Microbubble-Based Fiber Optofluidic Interferometer for Sensing

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***Abstract*—A fiber optofluidic interferometer based on an optical microbubble-on-tip (μBoT) structure is developed. The generation process and sensing mechanism of this μBoT sensor are very different from traditional optical fiber sensors. The μBoT is a hybrid solid/liquid/gas microstructure generated by heating a fiber tip with laser and can be easily regenerated with low cost and good repeatability. Carbon nanotube film is optically deposited on the fiber end face to increase the laser absorption, thus enhances the efficiency of the μBoT generation. The diameter of the μBoT interferometer increases with time, leading to a decrease in the free spectral range (FSR). By measuring the FSR, the temperature and flow rate sensing are demonstrated. A temperature sensitivity of -1146 pm/°C is achieved, which is two orders of magnitude higher than that of the widely used fiber Bragg gratings. A lower limit of detection of 10 nL/min and a resolution of 0.03 nL/min for the flow rate sensing are obtained, which is better than that of the state-of-the-art microfluidic flowmeters. Our study will open a door to the development of novel reconfigurable fiber optofluidic sensors.**

***Index Terms*—Fiber optic sensors, interference, optofluidics.**

I. Introduction

TRADITIONAL optical fiber sensors (OFS) such as fiber Fabry-Perot (FFP) [1] and fiber Bragg grating (FBG) sensors [2] were invented decades ago and have been fabricated in optical fibers by various micromachining technologies [3]–[5]. In addition, various materials [6], [7] were incorporated with optical fibers to enhance the sensing performance or to extend the versatility. However, all these OFS were based on pure solid materials, which inherently have drawbacks of low flexibility and are lack of reconfigurability.

Combining optofluidics [8]–[11] with optical fiber sensors leads to a new research topic, i.e., the so-called fiber optofluidic (FoF) sensors [12], [13]. Previous efforts were made on the FoF sensors by using conventional OFS to detect analyte in the microfluidic environment. Recently, people started to develop FoF sensors by employing the unique properties of the non-solid materials for reconfigurability [11]. Bykov *et al.* developed a flying particle sensor by optically trapping a single microparticle in air within the hollow-core photonic crystal fiber [12]. Gong *et al.* demonstrated a sensitive FoF flow rate sensor by using a trapped microparticle in water [13]. Based on the non-solid materials and optical forces, these sensors were entirely different in operating principle from traditional OFS.

Recently microbubbles with solid shell were developed by precise fusion splicing method and very high sensitivity for strain sensing was demonstrated [5]. Sumetsky *et al.* also reported a solid microbubble resonator based on a micro capillary [14]. The device cannot be changed once it was produced. In this paper, we proposed and demonstrated a reconfigurable fiber optofluidic sensor based on a gas microbubble in liquid, which was generated by laser heating on a fiber tip (μBoT) in the microfluidic environment. Our studies show that the μBoT generation is simple, highly reproducible, and of low cost. The μBoT acts as a micro-interferometer, which can be used to detect temperature and flow rate with superior performance.

II. Experimental Setup and Principle

Experimentally, two laser beams were delivered to the same fiber tip through a wavelength division multiplexer (WDM). A heating beam at 980 nm was employed to heat microfluid and generated the microbubble on the fiber tip. The laser power was tunable from 0 to 300 mW. A tunable laser from an optical spectrum analyzer (OSA) (OPT162, 1505 nm–1630 nm, Agilent) was used to monitor the changes of the reflective interference spectra that resulted from the evolution of the microbubble. The interference occurred between the Fresnel reflections from the solid/air surface and the other from the air/liquid surface.

Even though a bare single mode fiber (SMF) tip is sufficient to generate a μBoT, a large heating laser power is required. To reduce the power, a carbon nanotube (CNT) film was deposited on the fiber tip to increase the absorption of pump laser, thus easing the μBoT generation [15], [16].

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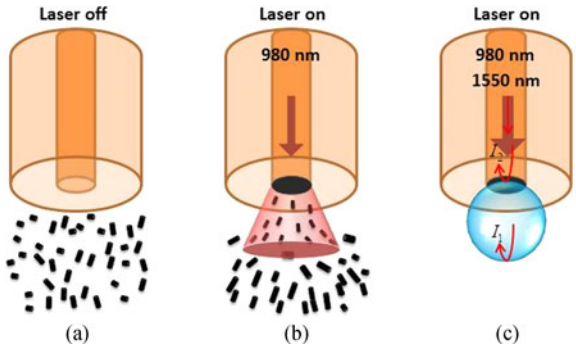

Fig. 1. Formation of the fiber optofluidic microbubble-on-tip sensor. CNTs were randomly distributed in solution with 980 nm laser switched off (a) and can be deposited near the fiber core area with the laser on (b). The μBoT structure, acting as a micro-interferometer, can be generated with a laser pump at 980 nm (c), while using a probe beam at the 1550 nm band for spectral analysis.

We chose CNT because it has a low conductivity of $\sim$1.52 W/(m·K) in the radial direction and also a low specific heat capacity of 0.7 J/(g·K) [17], [18], which are helpful for increasing the absorption of pump laser and introducing a large temperature increment for the μBoT generation. The coating process is simple, as described below. First an SMF was cleaved, washed with DI water, and dried under an ultraviolet (UV) irradiation (90 mW/cm^2) for 20 min. Then the fiber tip was inserted into the CNT solution 2 cm below the solution surface. The CNT working solution was obtained by diluting 18 μL of CNT stock solution (9.0 w.t.% multiwall carbon nanotubes (MWNTs) and 1.8 w.t.% dispersant TNWDIS, Timesnano Inc.) in 10 mL of deionized (DI) water. At the beginning, the CNTs in the solution were randomly distributed, as illustrated in Fig. 1(a). Then a continuous-wave (cw) 980 nm diode laser was switched on with a power of 20 mW and the CNTs were coated on the fiber tip [19] (see Fig. 1(b)) for 15 min. While keeping the laser on, the fiber tip was pulled out of the CNT solution at a slow speed of 0.01 mm/s and the residual solution on the tip was air-dried to solidify the CNT film and to improve the stability of the film for sensing. The efficiency of the deposition was improved during the pulling out process due to the local thermal effect near the surface of the solution [20].

The coated fiber tip was inserted into a microfluidic channel with a cross-section of 300 μm $\times$ 300 μm, in which the deionized (DI) water was flowed through. When the laser power was absorbed by the CNTs, a sharp temperature rise was generated in a very low volume near the fiber tip. A microbubble was generated thanks to the increment of the gas content around the fiber tip, either from the separation of dissolved gas from the liquid or from the vaporization of the liquid as the temperature increased (see Fig. 1(c)). Then the microbubble kept on growing if the pump laser power was sustained. The growth rate of the microbubble would change when the ambient parameters change. The FSR of the microbubble at a fixed heating time was used as the sensing signal. For temperature sensing, the gas for microbubble mainly came from the vaporization of the liquid, similar to boiling the water by heating. For flow rate sensing, the gas mainly came from dissolved gas delivered together with the microfluid and can be refreshed as the fluid flows. High

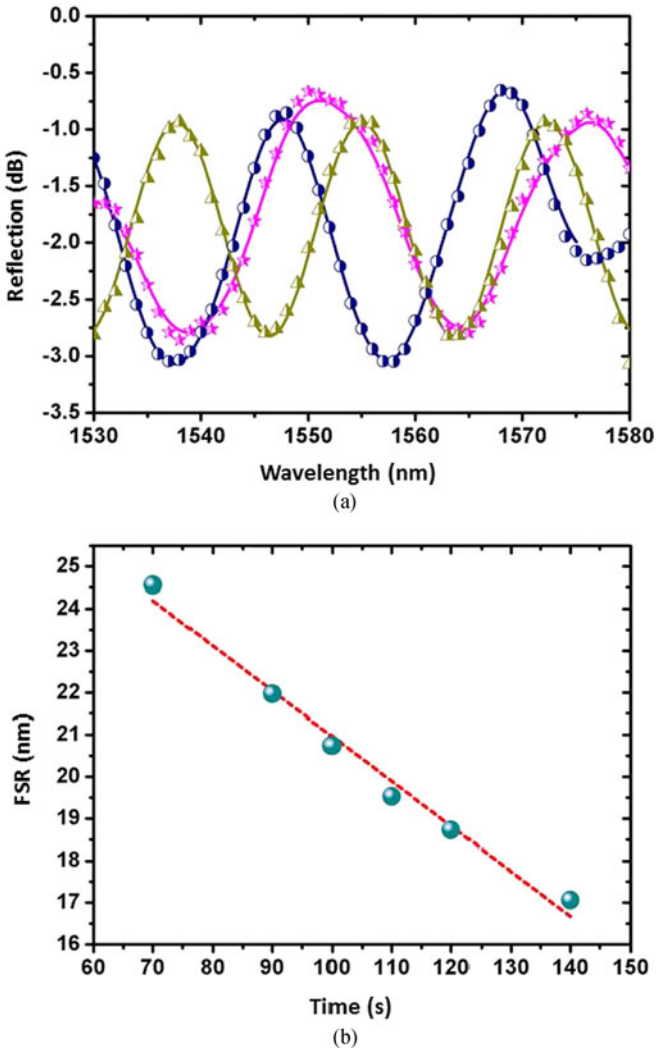

Fig. 2. (a) The interference spectra of the fiber optofluidic microbubble-on-tip structure and (b) FSR versus heating time.

sensitivity was achieved thanks to the photothermal effect of CNT and the small volume of the microbubble.

The reflective spectra from the μBoT structure at heating time of 70 s, 100 s and 140 s are shown in Fig. 2(a), while the ambient temperature was kept constant. The laser power for heating was 221 mW. The fringe contrast was not as high as solid fiber micro-interferometers, but was sufficient to determine the free spectral range (FSR) of the interference spectra. A compromise between increasing the absorption by CNTs and keeping good interference fringes should be considered during the CNTs deposition. The FSR decreases with a good linearity ($R^2 \sim 0.98$) as the microbubble grows in time (see Fig. 2(b)).

The diameter of the microbubble was also monitored by a charge-coupled device (CCD) camera. An example of the temporal evolution of the microbubble is shown in Fig. 3. Three microscopic images of the microbubbles, corresponds to that of the spectra in Fig. 2(a), are shown in the inset of Fig. 3. There was a sharp nonlinear increase in diameter at the beginning because a fast temperature rise was obtained when the laser was switched on. Then the diameter increased with time with a good

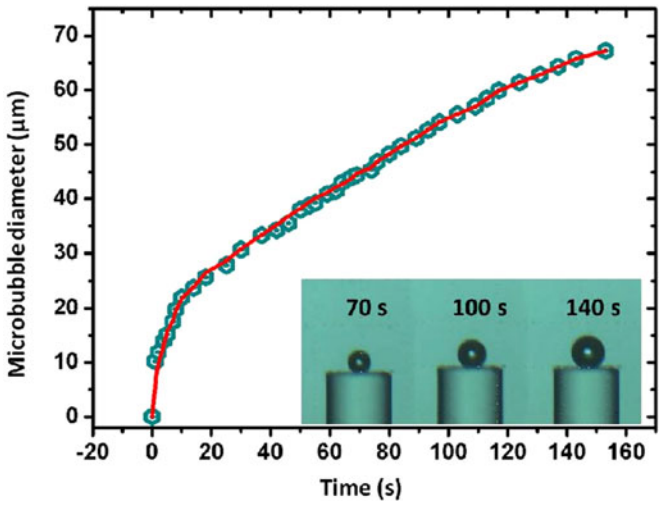

Fig. 3. The generation of the fiber optofluidic microbubble-on-tip for 150 s.

linearity ($R^2 \sim 0.99$) between 50 s and 150 s. When the diameter was large, the temperature rise became slow and gradually got balanced. Since the FSR changed linearly over certain range of heating time, detecting FSR in this time range would not influence on the calibration curve for sensing.

The FSR can be expressed by $FSR = \lambda_1\lambda_2 / (2nL_{eff})$, with n and L_{eff} the refractive index and the effective length of the microbubble interferometer, respectively. The FSR is inversely proportional to the diameter of the microbubble. Therefore, in principle, measuring the FSR is equivalent to detecting the diameter of the microbubble by a CCD camera. For the μBoT-based sensing, the imaging detection method is more cost-effective, while the spectroscopic detection method has higher resolution. Here, we would employ the spectral detection method to achieve higher sensing performance. As the microbubble in our experiment grows with a relative slow speed, the measurement time of FSR, which can be determined easily within sub-100 ms level, can be neglected and will not influence the sensing performance.

III. Results and Discussion

Ambient temperature measurement was demonstrated by the μBoT sensor. The fiber was fixed with the microfluidic chip and the whole chip was immersed into a thermostatic water bath for temperature calibration. The microfluid did not flow in the temperature sensing experiment. At each temperature, the 980 nm laser was turned on with a power of 146 mW and the reflective spectra of microbubble was recorded after heating for 60 s. Fig. 4(a) shows the FSR changes as a function of the temperature. Temperature was measured between 25 °C and 45°C, which covers the temperature range of human body and is significant for the human-on-a-chip or organ-on-a-chip applications [21], [22]. Note that the temperature measured here is not only that of the microfluid, but the ambient temperature. In most microfluidic applications, the reagents are firstly warmed up to the room temperature so that the temperature of the microfluid is usually the same as the ambient temperature.

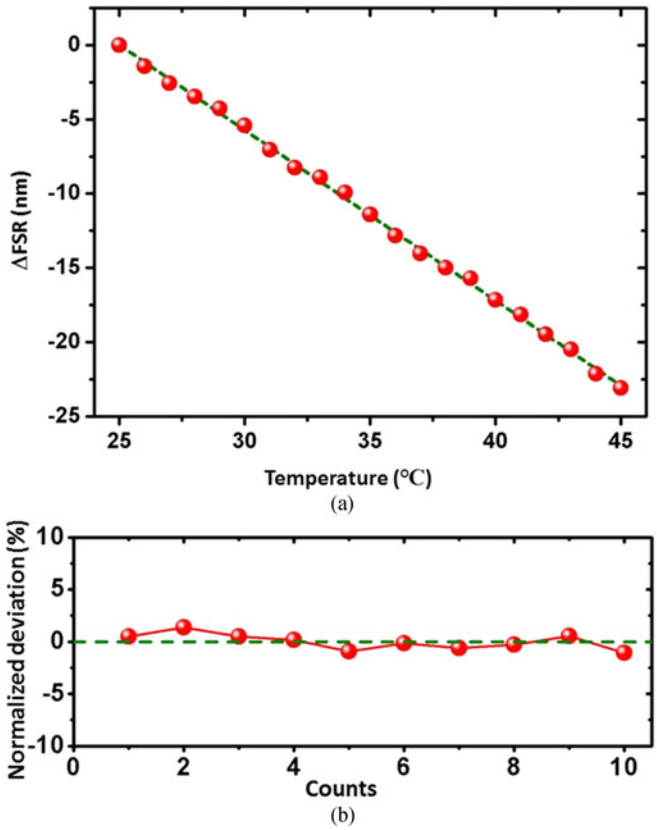

Fig. 4. (a) FSR changes versus temperature with a heating time of 60 s at 146 mW of 980 nm pump and (b) the repeatability of the same probe for 10 tests.

At higher temperature, the microbubble is larger so that the FSR is smaller.

A good linearity ($R^2 \sim 0.998$) is achieved and the sensitivity is -1146 pm/°C, two orders of magnitude higher than FBGs [23]. The sensitivity (δ) is determined by the slope of the linear fitting curve from data in Figs. 4(a), $\delta = \Delta y/\Delta x$. x is the parameter to be tested and y is the output signal, ΔFSR in this work. By using the same probe, good repeatability was confirmed by 10 times of tests at 25 °C. Between two tests, the pump laser was switched off to let the microbubble disappear. The FSR was statistically determined to be 29.358 ± 0.224 nm, corresponding to a deviation of ±0.76% (see Fig. 4(b)). The deviation of FSR was calculated by $D = (FSR - \overline{FSR})/\overline{FSR} \times 100\%$, where $\overline{FSR}$ was the statistically averaged FSR over 10 tests. The accuracy can be roughly evaluated by the statistical deviation of ±0.76%. The theoretical temperature resolution can be determined to be about 0.002 °C by the sensitivity of 1146 pm/°C and the spectral resolution of 2 pm. Then three different probes were tested for temperature sensing, indicating good consistency in the sensor's fabrication process (see Fig. 5). The fluctuation might be from the instability of CNT film after many times of tests, as well as the instability of the syringe pump and the microfluid, limiting the accuracy of the sensor. It can be improved by optimizing the deposition process, such as drying for a relatively long time after coating or improving the

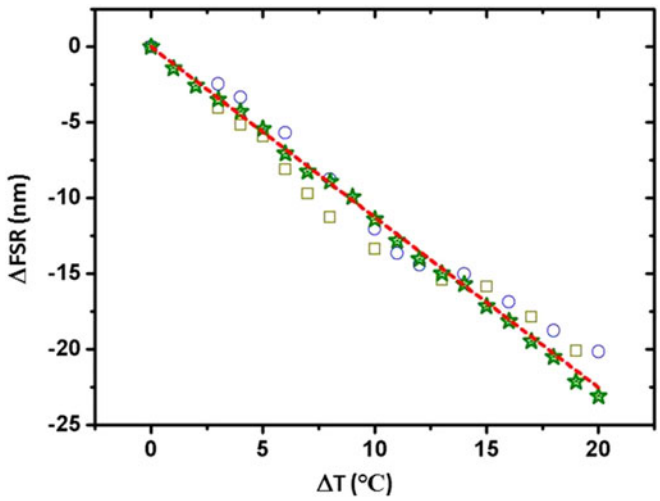

Fig. 5. Repeatability of the calibration curves for three different μBoT temperature probes.

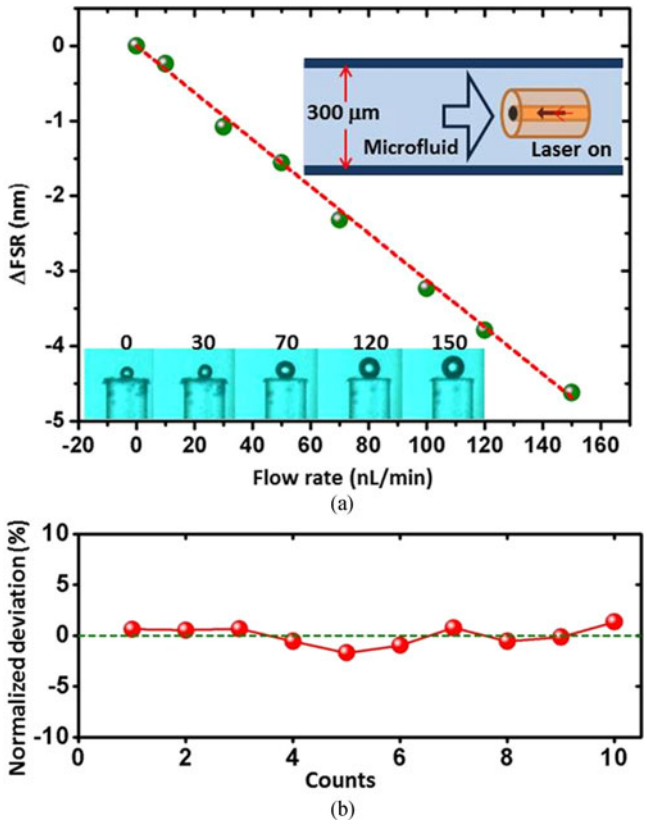

Fig. 6. (a) FSR changes as a function of flow rate with a heating time of 120 s at 221 mW of 980 nm pump and (b) the repeatability of the same probe for 10 tests.

adherence strength by an interlayer between fiber face and the CNT film.

For the microfluidic flow rate sensing, we used a syringe pump (Harvard Apparatus) to precisely control the microfluid, which flowed toward the fiber tip, as shown in the insert of Fig. 6(a). The stability of the syringe pump is very good so that the flow rate variation can be neglected. The fiber was adjusted to the center of the cross section in the microchannel. We used a pump power of 221 mW. The FSR, recorded at a heating time of 120 s, is shown as a function of the flow rate in Fig. 6(a). Flow rate of 0 – 150 nL/min was measured. The inset shows the microscopic images of μBoTs under 0, 30 nL/min, 70 nL/min, 120 nL/min and 150 nL/min, respectively. This flow rate range is important for the microfluidic applications such as separation-based sensing of neurotransmitters and certain quick analysis system [24], [25]. A good linearity ($R^2 \sim 0.997$) is achieved and the sensitivity is -31 pm/(nL/min), which is determined by the slope of the linear fitting curve. The spectral resolution of the OSA is 1 pm, so the theoretical resolution for the flow rate sensing can be calculated to be as low as 0.03 nL/min, which is much better than the state-of-the-art microfluidic flowmeters with resolution at nanoliter-per-minute level [13], [26]–[28]. 10 times of tests were performed with a flow rate of 120 nL/min and good repeatability was achieved, with a statistical deviation of $\pm 0.94\%$ (see Fig. 6(b)). The deviation is low and acceptable for sensing. The deviation may originate from the instability of the syringe pump or the non-uniformity of the dissolved gas in the microfluid.

In the flow rate sensing experiment, the microfluidic flow takes away part of the laser-generated heat, therefore, the microbubble was supposed to grow slower at higher flow rate. However, the opposite was surprisingly observed, that is, the microbubble grows faster at higher flow rate, leading to a smaller FSR at a fixed heating time (see Fig. 6(a)). The potential explanation is that the dissolved air in the liquid was delivered more efficiently at higher flow rate, leading to a faster growth of the microbubble. Therefore, the air solubility was initialized by ultrasonic bathing in both temperature and flow rate sensing. For practical use, the calibration needs to be performed at first by using the same sample to be test to make sure the air solubility is the same. The upper detection limit of flow rate was constrained by the maximum diameter of the microbubble, which was determined by the balance between the laser-induced heat, the surface tension and the fresh air supply from the microfluid flow.

Generally, the performance of the fiber optofluidic μBoT sensors can be further improved. First, enhancing the fringe contrast of the interference fringes is helpful to precisely determine the wavelength of interference fringes. The fringe contrast can be enhanced by polishing the fiber tip and thus getting a more uniform coating, and/or by using a solution with a large refractive index instead of DI water to increase the Fresnel reflection from the gas/liquid surface. Second, other materials instead of CNTs are worth being explored in order to further enhance the stability, repeatability, and also the lifetime of the sensor. Third, other sensing output, such as the diameter of the microbubble monitored by imaging, can be employed instead of measuring the FSR. Alternatively, the dynamic fringes from the μBoT sensor can also be measured by using a fixed wavelength laser and a photodetector [29], as the effective length of the μBoT changes much faster and larger than that of the traditional solid fiber micro-interferometers. At current stage, the μBoT interferometer was not ready for the simultaneous measurement of temperature and flow rate. Keeping the ambient temperature constant is a good way, since the FSR changes with both temperature and flow rate. Potential methods for dual-parameter

sensing include the sensitivity coefficient matrix method [30], [31], hybrid sensing structure with two mechanisms [32], etc.

IV. Summary

A novel type of fiber optofluidic microbubble-on-tip sensor has been developed and is much more sensitive than the traditional solid fiber-optic micro interferometers. By laser heating, the microbubble was generated in liquid with good reconfigurability and repeatability. CNT film was deposited on the fiber tip to enhance the laser absorption and thus improve the efficiency of the microbubble generation. Ultrahigh sensitivities of -1146 nm/°C and -31 pm/(nL/min) have been achieved for temperature and flow rate sensing, respectively. In addition to the sensing applications, the liquid-gas interface of the microbubble in liquid might have the potential to act as a chemical microreactor [33].

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