Coating configuration dependency of fiber optic silver coated U-bent plasmonic sensor surfaces in concentration sensing measurements*

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The sensitivity of the plasmonic sensor surfaces can be enhanced significantly if the sensor region is bent to U shape. Here, an experimental verification is done to evaluate the performance variations of the U-bent plasmonic silver coated sensor surfaces with different coating orientations inside the vacuum coating chamber. The performances of uncoated, partially coated as well as fully coated optical fiber U-bent surfaces are analyzed. The results show that the coating configuration of the U-bent surfaces can affect the sensing property. A fully silver coated sensor region outperforms the other two types of sensor surfaces with better sensing capability.
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Fiber optic sensing technology is finding applications in biomedical and chemical sensing requirements with potential sensing capabilities[1-6]. Numerous configurations and architectures of fiber based sensors have been discussed widely[7-11]. The U-bent shaped plasmonic fiber sensors are considered as the best option with the maximum sensing capability. Theoretical and experimental results are available for U-bent fiber optic sensing configurations and their better sensing properties[12-18]. In the available literature, the U-bent plasmonic sensor surfaces were mentioned to be fabricated as a point sensor with a plasmonic nano layer on the outer surface of the bent portion of the uncladded region. For silver coated fiber optic plasmonic sensors, the optimum plasmonic layer thickness for the best performance is ~ 50 nm[19]. The metallic sensing layer on the surface of the uncladded region of the fiber was deposited either by thermal evaporation method or by RF sputtering method under high vacuum[20,21].

While fabricating fiber optic plasmonic U-bent sensors by thermal evaporation method, the performance variations depending on the coating surface distribution and coating orientations are essential. These aspects are less discussed in the available literature. In the present studies, performance characterizations of the silver coated plasmonic U-bent sensors are carried out considering these aspects. Inside the vacuum coating unit, the uncladded U-bent structures can be placed at different locations with respect to the thermally evaporating material to get the plasmonic layer coating. In addition to this, plasmonic coating can be obtained on the entire uncladded portion of the U-bent fiber for a different coating orientation. An experimental verification is performed here to identify how the placement and orientations of the uncladded fiber U-bent structures inside the coating unit affect the performance of the developed sensor.

Fig.1 shows the simplified two-dimensional diagram of the electromagnetic wave propagating through an optical fiber with a plasmonic sensor surface.

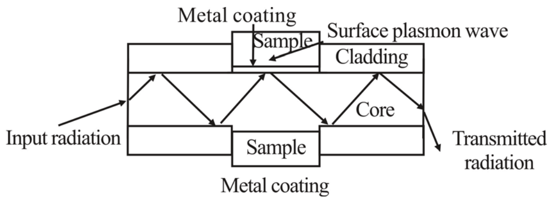

Fig.1 Schematic of radiation propagation in a straight SPR sensor probe

The uncladded portion of the fiber coated with a nano metal layer can act as the sensor surface, as the evanescent wave penetrating the uncladded portion of the fiber can excite the surface plasmon oscillations in the metallic layer. The surface plasmon resonance condition[22] is given by

$$\left(\frac{\omega}{c}\right)\sqrt{\varepsilon_{\rm p}}\sin\theta_{\rm res}=\frac{\omega}{c}\left(\frac{\varepsilon_{\rm m}\varepsilon_{\rm s}}{\varepsilon_{\rm m}+\varepsilon_{\rm s}}\right)^{1/2}, \quad (1)$$

where $\varepsilon_{\rm m}$ is the dielectric constant of the metal, $\varepsilon_{\rm s}$ is the

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dielectric constant of the sample and ε_p is the dielectric constant of the glass core of the fiber. ω and c are the frequency and velocity of the incident electromagnetic wave. θ_{res} is the angle of incidence for which the total internal reflection can take place at the core cladding interface. Hence by Eq.(1), resonance condition can be achieved either by changing the angle of incidence and keeping the frequency as a constant or by changing the frequency of input electromagnetic radiation at a constant angle of incidence.

Reported results reveal that bent plasmonic fiber surfaces are yielding better performance parameters[23]. The maximum sensitive fiber sensor surface is formed from the U-bent geometrical configuration. Theoretical treatments of U-bent plasmonic sensor surfaces are extensively done[24]. In these studies, optimum plasmonic coating thickness is assumed to be at the outer bent portion of the uncladded region and the propagation of the radiation is assumed to be in a plane inside the core of the fiber. In the present studies, U-bent structures are fabricated for plasmonic sensing, keeping variations when mounting these sensor surfaces inside the vacuum coating unit for silver layer coating. Characterization of these sensor surfaces is performed to see the performance changes of these sensor surfaces from the predicted optimum values.

For fabricating the plasmonic sensor surfaces, we selected plastic cladded silica (PCS) fiber with 400 μm core diameter. The sufficiently high light carrying capacity and the ease of removing the cladding make the multimode PCS fiber as a suitable sensor surface choice. The polymer coating on the fiber is removed using a sharp razor blade. For removing the cladding, the uncoated portion of the fiber is immersed in acetone and wiped carefully using a lint-free tissue repeatedly. The cladding can also be removed by burning torch method. For getting the U-bent fibers, the cladded portion of the fiber is subjected to a burning torch and bent gradually. The fiber cladding gets removed in this process and the core gets bent to U shape with the desired radius. The bent structures formed in this way are permanent. The U-bent fibers are now subjected to thermal evaporation coating.

For this purpose, in this experiment the fibers are first uncladded to 3 cm and then slowly bent to form a U shape with desired radius by exposing the uncladded portions to the blue flame of an alcohol burner. The sensor surface areas thus formed are subjected to thermal evaporation coating using a vacuum coating unit (Indian High Vacuum Pumps Model 12CU). In this work, seven U-bent structures are fabricated for the sensing studies with a bent radius of 1 cm. One of them is kept uncoated (U0). Five are mounted above the silver-carrying boat at slightly varying heights, 12—14.5 cm, from the evaporating material. In this mounting configuration, the fibers are getting coated with silver on the bent surface once (U1—U5). Hence the coating of the plasmonic layer on these surfaces can be considered to be partial.

The sixth U-bent structure (U6) is coated in a different way. In this case, both the bent surfaces of the fiber are coated with silver by performing the coating twice. Sensing studies are carried out on these seven types of U-bent structures and the variations in performance are recorded. The schematic of the fiber orientations inside the vacuum coating chamber is shown in Fig.2. The photographs of the different sensor surfaces fabricated are given in Fig.3.

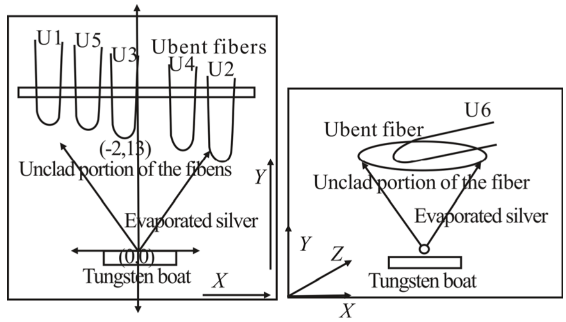

Fig.2 Uncladded U-bent orientations inside the vacuum coating unit

The 925 silver sample bought from authorized selling agency is used as the coating material. A small piece of silver placed in a tungsten boat inside the vacuum chamber is heated to evaporate the silver. The vacuum unit is operated and a high vacuum at the order of 2.5×10^{-5} mbar is achieved. After reaching this vacuum, the coating is done by increasing the heater current gradually. The current is increased up to 75—80 A at which the temperature of the silver carrying Tungsten boat reaches ~ 2 000°C and the pressure in the vacuum unit starts to increase because of the presence of the evaporated silver molecules. The uncladded fiber surfaces exposed to the evaporated silver slowly form a layer of silver as the cladding. The coating is done for a time duration till the thickness monitor shows the required film thickness of ~50 nm. After reaching the required silver layer thickness, the current is decreased to stop the thermal evaporation of the silver. The system is then allowed to cool down. In the case of the U6, the U-bent portion of the fiber is again placed with its uncoated portion facing down and once again the silver coating is done so that a fully coated U-bent structure is obtained. The photograph of the fabricated U-bent structure is shown in Fig.3.

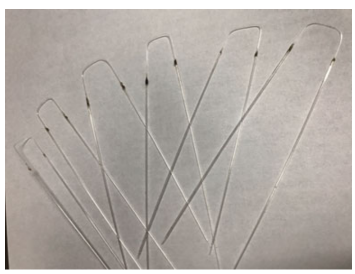

Fig.3 Photograph of U-bent fibers

The experimental setup used for the plasmonic sensing studies is given in Fig.4. Care is taken to keep the experimental setup undisturbed throughout the measurements and freshly prepared fibers are taken for the measurements. The processed core of the fiber is placed in contact with the sample solution. The input end of the fiber is fed to a white light source (Yogokawa-AQ4305) emitting in the range of 400—1 800 nm. The output of the fiber is connected to Ocean Optics (HR 4000) spectrometer interfaced with a PC.

The processed fiber is allowed to interact with the sample solution that is kept in a glass cell with dimensions of 12.0 cm×4.0 cm×4.0 cm. The absorption spectrum due to the interaction of the sensor surface with the sample solution is recorded.

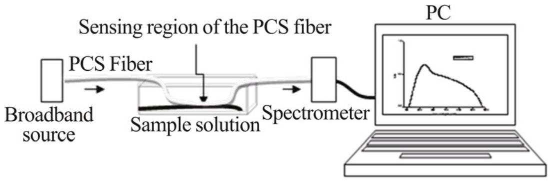

Fig.4 The experimental setup

Potassium permanganate ($KMnO_4$) is taken as the sample and the sample solution with different concentrations is prepared for sensing measurements. The absorption spectrum of the $KMnO_4$ solution in distilled water is taken using a spectrophotometer (Jasco 570V) and is given in Fig.5. Further, molar solution of the sample solution is prepared in distilled water and used as the stock solution. The stock solution is diluted to get different orders of concentrations (with molarity ranging from 10^{-2} to 10^{-8}). The response of the sensor to each sample concentration is monitored and recorded. The uncoated U-bent, partially coated U-bent and fully coated U-bent structures are subjected to concentration sensing studies. The experimental observations are plotted in Fig.6.

The results give an inference regarding the method of plasmonic coating preferred for U-bent structures. First of all, for all the types of plasmonic U-bent structures fabricated, blue shift with respect to the peak absorption wavelength (526 nm) of the sample solution is observed. This indicates the presence of a continuous silver plasmonic layer in each of the coated sensor surfaces fabricated.

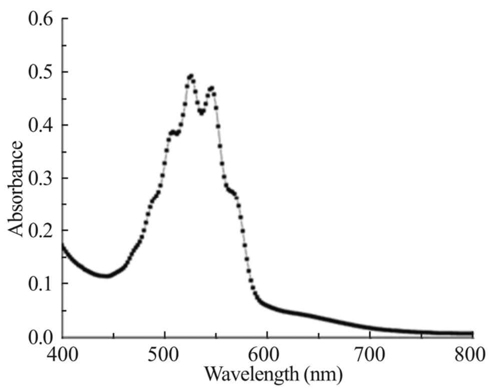

Fig.5 $KMnO_4$ absorption spectrum

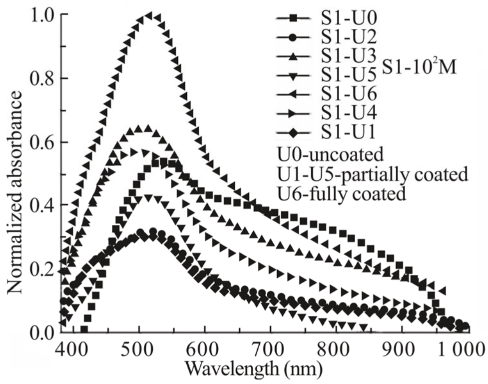

Fig.6 Absorbance curves of different U-bent structures

We can see from Fig.6 that it is the fully coated U-bent structure that possesses enhanced absorption compared to partially coated one as well as uncoated U-bent structures. The results for the partially coated U-bent structures show that the placement position of the U-bent structure above the coating material affects the sensing property. In Fig.2, it is shown that the bent surface exposed for coating is placed at different heights from the sample material for coating. The U-bent structures are placed above the sample material with orientation in the reference *XY* plane as U1 (−5, 14.5), U2 (10, 12), U3 (−2, 13), U4 (6, 13.5), U5 (−4, 14) (Refer to Fig.2). In addition to height wise variation, the transverse location of the U-bent structures also needs to be noted. This means that U5, U2 and U1 are farther away from the coating material in the *X* and *Y* directions. Hence as seen in Fig.6, the maximum enhancement in the signal is observed for the U3 kept just above the evaporating material with optimum distance of separation. Here, the thickness monitor's crystal surface is placed at a height equal to that of the exposed surface of U3. Hence the measured optimum thickness of silver plasmonic layer coating of 50 nm is obtained for U3. The response of U1 and U2 shows that the absorption pattern is similar to that of the uncoated U-bent fiber. From Fig.2, it is clear that these fibers are kept transversely and longitudinally away from the silver evaporation point. These observations imply that with respect to the placement of the fibers above the coating material, a gradation in coating layer thickness is possible, which in turn affects the sensing capability of the sensor. The bent portion exposed to the evaporating silver away from the optimum distance, which is monitored by the digital thickness monitor, is getting a non-optimum plasmonic coating in the uncladded portion, which results in decreased plasmonic absorption. The inference obtained from these measurements is that if partially coated U-bent sensors are developed for point sensing applications, the better option is to keep the bent portion exposed to the silver vapor at a height of (13±0.5) cm and also to keep a transverse orientation defined by a circle with radius of (2±0.5) cm from the coating sample location, along with a digital thickness monitoring setup that measures an optimum silver layer thickness of ~50 nm.

Further, for partially coated U-bent structures, only the lower bent portion is getting the coating material exposure, while the inner surface of the U-bent structures is without any plasmonic layer. Hence the results obtained also indicate that plasmonic enhancement in absorption for partially coated U-bent structures is less compared to a fully coated U-bent sensor surface (U6). Theoretical treatments on bent plasmonic fiber structures consider the confinement of electric vectors of the guided rays in the plane of bending[25]. But in practical cases, occurrence of electric vectors deviating from the plane of bending is possible, which can induce plasmonic propagation of the fields, if a metallic plasmonic layer is available around the entire core surface of the bent portion. This explains the highest enhanced absorption by the U6.

Based on the above studies, the enhanced absorption for the U-bent plasmonic sensor surface structure and its safer geometrical structure for the integration in sensor devices make the U6 a preferred choice for concentration sensing applications compared to the other structures explained. Hence, detailed results of $KMnO_4$ concentration sensing using the U6 are given in Fig.7.

Here, as shown in Fig.8 for the concentration changes from 10^{-6} mol/L to 10^{-2} mol/L, the wavelength shift observed is from 487 nm to 522 nm. The observed red shift with respect to concentration confirms the reported results[21,26]. The absorbance also increases gradually in this range. As the concentration of the sample reaches 0.01 mol/L, the peak absorbing wavelength shifts to 522 nm and the absorbance also increases to the highest value as shown in Fig.9. Hence for safer integration and predictable sensor behavior, a U6 structure can be adopted compared to other sensor surface structures.

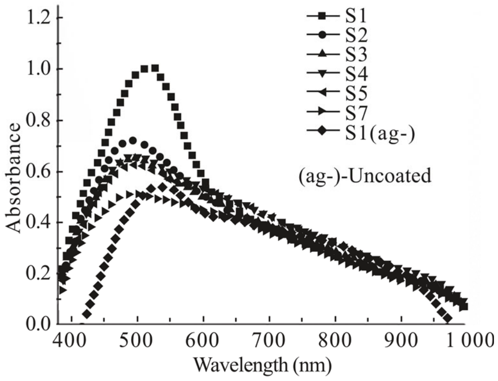

Fig.7 Concentration sensing measurement results using fully silver coated U-bent plasmonic sensor surface (U6)

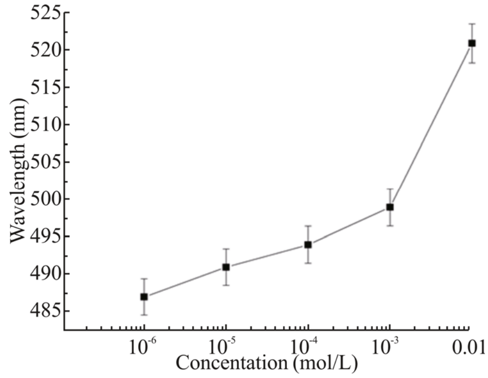

Fig.8 Wavelength dependence on concentration in the case of a fully silver coated plasmonic U-bent sensor (U6)

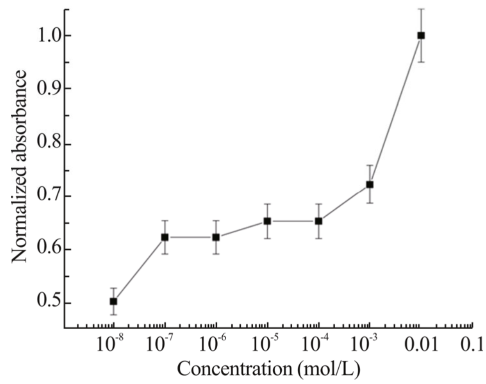

Fig.9 Absorbance dependence on concentration in the case of a fully silver coated plasmonic U-bent sensor (U6)

The studies presented here reveal the sensitivity dependence of the plasmonic surfaces on coating orientations inside the vacuum coating chamber. The point sensor method of the U-bent coating provides a partial nano layer plasmonic surface beneath the U-bent portion. If the U-bent surface can be fully covered with a metallic nano layer, enhancement in absorption can be observed compared to the partially coated fibers. The theoretical explanations of these observations can be further explored.

References

[1] Zhang H and Kulkarni A, J Nanosci. Nanotechnol. **11**, 5939 (2011).

[2] Adolfo J. Rodríguez and Carlos R. Zamarreño, Sensors (Basel) **14**, 4060 (2014).

[3] Jiri Hromadka and Begum Tokay, Sensors and Actuators B: Chemical **221**, 891 (2015).

[4] Aitor Urrutia and Kartheka Bojan, Journal of Sensors **2016**, 8129387 (2016).

[5] Jesús M.Corres and AnaiSanzFrancisco, Sensors and Actuators B: Chemical **131**, 633 (2008).

Chemical **272**, 160 (2018).

[7] Longjiang Zhao and Lan Jiang, Sensors **11**, 54 (2011).

[8] Violeta A. Márquez-Cruz and Juan A. Hernández-Cordero, Optics Express **22**, 3028 (2014).

[9] Shun Wang and Shuhui Liu, Optics Express **25**, 14389 (2017).

[10] Bart Peeters, AIP Conference Proceedings **1600**, 39 (2014).

[11] Marie Pospíšilová and Gabriela Kuncová, Sensors **15**, 25208 (2015).

[12] Gowri A. and V. V. R. Sai, Optical Sensors **10231**, 1023113 (2017).

[13] Mohd Anwar Sawawi, Elfed Lewis and Sinead O' Keeffe, IEEE Sensors, 11592 (2015).

[14] Yuqiang Fan, George Wu and Wanting Wei, Optics Express **20**, 26127 (2012).

[15] Jianrong Zhang, Hairong Liu and Xinkun Wu, Optoelectron. China **2**, 2009.

[16] Huixin Zhang, Lishuang Feng and Yulong Hou, Optical Fiber Technology **24**, 135 (2015).

[17] B. D. Gupta and R. K. Verma, Journal of Sensors **2009**, 979761 (2009).

[18] Elizaveta Klantsataya and Peipei Jia, Sensors (Basel) **17**, 12 (2017).

[19] Tuan Guo, Journal of Lightwave Technology **35**, 16 (2017).

[20] Christopher C and Subrahmanyam A, Plasmonics **13**, 493 (2018).

[21] Sruthy P Usha and Satendra K, Materials **8**, 2204 (2015).

[22] Anuj K Sharma, Rajan Jha and B. D. Gupta, IEEE Sensors Journal **7**, 1118 (2007).

[23] R.K. Verma and B.D. Gupta, Surface Plasmon Resonance Based U-shaped Fiber Optic Sensor with Enhanced Sensitivity, ICOP 2009 International Conference on Optics and Photonics, 2009.

[24] R.K. Verma and B.D. Gupta, J. Phys. D: Appl. Phys. **41**, 095106 (2008).

[25] B.D. Gupta, Fiber Optic Sensors Based on Plasmonics, 1st ed., Singapore, World Scientific, 2015.

[26] T Kundu and V V R Sai, PRAMANA-Journal of Physics **75**, 1099 (2010).