fabricated using polymeric substrates, which do not possess the high refractive indices necessary for strong optical confinement. It is worth noting that Corning [11] and Schott [12] have recently presented ultra-thin flexible glasses, so, there is a chance of a future development of an all-glass flexible photonics. The fabricated systems should operate in several deformation conditions i.e. bending, folding, rolling, twisting, stretching, and compression [13]. To succeed, the following steps are crucial: i) design and fabrication of appropriate flexible substrate; ii) realization of planar waveguides and photonic crystals deposited on the flexible substrates reducing radiative losses and maintaining a constant adhesion under mechanical deformation.

Numerous deposition techniques are used to fabricate photonic devices based on glasses and the RF-magnetron sputtering technique has demonstrated to be a suitable method for glass deposition on large areas for optoelectronic devices [14, 15] also on polymeric substrates [16, 17].

We have, indeed, shown that RF sputtering deposition is a suitable technique to fabricate a fully Er\textsuperscript{3+} doped monolithic 1-D dielectric microwire for coherent emission based on SiO\textsubscript{2}/TiO\textsubscript{2} multilayer structures on SiO\textsubscript{2} substrates [15]. Moreover, it is also proved that it is possible to fabricated SiO\textsubscript{2} based planar waveguides doped with erbium by rf-sputtering [14].

However, when polymeric substrates are employed, the temperature of the sample holder during the deposition process becomes a crucial parameter that has to be tailored [18] so different deposition protocols have to be defined when these kinds of substrate are used.

Here we present some results concerning the fabrication of novel flexible optical layers by radio frequency sputtering deposition. In particular, 1D photonic crystals and planar waveguides, both passive and activated by rare earth ions will be discussed. The general idea is to develop a viable technological way to transform intrinsically rigid or brittle materials into highly mechanically flexible and optically functional systems.

### 2. EXPERIMENTAL

The samples were prepared by multi target rf-sputtering technique using Poly-methyl methacrylate (PMMA), Polyether ether ketone (PEEK), silicon and silica substrates. The silica and silicon substrate have dimensions 7x3.5 cm while the PMMA and PEEK substrates have dimensions 1x3.5cm. The sample labels with the different substrates used and the description of the fabricated structures are summarized in table 1.

<table>
<thead>
<tr>
<th>Sample Label</th>
<th>Substrate</th>
<th>Structure</th>
</tr>
</thead>
<tbody>
<tr>
<td>OS6</td>
<td>Si</td>
<td>Multilayer structure consisting of 7 couple SiO\textsubscript{2} and TiO\textsubscript{2} layers.</td>
</tr>
<tr>
<td></td>
<td>SiO\textsubscript{2}</td>
<td></td>
</tr>
<tr>
<td></td>
<td>PMMA</td>
<td></td>
</tr>
<tr>
<td></td>
<td>PEEK</td>
<td></td>
</tr>
<tr>
<td>FW01</td>
<td>Si</td>
<td>Planar waveguide with a buffer layer of SiO\textsubscript{2} of 4 ( \mu )m deposited directly on the substrates and an active waveguiding layer of SiO\textsubscript{2}-HfO\textsubscript{2}:Er\textsuperscript{3+}</td>
</tr>
<tr>
<td></td>
<td>SiO\textsubscript{2}</td>
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<tr>
<td></td>
<td>PEEK</td>
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</tbody>
</table>

Sputtering deposition of the multilayer structures labelled OS6 and consisting on 7 couples (14 single layers) of SiO\textsubscript{2} and TiO\textsubscript{2} films were performed by sputtering alternatively changing a 15x5cm titania and 15x5cm silica targets. The residual pressure before the deposition is 4.5x10\textsuperscript{-7} mbar. During the deposition procedure, the substrates were not heated and the temperature of the sample holder during the deposition is kept at 30 °C. The sputtering conditions were an Ar gas pressure of 5.4 \( \times 10\textsuperscript{3} \) mbar, an applied rf power of 110 W for both silica and titania targets. To monitor the thickness of the layers during the deposition, two quartz microbalances Inficon instruments thickness monitor model SQM-160, faced
on the two targets, were employed. Thickness monitoring was calibrated for the two kinds of materials by a long deposition process (24 hrs of deposition) and by directly measuring the thickness of the deposited layer by an m-line apparatus [19, 20]. The final resolution on the effective thickness obtained by this quartz microbalance is about 1 Angstrom. More details are available in reference [15]. The total thickness of the OS6 sample is 1μm. Sputtering deposition of the planar waveguide labelled FW01 was performed by sputtering. As reported in Table 1 first a silica layer of about 4 μm acting as buffer layer was deposited on the different substrates. Then the active layer SiO2-HfO2:Er3+ was prepared sputtering at the same time 15×5cm hafnia and 15×5cm silica targets, on which metallic erbium pieces were placed, and moving the substrates above the targets. The sputtering conditions were an Ar gas pressure of 5.4×10⁻³ mbar and an applied rf power of 90 W for both silica and hafnia targets [14, 15]. All the samples were not heated after the deposition.

The absorption spectra in the VIS-NIR region of the samples was obtained by using a double beam Varian-Cary spectrophotometer with a resolution of 2 nm [15]. Atomic Force Microscopy (AFM) images were acquired with a Solver Px Scanning Probe Microscope from NT-MDT. AFM topographies were acquired in semi-contact mode with a silicon tip ~219.8 KHz, Q338 with a nominal radius of less than 10 nm. Analyses were performed in the “center” of each sample. Wide areas, 20x20 micron, were scanned at lower resolution (256x256 pixels) in order to avoid particles and determine the best area to characterize. Analyses were performed on different scanning areas, 10x10 μm², 5x5 μm² and 2x2 μm² with a resolution of 512x512 pixels. Wider areas (100x100 μm²) were also scanned to look for any scratch or creep occurring on the surface layer due to the bending of the substrates.

Optical parameters of the planar waveguide at 543 and 633nm were measured in TE and TM polarization by an m-line apparatus based on the prism coupling technique [14, 19, 20]. 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 TE₀ 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 [6, 14, 15].

### 3. RESULTS AND DISCUSSION

#### 3.1 Multilayer 1D structures.

Figure 1 shows the photos of the OS6 multilayer structure consisting of 7 couple SiO₂ and TiO₂ layers deposited on a PMMA substrate. The sample is shown under bending (left) and after bending (right).

![Figure 1. OS6 multilayer structure consisting of 7 couple SiO₂/TiO₂ layers deposited on a PMMA substrate. The sample is shown under bending (left) and after bending (right).](image-url)
It is evident that the adhesion of the coating is not influenced by the bending.

A deeper investigation has been performed by optical microscope inspection. Figure 2 shows the optical microscope images of the OS6 multilayer structure. On the left it is shown the unbent region of the sample while, on the right, it is reported the status of the bent region of the sample.

From Fig. 2 it is possible to identify punctual defects on the coating, probably due to not perfect cleaning of the substrates before the deposition, presence of contamination on the surface of the substrates could results in this kind of imperfections. The cleaning protocol employed for conventional SiO<sub>2</sub> and Si substrates require a heating at 120°C for some minutes in vacuum at 10<sup>-6</sup> mbar just before the deposition. This cleaning step could damage the polymeric substrate and was not performed. In this case the surface of the substrates was cleaned just with deionized water and ethanol.

It is also evident from Fig 2 that after the bending, the film is not delaminated and it still adherent to the substrate. However, some patterns appear. These ripples are probably induced on the coating by a surface change of the PMMA substrate. This pattern appears, in fact, also on the bare PMMA substrate and do not seems to influence the mechanical stability of the coating.

Figure 3 shows the AFM images of the three uncoated substrates silica, silicon and PMMA and of the three OS6-multilayer samples deposited on these substrates. In the case of the sample deposited on PMMA substrate, the AFM measurements were performed after bending as reported in Figure 1. Figure 4 shows the behavior of the Average Roughness (Ra) and Root Mean Square Roughness (Rq) on different scan area for the three substrates and relative three coating.

Figures 3 and 4 evidence that the deposited samples exhibit homogeneous surface roughness. Rq and Ra are, indeed, similar for the four scan areas and this fact indicate a homogeneous surface roughness. The roughness rises from about 4 to 5 nm for silicon and silica and from 2 nm for PMMA.

In the sample OS6 deposited on the different substrates, inspection performed on a wider (100x100 μm<sup>2</sup>) area, not reported in the figures, does not show many defects.
Figure 3: AFM scan area images of the three uncoated substrates silica, silicon and PMMA and of the three OS6-multilayer samples deposited on these substrates. In the case of the sample deposited on PMMA substrate the AFM measurements were performed after bending as reported in figure 1. The scan area is of 2x2 µm². The vertical bar indicates the height range.
Figure 4. Average Roughness (Ra) and Root Mean Square Roughness (Rq) on different scan area for the three substrates silica, silicon and PMMA and relative coating.
Figure 5. Transmittance spectrum of the OS6 sample deposited on silica and PMMA substrates after bending as reported in Figure 1.

Figure 5 shows the transmittance spectra of the OS6 sample deposited on silica and PMMA substrates after bending as reported in Figure 1. The transmittance measurement performed on the sample OS6 does not put in evidence any substantial differences comparing the PMMA and silica substrate and only modifications due to the different optical response of the substrates. It is worth to noting that, the transmittance spectra do not change even after more than ten bending cycles.

3.2 Planar waveguides.

Figure 6 shows the prism coupling setup for the m-line measurement performed on the FW01 planar waveguide. To ensure the prism coupling and avoid damaging the prism a bare microscope slide was placed between the sample and the piston [19, 20].
Figure 6. Prism coupling configuration used for the m-line measurement at 632.8 nm for the FW01 planar waveguide deposited on PEEK substrate. The positions of the FW1 waveguide, the microscope slide and the piston of the m-line are indicated.

Figure 7 shows the M-line spectra obtained at 632.8 nm and 543.5 nm in TE and TM polarization for the FW01 waveguide. The waveguide supports 2 modes at both wavelengths. The thickness of the waveguiding film is 1.2±0.1 μm with a refractive index of 1.518±0.005 @ 632.8 nm and 1.523±0.005 @ 543.5 nm. The refractive indexes measured in TE and TM polarization at both wavelengths are equivalent, within the experimental uncertainty indicating that birefringence is negligible. The measured values of the refractive index of the buffer layer (labelled n_s) and the values of the effective refractive index of the modes are also reported.

It is evident in Figure 6 that the light propagates only few millimeters out from the prisms indicating an attenuation coefficient higher than 6 dB/cm. It is important to note that this high value of the attenuation coefficient is obtained also on the FW01 planar waveguide deposited on the other substrates. This indicates that losses are not due to different roughness of the films or effect of bending, but that they are due to the nature of the sputtered films, independently on the employed substrates. This fact is under investigation.

Figure 8 shows the photoluminescence spectra related to the \( ^{4}I_{13/2} \rightarrow ^{4}I_{15/2} \) transition of the Er\(^{3+}\) ions obtained for the FW01 planar waveguide in waveguiding configuration exciting the TE\(_{0}\) mode at 514.5 nm with an excitation power of 100 mW on the prism.
Figure 7. M-line spectra obtained at 632.8 nm and 543.5 nm in TE and TM polarization for the FW01 waveguide deposited on PEEK substrate.

Figure 8. \(^{4}_{1}S_{3/2} \rightarrow ^{4}_{1}I_{3/2}\) photoluminescence spectrum from the FW01 planar waveguide activated by Er\(^{3+}\) ions. The emission is recorded in waveguiding configuration upon excitation of the TE\(_0\) mode at 514.5 nm with an excitation power of 100mW on the prism.
The emission spectrum is characteristic of erbium ion in SiO$_2$-HfO$_2$:Er$^{3+}$ glass-ceramics [21]. Well resolved Stark components are, in fact, present in the spectrum. The order of the crystalline environment reduces the inhomogeneous broadening typical of emission spectra of rare earth ions in glasses [21, 22].

The same emission features are observed for all the FW01 planar waveguide, independently on the substrates.

In similar SiO$_2$-HfO$_2$ based planar waveguides fabricated by rf-sputtering deposition using a multicomponent target, as reported in [14, 23], structural characterization and rare earth emission spectra confirmed that the matrix was completely amorphous. In the present case a different fabrication protocol is used. The sample is heat treated after deposition and a pure HfO$_2$ target is employed instead of a multicomponent target. With this new protocol, it is possible to deposit a glass-ceramic film on polymeric substrate and without thermal treatment it is possible to obtain a crystalline environment for the rare earth ions [21].

4. CONCLUSIONS

The protocols based on rf-sputtering deposition for fabrication of glass based multilayer structures and optical planar waveguides activated by Er$^{3+}$ ions are defined. A 1D photonic crystal based on a SiO$_2$/TiO$_2$ multilayer structure was fabricated. Optical microscopy and AFM measurements confirm the good adhesion of the glass on the PMMA substrate also after bending. No cracks are observed on the multilayer structure. Transmittance spectra do not put in evidence differences between the sample deposit on the SiO$_2$ substrate and PMMA even after bending.

A glass ceramic planar waveguide composed by a buffer layer of SiO$_2$ of 4 μm deposited directly on the substrates and an active waveguiding layer of SiO$_2$-HfO$_2$:Er$^{3+}$ was fabricated on SiO$_2$, Si, PMMA and PEEK substrates. M-line spectroscopy was used to characterize the optical features of the sample. The waveguide supports 2 modes at 632.8 and 543.5 nm. The attenuation coefficient is higher than 6dB/cm and no difference on the optical parameter was observed on different substrates and after bending. The emission spectrum in the 1.5μm region, characteristic of erbium ion embedded in crystalline environment, is obtained. Further measurements are in progress to estimate the dimension of the nanocrystals.

Optimization of the fabrication protocol of the planar waveguide to reduce the attenuation coefficient and obtain the correct thickness to support a mode at 1550nm is also in progress.

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