Liquid Concentration Sensing Properties of Microfibers with a Nanoscale-Structured Film

ZHOU Guo-Rui(周国瑞), LV Hai-Bing(吕海兵), YUAN Xiao-Dong(袁晓东), ZHOU Hai(周海), LIU Hao(刘昊), LI Ke-Xin(李可欣), CHENG Xiao-Feng(程晓锋), MIAO Xin-Xiang(苗心向)**

Laser Fusion Research Center, China Academy of Engineering Physics, Mianyang 621900

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A type of compact solution concentration sensor based on a microfiber with a nanoscale-structured film is proposed and demonstrated experimentally. Additional loss at different solution concentrations is calculated by means of the three-dimensional finite-difference time-domain (3D-FDTD) method. The microfiber is fabricated by using the flame-heated scanning technique. Nanoscale-structured film is coated on the microfiber surface, which is assembled as a sensing unit. The sensitivity of this kind of sensor increases with the decreasing diameters of the microfiber. When the diameter of the microfiber is 2 μm, a minimum concentration sensitivity of 1% (under 450 s measuring time) is demonstrated in the experiment. Higher sensitivity can be attained when the solution concentration is higher. The sensing properties of this microfiber with the nanoscale-structured film may provide opportunities for new applications in optical sensing devices.

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Solution concentration is one of the main parameters to denote the solution characteristics. The solution concentration must be measured with high accuracy for a great many applications, such as the textile and food industries, and environmental and chemical processing, where the content of impurities in liquid is an important factor. There are some traditional methods of solutions concentration monitoring, such as amperometric sensors,[1,2] ion-sensitive electrodes, and mass spectrometry.

Among the broad coverage of optical fiber research, micro/nanofibers have been attracting increasing interest for both fundamental studies and technological applications in the last few years. For photonics, micro/nanofibers provide favorable properties such as enhanced evanescent fields, tight light confinement and large waveguide dispersions.[3] Furthermore, micro/nanofibers, as one of the most important integrated photonic devices, are used as sensors,[4−13] lasers,[14] subwavelength waveguiding,[15] and so on.[16−19] A nanoscale sensor is one of the most promising applications of micro/nanofibers. Compared with other approaches, optical sensing based on micro/nanofibers may offer higher sensitivity, faster response, and better immunity to electromagnetic interference.

One technique potentially capable of meeting the demand of novel monitoring solution concentration is based on microfibers with nanoscale-structured film sensing, as presented in this work. When the composition of the analyte solutions adheres to a nanoscale-structure film, the light transmitting through microfibers with a nanoscale-structured film will be disturbed due to the change of the film's refractive index. The sensing unit is promising due to its low cost, easy fabrication and compact structure.

For basic consideration, it is assumed that the microfiber with a nanoscale-structured film in solution has a circular cross section and a step-index profile. Film with a loose and porous structure is coated on the microfiber surface. In addition, the refractive index (RI) of the microfiber is 1.465. The principle of the new type of sensors and the RI distribution of the entire sensing system are shown in Fig. 1.

The three-dimensional finite-difference time-domain (3D-FDTD) method is used to calculate the additional loss at different concentrations. The wavelength of the light transmitted along the microfiber was assumed to be 1.55 μm in the computational domain, which was discretized into a uniform orthogonal three-dimensional mesh with a cell size of 1/40 of the wavelength, terminated by perfectly matched layer boundaries. Different solution concentrations may cause different perturbations, which lead to different losses of sensing microfiber. The additional loss at different concentrations with respect to the RI of the nanoscale-structured film was calculated (450 s measuring time). As shown in the inset of Fig. 2, clearly the major light energy was confined inside a 2.0-μm-diameter microfiber and it left an amount of light guided inside the nanoscale-structured film as the evanescent wave. In Fig. 2, the additional loss caused by solute increased with solution concentration. When the solution concentration was 12.5%, the additional losses of 1.5 μm, 2.0 μm and 2.5μm-diameter microfibers were 0.4 dB/mm, 0.2 dB/mm and 0.11 dB/mm, respectively. The sensitivity of 1.5μm-diameter microfiber was far more than that

**Corresponding author. Email: miaoxinxiang.714@163.com

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of 2.0μm and 2.5μm-diameter microfiber. However, higher sensitivity means a lower dynamic range, suitable sensitivity and dynamic range should be selected by controlling the diameters of the microfiber in the experiment.

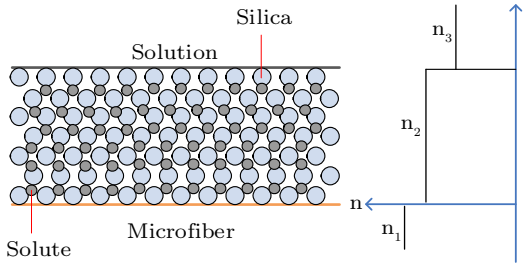


Fig. 1. Principle of solute adhered to the nanoscale-structured film and the corresponding RI distribution.

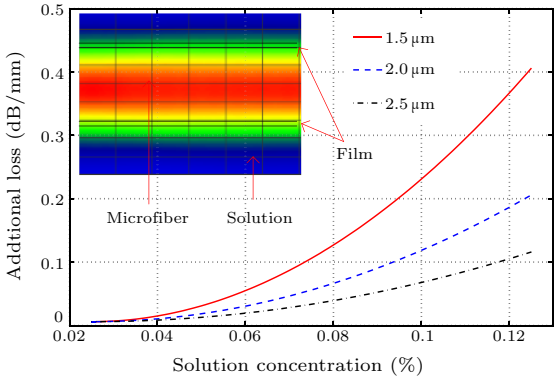


Fig. 2. Relationship between the additional losses of microfiber with different diameters and solution concentrations. The inset shows the power distribution of a 2.0-μm-diameter microfiber with a nanoscale-structured film which was immersed into absolute ethyl alcohol.

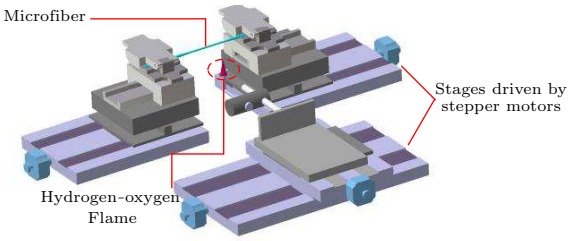


Fig. 3. The illustration of the microfiber drawing method by using the flame-heated scanning technique. The single-mode optical fiber was placed into where the suitable temperature of the hydrogen-oxygen flame was by using 3D translation systems. Single-mode fiber was drawn down to the microfiber based on the flame.

Here we introduce a three-step process for fabricating microfiber with nanoscale-structured film: drawing a standard fiber (Corning SMF28) to microfiber, coating nanoscale-structured film prepared by the sol-gel process on the surface of the microfiber, and assembling microfiber as a sensing unit. The microfiber used in this work was fabricated by the hydrogen-oxygen flame-heated scanning fiber drawing method[20] in an airtight environment, as shown in Fig. 3. Firstly, a standard fiber fixed on 2D translation systems was placed into a hydrogen-oxygen flame by an adjustable platform driven by stepper motors. The temperature of the heated fiber can be controlled by high-precision stepper motors. The hydrogen-oxygen flame fixed on stages and standard fiber moved with certain speeds controlled by a computer program, respectively. Compared with the laser drawing method, the flame-heated scanning method has certain advantages. Both theoretical and experimental results show that the laser power required for drawing microfibers with a uniform diameter is impractically large.[21]

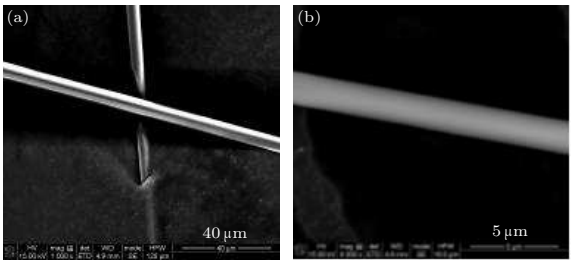


Fig. 4. SEM images of microfibers: (a) 8-μm-diameter microfibers, with a microfiber buried into the substrate, (b) a 2.0-μm-diameter microfiber.

The temperature in the drawing region and the speed of stepper motors can be controlled by a computer program to obtain different-diameter microfibers with good diameter uniformity. The uniform and smooth microfibers with diameters ranging from 2 μm to 8 μm are shown in Figs. 4(a) and 4(b). The higher-magnification scanning electron microscope (SEM) images of 8-μm-diameter and 2.0-μm-diameter microfibers indicate no visible irregularity on the surface of the microfibers. It can be seen clearly that microfibers fabricated by the method have good uniformity of diameter and surface smoothness.

In the experiment, a thin sol-gel film was coated on the surface of drawn microfibers by using the sol-gel process. A standard fiber was fixed on a glass slide, and a microfiber connected with a standard fiber cannot be supported by a glass slide. The entire system was encapsulated as a sensing unit. The sensing unit was washed by alcohol to keep the microfibers clean and then the thin sol-gel film was coated by the dip coating process. The sol-gel film consisted of a layer of porous, near-spherical silica particles, 10–40 nm in diameter, randomly stacked on the substrate surface. Such a highly porous film had highly specific surface areas and was therefore very susceptible to the solute in the solution. The film derived by the dip coating process with a low refractive index had controlled porosity, which could be adjusted from 65% to 12% continuously. The withdrawal rate was adjusted to give a suitable thickness. In the experiment, the withdraw rate U was 300 mm/min; and the liquid concentration η was 1.5%. The atomic force

microscope (AFM) images of the surface morphologies of a 1.5μm-diameter microfiber and the microfiber with nanoscale-structured film are shown in Fig. 5. The surface morphologies of microfiber are smooth in Fig. 5(a), while Fig. 5(b) indicates that the nanoscale-structured film is porous, and the roughness of the films is larger than that of microfibers.

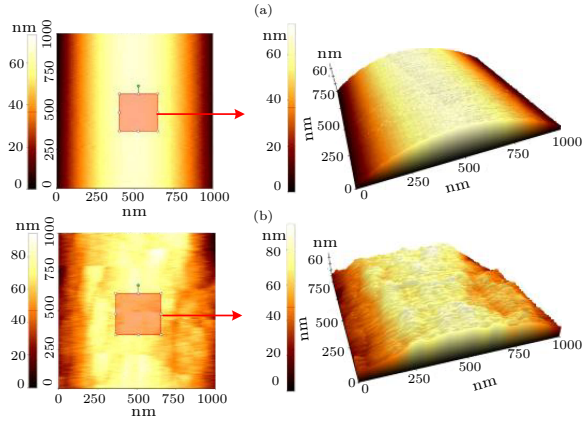


Fig. 5. AFM images of the surface morphologies: (a) a 1.5 μm-diameter microfiber, (b) a 1.5 μm-diameter microfiber with nanoscale-structured film.

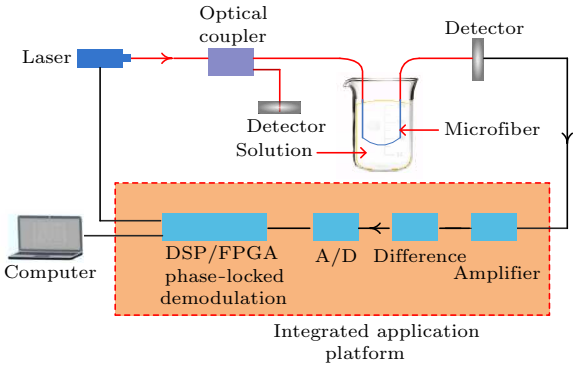


Fig. 6. Experimental setup of concentration sensing based on the microfiber with a nanoscale-structured film.

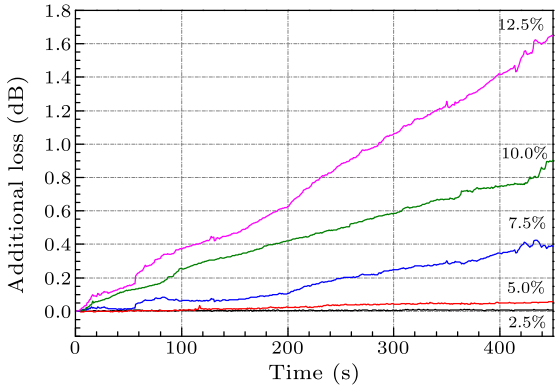


Fig. 7. The experimental results of the additional losses caused by the solution when the assembled microfiber was immersed into the solution.

The 2.0-μm-diameter 9 mm-length microfiber with a nanoscale-structured film has been assembled. The experimental setup has been designed and constructed to demonstrate the feasibility of concentration sensing with microfiber. The entire experimental setup is simple and compact as shown in Fig. 6. The microfiber with a nanoscale-structured film has been connected in the system. A 1.55-μm-wavelength laser was used as the source. Detectors, amplifier, difference, A/D and DSP/FPGA phase-locked demodulation were integrated into the application platform in which the phase-locked amplification technology was used to further improve measurement accuracy of the additional loss. Another beam of light was separated from the incident light by an optical coupler, which served as a reference light to improve the measurement accuracy.

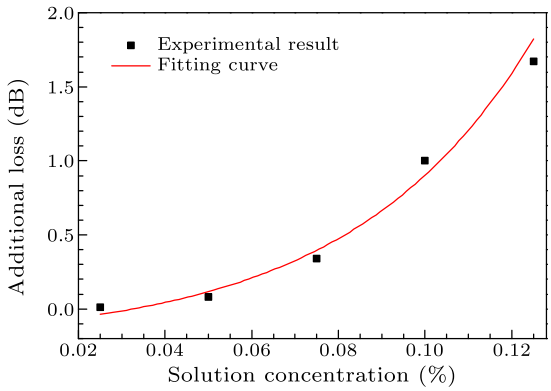


Fig. 8. The measured additional loss caused by solution at different concentrations when the measuring time was 450 s.

Vacuum pump oil (Leybold LVO130) was dissolved in absolute ethyl alcohol (CH_3CH_2OH) at 2.5%, 5%, 7.5%, 10% and 12.5% volume concentration, and produced a clear solution. The assembled microfiber was immersed into the measured solution. The output power for concentration sensing was recorded by a compiled software at different volume concentrations. In experiment, it was found that the static stability ΔE_1 of output power of the laser reached about 3.5% due to the change of temperature and the fluctuation of the supply current. The measured power uncertainty ΔE_2 of the detector was 2.2%, and measurement accuracy of the entire system $\Delta E = \sqrt{\Delta E_1^2 + \Delta E_2^2}$ was 4.1%. Therefore, monitoring the reference light power can eliminate power fluctuation of the laser ($\Delta E_1 = 0$), and measurement accuracy of the entire system increased by 46%. The experimental results of additional losses caused by solutions at different concentrations are illustrated in Fig. 7. As shown in Fig. 7, the variation of additional losses involved two phases roughly: the fast growth stage (about 1–120 s) and the linear growth stage (about 120–450 s). It can be explained that the adsorption rate of the nanoscale-structured film was considerably high at the beginning of the measuring state. At the same time, it can be seen clearly that the additional

loss caused by solute in solution increased dramatically with solution concentration as shown in Fig. 8. The reason was that more solute in solution adhered to nanoscale-structured film as the solution concentration increased, and more evanescent wave energy was no longer tightly confined inside, which lead to the increase of additional loss. When the solution concentration was 12.5%, the additional loss (1.67 dB) was close to the theoretical result (1.8 dB) in Fig. 2. The measurement inaccuracy of microfiber diameter may be the main factor which leads to the errors. The measuring accuracy integrated into the application platform was about 0.004 dB. The minimum measuring sensitivity of the entire system was 1% when the measuring time was 450 s, and the concentration sensitivity of the entire system increased dramatically as the solution concentration increased. Therefore, it indicates that the sensing unit works more precisely at higher solution concentration.

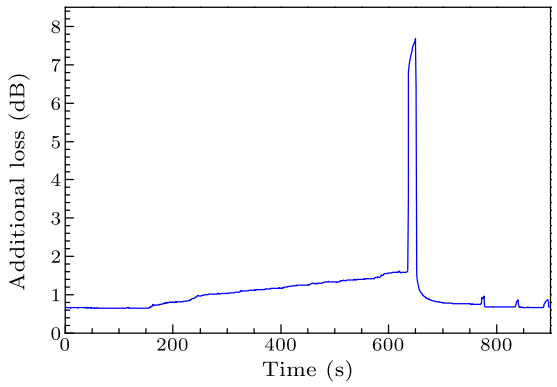


Fig. 9. The additional loss varying with the measuring time when the microfiber experienced five states to demonstrate the feasibility of alcohol washing.

To confirm the reusability of microfiber with a nanoscale-structured film, we washed the sensing unit with alcohol. The experimental results are shown in Fig. 9. The washing process of the sensing unit consisted of an initial state, a measuring state, taking out the microfiber, alcohol washing and free state. The sensing unit was immersed into a 10% solution for 500 s measuring time, and then it was taken out from the solution. At the same time, the additional loss increased dramatically, which may be attributed to two factors. For one thing, the bend loss of the microfiber increased dramatically due to solution tension. For another thing, nonuniformity of the refractive index of the nanoscale-structured film lead to the increase of additional loss. In the process of washing the sensing unit with alcohol, the additional loss dropped dramatically. After alcohol washing, the additional loss of the microfiber was in a free state. It can be seen clearly that the additional loss in the initial state was roughly the same as that in the free state. In the experiment, it was found that the additional loss increased dramatically when the washing time was longer than 20. The reason was that the nanoscale-structured film had been destroyed in the process of alcohol washing.

In summary, this research was driven by the need for a novel, sensitive and cost-effective monitoring method of solution concentration. Microfiber with a nanoscale-structured film was investigated to verify the potential applicability of the evanescent field sensing for the task. The microfiber with a nanoscale-structured film was assembled as the sensing unit and showed a remarkable stability when the solution concentration varied from 2.5% to 12.5%. Minimum concentration sensitivity of 1% (under 450 s measuring time) has been demonstrated in the experiment. Higher resolution can be attained when the solution concentration was higher. The sensing properties of the sensing unit may provide opportunities for a new application of optical sensing devices.

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