Multilayer CVD-Graphene and MoS$_2$ Ethanol Sensing and Characterization using Kretschmann-based SPR

P Suthitha Menon, Senior Member, IEEE, Nur Akmar Jamil, Gan Siew Mei, Ahmad Rifqi Md Zain, Daniel Hewak, Chung-Che Huang, Mohd Ambri Mohamed, Burhanuddin Yeop Majlis, Senior Member, IEEE, Ravi K. Mishra, Srinivasan Raghavan, Navakanta Bhat, Fellow, IEEE

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 (TMDS) 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 biochemical 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\textsubscript{2} for improvement in biosensing 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 ($R_{\text{min}}$) 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 et al. [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.

![Fig. 1. The simulated graphene/MoS\textsubscript{2}-coated Au-K-SPR sensor using Lumerical’s FDTD.](image)

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 ($\Delta \theta_{\text{res}}$) to the change in the refractive index of the sensing medium ($\Delta n_c$). In this paper, $\Delta n_c$ is assumed to be 0.0096 and 0.0116. The sensitivity can be given by:

$$S = \frac{\Delta \theta_{\text{res}}}{\Delta n_c}$$  \hspace{1cm} (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:

$$D.A = \frac{\Delta \theta_{\text{res}}}{\Delta \theta_{0.5}}$$  \hspace{1cm} (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\textsuperscript{-1} is governed by the sensitivity and the spectral width of the SPR curve at 50% reflectivity, which is given by:

$$Q = \frac{S}{\Delta \theta_{0.5}}$$  \hspace{1cm} (3)

Next, the schematic of the K-SPR detection method with the presence of chromium (Cr), gold (Au) and graphene/MoS\textsubscript{2} layers for the detection of ethanol is shown in Fig. 2. The shift in the resonance angle value, $\Delta \theta_{\text{res}}$ 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 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].

![Fig. 2. Schematic representation of the graphene/MoS\textsubscript{2} surface plasmon resonance (SPR) ethanol sensing technique based on the Kretschmann configuration](image)

B. Material

The multilayer CVD graphene grown on Cu foils and MoS\textsubscript{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\textsubscript{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 100x 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 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.

Fig. 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.

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].

Table 1 is produced by taking the resonance angle (Δθres) that conform to the change in the refractive index of sensing layers, with Rmin and spectral width of the K-SPR curve values in water and ethanol. A lower value of the minimum reflectance (Rmin) with reduced spectral width Δθ0.5 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 Δθ0.5 becomes narrower, more profound, and more effective in detecting resonance shifts at 785 nm compared to 670 nm. These Δθ0.5 values are calculated as low and high, with the lower and upper levels marked as low and high respectively. Δθ0.5 shows smaller values at 785 nm than 670 nm at a range of 2.4452 to 4.8905.

Moreover, Rmin 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 Rmin 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 Δθres value in the sensor layer ranges from 1.3284 to 1.3309 RIU and 1.34 – 1.3405 RIU for both wavelengths and is

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

Fig. 5. Comparison of K-SPR curves for water versus 10% ethanol detection for multilayer graphene/Au SPR sensor at 785 nm optical wavelength.
observed that the sensitivity increases after multilayers of graphene are added with values ranging from 133.09° to 192.03°/RIU. 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 $R_{\text{min}}$ and $\Delta \theta$ 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.

<table>
<thead>
<tr>
<th>Number of graphene layers</th>
<th>670 nm</th>
<th>785 nm</th>
</tr>
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<tbody>
<tr>
<td>$R_{\text{min}}$ for water</td>
<td>0.0128</td>
<td>0.0103</td>
</tr>
<tr>
<td>$\Delta \theta$ for water</td>
<td>2.4452</td>
<td>0.0128</td>
</tr>
<tr>
<td>$R_{\text{min}}$ for 10% ethanol</td>
<td>0.0502</td>
<td>0.0473</td>
</tr>
<tr>
<td>$\Delta \theta$ for 10% ethanol</td>
<td>3.0848</td>
<td>6.3577</td>
</tr>
</tbody>
</table>

Figs. 6 and 7. Minimum reflectivity ($R_{\text{min}}$) 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.
Fig. 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.

Fig. 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.

Fig. 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.

Further analysis was performed to analyze the SPR reflectance curves by considering Au/7 layers of graphene and Au/3 layers of MoS$_2$ K-SPR sensor with different ethanol concentrations in water at optical wavelength of 670 nm.

**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 (E$_1$ 2g) at the 383.49 cm$^{-1}$ and the S out-of-plane vibration (A$_1$ 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$_2$.
Fig. 12. Raman spectra of the bilayer graphene on the Au-coated SPR sensor.

Fig. 13. Raman spectra of the bilayer MoS$_2$ on the Au-coated SPR sensor.

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

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 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.

Fig. 15. The characterized K-SPR curve for graphene/Au-based sensor slide at 670 nm
The resulting layer width, where \(\ell\) refers to an increase in thickness for every transfer process, is also constructed on a linear curve, \(d = \ell \times N + b\), representing a constant gap between the graph and the SPR sensor. The obtained SPR thicknesses depend 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 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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REFERENCES

P Susthitha Menon (M’08–SM’14) obtained her B.Eng (Hons) and MSc (Microelectronics) degree from the National University of Malaysia (UKM) in 1998 and 2005 respectively. She obtained her Ph.D. degree (Optoelectronics) with Distinction in 2008, at the Institute of Microengineering and Nanoelectronics (IMEN), UKM, researching on InGaAs/InP p-i-n photodiodes. She was attached to Intel Malaysia as a Product Engineer from 1999 till 2002; during which she had a working stint as a New Product Integration (NPI) Engineer at Intel’s Systems Manufacturing Technology Division (SMTD) in Oregon, USA. She joined IMEN, UKM as a postdoctoral fellow in 2008 and worked on the development of GaAs/InP-based long-wavelength vertical-cavity surface emitting lasers (LW- VCSELs). Since 2009 till now, she is a senior research fellow at IMEN, UKM. She is the principal investigator for 4 national research projects, co-PI for one international research project, author of four book chapters, more than 200 indexed articles and 4 patents. Her current research interests are in the field of plasmonics, nanophotonics, optoelectronic devices, optical biosensing and imaging, and robust engineering optimization. She is currently the Vice Chair of the Region 10 IEEE Electron Devices Society (EDS) Subcommittee for Regions/Chapters and immediate past chair of IEEE EDS Malaysia Chapter.

Nur Akmar Jamil received her B.Eng. (Electrical) degree from Universiti Teknologi Malaysia in 2006. She received her M.Eng. Electrical – Telecommunication) degree from UKM in 2012. She worked as a system engineer at Technology Asian Resources-GET Sdn Bhd from 2007-2009. Then, she worked as a lecturer at Twintech International University College of Technology from 2010-2013. After that, she was attached as a lecturer in UCSI University. She is currently a PhD student at IMEN, UKM specializing in the field of Krestchmann-based surface plasmon resonance (SPR) sensing.

Gan Siew Mei received her BSc degree in Industrial Biotechnology from Universiti Malaysia Pahang (UMP) in 2013. Then she graduated with an MSc degree in Advanced Materials from UMP in 2016. She was a research assistant at IMEN, UKM.

Ahmad Rifqi Md Zain is an Associate Professor of Electronics Engineering at IMEN, UKM. He received his BEng and MSc degree from Coventry University and Glasgow University, UK in 2002 and 2005 respectively. He has worked with Marconi Communication as a hardware engineer from 2002-2004. In 2009, he received his PhD degree in Electronics Engineering from Glasgow University. After completing his postdoctoral studies in 2012 at Bristol University in GaN-based optical biosensors, he joined IMEN, UKM in 2014. In 2017, he joined Prof Marko Loncar at School of Engineering and Applied Sciences (SEAS), Harvard University as a postdoctoral research fellow in applied physics until 2019, working on Quantum Information. His research interests are in nanophotonics, optoelectronics and nanofabrication, optical imaging at infra-red region, 2D materials, optical based biosensors, 1D/2D PhC, optical MEMS sensors, environmental and agricultural based sensors, integrated optics and nanofabrication. He is a member of OSA, IEEE, IET and SPIE. He has been awarded several research grants nationally and internationally.

Daniel W Hewak, FlInstP, is Professor of Optoelectronics at the University of Southampton, and currently specializes in the production and application of advanced materials, thin films and optical fibres. Today, his research is leading to optical and composition fibres for sensing, medical and aerospace, 2D materials, a new generation of chalcogenide-glass lasers and solar-cell, thermoelectric, display and memory devices. This diversity is a testament to the truly advanced functionality of chalcogenides. He has a long-standing funded collaboration with the Universities of Surrey, Manchester, Cambridge, Bristol, Heriot-Watt, Exeter and Oxford. With reference to the ISI Web of Knowledge, he has 184 publications that have been cited over 3,152 times. His h-index is 33. He is PI on the £2.5M (EP/M015130/1), Manufacturing and Application of Next Generation Chalcogenide (Champ) and Co-I on the £10M (EP/N00762X/1) National Hub in High Value Photonic Manufacturing whose mandate is to support UK industry.

Chung-Chee Huang obtained his PhD in optoelectronics from the Optoelectronics Research Centre (ORC) at the University of Southampton in 2005. Currently, he is a Senior Research Fellow at the ORC, leading research in the fabrication of functional chalcogenides and emerging 2D/nano materials by chemical vapour deposition (CVD), atomic layer deposition (ALD) and van der Waals epitaxy (VdWE), including the successful fabrication of TMDCs and graphene at the wafer-scale. He has established more than 60 active academic/industrial collaborations in the area of 2D/nano materials for applications in photonic devices, phase-change memory, thin-film solar cells, photo-catalyst, nano-electronics and quantum technologies. He has been a research Co-I of some EPSRC and industrially funded grants. He has published over 120 journal and conference papers and holds one patent for CVD growth of chalcogenide materials.

Mohd Ambri Mohamed received his B.Eng in materials engineering from Tokyo University of Science in 2004 and MSc in the same field from Japan Advanced Institute of Science and Technology (JAIST) in 2007. He obtained his PhD degree from JAIST as well in 2010. He is currently the Deputy Director of IMEN, UKM since 2018. His current interests are Carbon electronics, Graphene and 2D related materials, III-V Semiconductors, MBE technology, Spintronics, Energy Harvesters, Materials Growth, Nano Devices and Characterizations.
Burhanuddin Yeop Majlis (SM’09) is a Professor of Microelectronics at IMEN, UKM. He received his PhD in Microelectronics from University of Durham, UK in 1988, MSc in microelectronics from University of Wales, UK in 1980, and BS (Hons.) in Physics from UKM in 1979. He was a Deputy Dean of Engineering Faculty from 1995 until 1997. He is also a Research Fellow of Telekom Malaysia Research & Development Division, and he was the director of UKM-TM Microelectronics Research Centre at the Faculty of Engineering, UKM. He was responsible in setting up of the clean room for research at UKM. He had attended intensive industrial training in GaAs MMIC at GEC-Marconi Material Technology Ltd. United Kingdom. He is a senior member of IEEE and the Chair of IEEE EDS Malaysia Chapter from 1994 to 2006. He also a Fellow Member of Malaysian Solid State Science and Technology Society (FMASS). He is the founder chairman and past President of Malaysia Nanotechnology Association (MNA), established in 2007. He initiated research in microfabrication and microsensors at UKM in 1995 and has also initiated research in GaAs technology with Telekom Malaysia. In 2001 he started research in MEMS with substantial research funding of US$10 million from Ministry of Science, Technology and Innovation. His current interests are design and fabrication of MEMS sensor, RFMEMS, BioMEMS and microenergy. He has published four text books in electronics and one book on Integrated Circuits Fabrication Technology for undergraduate courses and more than 400 academic research papers. He is the founder of Institute of Microengineering and Nanoelectronics (IMEN) in 2002. In 2019, he was awarded Achievement in Academic Award 2019 from the Institution of Engineering and Technology (IET) Malaysia.

Ravi K Mishra received his M.Tech degree in Microelectronics from the Indian Institute of Technology Bombay in India. He is currently a PhD student at the Center for Nano Science and Engineering IISc since 2016. His research interest include growth of 2D materials.

Srinivasan Raghavan received his B.E. in Metallurgy VNIT, Nagpur, India, ME degree in Metallurgy from the Indian Institute of Science (IISc), Bangalore, India and PhD degree in Materials Science and Engineering from the Pennsylvania State University, USA. He is currently a Professor at the Centre for Nanoscience and Engineering (CeNSE), IISc Bangalore with research interests in electronics, GaN technology, solar cells, device modeling & simulation, low-dimensional semiconductors, metal-oxides, nitrides, technology development and CVD reactor for GaN.

Navakanta Bhat (F’19) received his B.E. in Electronics and Communication from SJCE, University of Mysore in 1989, M.Tech. in Microelectronics from IIT Bombay in 1992 and Ph.D. in Electrical Engineering from Stanford University in 1996. Then he worked at Motorola’s Networking and Computing Systems Group under Advanced Products R&D Lab (APRDL) in Austin, TX until 1999. At Motorola he worked on logic technology development and he was responsible for developing high performance transistor design and dual gate oxide technology.

He joined the Indian Institute of Science, Bangalore in 1999 where he is currently a Professor and Chair, Centre for Nano Science and Engineering. His current research is focused on Nanoelectronics device technology, Biosensors for point of care diagnostics and Gas sensors for pollution monitoring. He has 240 research publications in international journals and conferences and 10 granted US patents and 14 pending patents to his credit. He was instrumental in creating the National Nanofabrication Centre (NNfC) at IISc, Bangalore, benchmarked against the best university facilities in the world. He served as the chairman of NNfC administration committee from 2010 to 2015.

He is a Fellow of the Indian National Academy of Engineering. He has received the Young Engineer Award (2003) from the Indian National Academy of Engineering, Swarnajayanti fellowship (2005) from the Department of Science and Technology, Govt. of India and Prof. Satish Dhawan award (2005) from the Govt. of Karnataka. He is also the recipient of IBM Faculty award 2007 and Outstanding Research Investigator award (2010) from DAE. For his translational research work, he has received the prestigious Dr. Abdul Kalam Technology Innovation National Fellowship (2018) and Prof. Rustum Choksi award for Excellence in Engineering Research (2017). He is a Fellow of IEEE, and is currently (2016-2019) a member of the Board of Governors of the IEEE Electron Devices Society and also the Chair of Nanotechnology technical committee. He is also the founder and promoter of a startup company, PathShodh Healthcare Pvt Ltd (www.pathshodh.com).