Microstructures made in optical fiber with femtosecond laser

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Different types of microstructures including microchannels and microslots were made in optical fibers using femtosecond laser inscription and chemical etching. Integrated with UV-inscribed fiber Bragg gratings, these microstructures have miniature, robustness and high sensitivity features and have been used to implement novel devices for various sensing applications. The fiber microchannels were used to detect the refractive index change of liquid presenting sensitivities up to 7.4 nm/refractive index unit (RIU) and 166.7 dB/RIU based on wavelength and power detection, respectively. A microslot-in-fiber based liquid core waveguide as a refractometer has been proposed and the device was used to measure refractive index, and a sensitivity up to 945 nm/RIU (10⁻⁶/pm) was obtained. By filling epoxy in the microslot and subsequent UV light curing, a hybrid waveguide grating structure with polymer core and glass cladding was fabricated. The obtained device was highly thermal responsive, demonstrating a linear coefficient of 211 pm/°C.

Keywords: ultrafast laser; optical fiber; fiber Bragg grating

1. Introduction

Recently, microfluidic devices have attracted considerable interest due to their unique advantages in detection and monitoring costly and difficult-to-obtain biochemical or biomedical samples/reagents, such as proteins, enzymes, DNA, etc. Their applications have covered a wide range of measurements including cell manipulation and sensing, flow cytometry, immunoassays, DNA analysis and so on [1]. Conventional microfluidic devices are usually fabricated using lithography or molding methods. However, depending on materials being used and applications, all these methods have their limitations. Recently, a newly emerged technique, femtosecond (fs) laser-assisted chemical etching, has received much attention since it provides a much easier and more efficient way to micromachine silica/glass-based materials to make microfluidic devices [2–6]. With the advantage of high spatial resolution and three-dimensional (3D) translation capability inherent to the fs radiation energy deposition system, arbitrary microstructure shapes and sizes could be defined by programming the movement of the focused fs-laser beam in the material. It was reported that patterned areas by the fs-laser exhibit a remarkable high etching rate compared to the pristine material with contrast ratio up to 100:1 [5]. The combination of fs-laser patterning and chemical etching could be an effective technique for engraving microstructures in a wide range of optical materials without using traditional cumbersome
lithography. However, most reports so far have been concentrating on bulk or planar substrates and few considered the integration with optical detection. Silica glass materials are predominant optical materials and used for many optical/photonic devices. It is thus very intriguing to integrate the microfluidic and the optical device in optical fibers and waveguides. In such structures, the fiber or the waveguide constitutes the backbone for delivery of input/output light and effective light–fluid interaction can be achieved when the microfluidic channel intercepts the optical pathway. With this respect, such hybrid microfluidic/optic structures could represent themselves as devices of low cost, miniature size and high sensitivity for sensing bio-reactions, especially for in situ synchronized delivery of the reagents. Fiber Bragg gratings (FBGs) made in chemically etched [7,8] or mechanically side polished [9,10] optical fiber and microstructured fiber [11] have been reported as refractive index based optical biochemical sensors using evanescent wave [7–11], thus they present themselves as ideal candidates for this integration.

In this paper, the development of microstructures in optical fibers fabricated using fs-laser and their sensing applications are reported. In Section 2, we show how the hybrid microchannel/FBG structures were fabricated in optical fibers using fs laser-assisted chemical etching. In Section 3, we demonstrate the liquid refractive index sensing using the integrated microfluidic channels with FBGs. In Section 4, we explore the concept of hybrid waveguide and its usage for sensing liquid of high refractive index and a hybrid waveguide grating structure with a polymer core and a glass cladding. Finally in Section 5, the work will be concluded with discussion for future exploitation.

2. Fabrication of hybrid microchannel/FBG devices

The fabrication process for microchannel devices in optical fiber involves two main steps: (1) fs-laser patterning of designed microfluidic channel structures in the fiber using focused high power pulses; (2) chemical etching of the patterned fiber samples to transform the pre-UV-inscribed FBG structures into microfluidic channels.

2.2. Micro-channel patterning using high power fs-laser pulse

Figure 1 shows the schematic of the fs-laser inscription system. In the fs-laser patterning process, the fs pulses (center wavelength = 800 nm) were focused onto the fiber by a ×100 objective lens with a NA of 0.55 and a working distance of 13 mm. The laser pulse width

Figure 1. Schematic of the fs inscription system.
was about 110 fs with repetition rate of 1 kHz. The focused spot size was \( \sim 1.5 \mu m \) and the pulse energy on the fiber can be tuned down to 10 nJ. The fiber was mounted on a dual-axes air-bearing translation stage so that the desired modification structure can be written by translating the fiber with respect to the fs-laser beam.

For an initial trial, the fs-laser inscription was performed on the fiber without FBG structure. Initially, the focus of the laser beam was outside of the fiber and then the fiber was moved in at 10 \( \mu m/s \) along the beam direction. A single-pass exposure over a distance much larger than the fiber diameter was carried out to ensure the inscription process covered the entire cross-section of the fiber. With the beam focus running through the whole diameter of the fiber, we presume a straight-line track transversely through the fiber core would be left. An optical microscope of high resolution was then used to visually inspect the inscribed feature within the fiber. As shown in Figure 2a, the fs-laser induced index modification is clearly visible, but it only covers half of the fiber section plane and a more dispersed modification was observed at the inner end (close to the core the fiber). The fs-laser inscription process was repeated on a new fiber sample whereby the fiber was translated in the reverse direction and a similar half-track feature was seen on the fiber. This clearly indicates that the beam expands as it approaches the fiber core and subsequently fades away as it penetrates further into the fiber. This defocusing effect is attributed to the cylindrical shape of the cladding which produces a distortion to the focus of the fs-laser beam, reducing the inscription efficiency across the fiber.

In order to eliminate the defocusing effect, a simple approach was adopted whereby the fiber surface geometry, presented to the path of the incident fs-laser beam, is rectified by using a closely-attached microscope glass slide (thickness = 125 \( \mu m \)) with index matching liquid, as shown in Figure 3. Conceptually, this is similar to the case when an oil-immersion lens is used, rectifying the fiber curved surface and alleviating the defocusing-induced distortions. A new inscription was made using this arrangement. Figure 2b shows the image of an undistorted straight-line track cross the whole fiber, indicating that the fs-beam can now remain focused as it penetrates the fiber.

With a focused fs-beam and the fine-controlling of the two-dimensional translation stage, microstructures of various geometries could be made in the optical fiber. Apart from a straight line, we show in Figure 4 three other patterns of microchannels made
Figure 3. Schematic of using an adaptive glass slide and index matching gel to eliminate the defocusing effect of the cylindrical shape of the fiber cladding.

Figure 4. Images of engraved microfluidic channels in optical fiber. (a) 2 × 250 μm dual-U-shape channel. (b) Dual-line channels of diameter of only ∼4 μm, with separation between lines is 500 μm. (c) Image of cross-section of the microslot, taken at the top of the slot channel and (d) taken from the middle at the same level to the core; note, the fringes of the FBG are also visible.

in the optical fiber. The three designed microchannel profiles include a dual-U-shape of 2 × 250 μm long, a dual-line across the fiber separated by 500 μm and a 125 × 500 μm slot in parallel to the fiber axis. It should be noted that all these structures were made overlapping with a FBG in the fiber.

2.3. Microchannel engraving with HF etching

Post fs-laser inscription, the FBG fiber samples with patterns for the microfluidic channels were chemically etched in a 5% HF acid solution assisted by an ultrasonic bathing to enhance the penetration of HF acid into the etched structures. In the meantime, the transmission of the fiber was monitored by an optical spectrum analyzer and a broadband source. Figure 5a plots the transmission of the FBG with the dual-U-shape channel against
3. Refractive index sensing

Since the microchannels can be positioned closely around the fiber core containing the FBGs, the mode field would interact with the filling agent in the channels, instigating disturbance to the mode properties and thus affecting the transmission spectra of the FBGs. We evaluated their responses to SRI and characterized them in terms of sensitivity and spectral change using index matching liquids with indices from 1.33 to 1.473. When the device was submerged in a set of index liquids, both the transmission and the reflection spectra were recorded. Figure 6 shows the transmission spectra of the FBG integrated with the dual-line micro-channel as shown in Figure 4b. As the etched hole is only 4 μm wide, the two narrow channels act as two phase shifts in the FBG structure and multiple resonances can be seen in the transmission spectrum as shown by the bottom trace in Figure 6a. This device was then immersed in the index matching liquids with different indices. In the experiment, we observed that when SRI increased, the multiple resonances shifted...
towards longer wavelength side. As the SRI approaching to the index of the fiber core, the phase shift effect induced by the holes was reduced accordingly, resulting in decreasing of the visibility of the resonances. Eventually, the resonances completely vanished and a spectrum of a single broad reflection peak was resumed, as shown in the middle trace of Figure 6a. Figure 6b plots the SRI-induced shifts of two resonant peaks in Figure 6a when the index was changed from 1.33 to 1.473. Both curves are nonlinear and the sensitivity increases for larger SRI. The maximum SRI sensitivity estimated from the curve is about 7.4 nm/refractive index unit (RIU), which is not particularly high compared to that of FBGs with the whole cladding being etched off [7,8]. However, the sensitivity could be improved with optimized microchannels, allowing for enhanced overlapping between the mode field and the liquid. More importantly, the integration of microchannels on the FBG will not just provide high SRI sensitivity but also retain the robustness of the whole device.

The presence of a microchannel within the fiber core introduces a scattering loss to the propagating core mode, whereas, on the other hand, the refractive index of the material infused in the channel determines the amount of the scattering, leading to a variation to the transmitted (or reflected) power. Therefore, measurement of optical power is another means to detect SRI change. Figure 7a shows the reflection spectra of an FBG integrated with the dual-U-shape microchannel for different SRIIs, clearly showing that the reflected power of the FBG increases with larger SRI. Figure 7b plots the reflection (the peak around 1536.9 nm, with the resolution of the optical spectrum analyzer set at 0.1 nm) with respect to SRI and an 11 dB difference is apparent for SRI from 1.33 to 1.44, giving an average sensitivity of 166.7 dB/RIU.

4. Hybrid waveguide using a microslot

4.1. Liquid core fiber Bragg grating

The measurement discussed in the previous section was carried out for SRI below that of the optical fiber and similar to other types of optical-fiber-based devices, the resonance peaks suffer from oscillation and diminishing when SRI is larger than that of the fiber. The

Figure 6. (a) Transmission spectra of the FBG with the dual-line channels: top trace – before etching; bottom trace – after etching and submerged in liquid of SRI = 1.339; middle trace – showing an elimination of the phase shift effect at SRI = 1.473 and recovered back to single reflection peak. (b) SRI sensitivity plots of two central resonant peaks, showing a nonlinear characteristic and the peak at shorter wavelength exhibiting slightly high SRI sensitivity.
microslot based liquid core device described in this section employs the liquid as the core of a waveguide and allows for measuring SRI beyond this limit.

Figure 8 shows the geometry of the microslot engraved along an FBG. With an opening on both sides, the liquid needs only to travel the radius of the fiber to fill the slot and form the liquid core slab waveguide. Here we use the liquid core waveguide as a refractometer to test SRI changes. To maximize the interaction between the optical signals propagating in the core and the liquid, the slot was purposely fabricated across the core. With commercial software, FEMLAB, we simulated its performance. Figure 9a plots its effective index against SRI. In the simulation, the radius and refractive index of the fiber are 62.5 μm and 1.44403 for the cladding and 4.15 μm and 1.4512 for the core, respectively. For a given slot, two regions with very different slopes can be seen with a changeover around the index of the fiber material. By examining the evolution of the field distribution (Figure 9b) with respect to the SRI, we can gain a clear understanding of the difference between these two regions. In the low SRI region, light is still confined in the glass and only evanescent wave penetrates into the liquid, while for the higher SRI region, the light exists mainly in the liquid and the effective index is dependent on the liquid. The influence of the opening size of the slot is also illustrated in Figure 9a and larger slot presents a higher sensitivity to SRI.

A microslot similar to Figure 4c was made along a 500 μm long FBG with an open slot height of ~2 μm only. The response of the FBG/microslot device to SRI was tested with a series of index matching liquid by immersing the device in the liquid and the reflection spectrum was monitored with an optical spectrum analyzer. After the sample was measured
for each liquid, the device was rinsed with acetone, methanol and water in turn for several times until the original spectrum was restored, making sure no residue liquid was left in the slot. Shown in Figures 10a and 10c are the spectra of the grating for successively increased SRI. In agreement with simulation results, two distinguishable regimes can be seen with a boundary around an index of 1.46. Initially, only the main Bragg peak appears while for SRI larger than 1.46, new reflection peaks emerge at longer wavelengths as marked by arrows in Figure 10c. No reflection from the fiber end was noticed because the index difference between the liquid and the fiber was so small.

These two regimes can also be distinguished by a close examination of the main Bragg peak around 1460 nm. Figure 10b gives its response to a variation of the SRI in terms of its wavelength and the strength. In the low SRI region, both change almost monotonically with a trend similar to previous reports [12,13] and the sensitivity reaches 116 nm/RIU (or $8.6 \times 10^{-6}$/pm) for SRI changing from 1.44 to 1.46. Once in high SRI regime, the scenario changes substantially, showing obvious less reflectivity and unpredictable wavelength shift. In this regime, the SRI is larger than that of the fiber, and thus a considerable part of light will be coupled to the liquid making the liquid a guiding part of a complex waveguide, which consists of the fiber cladding, the fiber core and the liquid. For one mode, the fiber core and liquid will serve as the core with the majority of the mode field still dwelling in the glass region since the liquid slab is very thin. The coupling from upstream fiber to this mode is large because it is basically a minor modification to the original fundamental mode. The peak around 1460 nm should arise from this mode and the influence by the liquid is small.

In the high SRI regime, the liquid in the slot begins to support propagation modes with fiber glass as its cladding and the number of modes supported by this waveguide depends on the index of the liquid, as indicated by the increasing number of peaks that emerge in the reflection spectrum in Figure 10c. The rightmost peak belongs to the fundamental mode since its effective index is the largest. Several wavelength bands were scanned using an optical spectrum analyzer set at high resolution (0.05 nm) and then concatenated to decrease the noise and ensure no visible peaks for a quite wide wavelength range and the obtained rightmost peak is accurate. It can be seen that, with increasing SRI, the peak gradually becomes broader with full width half amplitude changed from 1.6 nm for SRI
of 1.478 to 5.9 nm for 1.55. At the same time, the strength of the peak decreases, caused by the decreasing coupling from the un-etched fiber section. This could be improved by implementing a round tunnel in the central portion of the slot with the same fs inscription and chemical etching method and it is possible to realize a single mode liquid core waveguide.

More significant is the wavelength shift of the peak with changing of SRI as revealed by the linearly aligned hollow diamonds in Figure 10d. Using simulated effective indices for a 1 μm slot of Figure 2a, the Bragg wavelengths were also calculated and the results shown
as blue squares in Figure 10d exhibit excellent agreement with the experiment in terms of the slope. The offset may arise from the dispersion of the liquids since their indices are given for visible light instead of the infrared. Theoretically the turning point from the low to high SRI regime is around the refractive index of the fiber core, i.e. 1.45 (Figure 9a), whereas we observed the turning point in the experiment around 1.46. This difference could be caused by the dispersion of the liquid from the visible wavelength to the infrared. With this amending, better overlaps with the experimental results were obtained, as shown by red stars in Figure 10d. Evaluation of another device which used a 1550 nm FBG was also performed and the results are included in Figure 10d, with the average sensitivity of 945 nm/RIU (1.0 × 10⁻⁶/pm). Figure 10e gives the sensitivity for the Bragg peak of the fundamental mode in low and high SRI regimes. Apparently, much higher sensitivity is shown in the high SRI region.

4.2. Polymer core hybrid waveguide grating

As explained, with high refractive index material filled in the slot, a hybrid slab-waveguide with the filling material as its core and the glass as its cladding can be produced. In this section, we fill the microslot with polymer, which not only presents higher refractive index than the glass but can also undergo transition from liquid or viscous amorphous state to solid state, making them superb candidates as the filling material in the hybrid waveguide. Though a range of polymers can be used for filling, we used the UV-curable epoxy for fiber recoating (Desolite 950-200 from DSM Desotech Inc) for the initial work and cured it with a fiber recoater machine (Vytran RC-100). Figure 11a shows the reflection spectrum of the grating at different stages of the experiment. Before infusion, there was only one main Bragg peak at 1548 nm, while after the epoxy was filled, a new peak was noticed appearing at longer wavelength around 1583 nm, arising from the newly formed hybrid waveguide. The evanescent wave of light confined by the hybrid waveguide can extend to the neighboring glass material, where the periodic grating structure still remains (only 1–2 μm of the core was used for the slot), generating Bragg reflection of the hybrid slab waveguide. The spectrum of the slab waveguide grating was then simulated with a commercial software IFO (Optiwave Inc.), disclosing 1.51 for the refractive index of the epoxy. After the epoxy was cured by the fiber recoater, the peak further moved to 1599 nm. This further shifting suggests increment of the refractive index of the epoxy when it turned into solid state, which turned out to be 1.53 by the simulation. At this moment, due to the large difference between the core and cladding, light was so well confined in the polymer core region that the evanescent wave in the glass became negligible and the influence of the grating was much reduced, leading to a much weakened Bragg peak as demonstrated by the top spectrum in Figure 11a.

The thermal response of the in-fiber hybrid slab waveguide grating was then investigated. Figure 11b shows the spectral evolution of the Bragg grating for successively increased temperature. As can be seen clearly, the Bragg peak moves to shorter wavelength as the temperature is increased, indicating decreased refractive index of the epoxy. In the mean time, the peak becomes stronger, caused by more portion of the light in the glass cladding since the smaller difference of refractive index between the glass and the epoxy. Figure 11c gives the variation of the Bragg wavelength with respect to the temperature, revealing an almost linear relation with a coefficient of 211 pm/°C. In comparison with FBGs in polymer optical fiber, the hybrid waveguide grating shows the capability to demodulate variation of temperature not only by means of wavelength shift but also by the change to the amplitude of the Bragg peak. Moreover, with the large but negative thermal
response compared to the normal FBG in glass, which can still be found at the same location, the hybrid waveguide grating demonstrates great promise in implementing compact and thermal-compensated sensors. The strain response was also tested and the result is shown in Figure 11d, showing an almost linear relation with a coefficient about 1.3 pm/με, similar to that of FBGs in polymer optical fiber [14].

5. Conclusions

In conclusion, we have reported the design and fabrication of in-fiber microfluidic/optic integrated devices utilizing UV-inscribed FBGs and microchannel structures realized by fs-laser patterning and chemical etching. FBG based microchannel devices have been used to measure SRI change based on FBG wavelength shift detection and achieved maximum sensitivity of 7.4 nm/RUI and 166.7 dB/RUI in the refractive index region around that of the fiber. The microfluidic channel clearly sensitizes the FBG structure to SRI as it effectively introduces the interaction between the fluid and the evanescent field of the FBG. Hybrid waveguide gratings are proposed based on microslot through the fiber and they can be used to sense SRI. Simulation results show the device is effective in a broad SRI range and presents improved sensitivity in large SRI region. Compared to evanescent wave based refractometers, the sensing refractive index range of the device is extended to 1.55
and sensitivity up to 945 nm/RIU (or $1.0 \times 10^{-6}$/pm) is achieved. By filling the slot with epoxy and subsequent UV-curing, a hybrid slab waveguide grating structure with a polymer core and a glass cladding was achieved. The device was characterized in terms of strain and thermal response, giving linear coefficients of 211 pm/°C and 1.3 pm/µε, respectively.

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