



Sensitivity Enhancement by the Ag/Few-Layer Black Phosphorus/Mxene Structure in Surface Plasmon Resonance Biochemical Sensor

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Abstract

A highly sensitive surface plasmon resonance biosensor composed of the hybrid black phosphorus-MXene ($\text{Ti}_3\text{C}_2\text{T}_x$) structure and Ag bimetal layers is designed and investigated analytically by the angular interrogation technique. The use of bilayer Ag enhances the sensitivity compared to a single metal layer. Black phosphorus enhances the light-matter interaction due to its layer-dependent direct and tunable bandgap. The thickness of the BP and Ag layers and the number of $\text{Ti}_3\text{C}_2\text{T}_x$ sheets are optimized to maximize the sensitivity. It covers a wide range of refractive index (RI) from 1.330 to 1.335, for which an optimized angular sensitivity of $450^\circ/\text{RIU}$ is observed from the Ag bimetal MXene-based SPR biosensor. It can be fabricated as a sensor chip for efficient sensing of analytes or biochemical molecules by carefully controlling the surface termination of $\text{Ti}_3\text{C}_2\text{T}_x$. The encouraging results from this research underscore the high potential of this structure in surface plasmon resonance biosensors, which is crucial for the early detection of biological diseases. By overcoming the limitations of traditional sensors, this innovative structure boasts both high sensitivity and rapid response speed, thereby demonstrating the remarkable potential for market adoption and application in biomedical research, ultimately enhancing the effectiveness of disease treatment through earlier interventions.

Keywords Black phosphorus · MXene · Angular interrogation · Surface plasmon resonance · Sensitivity

Introduction

Recently, the promotion of interactions between biological analytes and detection platforms for the purpose of flexible detection has become a hot research topic in the field of biotechnology [3–7]. Optical biosensors based on the surface

plasmon resonance (SPR) effect have gained widespread popularity in various applications, including drug discovery, enzyme detection, and medical diagnosis [2, 50]. Surface plasmons (SPPs) are electron-density oscillations occurring at the interface between a metal and dielectric characterized by negative and positive permittivity, respectively [6, 8–11]. In SPR biosensors, the reversible adsorption of biomolecules (analyte) on the sensing surface (metal/dielectric interface) alters the local refractive index (RI) and the SPR condition.

SPR biosensors can be categorized into two prism coupling types, Otto and Kretschmann configurations. Both configurations operate based on the angular interrogation technique. The Kretschmann configuration is simple and produces a high signal-to-noise ratio (SNR) with single radiation. In traditional SPR sensors, metallic materials such as silver (Ag), gold (Au), aluminum (Al), and copper (Cu) are employed to produce the plasmonic effects for sensing [11–14]. Recently, 2D materials such as graphene, MoS_2 , and black phosphorous (BP) have been proposed for SPR biosensors [15, 25, 27–29,] because 2D materials can enhance the sensitivity. MXene

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($\text{Ti}_3\text{C}_2\text{T}_x$) contains functional groups making it suitable for the detection of certain analytes [31–33]. Wu et al. have designed an SPR biosensor by increasing the BP layer to enhance the sensitivity caused by light absorption of graphene [14]. The sensitivity increases to a maximum of $217^\circ/\text{RIU}$ when the number of graphene layers is 5. Singh et al. have designed an SPR chemical sensor with black phosphorus and graphene together with a gold bilayer for enhanced sensitivity [44]. Mudgal et al. have proposed a highly sensitive SPR biosensor based on silver (Ag), barium titanate (BaTiO_3), graphene, and affinity layer for the detection of *Pseudomonas* bacteria [49].

Black phosphorous (BP) is an allotrope of phosphorus having a honeycomb lattice structure bounded covalently within the layer through van der Waals forces. The structure provides easy exfoliation of thin phosphorene layers. BP possesses a direct and tunable band gap ranging from 0.3 eV (bulk) to 2 eV (monolayer), making it highly versatile for electronic and optoelectronic device applications [16, 17]. In addition, the direction-dependent crystallographic structure of BP results in the anisotropic behavior which makes BP quite useful for direction-dependant applications [20]. BP's biocompatibility also opens the possibilities for its uses for the applications in biomedicine such as drug delivery. MXene is an emerging two-dimensional material known for its unique physiochemical characteristics and ultrathin structures, surpassing graphene and other 2D materials. MXene is more advantageous than other 2D materials due to its two main properties: metallic nature and hydrophilicity [32, 33]. MXene is used in SPR because it increases the biomolecule adsorption rate and prevents the metal layer from oxidation. These characteristics make them promising candidates for exemplary performance in biosensing devices.

The hybrid structure of BP and MXene ($\text{Ti}_3\text{C}_2\text{T}_x$) can be used in the SPR sensor to enhance the sensing properties for adsorbed analytes due to the functionalized surface of $\text{Ti}_3\text{C}_2\text{T}_x$ and enhanced light-matter interactions rendered by BP [1, 29, 31]. Their high success rate in detecting bacteria, DNA, viruses, antibodies, lipids, peptides, and ions [22, 22, 24, 33] encourages us to undertake *Pseudomonas* bacteria detection in this study. The affinity layer is placed just below the sensing medium. It bonds with *Pseudomonas* bacteria [52]. Many *Pseudomonas* bacteria were attached to the affinity layer for carbon sources such as toluene and nicotine. The adhesion of *Pseudomonas* bacteria to hydrophilic metals was studied. When the sensor is exposed to different analytes for detection, the physical properties of the material at the end of the sensor, such as hydrophilic ability and molecular absorption capacity, can be utilized to achieve interaction between the sensor and the analyte. This enables the acquisition of offset resonance curves for analytes with different refractive indices, achieving the purpose of detection and identification.

After vertically stacking different 2D-layered materials together, van der Waals heterostructures (vdWh) and superlattices are formed. These structures can manipulate optical and electronic properties based on the interaction between their layers, allowing for the development of next-generation devices. Herein, the hybrid prism biochemical sensor consisting of low refractive index CaF_2 , bilayer metallic silver [43], and 2D materials black phosphorus and MXene ($\text{Ti}_3\text{C}_2\text{T}_x$) is designed, and the SPR sensor based on the Kretschmann configuration is numerically analyzed under visible-light irradiation by angular interrogation, which is designed to stimulate surface plasmon resonance to enhance the sensitivity. The proposed SPR biosensor performance is realized theoretically through COMSOL software. By optimizing the structural parameters, the best sensitivity is attained from the structure with nine layers of black phosphorus and 7-nm-thick silver. The best sensitivity at 662 nm is $450^\circ/\text{RIU}$, which is 3.9 and 2.5 times higher than that of the prismatic sensors based on Ag-BP structures and conventional single-layer metal, respectively. Our results provide insights into the excitation of SPR and the development of biochemical sensors with enhanced sensitivity.

Design and Theoretical Model

The SPR biochemical sensor composed of few-layer BP-MXene is depicted schematically in Fig. 1. In this structure, CaF_2 glass ($n_p = 1.43286$ at $\lambda = 662$ nm) is chosen for the coupling prism, and the 40-nm-thick silver (Ag) metal film excites SPP.

According to the Drude model, the dielectric constant of Ag can be determined by Eq. (1) [40]:

$$n_m = \sqrt{\epsilon_m} = \left[1 - \frac{\lambda^2 \lambda_c}{\lambda_p^2 (\lambda_c + i\lambda)} \right]^{1/2}, \quad (1)$$

where λ_p and λ_c represent the plasma and collision wavelengths, and the values of λ_p and λ_c for Ag are 1.4541×10^{-7} m and 1.7614×10^{-5} m, respectively.

Black phosphorus (BP), as a promising new 2D material, joined the 2D material family since 2014 and has rapidly attracted significant attentions due to its widely tunable and direct bandgap, high carrier mobility, and remarkable electrical, optical, and phonon properties [25, 26, 41]. However, BP is a readily oxidizable material in ambient conditions which needs a protective layer to prevent it from oxidation. Here, we chose Ag to be coated on BP as a protective layer to compensate for its easy oxidative volatilization and further improve sensitivity. Therefore, the sensor structure of this design is coated with a few layers of BP (5.85 nm, containing nearly nine layers) which is coated on the Ag film, and an Ag layer is deposited on the few-layer BP to prevent oxidation. The fifth layer is $\text{Ti}_3\text{C}_2\text{T}_x$

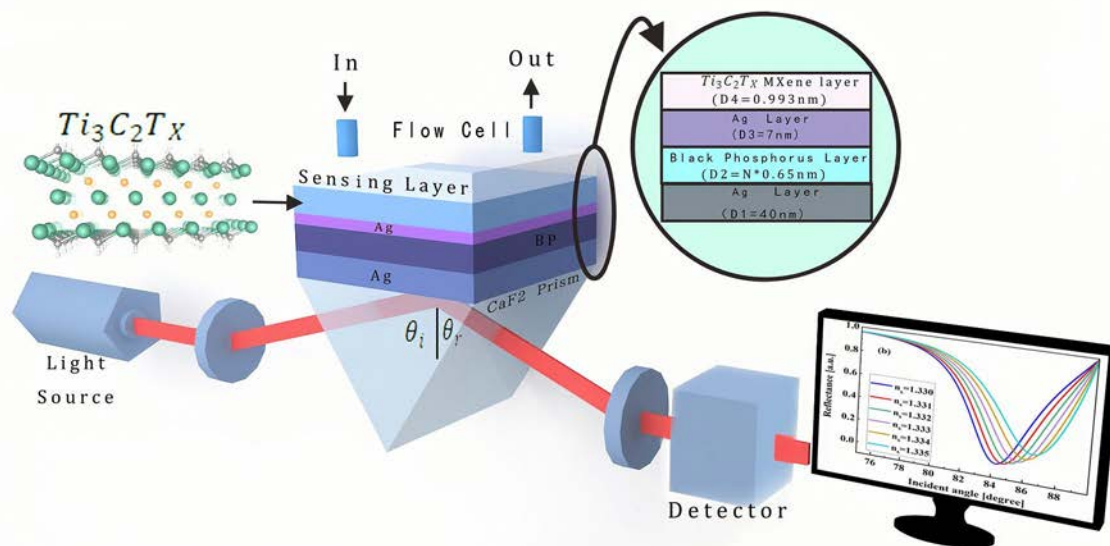


Fig. 1 Schematic diagram of the SPR sensor and detection setup

with a refractive index (RI) of $2.38 + 1.33i$ on the Ag layer to avoid the oxidation of Ag [30, 34]. The $Ti_3C_2T_x$ layer deposited on Ag as the BRE layer can be used to sense biochemical molecules and analytes [41]. The hydrophilic functional group (i.e., $-OH, -O, -F$) on $Ti_3C_2T_x$ captures water molecules for detecting biochemical molecules in an aqueous solution with enhanced sensitivity [19]. At the same time, the layered $Ti_3C_2T_x$ has a larger surface area allowing the attachment of more biochemical molecules/analytes. The thickness and refractive index of the sensor at $\lambda = 662\text{ nm}$ are shown in Table 1.

Enzymes, proteins, and DNA can be detected. The sensing medium is the last layer, and in an aqueous solution, the RI is $1.33 + \Delta n_{SM}$, where $\Delta n_{SM} = 0.005$ is the RI shift due to analyte adsorption on $Ti_3C_2T_x$ [19, 41]. The RI range is between 1.330 and 1.335, attributable to variations in the mass ratios of

solutes in the phosphate buffer saline (PBS) solution [19]. The reflectivity of the multilayer structure is derived by Eq. (2) [41]:

$$r_{pm_1d_1m_2d_2} = \frac{r_{pm_1} + r_{m_1d_1m_2d_2} e^{2iK_{m_1x}d_{m_1}}}{1 + r_{pm_1} r_{m_1d_1m_2d_2} e^{2iK_{m_1x}d_{m_1}}}, \tag{2}$$

and

$$K_{ix} = \sqrt{\left(\frac{2\pi}{\lambda}\right)^2 \epsilon_i - K_z^2}; i = p, m_1, d_1, m_2, d_2 \tag{3}$$

$$r_{pm} = \frac{(\epsilon_m K_{px} - \epsilon_p K_{mx})}{(\epsilon_m K_{px} + \epsilon_p K_{mx})}; r_{md} = \frac{(\epsilon_d K_{mx} - \epsilon_m K_{dx})}{(\epsilon_d K_{mx} + \epsilon_m K_{dx})}, \tag{4}$$

where p, m , and d denote the prism, metal, and dielectrics, respectively.

The resonance dip in the reflectance curve occurs when the resonance condition is satisfied, as shown by Eq. (2). Therefore, the coupling equation for incident light and SPs at the metal–dielectric interface can be derived from Eq. (2) as follows:

$$k_z = k_{sp} = \frac{2\pi}{\lambda} n_p \sin \theta_{spr} = \text{real}\left(\frac{2\pi}{\lambda} \sqrt{\frac{\epsilon_m \epsilon_s}{\epsilon_m + \epsilon_s}}\right) \tag{5}$$

In Eq. (5), n_p is the RI of the substrate (glass/prism), and λ is the wavelength. The dielectric constants of the metal layer and sensing (analyte) layers are ϵ_m and ϵ_s , respectively. The

Table 1 Thickness and refractive index of sensors at $\lambda = 662\text{ nm}$

Layers	Materials	Refractive index ($\lambda = 662\text{ nm}$)		Thickness (nm)	Ref
		Real (n)	Imaginary (k)		
I	Prism CaF_2	1.43286	—	—	[21]
II	Metal Ag	0.04944	4.5027	40	[41]
III	2D material BP	3.8337	0.015215	0.65	[1]
IV	Metal Ag	0.04944	4.5027	7	[41]
V	Mxene $Ti_3C_2T_x$			0.993	[42]

angle at which the SPR curve dips (R_{\min}) is the resonance angle (SPR angle), which occurs due to the transition in the RI of the analyte.

A comparative analysis of several prisms [21] in terms of the reflectance curves indicates that the CaF_2 prism has the largest SPR angle, and it is chosen as a light coupling glass at 662 nm. The structure consists of layers of Ag, BP, Ag, and $\text{Ti}_3\text{C}_2\text{T}_x$ on top of the coupling prism, with Ag serving as the SPR-active metal layer. $\text{Ti}_3\text{C}_2\text{T}_x$ is considered the last layer that interacts directly with the analyte. The dielectric constants of $\text{Ti}_3\text{C}_2\text{T}_x$, Ag, CaF_2 , and BP at 662 nm are extracted from Refs. [44–46].

The essential sensor characteristic, sensitivity, illustrates how the resonance angle changes in response to the sensing medium's RI changes. Its value should be greater for the SPR sensor. The sensitivity (S) is calculated by Eq. (6) [44]:

$$S = \frac{\Delta\theta_{SPR}}{\Delta n_s}, \quad (6)$$

The figure of merit (FOM) is an important index to analyze the comprehensive properties of sensors according to Eq. (7) [47]:

$$FOM = \frac{S}{FWHM}, \quad (7)$$

Detection accuracy (D.A.) is the reciprocal of the FWHM [48]. Signal-to-noise ratio (SNR) and detection limit (DL) are also considered important performance parameters [47]. SNR is used to measure the ratio of signal to noise, with

higher SNR indicating better performance of the sensor. DL represents the lowest amount of analyte that can be reliably detected by the proposed sensor, with a preference for smaller values [47]. The D.A., SNR, and DL of the sensor have been investigated [44, 47, 51] and calculated as:

$$DA = \frac{1}{FWHM}, \quad (8)$$

$$SNR = \frac{\Delta\theta_{SPR}}{FWHM}, \quad (9)$$

$$DL = \frac{\Delta n_s}{\Delta\theta_{SPR}} \times 0.001^\circ. \quad (10)$$

Fabrication Steps

The possible fabrication steps have been shown here step by step and can be illustrated in Fig. 2.

The proposed SPR sensor is composed of a multilayer structure based on the Kretschmann configuration. The layers of Ag, BP, and $\text{Ti}_3\text{C}_2\text{T}_x$ may be deposited by physical vapor deposition (PVD) [49], chemical vapor deposition (CVD), and plasma fluorination.

Finally, a sensing medium has been established for the analyte-biochemical molecules interaction. After the manufacturing process, the SPR chip is placed above the prism, and experimental calculations are found using the SPR sensor setup.

Fig. 2 Schematic diagram of the manufacturing steps of the proposed sensor

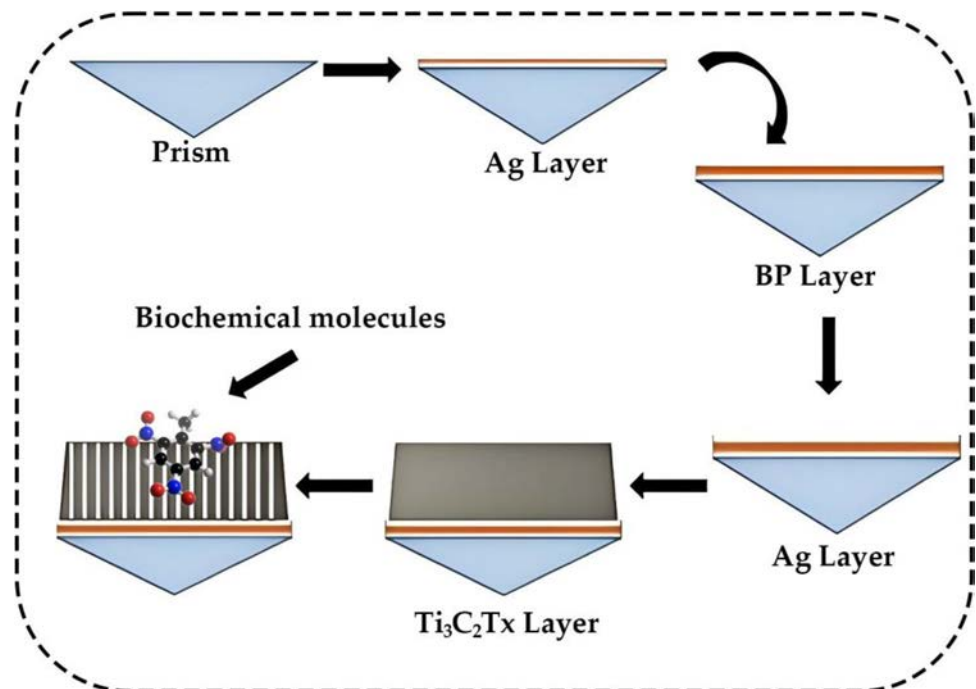
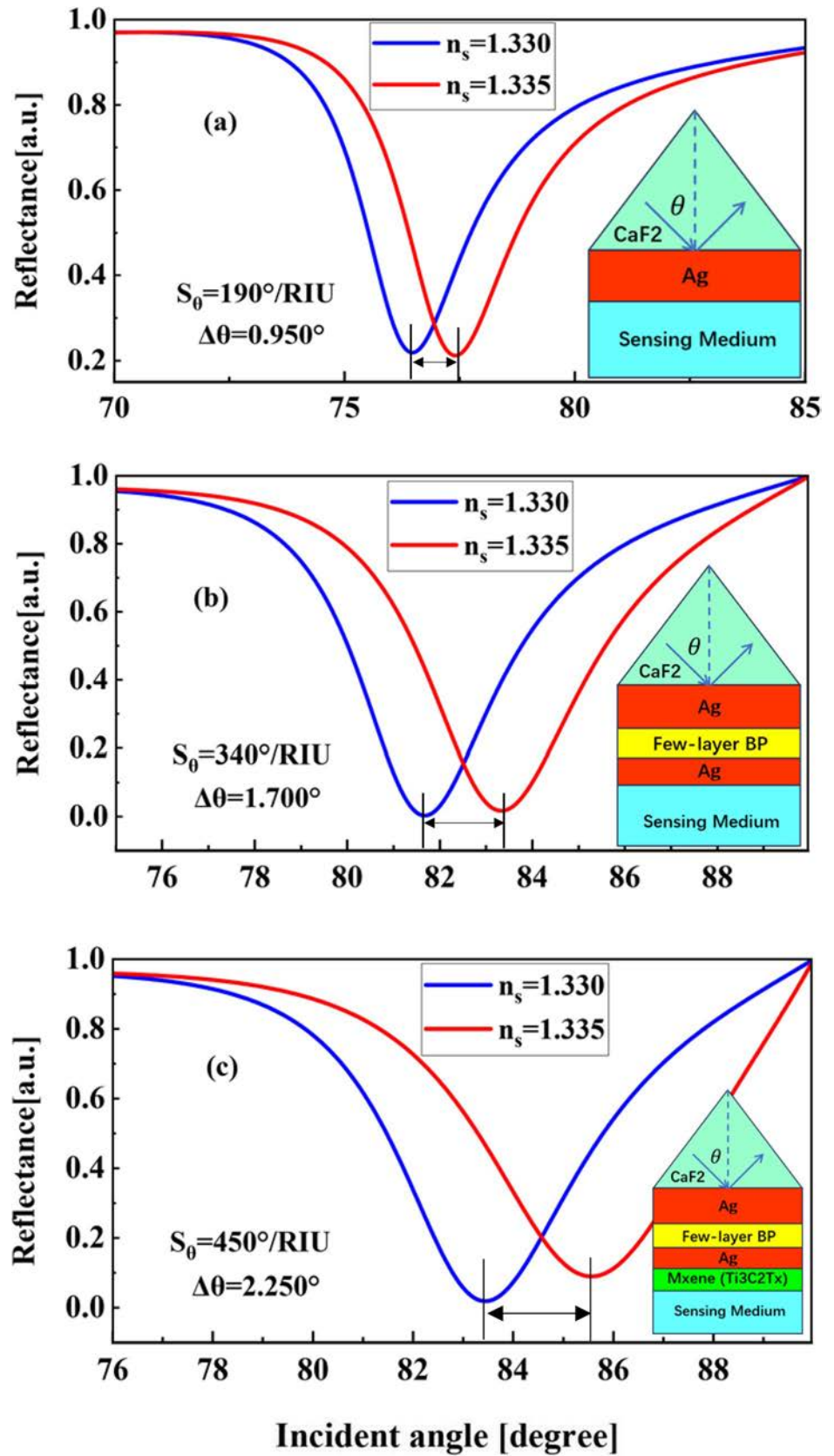


Fig. 3 Variation of the reflectance with respect to the incident angle: **a** conventional biochemical sensor with a single Ag film, **b** biochemical sensor with two Ag—5.85-nm-thick few-layer BP, and **c** biochemical sensor with the 5-nm-thick BP—Ti₃C₂T_x heterostructure



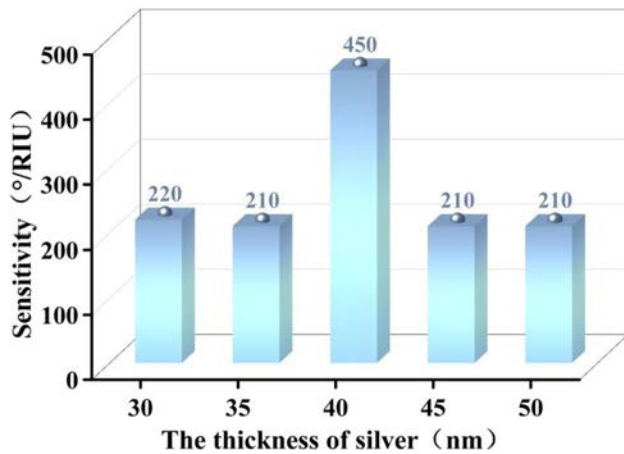


Fig. 4 Sensitivity of the first layer of silver at different thicknesses

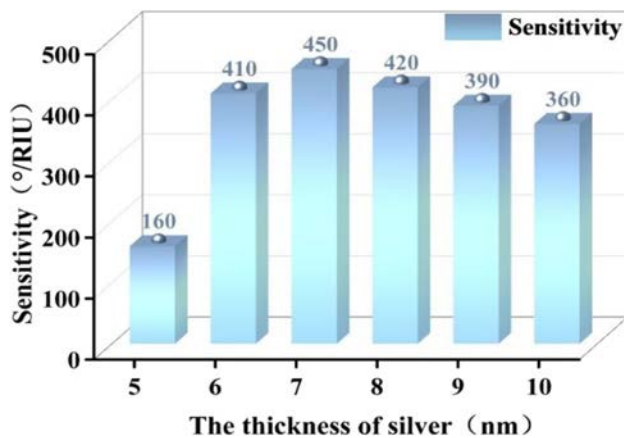


Fig. 5 Sensitivity of silver layers with different thicknesses

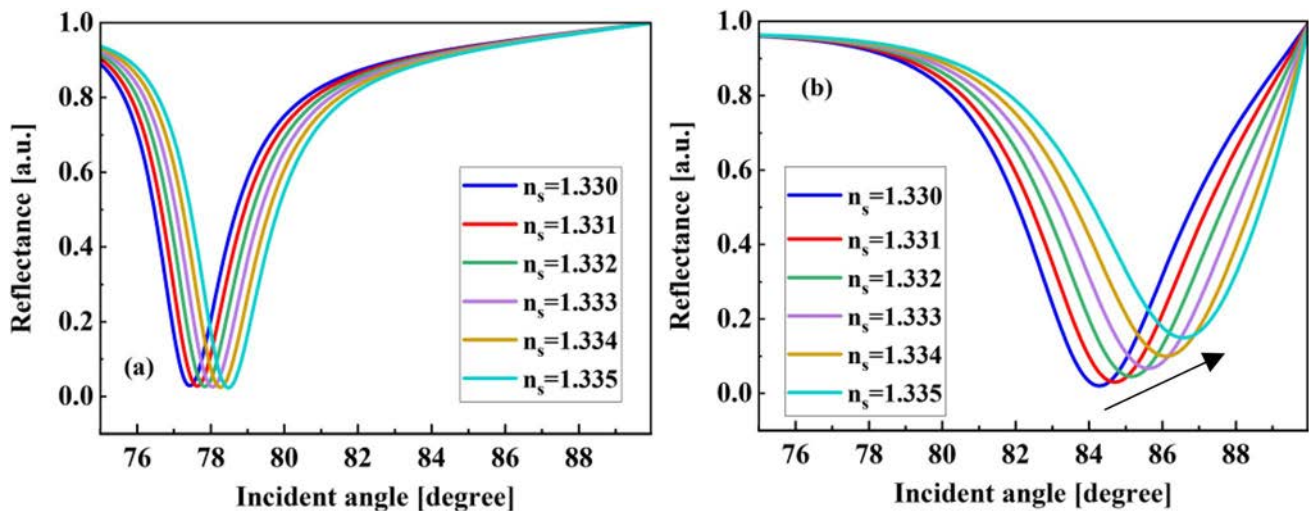


Fig. 6 a Variation of reflectance with respect to the incident angle for the refractive index range from 1.330 to 1.335 and b BP layers

Results and Discussion

The coupling prism is made of CaF_2 because the relatively low RI can improve the sensitivity. The reflectance graphs as a function of the incident angle are acquired from the single-layer and multilayer sensors, as shown in Fig. 3. The schematic of the traditional SPR biochemical sensor based on the Kretschmann geometry comprising the sensing medium, Ag layer, and CaF_2 (shown in insets) is shown in Fig. 3a. As the RI of the sensing medium varies from $n=1.330$ to 1.335, red-shifting is observed from the reflectance spectra [1]. The sensitivity reaches a maximum of $190^\circ/\text{RIU}$, but it falls short in detecting biomolecules with extremely low concentrations.

To boost the performance of the RI sensor, nine layers of BP are applied to the Ag layer in the conventional structure. Additionally, a 7-nm-thick silver layer is added to the top of the BP to mitigate oxidation in air. The results show that the sensitivity of the structure with 5.85-nm-thick BP and 7-nm-thick Ag is improved, as shown in Fig. 3b. The best sensitivity achieved is $340^\circ/\text{RIU}$. For further improvement, a $\text{Ti}_3\text{C}_2\text{T}_x$ layer with a thickness of 0.993 nm is coated on the 7-nm-thick Ag. The SPR resonance curve in Fig. 3c discloses a sensitivity as high as $450^\circ/\text{RIU}$.

The thickness of other materials is controlled, while the thickness of the first Ag layer is set to 30 nm, 35 nm, 40 nm, 45 nm, and 50 nm respectively. The sensitivities for the different Ag thicknesses are shown in Fig. 4. The results indicate that the sensitivity is the highest ($450^\circ/\text{RIU}$) when the silver thickness is 40 nm.

The Ag coating with a thickness of 40 nm on black phosphorus as the third layer does not produce the surface plasmon resonance effects. Therefore, in order to increase the sensitivity, the thickness of the third Ag layer is optimized.

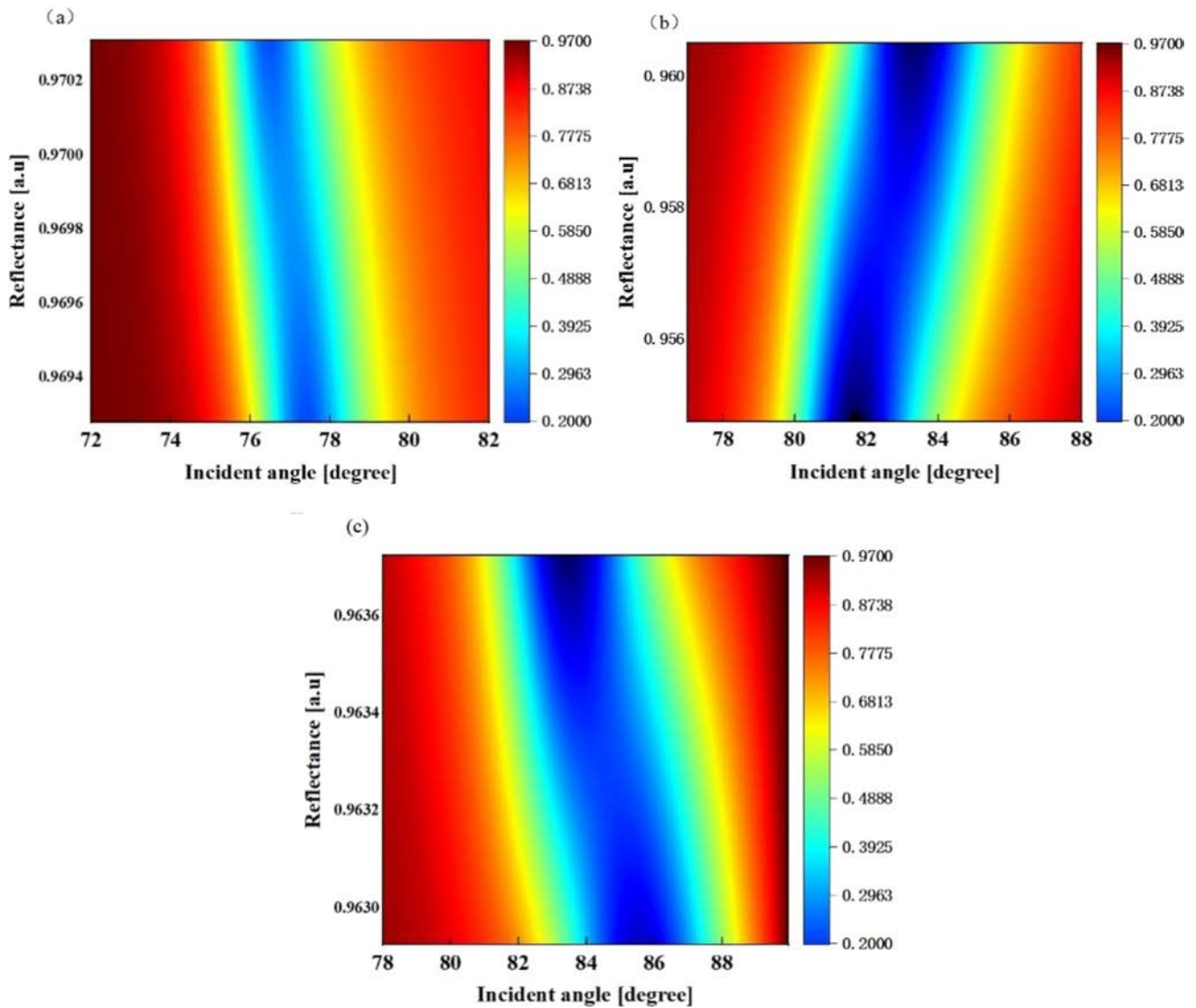


Fig. 7 Parametric contour plots: **a** CaF₂-Ag, **b** CaF₂-Ag-BP-Ag, and **c** CaF₂-Ag-BP-Ag-Ti₃C₂T_x

Table 2 Comparison of sensing performance at $\lambda = 662$ nm

Configuration (CaF ₂ +)	$\Delta\theta_{SPR}$ (degree)	Sensitivity (°/RIU)	Analyte RI range ($n = 0.005$)
Ag	0.95	190	1.330 to 1.335
Ag + BP + Ag	1.70	340	1.330 to 1.335
Ag + BP + Ag + Ti ₃ C ₂ T _x	2.25	450	1.330 to 1.335

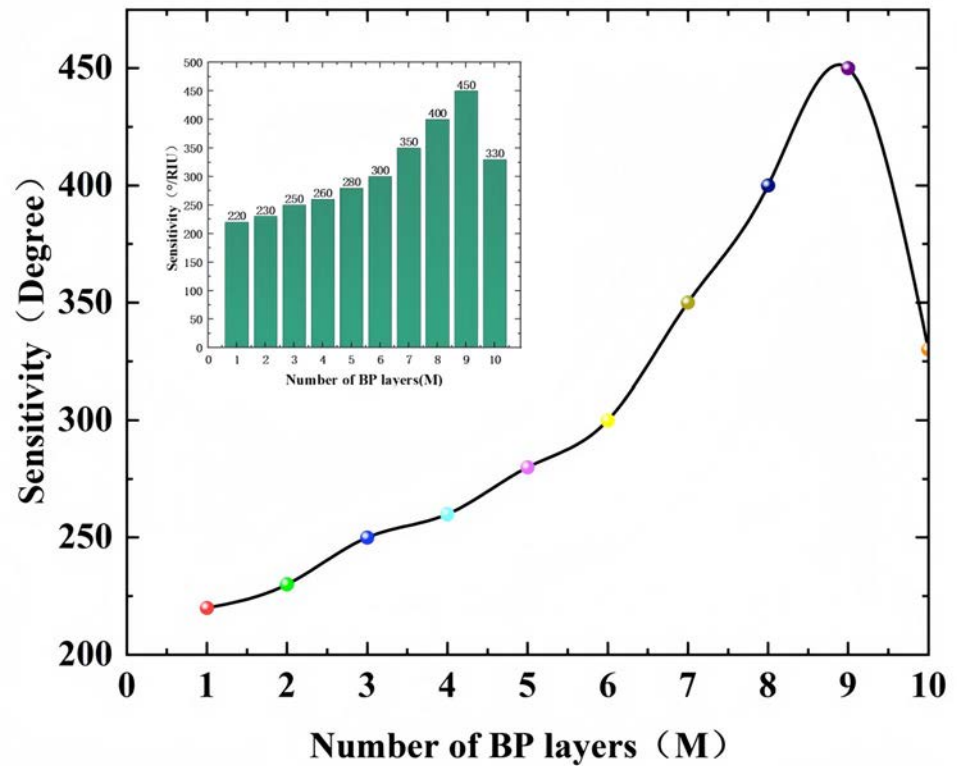
The sensitivity for different Ag thicknesses is shown in Fig. 5. The sensitivity increases as the thickness of the Ag layer increases, but the sensitivity decreases when the thickness exceeds 7 nm. The results show that the sensitivity is the highest (450°/RIU) when the silver thickness is 7 nm.

Figure 6a shows the responses of the structures with two-layer Ag and single-layer Ti₃C₂T_x, but without BP. Figure 6b shows the reflectance for different incidence angles of the structure

Table 3 Sensor structure with their computed performance factors

Configuration (CaF ₂ +)	FWHM (degree)	DA (degree ⁻¹)	FOM (RIU ⁻¹)	DL ($\times 10^{-6}$)	SNR (dB)
Ag	2.89	0.346	65.74	2.38	0.48
Ag + BP + Ag	3.68	0.272	92.39	2.94	0.46
Ag + BP + Ag + Ti ₃ C ₂ T _x	4.35	0.23	103.45	5.26	0.33

Fig. 8 Sensitivity of $M=9$ and $N=1$ for different refractive indexes



comprising $\text{Ag}+\text{BP}+\text{Ag}+\text{Ti}_3\text{C}_2\text{T}_x$ within the RI range from 1.330 to 1.335. The wave valley moves toward a larger incidence angle as the RI increases. The resonance angles are 84.3° , 84.7° , 85.15° , 85.6° , 86.1° , and 82.15° for $n_s = 1.330$, 1.331, 1.332, 1.333, 1.334, and 1.335, respectively. Figure 7 presents the contour diagrams illustrating the relationship between the incident angle and reflectance of the different structures.

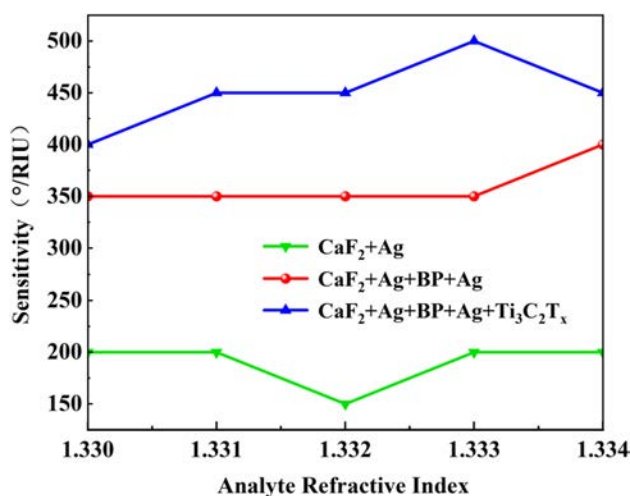


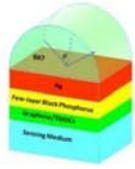
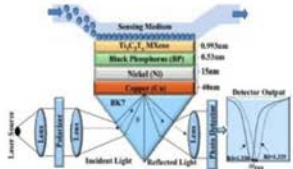
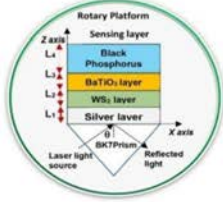
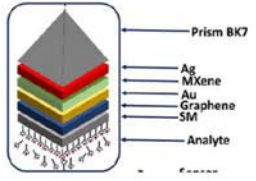
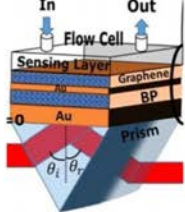
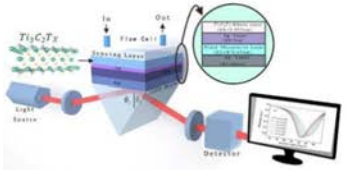
Fig. 9 Optimization of the sensitivity of different layer configurations for the RI range between 1.330 and 1.334 RIU

The sensitivities of conventional, bimetallic, and composite structures at a wavelength λ of 662 nm are shown in Table 2. The highest sensitivity of 450 ($^\circ/\text{RIU}$) is achieved for the analyte RI change of $\Delta n_s = 0.001$. The structure ($\text{CaF}_2 + \text{Ag} + \text{BP} + \text{Ag} + \text{Ti}_3\text{C}_2\text{T}_x$) shows a shift in the resonance angle of $\Delta\theta_{\text{SPR}} = 2.25^\circ$ when the analyte RI changes from 1.330 to 1.335, indicating that the sensitivity is indeed impacted by the layer number. The values computed for different performance factors are shown in Table 3.

To elucidate the physical mechanism between the number of black phosphorus layers and sensitivity, Fig. 8 shows the effects of the number of BP layers (M) with that of $\text{Ti}_3\text{C}_2\text{T}_x$ ($N=1$) unchanged. The refractive index of the sensing medium is set to $1.33 + \Delta n$. The sensitivity increases initially to a maximum of $450^\circ/\text{RIU}$ when the number of BP layers is 9 ($M=9$) but declines subsequently when $M > 9$.

Figure 9 shows the analysis for the RI range between 1.330 and 1.334. In the analysis, the sensitivity of a single layer of metallic silver is derived for different refractive indexes. The same is performed on the BP layer and titanium carbide layer, and the sensitivities are calculated separately. The sensitivity of the sensor increases gradually with the number of layers. Table 4 compares the performance of different sensors confirming the superiority of our sensor.

Table 4 Comparative analysis of sensitivity with relevant research work

References	Configuration	Sensitivity (°/RIU)	Structures
[1]	BP/Graphene/MoS ₂ /WS ₂ / MoSe ₂ /WSe ₂	279	
[4]	Cu/Ni/BP/MXene	304.4	
[2]	WS ₂ /BaTiO ₃ /BP	370	
[3]	Prism/Ag/MXene/ Au/Graphene	301.2	
[45]	Prism/Au/BP/Au/S ensing Layer	218	
This work	CaF ₂ /Ag/BP/Ag/Ti ₃ C ₂ T _x	450	

Conclusions

An SPR biochemical sensor composed of a CaF₂ prism, BP, MXene, and bimetallic Ag is designed to enhance the sensitivity. The sensitivity of the structure with silver coated on the CaF₂ prism is 190°/RIU. By introducing BP and another Ag layer, the sensitivity increases to 340°/

RIU. When Ti₃C₂T_x 2D is coated as a protective layer on the third Ag layer, the sensitivity is further improved. The heterostructure consisting of 0.993 nm-thick Ti₃C₂T_x and BP shows the highest sensitivity of 450°/RIU. Compared to the conventional structure with one silver film, the sensitivity of the SPR comprising Ag, few-layer BP, and Ti₃C₂T_x is improved by nearly 3.9 times. The results provide

insights into the design of high-performance SPR sensors for biochemical applications. In the future, SPR sensors will integrate a wide range of cutting-edge technologies, including computing, microfluidics, AI, and bioengineering, in order to improve their performance. Furthermore, the utilization of optical waveguide technology will enable the creation of efficient and miniature sensing elements, facilitating multi-sensor integration and further enhancing sensing capabilities.

Author Contributions A.Jingwei Lv: Conceptualization, Writing- Reviewing and Editing. B.Ying Yu: Conceptualization, Methodology, Software, Writing-Original Draft. C.Chao Mi: Investigation, Validation. D.Debao Wang: Formal analysis. E.Wei Li: Funding acquisition, Validation. F.Yanru Ren: Investigation. G.Jianxin Wang: Conceptualization. H.Wei Liu: Resources. I.Paul K. Chu: Project administration. J.Chao Liu: Resources, Writing- Reviewing and Editing. All authors reviewed the manuscript.

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Data Availability No datasets were generated or analysed during the current study.

Declarations

Competing Interests The authors declare no competing interests.

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