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Lab-on-tip based on photothermal microbubble generation for concentration detection

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ABSTRACT

Lab-on-fiber is an emerging technology using compact fiber microstructures smaller than their lab-ona-chip counterparts. In this paper, we report a lab-on-tip (LoT) technology for concentration detection based on a photothermal gas microbubble, which is generated by locally heating the fiber tip with a gold nanofilm. The temporal growth process of the microbubble is repeatable, and the parametric influence is investigated. The diameter of the microbubble is monitored by imaging, and its increment is detected as a function of concentration. The reflection spectrum of the microbubble as a micro-interferometer is recorded and the free spectral range (FSR) can also be used for concentration detection. Solutions of sucrose and hydrogen peroxide are used as examples to evaluate sensing performance. For sucrose detection, a dynamic range of 0.5 wt% to 50 wt% is obtained. For hydrogen peroxide detection, an unprecedented dynamic range of 5 orders of magnitude, 10^{-5} M \sim 1 M, is achieved. The demonstrated performance is exceptional and the method is simple and cost effective.

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1. Introduction

Lab-on-fiber (LoF) technology is a highly integrated, versatile sensing platform that uses optical fiber [1-3]. LoF has the advantages of miniature size, low cost, high sensitivity, etc., thereby making it well suited for a wide range of chemical and biological sensing applications. According to the structure employed, LoF can be divided into 3 categories: the lab-around-fiber, the lab-in-fiber, and the lab-on-tip (LoT) [4]. LoT uses the end-facet of an optical fiber as the substrate to integrate functional materials and/or micro/nano structures; it has attracted much attention for biochemical detection, especially for in vivo applications.

Previously, LoT structures were fabricated on the fiber tip by either *bottom-up* or *top-down* approaches [4]. The former includes technologies such as sol-gel processing [5], self-assembly (SA) [6,7], and electroplating [8], which are cost effective. The latter often employs microfabrication technologies such as electron-beam lithography (EBL) [9,10], focused ion beam (FIB) milling [11],

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http://dx.doi.org/10.1016/j.snb.2017.09.055 0925-4005/© 2017 Elsevier B.V. All rights reserved. and reactive ion etching (RIE) [12], which can fabricate fine periodic structures with high sensing performance, but at the expense of cost and fabrication process complexity. LoT can be operated by various mechanisms, including surface-enhanced Raman scattering (SERS) [13,14], whispering gallery mode (WGM) [15], and localized surface plasmon resonance (LSPR) [10]. To the best of our knowledge, the photothermal effects of the LoT applications have not been explored.

In this paper, we report an easy-to-fabricate and easy-to-use LoT sensor based on a gas microbubble growing on the fiber tip. A fiber-coupled laser was used for heating the fiber tip, and a gas microbubble was photothermally generated. A gold nanofilm was deposited on the fiber tip to enhance the efficiency of microbubble formation, thanks to the photothermal effect of gold [16–19]. The growth process was recorded by imaging, and the diameter change of the microbubble over a certain period was monitored as a function of the concentration. The concentration of the solution can influence the growth rate of the gas microbubble because of two factors. One is the evaporation of liquid near the fiber tip, and the other is gas generated from the heat-induced chemical decomposition. Solutions of sucrose and hydrogen peroxide (H₂O₂) were used as samples for testing, and the experimental results confirmed the high sensing performance, including a large dynamic range and high sensitivity. In contrast to solid microbubbles [15,20], the gas

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Fig. 1. The schematic structure and principle of the microbubble-LoT technology.



Fig. 2. (a) The temporal growth of the microbubble and (b) the diameter variations by absolute and relative measurements.

microbubble can be regenerated in liquid with good repeatability, which makes the microbubble reconfigurable and suitable for sensing applications. To the best of our knowledge, this study is the first to report the development of the microbubble-LoT for concentration detection. This technique has many advantages such as high sensing performance, ease of fabrication and use, and low cost.

2. Methods

2.1. Generation of microbubble-LoT

The process for fabricating the microbubble-LoT is simple. A single mode fiber with a diameter of 125 µm was cleaved and washed for 5 min using an ultrasonic cleaning machine. A gold nanofilm was coated on the fiber tip to increase the laser absorption and enhance the microbubble generation efficiency. The gold nanofilm was deposited by a compact, cost-effective sputter coater, which is often used for improving the image quality of scanning electronic microscope (SEM) samples. A sputter gun was installed on top of the chamber with a target of gold purity >99.99%. The prepared fiber tips were placed 5 cm under the target. The coating was performed at a working pressure of 5×10^{-3} Pa. The coating speed can be controlled by adjusting the sputter current between 0 and 30 mA. The average coating speed was approximately 0.1 nm per second with a current of 9 mA. By keeping the current constant at 9 mA, the thickness can be adjusted according to the coating time; here, we tried the coating time of 80 s and 160 s in the following experiments.

The schematic structure of microbubble generation is shown in Fig. 1. The coated fiber tip was inserted into the capillary with a square cross-section of $1 \text{ mm} \times 1 \text{ mm}$ and put at the center of the channel; this was filled with deionized (DI) water or other kinds of liquid samples under test. Gas dissolved in the liquid may accelerate the generation speed of the microbubble. Therefore, the dissolved gas was removed by processing the liquid under test in an ultrasonic bath for 15 min. The liquid was withdrawn into the capillary by using a syringe pump or through the capillary force. During the experiment, the liquid was kept static, i.e., without flow. A continuous-wave (CW), 1550 nm laser was coupled into the fiber and heated the liquid near the core of the fiber tip. There are several reasons for choosing the 1550 nm laser. First, 1550 nm is the common wavelength for optical fiber communication (OFC), so the laser, the optical fiber, and other devices such as fiber coupler are cost-effective and compatible with each other. Further, light at 1550 nm can be amplified with an Erbium-doped fiber amplifier (EDFA). Third, the absorption of water at $1550 \text{ nm} (10.9 \text{ cm}^{-1})$ is ~ 20 times higher than that at 980 nm (0.5 cm⁻¹) [21]. The latter is another frequently-used wavelength for OFC. Actually we tried similar experiments with a 980 nm laser, it did not work and no microbubbles can be generated, which means the total absorption of water and the nanofilm is higher at 1550 nm. On the other hand, it was much more difficult to generate microbubbles if no gold nanofilm was coated on the fiber tip. This observation confirmed that the gold nanofilm is helpful to further increase the absorption and to generate heat.

A microbubble can be generated on the tip and its diameter, *d*, increases with time, as shown in the inset of Fig. 1. The growth process was monitored using an inverted optical microscope (Model DSZ5000X, Novel Optics Inc., China) with an objective (10X, numerical aperture (NA): 0.25, working distance: 10 mm), as well as a low-cost CCD camera (310,000 pixels, 24 frames per second).

2.2. Temporal growth of microbubble

A typical example of the temporal evolution of the microbubble is shown in Fig. 2(a) when heating with a continuous wave laser at 1550 nm. This wavelength was chosen because of its strong absorp-

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