Optical Materials: X 19 (2023) 100241

Contents lists available at ScienceDirect

Optical Materials: X

journal homepage: www.journals.elsevier.com/optical-materials-x

Under bending optical assessment of flexible glass based multilayer structures fabricated on polymeric substrates

Giacomo Zanetti [a,d,*], Alice Carlotto [a], Thi Ngoc Lam Tran [a,b,c], Anna Szczurek [e], Bartosz Babiarczuk [e], Osman Sayginer [f], Stefano Varas [a], Justyna Krzak [e], Oreste Bursi [g,a], Daniele Zonta [g,a], Giacomo Baldi [d], Matteo Bonomo [h], Simone Galliano [h], Claudia Barolo [h,i], Nicola Bazzanella [j], Silvia Maria Pietralunga [k], Alessandro Chiasera [a,**]

[a] IFN-CNR, CSMFO Lab. and FBK Photonics Unit, Via alla Cascata 56/C, 38123, Povo, TN, Italy
[b] Dept. of Physics, Politecnico di Milano, P.zza L. da Vinci 32, 20133, Milan, Italy
[c] Dept. of Materials Technology, Faculty of Applied Sciences, Ho Chi Minh City University of Technology and Education, Vo Van Ngan Str. 1, Thu Duc District, 720214, Ho Chi Minh City, Viet Nam
[d] Department of Physics, University of Trento, Via Sommarive 14, 38123, Povo, TN, Italy
[e] Dept. of Mechanics, Materials and Biomedical Engineering, Wroclaw University of Science and Technology, Smoluchowskiego 25, Wroclaw, 50-370, Poland
[f] Biological Imaging and TranslaTUM, Technische Universität München, Ismaninger Str. 22, Munich, D-81675, Germany
[g] Dept. of Civil, Environmental and Mechanical Engineering, University of Trento, via Mesiano 77, Trento, 38123, Italy
[h] Department of Chemistry, NIS Interdepartmental Centre and INSTM Reference Centre, University of Turin, Via Pietro Giuria 7, Turin, 10125, Italy
[i] Istituto di Scienza, Tecnologia e Sostenibilità per lo sviluppo dei Materiali Ceramici (ISSMC-CNR), Via Granarolo 64, 48018, Faenza, RA, Italy
[j] Laboratorio Idrogeno Energia Ambiente (IdEA), Department of Physics, University of Trento, Via Sommarive 14, 38123, Povo, TN, Italy
[k] IFN-CNR, P.zza Leonardo da Vinci 32, Milan, 20133, Italy

ARTICLE INFO

Keywords:
Flexible photonics
1D photonic crystal
Optical properties under bending
RF-Sputtering
TiO_2 thin films
SiO_2 thin films

ABSTRACT

We present the results of the spectral transmittance and reflectance of a SiO_2/TiO_2 1D photonic crystal deposited by Radio Frequency sputtering on a flexible polymeric substrate, which shows a blue-shift in its transmittance stopband proportional to the incidence angle when bent. An adjustable sample holder was designed to regulate the bending and keep the sample in a bent condition. Different angles of incidence were also achieved through variable angle reflectance, where an increase in incidence angle led to the blue-shift and narrowing of the transmittance stopband. We addressed the sample's resistance against bending wear and tear by comparing the transmittance spectra acquired for the flat sample before and after the measurements made in bent configurations. An important stability has been observed.

1. Introduction

The field of thin optical films deposited on flexible substrates has been rapidly growing in the past few years thanks to the great potentiality of these systems for developing applications in various areas, expanding the already broad spectrum of applications of rigid thin films. Sensing, amplification and flexible screens for augmented reality are only some of the high interest examples [1–6].

Different protocols can be employed to realize thin films, such as sol-gel [1,7,8] and Radio Frequency (RF) sputtering [9], which has already been demonstrated to be a suitable technique for the fabrication of 1D photonic structures on flexible substrates [2,10–14].

The development of flexible photonic devices not only requires specific fabrication protocols, but also different design approaches and characterization procedures [15–18]. In particular, the optical and spectroscopic assessment must be performed both before and after the bending, as well as during the bending procedures in order to put in evidence possible interactions between mechanical stress induced by bending and optical or spectroscopic features [2]. To do so, sample holders designed to monitor the properties of the sample in bent configurations must be developed.

In this paper, the spectral transmittance of a glass based multilayer

* Corresponding author. IFN-CNR, CSMFO Lab. and FBK Photonics Unit, Via alla Cascata 56/C, 38123, Povo TN, Italy.
** Corresponding author.
E-mail addresses: gzanetti@fbk.eu (G. Zanetti), chiasera@fbk.eu (A. Chiasera).

https://doi.org/10.1016/j.omx.2023.100241
Received 24 May 2023; Received in revised form 6 June 2023; Accepted 8 June 2023
Available online 20 June 2023

structure fabricated on a polymeric substrate is studied under different bending conditions, and the results are compared to the ones obtained via variable angle reflectance technique. For this purpose, a sample holder has been designed and fabricated to monitor the properties of the sample in bent configurations. Also, the 1-Dimensional Photonic Crystal's (1D-PC) stability against bending wear and tear is tested.

2. Experimental

The 1D-PC was fabricated by multi target non-reactive RF-sputtering technique on a self-standing polymeric flexible substrate (76 mm x 25 mm x 1 mm). Simultaneously, the deposition was performed on a silicon substrate for Scanning Electron Microscopy characterization. Before the deposition, the silicon substrate was cleaned using an ultrasonic bath in deionized water. Afterwards, both substrates were cleansed with isopropanol and dried in nitrogen. The residual pressure in the sputtering chamber before the deposition was $4.5 \cdot 10^{-7}$ mbar. Inside the chamber, a target plate (15 cm × 5 cm) of SiO_2 and an identical one of TiO_2 were placed in order to alternate the deposited materials: once the first layer is formed, the sample is moved over the other target plate and the deposition of the second layer starts; this process is iterated until all the layers are deposited. The deposition process was performed in an Ar environment ($5.4 \cdot 10^{-3}$ mbar) with a set RF power of 100 W for both targets, the substrates were not heated, and the holder's temperature was kept at 30 °C. Starting from a SiO_2 layer, a total of 10 alternating layers was deposited. To monitor the thickness of the layers during the deposition, two quartz microbalances INFICON model SQM-160, each facing one of the targets, were employed. More details about the deposition setup and process are available in Ref. [19]. Eventually, an 8-hour heat treatment was performed, in air, at 150 °C.

Scanning Electron Microscopy (SEM) analyses were carried out, on the 1D-PC grown on silicon substrate, using a JEOL JSM-7001F SEM-FEG instrument. Measurements were performed at 20 keV electron beam energy and 10 mm of working distance. The thicknesses of single layers were estimated by processing the cross-sectional SEM micrographs, resulting in a thickness of 83 ± 2 nm for the SiO_2 layers and 51 ± 3 nm for the TiO_2 ones. The analysis was performed on the film cross-section fixing the sample on a 90 ° stub and using Backscattering Electrons (BSE) images to better separate the different SiO_2 and TiO_2 layers from the substrate (Fig. 1). To have a conductive surface, a layer of about 15 nm of carbon was deposited on the sample.

To study the optical properties of the 1D-PC deposited on the polymeric substrate under bending, we developed a sample holder adjustable in size, thus allowing us to regulate the bending of the substrate and keep the sample in a bent condition during the measurements. This sample holder and a possible configuration are reported in Fig. 2.

Transmission spectra and reflectance spectra at different angles in the Vis-NIR region (300-1200 nm) were obtained using a double beam Varian-Cary 5000 spectrophotometer with a resolution of 2 nm. We studied a fixed point of the sample to avoid the deviations that could have arisen from the sample's inhomogeneities; in particular, the spot chosen was at approximately 1.4 cm from the short edge of the sample and centred in the other direction. Furthermore, we used a 2x2 mm slit to limit the shined area and always kept the 1D-PC as close to it as possible. The machine is also capable of performing variable angle reflectance, allowing us to compare the dependence of the reflectance on the angle of incidence when the sample is flat, with the results obtained by changing the incidence angle through bending.

The angles of incidence in the case of bent surface were estimated with a simple code that approximates the bent shape with a parabola. Through geometrical considerations, we were able to show that, in the range studied, the measured angles were always within 1.5 ° from the estimated values. In any case, the objective was not to quantify this angle dependence, but rather to study it qualitatively: the angles are therefore to be taken as a rough index that represents how bent the sample is, also because the discrete size of the sampling beam means that the measurements are made on a portion of the 1D-PC that does not have a single incidence angle.

Before and after the measurements in bent configurations, we acquired the spectrum of the flat layout to see if the bending procedure altered the sample's properties at rest. Also, since the curved shape implies that some points are closer to the slit than others, and thus are shined by a beam that has diverged less, we checked whether the distance from the slit influenced the results.

3. Results and discussion

The variable angle reflectance spectra are shown in Fig. 3. As the angle increases, the high-reflectance band shifts to higher frequencies and narrows. Note that the first angle is 20 °, as it is the smallest allowed by the instrument used.

Then, the spectral response of the fixed point we had chosen (see the previous section) was studied in different bending conditions, and is shown in Fig. 4. This measuring procedure allows to see the influence of the bending only, because it rules out the dependence on the possible inhomogeneities of the 1D-PC.

From Fig. 4, it can be deduced that increasing the bending of the sample increases the blue-shift of the resonance, as previously observed with variable angle reflectance measurements. What is different, though, is the fact that here no narrowing of the bands is observed. Since we have carefully kept the sampled point the same during the measurements, and the light beam is quite collimated, we could attribute the narrowing effect, previously observed, to the procedure used in reflectance measurements, where the light is spread on a much broader portion of the sample. This means that, to study the optical properties of the bent 1D-PC, variable angle reflectance is not a valid choice: for the same incidence angles, the results are, in general, different between variable angle reflectance and the transmittance measurements obtained by bending the sample.

To evaluate the stability of the sample against bending, we measured

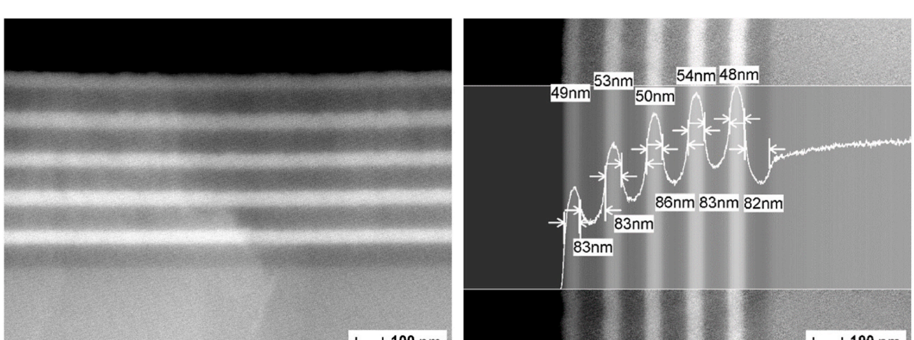

Fig. 1. Cross-sectional SEM micrograph of the 1D photonic crystal and layers' thickness estimation.

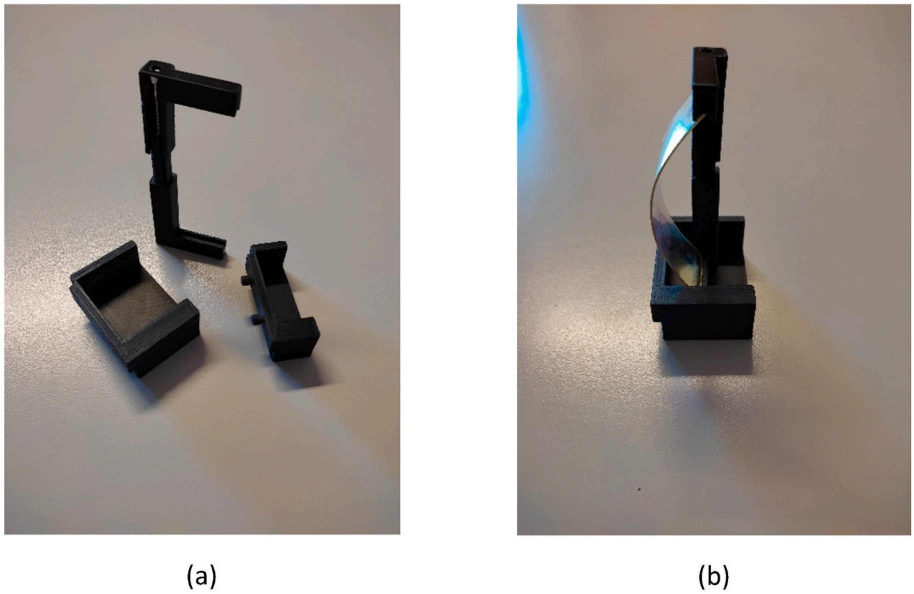

Fig. 2. (a) Picture of the holder with two supports; (b) support and sample holder with the 1D-PC mounted in a bent configuration.

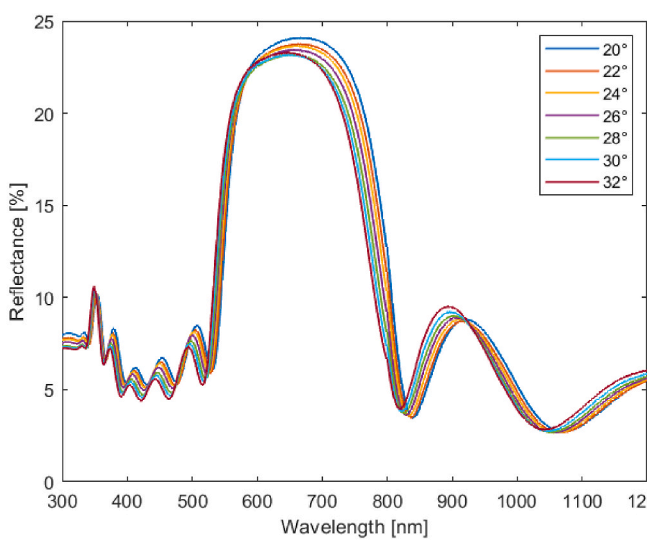

Fig. 3. Variable angle reflectance made on the 1D-PC.

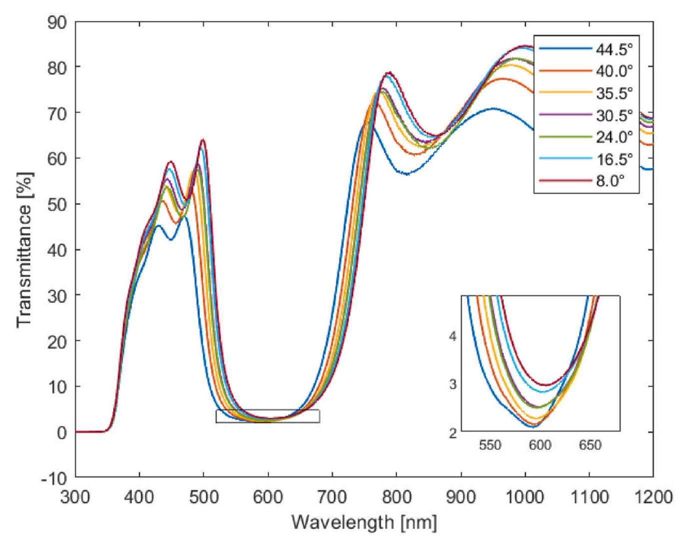

Fig. 4. Transmittance spectra under different bending conditions. The angles have been estimated using a parabolic model.

the transmittance spectrum of three arbitrary points of the sample before and after repeatedly bending it. In particular, the configurations of Fig. 4 were reached roughly 10 times each and held for periods of time always longer than 2 minutes. The corresponding spectra are reported in Fig. 5: the bending did not sensibly alter the position of the stopband for any of the considered points, although the film suffered cracking damage. Despite this, SEM analysis did not show any alteration of the layers as a result of bending. Additionally, the sample responded elastically to bending below the 30.5 ° configuration.

Finally, we wanted to investigate if any of the observed effects could depend on the different distances between slit and sample in the various bending configurations. Ideally, the point that is being investigated should stay as close as possible to the slit, in order to avoid dispersion of the light and hence more surface exposed, which also means that light enters the sample at a wider range of angles. However, since we needed to accommodate the bent sample, the distance between the slit and the examined point of the 1D-PC depended on the configuration. We tested the two extremal configurations, namely at maximum possible bending and at almost zero bending (the polymer couldn't stay straight due to the previous stresses, which resulted in a slight bending). These measurements have been done using the leftmost support shown in Fig. 2(a) and the point sampled was the one selected at the beginning. In particular, the "close" condition is the one that allows the least distance between sample and slit, "medium" has the holder in the middle of the support, while "far" has it at the support's edge. The support was approximately 3.7 cm long.

What we can see is that the distance from the slit seems to red-shift the band, but the effect becomes significant only after a certain spacing value. In fact, the "close" and "medium" configurations in Fig. 6(a) have the exact same transmittance. An important variation is observed only if we compare them with the "far" configuration. If we compare the two pictures, having a bigger angle makes the dependence on the distance greater. We believe it is because the light impinges on a broader area of the 1D-PC if it is more bent, resulting in a wider range of incident angles with respect to the slightly bent case.

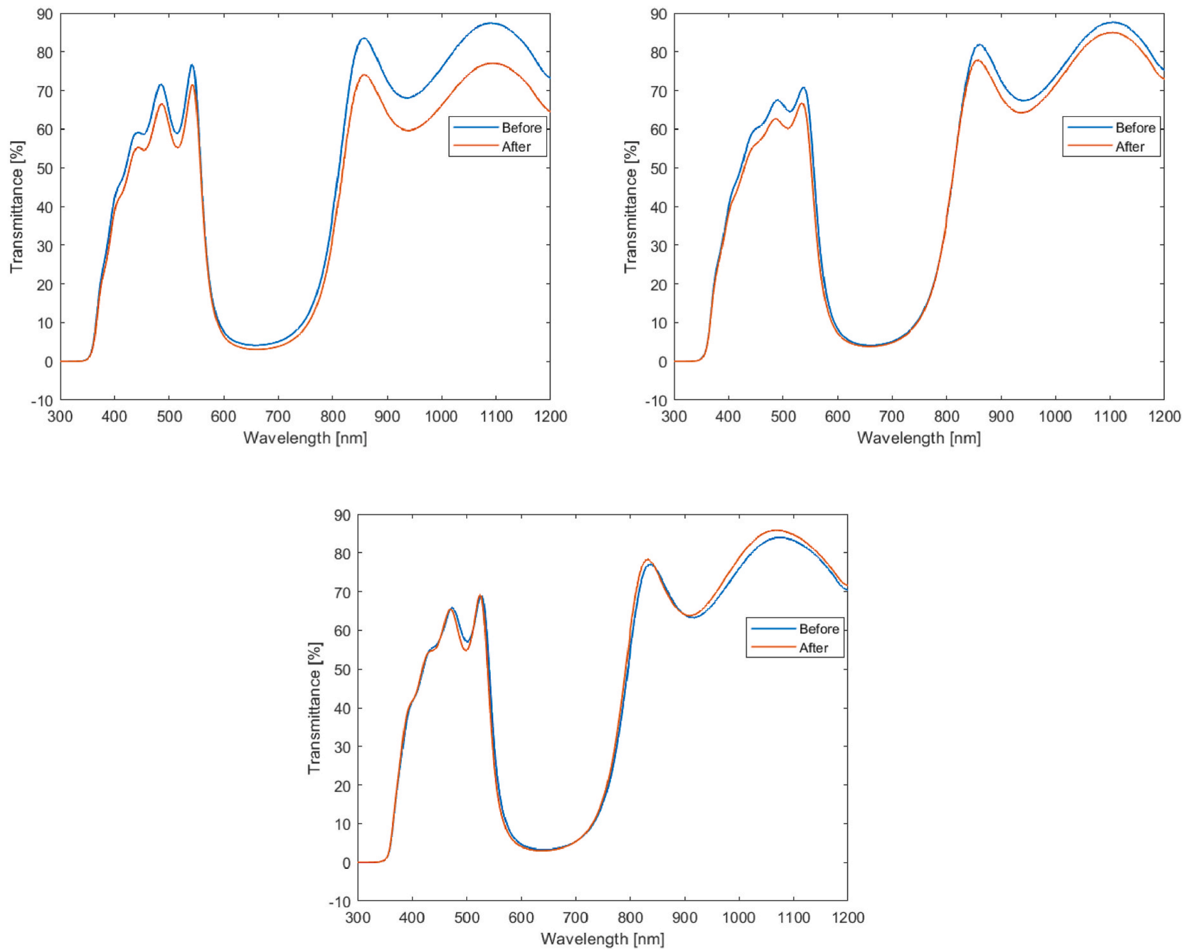

Fig. 5. Comparison between transmittance spectra before and after bending for three points of the 1D-PC.

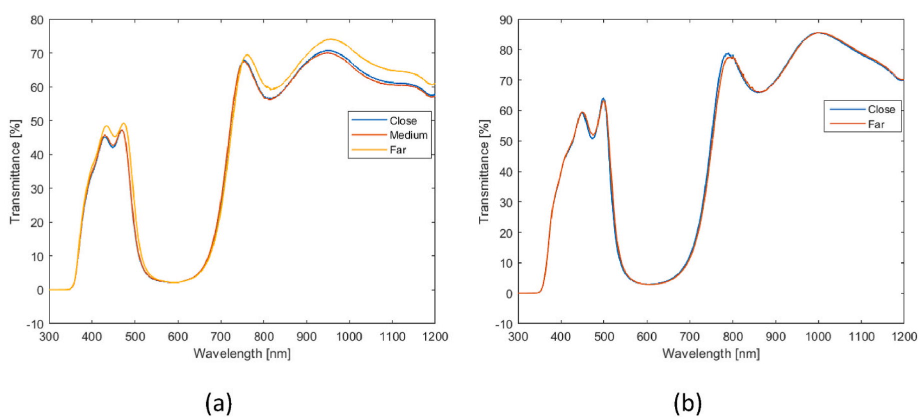

Fig. 6. Transmittance of the fixed sample point at different distances from the slit. In (a), the estimated angle is 44.5 °, while it is 8.0 ° in (b).

4. Conclusions

Flexible glass based multilayer structures extend the scope of photonic applications by removing the constraint of a rigid device. In order to tap into this potential, we must understand what consequences bending has on the optical properties of photonic structures, especially those based on planar configuration designs. In this paper, we reported the phenomenology of a 10-layer SiO_2/TiO_2 1D-PC deposited by RF-sputtering technique on a flexible polymeric substrate. The main findings are the slight blue-shift proportional to the bending and the impossibility to study bent configurations by directly employing variable angle reflectance, as it yields results that behave differently from the spectra obtained by physically bending the sample. Moreover, the size of the sampled region greatly affects the optical properties, as it determines the range of incidence angles at which a light beam enters the 1D-PC. This is evident from the change in transmittance spectra collected at different distances between slit and sample surface. The studied structure also manifests great stability against bending wear and tear.

CRediT authorship contribution statement

Giacomo Zanetti: Conceptualization, Methodology, Software, Writing – original draft, Figure creation.. **Alice Carlotto:** Investigation, Writing – review & editing, Figure creation.. **Thi Ngoc Lam Tran:** Investigation, Writing – review & editing, Figure creation. **Anna Szczurek:** Investigation, Writing – review & editing, Figure creation.. **Bartosz Babiarczuk:** Investigation, Writing – review & editing, Figure creation.. **Osman Sayginer:** Investigation, Writing – review & editing, Figure creation.. **Stefano Varas:** Investigation, Software, Technical support.. **Justyna Krzak:** Writing – review & editing. **Oreste Bursi:** Writing – review & editing. **Daniele Zonta:** Writing – review & editing. **Giacomo Baldi:** Writing – review & editing. **Matteo Bonomo:** Research coordination, Scientific management, and manuscript revision.. **Simone Galliano:** Writing – review & editing. **Claudia Barolo:** Writing – review & editing. **Nicola Bazzanella:** Investigation, Software, Technical Support. **Silvia Maria Pietralunga:** Research coordination, Scientific management, and manuscript revision.. **Alessandro Chiasera:** Research coordination, Scientific management, and manuscript revision.

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.

Data availability

Data will be made available on request.

Acknowledgements

This research is supported by the projects: FESR-PON 2014-2020 BEST4U ARS01_00519; CNR-PAS "Flexible Photonics" (2020-2022); NAWA PPN/IWA/2018/1/00104; MIUR- 'Departments of Excellence' L 232/2016; ERC-H2020 PAIDEIA GA 816313; "Nuovi Concetti, mAteriali e tecnologie per l'iNtegrazione del fotoVoltAico negli edifici in uno scenario di generazione diffuSa" CANVAS and NAWA-MAECI Canaletto (2022-2023). M.B., S.G. and C.B. acknowledge support from the Project CH4.0 under the MUR program "Dipartimenti di Eccellenza 2023–2027" (CUP D13C22003520001).

References

[1] Y. Jestin, C. Armellini, A. Chiasera, A. Chiappini, M. Ferrari, E. Moser, R. Retoux, G.C. Righini, Low-loss optical Er3+-activated glass-ceramics planar waveguides fabricated by bottom-up approach, Appl. Phys. Lett. 91 (2007), 071909, https://doi.org/10.1063/1.2771537.

[2] O. Sayginer, A. Chiasera, L. Zur, S. Varas, L. Thi Ngoc Tran, C. Armellini, M. Ferrari, O.S. Bursi, Fabrication, modelling and assessment of hybrid 1-D elastic Fabry Perot microcavity for mechanical sensing applications, Ceram. Int. 45 (2019) 7785–7788, https://doi.org/10.1016/J.CERAMINT.2019.01.083.

[3] A.Q. Le Quang, E. Besson, R. Hierle, A. Mehdi, C. Reyé, R. Corriu, J. Zyss, S. Pietralunga, I. Ledoux, Erbium-doped polymer-based materials and waveguides for amplification at 1,55 µm, in: Y. Sidorin, C.A. Waechter (Eds.), Integr. Opt. Devices, Mater. Technol. X, SPIE, 2006, 612302, https://doi.org/10.1117/12.644655.

[4] L. Eisen, M. Meyklyar, M. Golub, A.A. Friesem, I. Gurwich, V. Weiss, Planar configuration for image projection, Appl. Opt. 45 (2006) 4005, https://doi.org/10.1364/AO.45.004005.

[5] Y.-H. Lee, T. Zhan, S.-T. Wu, Prospects and challenges in augmented reality displays, Virtual Real, Intell. Hardw. 1 (2019) 10–20, https://doi.org/10.3724/SP.J.2096-5796.2018.0009.

[6] J.L. Ferrari, K.D.O. Lima, R.R. Gonçalves, Refractive indexes and spectroscopic properties to design Er3+-doped SiO2-Ta2O5 films as multifunctional planar waveguide platforms for optical sensors and amplifiers, ACS Omega 6 (2021) 8784–8796, https://doi.org/10.1021/acsomega.0c05351.

[7] R.R. Gonçalves, G. Carturan, L. Zampedri, M. Ferrari, M. Montagna, A. Chiasera, G. C. Righini, S. Pelli, S.J.L. Ribeiro, Y. Messaddeq, Sol-gel Er-doped SiO2–HfO2 planar waveguides: a viable system for 1.5 µm application, Appl. Phys. Lett. 81 (2002) 28, https://doi.org/10.1063/1.1489477.

[8] Y. Jestin, C. Arfuso-Duverger, C. Armellini, B. Boulard, A. Chiappini, A. Chiasera, M. Ferrari, E. Moser, G. Nunzi Conti, S. Pelli, O. Peron, R. Retoux, G.C. Righini, Ceramization of erbium activated planar waveguides by bottom up technique, in: S. Jiang, M.J.F. Digonnet (Eds.), Opt. Components Mater. IV, SPIE, 2007, 646909, https://doi.org/10.1117/12.702057.

[9] C. Tosello, F. Rossi, S. Ronchin, R. Rolli, G.C. Righini, F. Pozzi, S. Pelli, M. Fossi, E. Moser, M. Montagna, M. Ferrari, C. Duverger, A. Chiappini, C. De Bernardi, Erbium-activated silica–titania planar waveguides on silica-on-silicon substrates prepared by rf sputtering, J. Non-Cryst. Solids 284 (2001) 230–236, https://doi.org/10.1016/S0022-3093(01)00407-0.

[10] A. Chiasera, O. Sayginer, E. Iacob, A. Szczurek, S. Varas, J. Krzak, O.S. Bursi, D. Zonta, A. Lukowiak, G.C. Righini, M. Ferrari, Flexible photonics: RF-sputtering fabrication of glass-based systems operating under mechanical deformation conditions, in: S. Taccheo, M. Ferrari, J.I. Mackenzie (Eds.), Fiber Lasers Glas. Photonics Mater. Through Appl. II, SPIE, 2020, p. 3, https://doi.org/10.1117/12.2551472.

[11] A. Chiasera, A. Szczurek, L.T.N. Tran, K. Startek, O. Saynger, S. Varas, C. Armellini, A. Chiappini, A. Carpentiero, D. Zonta, O. Bursi, R. Ramponi, M. Bollani, F. Scotognella, G. Macrelli, J. Krzak, G.C. Righini, M. Ferrari, A. Lukowiak, Flexible photonics: transform rigid materials into mechanically flexible and optically functional systems, in: M.J. Digonnet, S. Jiang (Eds.), Opt. Components Mater. XVIII, SPIE, 2021, p. 24, https://doi.org/10.1117/12.2577860.

[12] A. Szczurek, L.T.N. Tran, S. Varas, D. Lewandowski, A. Gąsiorek, B. Babiarczuk, A. Carlotto, A. Chiasera, M. Ferrari, A. Łukowiak, J. Krzak, SiO2-TiO2 hybrid coatings applied on polymeric materials for flexible photonics applications, in: S. Taccheo, M. Ferrari, A.B. Seddon (Eds.), Fiber Lasers Glas. Photonics Mater. Through Appl. III, SPIE, 2022, p. 11, https://doi.org/10.1117/12.2621465.

[13] A. Carlotto, O. Sayginer, H. Chen, T.N.L. Tran, R. Dell'Anna, A. Szczurek, S. Varas, B. Babiarczuk, J. Krzak, O.S. Bursi, D. Zonta, A. Lukowiak, G.C. Righini, M. Ferrari, S.M. Pietralunga, A. Chiasera, RF-sputtering fabrication of flexible glass-based 1D photonic crystals, in: S. Taccheo, M. Ferrari, A.B. Seddon (Eds.), Fiber Lasers Glas. Photonics Mater. Through Appl. III, SPIE, 2022, p. 9, https://doi.org/10.1117/12.2621281.

[14] T.N.L. Tran, A. Szczurek, S. Varas, C. Armellini, F. Scotognella, A. Chiasera, M. Ferrari, G.C. Righini, A. Lukowiak, Rare-earth activated SnO2 photoluminescent thin films on flexible glass: synthesis, deposition and characterization, Opt. Mater. 124 (2022), 111978, https://doi.org/10.1016/J.OPTMAT.2022.111978.

[15] H. Zuo, S. Yu, T. Gu, J. Hu, Low loss, flexible single-mode polymer photonics, Opt Express 27 (2019), 11152, https://doi.org/10.1364/OE.27.011152.

[16] D.W. Mohammed, R. Waddingham, A.J. Flewitt, K.A. Sierros, J. Bowen, S. N. Kukureka, Mechanical properties of amorphous indium–gallium–zinc oxide thin films on compliant substrates for flexible optoelectronic devices, Thin Solid Films 594 (2015) 197–204, https://doi.org/10.1016/j.tsf.2015.09.052.

[17] W.J. Lee, S. Bera, P.K. Song, J.W. Lee, W. Dai, H.C. Kim, C.S. Kim, S.H. Kwon, Optimization of bending durability of Ti-ZnO thin films on flexible glass substrates with highly enhanced optoelectronic characteristics by atomic layer deposition, Jpn. J. Appl. Phys. 58 (2019), https://doi.org/10.7567/1347-4065/ab1cf4.

[18] Lukowiak Anna, Shibin Jiang, Maurizio Ferrari, Foreword, Opt. Mater. 129 (2022), 112555, https://doi.org/10.1016/j.optmat.2022.112555. ISSN 0925-3467, https://www.sciencedirect.com/science/article/pii/S0925346722005894.

[19] L. Moscardi, S. Varas, A. Chiasera, F. Scotognella, M. Guizzardi, J. Eur.Opt. Society-Rapid Publ. (2022), https://doi.org/10.1051/jeos/2022009.