Performance of Single Mode Tapered Optical Fiber Sensor Based on Localized Surface Plasmon Resonance (LSPR) for Various Coating Time

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Abstract. Tapered optical fiber (TOF) sensor based on localized surface plasmon resonance is demonstrated for refractive index sensing application. The single-mode fiber (G652D) with 10/125 µm of core/core-cladding diameter was tapered by flame brushing method and achieved a waist diameter of approximately 15 µm. Mercapto group (-SH) of 3-mercaptopropyltrimethoxysilane (MPTMS) was utilized for immobilization of gold nanoparticles (AuNPs) by self-assemble monolayer (SAM) method on fiber surface. Evanescent field at the TOF sensing region excites the localized surface plasmons of AuNPs, with average mean diameter 15 ± 2 nm as evidence by scanning electron microscopy (SEM). The effect on deposition time was examined on three different samples; S1, S2 and S3 with 24h, 48h and 72h of coating time respectively. TOF sensor performance was evaluated with surrounding refractive indices ranges from 1.3324 to 1.4254. The sensing mechanism is based on the modulation of output light signal that represent by transmission shifting spectra. The optimum sensitivity was obtained for S2 at 18 nm/RIU with good repeatability, reversibility and stability.

1. Introduction

The use of optical fibers as sensing device has wide applications ranging in physical, chemical and biological applications to sense temperature [1], gas [2], humidity [3] as well as pH [4]. Fiber optics sensor offers many advantages in terms of sensitivity, responsiveness, ease of handling, compact in size and immune to electromagnetic interference. Among many kind of fiber optics-based sensor, tapered optical fiber (TOF) offers the simplest fabrication methods. The evanescent wave propagates along the core-cladding interface at tapered region (reduced diameter section) can interact directly with the surrounding. The direct interaction between evanescent waves with the surrounding media will subsequently provide significant information for sensing purposes.

The combinations of optical fiber sensor and localized surface plasmon resonance (LSPR) have been utilized for the design of simple sensors that are sensitive to changes in the refractive index of surrounding analyte. Among many metallic materials, gold is considered as one of the most popular material for plasmonic application in visible wavelength region compared to other metals like silver or aluminum due to its inert properties which contributed for its stability [5]. In particular, gold-deposited TOF causes the evanescent field produced by guided rays excites the surface plasmons phenomenon at the metal-dielectric sensing layer interface, which is further exploited as LSPR based sensor.
In this study, we examine the effect of coating time on performance of single-mode TOF sensor based on LSPR effect. The optical fiber was modified by tapering down the diameter of a small section on the fiber line in order to promote the evanescent wave on fiber-surrounding boundary. Immobilization of gold nanoparticles by self-assemble monolayer on tapered region act as sensitive layer to excite LSPR. The performance of the sensor was examined by transmission shifting which represent its sensitivity, and detail examination on the stability, repeatability and reversibility of the designed sensor is presented in this work.

2. Materials and Method

2.1 Gold nanoparticles synthesis and deposition
Gold nanoparticles (AuNPs) were prepared based on the reduction reaction of gold chloride (HAuCl$_4$) by sodium citrate (Na$_3$C$_6$H$_5$O$_7$) which known as Turkevich method. 2 ml of 1 vol% sodium citrate solution was added rapidly in boiling solution of 20 ml 1M HAuCl$_4$ which caused the color changes of the solution from light-yellowish to colorless. Further stirring shows the ruby color appearance which indicated the AuNPs was successfully produced. The final solution was cool down in room temperature before dip coating process take place. The particle size analyzer (PSA) analysis was obtained with average size diameter of 15 nm.

The cleaned tapered fiber region was immersed in sodium hydroxide, NaOH for an hour to activate the fiber surface with –OH functional groups. 3-mercaptopropyltrimethoxysilane (MPTMS) with mercapto group (-SH) was utilized as a silane agent to immobilized the AuNPs. 1 vol% of MPTMS in ethanol was introduced on the taper fiber for 12hours. The thiol-functionalized tapered fiber surface were rinsed with ethanol to remove unbound monomer and dried. After that, the tapered fiber was coated with the prepared AuNPs by dip coating method for 24h, 48h and 72h on three different fiber samples denoted as S1, S2 and S3, respectively. Upon completion of the coating process, the Au-coated fiber was rinsed with ethanol and dried.

Figure 1 illustrates the schematic diagram of self-assemble monolayer (SAM) of MPTMS on TOF surface. Halas and his co-workers [6] reported Au coverage on silica surface influences by its terminal which obtained that mercapto (-SH) group; one of hydrophilic group, as a good anchor for AuNPs. The SAM also promotes highly smooth Au distribution [7].

2.2 Experimental setup
Figure 2 illustrates the schematic diagram of LSPR sensing setup. Halogen-tungsten light source (HL2000) with wavelength range of 360 nm to 2000 nm was used as input source. The single-mode fiber (G652D) with 10/125 µm of core/core-cladding diameter was tapered by flame brushing method. The optical fiber was tapered down to approximately 15 µm of diameter size at 25 nm stripped fiber region. One end of the TOF was connected to the light source, and the other end was connected to CCS175 Thorlabs spectrometer with wavelength range of 499 nm to 1100 nm. The tapered region was placed on sample holder as shown in the diagram. Thorlabs OSA software was used to interpret and analysis data spectrum collected from the output signals of the sensor.
3. Result and Discussion
The ability of TOF LSPR based sensor with different coating time to detect changes in the surrounding refractive index was studied. Surrounding refractive index was controlled through the use of ethylene glycol (EG) solutions in various concentrations. The relation between refractive index and EG solution’s concentration in ranges of 1.3324 to 1.4254 is shown in figure 3.

The sensor response towards different refractive index values starting from $n_1=1.3324$, $n_2=1.3523$, $n_3=1.3728$, $n_4=1.3931$, $n_5=1.4119$ to $n_6=1.4254$ were examined. Transmission spectra of all S1, S2 and S3 were recorded and shown in figure 4. The transmitted light intensity decreases with the increment of refractive index of surrounding medium. From the spectra, it is also observed that the locations of the dip were shifted to a higher wavelength. A linear regression method was employed to analyze the relationship between the transmission shifts with refractive index of surrounding as shown in figure 5. The best-fitting linear slope was determined and denoted as the sensitivity of the sensor, which reflect the overall performance of the developed sensor. Based on figure 5, S2 was reported with highest sensitivity of 18 nm/RIU compared to S1 and S3 with 9 nm/RIU and 2 nm/RIU respectively.
Figure 4. Transmission spectra of LSPR based TOF sensor for (a) S1; (b) S2 and (c) S3.

Figure 5. The linear slope for refractive index sensitivity based on sensor response of TOF.

Analysis shows that the highest sensitivity of TOF sensor was calculated at 18 nm/RIU for 48h deposition time. Shorter deposition time may result in poor SPR effect due to lower surface density of AuNPs. On the other hand, excessive amount of AuNPs deposited on the surface of tapered fiber will results in agglomeration of AuNPs. This phenomenon will subsequently decrease the sensitivity of the fiber, which is confirmed by the data plotted in figure 5. Based on the result, further characterization on the surface morphology, stability, reversibility and repeatability of S2 sensor were examined. Figure 6 shows the SEM image of (a) surface morphology of the coated fiber and (b) its cross-section which represent the coating thickness. Random distribution of immobilization AuNPs on TOF surface was obtained with 15 ± 2 nm of mean diameter which measured based on histogram analysis. The thickness of deposited Au layer was obtained with about ~53 nm.
The output responses of S2 were continuously monitored for 70 minutes to check the sensor stability in three different solutions selected at low, medium and high refractive indices levels. The controlled solutions were fixed at RI of 1.3728 (red), 1.3523 (blue) and 1.4254 (yellow). As illustrated in figure 7, the peak intensities of the sensor have shown to be constant over the test period with a little variation of ±0.017 a.u for the maximum peak-to-peak intensity value.

Repeatability of the sensor was examined by monitoring its output spectra for 3 repeated cycles with respect to surrounding’s RI ranges from 1.3324 to 1.4254 as shown in figure 8 (a). A good repeatability is shown based on the minimal variation in output intensity among the tested cycles. The largest deviation was observed when the sensor was immersed in solution with refractive index of n2=1.3523 with standard deviation of ±0.00594 a.u. Finally, reversibility test was carried out by recording the output transmission of the sensor with respect to both ascending and descending order of surrounding’s RI as shown in figure 8 (b). Result shows that the sensor repeated the same transmitted intensity as with little variation of ±0.008 a.u.
Figure 8. The analysis of (a) repeatability and (b) reversibility of LSPR based sensor for S2.

4. Conclusion
Gold nanoparticles (AuNPs) were successfully synthesized by using Turkevich’s method with average diameter of 15 nm, as evidenced particle size analyzer (PSA) analysis. The immobilization of AuNPs on the TOF surface was successfully carried out and verified by scanning electron microscopy (SEM) characterization. Based on the output spectra analysis, sensor S2 with 48h coating period showed the best sensitivity calculated at 18nm/RIU corresponding to 53 nm of coating thickness. Further analysis shows that the proposed sensor exhibits good stability, repeatability as well as reversibility with significantly low level of variation.

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