ZnO microwire-based fiber-tip Fabry-Pérot interferometer for deep ultraviolet sensing

Han Wu, Ying Wang, Lichao Zhang, Kuikui Guo, Yijian Huang, Changrui Liao, and Yiping Wang, *Senior Member, IEEE, Senior Member, OSA*

***Abstract*—We propose a simple deep ultraviolet (UV) sensor consisting of a conventional single-mode optical fiber capped with a ZnO microwire. The ZnO microwire positioned on the fiber-tip acts as a Fabry-Pérot interferometer, of which the reflection spectrum can be employed for UV light monitoring. When ZnO microwire is exposed to UV irradiation, variations in the concentration of photogenerated carriers will result in the change of the refractive index (RI) of the ZnO microwire, and thus interference wavelengths of the proposed device exhibits red-shift with a sensitivity of 0.288 nm/(W·cm^{-2}) under 266-nm deep UV laser irradiation. Meanwhile, a fast response time of 0.56 ms is experimentally obtained. The results may pave a new way for fast responsive, highly spatial-resolved optical fiber UV sensors.**

***Index Terms*—ZnO microwire, UV sensor, optical fiber sensor, Fabry-Pérot interferometer**

I. INTRODUCTION

Ultraviolet (UV) light has been widely used in the fields of medical treatments, defect detection, water purification, UV curing and printing, and the necessity of quantitatively detecting and monitoring of UV light in these cases has drawn increasing research interests. UV sensors can reduce UV-related accidents for safety in certain fields, such as UV light emitted by electric leakage and high-power generation[1]. To date, most reported UV sensors have been demonstrating responses to UV wavelengths above 300 nm [2,3], few works have been focusing on the wavelength down to the deep UV region. As a matter of fact, the performance of UV light is affected by its wavelength in certain applications. For instance, research have indicated that pulsed UV laser with operation wavelength of 266 nm are much more effective in activation of Bovine viral diarrhea virus (BVDV) suspended in (fetal) bovine serum (FBS) than 355-nm UV laser [4]. And in the pharmacy developments, exposure of Chlorpromazine Hydrochloride (CPZ) to a 266 nm laser beam yields rapidly a large number of new and stable species of products with antibacterial properties [5]. Deep UV light is highly applicable in the fields of biological technology and medical industry. Therefore, UV sensors have been widely investigated in recent years. Shape-memory polymers has been used to build UV sensor for real-time monitoring, such as a stretchable fiber Bragg grating (FBG) coated with azobenzene moiety [6], or azobenzene-polymer-capped optical-fiber end [7]. These sensors are based on the UV introduced tensile stress, and the response time are in the level of seconds, which are not applicable in some cases where fast response is required. Optical resonators or microcavities fabricated with silica materials are also demonstrated for UV sensing, where thermal effect caused by the UV light illumination is utilized, which leads to the index change of silica material [8]. The response time of such a sensor is even longer, which is about 8 seconds.

ZnO micro/nanowires (MNWs), as important representatives of one-dimensional wide bandgap semiconductors, have attracted great attention in the development of novel optoelectronic and nanophotonic devices, such as transistors [9,10], light-emitting diodes [11], lasers [12-15], solar cells [16,17], and biosensors [18, 19]. Particularly, ZnO MNWs also exhibit exciting applications in UV photodetection due to their excellent electronic and optical properties, such as giant photoconductive gain, high sensitivity and efficient waveguide of light [20,22]. From the discovery of UV photo-sensitivity of ZnO nanowires as a photodetector (PD) by Kind et al in 2002 [23], numerous efforts have been devoted to develop ZnO MNWs-based UV sensors or PDs, aiming to achieve fast response, high sensitivity or responsivity, and multi-functionality [24-26]. However, the response time of ZnO-based UV sensors is always limited by the carrier mobility and high electrical resistance. To improve the response time, the optical properties of ZnO MNWs, such as transient refractive index (RI) change induced by the generation of photocarriers, can be readily considered to build UV sensors. A ZnO nanowire-based microfiber coupler has been demonstrated for UV sensing with a drastically reduced response time down to the sub-millisecond level [5].

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(Corresponding author: Ying Wang.)

The authors are with The Guangdong and Hong Kong Joint Research Centre for Optical Fibre Sensors, College of Physics and Optoelectronic Engineering, Shenzhen University, Shenzhen 518060, China and the Key Laboratory of Optoelectronic Devices and Systems of Ministry of Education and Guangdong Province, Shenzhen University, Shenzhen 518060, China (e-mail: wuhan2018@email.szu.edu.cn; yingwang@szu.edu.cn; lczhang5354@szu.edu.cn; 2150190116@email.szu.edu.cn; huangyijian@email.szu.edu.cn; cliao@szu.edu.cn; ypwang@szu.edu.cn).

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In this work, a ZnO microwire-based fiber-tip Fabry-Pérot interferometer (FPI) is proposed for deep UV sensing. The FPI is formed by the two flat end facets of the ZnO microwire, which is capped to the cleaved end of a single mode fiber (SMF) through a short section of silica tube. The proposed device exhibits a redshift in the reflection interference spectrum under 266-nm UV laser irradiation due to the photocarrier induced transient RI change of the ZnO microwire, and a sensitivity of 0.288 nm/(W·cm^{-2}) and a response time of 0.56 ms are obtained, respectively. These results indicate that the proposed sensor suggests a solution for the buildup of fast responsive, highly spatial resolved UV sensor, which may find potential applications in pharmacy developments and biological industries.

II. Device design and Experimental Setup

The ZnO microwires used here are synthesized by a simple chemical vapor deposition (CVD) method. A mixture of high purity ZnO and graphite powders (1:1 in mass ratio) is used as the source material, which is put into a quartz boat (3cm×1.5cm×1cm) covered by a silicon chip (4cm×2cm). Then the boat is transferred to a glass tube that placed in a high temperature furnace operated at 1150 ℃ under normal atmospheric pressure. For a duration of 40 minutes, ZnO microwires with lengths ranging from 100 to 1000 μm and diameters between 1 and 10 μm are obtained successfully via the abovementioned CVD method. The typical scanning electron microscopy (SEM) image and scanning transmission electron microscopy (STEM) image of a single ZnO microwire are shown in Figs. 1(a) and 1(b), respectively, and the top-left inset of Fig.1(b) shows the selected area electron diffraction (SAED) pattern. Figure 1(c) shows the X-ray diffraction (XRD) pattern, where the detected signals of quartz originate from the used substrate. Figures 1(d)-(f) show the elemental mapping of O, Zn and the electron diffraction X- ray (EDX) spectrum, respectively. These results confirm that the obtained microwires are hexagonal ZnO microwires.

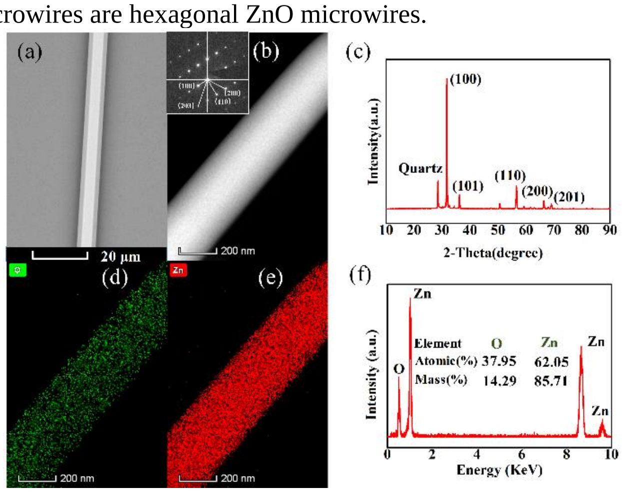

Fig. 1: (a) SEM and (b) STEM image of a single ZnO microwire synthesized by CVD method. Top-left inset in (b) shows the SAED pattern. (c) XRD pattern of the synthesized ZnO microwires. (d)-(f) Elemental mapping of O,Zn and EDX spectrum,respectively.

Figure.2 shows the schematic of the proposed ZnO microwire-based fiber-tip FPI, where the ZnO microwire is integrated in the end facet of a SMF and is held by a short section of silica tube that fusion spliced with the SMF. The left end of ZnO microwire is closely attached to the fiber core of SMF. Thus, white light propagated in the fiber core is reflected at the silica/ZnO interface and the ZnO/air interface, respectively, and forms interference in the reflection spectrum. When illuminated by UV light, photocarriers are generated in the ZnO microwire of the fiber-tip FPI and accordingly interference wavelengths will change due to photocarrier induced transient RI variation.

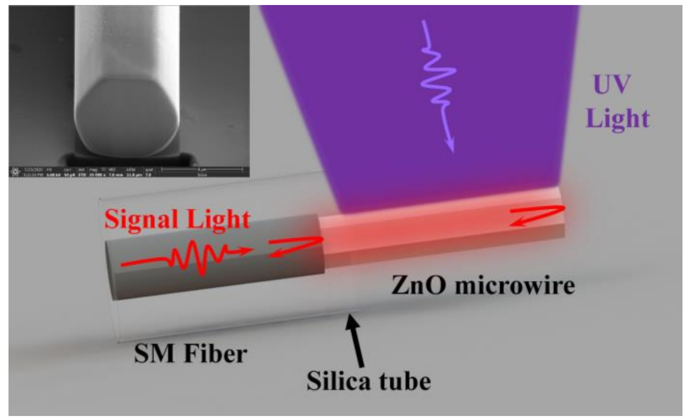

Fig. 2 Schematic of the ZnO microwire-based fiber-tip Fabry- Pérot interferometer for UV light sensing. Top-left inset shows a SEM image of the FIB milled end facet of ZnO microwire.

For the fabrication of fiber-tip FPI, both ends of a ZnO microwire are etched by focused ion beam (FIB) milling to enhance their parallelism, smoothness and flatness, and a typical SEM image is shown in top-left inset of Fig. 2 to illustrate the end facet quality more directly. Meanwhile, the lengths of ZnO microwire can also be controlled precisely by FIB milling. To provide an appropriate fiber-tip for microwire integration, a 30-μm-long silica tube with an inner/outer diameter of 9/125 μm (TSP010150, Polymicro, Arizona, USA), respectively, is fusion spliced with the cleaved end of a conventional SMF (SMF28e, Corning Inc). The other end of the SMF is connected to an optical circulator, which is also the junction of a supercontinuum light (WhiteLase Micro, NKT Photonics) and an optical spectrum analyzer (OSA, AQ6370C, Yokogawa), to observe its reflection spectrum. Then the FIB milled ZnO microwire with required diameter and length is picked up and transferred into the silica tube by a tungsten probe with the help of an optical microscopy. With moving the microwire towards the fiber core, a two-beam interference in the reflection spectrum can be observed unless the inserted end of ZnO microwire is closely attached to the SMF fiber core, which can be monitored by OSA in real-time. The inserted end of ZnO microwire in the silica tube can be encapsulated by a micro-drop of UV curable adhesive or by arc-discharge collapsing of the silica tube.

The experimental setup for UV photodetection with the proposed device is shown Fig. 3. Bottom-right inset of Fig. 3 shows the microscopic image of a fabricated fiber-tip FPI with a ZnO microwire length of 100 μm. Similar to that during fabrication, the light source, the input SMF of the fiber-tip FPI and an OSA spanning a wavelength range of 900-1700 nm are connected with an optical circulator to record the reflection spectrum of the device in real time. The fiber-tip FPI is fixed on a glass substrate to avoid possible vibration, and 266-nm UV laser beam is focused onto the ZnO microwire of the fiber-tip FPI by a UV lens with a focal length of 10 cm. The UV laser power can be adjusted continuously from 0 to 80 mW by the combination of a half-wave plate and a Glen lens. For dynamic response measurement of the device, the light source is replaced by a tunable laser (Model 81940A, Agilent Technologies) operated in a wavelength range between 1520 and 1620 nm, and the UV laser beam is chopped to pulse trains by an optical chopper (MC2000B-EC, Duty Cycle 50%, Thorlabs) with variable repetition rates.

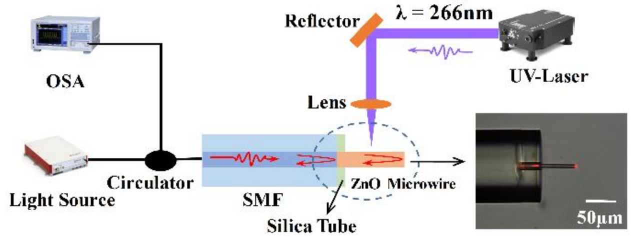

Fig. 3: Experimental setup for UV photodetection with the ZnO microwire-based fiber-tip FPI. UV laser wavelength is 266 nm. Bottom-right inset shows a fiber-tip FPI with a microwire length of 100 μm.

III. Results and Discussion

Reflection spectra of the fabricated devices with the same microwire diameter of 6.6 μm and different microwire lengths of 50, 80 and 100 μm are plotted in Fig.4(a), respectively, where it can be seen that the free spectral range (FSR) decreases with the length of ZnO microwire increases. Generally, the FSR can be described as $\boldsymbol{FSR}=\lambda^2/2\boldsymbol{nl}$, where λ, n and L are the inspected wavelength in vacuum, RI and length of ZnO microwire, respectively. Thus， the FSR for above mentioned devices can be calculated to be 12nm, 7.5 nm and 6 nm, respectively, at around the wavelength of 1550 nm, and the experimentally obtained results are in good agreement with the calculated ones.

When the fiber-tip FPI is exposed to UV illumination, it exhibits redshift of the interference wavelengths in the reflection spectrum with UV light intensity increasing. Figure 4(b) shows the reflection spectra variation for a fiber-tip FPI with a ZnO microwire diameter of 6.6 μm and length of 50 μm that irradiated by 266-nm UV laser beam with intensities increases from 0 to 25.48 W·cm^{-2}, where apparent wavelength shift from 1549.6 nm to 1556.5 nm can be observed. To illustrate the relationship between the UV sensitivity and the diameter of ZnO microwire , three samples with microwire diameters of 4.5, 5.5 and 6.6 μm, respectively, are illuminated by UV light with changing the intensity from 0 to 38.22 W·cm^{-2} gradually, and the shift of interference wavelength that closest to 1550 nm for each sample is plotted in Fig. 4(c) with respect to the UV light intensity accordingly. Sensitivities of these samples can be determined to be 0.260, 0.263 and 0.284 nm/(W·cm^{-2}) through linear fitting, respectively. Figure 4(d) shows the wavelength shift as a function of UV light intensity for another three samples with ZnO microwire lengths of 50, 80 and 100 μm, respectively, where the diameters of ZnO microwires are kept the same as 6.6 μm. Through linear fitting, the sensitivities are determined to be 0.268 nm/(W·cm^{-2}), 0.283 nm/(W·cm^{-2}) and 0.288 nm/(W·cm^{-2}) for these samples, respectively. Considering the experimental errors, the UV sensitivities for all of the fabricated samples are almost the same, as indicated in Figs.4(c) and 4(d), which implies that the UV sensitivity of the proposed fiber-tip FPI is independent on the diameter and length of ZnO microwires.

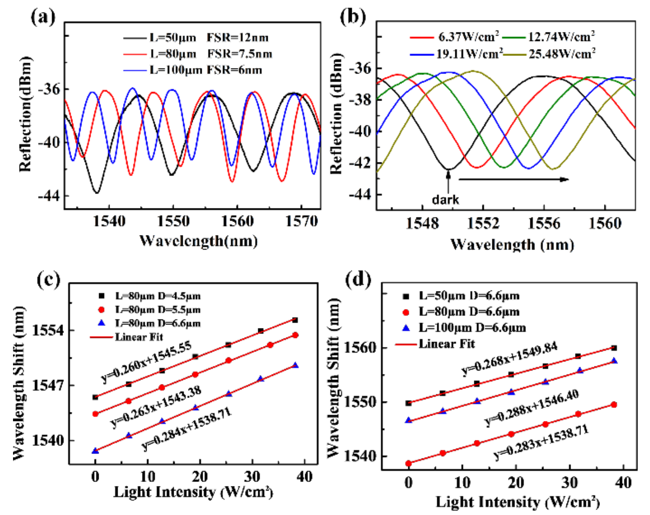

Fig. 4: (a) Reflection spectra for the fiber-tip FPIs with ZnO microwire diameter of 6.6 μm and lengths of 50, 80 and 100 μm, respectively. (b) Interference wavelength redshift in the reflection spectrum for a fiber-tip FPI with a ZnO microwire diameter of 6.6 μm and length of 50 μm under UV illumination. The UV light intensity increases from 0 to 25.48 W·cm^{-2}. (c) Wavelength shift as a function of UV light intensity for fiber-tip FPIs with ZnO microwire diameters of 4.5, 5.5 and 6.6 μm, respectively. Lengths of all ZnO mirowires are kept the same as 80 μm. (d) Wavelength shift as a function of UV light intensity for fiber-tip FPIs with ZnO microwire lengths.

The redshift of interference wavelengths of the proposed fiber-tip FPI devices under UV illumination is mainly attributed to the photocarrier induced transient RI change in ZnO microwire. As is known, the ZnO MNW exhibits ~90% reflectance in the visible spectrum, compared to ~18% in the UV wavelength region [25]. The strong absorbance of UV photons results in the transition of a large number of valance electrons from the valance band to the conduction band and generate large amounts of photocarriers in the forms of photo-generated electrons and vacancies in ZnO MNW. These carriers can cause notable changes in absorption coefficient and produce a considerable RI change in ZnO MNW according to the Krames-Kronig relationship [27],

$$\Delta n(N,P,E)=\frac{2c\hbar}{e^2}P\int_0^{\infty}\frac{\Delta\alpha(N,P,E')}{E'^2-E^2}dE' \qquad (1)$$

where N and P are concentrations of free electrons and vacancies, respectively. c, $\hbar$, e, E and $P\int$ are respectively the speed of light in vacuum, Planck's constant, electron charge, photon energy and principal value of the integral. Here, indicates the variation in the absorption coefficient, which is always positive in ZnO MNWs due to the bandgap shrinkage effect in the case of relatively high photocarrier concentrations [25, 27-29]. This causes the RI increase in ZnO microwires with UV light intensity increasing, according to Eq. (1), and

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thus results in the optical path elongation in the fiber-tip FPIs and the redshift of the interference wavelengths.

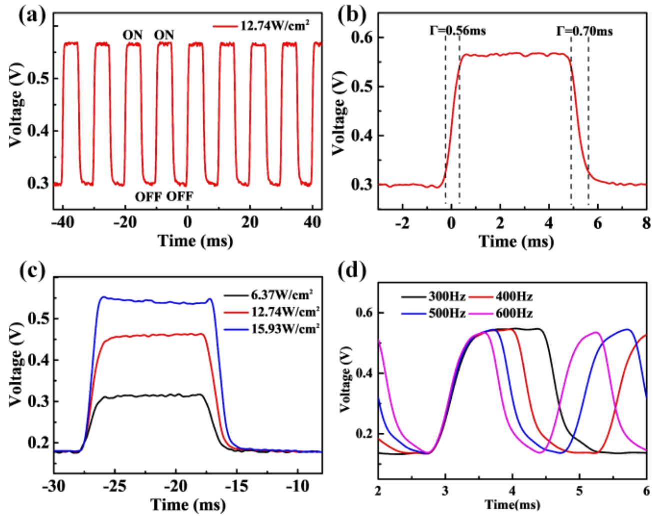

Fig. 5: (a) Reproducible on/off switching for a fiber-tip FPI with a ZnO microwire diameter of 6.6 μm and length of 50 μm illuminated by chopped 266-nm pulse train with a repetition rate of 100 Hz and a light intensity of 12.74 W/cm^2. (b) Magnification of the temporal response for one period shown in (a). The rise and decay time is 0.56 ms and 0.70 ms, respectively. (c) Temporal response versus UV light intensity for the sample. Repetition rate: 50 Hz. (d) Temporal response versus repetition rate for the sample. UV light intensity: 15.93 W/cm^2.

To demonstrate the dynamic response of the proposed device, a sample with the ZnO microwire diameter of 6.6 μm and length of 50 μm, of which the reflection spectrum is shown in Fig. 4(a), is illuminated with chopped pulse trains of UV light. The light source shown in Fig. 3 is replaced by 1547.5-nm light delivered from the aforementioned tunable laser here, and the OSA is substituted to a commercial available photodetection (MODE 3054, Tektronix) that connected with an oscilloscope (MDO 3054, Tektronix) to monitoring the variation of the output optical signal that reflected by the sample. The reflected 1547.5-nm light from the sample can be detected by the abovementioned photodetection and converted to electric voltage, which is then presented by the oscilloscope in real-time. The output wavelength of tunable laser is set to the wavelength corresponding to the position of 3-dB difference compared to the interference peak nearest to 1550 nm in the reflection spectrum. Figure 5(a) shows the on/off switching characteristics for the sample under the illumination of chopped 266-nm pulse train with a repetition rate of 100 Hz and a light intensity of 12.74 W/cm^2, which exhibits good reproducibility. A 0.3V offset of the voltage output can be observed, which is originated from the reflected interrogation light. One period in Fig. 5(a) is shown in Fig. 5(b) for clarity, where the rise time and decay time, defined as the time interval that the output voltage increases from 10% to 90% (or decreases from 90% to 10%) of the voltage difference between the high and low level, can be measured to be 0.56 ms and 0.70 ms, respectively. Obviously, the obtained response time is two or more orders of magnitude lower than that of electrical bridge-based ZnO UV sensors reported previously [20]. Actually, the response time of the proposed device is increased significantly by the trapping effect, photocarrier diffusion and recombination processes [25, 30, 31]. Fig.5(c) shows the evolution of photoelectric conversion voltage for the sample under varying UV light intensities at a frequency of 50 Hz. The output voltage increases with increasing the UV light intensity, which agrees with previous result of interference wavelength redshift of the sample, as illustrated in Fig. 4(b). Figure 5(d) shows the temporal response of the sample illuminated with UV light intensity of 15.93 W/cm^2 at various chopping frequencies. Evidently, the rising edge remains constant as the frequency increases. However, the voltage breaks away to recover other than reach to saturation when the frequency is increased to be 700Hz, which is mainly limited by the response time of the proposed structure and implies its upper limit of operation frequencies.

IV. Conclusions

A ZnO microwire-based fiber-tip FPI is proposed and demonstrated for UV sensor. The device is fabricated simply by integrating a single ZnO microwire in the end facet of a conventional SMF, where the microwire acts as a FPI and optical demodulation scheme can be adopted. Experimental results show that the sensitivity of the device for UV sensor is independent on the diameter and length of ZnO microwire, and the highest sensitivity obtained is 0.288 nm/(W·cm^{-2}). And the rise time and decay time is measured to be 0.56 ms and 0.70 ms, respectively, for a device with ZnO microwire diameter of 6.6 μm and length of 50 μm. The proposed structure aims to pave a new way for fast responsive, highly spatial-resolved and optical fiber-compatible sensor.

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Han Wu received the B.Sc. degree in School of Science, Wuhan Institute of Technology, Wuhan, China, in2018. Since 2018, he has been working toward the M.S. degree optical engineering in the School of Photoelectric Engineering, Shenzhen University, Shenzhen, China. His research interests are optical fiber sensing and femtosecond laser micromachining.

Ying Wang was born in Henan, China, in 1983. He received the B.S. degree in applied physics and the Ph.D. degree in physical electronics from the Huazhong University of Science and Technology, Wuhan, China, in 2004 and 2010, respectively. From 2010 to 2011, he worked with the Department of Electrical Engineering, Hong Kong Polytechnic University, Hong Kong, as a Research Associate. And from 2011 to 2015, he worked with the School of Science, Wuhan Institute of Technology, Wuhan, China. Since 2015, he has been with the College of Physics and Optoelectronic Engineering, Shenzhen University, Shenzhen, China, as an Associate Professor. His research interests include optical fiber sensors, photonic crystal fibers and devices, and femtosecond laser micromachining.

Lichao Zhang received the B.E. degree in materials shaping and controlling engineering from Henan University of Science and Technology in 2008, and Ph.D. degree in physical electronics from Huazhong University of Science and Technology (Wuhan, China) in 2014. She worked as a postdoctoral fellow in Professor Xiaocong Yuan's group in Shenzhen University during 2014-2017, and joined in Yiping Wang's group in Shenzhen University in 2018, working as a postdoctoral fellow. Her current research interests include optical fiber grating and femtosecond laser micromachining.

Kuikui Guo received the B.S. degree from the Institute of Photoelectric Information, Changchun University of Science and Technology, Changchun, China, in 2013. He is currently working toward the Ph.D. degree in optical engineering in Shenzhen University, Shenzhen, China. From 2013 to 2015, he worked in Accu-Tech Co., Ltd., Beijing, China, as an Optical Engineer. His current research interests include optical fiber laser sensors and fiber Bragg gratings.

Yijian Huang was received the B.S. degree in optical information science and technology from East China Jiaotong University, Nanchang, China, in 2013.Since 2014, he has been working toward the Ph.D. degree in optical engineering in the School of Photoelectric Engineering, Shenzhen University, Shenzhen, China. His research focuses on photonic crystal fiber-based sensors and electrooptic switch.

Changrui Liao was born in Shandong, China, in 1984. He received the B. Eng. degree in optical engineering and the M.S. degree in physical electronics from the Huazhong University of Science and Technology, Wuhan, China, in 2005 and 2007, respectively, and the Ph.D. degree in electrical engineering from the Hong Kong Polytechnic University, Hong Kong, in 2012. He is currently with Shenzhen University, Shenzhen, China, as an Associate Professor. His current research interests include femtosecond laser micromachining, optical fiber sensors, and opto-microfluidics.

Yiping Wang (SM'11) was born in Chongqing, China, in 1971. He received the B.Eng. degree in precision instrument engineering from Xi'an Institute of Technology, Xi'an, China, in 1995, and the M.S. and Ph.D. degrees in optical engineering from Chongqing University, Chongqing, China, in 2000 and 2003, respectively. From 2003 to 2005, he was with Shanghai Jiao Tong University, Shanghai, China, as a Postdoctoral Fellow. From 2005 to 2007, he was with the Hong Kong Polytechnic University, as a Postdoctoral Fellow. From 2007 to 2009, he was with the Institute of Photonic Technology, Jena, Germany, as a Humboldt Research Fellow. From 2009 to 2011, he was with the Opto-electronics Research Centre, University of Southampton, Southampton, U.K., as a Marie Curie Fellow. Since 2012, he has been with Shenzhen University, Shenzhen, China, as a Distinguished Professor. He has authored or co-authored 1 book, 21 patent applications, and more than 240 journal and conference papers. His current research interests include optical fiber sensors, fiber gratings, and photonic crystal fibers.

He is a Senior Member of the Optical Society of America and the Chinese Optical Society.