Received 9 July 2020; revised 26 August 2020; accepted 27 August 2020. Date of publication 7 September 2020;
date of current version 4 November 2020. The review of this article was arranged by Editor A. Nathan.

Digital Object Identifier 10.1109/JEDS.2020.3022036

Multilayer CVD-Graphene and MoS_2 Ethanol Sensing and Characterization Using Kretschmann-Based SPR

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This work was supported in part by the Malaysian Ministry of Education and Universiti Kebangsaan Malaysia under Grant FRGS/1/2019/STG02/UKM/02/8 and Grant DIP-2016-022, and in part by the Engineering and Physical Sciences Research Council, U.K., through the Future Photonics Manufacturing Hub, University of Southampton under Grant EPSRC EP/N00762X/1, for graphene 2D materials.

ABSTRACT The Kretschmann-based surface plasmon resonance (K-SPR) sensor was developed using multilayer graphene and molybdenum disulphide (MoS_2) structures on a plasmonic gold (Au) layer for ethanol detection. In this configuration, the SPR spectra of minimum reflectance versus SPR angle was used to determine the sensitivity, detection accuracy and quality factor as the main figure of merit (FOM). Both graphene and MoS_2 were used as hybrid detection layers to enhance the ethanol sensing performance using Finite Difference Time Domain (FDTD). The multilayer graphene/Au and MoS_2/Au sensors gave a maximum sensitivity of 192.03°/RIU and 153.25°/RIU respectively at 785 nm optical wavelength. In terms of material characterization using the K-SPR technique, chemical vapor deposition (CVD)-grown graphene on Au, had a thickness of 1.17 nm with real and imaginary refractive indices of 2.85, 0.74, and 3.1, 1.19, respectively, at optical wavelengths of 670 nm and 785 nm.

INDEX TERMS Ethanol sensor, FDTD, graphene, Kretschmann configuration, MoS_2, refractive index, surface plasmon resonance.

I. INTRODUCTION

Two-dimensional (2D) materials, including graphene and transition metal dichalcogenides (TMDs), have recently gained profound interest due to their captivating electrical, optical and thermal properties. Graphene is a thin 2D sheet of carbon atoms which are arranged in honeycomb form. Graphene has several exclusive features such as atomic thickness, high strength, transparency, high mobility of electrons, tightness, and high quantum efficiency. Besides, graphene plays an essential role as an appropriate candidate as the top dielectric layer in Kretschmann-based surface plasmon resonance (K-SPR) sensors, due to its outstanding array and superior surface area. In addition to graphene, greater attention was given to increasing the efficiency of K-SPR sensors using 2D transition metal dichalcogenides (TMDC) such as molybdenum disulphide (MoS_2). MoS_2 is a suitable biosensing material candidate due to its high optical absorption efficiency (∼5 %), which further reduces the SPR resonance curve's dip [1].

The characterization of graphene and MoS_2 material characteristics such as thickness and refractive index is critical for both laboratory and mass production. Some common methods for measuring graphene thickness are such as Raman spectroscopy, optical contrast analysis, and atomic force microscopy (AFM). The normal reported literature value for single-layer graphene thickness is 0.335 nm. Else than

sensing, the K-SPR, an optical surface-sensitive analysis technique using a full angular spectral range, can also be used to measure ultra-thin film properties. Although the K-SPR technique is typically correlated with the analysis of bio-chemical interactions, it also shows outstanding performance in characterizing thin nanoscale layers as well [2]. The SPR method is an optical technique, using noble metal layers such as gold (Au) on quartz-based sensors to generate a sensitive propagating plasmonic wave region on metal surfaces. Small changes adjacent to an area of about 300 nm are susceptible to sensing. The Au-coated SPR with graphene and MoS_2 coating sensor also offer excellent absorption of biomolecules, increasing the sensing performance of biochemical systems using various surface analyses. Our previous work on K-SPR involves the bio-sensing of various analytes [3]–[8]. A graphene-coated SPR sensor has more sensitivity than a conventional K-SPR biosensor and the K-SPR technique can be used to characterize graphene film properties to obtain parameters such as thickness and refractive index.

In this study, a numerical setup of the Kretschmann-based SPR using plasmonic gold (Au) with multilayer graphene and MoS_2 layers was examined to explore a new window for ethanol detection. Hybrid Au-graphene and Au-MoS_2 layers were used for ethanol sensing by observing the change in SPR angle, minimum reflectance and SPR spectral width of reflectivity. Next, the effect of adding graphene and MoS_2 was also analyzed. Also, the usage of K-SPR technique for the characterization of graphene layers grown by chemical vapor deposition (CVD) is also disclosed where the film thickness as well as real and imaginary refractive index values were obtained. The sensitivity variation study of the proposed sensor is evaluated in line with the increase in the refractive index. The simulation data from numerical modeling was compared with the results achieved at optical wavelengths of 670 nm and 785 nm. The method obtained can be used for the physical characterization of graphene and MoS_2 for improvement in bio-sensing applications. This work is an extension of our recently published work [7].

II. METHOD AND PROCEDURES

A. FDTD SIMULATION

Initially, the K-SPR sensor was numerically simulated with Lumerical's Finite Difference Time Domain (FDTD), as shown in Fig. 1. The setup is composed of five components: BK7 glass, 1.5 nm-thick chromium (Cr), 50 nm-thick gold (Au) layer, and graphene (Gr) with three, five and seven layers. From our previous work, 50-nm thick Au layer with Cr as an adhesion layer was proven to have the best sensitivity of 80.56°/RIU at 670 nm with minimum reflectivity (R_{min}) of 0.15 and resonance angle of 70.2°; hence it is used in this work as well [9]. The simulated refractive index of water and the sensing medium is 1.3309 and 1.3284 respectively (before adsorption) as well as 1.3405 and 1.34 respectively (with 10% ethanol absorption) at optical wavelengths of 670 nm and 785nm, obtained from Kameoka

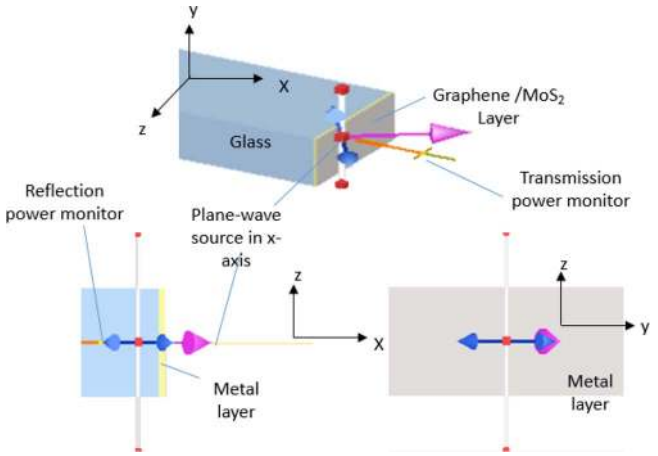

FIGURE 1. The simulated graphene/MoS_2-coated Au-K-SPR sensor using Lumerical's FDTD.

and Craighead [10]. All simulation results were verified with 100% analytical and numerical results. Graphene thickness is given as L = 0.34 nm, where L is the thickness of one graphene layer [11]. The sweeping parameter of the source angle of the incident light was from 36° to 80° and set as a plane wave source as Bloch or regular form in the preferred wavelengths. The sensing medium is a mixture of Millipore water with ethanol concentrations of 1%, 2%, 5% and 10%. In the subsequent experimental work using Bionavis SPR Navi-200L, the ethanol samples are loaded into a syringe and immediately injected into the flow cell to avoid the evaporation of ethanol during the detection.

Sensitivity, detection accuracy and quality factor are the main performance parameters of the K-SPR sensor [9]. The sensitivity (S) is defined as the ratio of incident angle shift of the SPR ($\Delta\theta_{res}$) to the change in the refractive index of the sensing medium (Δn_c). In this paper, Δn_c is assumed to be 0.0096 and 0.0116. The sensitivity can be given by:

$$S = \Delta\theta_{res}/\Delta n_c \tag{1}$$

with units of deg/RIU. The detection accuracy (D.A.) is also referred to as the signal-to-noise ratio (SNR). The SNR can be calculated and given by:

$$D.A = \Delta\theta_{res}/\Delta\theta_{0.5} \tag{2}$$

where $\Delta\theta_{0.5}$ is the spectral width at 50% reflectivity of the SPR curve. The quality factor (Q) with units of RIU^{-1} is governed by the sensitivity and the spectral width of the SPR curve at 50% reflectivity, which is given by:

$$Q = S/\Delta\theta_{0.5} \tag{3}$$

Next, the schematic of the K-SPR detection method with the presence of chromium (Cr), gold (Au) and graphene/MoS_2 layers for the detection of ethanol is shown in Fig. 2. The shift in the resonance angle value, $\Delta\theta_{res}$ is directly correlated to the presence of ethanol in the sample. Another SPR parameter is $\Delta\theta_{0.5}$, which refers to the difference between the minimum resonance angle and

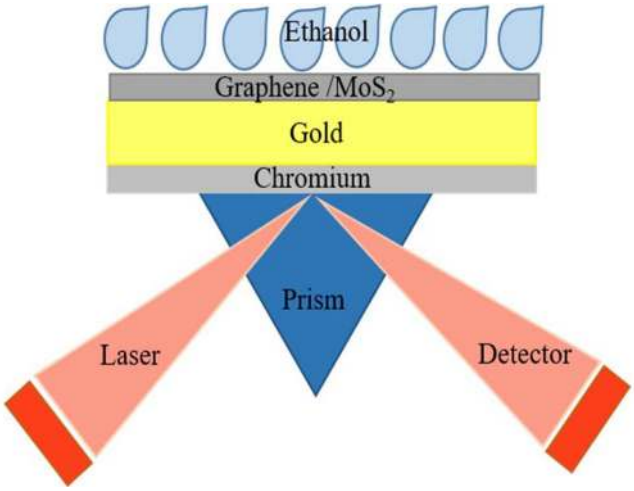

FIGURE 2. Schematic representation of the graphene/MoS_2 surface plasmon resonance (SPR) ethanol sensing technique based on the Kretschmann configuration.

mid-reflection angle in the SPR curve. A broad spectrum of reflection that becomes narrower with a deeper resonance peak can effectively detect resonance shifts due to the presence of analytes [12].

B. MATERIAL

The multilayer CVD graphene grown on Cu foils and MoS_2 grown on quartz glass samples were obtained from our collaborators at University of Southampton and Indian Institute of Sciences, Bangalore (IISc). Graphene and MoS_2 were transferred to the sensor slides comprising of BK7 glass, Cr adhesion layer and Au coating layer (obtained directly from BioNavis Ltd.) with a polymer assistance, using polystyrene (PS), wet transfer procedure [13], [14].

C. CHARACTERIZATION

Raman measurement was performed with a 532 nm frequency confocal Raman microscope (DXR2xi, Thermo Scientific) to assess the quality of the transferred CVD-graphene films. The spectra was generated at ambient temperature using a 100× microscope lens in backscattering geometry. The power of the excitation was 7mW. In non-contact mode, the NX10 Park System was used to make AFM measurements approximating the height of the transferred CVD-graphene layers. The optimum scan region of the device was 10 μm × 10 μm. The BioNavis-SPR Navi200-L equipment with Kretschmann configuration was used to perform the SPR measurements with optical wavelengths of 670 nm and 785 nm at 24°C room temperature. The measuring beam point is about 0.5 mm in diameter and the data is summed.

III. RESULTS AND DISCUSSION

A. NUMERICAL ANALYSIS

Fig. 3 compares the K-SPR spectra obtained experimentally and with FDTD simulation using a 50 nm-thick Au-coated

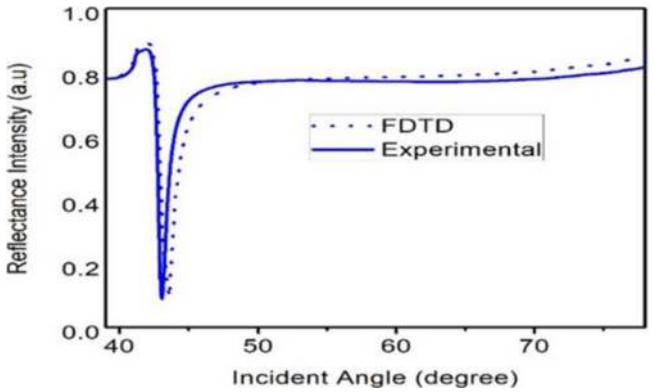

FIGURE 3. Comparison of the K-SPR curve obtained via FDTD simulation (dotted curve) and experimentally (solid curve) using Bionavis SPR Navi-200 measured at 670 nm optical wavelength with less than 5% difference in reflectance intensity and incident angle.

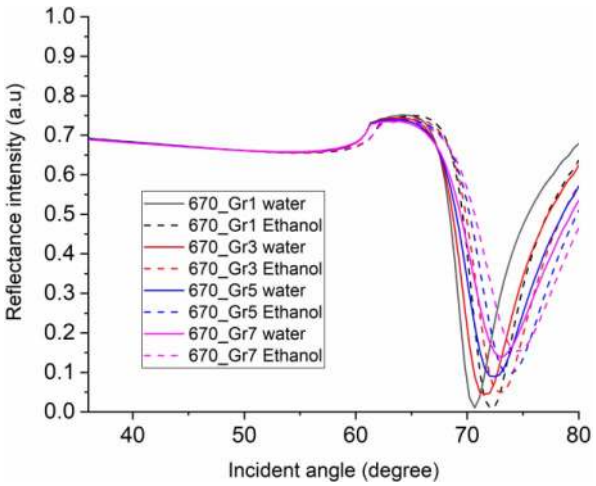

FIGURE 4. Comparison of K-SPR curves for water versus 10% ethanol detection for multilayer graphene/Au SPR sensor at 670 nm optical wavelength.

with graphene on BK7 glass at optical wavelength of 670 nm. It shows good agreement with only less than 5% error in the reflectance intensity and incident angle. Since the FDTD method solves Maxwell's equations with no approximations, the main source of error could be due to numerical error caused by discretization of space and time. This can be rectified using a very fine mesh albeit at the expense of increased memory requirements and simulation time. Low frequency drift and other forms of noise could also contribute to this error.

In this work, the difference in the SPR curve for different sensing layers comprising of multiple layers of graphene at 670 nm and 785 nm is plotted in Fig. 4 and Fig. 5, respectively. The addition of 10% ethanol concentration red-shifts the SPR spectra as compared to sensing with water only. The 785 nm reflectance SPR curves are noticeably narrower than the 670 nm. The longer wavelength is capable of generating lower resonance peaks, and the resonance angle shift is smaller [15].

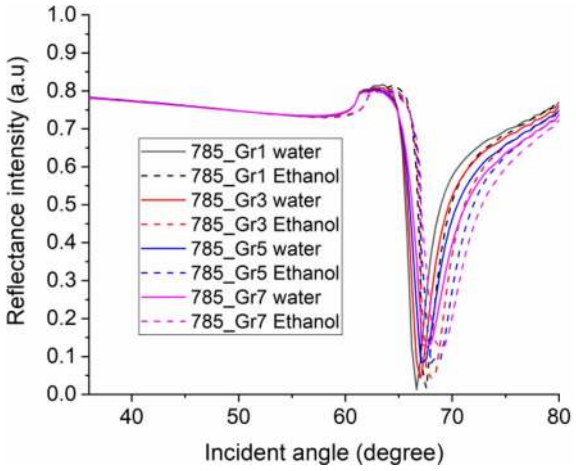

FIGURE 5. Comparison of K-SPR curves for water versus 10% ethanol detection for multilayer graphene/Au SPR sensor at 785 nm optical wavelength.

TABLE 1. Minimum reflectance, R_{min} and resonance angle shift at 50%, $\Delta\theta_{0.5}$ for multilayer graphene/Au-based K-SPR sensor at 670 and 785 nm wavelength.

Number of graphene layers	670 nm		785 nm	
	R_{min} for water	$\Delta\Theta_{0.5}$ for water	R_{min} for 10% ethanol	$\Delta\Theta_{0.5}$ for 10% ethanol
1	0.0128	2.44528	0.0103	4.89056
3	0.0502	3.084815	0.0473	6.357729
5	0.0924	3.624031	0.0894	7.574099
7	0.1251	4.363885	0.1393	8.313953

Table 1 is produced by taking the resonance angle ($\Delta\theta_{res}$) that conform to the change in the refractive index of sensing layers, with R_{min} and spectral width of the K-SPR curve values in water and ethanol. A lower value of the minimum reflectance (R_{min}) with reduced spectral width $\Delta\theta_{0.5}$ exhibits higher efficiency of the biosensor. For structures with different number of graphene layers, one can see that the full range of reflections of the $\Delta\theta_{0.5}$ becomes narrower, more profound, and more effective in detecting resonance shifts at 785 nm compared to 670 nm. These $\Delta\theta_{0.5}$ values are calculated as low and high, with the lower and upper levels marked as low and high respectively. $\Delta\theta_{0.5}$ shows smaller values at 785 nm than 670 nm at a range of 2.4452 to 4.8905.

Moreover, R_{min} increases after the graphene layer was added due to the quality factor, and a significant factor enhances the detection accuracy of the proposed K-SPR sensor in Eq. (3) and (4). One layer of graphene shows the lowest R_{min} value of 0.0103 and 0.0128 at 670 nm and 785 nm, respectively [16].

Sensitivity for different wavelengths is summarized in Table 2. The $\Delta\theta_{res}$ value in the sensor layer ranges from 1.3284 to 1.3309 RIU and 1.34 – 1.3405 RIU for both wavelengths and is calculated based on Eq. (1), (2) and (3),

TABLE 2. Sensitivity (S), detection accuracy (D.A.) and quality factor (Q.F.) for multilayer graphene/Au-based SPR K-sensor at 670 and 785 nm wavelength for 10% ethanol detection.

Number of graphene layers	670 nm			785 nm		
	S	D.A	Q.F. (RIU)	S	D.A	Q.F. (RIU)
1	133.09	0.26	27.214	123.45	0.40	50.487
3	150.46	0.22	23.667	150.88	0.39	48.913
5	159.14	0.2	21.011	164.60	0.37	45.42
7	185.18	0.21	22.274	192.03	0.35	44.005

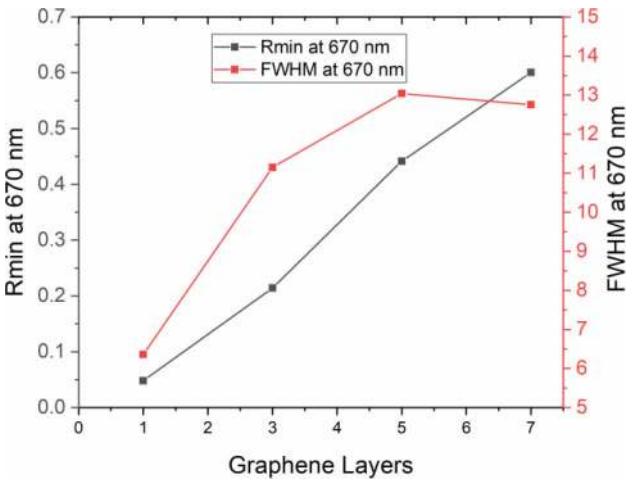

FIGURE 6. Minimum reflectivity (R_{min}) and Sensitivity (S) of the multilayer Graphene/Au-based K-SPR sensor versus increasing number of graphene layers for the detection of 10% ethanol at optical wavelength of 670 nm.

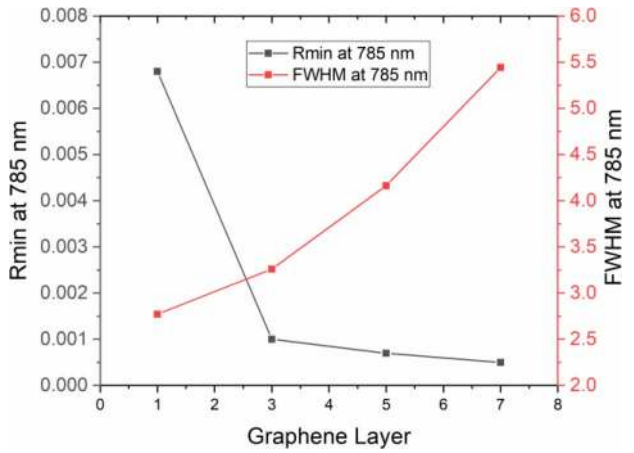

FIGURE 7. Minimum reflectivity (R_{min}) and Sensitivity (S) of the multilayer Graphene/Au-based K-SPR sensor versus increasing number of graphene layers for the detection of 10% ethanol at optical wavelength of 785 nm.

respectively as well as portrayed in Fig. 6 and Fig. 7. The increased K-SPR angle results in the sensitivity of the sensor being increased, as the sensitivity is relative to the number of graphene layers. It is observed that the sensitivity increases

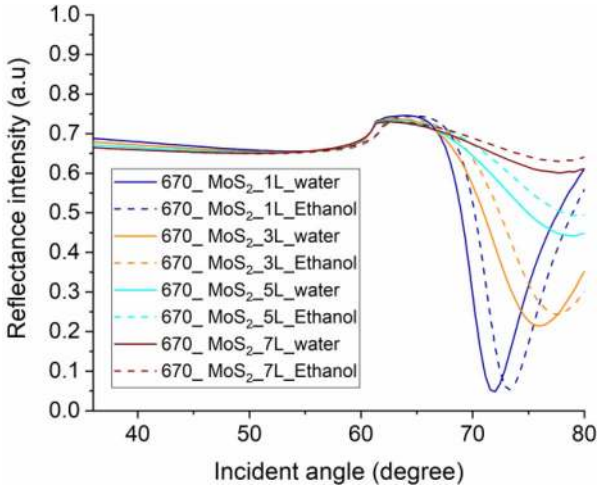

FIGURE 8. Comparison of K-SPR curves for water versus 10% ethanol detection for Au/multilayer MoS_2-based SPR sensor at 670 nm optical wavelength.

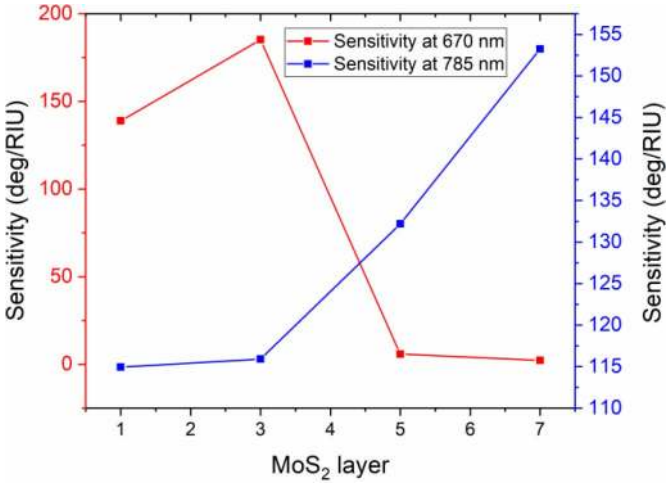

FIGURE 10. Sensitivities of MoS_2/Au-based K-SPR ethanol sensor versus number of MoS_2 layers at 670 and 785 nm optical wavelength for 10% ethanol concentration.

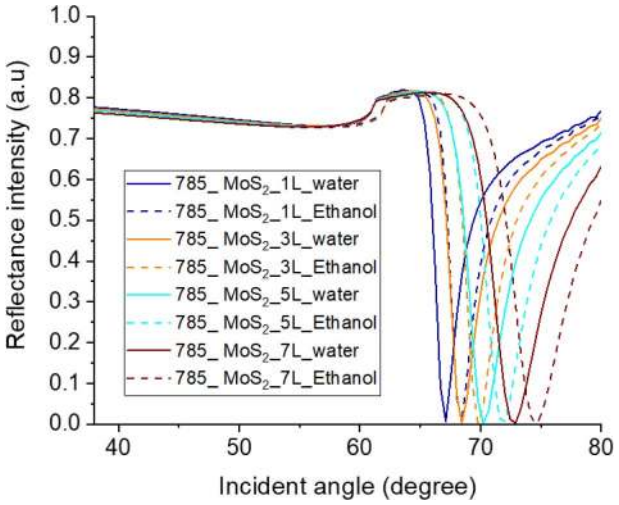

FIGURE 9. Comparison of SPR curves for water versus 10% ethanol detection for multilayer MoS_2/Au-based K-SPR sensor at 785 nm optical wavelength.

after multi layers of graphene are added with values ranging from 133.09∘/RIU to 185.18∘/RIU and 123.45∘/RIU to 192.03∘/RIU for 670 nm and 785 nm respectively. However, the detection accuracy and quality factor remain unchanged owing to constructive interference from various layers due to radiation regions where thin flakes are much smaller than the light wavelength [17].

Further simulation is performed to analyze SPR curves for ethanol concentrations of Au-coated MoS_2 structures as shown in Fig. 8 and Fig. 9. Each thickness of MoS_2 layer is considered as 0.65 nm (M × 0.65 where M is the number of MoS_2 layers). It is clearly observed that the changing refractive index of the sensing medium increases the resonance angle. This is because of the larger bandgap of MoS_2, higher optical absorption and its more significant working function (5.1eV) compared to graphene [18]. The values of R_{min} and $\Delta\theta_{0.5}$ have increased with the addition of MoS_2 coating on the Au metal layer. It is observed from the simulation results, that there is a substantial alteration in the angular dip and of the resonance curves if more MoS_2 layers are added. It is claimed that the main cause of the shifting of the SPR curve is because of the absorptive plasmon damping related to the MoS_2 layer. As the spectral width of the K-SPR curve rises, the analytes become more challenging to be sensed and the performance properties such as detection accuracy and the quality of the SPR sensor degrade. In order to achieve a maximum sensing performance, the optimal number of MoS_2 layers must therefore be carefully designated. As seen in Fig. 8, the sensitivity increased and subsequently decreased after 3 layers of MoS_2 at 670 nm.

It is shown in Fig. 9 that the resonance curve shifts from 68.44° to 74.67° for ethanol sensing using a single MoS_2 layer till 7 layers of MoS_2, corresponding to a substantial angular shift of 6.22°. It offers an effective ethanol sensing of a broader range. The maximum sensitivity at 670 nm optical wavelength is traced along the primary vertical axis, whereas the sensitivity at 785 nm is plotted in the secondary vertical axis as shown in Fig. 10. Therefore, wavelength 785 nm is considered for optimized performance for MoS_2-coated gold sensor with maximum sensitivity of 153.25°/RIU.

Further analysis was performed to analyze the SPR reflectance curves by considering Au/7 layers of graphene and Au/3 layers of MoS_2 at 670 nm, which is shown in Fig. 11. Different ethanol concentrations (1%, 2%, 5% and 10%) can be prepared in the Millipore water. It is shown that the angular sensitivity increases linearly with ethanol concentration for graphene ($R^2 = 0.99879$) and MoS_2 ($R^2 = 0.91741$). This is probably due to the significant absorbance by the graphene and MoS_2 layer of different analytes near to the analyte interface.

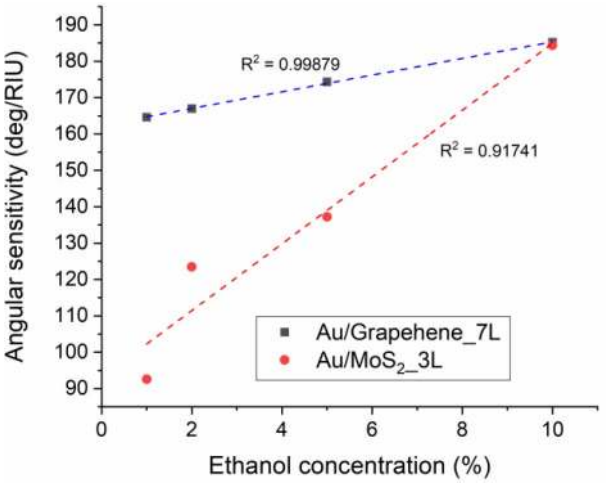

FIGURE 11. Variation of angular sensitivity of Au/7 layers of graphene and Au/3 layers of MoS_2K-SPR sensor with different ethanol concentrations in water at optical wavelength of 670 nm.

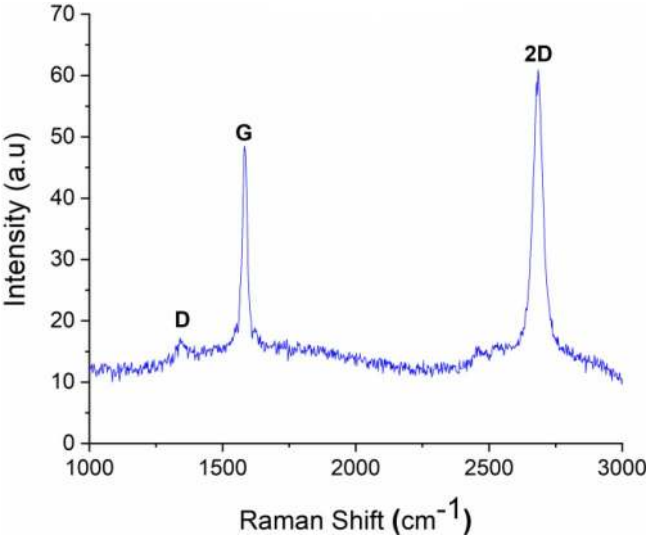

FIGURE 12. Raman spectra of the bilayer graphene on the Au-coated SPR sensor.

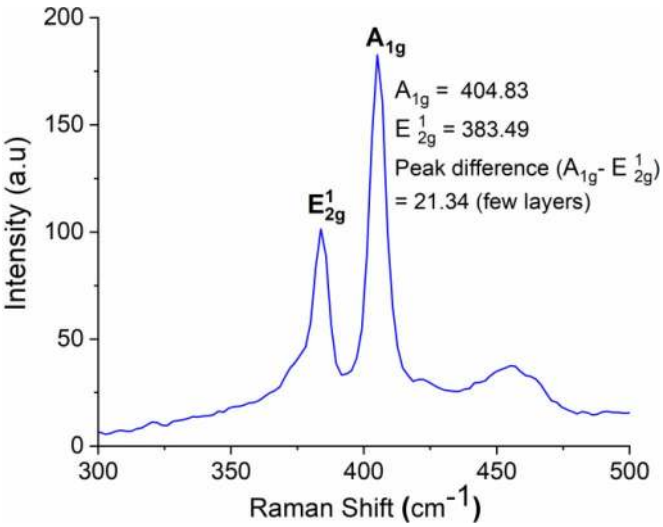

FIGURE 13. Raman spectra of the bilayer MoS_2 on the Au-coated SPR sensor.

B. THICKNESS AND REFRACTIVE INDEX

Raman spectroscopy was performed to detect graphene's presence and to validate the efficiency of the transmission mode. The Raman spectrum from the CVD-synthesized graphene layer comprises three signature bands: D, G, and 2D. Heteroatoms, vacancies, or other defects in the first-order dispersion activate the D band. G and 2D groups are associated with the input excitation of the sp2 carbon atoms in the monolayer graphene and the stacking arrangement along the graphene c axis. Thus, the graphene thickness ratio of the 2D band to the G band can be accurately estimated from the I_{2D} / I_G as follows: I_{2D} / $I_G = 2$ for the monolayer, I_{2D} / $I_G = 1$ for the bilayer, and I_{2D} / $I_G < 1$ for the multilayer structure [16].

Raman peaks at a wavenumber of approximately 1340/cm, 1585/cm, and 2684/cm can be traced to the graphene peaks D, G, and 2D, thus demonstrating the deposition of graphene to the SPR sensor. The graphene intensity ratio is 1.69, indicating the presence of bilayer graphene as shown in Fig. 12.

Further again, Fig. 13 displays the spectra of the Raman scattering between 300 and 500 cm^{-1} for the deposited MoS_2 layer on Au-coated SPR sensor. In general, this occurs depending on the preparation method of the MoS_2 flakes. The mechanically exfoliated MoS_2 flakes, for instance, have a maximum difference of 18 cm^{-1}. The difference in CVD-grown flakes can, on the other hand, have higher yields, such as 22 cm^{-1} for monolayer flakes [19].

MoS_2 has two distinctive Raman summits corresponding to the Mo and S atoms in-plane vibration (E1 2 g) at the 383.49 cm^{-1} and the S out-of-plane vibration (A1 g) at the 404.83 cm^{-1} level, which is used to indicate the number of layers by a change in the difference between these two peaks. The MoS_2 intensity ratio is 21.34, indicating bilayer of MoS_2.

Next, the measurement of AFM in Fig. 14 shows that the graphene is about 3 nm thick. This approximate is significantly larger than the reported graphene thickness value from the literature, probably caused by some surface polymer residues. This common issue often occurs when measuring graphene thickness using AFM. After all, AFM is not an ideal method for characterizing graphene layer height and thus requires a different approach such as K-SPR measurement. Therefore, the SPR BioNavis Navi-200L was used instead to obtain the thickness and optical coefficients of the transferred graphene layers.

Fig. 15 and Fig. 16 display the K-SPR curve of a bare gold sensor and an Au-graphene sensor assessed at 670 nm and 785 nm. The red SPR curve indicates the SPR excitement of the Au and the Au-graphene and the resonance peak is at 43.54° and 42.75° at 670 nm and 785 nm, respectively. Meanwhile, the resonance peak in black-colored line

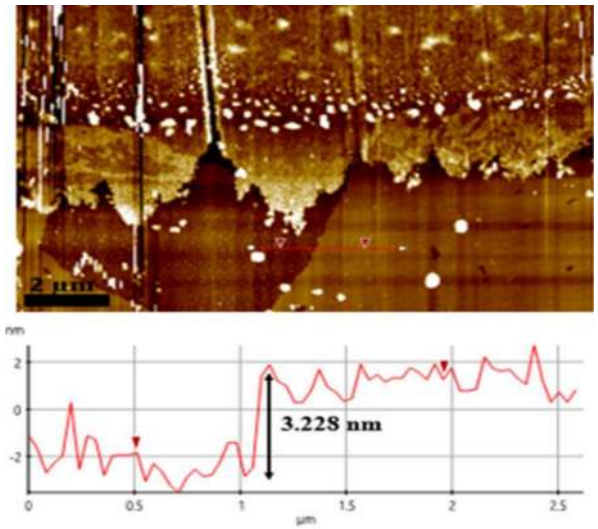

FIGURE 14. AFM image of graphene on Au-coated K-SPR sensor slide.

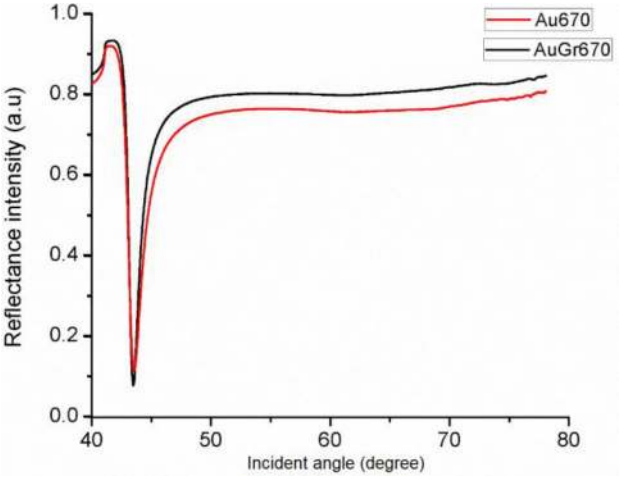

FIGURE 15. The characterized K-SPR curve for graphene/Au-based sensor slide at 670 nm.

was obtained from the graphene layer sample at the top of the gold SPR sensor and the resonance peak was 43.56° and 42.67° respectively for both wavelengths. The resulting SPR thickness is mapped based on the number of transfer processes involved. The complex refractive index (n, k) of the graphene layer was measured using a resonance surface value of 670 nm and 785 nm on an Au-coated sensor and is reported to be 2.85, 0.74, as well as 3.1, 1.19 respectively. These are about the same as the literary value of the graphene's refractive index.

The resulting layer width, where l refers to an increase in thickness for every transfer process, is also constructed on a linear curve, $d = l \times N + b$, representing a constant gap between the graph and the SPR sensor. The obtained SPR thicknesses depends on the number of transfer processes as reported by Jussila *et al.* [2] where the thickness of graphene layer on Au-coated SPR sensor was 0.31 nm. Moreover, the reported value of 0.335 nm for the graphene-graphene layer difference in graphite is in good agreement. Thus, the fitting

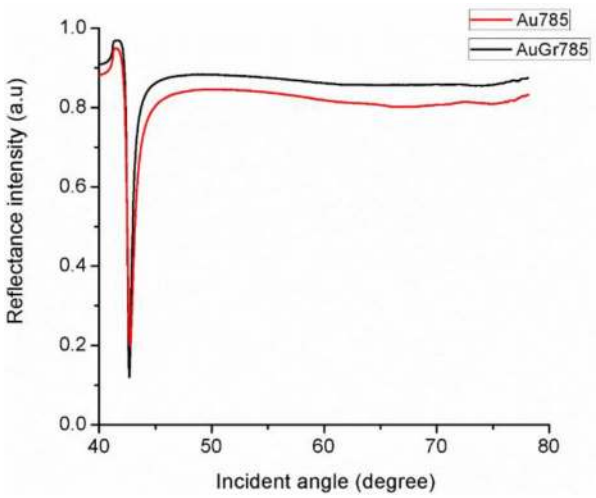

FIGURE 16. The characterized K-SPR curve for graphene/Au-based sensor slide at 785 nm.

constant of b is 0.42 [2]. Therefore, the thickness of graphene is 1.17 nm, indicating a bilayer graphene that matches the Raman spectroscopy measurement.

IV. CONCLUSION

In summary, a numerical analysis of the effects of adding graphene and MoS_2 layers on the Au-coated SPR sensor for the detection of ethanol to the sensitivity parameters was examined. The study aims to detect the presence of ethanol by observing an alteration in the minimum reflectance and maximum spectral spectrum of the SPR curve based on the Kretschmann method. Due to their biocompatibility and chemical stability properties, both graphene and MoS_2 thin films play a vital role in the progress of optical sensors. The sensitivity of 192.03°/RIU and 153.25°/RIU at 670 nm and 785 nm for the proposed sensor was observed numerically. The measured complex refractive index of the CVD-grown graphene layer on the Au-coated K-SPR sensor at 670 nm and 785 nm are 2.85, 0.74, and 3.1, 1.19, respectively. The measured thickness of graphene using K-SPR was 1.71 nm indicating the presence of bilayer graphene. In our future work, we intend to develop K-SPR biosensors with graphene and other two-dimensional materials, such as WS_2.

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