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Optofluidic waveguides: II. Fabrication and structures

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Abstract

We review fabrication methods and common structures for optofluidic waveguides, defined as structures capable of optical confinement and transmission through fluid filled cores. Cited structures include those based on total internal reflection, metallic coatings, and interference based confinement. Configurations include optical fibers and waveguides fabricated on flat substrates (integrated waveguides). Some examples of optofluidic waveguides that are included in this review are Photonic Crystal Fibers (PCFs) and two-dimensional photonic crystal arrays, Bragg fibers and waveguides, and Anti Resonant Reflecting Optical Waveguides (ARROWs). An emphasis is placed on integrated ARROWs fabricated using a thin-film deposition process, which illustrates how optofluidic waveguides can be combined with other microfluidic elements in the creation of lab-on-a-chip devices.

Keywords

Optofluidics; Integrated optics; Waveguides; Photonic crystals; Bragg

1 Introduction

Light guiding through a fluid is a natural phenomenon that takes place every time a ray of light enters a free flowing stream of water. Tyndell recognized this as one of the first examples of total internal reflection (Hecht 1998). For decades after these observations, optical waveguiding was relegated primarily to solid materials, especially silica based glasses. Years of research in this area of course culminated in optical fiber with incredible purity and light transmission properties. Until recently, research in fluid waveguiding has been somewhat sporadic, coming either from attempts to compete with glass optical fiber or to build light based fluid sensors. The past decade, however, has seen a surge in interest in the topic (Psaltis et al. 2006; Monat et al. 2007). This coincides somewhat with the introduction of photonic crystal structures in many different variations. These structures were originally designed to allow for guiding through air, but their operating principles can also be applied to other low refractive index materials like water. Optofluidic devices have also benefited from the technology development that has taken place in the telecommunication and microelectronic industries. Techniques used to create all-glass optical fiber are now used to create hollow waveguiding fibers that can be filled with

liquids. Semiconductor fabrication has been adopted to create mechanical structures on chips, including waveguides and photonic circuits and fluid based labs-on-a-chip.

Optofluidic waveguides have found applications in liquid analysis in which optical beams are used to probe lengths of a fluid column. Guided modes within the waveguides can confine light into small areas, leading to high intensities over long distances. Fields such as analytical chemistry, toxicology, and environmental monitoring can benefit from liquid waveguide based systems. Biosensing is another promising application. The small dimensions and fluid volumes in an optofluidic waveguide are suitable for very high sensitivity detection including single molecule detection (Craighead 2006). Recently a number of active optical devices have also emerged that rely on these waveguides including fluidic dye lasers (Balslev and Kristensen 2005).

This paper reviews fabrication techniques and structures for optofluidic waveguides including those that are fiber based and those that can be integrated on a chip. Structures will be included that have demonstrated light guiding through a liquid medium or through an air medium (with a suitable light confining mechanism for fluid guiding). While no paper can hope to be entirely inclusive, the intention is to provide the reader with an understanding of what has already been made and insight into what future optofluidic based waveguides are possible, given our current fabrication methodologies. The waveguides cited include a variety of light confinement mechanisms. In the context of microfluidics and the integration of optofluidic waveguides with other optofluidic devices, we will include an emphasis on the integrated waveguides our research groups have recently developed. These planar Anti Resonant Reflecting Optical Waveguides (ARROWs) provide a good example of marrying multiple microfluidic and optical functions on a compact platform.

This paper is organized as follows. Section 2 provides a very brief review of the two most common waveguiding mechanisms. Sections 3 and 4 then provide a review of fabrication methods and structures, first for fiber based waveguides and then on-chip implementations. These sections are organized according to general fabrication approach, with specific structures using various waveguiding mechanisms. Section 5 concentrates on planar ARROW waveguides and on-chip test platforms constructed using thin-film deposition and sacrificial etching.

2 Brief review of waveguiding mechanisms

This section is meant simply to provide some basic definitions of waveguiding mechanisms so they can be referred to in the text. The physics of these mechanisms are described more completely in the preceding paper, Optofluidic Waveguides: I. Concepts and Implementations. The majority of waveguides rely on either Total Internal Reflection (TIR) or wave interference for confinement of optical beams. TIR based waveguides surround a high refractive index core material with a low refractive index cladding material. Achieving TIR with a fluid based core is challenging, at least when considering the silicon dioxide based glasses typically used in most optical applications. This is because silicon dioxide has a refractive index of approximately 1.46, whereas most fluids have refractive indexes that are lower (water's is 1.33). Creating TIR based optofluidic waveguides has required using alternate materials or creativity in structure design.

Another approach to waveguiding is the use of wave interferences to confine an optical wave. In this case, multiple layers of material are used as the waveguide cladding. These layers create multiple reflections of the electric field that can interfere constructively or destructively. The key idea is that near-perfect reflection into the original medium can be achieved even if that medium (i.e. water) has a lower index than all of the cladding layer materials (i.e. silicon dioxide).

3 Fibers

Optofluidic fibers have in common a circular cross-section with a hollow core that can be filled with fluid. The outer walls of most fiber are formed from glass, although polymer fibers are becoming more common. Small diameter fibers are formed by first constructing a preform, which is a macroscopic scale version of the structure of interest. A typical preform might be 1 m long and 2 cm in diameter. For a simple hollow glass fiber or capillary, the preform would look like a glass cylinder with a hole in its center. Preforms can be made by extruding glass through a mold or drilling holes through a glass rod. Fiber is created by drawing down the preform in a drawing tower at high temperatures which dramatically increases its length and reduces its cross sectional area while maintaining the geometric proportions set by the preform (Hecht 2002). The fibers cited in Sects. 3.1 and 3.2 have simple structures with a single hole in their center and walls made from a single material. Section 3.3 describes more complicated structured fiber that could contain many holes or have walls made from a stack of different materials.

3.1 Uncoated fibers

3.1.1 Glass fibers with high index liquids—Some of the earliest experiments involving light guiding in a physically confined fluid used glass or quartz fibers filled with high refractive index liquids (Stone 1972). Done in the early 1970s, this work was driven by the desire to create low-loss fibers for optical communications. The fibers were drawn from preforms down to inner diameter dimensions of approximately 75 μm . Fibers hundreds of meters in length were constructed and tested. Given the refractive index of quartz and glass ($n \approx 1.46$), in order to achieve total internal reflection, the tubes were filled with a combination of tetrachloroethylene ($n = 1.50$) and carbon tetrachloride ($n = 1.458$)—see Fig. 1a. Light was launched into the tubes using a microscope objective to focus through the glass window of a filling cell which contained the high index fluid and mechanically positioned the fibers. While relatively low loss optical transmission was achieved (2×10^{-4} dB/cm for 840 nm light), improvements in all-silica fiber rendered liquid core fiber obsolete for long haul optical communications.

3.1.2 Glass capillaries with reflection at the outer air-glass interface—Glass capillaries filled with low refractive index liquids (low relative to glass) have been used as waveguides in which the light guiding mechanism was the total internal reflection at the glass-air interface of the capillaries' outer edge (Tsunoda et al. 1989)—see Fig. 1b. Light propagating down the length of the tubes was thus able to interact with the fluid, although the fluid-glass interface provided no light guiding. Capillaries with inner diameters ranging between 340 and 215 μm were used. Light was launched into the capillaries through a 50- μm core quartz optical fiber. The optical fiber was positioned into one arm of a T-shaped metal fixture, the glass capillary held in place in a second arm, and liquid injected through the third arm. Researchers reported that light transmissivity through the capillaries was highly dependent on the glass capillary's surface conditions.

3.1.3 Teflon[®] AF fibers—These are examples of fibers made with polymer sidewalls (Altkorn et al. 1997a; Dallas and Dasgupta 2004)—see Fig. 1c. Teflon[®] AF is an amorphous copolymer of polytetrafluoroethylene (PTFE) with a low refractive index ($n = 1.29\text{--}1.31$). Teflon[®] AF fibers filled with aqueous solutions ($n = 1.33$) have TIR confinement at the fluid-Teflon[®] interface. Fibers are formed by melting Teflon[®] pellets into a preform and drawing capillaries in much the same way glass capillaries are made. Researchers report smooth Teflon[®] sidewalls capable of low loss light propagation. Capillary dimensions are dependent on drawing conditions but typical inner diameters are around 250 μm . An alternative fabrication method consists of using a glass capillary coated internally with

Teflon[®] (see Sect. 3.2.2) and then removing the outer glass using hydrofluoric acid (Dasgupta et al. 1998). Light and fluid are typically injected into these fibers through separate arms of T-shaped fixtures. Teflon[®] AF tubing intended as a water filled light guide is now commercially available from companies such as Biogeneral (<http://www.biogeneral.com/teflon.asp>).

3.2 Coated fibers

3.2.1 Glass capillaries coated externally by a low index polymer—Glass capillaries have also been coated externally by low refractive index polymers, specifically Teflon[®] AF, to produce TIR at the glass–Teflon[®] interface (Altkorn et al. 1997b; Wang et al. 2001)—see Fig. 2a. Teflon[®] AF is applied to the glass surface using a liquid solution available from Dupont Corporation (http://www2.dupont.com/Teflon_Industrial/en_US/products/product_by_name/teflon_af/index.html) that solidifies to an approximately 15 μm thick film coating. Characterization systems were constructed using 530 μm inner diameter glass tubes. Optical injection and fluid filling are commonly done using a T-fixture and optical fiber. Teflon[®] coated capillaries have been used for analysis of liquids with light launched along the length of the capillary and for light launched perpendicular to the capillary. In the latter arrangement, a fluorescence signal is excited and guided down the length of the capillary. Teflon[®] AF coated light guiding capillaries are now commercially available from companies such as Polymicro Technologies (http://www.polymicro.com/products/opticalfibers/products_opticalfibers_fsu_flu.htm).

3.2.2 Glass capillaries coated internally by low index polymer—Teflon[®] AF coatings have also been applied to the inner wall of glass capillaries, making them capable of total internal reflection when filled with aqueous solutions ($n = 1.33$) (Dreß and Franke 1996; Schelle et al. 1999)—see Fig. 2b. The Teflon[®] layer is applied through dip coating a liquid solution. Thicknesses for the Teflon[®] coating range from 50 to 7 μm depending on the coating conditions with total capillary lengths on the order of tens of centimeters. Researchers have reported coating capillaries with inner diameters varying between 0.36 and 4 mm, with the smaller diameter capillaries requiring pumping systems to pressurize the coating solution in order to coat long lengths. Optical launching has been done with a T-fixture and optical fiber or by simply placing an optical fiber inside large diameter capillary tubes.

3.2.3 Metal coating on outside of glass fibers—The idea behind these structures is straightforward. By coating the inside or outside of a glass wall with a highly reflective metallic layer, light propagation loss should be reduced compared to the uncoated case. Light guiding and confinement was successfully demonstrated in glass capillaries which had their outer walls coated by a layer of silver (Wang et al. 1991)—see Fig. 2c. The capillaries had an inner diameter of 75 μm and an outer diameter of 364 μm . The silver coating was applied directly to the glass using a redox reaction of $\text{Ag}(\text{NH}_3)_2^+$ and glucose and then painted over with black paint to provide physical protection. Windows separated by 0.8 mm were opened in the silver coating to provide access for optical beams. The capillary was filled with an aqueous solution and then a beam from a He–Ne laser introduced perpendicular to the capillary through one of the window openings. The beam was monitored by a detector located at the opening of the second window. The multiple reflections of the laser at the glass–silver interface allowed the beam to interact with the fluid over a much longer path length than would be possible in a single-pass measurement system. When the silver coated capillaries were used for optical absorption measurements, signal-to-noise ratios were dramatically increased compared to single-pass arrangements.

3.2.4 Metal on inside of glass fibers—The inner wall of glass capillaries has also been coated with metallic layers in order to produce a high reflectivity surface (Mohebbi et al. 2002)—see Fig. 2d. Silver layers several hundred nanometers thick were applied to tubes with inner diameters of 250 μm using a liquid-phase deposition process similar to the one described in Sect. 3.2.3 to coat outer tube walls. To achieve even coating of 50 cm long capillaries, the silver coating solution was pumped through the capillary with a peristaltic pump. Optical measurements for the resulting structure were made when the capillary was filled with air and showed an attenuation of 0.004 dB/cm at a wavelength of 800 nm. Although measurements were done on these capillaries when filled with air, guiding could also be accomplished when they were filled with low index fluids.

3.3 Structured fibers

3.3.1 Bragg fiber—Bragg fibers rely on reflective dielectric cladding layers that coat the inner walls of a hollow tube—see Fig. 3a. These structures utilize interference-based waveguiding and the dielectric layers must be periodic and of controlled thicknesses and composition. Bragg fibers have been made in a number of ways. One method involved the deposition of As_2Se_3 layers onto poly(ether sulphone) (PES) films (Fink et al. 1998; Temelkuran et al. 2002). These films were rolled into a hollow multilayer tube that acted as a preform. This preform was then placed in an optical fiber drawing tower and drawn down so that the As_2Se_3 and PES films were 270 and 900 nm thick, respectively, and the fiber had a central hollow core of 275 μm . These Bragg layers can be designed to be reflective for all angles of incidence (omnidirectional guiding) and light can even propagate around bends with very little loss. This particular design and fabrication method has become known as the Omniguide and these types of fibers are now commercially available (<http://www.omni-guide.com/>). Bragg fibers have also been made using all-polymer dielectric layers (Pone et al. 2006). Fibers were drawn from preforms that were created through the rolling method mentioned above or through solvent evaporating alternating polymer layers on the inside of a rotating tube. Bragg fibers were also constructed in a coating procedure that mimics the silver coated capillaries cited above. Layers of cadmium sulfide and lead sulfide were placed over silver coatings using a liquid-phase deposition process with specific applications in the infrared wavelengths (Gopal and Harrington 2003). One millimeter inner diameter tubes with these coating exhibited an attenuation of 6×10^{-4} dB/cm at a wavelength of 1.55 μm when filled with air. Liquid-filled Bragg fibers have not yet been reported, but given that waveguiding takes place when filled with air, it should also occur with aqueous cores.

3.3.2 Photonic crystal fibers—Photonic crystal fibers (PCFs) utilize a two dimensional, periodic array of low refractive index material imbedded inside a high index material to guide light down the length of a fiber based on an interference mechanism—see Fig. 3b. PCFs have been most commonly realized using silica as the high index matrix material with air holes used as the low index array (Knight 2003). The spatial structure of the air/silica index variation determines the propagation properties in the PCF along the length of the fiber including allowable guided light wavelengths. PCFs are fabricated by drawing preforms and one common method for constructing glass preforms is by stacking glass capillaries and rods together into a desired geometry. In addition to silica–air PCFs, polymer based PCFs can also be created through similar manufacturing processes (Cox et al. 2006). PCF cross sectional designs can be quite varied, but one popular design uses a large hollow core in the center of a two-dimensional array. Typical diameters of this center core are between 5 and 20 μm . Researchers have demonstrated that the voids of PCFs can be filled with low refractive index fluids that allow for low loss transmission down long lengths of fiber and very long interaction lengths between the light and the fluid (Jensen et al. 2004; Martelli et al. 2005; Rindorf et al. 2006). Launching light into these fluid filled fibers can be

accomplished with the same type of T fixtures used when evaluating glass capillaries (described above). PCFs have become very ubiquitous and are now commercially available from a number of companies in standard and custom configurations (<http://www.crystal-fibre.com/products/airguide.shtm>, <http://www.newport.com/Photonic-Crystal-Fibers/323352/1033/catalog.aspx>) (Table 1).

4 Integrated waveguides

Integrated waveguides are by definition constructed on planar substrates, usually of silicon, glass, or quartz. Whereas fiber relies on the forming and drawing of preforms, integrated waveguides are made using micromachining procedures borrowed from the semiconductor industry. Common processes include lithographic patterning, thin-film deposition, and etching. The cross sections of integrated waveguides are more often rectangular than circular because of the way they are fabricated. The substrates these waveguides are made on are usually meant to be cut into chips with dimensions of only a few centimeters. This limits the length of integrated waveguides, and they are typically much shorter than the lengths of fiber most researchers would work with. Consequently, integrated waveguides with relatively high optical losses may still be interesting because on-chip systems may only require short optical path lengths. The waveguides cited in this section are grouped according to shared fabrication methods, although, due to the wide variety of micromachining processes available, it can be difficult to categorize the structures.

4.1 Wafer bonding

Wafer bonding is a common technique in the Micro-Electro-Mechanical-Systems (MEMS) field used to join two substrates together (Liu 2006). This technique is used to form hollow waveguide channels by etching trenches into the surface of one substrate and then joining it with a second substrate. The trenches are covered by the second substrate, forming enclosed channels. Wafer bonding requires very thorough surface cleaning and an environment free of particles. The bonding process is done at high temperatures so chemical bonds between the substrates can be formed, making the structure mechanically strong. Common wafer bonded materials include silicon, glass, and polymers like polymethylmethacrylate (PMMA).

4.1.1 Glass capillaries (“leaky waveguides”)—Perhaps the simplest examples of wafer bonded optofluidic waveguides are the so-called “leaky” waveguides formed with two glass substrates (Fouckhardt et al. 2001)—see Fig. 4a. Trenches 30 μm deep and 60 μm wide were etched into the surfaces of both substrates using hydrofluoric acid. The substrates were positioned so that the trenches were aligned to each other and then the wafers bonded together. The cross-section of the resulting waveguides was somewhat elliptical in shape. The glass substrates were cut and polished perpendicular to the waveguides to form two smooth facets on either side of a chip. Light was launched into one of the facets and transmission measured out the other side of the facet. When filled with dioxane, the waveguides exhibited a loss of 1.4 dB/cm using 840 nm light. In “leaky waveguiding” the reflection of light beams at the fluid–glass interface is only partial so optical losses are higher than in TIR structures. Loss is highly dependent on the diameter of the fluid filled core, increasing rapidly for core diameters below 30 μm . Simple glass capillaries can also serve as leaky waveguides, but since a high-loss waveguide has more utility with on-chip applications, they are only mentioned in the context of integrated waveguides.

4.1.2 Teflon coated microchannels in silicon—Wafer bonding was combined with Teflon[®] AF coating to create integrated TIR-based waveguides on silicon (Datta et al. 2003)—see Fig. 4b. The standard micromachining technique of potassium hydroxide wet etching

was used to form trenches 500 μm wide and 180 μm deep into a silicon substrate. These trenches were spin coated with a Teflon[®] AF solution and then baked at approximately 300°C to drive off solvents and solidify the Teflon[®]. Films of over 3 μm in thickness were produced, although for some types of Teflon[®] AF solutions this had to be done with multiple spin coatings. It should be noted that a coating of perfluorooctyltrimethoxysilane was applied to the silicon immediately before the spin coating to promote Teflon[®] adhesion. A glass substrate was also covered with a Teflon[®] film using the same procedure. The glass and silicon substrates were then bonded together at 300°C with their Teflon[®] surfaces in intimate contact. In this way, enclosed and water-tight Teflon[®] coated channels up to 2.3 cm long were produced. Optical testing of channels was conducted by placing a bundle of optical fibers directing into the channel and sealing the fibers in place with a silicone rubber. When filled with deionized water, the optical loss in the Teflon[®] coated channels was reported to be 1.09 dB/cm using 660 nm light. Researchers reported that the cured Teflon[®] coating produces a smooth surface, although not as smooth as the surface found in Teflon[®] capillary tubing.

4.1.3 Metal coated silicon microchannels—Wafer bonding was also combined with a thin film metal coating to create integrated waveguides in which the inner walls were coated in metal (Jenkins et al. 2003)—see Fig. 4c. Deep reactive ion etching (RIE) was done on silicon or Silicon-On-Insulator (SOI) wafer to form trenches 250 μm wide and 50 μm deep. CVD coated copper or evaporated gold coatings are then deposited inside the trench. A second silicon wafer also covered in the metal was then bonded over the first to form an enclosed channel. Researchers report low attenuation losses (<0.1 dB/cm) for these relatively large cross sectional area structures. Measurements were made using 1,550 nm light when the channel was filled with air. Guiding would also be possible, however, when the channels were filled with liquid.

4.1.4 Integrated Bragg waveguides in silicon—Bragg waveguides consisting of hollow microchannels surrounded by periodic dielectric layers have also been fabricated using wafer bonding (Lo et al. 2004)—see Fig. 4d. A thin and shallow trench was etched into a silicon wafer and then CVD coated with six pairs of alternating silicon/silicon dioxide layers. A second unetched silicon wafer was coated with the same layers and the two wafers bonded together resulting in hollow microchannels 1.3 μm wide and 1.2 μm tall. The silicon wafer was cut perpendicular to the waveguides and smooth facets formed by polishing. Waveguides as long as 2.9 cm were measured and propagation losses in air were around 1.7 dB/cm when tested using 1,550 nm light. Guiding in these structures should also occur for fluid filled cores. Bragg waveguides rely on interference based confinement to achieve guiding in such small cross sections.

4.1.5 ARROWS in silicon—ARROW waveguides are also based on an interference effect, but do not require the periodicity of a photonic crystal. Consequently, a single dielectric layer is sufficient to provide low-loss propagation—see Fig. 4e. A detailed description of our groups' fabrication method of ARROW structures is given in Sect. 5, but we note here the use of wafer bonding for ARROW construction. Researchers reactive ion etched 150 μm wide, 150 μm deep trenches into a silicon wafer and coated the trench with alternating layers of silicon dioxide and silicon nitride deposited through CVD. Only one pair of oxide and nitride layers was used. The etched wafer was bonded to an unetched wafer coated with similar layers forming a hollow waveguide that could be filled with fluid (Campopiano et al. 2004; Bernini et al. 2007). Structures were tested by inserting optical fibers directly into openings made at the ends of the hollow channels. Side arms that intersected the waveguides were also etched into the silicon so that liquids could be injected in and out of the channels.

4.1.6 Nanoporous cladding on glass—TIR based waveguiding is also possible using nanoporous films with very low refractive indexes (compared to water) attached to planar substrates (Risk et al. 2004). This idea was demonstrated by using a solution containing poly(methylsilsequioxane) (PMSSQ), PS-PEG, and the solvent 1-methoxy-2-propanol acetate. The solution was spin coated on a substrate and heated to 450°C to remove the solvent and dissolve the PMSSQ. When the PMSSQ is removed, nanoscopic air pores 10–15 nm in diameter are left in the PS-PEG. While PS-PEG has a refractive index of 1.37, the effective index of the nanoporous film is an average of the PS-PEG index and the refractive index of the pores ($n = 1$). Changing the ratios of PMSSQ to PSPEG in the solution changes the density of air pores and can allow the tuning of the subsequent film refractive index. Researchers report that films with refractive indexes ranging from 1.37 to 1.15 have been produced. Liquid waveguiding was demonstrated by coating two silica wafers with a nanoporous film 0.8 μm thick with a refractive index $n = 1.27$. The two wafers were placed in a holder that kept them 50 μm apart with their nanoporous films facing each other—see Fig. 4f. Water was placed in the 50 μm gap and light introduced into the gap through a cylindrical lens. This arrangement produced only one-dimensional confinement but allowed researchers to measure the optical modes and waveguide loss parameters produced by the films, which were reported as better than 1 dB/cm. If thicker nanoporous films were desired, multiple spin coatings could be done. Although an enclosed channel has not been demonstrated, this structure has been categorized with other wafer bonded structures because enclosed waveguides could likely be made using that method—similar to what was done with the Teflon[®] AF coated microchannels.

4.2 Nanoscale etching

The waveguides discussed in Sect. 4.1 all feature core dimensions of ten to hundreds of micrometers. With the advent of lithography and etch tools capable of producing nanometer scale features, a couple of new optofluidic waveguiding structures have emerged.

4.2.1 Slot waveguides—In slot waveguides, index guiding can be realized in nanoscale cross sections of a low index medium (Xu et al. 2004; Dell’Olio and Passaro 2007)—see Fig. 5a. The structure of a slot waveguide consists of a high index material surrounded by a low index material. A trench approximately 100 nm wide is etched into the center of the high index material forming the low index region. If the trench dimensions are small enough, a substantial amount of the guided optical mode is present in the low-index region through field evanescence. The advantage of slot waveguides is that their modes are actual mode solutions of the Helmholtz equation. That should, in principle, allow for lossless propagation of a sizable fraction of the power within a nanoscale channel. Slot waveguides have been made using SOI wafers in which a ridge of silicon is etched down to the buried oxide layer through reactive ion etching. A 100 wide nm trench is then etched around 300 nm deep into the silicon ridge, also through reactive ion etching. The resulting silicon cross section thus has silicon dioxide below it and is surrounded by air above. The top of the silicon can also be coated in chemical vapor deposited silicon dioxide and the conformal nature of the deposition will seal the narrow slot from above, leaving a void surrounded by solid material. Waveguiding takes place when infrared light is launched down the silicon ridge and confined by the lower index materials surrounding it. To this point, slot waveguides have been demonstrated only with air filling the nanoscale slot, but guiding would also be possible with low index liquids fluids filling this space.

4.2.2 Planar photonic crystal waveguides—Photonic crystal structures analogous to PBFs can be constructed in two-dimensional arrays on the surface of a substrate—see Fig. 5b. Typical fabrication involves the reactive ion etching of large numbers of nanometer scale holes (i.e. 200 nm) into silicon. Light can be confined in waveguides along the surface

of the wafer with TIR providing the confinement above and below the etched silicon structure, and the photonic crystal array providing the confinement on the left and right. Many of these integrated waveguides are created on SOI wafers so there is a silicon dioxide layer underneath the patterned silicon layer. Light can be coupled into and out of these photonic crystal waveguides through TIR based ridge waveguides etched into silicon. Researchers have been able to fill the etched voids of the photonic crystals with fluids and use them as fluidic sensors (Chow et al. 2004; Skivesen et al. 2007) as well as fluidic controlled photonic devices (Erickson et al. 2006). To selectively control the flow of fluids over the photonic crystal structures, silicone molds containing fluid channels are aligned and attached to the silicon substrate.

4.3 Liquid–liquid waveguides (L^2 s)

Another method for creating integrated optofluidic waveguides with TIR confinement is to use two different liquids with different refractive indices inside a larger fluidic channel—see Fig. 6. This concept was first demonstrated using CaCl_2 ($n = 1.445$) and water as core/cladding liquids, respectively, flowing in Poly(dimethylsiloxane) (PDMS) ($n = 1.4$) channels (Wolfe et al. 2004, 2005; Brown et al. 2006). The PDMS channels were created by pouring a liquid pre-polymer of PDMS over a lithographically patterned silicon wafer (McDonald and Whitesides 2002). After the PDMS was hardened, it was peeled off of the silicon wafer retaining the patterned features—in this case 100 μm deep and approximately 100 μm wide trenches. The PDMS was then placed on another PDMS coated substrate to form channels surrounded by PDMS. Designs for L^2 chips included a central waveguiding microchannel that is aligned to a multimode fiber inserted into a slot in the PDMS layers. Core fluid is first injected into the central channel through another tapered channel. Cladding fluid is then injected on either side of the central channel, resulting in the core fluid being surrounded by cladding fluid on the left and right and PDMS above and below. The fluids exhibited relatively slow mixing along the channel due to laminar flow conditions with the central core width being approximately 10 μm . Channel tapers and bends and large fluid inlets are all easily patterned into the PDMS. In addition to waveguides, several other optical functions have been demonstrated using the L^2 concept including an optical switch, a fluorescent light source, optical splitters, and wavelength filters.

4.4 Buckling

Our last example of integrated waveguides in this section are Bragg waveguides created through the delamination of polyamide-imide and $\text{Ge}_{0.33}\text{As}_{0.12}\text{Se}_{0.55}$ thin-film layers that formed small buckles at a substrate's surface (DeCorby et al. 2007)—see Fig. 7. The buckles created air gaps surrounded by the thin films and their width was controlled by patterning a silver layer sandwiched between the other films. Waveguides approximately 20 μm wide and 3 μm tall were made with this method on silicon substrates. Cleaving through the silicon perpendicular to the waveguides provided optical access to the hollow waveguide cores. Losses as low as 15 dB/cm were measured for air cores using 1,550 nm light (Table 2).

5 Planar integrated ARROWs

Our research groups at BYU and UCSC have recently developed ARROW based optofluidic waveguides using a thin-film deposition technique. We have purposely pursued making waveguides with small hollow cross-sectional areas (approximately $10\ \mu\text{m} \times 4\ \mu\text{m}$). These small areas limit the total fluid contained in the waveguides and allow for analysis of small analyte volumes with single-particle sensitivities. Such small cross-sections require good optical confinement to achieve reasonable optical losses, which ARROW dielectric layers have proven to provide. We have had to develop a new fabrication approach for these

waveguides, however, to enable our size constraints and allow the flexibility to create multiple optical elements on the same chip.

5.1 Thin-film planar fabrication and structures

An ideal fabrication method for optofluidic lab-on-a-chip elements could be used with a variety of materials (i.e., semiconductors, metals and glasses) and allow ready access to fluidic channels at the surface of a wafer. It would also allow for the creation of hollow channels for fluid manipulation, solid and fluid filled waveguides for routing optical signals, and metallic lines and pads for routing electrical signals. In the interest of cost, it should not stray too far from standard procedures used in the microelectronics industry. In this section, we describe a procedure that satisfies all of these requirements which was developed for integrated ARROWs. The methodology is flexible enough that it can also be used to produce other microfluidic devices.

The heart of our process is the creation of hollow tubes by surrounding a sacrificial core with silicon dioxide and/or silicon nitride layers and then removing the sacrificial layer with acid etching. This process is depicted in Fig. 8. First, a substrate is coated with silicon dioxide or nitride layers using plasma enhanced chemical vapor deposition (PECVD). These layers are grown to specific thicknesses in order to achieve ARROW based optical confinement. This process takes place at approximately 250°C. A thin layer of sacrificial material is then deposited and defined into a thin line using photolithography and etching. A variety of sacrificial materials may be used, including photosensitive polymers and metals. Layers of PECVD oxide and nitride are then grown over the sacrificial material. The conformal nature of this process is important to ensure that the sacrificial layer is completely enclosed. The final step of the procedure is to expose the sacrificial material to an acid etch from either end of the patterned line. Upon completion of the etch, we are left with a hollow tube with walls composed of silicon dioxide and silicon nitride layers.

A number of sacrificial layers have been investigated in the context of our outlined fabrication process: aluminum (Yin et al. 2005b), SU8 (a photosensitive epoxy) (Yin et al. 2004), and photoresist (Barber et al. 2006a). Aluminum is most quickly removed using a nitric and hydrochloric acid etching solution while SU8 and photoresist are removed using a sulfuric acid and hydrogen peroxide solution. The different sacrificial materials result in differently shaped hollow core cross sections as illustrated in Fig. 9, providing flexibility when designing microfluidic devices. The hollow ARROWs shown in Fig. 9 depict openings that are very small, down to approximately 3 μm across. These diameters are more than an order of magnitude smaller than typical on-chip fluid channels produced by wafer bonding, which is the most common technique used for microfabrication. The alternating silicon dioxide and silicon nitride layers are evident in the images in Fig. 9. The layers nearest the hollow core are grown to thicknesses dictated by the ARROW confinement principle (Duguay et al. 1986) and are around 100–200 nm thick. The topmost layer is a silicon dioxide film grown to around 3 μm thick. The intention of this relatively thick top layer is to give the structure mechanical strength. The optical performance of the waveguide structures shown in Fig. 9 when filled with air and fluids is reported in the previous paper Optofluidic Waveguides: 1. Concepts and Implementations.

In order for structures made using our planar, thin film technique to be useful for fluid and light guiding, the channels must have smooth inner walls, be of reasonable length, and mechanically strong. The first criterion minimizes optical scatter or interruptions in fluid flow and is met by the conformal CVD coating as evident in Scanning Electron Microscope (SEM) micrographs shown in Fig. 9. In order to determine the ultimate mechanical strength of the hollow structures, a finite-element analysis was done using a commercially available software package (ANSYS 6.0). To provide the necessary stress and strain constants to the

software, a set of experiments was also done. The results of the model indicate that the critical failure pressure for a hollow channel can be given by the simple expression

$$P_c = 2S_t \left(\frac{t_h}{w} \right)^2 \quad (1)$$

where S_t is the tensile strength of the overcoat material, t_h is the thickness of the overcoat layer, and w is the width of the channel. This simple equation reveals the functional dependence of the pressure on the width and thickness, and agrees within 10% of the values calculated using the finite-element simulation when $t_h/w < 1/10$. Tests were done on real structures by varying their core width and overcoat thickness to confirm this expression.

From experiments and simulations, we have found that the following design considerations provide optimal channel stability: for rectangular microchannel geometries, a ratio of $t_h/w > 1/10$ is needed; for a trapezoidal channel structure, the ratio must be $t_h/w > 1/25$; and for arch (hemispherical) motifs, the ratio should be $t_h/w > 1/35$ (Hubbard et al. 2005; Barber et al. 2005; Lee et al. 2007).

Because hollow structures are formed through the chemical etching of a sacrificial layer, the ultimate channel length and fabrication time will be dependent on this chemical process. Etch times were investigated for aluminum and SU8 sacrificial cores patterned on silicon. Core width varied between 10 and 300 μm . A single layer of silicon dioxide 3.0 μm thick was deposited over the cores, the silicon substrate was cleaved, and then the aluminum core samples were placed in an aqua regia (3:1 mixture of hydrochloric and nitric acid) solution and the SU8 samples in Nanostrip, a stabilized solution of sulfuric acid and hydrogen peroxide. Samples were periodically removed from the acid solution, and the amount of sacrificial core that was etched was measured using an optical microscope. We found that the etch length as a function of time follows the equation

$$l(t) \approx \sqrt{2k_n D c_o t} \quad (2)$$

where $l(t)$ is the length of the channel etched in a given time, k_n is a constant relating to the geometry of the channel, D is the diffusion coefficient for etchant through the channel, and c_o is a constant relating the concentration of the most critical component of the etch solution. Equation 2 indicates the diffusion-limited nature of the etch mechanism. Moreover, the speed of etching increases with bath temperature, and wider channels etch faster than narrower ones (due to a change in the constant k_n related to channel geometry). Under optimized etch conditions, aluminum cores will clear at a rate of around 10 mm in 24 h, while the SU8 cores clear at a rate of around 1.5 mm per 24 h.

As seen in Fig. 9, the side cladding layers of the ARROWs built using our standard thin film deposition process do not terminate in an air layer, but rather a thick silicon dioxide layer extending to the left and right of the hollow core. If the sides of the waveguides could be surrounded by air, they would have lower optical loss. A way to produce such a structure is to etch a pedestal into the silicon wafer before thin-film waveguide formation, raising the waveguide core above the wafer surface. This results in an air termination to the side of the dielectric layer stack. Figure 10 shows an ARROW waveguide fabricated on top of a raised silicon pedestal. The structure shown in Fig. 10 required the same PECVD layer growth and sacrificial etching used to produce the ARROWs shown in Fig. 9, but represents an advance in the fabrication procedure and structure (Schmidt et al. 2006; Yin et al. 2006a). The pedestal etch was performed in a reactive ion etcher using CF_4 based plasma chemistry.

Careful control of the etch anisotropy and depth are needed to produce a properly sized pedestal, and photolithographic alignment of the core to the pedestal must also be monitored. To this point, lower losses have been achieved in pedestal ARROWs compared to their non-pedestal counterparts.

5.2 Solid and liquid core waveguides on the same substrate

In order to utilize integrated optofluidic waveguides in a compact lab-on-a-chip platform, it is important to be able to interface between fluid filled waveguides and standard solid waveguides. The solid waveguides serve the purpose of directing light on and off the chip and into and out of a fluidic media. An example optofluidic platform is shown in Fig. 11. Using solid waveguides allows for a T configuration in which light is injected down the length of a fluid filled waveguide from one arm of the T while fluid enters the waveguide through another arm. Solid waveguides also allow for perpendicular excitation of the fluid filled waveguides, critical for devices like fluorescence sensors (Yin et al. 2006b).

Our thin-film ARROW fabrication process lends itself well to the simultaneous creation of solid and hollow core waveguides. To form the solid core waveguides, first we fabricate the hollow core ARROWs on a silicon substrate as described previously. We then define the solid waveguides using photolithography and then etching approximately one micron into the top oxide layer using CF_4 -based reactive ion etching. Light is confined in the solid waveguide in the transverse direction by the ARROW layers and air above the waveguide. In the lateral direction, confinement is provided by etching into the top layer. To create an efficient interface between the solid-core and hollow-core waveguides, the optical modes should be vertically aligned. This constrains the choice of core height and dielectric layer thickness. Figure 12 shows an illustration of how solid core waveguides can be formed at the end of a hollow (liquid filled waveguide) and perpendicular to a hollow waveguide (Barber et al. 2006b). Figure 13 shows SEM micrographs of solid core to hollow core interfaces from various angles.

Theoretically, we can design ARROW layers for low loss transmission (99%) at these interfaces, either from solid to hollow or vice versa. However, in practice, this has been difficult to realize due to film imperfections and interface surfaces that are not perfectly vertical. Due to conformality challenges during PECVD growth, these kinds of parameters are very difficult to achieve on the side of a waveguide. Because of these problems, in real structures, we have only measured approximately 30% optical transmission through these interfaces. Future fabrication research will concentrate on improving these transitions (Hawkins et al. 2007).

5.3 Fabrication of other microfluidic components

The thin film fabrication method used to construct ARROWs can also produce an array of microfluidic elements and lends itself to high-density integration even without built-in waveguiding. Examples of microfluidic elements that have been fabricated this way included a capillary electrophoresis separation device (Peeni et al. 2005, 2006). The critical element of the device, two offset T junctions, is shown in Fig. 14 along with separation results. The open cross sections of the microchannels are approximately $10\text{ }\mu\text{m} \times 3\text{ }\mu\text{m}$. Fluorescein 5-isothiocyanate (FITC)-labeled amino acids and peptides have been separated in these devices. The microchannels were fabricated on glass substrates so that confocally filtered laser-induced fluorescence detection could be used. An Ar ion laser was focused through the substrate and used for excitation while a photomultiplier tube was used as a detector. Fluorescence was probed approximately 0.65 cm away from the junction region shown in Fig. 14. The specific separation results shown are for FITC-labeled glycine, phenylalanine and arginine.

Another example of a microfluidic device that has been made using our thin film fabrication method is an electroosmotic (EO) pump (Edwards et al. 2007). The small diameter channels we produce have a large back pressure and moving fluids through them using pressure based pumps like syringe pumps can be difficult. EO pumps are attractive because they are electric based and can generate flows in very small diameter channels. An EO pump can be built by directing the fluid flow generated from a large number of small diameter channels from one fluid reservoir into another. The design of such a pump is illustrated in Fig. 15a. Voltages are applied at both ends of the device, and the generated electric field produces fluid flow in the small diameter channels. Pressure in Reservoir 2 then pushes fluid into the larger diameter channel to the right. Pumps have been made on silicon, glass, and quartz substrates using photoresist/aluminum as the sacrificial material. Devices had 75 channels (5 μm in width and ~ 3.5 μm tall) feeding into a 50- μm -wide channel. Measured volumetric flow rates show that flow scales linearly with the electric field, as expected, and is dramatically increased by using multiple, small-diameter pumping channels. Figure 15b shows an SEM image of the parallel channel region of an electroosmotic pump.

Another attractive feature of our fabrication technique is that sacrificial channels can be formed and etched over a variety of surfaces and even over existing channels. This makes possible fluid channels that cross over each other and connect from one fluid layer to another through vias, much like metal layers connect in microelectronic circuit designs. To this point, we have demonstrated fluid crossovers for small numbers of channels and will continue to pursue vias and crossovers for advanced designs. Figure 16 illustrates one example of how optofluidic waveguides can be used in concert with other microfluidic elements to create sophisticated labs-on-a-chip. This particular chip would allow for three parallel capillary electrophoresis separations to take place with detection done optically through the optofluidic waveguides—eliminating the need for microscopes and drastically reducing the size scale of these operations.

6 Conclusion

Optofluidic waveguides will be an important component of future optofluidic devices and systems. Fluid filled fibers capable of guiding light have progressed dramatically in the past decade due to the introduction of new materials and photonic crystal structures. Teflon[®] coated capillaries, all Teflon[®] capillaries, Photonic Crystal Fibers, and Bragg fibers are now commercially available. They offer low loss transmission through a variety of fluids including aqueous solutions and are available with core diameters that vary between a few microns and hundreds of microns. The wide availability of these fibers has put them in the hands of many researchers and new applications and implementations appear frequently. One limitation of these fibers, however, is that they can be challenging to combine with other fluidic and optical elements, often resulting in bulky fixtures and systems.

One driver behind developing integrated optics and labs-on-a-chip was to alleviate some of the packaging challenges associated with liquid-filled fiber and combine many functions on a compact chip. On-chip fabrication has been challenging, however, and very few lab-on-a-chip systems are commercially available and certainly none that utilize optofluidic waveguides. This will likely change, however, as methods for making integrated photonic crystal structures, Bragg waveguides, and ARROWs become commercially viable. The planar ARROW structures reviewed here show how attractive large-scale integration of fluidic elements can be. On-chip microplumbing stands poised to follow the lead of microelectronics and large-scale electronic circuitry. Fields that should be impacted include medical diagnostics, analytical chemistry, biology, pharmacology, and photonics, along with a host of others still unforeseen.

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Fig. 1. Representations of cross sections for uncoated fibers containing fluid cores. **a** High index fluid in glass, **b** TIR at the glass–air interface, and **c** fiber formed from a low index polymer

Fig. 2. Representations of cross sections for coated glass fibers containing fluid cores. **a** An outer coating with a low index polymer, **b** an inner-wall coating with a low index polymer, **c** a metallic coating on the outside wall, and **d** a metallic coating on the inside wall

Fig. 3.

a Representative cross section of a Bragg fiber in which the cladding is made up of multiple periodic dielectric layers. **b** Cross section of a PCF in which a large number of small holes are patterned around a larger central core

Fig. 4.

Representations of cross sections for wafer-bonded integrated waveguides. **a** A “leaky mode” waveguide formed from glass substrates, **b** a microchannel coated with a low index polymer, **c** a microchannel coated with reflective metal, **d** a microchannel surrounded by Bragg layers, **e** a microchannel surrounded by only a single pair of ARROW layers, and **f** two substrates coated with a nanoporous film and separated by a fluid filled gap

Fig. 5. Representations of integrated waveguides fabricated through etching nanoscopic features in silicon. **a** Cross section of a slot waveguide where the n_1 region could contain a liquid and would be around 100 nm wide. **b** Top view of a 2D photonic crystal array made by drilling nanoscale holes into silicon

Fig. 6. Cross section representation of a microchannel filled with two liquids (n_1 and n_2), kept separate due to laminar flow in an L^2 waveguide. The n_3 region is PDMS

Fig. 7. Cross section representation of a hollow microchannel formed by the buckling of dielectric layers on the surface of a substrate

Fig. 8. Fabrication steps used to create hollow ARROW channels based on removal of a sacrificial core and conformal PECVD deposition

Fig. 9. SEM images of hollow ARROW waveguides formed by the removal of sacrificial. **a** Aluminum, **b** SU-8, and **c** reflowed photoresist

Fig. 10. Cross section SEM of ARROW waveguide fabricated on a raised silicon pedestal. The *top* dielectric cladding layers are thus surrounded by air

Fig. 11.

A simple test platform using fluid filled ARROWs and solid core waveguides to direct light on and off the chip. Critical interfaces are indicated including the T-junction formed near the end of the fluid filled waveguide and the perpendicular intersection

Fig. 12.

Illustration showing how solid core waveguides can be formed by etching into the thick silicon dioxide layer used to coat hollow ARROWs. **a** A solid-core waveguide at the termination of a hollow-core waveguide, used for launching light down the hollow-core waveguide. **b** A solid-core waveguide perpendicularly intersecting a hollow-core waveguide, used for exciting the hollow-core waveguide at a single location

Fig. 13. SEM images showing solid-core/hollow-core interfaces. **a** End-coupling interface between standard solid waveguide and hollow ARROW waveguide. **b** Angle view of perpendicular intersection

Fig. 14.

a SEM view of the intersecting channels that form the key element of an electrophoresis separation system; the scale bar is 20 μm . The *arrow* indicates the direction of fluid flow. **b** Separation of FITC-tagged amino acids at 660 V/cm in a device having channels modified with UltraTrol Dynamic Precoat HR; peaks are: 1 glycine, 2 phenylalanine, 3 FITC, and 4 arginine

Fig. 15.
a Illustration of an on-chip electroosmotic pumping device using closed channels formed from sacrificial etching. **b** Top view SEM showing small diameter microchannels intersecting a large diameter microchannel to form the pump

Fig. 16. Schematic diagram of a lab-on-a-chip system using optofluidic waveguides to perform three parallel capillary electrophoresis separations

Table 1

Summary of fiber based optofluidic waveguides

Waveguide description	Hollow core dimensions	Reported loss measurement	Wavelength used for testing	Tested in liquid or air	References
Glass fiber, high index liquids	75 μm diameter	2×10^{-4} dB/cm	840 nm	Liquid	Stone (1972)
Glass fiber, reflections at outer air-glass interface	340–215 μm diameter			Liquid	Tsunoda et al. (1989)
Teflon® AF	250 μm			Liquid	Altorn et al. (1997a), Dallas and Dasgupta (2004), Dasgupta et al. (1998), http://www.biogeneral.com/teflon.asp
Glass coated externally by polymer	530 μm			Liquid	Altorn et al. (1997b), Wang et al. (2001), http://www.polymicro.com/products/opticalfibers/products_opticalfibers_fsu_flu.htm
Glass coated internally by polymer	360–4,000 μm			Liquid	Dreß and Franke (1996), Schelle et al. (1999)
Glass coated externally by metal	75 μm			Liquid	Wang et al. (1991)
Glass coated internally by metal	250 μm	0.004 dB/cm	800 nm	Air	Mohebbi et al. (2002)
Bragg fiber	275 μm	6×10^{-4} dB/cm	1,550 nm	Air	Fink et al. (1998), Temelkuran et al. (2002), http://www.omni-guide.com/ , Pone et al. (2006), Gopal and Harrington (2003)
Photonic crystal fiber	5–20 μm			Liquid	Knight (2003), Cox et al. (2006), Jensen et al. (2004), Martelli et al. (2005), Rindorf et al. (2006), http://www.crystal-fibre.com/products/airguide.shm , http://www.newport.com/Photonic-Crystal-Fibers/323352/1033/catalog.aspx

Table 2

Summary of integrated optofluidic waveguides

Waveguide description	Hollow core dimensions	Reported loss measurement	Wavelength used for testing	Tested in liquid or air	References
Leaky channels in glass	30 $\mu\text{m} \times 60 \mu\text{m}$	1.4 dB/cm	840 nm	Liquid	Fouckhardt et al. (2001)
Teflon® coated silicon channels	500 $\mu\text{m} \times 180 \mu\text{m}$	1.09 dB/cm	660 nm	Liquid	Datta et al. (2003)
Metal coated silicon channels	250 $\mu\text{m} \times 50 \mu\text{m}$	<0.1 dB/cm	1,550 nm	Air	Jenkins et al. (2003)
Wafer bonded Bragg waveguides	1.3 $\mu\text{m} \times 1.2 \mu\text{m}$	1.7 dB/cm	1,550 nm	Air	Lo et al. (2004)
Wafer bonded ARROW's	150 $\mu\text{m} \times 150 \mu\text{m}$			Liquid	Campopiano et al. (2004), Bermiñi et al. (2007)
Nanoporous cladding on glass	50 μm gap			Liquid	Risk et al. (2004)
Slot waveguides	100 nm $\times$ 300 nm			Air	Xu et al. (2004), Dell'Olio and Passaro (2007)
Planar photonic crystals	200 nm voids in cladding			Liquid	Chow et al. (2004), Skivesen et al. (2007), Erickson et al. (2006)
L ² s	100 $\mu\text{m} \times 100 \mu\text{m}$			Liquid	Wolfe et al. (2004, 2005), Brown et al. (2006), McDonald and Whitesides (2002)
Buckled dielectrics	20 $\mu\text{m} \times 3 \mu\text{m}$	15 dB/cm	1,550 nm	Air	DeCorby et al. (2007)
Planar integrated ARROW's	10 $\mu\text{m} \times 4 \mu\text{m}$	1.1 dB/cm	633 nm	Liquid	Yin et al. (2004, 2005a, b, 2006a, b), Barber et al. (2005, 2006a, b), Duguay et al. (1986), Hubbard et al. (2005), Lee et al. (2007), Schmidt et al. (2006), Hawkins et al. (2007)