# Formation of PDMS films inside the holes of silica photonic crystal fibers

C. Markos [a, b], K. Vlachos [b], G. Kakarantzas [*a],
[a]Theoretical and Physical Chemistry Institute, National Hellenic Research Foundation, 48 Vas. Constantinou Ave., 11 635, Athens, Greece; [b] Computer Engineering and Informatics Dept., University of Patras, Building B, GR26500, Rio, Patra, Greece

## ABSTRACT

In this work, we demonstrate the formation of Poly-dimethylsiloxane (PDMS) films inside the holes of conventional silica photonic crystal fiber. The index guiding properties of the new PDMS-layer/Silica structures were investigated and optimized numerically using FDTD analysis. Films with thicknesses ranging from 100nm to 1μm were formed using different PDMS solution concentrations and different solution movement speeds. The thickness of films was very uniform along almost all the deposition length as indicated by Scanning Electron Microscopy (SEM) images and micro-Raman mapping.

**Keywords:** Photonic crystal fiber, hybrid fiber, PDMS film

## 1. INTRODUCTION

Photonic crystal fibers have many distinguished properties and characteristics compared to conventional single mode fibers[1]. This unique kind of optical fibers constitute of micrometer-scaled holes running along their entire length which usually are arranged in a hexagonal pattern[2]. This feature enables the infiltration of advanced materials and liquids such as liquid crystals[3, 4], high index liquids[5], biolayers[6, 7], ferrofluids[8], metals[9] as well as polymers[10, 11], into the air-holes of the PCF. Infiltration of the air-holes allows the modification the guiding properties so that tunable devices[12, 13]and various types of sensors[14] can be formed.

The PDMS elastomer is widely used in opto/microfluidics[15] and soft lithography. It owns very good optical properties such as high transparency over a wide range of wavelengths, lower refractive index (around 1.41) than fused silica and negligible birefringence. It also exhibits very good mechanical properties due to low Young's modulus; it is soft and deformable with no shrinkage, it bonds relatively well to glass and it can be dissolved to many organic solvents. The unique optical and mechanical properties of PDMS with the mature technology of PCFs can constitute an efficient route to development of compact cost-effectiveness tunable devices and sensors.

In this work, we demonstrate the formation of PDMS films inside the holes of a Hi-Bi silica photonic crystal fiber. The index guiding properties of the new PDMS-layer/Silica structures were investigated and optimized numerically using FDTD analysis. The details of PDMS film formation were established and the films were characterized using SEM imaging and micro-Raman 2D mapping.

## 2. EXPERIMENTAL

The Hi-Bi PCF PM-1550-01 from NKT Photonics was used. The fibre has a hole pitch of $\Lambda_P$ = 4.4 μm and hole diameter of $d_1$ = 2.2 μm for small holes and $d_2$ = 4.5 μm for the large holes. The outer diameter of the fibre is 125 μm. PDMS (Sylgard 184-Dow Corning) was prepared by mixing elastomer and curing agent at 10:1 ratio. We prepared solutions of different concentrations of PDMS in toluene. The PDMS/toluene solutions were inserted inside the holes of the PCF using capillary forces. Over pressure up to 10 bars was used for more viscous solutions with higher PDMS concentrations. In a variant of the well-known dip-coating process (see schematic diagram in Figure 1), the PDMS solutions were moved inside the PCF holes with the aid of a pressure gradient. Films with thicknesses ranging from 100nm up to 1μm were formed using different PDMS solution concentrations and different solution movement speeds.

*gkakaran@eie.gr;

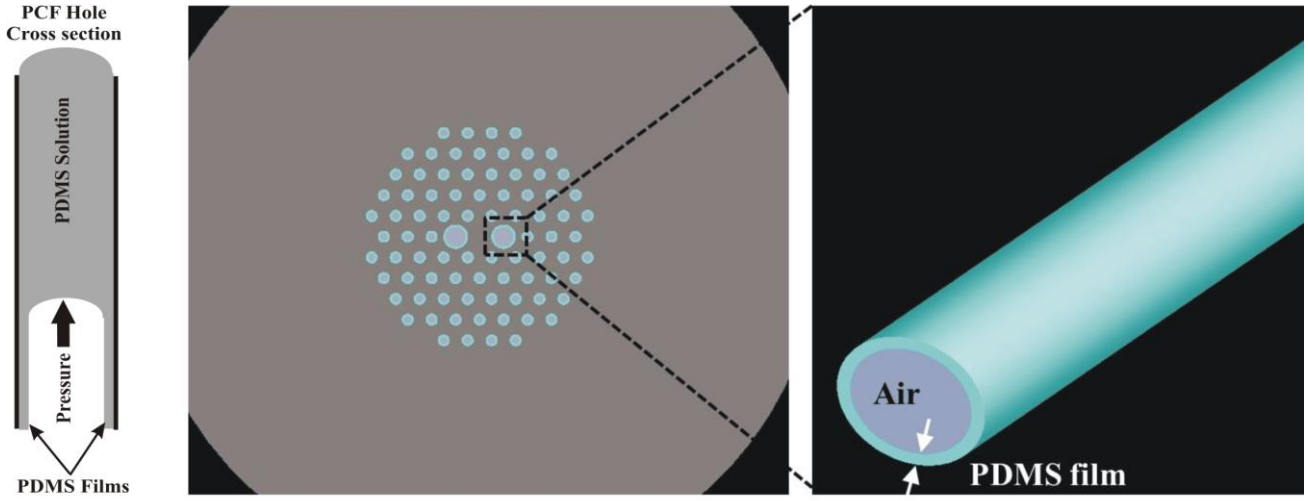


Figure 1. Schematic diagram of the method used for the PDMS film formation inside the holes of the PCF

## 3. RESULTS AND DISCUSSION

### 3.1 Simulation

The numerical investigation of both conventional and hybrid Hi-Bi PCF was done by employing a full vectorial finite difference method. The effective index of the fundamental guided mode was computed based on finite difference analysis using Yee's mesh and the index averaging technique utilizing perfectly matched layer (PML) boundary conditions[16] .To calculate the birefringence, in the hybrid PCF, a structure with the same structural parameter as the real fiber was created. In order to achieve accurate predictions in the numerical computation of the presented hybrid PDMS/Silica PCF cases, it is important to select the appropriate mesh spacing (grid size). With respect to the discretization scheme that was used in the calculation, reasonably accurate results can be obtained by keeping the mesh spacing at $\sim\lambda/15$ [17]. It should be mentioned that in this work we refer only to the phase birefringence. Dispersion of both materials (silica and PDMS) was included in our calculations using their Sellmeier equation[18]. The effective indices of both orthogonal polarizations (x and y polarization) of the fundamental mode is then calculated for a range of wavelengths from 1000 - 1700 nm. The birefringence B is obtained by taking the difference between the effective indices of the two different polarizations of the fundamental mode and from this the polarization beatlength for a wavelength λ, is defined as:

$$L_b = \frac{\lambda}{B} \tag{1}$$

The beatlengths of the Hi-Bi PCF with PDMS film thicknesses ranging from 100-500 nm together with the beatlengths of the PDMS free fiber were calculated using FTDT and they are shown in Figure 2. The presence of the PDMS films decreases the fiber's birefringence hence increasing the polarization beatlength. This is mainly due to lower mode confinement caused by the lower refractive index difference of silica and PDMS. The polarization beatlengths at λ=1550 nm are for different PDMS film thicknesses are shown in Table 1. The PDMS films extend the birefringence range over which the Hi-Bi fiber operates. Further tuning of the birefringence can be achieved thermally since the PDMS has a very high and linear thermo-optic coefficient ($-4.5\times10^{-4}$/°C) [13].

Table 1. Polarization beatlengths for different PDMS film thicknesses.

| Film Thickness (nm) | No PDMS | 100 | 200 | 300 | 400 | 500 |
|---|---|---|---|---|---|---|
| Beatlength (mm) | 2.85 | 3.40 | 4.10 | 5.02 | 6.25 | 8.03 |

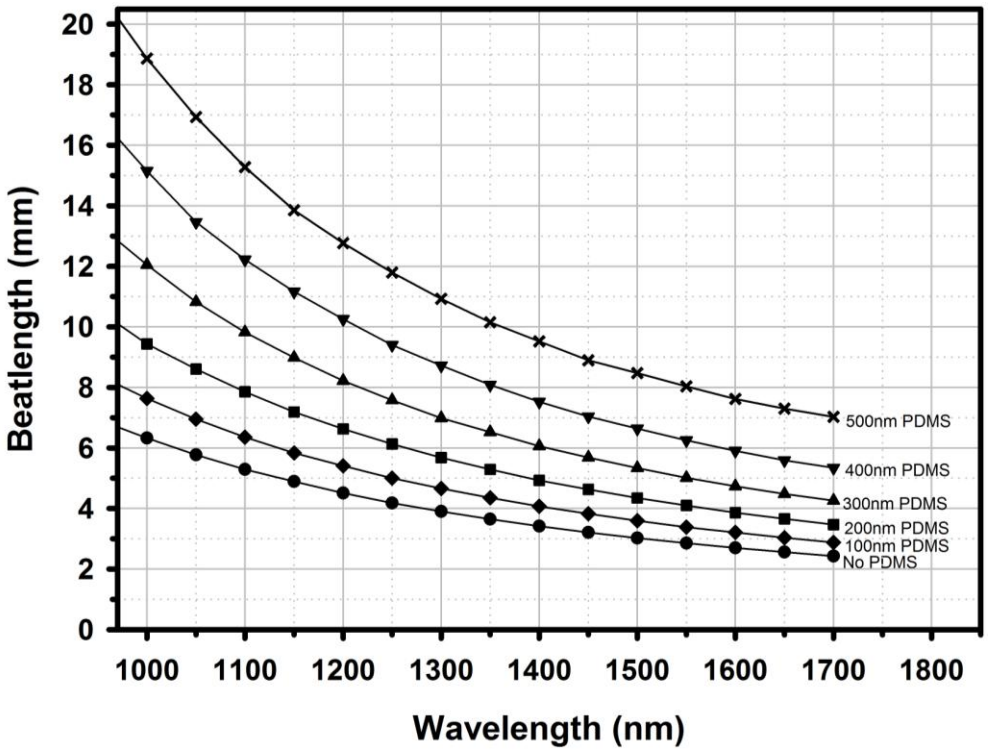


Figure 2. Beatlength vs wavelength for different PDMS film thicknesses (0-500nm) inside the holes of the PM-1550-01 fiber.

### 3.2 Experiment

In this section, we demonstrate the PDMS film formation inside the hole of a PCF. We prepared solutions of different concentrations of PDMS in toluene. Less dense (lower concentrations of PDMS in toluene) and faster moving solutions

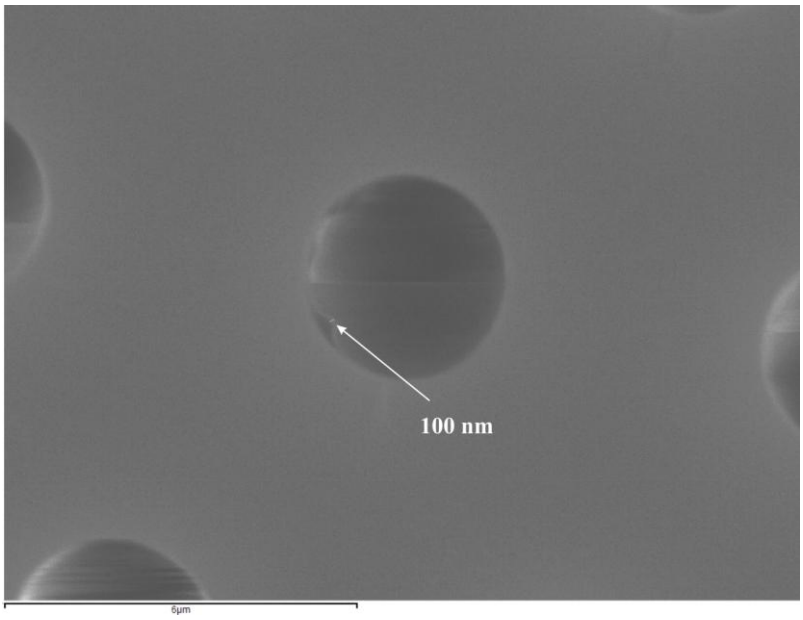


Figure 3. SEM photograph of 100 nm thick PDMS film inside the holes of a PCF.

were used for thinner film formation and thicker (with higher concentrations of PDMS in toluene) and slower moving solutions were used for thicker film formation. Figure 3 shows a SEM image of a 100 nm thick PDMS film inside a hole of a PCF. We used a PDMS/ Toluene solution at 1:1 ratio. A 15 cm long piece of PCF was carefully cleaved at both ends one of which was inserted inside the PDMS/ Toluene solution. The liquid was sucked inside the PCF holes over several cms with the aid of capillary forces. Then, the one end of the solution filled fiber was placed inside a pressure cell and the liquid was moved inside and out of the PCF holes by applying 10 bars air pressure.

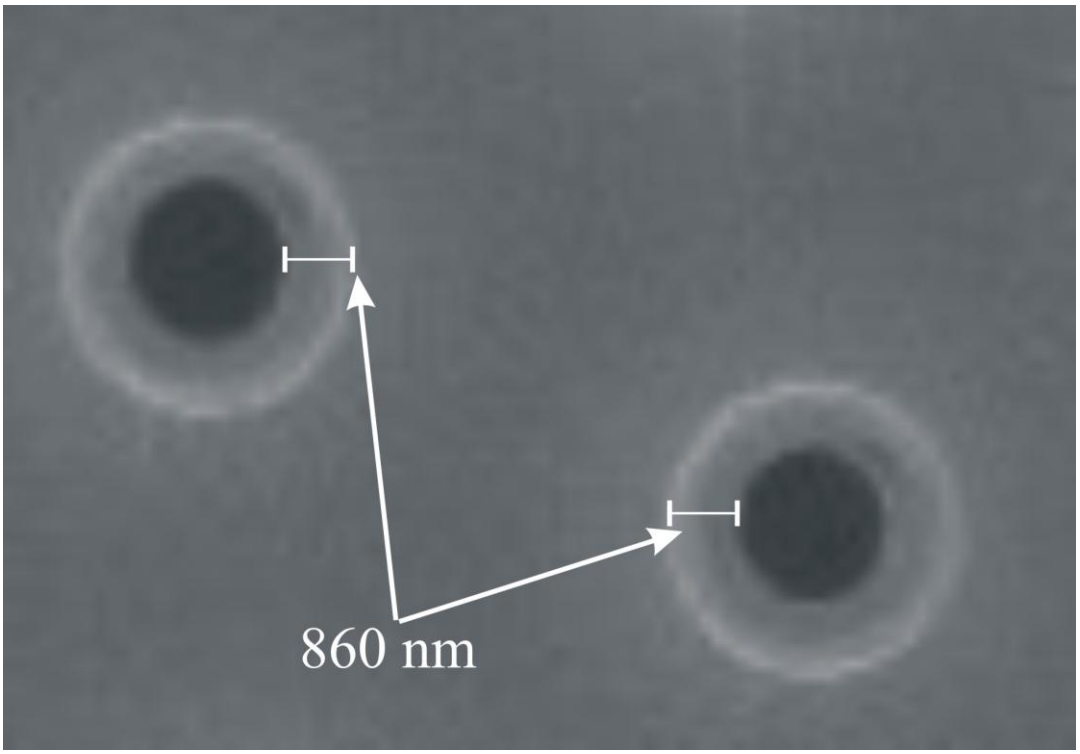


Figure 4. SEM photograph of 860 nm thick PDMS film inside the holes of a PCF.

Figure 4 shows a SEM image of a 860 nm thick PDMS film inside the holes of a PCF. We used a PDMS/ Toluene solution at 1:0.5 ratio which was more viscous than in the previous case of Figure 3. Over pressure of about 7 bars was used to place the liquid inside the holes. The liquid was moved inside and out of the PCF holes by applying 4 bars air pressure. The more viscous and slower moving PDMS/ Toluene solution formed thicker PDMS films. The PDMS film formation process will be quantified in the near future.

The mechanical properties of the silica glass and the PDMS elastomer are very different. Silica is a very hard glass and PDMS is a very soft and elastic polymeric material. As a result, the "explosive" process of cleaving the PCF may tear, wrinkle and even pull out the PDMS films formed inside the PCF holes, posing a difficulty in imaging the very thin films with SEM. Furthermore, the contrast between the silica glass and the PDMS elastomer is quite low for SEM imaging and the thin delicate PDMS films may also be too sensitive for the electron beam of the SEM. Alternatively, we employed the non destructive method of micro-Raman mapping for imaging the PDMS thin films. We used a Renishaw inVia Raman microscope with spatial scanning resolution of 100 nm. The laser beam of the Raman spectrometer at 488 nm was tightly focused through the objective of the microscope and scanned at a defined area around a PCF hole each time recording a Raman spectrum. We chose the very characteristic for PDMS and relatively strong Si-C symmetric stretch band at 711 $cm^{-1}$ to do the mapping. Figure 5 the spatial intensity distribution of the Si-C symmetric stretch band. The PDMS film cross section is deformed due to the fiber cleaving. Its thickness is estimated to be around 200 nm. The micro-Raman mapping is capable of giving PDMS film images in cases where no other imaging technique can be applied.

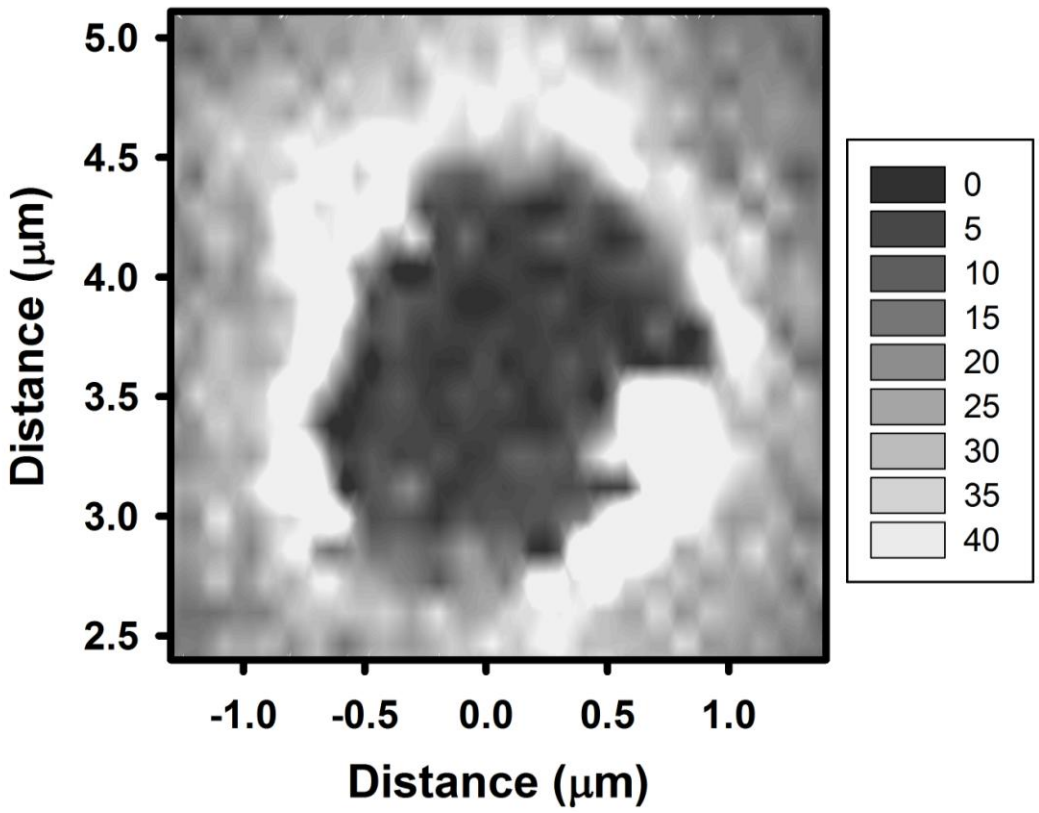


Figure 5. Micro-Raman mapping of PDMS films inside the holes of PCF.

## 4. CONCLUSIONS

We have presented results that report the formation of thin PDMS films inside the holes of a commercial Hi-Bi PCF. Films with thicknesses ranging from 100nm to 1μm were formed using different PDMS/toluene solution concentrations and different solution movement speeds. The index guiding properties of the PDMS-film/Silica structures were investigated and optimized numerically using FDTD analysis. The thickness of films was very uniform along almost all the deposition length as indicated by SEM images and micro-Raman mapping. The hybrid PDMS-layer/Silica structures retain to a large extend the advantages of both silica (regular structure, low loss) and polymer (bio-fictionalization, thermal tunability, ease of doping) PCFs, hence making them very attractive for bio-sensing and other applications.

## ACKNOWLEDGEMENTS

The authors acknowledge support from the Greek NSRF Program (MEDOUSA) with Grant No. 09SYN-24-769 and (SESAMO) of the European Defence Agency (EDA) with Grant No A-0931-RT-GC. The authors would like to thank Dr. D. Palles for his help with the micro-Raman mapping.

## REFERENCES


[1] Russell, P. St.J., "Photonic crystal fibers," Science 299, 358-362 (2003).

[2] Knight, J. C., Birks, T. A., Russell, P. St.J., Atkin, D. M., "All-silica single-mode fiber with photonic crystal cladding," Opt. Lett. 21, 1547-1549 (1996).

[3] Yuan, W., Wei, L., Alkeskjold, T. T., Bjarklev, A. and Bang, O., "Thermal tunability of photonic bandgaps in liquid crystal infiltrated microstructured polymer optical fibers," Opt. Express 17, 19356-19364 (2009).

[4] Zografopoulos, D. C., Kriezis, E. E. and Tsiboukis, T. D., "Tunable Highly Birefringent Bandgap-Guiding Liquid-Crystal Microstructured Fibers," J. Lightwave Technol. **24**, 3427–3432 (2006).

[5] Bise, R. T., Windeler, R. S., Kranz, K. S., Kerbage, C., Eggleton, B. J. and Trevor, D. J., "Tunable photonic band gap fiber," in Proc. Opt. Fiber Commun. Conf. (OFC), Anaheim, CA, pp. 466–468 (2002).

[6] Rindorf, L., Jensen, J. B., Dufva, M., Pedersen, L. H., Høiby, P. E. and Bang, O., "Photonic crystal fiber long-period gratings for biochemical sensing," Opt. Express **14**, 8224-8231 (2006).

[7] Markos, C., Yuan, W., Vlachos, K., Town, G. E. and Bang, O., "Label-free biosensing with high sensitivity in dual-core microstructured polymer optical fibers," Opt. Express 19, 7790-7798 (2011).

[8] Candiani, A., Konstantaki, M., Margulis, W. and Pissadakis, S., "A spectrally tunable microstructured optical fibre Bragg grating utilizing an infiltrated ferrofluid," Opt. Express 18, 24654-24660 (2010).

[9] Poulton, C. G., Schmidt, M. A., Pearce, G. J., Kakarantzas, G. and Russell, P. St.J., "Numerical study of guided modes in arrays of metallic nanowires," Opt. Lett. 32, 1647-1649 (2007).

[10] Westbrook, P. S., Eggleton, B. J., Windeler, R. S., Hale, A., Strasser, T. A. and Burdge, G. L., "Cladding-Mode Resonances in Hybrid Polymer-Silica Microstrucutred Optical Fiber Gratings," IEEE Photon. Technol. Lett. 12, 495–497 (2000).

[11] Markos, C., Vlachos, K. and Kakarantzas, G., "Bending loss and thermo-optic effect of a hybrid PDMS/silica photonic crystal fiber," Opt. Express 18, 24344-24351 (2010).

[12] Kerbage, C., Steinvurzel, P., Reyes, P., Westbrook, P. S., Windeler, R. S., Hale, A. and Eggleton, B. J., "Highly tunable birefringent microstructured optical fiber," Opt. Lett. 27, 842-844 (2002).

[13] Markos, C., Vlachos, K. and Kakarantzas, G., "Guiding and birefringent properties of a hybrid PDMS/Silica photonic crystal fiber" Proc. SPIE 7914, 791427 (2011).

[14] Qian, W., Zhao, C., He, S., Dong, X., Zhang, S., Zhang, Z., Jin, S., Guo, J. and Wei, H., "High-sensitivity temperature sensor based on an alcohol-filled photonic crystal fiber loop mirror," Opt. Lett. 36, 1548-1550 (2011).

[15] Fainman, Y., Lee, L. P., Psaltis, D. and Yang, C., [Optofluidics: Fundamentals, Devices, and Applications] McGraw-Hill, 7-28 (2010).

[16] Yu, C. P. and Chang, H. C., "Yee-mesh-based finite difference eigenmode solver with PML absorbing boundary conditions for optical waveguides and photonic crystal fibers," Opt. Express 12, 6165–6177 (2004).

[17] Zhu, Z. and Brown, T., "Full-vectorial finite-difference analysis of microstructured optical fibers," Opt. Express 10, 853–864 (2002).

[18] Schneider, F., Draheim, J., Kamberger, R. and Wallrabe, U., "Process and material properties of polydimethylsiloxane (PDMS) for Optical MEMS," Sens. Actuators A Phys. 151, 95–99 (2009).

[19] Cai, D., Neyer, A., Kuckuk, R., Heise, H.M., "Raman, mid-infrared, near-infrared and ultraviolet-visible spectroscopy pf PDMS silicone rubber for the characterization of polymer optical waveguide materials" J. Mol. Struct. 976, 274-81 (2010).