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To cite this article: Nurfatim Musa *et al* 2019 *J. Phys.: Conf. Ser.* **1371** 012017

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Tapered single-mode optical fiber-based on localized surface plasmon resonance as refractive index sensor

Nurfatin Musa¹, Hazri Bakhtiar^{1,2}, Ganesan Krishnan^{1,2}, Harith Ahmad⁴,
Sulaiman Wadi Harun³, Muhammad Safwan Abd Aziz^{*1,2}

¹ Department of Physics, Faculty of Science, Universiti Teknologi Malaysia, 81310 Skudai, Johor, Malaysia

² Laser Center, Ibnu Sina Institute for Scientific and Industrial Research, Universiti Teknologi Malaysia, 81310 Skudai, Johor, Malaysia

³ Photonics Engineering Laboratory, Department of Electrical Engineering, Faculty of Engineering, Universiti Malaya, 50603 Kuala Lumpur, Malaysia

⁴ Photonics Research Centre, University of Malaya, 50603 Kuala Lumpur, Malaysia

safwanaziz@utm.my

Abstract. A simple tapered single-mode optical fiber-based on localized surface plasmon resonance was proposed as a refractometer for liquid samples application. The tapered optical fiber (TOF) was fabricated by using a flame-brush method and achieved a waist diameter of 16.5 μm over the length about 20 mm. 3-mercaptopropyltrimethoxysilane (MPTMS) with a mercapto group (-SH) was utilized as a silane agent to immobilized the nanoparticles by strong covalent bonding between sulphur and gold. The sensing mechanism relies on the transmission shifting in the output spectra due to the evanescent field absorption by immobilized gold nanoparticles on TOF surface. The performance of TOF has been evaluated with the surrounding refractive index varied over the range from 1.3330 to 1.4229. The sensitivity of the sensor was recorded at 18 nm/RIU with good repeatability and stability for 70 minutes.

1. Introduction

Fiber optics have attracted a great deal of attention in the field of sensors for detecting various physical and chemical parameters. Of the wide variety of available fiber optics sensors, fiber optics refractometer is promising and attractive in both chemical and biotechnological applications. This particular type of sensor offers many advantages such as simplicity, low-cost system, high sensitivity, and high responsivity towards measurand. On the other hand, sensing activity in hostile environments is made possible due to electromagnetic interference immunity, and compact in size [1, 2].

Among many, tapered optical fibers (TOF) is considered as one of the common types of fiber-based sensors. The evanescent waves propagating on the tapered region of a TOF can interact directly with the surrounding, thus altering the output light signal with any changes introduced to the surrounding medium. In many cases, the evanescent waves interact with an intermediate thin film transducer, which was fabricated to be permeable to particular measurands. Such devices were designed to be highly sensitive towards the surrounding's refractive index RI and or color. As a consequence, one or more wavelengths of the guided light may be selectively absorbed or attenuated. A simple detection scheme could be further used to easily monitor the absorption or attenuation suffered by the guiding light.



In this work, a single-mode optical fiber was proposed as a refractometer to detect the refractive index of surrounding liquid medium based on localized surface plasmon resonance (LSPR) effect. The structure of the optical fiber was modified by tapering it down to smaller diameter, in order to produce evanescent field propagating along with the tapered structure. A thin layer of gold nanoparticles was immobilized on the tapered region as the sensitive coating. The performance of the sensor in term of its sensitivity, stability, repeatability, as well as reversibility, was examined.

2. Experimental Setup

2.1. Synthesis of gold nanocolloid

Gold nanoparticles (AuNPs) employed in this experiment were prepared by Turkevich method. This method is based on the reduction reaction between gold chloride (HAuCl_4) and sodium citrate ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$) respectively. 20 mL of 1.0 mM of HAuCl_4 was stirred and heated until boiled. Upon reached boiling state, a known amount of sodium citrate was added quickly to the solution. As a result, the yellowish color of gold chloride turned colorless before gradually changed to grey-purplish color. The solution was kept heated for several minutes until red-wine color appeared. After that, the heat source was removed and the solution was left at room temperature with further stirred for 10 minutes. The final solution was kept in the refrigerator to ensure the particles size was stable up to a month.

2.2. Tapered fiber LSPR sensor preparation

Standard telecommunication single-mode fiber (G652D) with core and cladding diameters of 10 μm and 125 μm was used for the sensor preparation. The tapered structure was fabricated by the flame-brushing method. During the tapering process, one side of the fiber was kept static and the other side was pulled while the fiber was heated back and forth horizontally by using butane torch (max temperature at 1430 $^\circ\text{C}$). Both sides of fiber were fixed by stage holder as shown in Fig. 1. Approximately 30 mm of the buffer layer was removed from the middle section of the prepared sensor by a mechanical stripper. The uncovered section of the fiber was exposed to the flame for softening purpose, while the fiber being pulled by a linear translational stage.

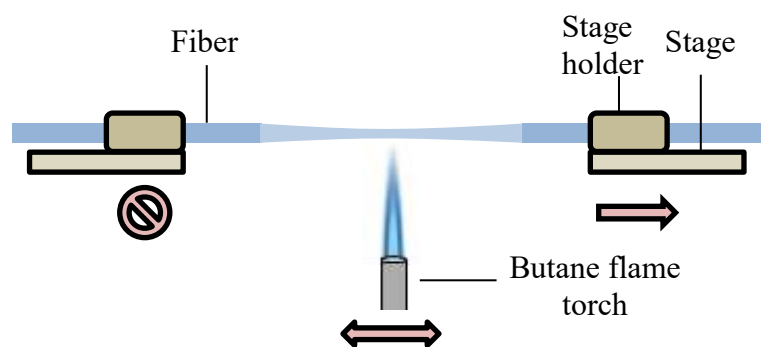


Fig. 1. Schematic diagram of tapered fabrication

The tapered fiber region was cleaned by methanol solution to remove unwanted debris from the fiber surface and let dry. The tapered fiber was then immersed in 1.0 M of sodium hydroxide, NaOH for an hour, to activate the fiber surface with -OH functional group. The tapered fiber was rinsed with distilled water thoroughly and blow-dried. Then, the prepared fiber was thiol-functionalized with 1% of MPTMS in ethanol for 12 hours. After this process, the fiber was again rinsed several times by ethanol to remove unbound monomer from the fiber surface and blow-dried. Afterwards, the tapered fiber was sent for coating with AuNPs for 48 hours through a dip coating process to form a metal nanoparticles layer on the tapered fiber surface. Finally, the coated fiber was rinsed with ethanol and dried. Fig. 2 shows the

schematic diagram of self-assembled monolayer (SAM) of MPTMS on tapered fiber. Ruby-color appearance was visible on the tapered fiber surface by naked eyes.

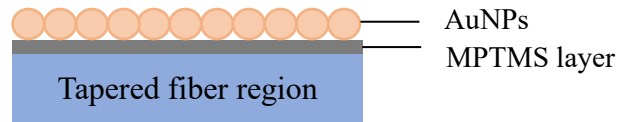


Fig. 2. Schematic diagram of SAM of MPTMS on the tapered fiber surface

2.3. *LSPR sensor setup*

Fig. 3 illustrates the schematic diagram of LSPR sensor system used in this work. One end of tapered fiber sensor was connected to a halogen-tungsten broadband light source and the other end was connected to C175 Thorlabs spectrometer. The spectrometer connected to the computer and the corresponding spectra were recorded in real-time. The sensitive tapered region of the fiber was immersed in solutions with different refractive indices ranges from 1.3330 to 1.4229. Table 1 indicates the refractive indices of the 5 prepared solutions represented in refractive index unit, RIU respectively.

Table 1. Refractive index of samples test

Samples	n1	n2	n3	n4	n5	n6
Refractive index unit, RIU	1.3324	1.3523	1.3728	1.3931	1.4119	1.4254

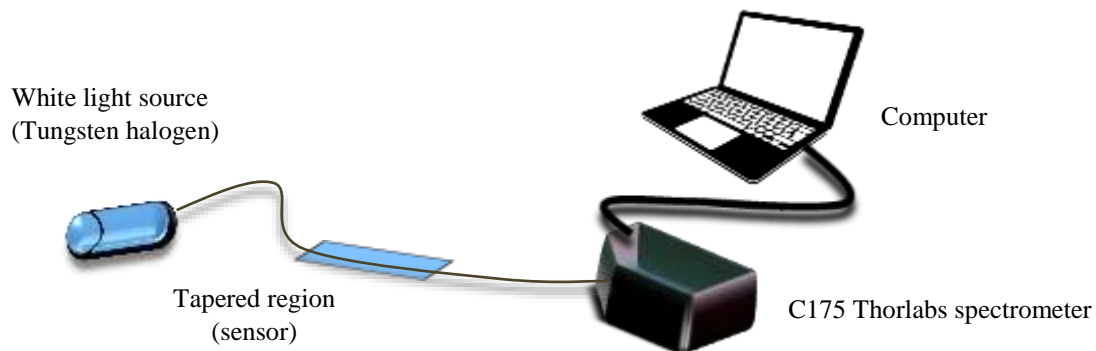


Fig. 3. Schematic diagram of LSPR sensing setup

3. Result and Discussion

3.1. *Gold nanoparticles immobilized on TOFs*

Immobilization of the prepared nanoparticles on the surface of the TOF is examined by scanning electron microscope. As shown, immobilized AuNPs on the surface of TOF is evidenced in Fig. 4(a) & (b) with different resolution. The random distribution and uniformity of AuNPs layer are obtained based

on the results. The mean diameter of deposited AuNPs was measured and shown in Fig. 4(c) which estimated to be 15 ± 2 nm.

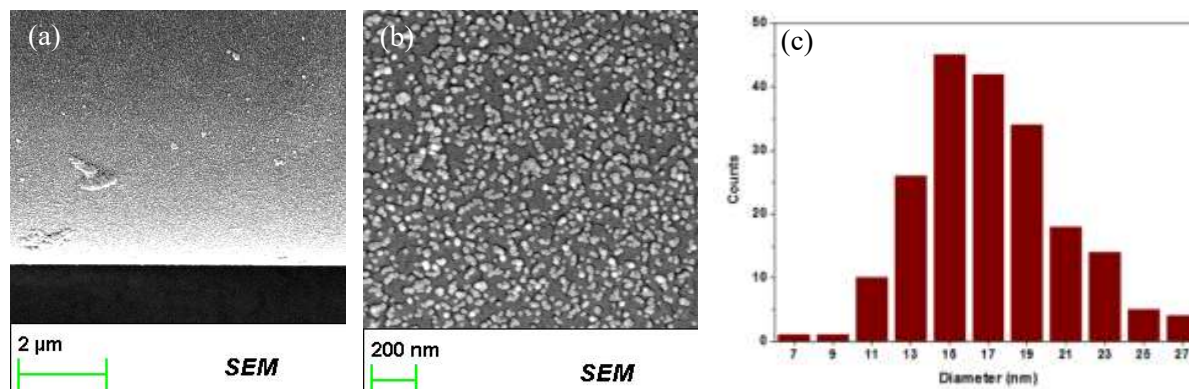


Fig. 4. (a) & (b) SEM images of Au immobilized on TOF surface; (c) Histogram analysis of Au average size based on SEM image

3.2. The sensitivity of TOF based LSPR sensor

Transmission spectra of the sensor were recorded against different refractive indices values of the controlled surrounding media. Fig. 5(a) indicates the output signals obtained from LSPR-based TOF. Based on the figure, it is shown that TOF has transmittance trends with the highest reading in n_1 which refer to the lowest refractive index followed by n_2 until n_6 . The transmission of light decreases with the increment of refractive index unit (RIU) of liquid samples due to the attenuation loss. On top of that, a significant shifting has been observed in the output spectrum as the surrounding refractive index varied from low to high due to the LSPR effect. Immobilized AuNPs on the surface of TOF acts as a new cladding and the excitation of surface plasmons on AuNPs occurs due to the presence of evanescent field at the metal-dielectric sensing layer interface. Resonance state was achieved when the evanescent field produced from incident light matched with the surface plasmons oscillation of the AuNPs, resulting in a sharp dip in the transmitted light intensity at a particular wavelength [3]. Applying the different refractive index at the surrounding surface may vary the resonance wavelength at each RIU based on the coupled light that exhibits the modulated transmittance spectrum.

In order to analyse the performance of TOF LSPR-based sensor, the spectral shift in the transmitted signal was recorded in response to refractive index changes. The result was plotted and shown in Fig. 5(b). It is shown that the proposed LSPR sensor exhibit a good linear dependency. A correlation coefficient, R^2 of 0.95490 was calculated with the corresponding sensitivity recorded at 18.4931 nm/RIU.

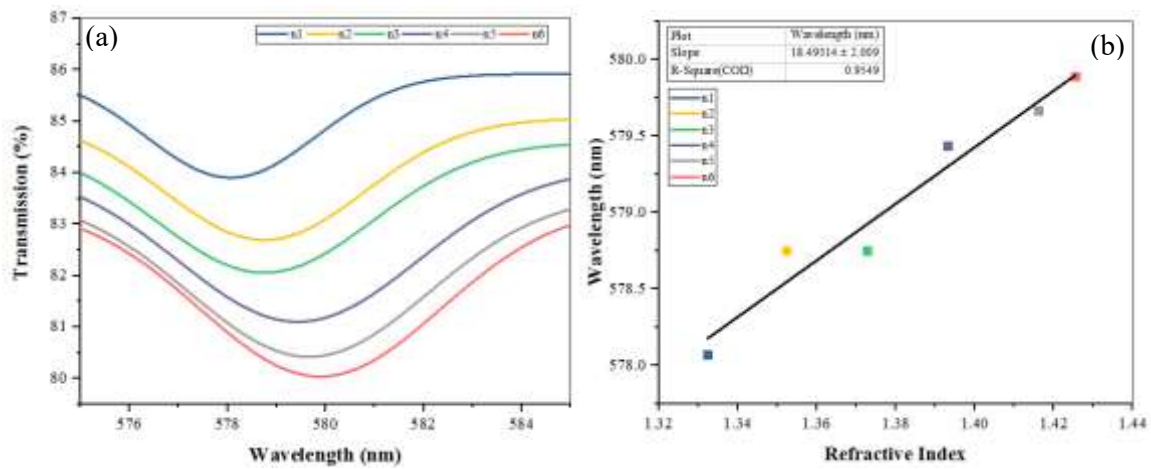


Fig. 5. (a) Transmission spectra for TSF; (b) The sensitivity analysis of TSF

3.3. Stability, Repeatability and Reversibility

The stability of the sensor was determined by continuously monitoring its output spectra for 70 minutes in 3 different solutions with RIU of 1.3324, 1.3523 and 1.4254. The controlled media was purposely selected at low, medium and high indices in order to test the actual stability of the sensor across the whole range of RIU. The peak intensities values for every reading were recorded and illustrated in Fig. 6. Result suggests that the output response of proposed LSPR-sensor is constant with very little variation over the period of 70 minutes.

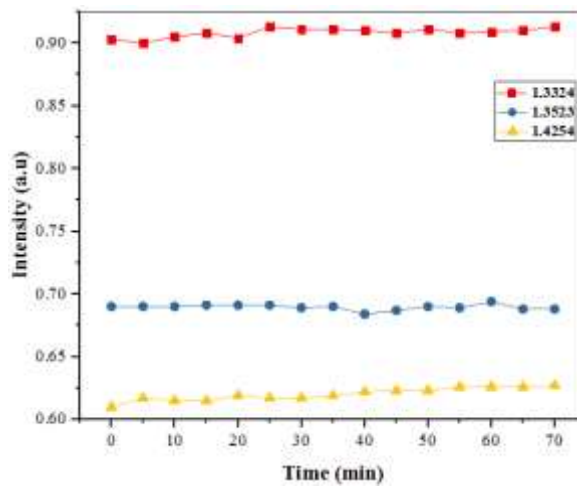


Fig. 6. Peak intensity of TSF LSPR-based sensor for 70 minutes

In order to evaluate the repeatability of the sensor, the output spectra were recorded for a complete 3 cycle periods. During the test, the sensor was subjected to the controlled refractive index solution ranges from 1.3324 to 1.4254. Fig. 7(a) shows the variations of intensities between 3 repeated cycles recorded against corresponding refractive index values. Fairly good repeatability is shown regarding the low degree of variation among those 3 cycles of measurements. To confirm the reversible performance of the sensor, measurements were made by both ascending and descending fashion. Refractive index of the surrounding medium was varied between 1.3324 to 1.4254 RIU and the corresponding transmission were recorded at 545.56 nm wavelength. Results are shown in Fig. 7(b) indicates that the sensor repeated

the same transmitted intensity with little variation. This confirms the reversible and stable behaviour of the device in detecting refractive index variation of the surrounding liquid medium.

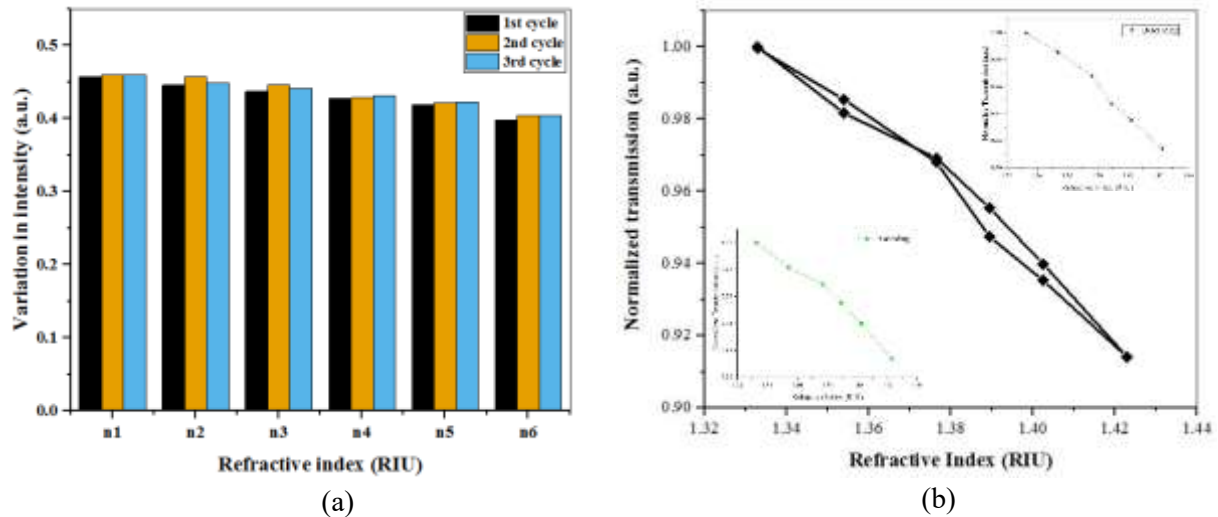


Fig. 7. (a) Histogram analysis of repeatability test for LSPR-based sensor; (b) Reversibility trends of LSPR-based sensor

4. Conclusion

In this work, Au nanoparticles were successfully synthesized by using turkevich method. The immobilization of Au on the TOF surface by SAM method was successfully employed which verified by SEM analysis. The proposed optical fiber sensor shows a relatively high sensitivity of 18.4931 nm/RIU, on top of that, it also shows good repeatability in 3 cycle periods, better stability for 70 minutes and great reversibility with a little variation. Further optimization of fiber geometry or coating parameter may result in more compact refractometric sensing with improved performance.

Acknowledgement

Authors would like to acknowledge the financial support under GUP Universiti Teknologi Malaysia through the Teir 1 fund with vote 18H67.

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