Liquid-filled microstructured polymer fibers as monolithic liquid-core array fibers

Jian Wang and Lili Wang*

State Key Laboratory of Transient Optics and Photonics, Xi'an Institute of Optics and Precision Mechanics, Chinese Academy of Sciences, Information Road 17, Xi'an, Shaanxi, 710119, China

*Corresponding author: liliwang@opt.ac.cn

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Liquid-filled microstructured polymer optical fibers (MPOFs) as monolithic liquid-core array fiber are proposed and prepared by injecting high-refractive-index liquid into the holes array of the MPOFs. One example for potential applications is demonstrated as a new kind of coherent imaging fiber. It provides great potential for applications in chemical sensing, biosensors, and endoscopy, particularly in bifunctional detection. © 2009 Optical Society of America

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1. Introduction

A number of liquid-core fibers were developed, and many research results about liquid-core fibers were reported, demonstrating great advantages in fields such as temperature sensor, Raman spectroscopy, fluorescence applications, and transmitting UV light for curing and industrial or medical purposes [1–7]. For example, a liquid-core optical fiber (LCOF) sensor based on the intensity-modulated principle as a temperature sensor had a high sensitivity in accurate temperature measurements and a tunable operational range by simply changing the concentration of the two liquid components [4]. Intensification factors of Raman spectroscopy in a long LCOF was 1000–3000 times greater than conventional sampling arrangements in spontaneous Raman spectroscopy, and the detection limit was reduced by 1000 for an aqueous absorbing sample by using a LCOF Raman cell made from Teflon AF 2400 [3]. Although LCOF has not been used in communications, people have been trying to reducing the loss of liquid fiber. Altkorn *et al.* fabricated a LCOF based on tubing made entirely of Teflon AF 2400, which could transmit visible light with low loss when filled with low refractive-index liquids such as water, methanol, and ethanol [1]. Recently, liquid-filled microstructured optical fibers (MOFs) have received intense attention due to the wide range of potential applications in various fields: on one hand, applications related to the active field of chemical and biological sensing of liquids using infiltrated MOFs, and on the other hand, changing cladding refractive index associated with the inserting liquid material into the microstructure of a solid-core MOF. Vienne *et al.* proposed a liquid fiber in which the hollow core of a microstructured fiber was filled with water, where visible light was efficiently guided [8]. Matos *et al.* demonstrated an experimental method for simultaneously and selectively filling the core and the cladding of a hollow-core photonic crystal fiber with different liquids and theoretically studied the coexistence of bandgap and index-guiding modes in such fibers [9]. While liquid-core fiber applications employing a single optical fiber have been applied to a wide variety of analytical determinations, they cannot be used for imaging.

If different liquids are filled in original two-dimensional (2-D) photonic crystal fiber, there will be two new types optical fibers: a monolithic liquid-core array, solid-cladding fiber and a monolithic solid-core array, liquid-cladding fiber. We are interested in what would happen when some liquids

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with high refractive index are injected into the 2-D photonic crystal fiber. The liquid-filled 2-D photonic crystal fiber with monolithic liquid-core array fibers (MLCAFs) may expand the liquid-core fibers' application domain to image transmission, parallel optical links, and optical splitting devices.

Imaging fiber bundles developed as flexible image carriers have important applications in human endoscopy, imaging optical interconnects, and sensing [10–16]. Microstructured polymer optical fibers (MPOFs) as a new type of imaging fiber were developed by van Eijkelenborg in 2004, with coherent imaging achieved either by guiding light in polymer cores between air holes or by guiding light in the air channels themselves [17]. With the further development of MPOFs, we believe that the MLCAF will make it of considerable value in image transmission. To the best of our knowledge, it has not yet been reported that the liquid-core fiber has the application of image transmission.

We present a highly ordered MLCAF consisting of 547 cores and/or 525 cores of high-refractive-index liquid (triphenyl phosphite and/or hexachloro-1, 3-butadiene) in a matrix of low-refractive-index cladding (poly[methyl methacrylate] [PMMA]). As an initial demonstration of the use of MLCAF, its imaging capability is observed. This kind of fiber is flexible, simple, light weight, and low cost, and high numerical aperture (NA) is expected from the proposed design, which can yield high collection efficiency and wide-field imaging. Potentially such liquid-core fiber could find useful applications in optical interaction, endoscopy, chemical sensors, and biosensors, particularly attractive for fabricating bifunctional fiber that has imaging and sensing capabilities.

2. Fabrication of Monolithic Liquid-Core Array Fiber

MPOFs with a 547-hole hexagonal array and a 525-hole rectangular array used in this study are fabricated by extrusion and draw technique. The optical grade PMMA pellets are extruded into specialized high-quality metal molds, in which preforms are made with a diameter of 70 mm, an air-hole diameter of 1.5 mm, and hole spacing of 1.9 mm. It is large enough to produce more than one hundred kilometer fibers with a diameter of 150 μm as previously reported [18]. The diameter of the resultant MPOF is mainly controlled by changing drawing speed and temperature. Scanning electron microscope photographs of the cross section of the MPOFs are shown in Fig. 1. It can be seen that the structure

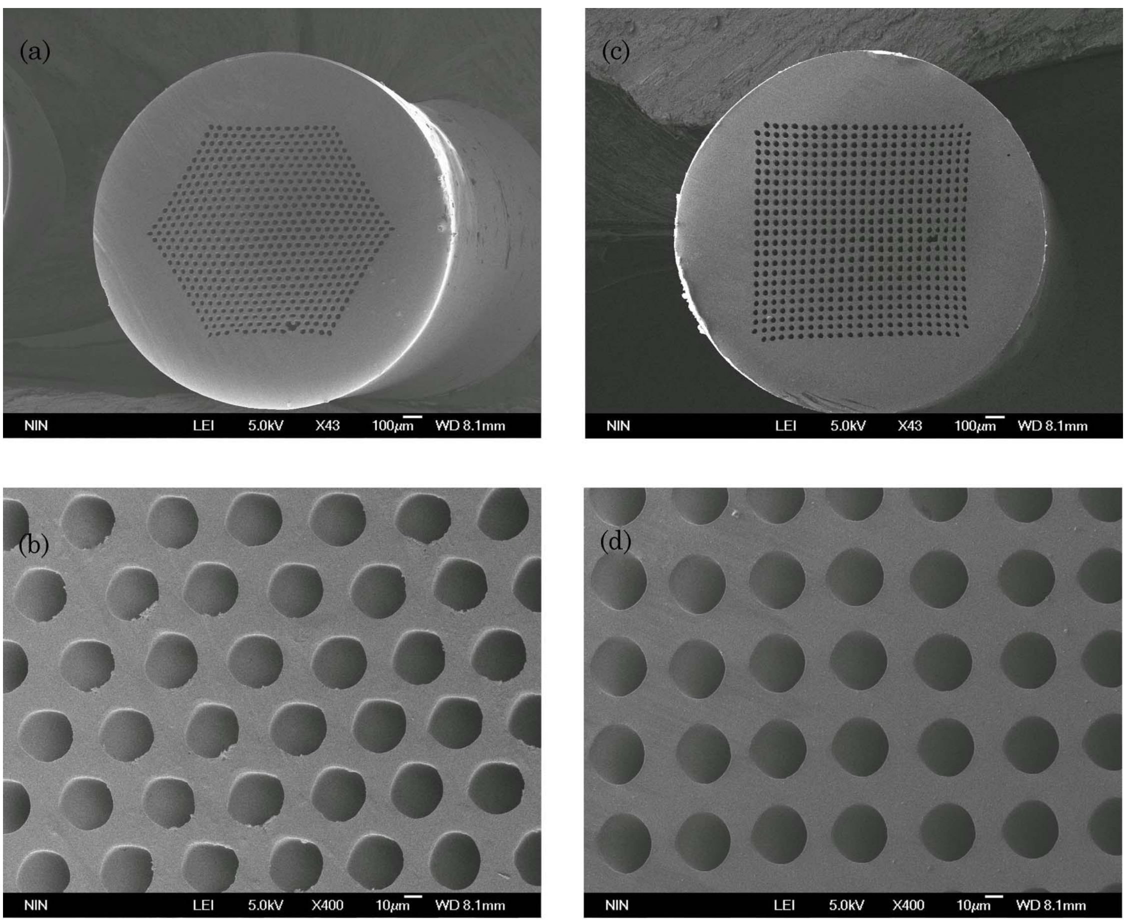
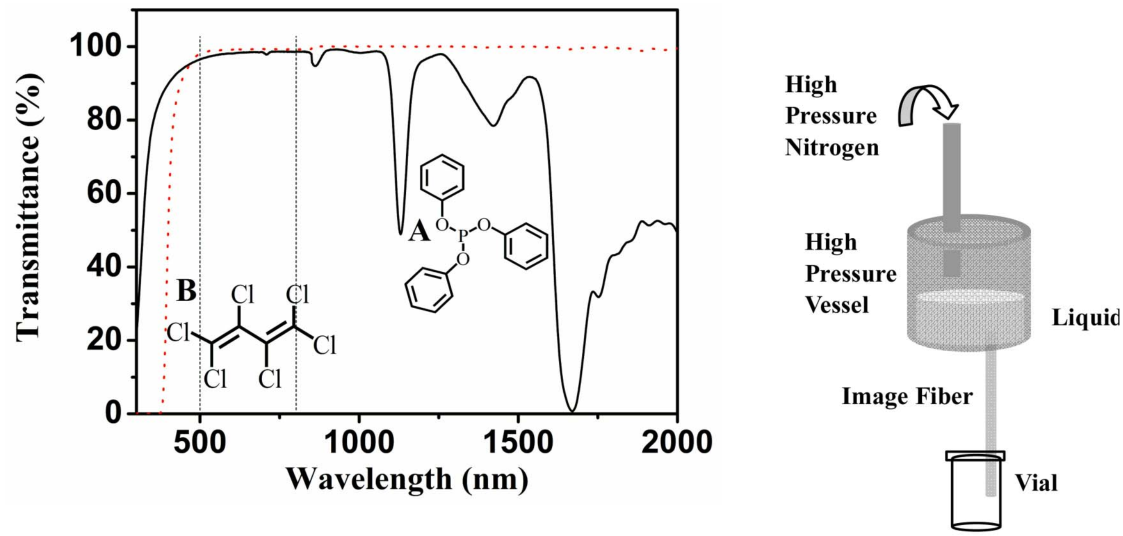


Fig. 1. Scanning electron micrograph of the cleaved end faces of MPOFs. (a) and (b) MPOF with a 547-hole hexagonal array in different magnifications. (c) and (d) MPOF with a 525-hole rectangular array in different magnifications.



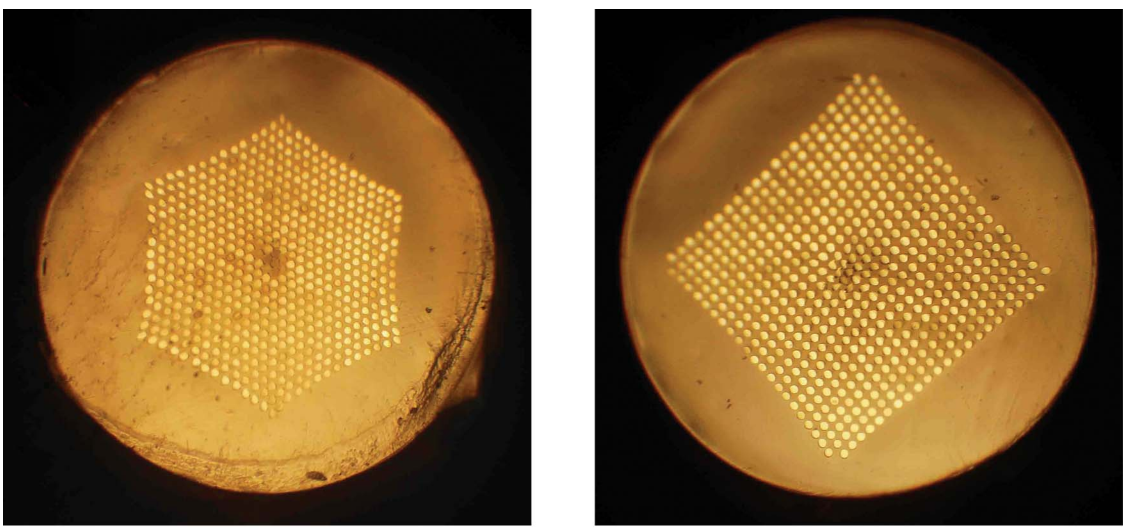
(a) (b)

Fig. 2. (Color online) (a) Transmittance of triphenyl phosphite (solid curve) and hexachloro-1, 3-butadiene (dotted curve). (b) Schematic of injecting liquid into the hole array of a fiber.

of the MPOFs and the shape of the holes have little deformations. Figures 1(a) and 1(b) are the images of MPOFs with a 547-hole hexagonal array and an outer diameter of 1.9 mm. Figures 1(c) and 1(d) are the images of the MPOFs with a 525-hole rectangular array and an outer diameter of 2.0 mm.

Because the refractive index of the PMMA is 1.49, it is essential that the liquid chosen as the core of MLCAF have a higher index than that of the PMMA to achieve a guiding structure. The imaging fiber requires a relatively high NA in order to capture as high a percentage as possible of the optical energy available from the image and reduce the cladding contribution to the fiber attenuation. In this study, two kinds of liquid (triphenyl phosphite and hexachloro-1, 3-butadiene) are chosen as the liquid-core material, having high boiling points of 360 °C and 212 °C, which is beneficial to retain the liquid in the holes of the fiber [19]. The refractive indexes of triphenyl phosphite and hexachloro-1, 3-butadiene are 1.59 and 1.56, which are much higher than that of PMMA (1.49). NA $[(n_{\mathrm{CORE}}{}^2 - n_{\mathrm{CLAD}}{}^2)^{0.5}]$ of these two kinds of liquid-filled MPOFs is evaluated to be equal to 0.55 and 0.46, respectively.

Both the liquid triphenyl phosphite and the hexachloro-1, 3-butadiene were normal commercial analytical reagent and were used directly from the bottle. Figure 2(a) is the transmittance of them



(a) (b)

Fig. 3. (Color online) Photos of MLCAFs illuminated on the distal end. (a) Hexagonal array with 547 liquid cores. (b) Rectangular array with 525 liquid cores.

measured by a UV/Vis spectrophotometer. In the region of 500–800 nm, the transmittance of triphenyl phosphite and hexachloro-1, 3-butadiene in a 1 cm cell is higher than 96% and 98%, respectively. In particular, hexachloro-1, 3-butadiene has high transmittance in the region of 500–2000 nm. It can be used as liquid-core material of visible and near-infrared imaging fiber.

The liquid was injected into the hole array of the MPOF by attaching one end of the fiber to 5 mL of triphenyl phosphite in a vessel under the pressure of 0.4 MPa nitrogen and placing the other end in a vial to load effluent liquid as shown in Fig. 2(b). When the liquid flowed out one end of the fiber, it demonstrated that some air channels had been fully filled with liquid. It needed another moment to ensure that all the air channels were fully occupied by liquid. The length of a 0.5 m fiber with an approximate diameter of 2.0 mm could be filled of triphenyl phosphite in 3 min and hexachloro-1, 3-butadiene in 1 min. The volume of the liquid held by the filled fiber was of the order of 1 mL. After the hollow-core array was fully filled with liquid, the end faces of the fiber were sealed by transparent film to avoid liquid evaporation, which had little effect on the image transmission. The interfaces of film and fiber were bound together with PMMA dissolved by acetone. The MLCAF was examined under a microscope to ensure no air bubbles were present, which could cause loss or completely suppress image transmission. By illuminating the distal end of the fiber, photos of the MLCAF with an approximately 15 cm length obtained by CCD camera through 4× microscope objective are shown in Fig. 3. It can be seen that light clearly transmits through liquid cores but not solid cladding.

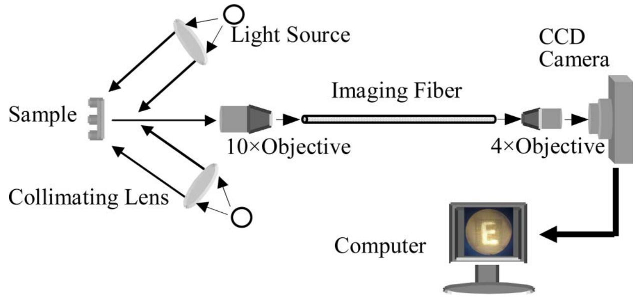
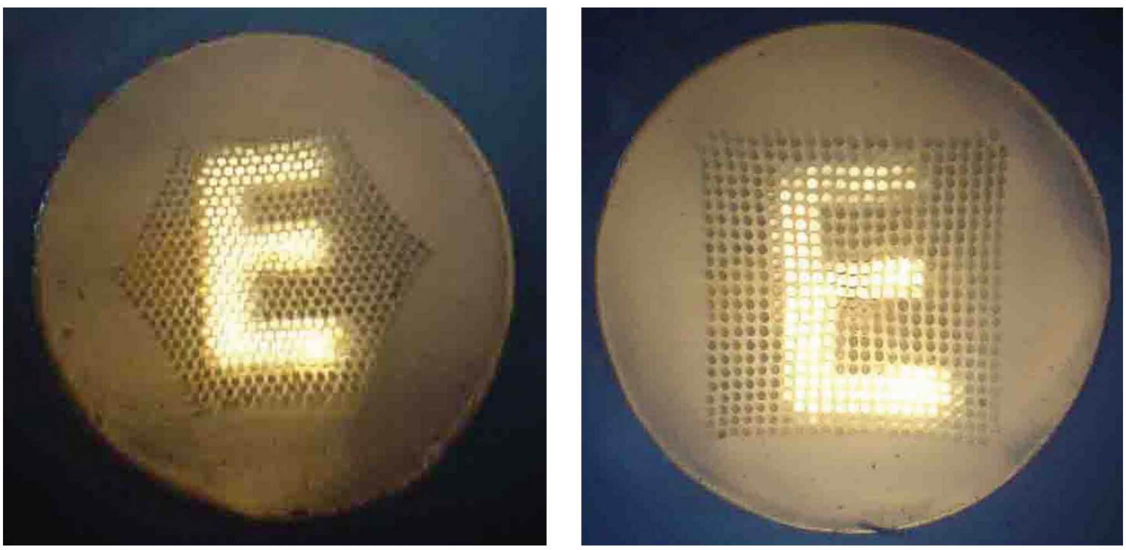


Fig. 4. (Color online) Schematic of the MLCAF imaging system.

3. Application of Monolithic Liquid-Core Array Fibers for Image Transmission

A homemade specialized assay system is shown in Fig. 4, which was used to observe fibers' imaging capabilities. The imaging system consists of two light sources, two collimating lenses, a MLCAF, microscope objective lenses, and a CCD camera. A thin aluminum screen with an "E" shape cut out as a sample is placed in front of light sources, and collimating lenses are placed between them, directing light from the light sources. This screen is imaged onto the left end face of the MLCAF (Fig. 4) through a 10× objective, which forms the image inverted and reduced in size by controlling the distance between the screen "E" and the 10× objective. By illuminating the aluminum screen "E" shape, the image is transmitted by the fiber and obtained by the CCD camera with a 4× microscope objective, which makes the image bigger in size.

Figure 5 shows two photos of image transmission by using two kinds of MLCAF. Figure 5(a) is obtained from a 547-pixel MLCAF that has an outer diameter



(a) (b)

Fig. 5. (Color online) CCD camera images of the "E"-shaped aluminum screen transmitted by (a) a MPOF with a hexagonal array of 547 liquid cores and (b) a MPOF with a rectangular array of 525 liquid cores, respectively.

of 1.9 mm, a 14.5 cm length, and a pixel diameter of approximately 30 μm [Fig. 1(b)]. Figure 5(b) is obtained from a 525-pixel MLCAF with an outer diameter of 2.0 mm, a 15.2 cm length, and a pixel diameter of approximately 32 μm [Fig. 1(d)]. As shown in Fig. 5, the structure of the liquid-core array is a spatially ordered arrangement, and the image is clearly transmitted in a coherent way, with the liquid cores guiding light by total internal reflection. The slight irregularities in the transmission pattern and some defects originate from the imperfections of the razor-blade fiber-cutting method. The results demonstrate that this new type of MLCAF has a great ability to transmit images. It is proven by experiment that these fibers with an approximate diameter of 2 mm could transmit images of more than 60 cm. The image is maintained down to a bending radius of approximately 1 cm, beyond which the overall transmission losses become significantly higher.

Conventional liquid-core fibers have applications in Raman spectroscopy and fluorescence spectroscopy, which are well-known. With the addition of the function of image transmission, the MLCAF is potentially developed as a bifunctional fiber for image transmission and chemical and/or biosensors. Moreover, a MLCAF's Raman spectroscopy intensity may be several dozen to several hundred times greater than a conventional single liquid-core fiber, because an MLCAF is equal to bundling hundreds of single liquid-core fibers. In many clinical situations, this fiber may provide diagnostic information. If the cladding of the MLCAF is made of Teflon AF 2400 ($n = 1.29$), virtually all the liquid could be used as the core material. Though it still has higher loss than would be expected for a perfect fiber, it proved that the array of the liquid-core fiber would be fabricated and has applications in image transmission. In addition, it has a larger emergence angle than that of MPOFs.

4. Conclusions

In summary, MLCAFs with 547 liquid cores and 525 liquid cores were successfully fabricated, and the imaging capabilities were demonstrated with clear image transmission and small bending radius. The image transmits through liquid-core arrays but not solid cores. This new kind of imaging fiber has advantages such as flexibility, light weight, low cost, and high NA. We believe that MLCAFs will play an important role in diverse spectroscopic applications, especially in bifunctional detection combined with image transmission and chemical or biochemical sensing.

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References

1. R. Altkorn, I. Koev, R. P. Van Duyne, and M. Litorja, "Low-loss liquid-core optical fiber for low-refractive-index liquids: fabrication, characterization, and application in Raman spectroscopy," Appl. Opt. **36**, 8992–8998 (1997).
2. G. E. Walrafen and J. Stone, "Intensification of spontaneous Raman spectra by use of liquid core optical fibers," Appl. Spectrosc. **26**, 585–589 (1972).
3. Y. J. Tian, L. Y. Zhang, J. Zuo, Z. W. Li, S. Q. Gao, and G. H. Lu, "Raman sensitivity enhancement for aqueous absorbing sample using Teflon-AF 2400 liquid core optical fibre cell," Anal. Chim. Acta **581**, 154–158 (2007).
4. Y. H. Xu, X. F. Chen, and Y. Zhu, "High sensitive temperature sensor using a liquid-core optical fiber with small refractive index difference between core and cladding materials," Sensors **8**, 1872–1878 (2008).
5. S. Yiou, P. Delaye, A. Rouvie, J. Chinaud, R. Frey, G. Roosen, P. Viale, S. Février, P. Roy, J.-L. Auguste, and J.-M. Blondy, "Stimulated Raman scattering in an ethanol core microstructured optical fiber," Opt. Express **13**, 4786–4791 (2005).
6. D. Qi and A. J. Berger, "Chemical concentration measurement in blood serum and urine samples using liquid-core optical fiber Raman spectroscopy," Appl. Opt. **46**, 1726–1734 (2007).
7. J. Y. Ye and M. Ishikawa, "Enhancing fluorescence detection with a photonic crystal structure in a total-internal-reflection configuration," Opt. Lett. **33**, 1729–1731 (2008).
8. G. Vienne, M. Yan, T. Luo, T. K. Liang, P. Ho, and C. Lin, "Liquid core fibers based on hollow core microstructured fibers," in *Proceedings of IEEE Conference on Lasers and Electro-Optics, Pacific Rim* (IEEE, 2005), pp. 551–552.
9. C. J. De Matos, C. M. B. Cordeiro, E. M. dos Santos, J. S. Ong, A. Bozolan, and C. H. B. Cruz, "Liquid-core, liquid-cladding photonic crystal fibers," Opt. Express **15**, 11207–11212 (2007).
10. T. J. Muldoon, M. C. Pierce, D. L. Nida, M. D. Williams, A. Gillenwater, and R. Richards-Kortum, "Subcellular-resolution molecular imaging within living tissue by fiber microendoscopy," Opt. Express **15**, 16413–16423 (2007).
11. K. S. Bronk, K. L. Michael, P. Pantano, and D. R. Walt, "Combined imaging and chemical sensing using a single optical imaging fiber," Anal. Chem. **67**, 2750–2757 (1995).
12. Y. Li, T. Wang, H. Kosaka, S. Kawai, and K. Kasahara, "Fiber-image-guide-based bit-parallel optical interconnects," Appl. Opt. **35**, 6920–6933 (1996).
13. T. Maj, A. G. Kirk, D. V. Plant, J. F. Ahadian, C. G. Fonstad, K. L. Lear, K. Tatah, M. S. Robinson, and J. A. Trezza, "Interconnection of a two-dimensional array of vertical cavity surface emitting lasers to a receiver array via a fiber image guide," Appl. Opt. **39**, 683–689 (2000).
14. C. Amatore, A. Chovin, P. Garrigue, L. Servant, N. Sojic, S. Szunerits, and L. Thouin, "Remote fluorescence imaging of dynamic concentration profiles with micrometer resolution using a coherent optical fiber bundle," Anal. Chem. **76**, 7202–7210 (2004).
15. M. E. Bosch, A. J. R. Sánchez , F. S. Rojas, and C. B. Ojeda, "Recent development in optical fiber biosensors," Sensors **7**, 797–859 (2007).
16. J. Ai and Y. Li, "Polymer fiber-image-guide-based embedded optical circuit board," Appl. Opt. **38**, 325–332 (1999).
17. M. A. van Eijkelenborg, "Imaging with microstructured polymer fibre," Opt. Express **12**, 342–346 (2004).
18. J. Wang, X. H. Yang, and L. L. Wang, "Fabrication and experimental observation of monolithic multi-air-core fiber array for image transmission," Opt. Express **16**, 7703–7708 (2008).
19. D. N. Payne and W. A. Gambling, "New low-loss liquid-core fiber waveguide," Electron. Lett. **8**, 374–376 (1972).