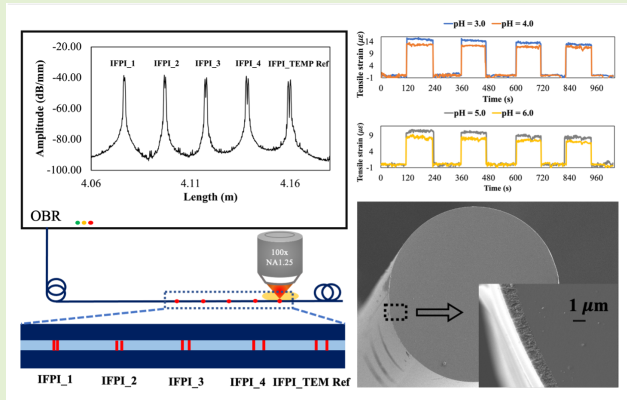
Multiplexable Fiber pH Sensors Enabled by Intrinsic Fabry-Peìrot Interferometer Array

Xinruo Yi, Kehao Zhao, and Kevin P. Chen

***Abstract*—This work presents multiplexable fiber optical sensors to perform multiple-point pH measurements. Using a femtosecond laser direct writing approach, array of intrinsic Fabry-Peìrot interferometer (IFPI) sensors were inscribed in standard single-mode fibers. A sol-gel dip coating process was used to deposit Palladium-doped Titanium Dioxide (Pd-TiO_2) sensory film on IFPI sensors to perform pH measurement, while an uncoated sensor was used to measure temperature of aquatic solutions to remove influence of temperature fluctuation on pH measurements. Pd nanoparticles in sensory films acts as catalyst to convert hydrogen ion in aquatic solutions Pd hydride, which produce strains to IFPI sensors. White light interferometry demodulation algorithm was applied to resolve strain exerted to IFPI sensors induced by pH value changes in aquatic solution. The sensor exhibits reversible and reproducible response in aquatic solution with pH value from 1.0 to 7.0 at the room temperature. The response time of the pH sensors was approximately 7 s for all measurements performed. The sensor technology demonstrated in this paper has a potential to perform multi-point pH and temperature measurements using a single fiber.**



***Index Terms*— Fabry-Perot, interferometers, optical fiber sensors, pH measurement.**

I. Introduction

PH VALUE is a fundamental chemical property for all aquatic solutions. The measurement of pH value is required for a large number of chemical, biomedical, and environmental analyses. Although the pH measurement can be performed using conventional paper strip or glass electrode, these existing sensors often incur low accuracy or long response. In addition, electrochemical sensors cannot produce valid results in harsh environments such as those for subsurface applications. To address the needs of pH measurement for harsh environment applications, fiber optical based pH sensors have been developed and studied since 1990s. These research efforts are based on well-established arguments that fiber optical sensors are resilience in harsh environment, immune to electromagnetic inference [1]. Optical fiber-based sensors also offer unique capability of sensor multiplexing that enable multiple point pH measurements using a single fiber. Construction of optical fiber pH sensors involves integration of pH-sensitive sensory materials on various optical fiber platforms. Change of pH values induce changes of optical absorption or physical expansion of sensory materials, which are measured by fiber sensors. In recent years, various metal-oxide dielectric membranes were studied as pH-sensing materials including tantalum pentoxide (Ta_2O_5) [2], zinc oxide (ZnO) [3], tin oxide (SnO_2) [4], and titanium dioxide (TiO_2) [5]. Based on hydrolysis and poly-condensation of metal-organic precursors, high purity and homogeneous oxide materials can be prepared by low-cost sol-gel technology at low temperatures using various wet chemical processes [6]. TiO_2-based vitreous coating obtained by sol-gel technology has been experimentally confirmed having applications in optics and electronics because of its excellent properties such as high refractive index, high dielectric constant, resistance to chemical and electrical interference [6], [7]. Even though TiO_2 has been demonstrated as one of the excellent sensing materials, pure TiO_2 nanostructure still shows the limitation of sensitivity [8]. In order to improve the sensitivity and durability of TiO_2 based optical fiber pH sensor, combination of palladium (Pd) was introduced. Pd are commonly used as the sensing material, due to its high sensitivity and selectivity to hydrogen element.

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The catalytic effect of Pd nanoparticles can reduce the sensor working temperature down to the room temperature [9].

Most of fiber optical pH sensors are based on evanescent wave interaction between guided light in fiber and sensory materials. The change of optical absorption in sensory materials induced by changes of pH value can be gauged using optical transmission measurements. However, pH sensor based on optical transmission drastically reduce multiplexing ability of fiber sensors, which significantly weaken rationale for industry to adapt the fiber sensors. In addition to the absorption-based approach, optical fiber pH sensors based on different kinds of fiber interferometers have been studied. These include both intrinsic and extrinsic fiber Fabry-Peìrot interferometers (FPIs) [10], [11] and Mach-Zehnder interferometers [12]. However, current approach to construct FPI-based pH sensors involve fusion of claddingless fiber coated with sensory materials with single-mode fibers or coating of sensory materials on fiber tips. They are difficult to fabricate and incur significant transmission loss for sensor multiplexing.

In this paper, we demonstrate a strain-based Intrinsic Fabry-Peìrot Interferometers (IFPIs) pH sensors, which are suitable for multiplexable pH measurements. Using a femtosecond laser direct writing scheme, pairs of Rayleigh enhanced backscattering points were used to form IFPI cavities. Pd-doped porous TiO_2 were coated on IFPI arrays as sensory materials. Using a white-light interferometry demodulation algorithm, multiple IFPI sensors can be demodulated simultaneously to resolve pH-induced strain change exerted on IFPI devices with minimal detection sensitivity up to 40 nε.

II. Fabrication and Characterization

A. Fabrication of Intrinsic Fabry-Peìrot Interferometer

A femtosecond (fs) Ti:Sapphire laser system (Coherent MIRA-D and RegA 9000) was used for IFPI sensor fabrication. The laser produced a linear polarized 270 fs pulses at 800-nm with 250 kHz repetition rate. Laser pulses were tightly focused inside the core of standard telecom single-mode fiber (Corning SMF-28e+) through a 100x oil-immersion objective as depicted in Fig. 1.

Laser-induced nanograting can be formed to enhance Rayleigh backscattering when on-target pulse energy exceeds 200-nJ. The detail fabrication process can be found in Ref. [13]. Five pairs of Rayleigh scattering points were induced by the laser to form five IFPI sensors. The Rayleigh backscattering profile of five IFPI sensors was characterized in real-time by using an optical backscattering reflectometer (Luna OBR 4600). A typical Rayleigh backscattering profile for sensor array fabricated in fiber is shown in Fig. 1. This OBR system was used during the overall fabrication process which could verify the location of those laser-induced backscattering points and also could monitor the optical loss of the optical fiber when IFPI sensors were formed.

The IFPI sensor array used in this work consist of five pairs of laser-induced reflectors with cavity length increased from 480 μm to 1080 μm with an increment of 150 μm. The spatial separation between each adjacent IFPI sensors was around 20 mm. The laser-induced scattering points produces ~50-dB enhancement of backscattering power above

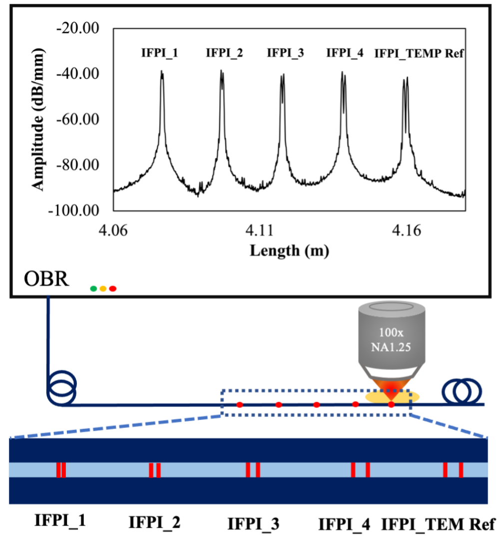

Fig. 1. The schematic illustration of IFPI sensors fabrication by femtosecond laser irradiation on optical fiber. The inset is the IFPI spectrum profile detected by OBR from enhanced Rayleigh scattering points. The initially designed five pairs of laser-induced reflectors with cavity length was increasing from 480 μm (IFPI_1) to 1080 μm (IFPI_TEMP Ref) with an increment of 150 μm. The separation between each adjacent sensor was designed to be 20mm.

the intrinsic Rayleigh backscattering signal in a standard single-mode optical fiber as shown in the inset image of Fig. 1. This increase of backscattering signal from the laser-induced scattering points are sufficient to produce significant interference fringe, which could be detected by spectrometer and subsequently demodulated for real-time determination of the cavity length.

B. Preparation of Pd-TiO_2 Sol-Gel Solution

A dip coating and sol-gel approach was used to integrate pH sensing films on IFPI sensors. To prepare TiO_2 precursor solution, 1.5 g $Ti(OCH(CH_3)_2)_4$ and 0.45 g HCl were mixed with 6 g ethanol and stirred for 10 min. The Pd precursor source for Pd is 1g of $PdCl_2$ ethanol solution, which was added into the TiO_2 precursor solution. To improve interaction between pH reagent liquid and the sensory film, 0.8g Pluronic F-127 co-polymer solution was added the precursor solution to create porous structures [14], [15]. The mixture was kept stirring overnight on top of a hot plate (60 °C) to obtain a homogeneous solution. The final mixture solution was cooled down to the room temperature for 24 hours before use. All chemicals mentioned above were purchased from Sigma-Aldrich.

C. Coating of Pd-TiO_2 Thin Films on IFPI Sensors

Optical fibers with IFPI sensor array were first thoroughly cleaned by methanol and then dried under nitrogen flow before dipped into prepared sol gel precursor solutions. The fiber was immersed in the precursor solution and coated through a dip coating procedure. Repeated dip coating processes were used to achieve desired film thickness (>1 μm). The coated length was 15 cm and dried in the air at the room temperature overnight to allow thorough hydrolysis process. Afterwards,

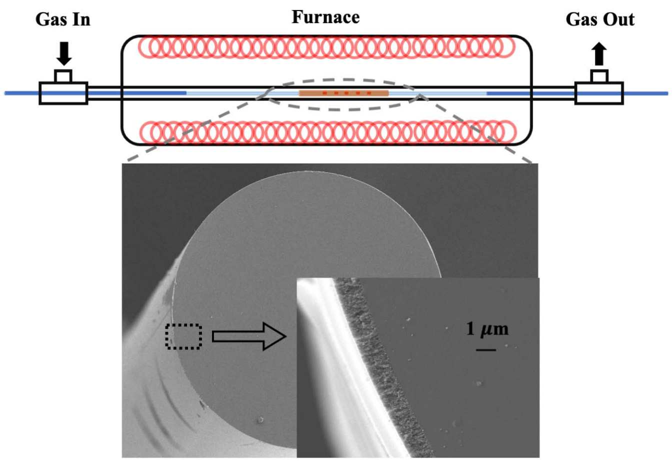

Fig. 2. The schematic illustration of Pd-doped TiO_2 thin films on IFPI sensors. The enlarging image is the SEM cross-section view of Pd-doped TiO_2 film.

the coated optical fiber was inserted into a tube furnace for annealing as shown in Fig. 2. The coated thin film was first heated up to 130 °C and stayed for 1-hour. Then the coated fiber was heated up to 600 °C at a rate of 3 °C per minute with 1-hour dwelling time to achieve calcination process. Once the calcination is completed, the temperature was cooled down to 300 °C at a rate of 3 °C per minute. When temperature cooled down to 300 °C, the air atmosphere was transferred to 5 vol. % combination of hydrogen and nitrogen to reduce PdO inside of TiO_2 film into Pd nanoparticles for another 1-hour [14], [16]. The cross-section view of Pd-doped TiO_2 coated optical fiber was investigated by Zeiss SIGMA VP scanning electron microscope (SEM) as shown in Fig. 2. The coating thickness was around 1.3 μm with a dense, uniform and homogenous finish.

III. Results and Discussion

To characterize the Pd-TiO_2 film coated IFPI sensors and their multiplexable performance, five IFPI sensors was inscribed by the femtosecond laser. Their reflection spectra were interrogated by an Er-doped fiber Amplified Spontaneous Emission (ASE) broadband source and a CCD spectrometer (Bayspec). Fig. 3 (a) shows a reflection spectrum (blue trace) of the sensor array. The envelope of the light source used for the interrogation is superimposed in Fig. 3(a) in black trace. The normalized reflection spectrum, after removing the light source envelope, reveal interference patterns of the IFPI sensor array shown as the orange trace in Fig. 3(a).

The cavity lengths of all IFPI sensors were determined in real-time through a non-zero-padded fast Fourier transform (FFT) and Buneman frequency estimation [17]. Based on the normalized IFPI sensor array spectrum as shown in Fig. 3 (a), the demodulated cavity length for these five IFPI sensors were 485 μm, 645 μm, 791 μm, 967 μm and 1118 μm, respectively, as shown in Fig. 3 (b). These demodulation results are consistent with the initial femtosecond laser fabrication setting as shown in the inset of Fig. 1.

To perform pH measurement, the fiber with IFPI sensor array was attached to a bridge-like holder as shown in Fig. 3 (c). Fiber sensors were immersed in sample solution for

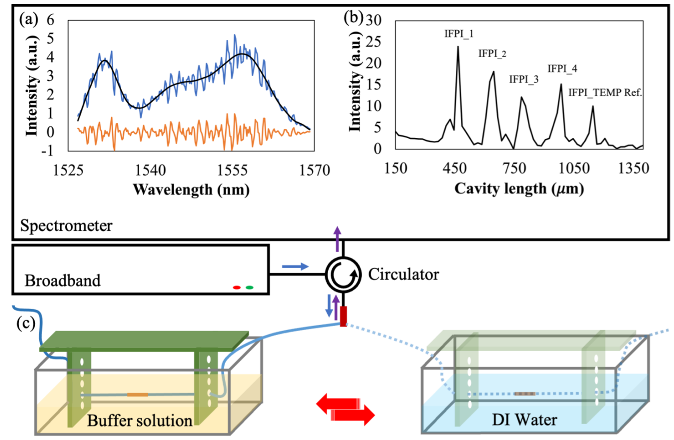

Fig. 3. The schematic illustration of pH sensing setup. (a) The reflection spectrum of sensor array was captured by the spectrometer (blue trace). The normalized interference spectrum of five IFPI sensors was plotted in orange trace. The spectrum of the ASE optical source was plotted in black line. (b) After demodulation, the corresponding IFPI cavity length would be able to access with 485 μm, 645 μm, 791 μm, 967 μm and 1118 μm, respectively. (c) The schematic diagram of pH measurement setup.

2 minutes and then rinse in DI water for another 2 minutes before reuses. All pH solutions tested in this work are standard pH solution purchased from Fisher Scientific without any additional dilution. The characteristics of un-doped and Pd-doped TiO_2 materials were both studied in this work and synthesized using the same sol-gel method.

In order to accurately characterize the response of the IFPI sensors, 4 out of 5 IFPI sensors (IFPI_1 to IFPI_4) inscribed in one optical fiber were coated with pH sensory thin film and the last one IFPI sensor with cavity length of 1118 μm was utilized as temperature reference without any coating (IFPI_TEMP Ref). Using Buneman frequency estimation [17], the smallest strain change exerted in fiber sensors, which can be resolved by the interrogation system, was estimated to be 64-nε. During the real-time cavity length monitoring, the response of Pd-doped TiO_2 sensory thin film coated IFPI sensors were obtained under different pH level from pH = 1.0 to pH = 7.0 as shown in Fig. 4 (red circles). For sensors coated with Pd-doped TiO_2, change of pH value from 1.0 to 7.0 yield significant tensile strain changes from ~30-$\mu\varepsilon$ (pH = 1.0) to ~6-$\mu\varepsilon$ (pH = 7.0). In order to exam the catalytic effect of Pd doping, the IFPI sensors coated with un-doped TiO_2 was also investigated. The strain response of un-doped TiO_2 coated IFPI sensors to pH level from1.0 to 7.0 changes from ~6-$\mu\varepsilon$ (pH = 1.0) to ~1-$\mu\varepsilon$ (pH = 7.0) as the series of blue triangles shown in Fig. 4. These results highlight importance of Pd as catalyst to amplify sensor responses. The temperature reference IFPI sensor was also monitored at all times. The result was plotted in Fig. 4 (green crosses). pH value changes from 1.0 to 7.0 yield a constant 0.43-$\mu\varepsilon$ strain change at room temperature. Using the uncoated IFPI, the fluence of temperature fluctuations of the aquatic solution can be eliminated leading to more accurate pH measurements.

These changes are consistent with all coated sensors. This tensile strain change when IFPI sensors exposed to various pH testing solutions could sufficiently prove the sensitivity of

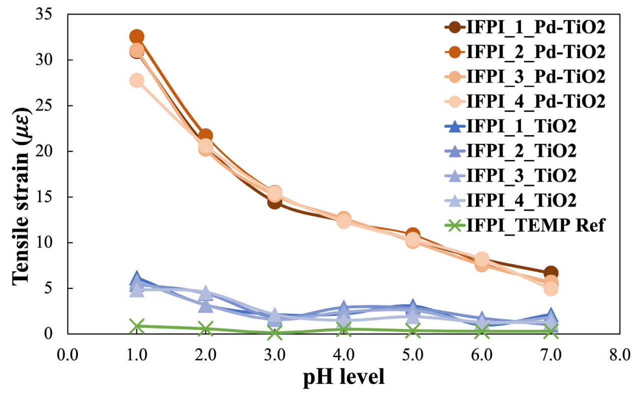

Fig. 4. The tensile strain response of IFPI sensors under various pH level from 1.0 to 7.0 with different pH-sensing coating materials.

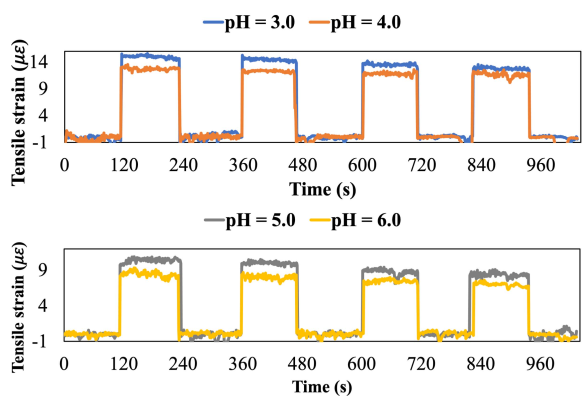

Fig. 5. The endurance of IFPI sensors with cyclic IFPI sensors immersion under pH measurement of 3.0, 4.0, 5.0 and 6.0.

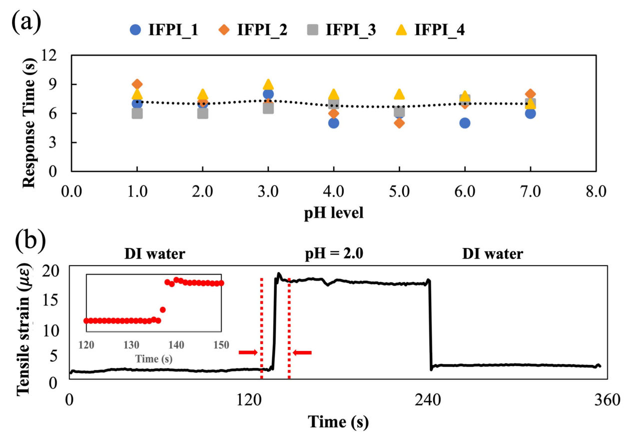

Fig. 6. (a) The response time of Pd-doped TiO_2 coated IFPI sensors under various pH level from 1.0 to 7.0. (b) Response time of a pH sensor (IFPI_4) under pH = 2.0 with respect to DI water.

Pd-doped TiO_2 film. A large tensile strain can be observed when IFPI sensors exposed to a low value pH solution. The tensile strain is proportioned to the concentration of H^+ ions. The underlying mechanism of hydrogen-induced tensile strain exerted onto the Pd-doped TiO_2 coated IFPI sensors can be attributed to the Pd hydride formation inside the TiO_2 film matrix which would stretch the FP cavity to induce the tensile strain. Those dispersed Pd nanoparticles on a TiO_2 film with a large surface area has been proved to effectively prevent the agglomeration and to increase the accessible surface area of reaction [9]. During the pH-sensing film annealing process, the intrinsic defects such as oxygen vacancies exist in the non-stoichiometric form of TiO_2 material [18]. When hydrogen gas is introduced, activated hydrogen atoms reduce PdO to metallic Pd which returning electrons back to TiO_2 where reduce Ti^{4+}ions to Ti^{3+} ions [18]. When the sensor submerged into pH testing solutions, the trapped electrons in titanium lattice site was released and reduced H^+ from aqueous solution back to H atoms assisted by Pd nanoparticles as catalysts [19]. Then the adsorption of hydrogen near Pd nanoparticles occupied interstitial sites in the Pd lattice that triggered the formation of Pd hydride.

The reversibility and reproducibility of IFPI sensors were also investigated by repeatedly immersing IFPI sensors into certain pH buffer solution and DI water with 2 min interval at room temperature for a period of time. The tensile strain time-dependent response of an IFPI sensor with cavity length of 791 μm was demonstrated at Fig. 5. The response of IFPI sensors to the multiple cyclic immersion at pH level of 3.0 $\sim$ 6.0 were selected to demonstrate. This result shows an excellent reversibility of Pd-TiO_2 coated IFPI sensors under acid range. The tensile strain truly had a decedent tendency when submerged under aqueous atmosphere for a long time. This phenomenon broadly monitored in this work which would explain by the intrinsic hydrophobicity feature of optical fiber.

Response time was another important factor to evaluate the performance of sensor behavior. The definition of response time for pH measurement was the duration of the tensile strain change reaching the saturation point when IFPI sensors moved from DI water and immersed into pH testing solution. The response time of four Pd-TiO_2 coated IFPI sensors to various pH level were demonstrated in Fig. 6. Compared to hydrogel-based fiber-optic pH sensor with the response time up to 24 s[20]–[22]. The response time of Pd-TiO_2 coated IFPI sensors proposed in this work can roughly stay constant at different level of pH values. The average response time of IFPI sensors monitored in various pH level was around 7 s with the maximum standard deviation of 2.8 s. The reason for this fast response time could be explain by the nanoparticle of Pd prepared for a better catalytic activity rather than using the thermal cross-linked polymers.

IV. Conclusion

In summary, this paper demonstrates a femtosecond laser-induced intrinsic Fabry-Peìrot interferometric sensor array that can perform multi-points pH and temperature measurement in aquatic solutions. A low-cost sol gel coating technique was used to homogeneously deposit Pd-doped TiO_2 metal oxide film on fiber sensors as pH sensory materials. The pH was tested in a range of 1.0 to 7.0 by directly monitoring Fabry-Peìrot cavity length change using a white-light demodulation algorithm. The sensor exhibits a good reversibility and reproducibility under aqueous solution with various acidity. The response time was observed constantly for all pH levels are 7 s. The sensor technology demonstrated in this paper has a potential to perform multi-point pH measurements using a single fiber.

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