A Polyaniline-Coated Integrated Microfiber Resonator for UV Detection

We present a UV detector based on an integrated microfiber resonator. In fabrication, a thin layer of polyaniline (PAni) is deposited on a ~1-mm-diameter microfiber ring resonator-the sensing region of the detector. Red shift is observed in the output spectrum when PAni is irradiated with a UV light with a peak wavelength at 365 nm. This phenomenon can be due to the high absorbance in the UV region and photothermal effect of PAni. The wavelength shift is linearly proportional to the UV light intensity and the measured sensitivity is 6.61 nm/(W·cm^{-2}).

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SECTION I
INTRODUCTION

MICROFIBER device are well known for their simple fabrication process, high flexibility, strong evanescent field and high sensitivity to variation in the ambient condition. Taking advantage of those properties, different microfiber devices have been developed for numerous applications for instance electrically controlled tunable microfiber filter [1], [2], industrial sensors for numerous measurands such as refractive index, temperature [3], [4], [5] and biochemical substances [6]. Hybrid microfiber devices that integrate more than one optical structure have also been proposed. For example, a combined Mach Zehnder/Sagnac Interferometer structure fabricated from a single microfiber has been experimentally demonstrated. Such device possesses a spectral properties suitable for optical add/drop filtering in the WDM network [7]. Fiber-optic sensor incorporated with PAni has been suggested for chemical detection. The output intensity of the sensor with different chemical vapours due to the variation of light absorbance and refractive index of PAni vary when it is exposed to different chemical vapours [8].

One of the commonly used detection methods in microfiber sensors is spectral shift detection in which the resonance wavelength in the output spectrum linearly shifts with the parameter of interest. For example, the resonance wavelength red-shifts as the ambient temperature of the microfiber resonator increases. The strong temperature dependence of optical microfiber devices can be well explained by the thermal expansion effect and thermo-optic effect in silica glass. Recently, microfiber current sensors have been demonstrated based on the idea of thermally induced resonance wavelength shift in Microfiber Resonator [9], [10]. A thin conductor wire is wrapped by a microfiber resonator in which the temperature is influenced by the heat generated by the conductor wire when there is a current flow. Thus, output spectrum of the resonator red-shifts with temperature increase.

Some optical fiber devices suffer from intermodal interference between fundamental mode and higher order modes or different polarization modes in waveguides which often results in undesired output spectrum. Spectrum Differential Integration (SDI) analyzing method is proposed to provide accurate determination of wavelength shift in the spectrum [11], [12].

In this work, we present a UV detector fabricated from an Integrated Microfiber Resonator (IMR) that comprises of an MKR in a Sagnac loop reflector. The MKR is the sensing part of the device and is coated with PAni which has high absorbance in the UV region. When exposed to an On-Off modulated UV light, spectral shift in the output spectrum is observed and analyzed using SDI method to eliminate the influence of intermodal interference in the microfiber system.

SECTION II
EXPERIMENTAL METHODS

Preparation of the PAni coated Integrated Microfiber Resonator first involves two independent processes: (A) Preparation of PAni solution and (B) Fabrication of integrated microfiber structure. Procedures of both processes are detailed below.

A. Preparation of PAni Solution

PAni was synthesized using aniline (Ani) as monomer, dioctyl sodium sulfosuccinate (AOT) as dopant and ammonium persulphate (APS) as oxidant through chemical oxidative method. First, Ani was added into the AOT acidic solution and stirred for 2 hours. Then, APS solution is slowly added to the Ani/AOT solution. Polymerization was carried out at low temperature for 24 hours. The resultant sample was washed with distilled water for a few times to remove the unreacted AOT, APS and monomers. Extraction and dilution were carried out to obtain the desired PAni concentration [13]. Finally, the resulted PAni was characterized by Ultraviolet-Visible (UV-Vis) and Fourier Transform Infrared (FTIR) spectroscopy. Fig. 1(a)–(c) show the chemical structures of the general PAni and PAni in quinoid and benzenoid respectively. The presence of benzenoids and quinoids form in the PAni chains are significant property of a conducting form of a PAni emeraldine salt. These chemical structures can be determined through the characterization of functional group observed from the FTIR peaks while the special conjugated property (quinoid and benzenoid rings) of PAni can be determined from the UV-Vis spectrum.

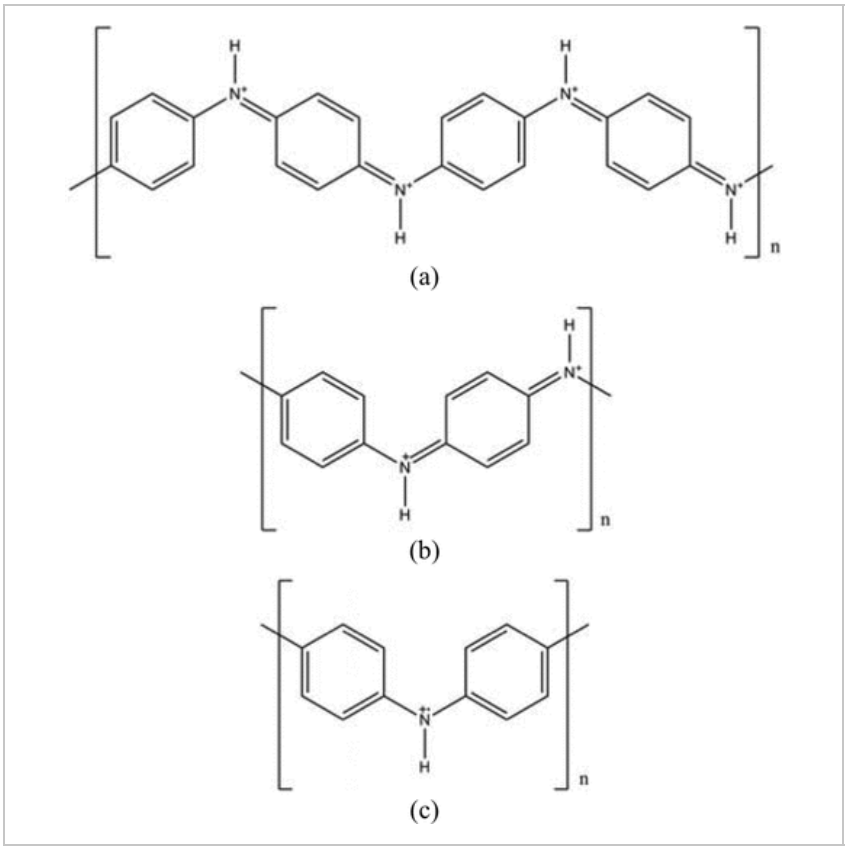

Fig. 1. Chemical structure. (a) General PAni and PAni in both (b) quinoid, and (c) benzenoid forms.

In the absorbance spectrum Fig. 2(a), two peaks constitute the absorbance in the UV region. The peak at 353 nm corresponds to the $\pi - \pi^*$ transition of benzenoid rings while the shoulder peak at 434 nm is due to localized polaron bands of protonated PAni [14]. For FTIR spectrum shown in Fig. 2(b), peaks at 1614 cm^{-1} and 1460 cm^{-1} can be attributed to quinoid and benzenoid rings vibration in the polymer chain, respectively. Furthermore, it was noted that the peaks at 1207 cm^{-1} and 1182 cm^{-1} indicate that the C-N stretching in bipolaron polymer is highly doped and exist in conducting form. The peaks at $\sim$3600-3000 cm^{-1} and $\sim$3000-2800 cm^{-1} correspond to the-N-H and-C-H stretching vibrations of PAni, respectively. These results reveal the PAni exist in the emeraldine salt form (conducting) [14], [15]. Both UV-Vis and FTIR spectra confirmed the chemical structure of the resulted PAni.

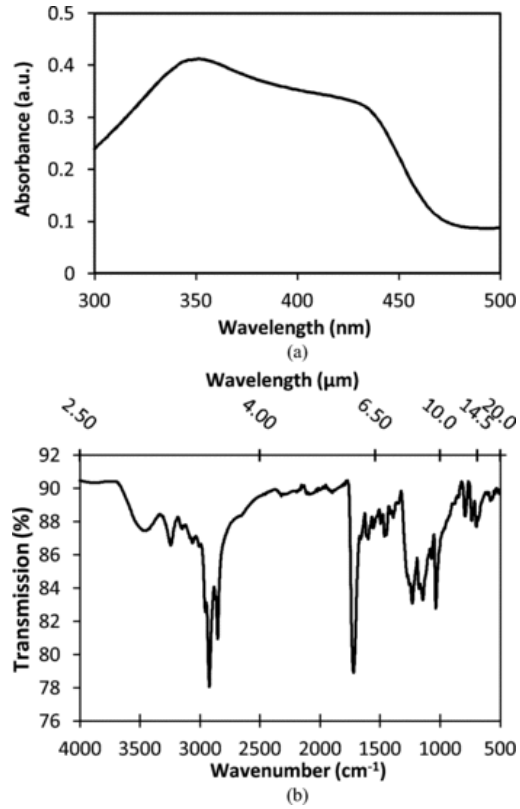

Fig. 2. Chemical structure determination of PAni. (a) UV absorbance. (b) FTIR spectrum.

B. Device Fabrication

Fig. 3(a) provides a schematic illustration of fabrication flow for the proposed UV detector. (i) First, a thin layer of low index Teflon (Dupont, 400S2-100-1, $\mathrm{RI}\sim 1.29$) with a thickness in the range of 12–14 $\mu\mathrm{m}$ was deposited on a 75×25 mm glass substrate using drop coating method. (ii) Then, a $\sim 5\mathrm{cm}$ long microfiber with waist diameter of 3–4 $\mu\mathrm{m}$ was fabricated from an SMF-28 fiber based on flame brushing technique [16]. By using the running end of the microfiber, an overhand knot was made at the center part of the microfiber to create an MKR. The untapered end of the microfiber was then twisted to form a Sagnac loop with the MKR located inside to complete the IMR. The dimension of the Sagnac loop is approximately 4×10 mm^2. The IMR was then laid on the glass substrate and the low-index layer acts as the underclad for the microfiber structure.

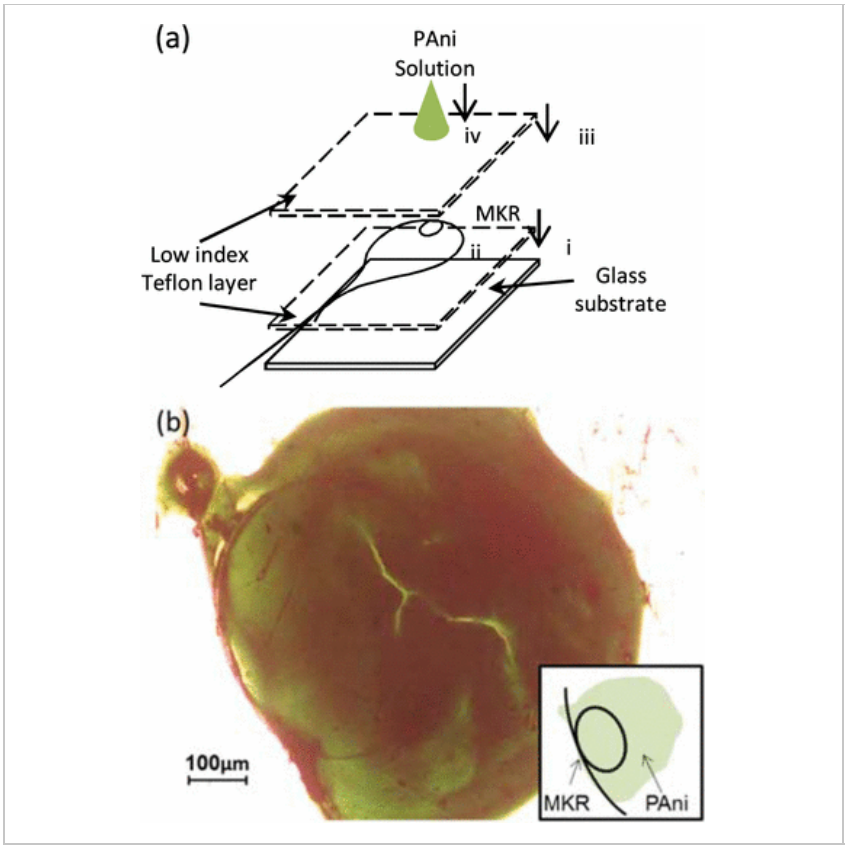

Fig. 3. (a) Fabrication steps of the proposed detector. Sequence is stated by the Roman numbers. (b) Microscope image of an MKR coated with a thin layer of PAni. There is a thin layer of low index Teflon in between PAni layer and MKR to prevent light in MKR from total leakage loss into the PAni. Inset: illustration of the proposed detector.

(iii) After that a Teflon $(\mathrm{RI}\sim 1.29)$ liquid solution was poured on the IMR to create another low-index layer to cover and protect the MKR. The output spectrum of the device varies when the Teflon liquid solution was applied. The solution was left to dry for 30 minutes. After that, the device was embedded in the solid Teflon layer and the output spectrum was stable. Detailed description of the IMR fabrication is described in [3]. (iv) The synthesized PAni solution is deposited onto the knot region of the detector by applying a small drop of PAni solution onto the MKR and the droplet is left to dry for a few seconds. Generally, PAni has higher refractive index and it is optically lossy. The low-index Teflon layer between MKR and PAni/glass slide is to ensure total internal reflection of light within the MKR and to prevent the light from leakage loss to the PAni or glass slide that has higher refractive index than the microfiber. Fig. 3(b) shows a microscope image of an MKR coated with a layer of PAni. The Teflon layer between the MKR and PAni layer provides an optical protection to the MKR from the PAni which is an optically lossy material. Toluene, which is used as solvent in the PAni solution, is also a good solvent that can dissolve the Teflon layer. An excessive amount of PAni solution used in the deposition may dissolve the Teflon layer and result in a very high transmission loss in the microfiber system. The solubility of the Teflon layer can be prevented by expediting the evaporation process to minimize the evaporation time hence reduces the interaction time between toluene and Teflon layer. The deposition was conducted in a high temperature environment by heating the MKR on a hotplate. At a temperature of 50°C, the PAni dried within $\sim 4\mathrm{seconds}$. The transmission loss of the microfiber detector was monitored closely during the deposition process by connecting the proposed detector to a wideband source and an Optical Spectrum Analyser (OSA). During the deposition, we observed a small fluctuation in the output spectrum right after a PAni solution droplet was applied on the MKR. The spectrum stabilized after the droplet dried up. Fig. 4 shows the output spectra of the UV detector before and after the deposition of PAni. There is a small variation in the output spectrum while the spectral power remains almost at the same level. The wavelength shift is due to the micro structure changes of the MKR during the deposition of the PAni solution. The solvent of the PAni solution may dissolve some portion of the Teflon layer thus disrupting the embedded MKR structure and causes a variation in the output spectrum.

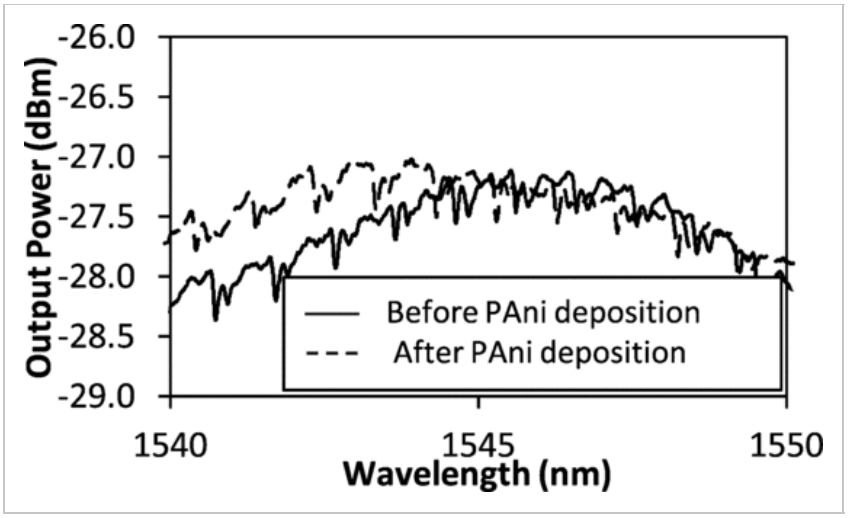

Fig. 4. Output spectra of the UV detector. Solid: before deposition of PAni. Dotted: after deposition of PAni.

SECTION III
Experimental Results

Fig. 5 shows the experimental setup of the proposed UV detector. EDFA provides a wideband source to the detector. Circulator is used to circulate the reflected light which is the output of the detector to the Optical Spectrum Analyser (OSA, Anritsu MS9710B). The UV sensitivity measurement of the proposed detector was carried out by irradiating the PAni coated MKR with an On-Off UV light from a mercury arc lamp (peak wavelength at 365 nm). The output spectrum of the detector was analyzed and recorded repetitively every 2 seconds by the OSA that is controlled by a computer using Labview data acquisition program. The UV intensity impinging on the detector is controlled by manipulating the distance between the diverging UV light source and the detector. At a distance of 6 mm, the spot size of the UV light at the source aperture is $\sim 1\mathrm{cm}$ in diameter and the area is $\sim 0.79\mathrm{cm}^2$. The UV light diverges and the spot size is linearly proportional to the distance between the light source and detector while the light intensity is inversely proportional to the square of distance. As a reference, the intensity of the UV was measured using a commercial power detector (P/N: UP12E-10S-H5-D0, Gentec-EO). The excited higher order modes in the microfiber structure may interfere with the fundamental mode which results in irregular interference fringes in the output spectrum and erroneous measurement of wavelength shift. This can be prevented by analyzing the output spectra using SDI method to eliminate the influence of intermodal interference in the microfiber structure.

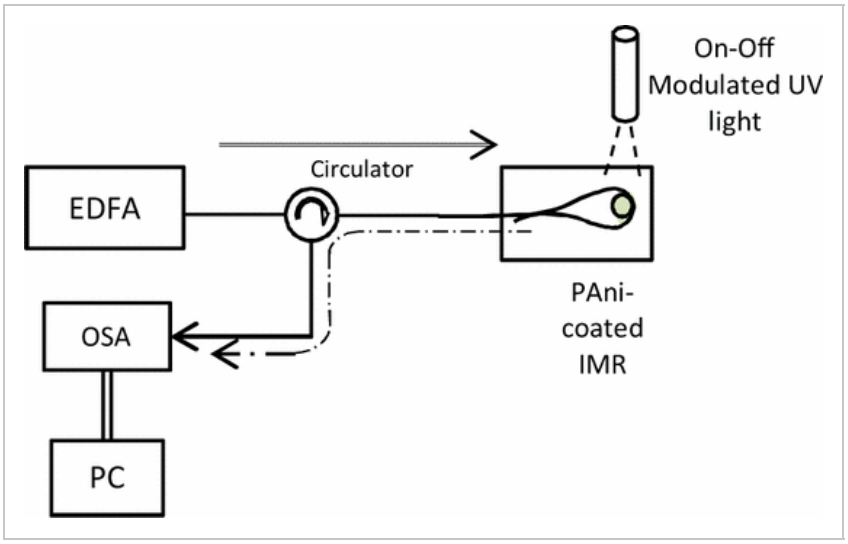

Fig. 5. Experimental setup of the proposed UV detector.

Fig. 6(a) shows the output response of the proposed detector to the modulated UV light with a period of 40 s and 50% duty cycle. The dotted square wave in the graph indicates the periodic UV irradiation on the detector. From the output curves, rapid red shifts in the spectrum occur at the moment when the detector is exposed to UV light driven by the heat generated from the PAni, a result of photothermal effect. Fig. 6(b) shows time evolution of the output spectra of the detector during the "On" state.

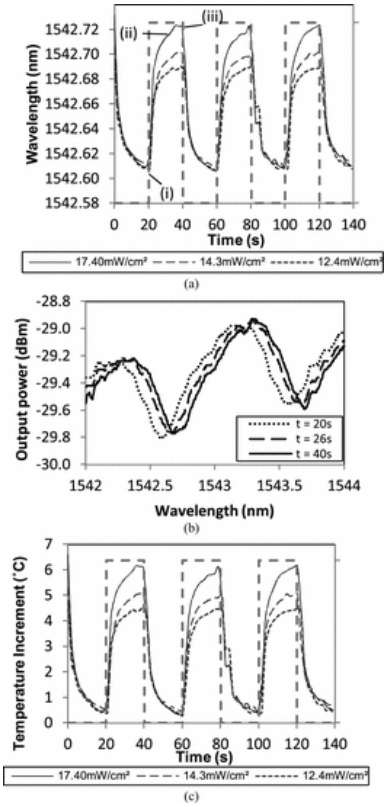

Fig. 6. (a) Resonance wavelength responses of the detector to on–off UV light of different light intensities: 12.4, 14.3, and 17.40 $\mathrm{mW/cm^2}$. (b) Output spectra of the detector for UV light intensity of 17.4 $\mathrm{mW/cm^2}$ during "on" state. (c) Calculated temperature increment at the detector.

The spectra correspond to UV irradiation intensity of 17.40 $\mathrm{mW/cm^2}$ at different duration labeled as (i)–(iii) in Fig. 6(a). The resonance wavelength decayed to its original position during the "Off" state. The short time constant can be attributed to the open top surface of the PAni to ambient air, allowing more dissipation of heat from the UV-irradiated PAni by convection. The magnitude of the oscillating output curve is linearly proportional to the intensity of the UV light. The peak-to-peak wavelength shifts for UV light intensities of 12.4 $\mathrm{mW/cm^2}$, 14.3 $\mathrm{mW/cm^2}$ and 17.40 $\mathrm{mW/cm^2}$ are 0.08 nm, 0.10 nm and 0.12 nm respectively, corresponding to a sensitivity of 6.61 nm/$(\mathrm{W \cdot cm^{-2}})$. The temperature sensitivity of this IMR was $\sim 20\mathrm{pm/^\circ C}$ which was calculated from the results of digital hotplate reading and the corresponding resonance wavelength shift of the sensor. The value agrees with temperature sensitivity reported in [3]. Based on this temperature sensitivity, a wavelength shift of 0.12 nm is equivalent to a temperature increment of $\sim 6^\circ \mathrm{C}$. Fig. 6(c) shows the time response of temperature increment at the sensing part of the detector, calculated based on data in Fig. 6(a).

Fig. 7 shows the resonance wavelength response to the exposure of On—Off UV light of increasing intensity. First, the UV source was placed at a distance of 17 mm from the detector corresponding to light intensity of $\sim 10\mathrm{mW/cm^2}$. The distance was reduced by 2 mm every 20 s to increase the UV exposure power on the UV sensor until it reached 2 mm. In this way, the UV light intensity at the detector was increased almost linearly from 10 $\mathrm{mW/cm^2}$ to 22.5 $\mathrm{mW/cm^2}$. The dotted square wave in Fig. 7 indicates the duty cycle of the UV. It is observed that the shift of the resonance wavelength becomes larger when the UV sensor is exposed to the UV light of higher intensity. Similar test was performed on the IMR without any deposition of PAni in the system. No significant variation in the output spectrum was observed in the test. Another test was performed on the PAni coated MKR using a modulated red light, a wavelength in the region where absorbance of PAni is low. The spectrum too is unaffected by the irradiation.

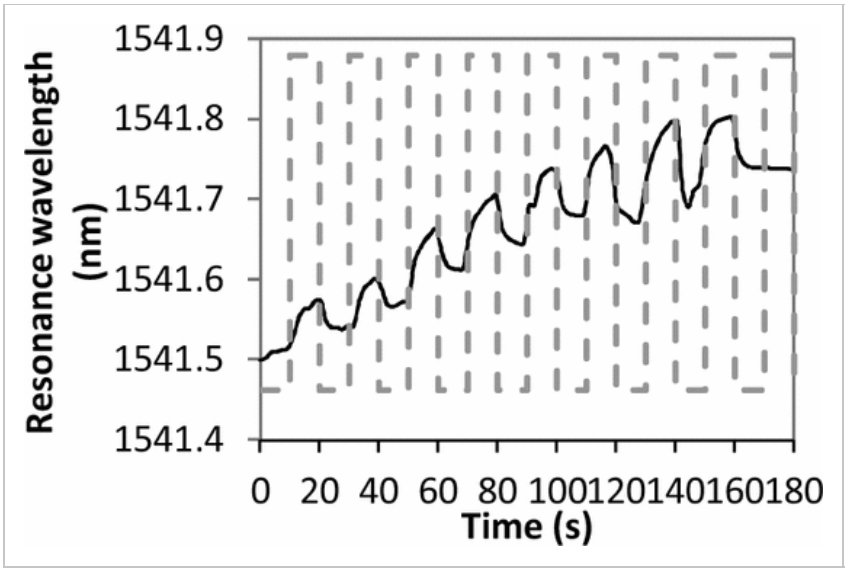

Fig. 7. Resonance wavelength responses to the exposure of UV light for on and off states at different exposure distance or power.

To improve the sensitivity and minimum detectable signal of the detector, a number of approaches can be taken for instance increasing the thickness of the PAni deposition so that more heat can be generated by the PAni and to trigger larger resonance wavelength shift in the detector. The thickness of Teflon layer that separates PAni from MKR can be reduced to decrease the temperature difference between them and improve the sensitivity of the detector.

SECTION IV
CONCLUSION

In conclusion, we presented a miniature PAni coated IMR using a microfiber. We have shown that the exposure of the detector to UV source will cause the resonance wavelength of the detector to shift to a longer wavelength with a sensitivity of 6.61 nm/$(\mathrm{W}\cdot\mathrm{cm}^{-2})$ due to the photothermal effect. In comparison with the conventional UV-detector, our proposed detector possesses numerous advantages including immunity to electromagnetic noise, compact size, good physical flexibility and simple fabrication technique. With the assistance of SDI method, the resonance wavelength shift can be accurately determined and subsequently provides accurate UV detection. The proposed UV detector offers solutions where conventional detectors are unsuitable. Based on our knowledge, novel PAni coated IMR using microfiber for UV detection is reported for the first time here.

FOOTNOTES

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Chemicals, Detectors, Fabrication, Optical resonators, Sensitivity, Temperature sensors

INSPEC: Controlled Indexing

fibre optic sensors, optical polymers, optical resonators

INSPEC: Non-Controlled Indexing

UV detection, UV detector, UV light intensity, UV region, microfiber ring resonator, photothermal effect, polyaniline-coated integrated microfiber resonator, red shift, sensing region, wavelength 365 nm, wavelength shift

Authors Keywords

Microfiber, UV, polyaniline, sensors

CORRECTIONS

None