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Abstract. This paper presents a simple fabrication method for a liquid-core optofluidic waveguide and demonstrates efficient light delivery and collection using optical fibers pressure-sealed into a polydimethylsiloxane matrix. Total optical loss as small as 3 dB from an input optical fiber with a core diameter of 9 μm to an output optical fiber with a core diameter of 62.5 μm connected one to another via a 2 cm long liquid-core optofluidic channel is achieved. This strategy allows rapid prototyping of optofluidic waveguides without requiring expensive microfabrication facilities and is suitable for applications that require an efficient optical sensing in a small liquid sample. As a demonstration, we examine the optical Faraday rotation of a fluid sample and identify advantages and limitations by comparison to a macroscopic system of similar length. © 2013 Society of Photo-Optical Instrumentation Engineers (SPIE) [DOI: [10.1117/1.OE.52.4.044404](https://doi.org/10.1117/1.OE.52.4.044404)]

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1 Introduction

A major trend in analytical science and engineering is toward miniaturization. Entire analytical measurement systems, such as mass spectrometers and DNA analysis systems, are shrinking to shoebox-sized or handheld devices. The revolution in “lab-on-a-chip” technologies is exemplified by the field of optofluidics. Optofluidics employs chip-based manufacturing methods to provide functional systems with samplers, valves, mixers, waveguides, lenses, and detectors integrated onto a chip for a broad range of applications. Notably, many of these applications require efficient optical coupling between the optical signal and the fluidic channel, which is why most demonstrations to date have relied on bulk optics where losses can be more easily mitigated. An alternate strategy, however, is using fiber optic coupling, which features various advantages including compactness, flexibility, and stability. Unfortunately, guiding light in and out of a microfluidic channel while preserving the functionality of a chip-integrated device still proves far from trivial. For example, Leistiko and Jensen¹ used a silicon V-groove to align an optical fiber into a planar waveguide through a beam splitter across a microfluidic channel but this approach finds limitations when it comes to properly sealing the microfluidic channels. Conversely, Chabinyt et al.² introduced a technique where the optical fiber is aligned to a master channel and a polymer is poured and cured to secure its position. This strategy solves the sealing problem but accurate alignment during the curing process is difficult. In an alternate approach, Camou et al.³ focused the beam into and out of the channel using chip-integrated polymeric optical lenses, but lens fabrication is relatively complex and the resulting

optical coupling is highly sensitive to the lens properties. Several others demonstrate the use of optical fibers to couple light into microfluidic channels.^{4–7} Most of them, however, still rely on bulk optics to collect light and optical fiber detection still suffers from low collection efficiency.

Here, we present a simple fabrication method of a liquid-core optofluidic waveguide and its efficient optical coupling to optical fibers to analyze liquids and liquid solutions. Our structure is based on polydimethylsiloxane (PDMS), a polymeric material of widespread use in microfluidic devices and suitable for a rapid mold fabrication process without requiring complex microfabrication facilities. Further, PDMS is simultaneously robust, flexible, transparent, and permeable, which makes it ideal for bio-related applications such as cell culturing.⁸ Using PDMS to fabricate a liquid-core waveguide results in a flexible system that can adjust its shape to fit the optical fiber. However, the inner porous network structure of PDMS makes it highly permeable compared to other polymeric materials. Diffusion of liquid vapor through PDMS has been reported,⁹ which leads to unwanted drying and convective flow within the sample volumes if there is no continuous sample liquid supply.¹⁰ Our work shows that this problem can be circumvented via the use of a glass barrier tube packaging. This approach allows us to produce stable, efficiently-coupled liquid-core waveguides up to 2 cm long and only ~ 80 μm diameter, a geometry suitable for measurements that simultaneously require a moderately small sample volume and a long optical path (such as high-throughput optical absorption) without continuous liquid sample supply. Also, we show that our design is compatible with polarization-sensitive observations, a feature we discuss through optical Faraday rotation (OFR) experiments in a fluid sample.

2 Design and Fabrication

Our approach to fabricate a liquid-core waveguide microfluidic channel is schematically shown in Fig. 1. We start by combining a commercial PDMS resin (SYLGARD 184, Dow Corning, Midland, Michigan) with its accompanying crosslink initiator in a 10:1 weight ratio. We then inject this PDMS prepolymer—still in fluid form—into a hollow glass barrier tube (inner diameter of ~ 3 mm), which, as we describe below, will serve as the system outer jacket. To form a microfluidic channel mold, we use a bare, 40 gauge ($\sim 78 \mu\text{m}$) Nickel-Chromium wire concentric with the glass barrier tube. As shown in Fig. 1, small plastic jackets (outer diameter of $\sim 900 \mu\text{m}$ with inner diameter of $\sim 125 \mu\text{m}$) positioned to coincide with the ends of the glass barrier tube are used to create the molds of two fiber ports. We apply a drop of photoresist on the inner end of each jacket to produce a smooth, tapered structure, which we then bake at 100°C for 30 min in an oven. After the PDMS is fully cured, we first remove the plastic jackets and pull the wire out of the PDMS matrix. The completed microfluidic channel is rinsed with acetone and isopropanol to remove any photoresist residue. We note that the chosen curing conditions have an impact on the chemical cross-linking reaction and, correspondingly, on the mechanical and optical properties of the resulting structure. As a matter of fact, the refractive index (n_{cl}) of PDMS typically increases with baking time and temperature; for the present case we have $n_{cl} = 1.445$ at 532 nm based on the interpolated data.¹¹ To deliver light into the microfluidic channel, we use a single-mode optical fiber (9 μm diameter core, 125 μm diameter clad); light collection was carried out with a multimode fiber (62.5 μm diameter core, 125 μm diameter clad). We will show below that this selection allows us to attain substantial light collection efficiency. The presented fabrication process can be modified to include liquid in and out ports for continuous liquid circulation. In this case, the glass barrier tube is not necessary to prevent liquid evaporation. As a mold for liquid in and out ports, we use extra wires in contact with the liquid waveguide core. Upon following a process similar to that described before, the wires are pulled after the PDMS is fully cured. Figure 1(d) shows an example of a liquid-core optofluidic waveguide aligned with input and output fibers and liquid in and out ports without a glass barrier tube.

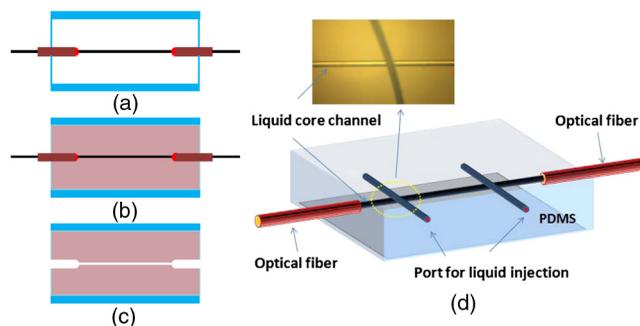


Fig. 1 Process flow-chart of the optofluidic waveguide fabrication. (a) Preparing a hollow tube with metal wire hanging in the center. (b) Injecting the liquid polydimethylsiloxane (PDMS) prepolymer into the tube and baking properly. (c) Pulling out the wire and the jackets. (d) Optofluidic waveguide structure with liquid in and out ports.

3 Results and Discussion

In this experiment, we examine Dimethyl Sulfoxide (DMSO) as the liquid core, with a refractive index of 1.479. This allows total internal reflection at the liquid core and PDMS cladding interface. The present PDMS optofluidic waveguide was tested on an optical table whereby optical fibers with 125 μm in diameter were prealigned and coupled with the optofluidic waveguide by means of fiber-holders mounted on manipulators through a microscope. Final alignment of the optical fibers with the optofluidic waveguide channel takes place automatically upon insertion into the interface section due to the tapered structure [Fig. 1(c)]. As shown in the microscope image of Fig. 2(a), the resulting structure shows a symmetric and smooth transition into the microfluidic channel section of the device. Also, due to the high elasticity of PDMS, this structure guarantees good sealing of the sample inside the channel.

3.1 Characterization of Liquid-Core Waveguides and Optical Fiber Coupling Efficiency

To test the device performance, we connect the input and output fibers to a light source (1 mW at 532 nm) and a power meter (Newport Model 1930C) as shown in Fig. 2(b). Two different optofluidic channels are prepared with and without a glass barrier tube and their optical transmission characteristics are measured. Figure 3 compares the measured transmission loss of a 2 cm long liquid-core waveguide (as measured from its joints with the 0.5 mm long input and output fibers) with and without a glass barrier tube. As shown in Fig. 3(a), without the glass barrier tube, the total transmission loss gradually increases. We also observe that the fluidic channel diameter decreases without creating any air bubbles in the channel as time passes. After 4 h, the channel is completely closed and the sample liquid has completely evaporated. Taking the fiber out from the PDMS matrix brings the channel back to its original shape, which leads us to hypothesize that rather than PDMS swelling, fluid

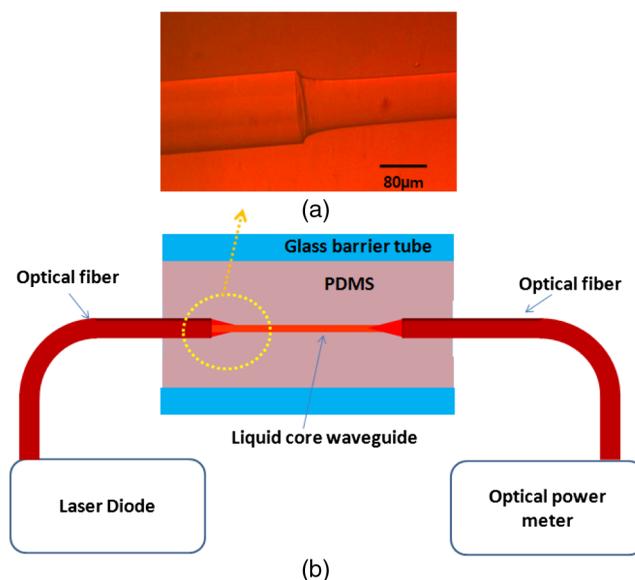


Fig. 2 (a) A microscope image of an optical fiber and optofluidic waveguide interface. (b) Schematic diagram for measuring optofluidic waveguide loss and coupling efficiency.

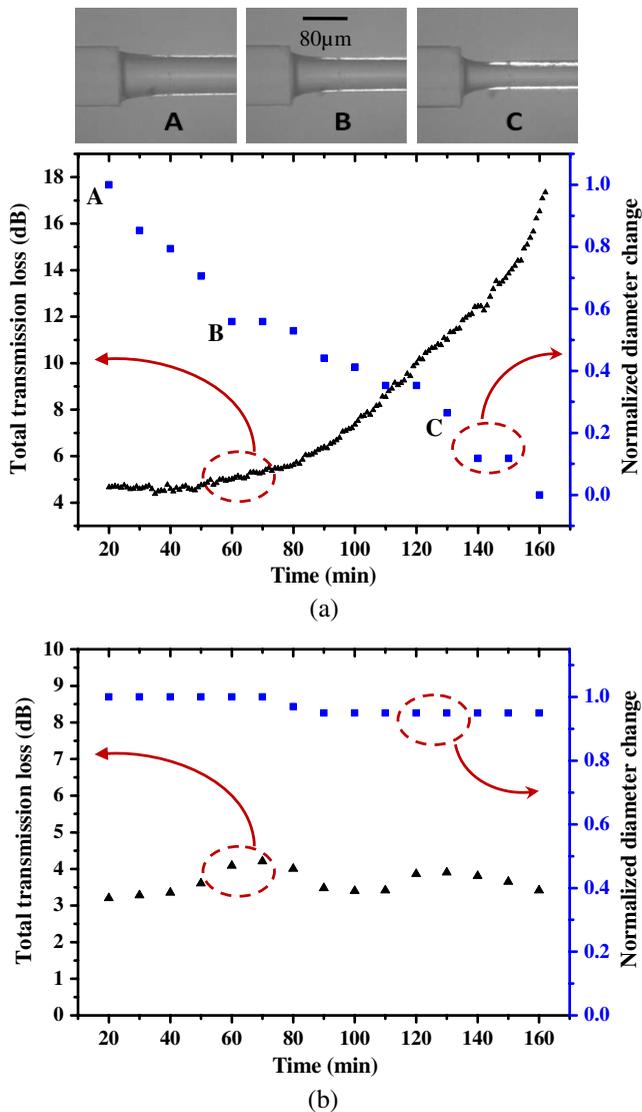


Fig. 3 Measured transmission loss and channel width changes versus time for optofluidic waveguide without (a) and with (b) a glass barrier tube. The right vertical axis in (a) and (b) quantifies the channel diameter change relative to its original diameter.

evaporation is responsible for the observed effects. Indeed, for the channel with a glass barrier tube as shown in Fig. 3(b), we find that the channel width does not change and the transmission loss is stable for several days.

Assuming that optical losses due to the fiber connectors and fibers are negligible, the total transmission loss mainly comes from propagation loss (due to fluid absorption, surface roughness of the liquid-core structure, PDMS cladding, modal propagation loss, etc.) and coupling loss between the liquid-core waveguide and the optical fibers at the input and output ports. The optical propagation loss in the liquid-core waveguide can be estimated by measuring the optical scattering loss along the liquid-core waveguide. A representative example using green laser light at the input is shown in Fig. 4 for the case of an optofluidic waveguide without a glass barrier tube; we find a net propagation loss of approximately 2 dB. A similar measurement when the glass barrier is present is difficult because of the cylindrical surface of the tube. However, we expect the optical coupling loss and

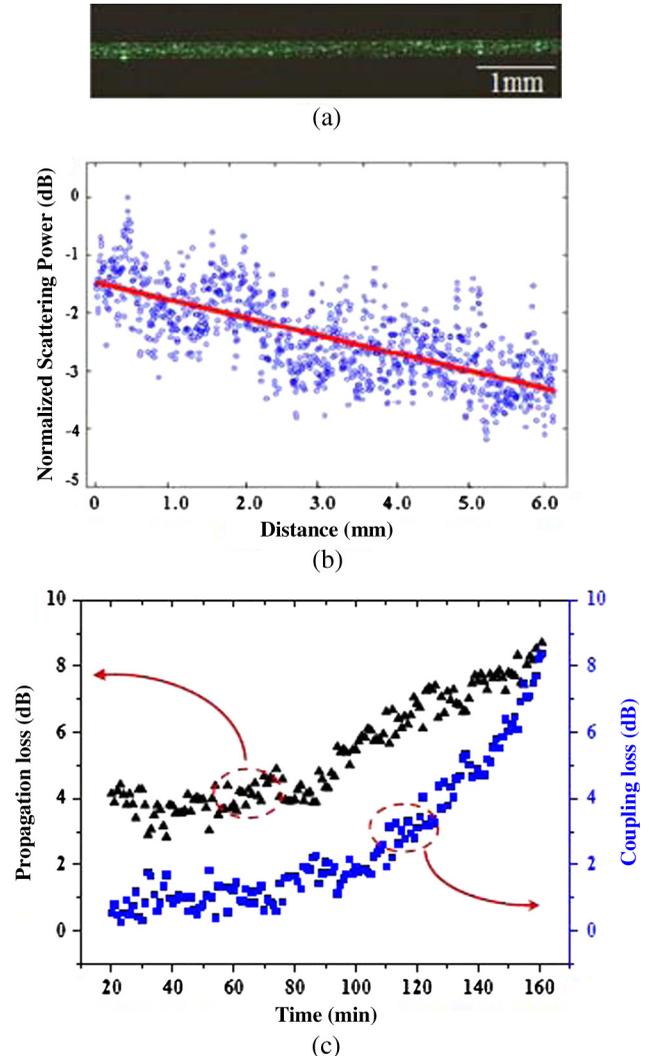


Fig. 4 (a) Microscope image of the optofluidic waveguide for green light ($\lambda = 532$ nm) input illumination. (b) Extracted light intensity along the optofluidic waveguide. (c) Propagation loss and coupling loss of the optofluidic waveguide without a glass barrier tube.

propagation loss to be similar to those found in the example case above (assuming the same channel width). The total optical coupling loss between the optical fibers and liquid-core waveguide is extracted by subtracting the optical propagation loss in the liquid-core structure from the total transmission loss [Fig. 4(c)]. For a liquid-core diameter of approximately $40 \mu\text{m}$, the propagation loss is dominant and the total coupling loss of the input and output sections is approximately 1 dB, demonstrating that the present structure can be effectively used to excite and collect optical signals in a liquid-core waveguide. As the channel shrinks further, however, the coupling loss and propagation loss estimated from the measurement increase.

3.2 Optical Faraday Rotation Measurements

With a total length approaching 2 cm and a volume of only 160 nL, our device structure is well-suited for applications that must reconcile the use of mass-limited samples with the need for a long optical path. Such is the case, for example, of optical absorption, where detection sensitivity grows linearly

with the probed sample length. Rather than absorption, however, here we investigate the device performance through the observation of OFR, i.e., the change in the direction of the plane of polarization of linearly polarized light upon crossing a magnetized sample. Phenomenologically, it is found that the rotation angle θ grows with the sample length L and the magnetic field B in the direction of light propagation according to the formula,

$$\theta = VLB,$$

where we use V to denote the so-called “Verdet constant”, more correctly, a known function of the illumination wavelength specific to the fluid under consideration.

To measure OFR, we integrate our device with the setup sketched in Fig. 5(a). We rely on a single-mode (multimode)

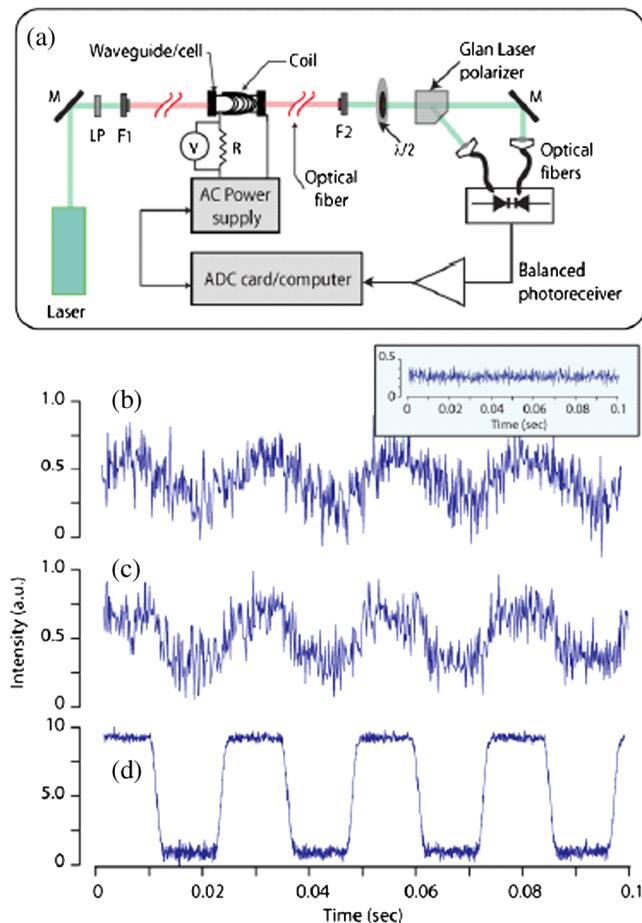


Fig. 5 (a) Optical Faraday rotation (OFR) experimental setup. LP, F, and M, respectively denote the linear polarizer, fiber optics, and mirror; R is a monitor resistor and $\lambda/2$ is a half-wave plate. Depending on the experiment, we replace the fiber-coupled device by an optical cell (see below). (b) OFR trace using the present fiber-optic-coupled waveguide filled with dimethyl sulfoxide (DMSO). (c) Same as in (b) but for the case of a DMSO-filled optical cell. Transmission and polarization of the output beam were altered to replicate the conditions in (a), i.e., 30% transmission, 15% linear polarization. (d) Reference OFR signal for “through-air” cell illumination and detection (95% transmission, 90% linear polarization). In (b)–(d), the total number of repeats is 10^3 , the applied magnetic field is $57 \mu\text{T}$ and the modulation frequency is 25 Hz. The figure insert [upper right of (b)] is the observed response in the absence of magnetic field. This result can be extended to all three cases when either the magnetic field is turned off or the light beam is blocked.

fiber for light delivery (collection) into (from) the fluidic waveguide as described above. As demonstrated in the previous measurement, the optofluidic waveguide structure with a glass barrier tube effectively maintains the optical transmission for a few days without any notable degradation. We use a half-wave plate and a Glan-Laser polarizer to split the output beam in two branches of identical intensity and opposite linear polarization, which, in turn, are guided into a differential photoreceiver. An eight-turn solenoid wound around the device serves as the magnetic field source; a 1Ω resistance in series with the solenoid is used to monitor the current (and thus the magnetic field) on the sample volume. Upon application of a B field, the ensuing change in the beam polarization translates into a nonzero intensity difference between the two beam branches, thus resulting in a Faraday rotation signal. We use a variable frequency AC source synchronous with the signal acquisition timing to modulate the field intensity at a predetermined, nonzero frequency, thus improving our detection sensitivity.

We note that Faraday rotation measurements provide a more comprehensive (and demanding) characterization of the device optical performance: As in absorption measurements, the amplitude of the OFR signal grows linearly with the light intensity at the photoreceiver and thus depends critically on the device transmission characteristics. On the other hand, Faraday rotation gives us the opportunity—absent in absorption measurements—to investigate the impact of the various device components on the beam polarization. We start with Fig. 5(b) where we show the DMSO OFR signal as determined using the present device for an acquisition time of 0.1 s and after 1000 repeats in the presence of a $57 \mu\text{T}$ magnetic field. As expected, the observed trace displays a periodic modulation at the chopper frequency, which disappears in the absence of illumination or applied magnetic field [insert trace in Fig. 5(b)]. The amplitude of this modulation is proportional to the Faraday rotation angle θ (approximately 5×10^{-6} radians for DMSO under the present conditions) and is indicative of the system sensitivity.

Comparison with the typical, macroscopic Faraday rotation experiment—where a beam of the same input power crosses a DMSO sample contained in a 4-mm diameter, 2-cm-long cell in the absence of fiber optics—shows that our device only manages to capture a small fraction (5%) of the signal otherwise available [Fig. 5(d)]. To understand the origin of this discrepancy, we conducted a set of tests that progressively altered this reference “macroscopic” geometry to closely emulate the one found in the PDMS device. For example, in an initial study we intercalated a filter in the trajectory of the output beam so as to reproduce the device losses (see above), only to observe a signal reduction proportional to the power drop in the light beam.¹² Notably, however, we find a virtually identical result even if, rather than direct illumination, we use a $9 \mu\text{m}$ core, single-mode optical fiber to deliver light into the cylindrical container. This observation (along with a direct inspection of the polarization and transmission properties of the single- and multimode fibers) points to the output fiber as the main component responsible for the signal loss. Indeed, Fig. 5(c) shows that an OFR signal comparable to that observed with the PDMS device is obtained using the reference cylindrical sample cell when the output beam traverses a multimode

fiber prior to detection (i.e., the transmission and polarization output in this experiment were similar to those obtained with the microfluidic device).

The above observations highlight an important tradeoff inherent to the use of the present geometry: light collection via a multimode fiber is well suited for light absorption studies of liquid samples, but the spatially nonuniform, mixed polarization is inadequate for high-sensitivity OFR or other polarization-dependent studies.

4 Conclusions

This paper demonstrates a simple fabrication method for a liquid-core optofluidic waveguide and its efficient optical coupling with optical fibers to analyze liquid solutions. The elasticity of PDMS allows a simple pressure-sealing of the optical fibers with the optofluidic channel, and efficient optical excitation into and extraction from a liquid-core waveguide. We achieve a total optical loss as small as 3 dB from an optical fiber with a core diameter of 9 μm to an optical fiber with a core diameter of 62.5 μm , bridged to the optical fibers through a 2 cm long liquid-core optofluidic waveguide. The total coupling loss between the optical fibers and the liquid-core waveguide was estimated at about 1 dB for the liquid-core waveguide (for a diameter of 40 μm or greater). To prevent liquid evaporation, we also employ a glass barrier tube, which shows stable optical transmission over several days. While the present design is limited to liquid samples with index of refraction higher than that of PDMS, our device can be adapted to monitoring other fluids (including water) if the walls of the microfluidic channel are coated with Teflon-AF ($n = 1.31$).⁴ Our approach promises to be useful for applications that require long interrogation times as well as long optical paths using mass-limited liquid samples. Besides absorption experiments, the latter includes polarization sensitive observations such as OFR, although in this latter case further work will be necessary to minimize the collection fiber impact on light polarization.

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