

Polymer Microring Resonators for Biosensing Applications by Nanoimprint Lithography

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ABSTRACT

Nowadays there is a permanently growing demand and need for simple, low cost, user friendly and real-time monitoring devices in biosensing technologies. In this context our contribution aims to demonstrate a fast fabrication process of centimeter-scale polymer microring resonators by UV nanoimprint lithography. A negative photoresist (SU-2) patterned by photolithography acts as a master mold in the imprinting process flow. High quality devices were designed and fabricated using rib waveguides with 1 μm by 1 μm width and height quadratic geometry. A controllable thickness of the residual layer with values down to 200 nm was achieved by tuning the thickness of the initial layer before imprint and the pattern dimensions of the master mold. The microrings with 1 μm gap in a racetrack configuration are targeted for operation in a single mode at visible wavelengths, where cheap light sources are available and water absorption is much lower than at infrared wavelengths.

Keywords: polymer photonic biosensor, UV- nanoimprint, optical waveguides, microring resonator.

1. INTRODUCTION

Integrated optical devices in polymeric materials have been the subject of great interest over the last decades. Obviously this is because of their extraordinary properties, with most importantly high optical transmittance, but also an easier fabrication process, flexibility and their potential for low-cost production [1]. Moreover, compared to semiconductor materials, it is possible to tune more easily their refractive index and furthermore different active dopants can be added to the polymeric material to fabricate devices for a wide range of applications. Considering their potential for bio-applications including sensing, the biocompatibility and surface functionalization of polymers play an important and crucial role in the material selection. Regarding the fabrication of polymer devices, different methods have been described in the literature so far. Among them, the conventional UV lithography technique has been extensively used to pattern the polymers. By exposing a photoresist to UV light through a mask, waveguides are generated after developing the unexposed areas. Unfortunately the resolution obtained with this technique is limited by the diffraction of light as well as by scattering and interference. Many novel fabrication techniques including nanoimprint lithography (NIL) have been developed in order to overcome the drawback of the limited resolution in photolithography. It has been reported that NIL with low-cost equipment can fabricate high-resolution nanopatterns down to sub-25 nm resolution on a large scale [2]. This replication technology is either thermal or UV based and uses a master mould that is pressed onto a polymer previously spun on a substrate. Consequently after the UV or thermal curing and the demoulding, the patterns of the mould are imprinted on the desired polymer. A crucial requirement in the imprinting process is the mould choice (e.g., silicon, fused silica, PDMS, resist) and it depends on the polymer physical properties, its chemical composition and moreover on the desired imprinted pattern.

Most of the earlier studies described in the literature considering polymer-based biosensor characterization and applications have been focused at infrared wavelengths [3, 4]. However, there is an urgent challenge and demand for the development of polymer photonic based sensors at visible wavelengths, taking into account that the absorption of water is about two thousand times lower in the visible range than in near infrared regions and that lower cost light sources are available in this range [5]. Along these lines, M Hiltunen *et al.* [6] have nicely demonstrated a polymeric slot waveguide with an integrated Young interferometer at 633 nm wavelength. Moreover M. H. M. Salleh *et al.* [7] have proved a dual disk resonator integrated with a microfluidic device at visible wavelengths. Although these studies are of tremendous importance, in their fabrication process for the master mould the structures were patterned by electron beam lithography followed by etching and anti-adhesion treatment. This process flow is more sophisticated which leads to extremely high mould costs. Therefore it counteracts the idea of low cost devices fabricated by NIL. The replacement of the mould with a cheaper one can be a solution to decrease the overall cost. Most likely, polymer moulds are very promising candidates to solve these problems.

In this contribution we demonstrate a low-cost fabrication process of centimeter-scale polymer microring resonators by UV nanoimprint lithography. A negative photoresist (SU-2 from Microchem Corporation) patterned by photolithography acts as a master mould in the imprinting process flow. For the fabrication of the master mould, different from the work of other groups [8] that used two steps of lithography followed by RIE,

we use single-step lithography without the etching step and the anti-adhesion treatment. Additionally a thermal treatment was applied to the master mould in order to decrease the surface roughness of the final imprinted structures.

It has been shown that using visible wavelengths of light within a biosensor, the gap between the bus waveguide and ring resonator is below 100 nm in order to enable an efficient coupling of light [7]. Thanks to the racetrack configuration of the microrings used in this study, we maintain the optical performance of the structure while extending the tolerance of the fabrication process by increasing the values of the gap to around 1 μm .

2. SIMULATION AND FABRICATION

As an initial step, prior to the design and physical fabrication of the resonators, simulations with the commercial Fimmwave software have been performed in order to estimate the resonator parameters for good performance at the wavelength $\lambda = 850 \text{ nm}$. Figure 1a depicts the cross-section of the rib waveguide used for the design and simulation. As a core material we used the hybrid polymer Ormocore [9] with a high refractive index of $n_{\text{core}} = 1.543$. Silicon wafers are used as substrates with a 3 μm thermal oxide on the top which act as cladding $n_{\text{cladding}} = 1.453$. Water was considered as upper cladding by default $n_{\text{water}} = 1.33$, which is the typical solution in a sensing application. As the number of the guided modes depends of the waveguide geometry, initially it has been determined that for single mode operation, the waveguide width and height, within a range of dimension tolerances can be fixed to 1 μm . The horizontal cross section plot of the E_x field shown in Fig. 1a illustrates that the maximum of the optical field is located in the core of the rib-waveguide with a quadratic geometry.

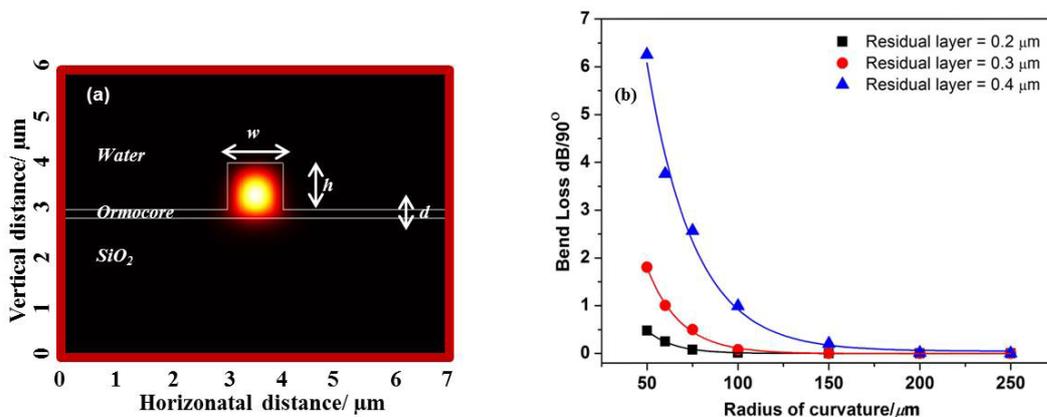


Figure 1: (a) The Fimmwave simulated intensity distribution of the fundamental TE -mode of the rib waveguide used in our design; (b) The bend losses of polymer rib waveguide with different residual layer dimensions.

As is very well known, for biosensing applications highly sensitive sensors are desired, therefore there is a need for microrings with high Q factors. Low losses will lead to higher Q factors, therefore in the fabrication process any kind of loss should be minimized, like e.g. the bend loss. This bend loss is mainly determined by the desired ring radius (and increases exponentially as the ring radius decreases) but will be also influenced by the thickness of the residual layer. In this context, the graph in Fig. 1b depicts the bend losses as a function of the radius for an increasing thickness of the residual layer. It can be seen that for a given radius value, the bend losses increase significantly with increasing thickness of the residual layer. Moreover, the FSR of the ring is inversely proportional to the radius of the ring. Therefore in order to get large FSR values we have to minimize the radius according to the bend losses tolerances. Our mask layout includes rings with radius tuned from 150 to 220 μm ; consequently the FSR was tuned from 0.51 to 0.35. Using a directional coupler configuration, the coupling efficiency between the straight waveguide and the ring was controlled by tuning the gap distance and the coupling length. For example, in order to achieve between 10% and 30% coupling efficiency, the coupling length should be larger than 180 μm for a waveguide with quadratic geometry with width and height of 1 μm , with 0.2 μm residual layer and for a fixed gap of 1 μm .

For the fabrication of the microrings we use a simple process flow mainly divided in two steps as illustrated in Fig. 2. The first step is represented by the fabrication of a resist master mould (Fig. 2a). After SU8-2 was spun on fused silica at 5600 rpm for 40 seconds, it was patterned by UV lithography. The SU8 film thickness was about 960 nm. After the development of the resist, in which the unexposed part of SU8-2 was removed, the substrates were hard baked at 180° for 3 minutes in order to decrease the surface roughness. Promotion of adhesion of SU8-2 on fused silica has been done by spin coating of Ti-primer at 3000 rpm for 40 seconds. The exposure, the development time, as well as the baking and the flood exposure time have been optimized in order to generate structures with a very good quality. This quality of the master mould, including its surface roughness, is essential in the imprinting process where all the patterns are imprinted in the desired polymer, including the defects. Figure 2b depicts the replication process started by spin coating of commercial Ormocore hybrid

polymer on a SiO₂ insulator on Si substrate. The 3 μm thermal oxide layer is thick enough in order to avoid light leakage towards the Si substrate. After the spinning and baking of theOrmopriime adhesion layer, Ormocore polymer film is then formed on the SiO₂ substrate again by a spin coating process. One can obtain Ormocore films with different film thickness by controlling the dilution of the concentration with a thinner (Ormothin /ma-T) and the spinning speed. From here in the imprinting process we have used films with a thickness of 0.8 μm and 0.5 μm respectively.

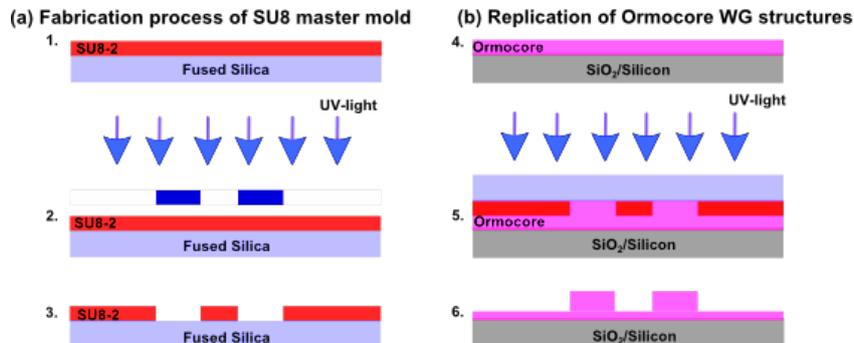


Figure 2. Fabrication process of microring resonators. (a) SU8 master mould has been prepared by photolithography steps 1 to 3. (b) Generation of Ormocore WGs by UV-NPL steps 4 to 6.

The SU8-2 patterned mould was then pressed against the Ormocore film on a Si substrate using a flip chip bonder by applying a force of 20 N. During the imprinting process, the Ormocore films were heated at 85°C for 10 minutes. Furthermore, around 3 minutes of UV illumination with an intensity of 30mW/cm² in a nitrogen atmosphere was applied to the bonded samples in order to cure the Ormocore layer. Finally, the replicated microrings were manually detached and were hardened by post-baking for 3 hours at 150°C.

3. CHARACTERIZATION

As already mentioned high Q factor microrings are desired for biosensing. Therefore there is a particular need to minimize losses in the fabrication process, a.o. by reducing the surface roughness and the dimensions of the residual layer. Figure 3 shows the SEM (scanning electron microscope) images of the fabricated SU8-2 master mould (top images) and of the replicated Ormocore microrings (bottom images). From the cross section in Fig. 3b, one sees that the SU8 generated pattern has vertical and smooth sidewalls.

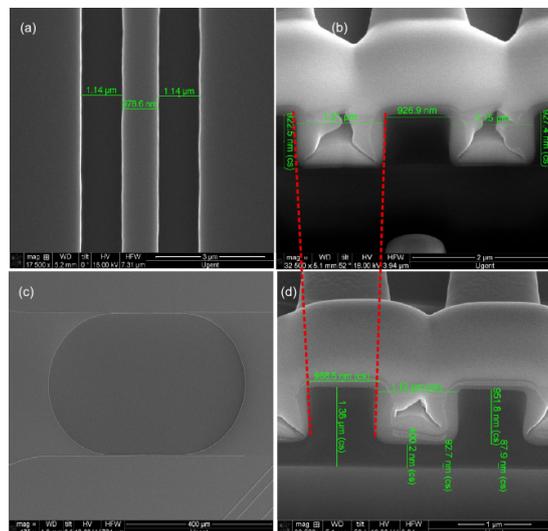


Figure 3. SEM images of the SU8-2 master mould: (a) top view and (b) cross section respectively. (c) SEM image of the imprinted Ormocore microring resonator and (d) a SEM cross-section in the gap region.

For only 2 seconds exposure time the gap in the SU8 patterns was around 927 nm, while the width and height of the microrings were 1.2 μm and 950 nm respectively. This pattern imprinted in the Ormocore films (with a thickness of 0.8 μm before imprinting) generated Ormocore microrings with a gap around 1.1 μm, for a waveguide width and height of 980 nm. In this case a residual layer of 0.4 μm was formed after imprinting. In order to decrease it, we have increased the exposure time for SU8 by 1 second only in a next step, in order to generate structures with a slightly larger width and therefore narrower gap. After imprinting in Ormocore (with an initial thickness of 0.5 μm before imprinting), microrings with a gap of 1 μm, and waveguide width and

height of 1.1 μm and 980 nm respectively were replicated. In this case the residual layer was about 0.2 μm . Figure 4a depicts a SEM image of the end facet of the Ormocore waveguide with vertical smooth sidewalls, which is beneficial to obtain low propagation loss.

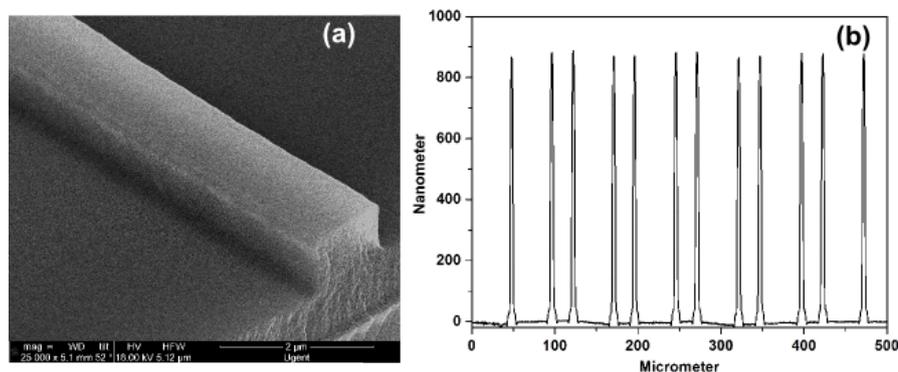


Figure 4: (a) SEM image of the end-facet of the Ormocore waveguide on SiO_2/Si substrate; (b) The height profile of Ormocore WGs measured with a Dektak profilometer.

We have generated large-scale imprinted areas with these structures. Figure 4b shows the height profile of 12 imprinted structures measured with a Dektak profilometer for a 500 μm scan area during 50 seconds. Considering the optical characterization of these devices, first measurements show positive results considering guiding the light and the transmission spectra of the rings with a Q factors around 12×10^4 and 0.35 nm FSR. However these studies are still under investigation in order to obtain improved results and will be the subject of a future contribution.

4. CONCLUSIONS

In summary we have fabricated centimetres large area of microrings with tailored dimensions around 1 μm width and height, 1 μm gap and controlled residual layer thickness down to 0.2 μm by UV nanoimprint lithography. They are targeted for operation at 850 nm and we expect higher quality factors and thus improved detection limit for biosensors.

ACKNOWLEDGEMENTS

The authors acknowledge the financial support of the FWO Grant G.0533.13N (2013-2015).

REFERENCES

- [1] H. Ma, A. K.-Y. Jen, and L. R. Dalton, "Polymer-based optical waveguides: Materials, processing, and devices," *Adv. Mater.* vol. 14, no.19, pp. 1339-1365, 2002.
- [2] S. Y. Chou, P. J. Krauss, and P. J. Renstrom, "Imprint of sub-25 nm vias and trenches in polymers," *Appl. Phys. Lett.*, vol. 67, no 21, pp. 3114-3119, Sep. 1995.
- [3] J. Hiltunen, M. Hiltunen, J. Puustinen, J. Lappalainen, and P. Karioja, "Fabrication of optical waveguides by imprinting: Usage of positive tone resist as a mould for UV curable polymer," *Opt. Express*, vol.17, no.25, pp. 22813-22822, Nov. 2009.
- [4] C.-Y. Chao, W. Fung, and L. J. Guo, "Polymer microring resonators for biochemical applications," *IEEE J. Sel. Topics Quantum Electron.*, vol.12, no.1, pp. 134-142, Jan./Feb., 2006.
- [5] J. Hiltunen, A. Kokkonen, J. Puustinen, M. Hiltunen, and J. Lappalainen, "UV-Imprinted single-mode waveguides with low loss at visible wavelength," *IEEE Photonics Technology Lett.*, vol. 25, no10, pp. 996-998, May 2013.
- [6] M. Hiltunen, J. Hiltunen, P. Stenberg, J. Petäjä, E. Heinonen, P. Vahimaa, and P. Karioja, "Polymeric slot waveguide at visible wavelength," *Opt. Letters*, vol.37, no. 21, pp. 4449-4451, Nov. 2012.
- [7] M. H. M. Salleh, A. Glidle, M. Sorel, J. Reboud, and J. M. Cooper, "Polymer dual ring resonators for label-free optical biosensing using microfluidics," *Chem. Commun.*, vol. 49, pp. 3095-3097, Jan. 2013.
- [8] K. D. Lee, S. W. Ahn, S. H. Kim, S. H. Lee, J. D. Park, P. W. Yoon, D. H. Kim, and S. S. Lee, "Nanoimprint technology for nano-structured optical devices," *Curr. Appl. Phys.*, vol. 6S1, pp. 149-153, 2006.
- [9] Ormocore Products, Micro Resist Technol. GmbH, Berlin, Germany 2014.