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Interface engineering in 2D materials for SERS sensing

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In recent years, the use of 2D materials as surface-enhanced Raman scattering (SERS) active substrates has been extensively investigated. This mini review emphasizes the tunable interface characteristics of 2D materials as SERS substrates. Notably, 2D materials exhibit a wealth of tunable properties, encompassing energy levels, band structures, and interlayer coupling, which confer upon them significant potential as versatile SERS substrates. We summarize the recent advances in tunable 2D materials as SERS active substrates, providing guidance for future research in developing tunable multifunctional molecular sensors based on 2D materials.

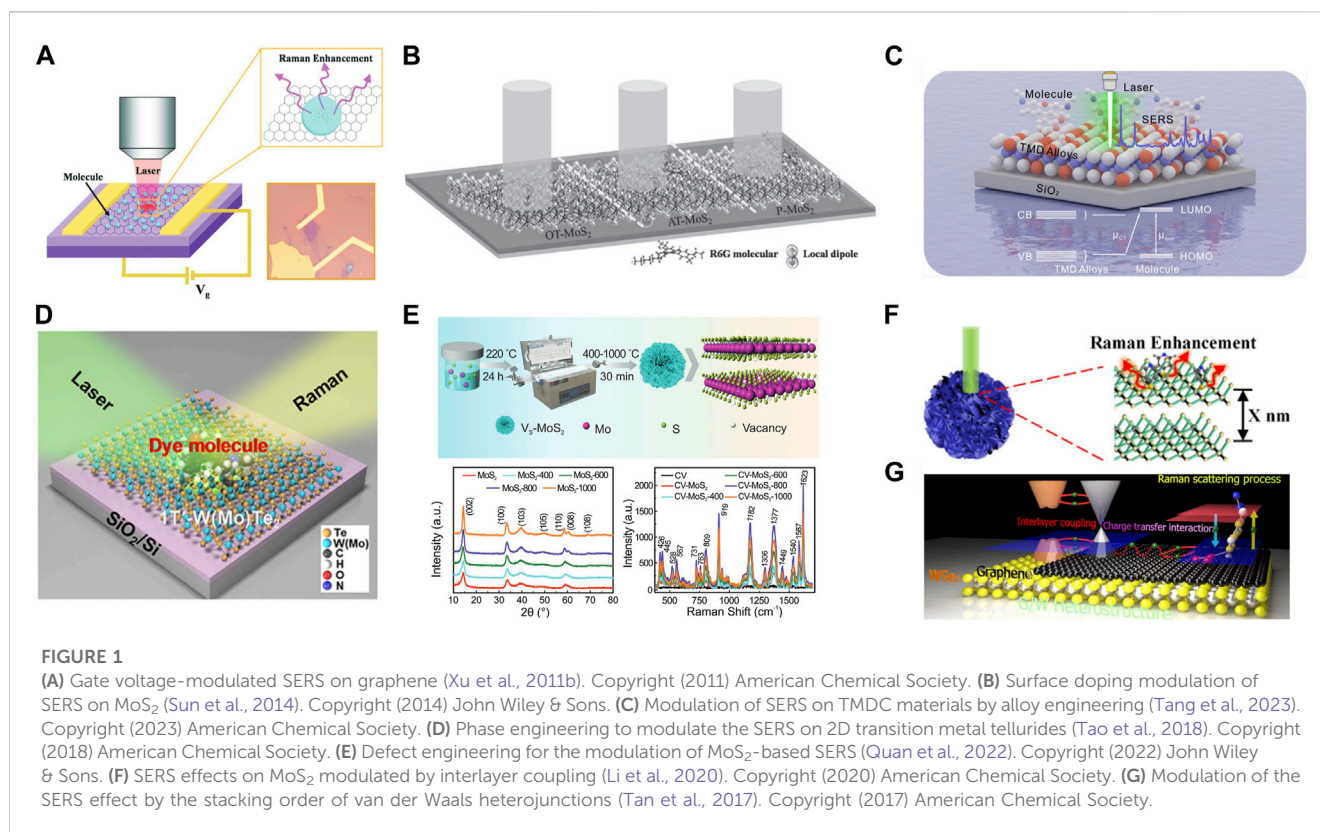
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Introduction

Raman spectroscopy was first discovered in 1928 (Raman and Krishnan, 1928). It relies on the interaction of light with the chemical bonds within a molecule, providing fingerprint information about molecular or lattice vibrations. This non-destructive molecular detection technique finds wide-ranging applications in various fields including chemical analysis, environmental protection, and food processing. However, because of the low Raman scattering cross-sections, the effectiveness of Raman spectroscopy is limited in detecting molecules with very low concentrations. Surface-enhanced Raman scattering (SERS) is a technique that can amplify the Raman signals of molecules on an active substrate, enabling even single-molecule detection. The generation of the SERS effect involves two primary enhancement mechanisms: electromagnetic mechanism (EM) and chemical mechanism (CM) (Campion and Kambhampati, 1998; Langer et al., 2020). The former achieves enhancement by utilizing surface plasma excitations to intensify the electromagnetic field on the SERS-active substrate, thus boosting the Raman signal intensity of molecules on the substrate surface (Schlücker, 2014). The latter relies on interactions between the SERS-active surfaces and molecules, such as charge transfer and exciton resonances. This interaction induces a modification in the electron density of the molecules, leading to heightened molecule polarization and thus amplified Raman scattering (Morton and Jensen, 2009; Lombardi and Birke, 2014).

The theory of EM has been extensively researched and demonstrated. In contrast, the understanding of CM remains largely limited to qualitative descriptions. The challenge lies in the fact that the Raman signal from chemical enhancement tends to be overshadowed by the Raman signal from electromagnetic enhancement, making it difficult to experimentally quantify the CM-induced Raman enhancement. In recent years, two-dimensional (2D) materials, such as graphene, hexagonal boron nitride, and transition metal dichalcogenides



(TMDCs) such as MoS₂, WSe₂, and MoTe₂, have demonstrated excellent SERS effects (Ling et al., 2014; Lee et al., 2016; Chen et al., 2020). Compared to noble metals, 2D materials possess atomic-level smooth surfaces, greater chemical stability, and exhibit stronger interactions with molecules. The primary mechanism by which 2D materials achieve SERS is considered to be CM, offering an ideal platform for understanding the CM mechanism in SERS. Moreover, because of unique properties such as the large surface-to-volume ratio and quantum confinement effect, 2D materials possess highly interface-tunable nature. Through precise modulation of the band structures and energy levels in 2D materials, optimized energy level alignment that facilitates the charge transfer and/or exciton resonances at the molecular-substrate interface can be achieved, culminating in a potent augmentation of the SERS effect. This tunability provides numerous avenues for the development of 2D materials as SERS-active substrates.

Herein, we present a comprehensive summary of recent research on achieving tunable SERS using 2D materials. We extensively discuss the capabilities of 2D material interface engineering in enhancing the performance of 2D materials as SERS-active substrates. This mini review serves as a guiding resource for the advancement of 2D materials as substrates for SERS applications and multifunctional optoelectronic sensing devices.

Electrostatic tuning

The tunable energy levels of 2D materials offer promising prospects for their application as tunable SERS-active

substrates. Notably, monolayer graphene can be effectively manipulated by applying gate voltage to induce hole or electron doping, thus allowing precise control of its Fermi levels (Figure 1A) (Xu et al., 2011b). Negative gate voltage shifts the Fermi level of graphene downward, bringing the energy gap between the graphene Fermi level and the lowest unoccupied molecular orbital (LUMO) of the probe molecule closer to the incident laser excitation (1.96 eV). This significantly enhances the charge transfer resonance between probe molecules (Co-Pc) and the substrate, thereby enhancing the Raman signal of the probe molecules. Conversely, positive gate voltage weakens the charge transfer and leads to signal attenuation. Importantly, this modulation is reversible. In their subsequent work, the authors have further investigated the electrical tuning effects on SERS under different gas atmospheres (Xu et al., 2011a).

Furthermore, a comparative study was conducted between graphene field-effect doping and chemical doping (Hao et al., 2013). Both methods exhibited similar trends in terms of electron and hole doping, with p-doped graphene showing greater SERS enhancement compared to n-doped graphene.

The electrical tuning approach is expected to be suitable for semiconducting 2D materials. For instance, the SERS effect of tungsten oxide thin films can be electrically regulated (Zhou et al., 2019). The modulation of oxygen vacancy defects in WO_{3-x} was realized by applying electric field. The defect energy levels introduced by the oxygen vacancy defects promoted the charge transfer between the substrate and the molecule, thus realizing the SERS enhancement.

Surface doping and modification

In addition to electrical methods, chemical doping can also be used to modulate the Fermi level of 2D materials, thereby influencing its SERS performance. By controlling the chemical doping of graphene with varying concentrations of nitrogen atoms, the Fermi level of graphene can be adjusted, leading to controlled variations in its SERS effect (Feng et al., 2016). Additionally, inducing graphene to react with ozone under ultraviolet radiation introduces oxygen-containing functional groups and p-type doping in graphene (Huh et al., 2011). The p-type doping shifts the Fermi level of graphene downward, increasing charge transfer between graphene and probe molecules (consistent with the electrostatic tuning of graphene-based SERS) (Xu et al., 2011b), thereby enhancing the Raman signal of the probe molecules.

Surface doping of MoS₂ can be achieved through oxygen plasma treatment as shown in Figure 1B. This treatment introduces disorder in the surface structure of MoS₂ (Sun et al., 2014), leading to the generation of localized dipoles and the adsorption of oxygen on the material surface. Oxygen doping on the surface of MoS₂ results in a reduction of the valence band, promoting charge transfer between probe molecules and the substrate, thereby enhancing the Raman signal.

Moreover, through the *in-situ* substitutional doping of WSe₂ with Re and Nb atoms, the SERS detection is greatly enhanced, enabling the molecular sensing at the femtomolar level. By precisely controlling the concentration and ratio of these doping elements, the electronic structure of WSe₂ can be tuned, allowing for the regulation of its SERS effect (Lv et al., 2022).

Alloy engineering

During the material growth process, alloy engineering, an effective method involving elemental composition manipulation, offers a promising avenue for controlling the band structure of materials. By introducing carbon (C) during the synthesis of boron nitride (BN), BCN can be synthesized (Liang et al., 2022). Through meticulous control of the introduced C content, precise modulation of the material's band structure is attainable, leading to enhanced SERS effects for a diverse range of probe dye molecules (e.g., rhodamine 6G, crystal violet, methylene blue, etc.).

Moreover, alloy engineering can be applied to control the sulfur and selenium element ratio in few-layer manganese phosphorus trichalcogenides (MnPS_{3-x}Se_x, 0 ≤ x ≤ 3) to achieve Raman signal modulation (Hou et al., 2020). By appropriately adjusting the element ratio, the material's band structures can be aligned with that of the probe molecules, resulting in a pronounced enhancement of the Raman signal. Subsequent investigations have further demonstrated the universality of this approach (Figure 1C), utilizing alloy engineering to control the band structures of TMDCs materials (Tang et al., 2023). Continuously tuning of energy levels in TMDC alloys (including WS_{2(1-x)}Se_{2x}, MoS_{2(1-x)}Se_{2x} and Mo_{1-x}W_xS₂) is achieved by adjusting the composition ratio, which offers the on-demand design of charge transfer resonances for specific probe molecules, and thus delivers the optimized SERS sensing. The alloy engineering in 2D SERS materials enables the

highly precise modulation of the bandgaps of the alloys and the energy level alignments at the interfaces, showing promises in high-performance SERS sensors for a large variety of target molecules.

Phase engineering

Phase is a unique tunable degree of freedom in 2D materials compared to conventional noble metal SERS materials. When a monolayer of MoX₂ (X = S, Se) undergoes a transition from 2H to 1T phase, a highly efficient charge transfer occurs between the Fermi level of the monolayer substrate and the highest occupied molecular orbital (HOMO) level of the probe molecule, leading to a significant enhancement of the probe molecule's Raman signal (Yin et al., 2017).

Phase engineering for SERS has also been observed in 2D transition metal tellurides, where 1T' phase MoTe₂ exhibits better SERS performance compared to the 2H phase (Figure 1D) (Tao et al., 2018). The semi-metallic 1T' phase not only enables the strong coupling between SERS substrate and the probe molecules, but also facilitates the charge transfer in SERS via a much higher density of states near the Fermi levels. Interestingly, the high surface activity of 1T' phase MoTe₂ facilitates the dichlorination reaction in HAuCl₄ solution, allowing the spontaneous fabrication of high-density gold nanoparticles (AuNPs) on 1T' MoTe₂ (Tao et al., 2021). In contrast, the dichlorination on 2H MoTe₂ only occurs at the defect sites. The strong coupling effects of CM and EM enhancements on the AuNPs-1T' MoTe₂ hybrid structure shows largely boosted SERS sensitivity (4 × 10⁻¹⁷ M limit of detection for rhodamine 6G). In a word, the phase modulation of 2D materials opens up new possibilities for their application as SERS-active substrates.

Incorporating vacancies

Defect engineering in 2D materials can also be utilized to modulate their SERS effects. Introducing oxygen doping into MoS₂, for example, enhances its performance as a SERS substrate (Zheng et al., 2017). Unlike the oxygen adsorption resulting from plasma treatment discussed earlier, oxygen doping induces partial oxidation of the material. Furthermore, their previous work demonstrated that the introduction of oxygen vacancies also effectively enhances Raman signal intensity (Cong et al., 2015). The enhanced Raman signals observed from oxygen doping and oxygen vacancies are attributed to the combined effects of charge transfer, molecular interactions, and exciton resonances.

Additionally, precise control over the concentration of sulfur vacancies in MoS₂ enables the manipulation of its band structure and the degree of internal carrier separation within the material, leading to an enhanced substrate-probe interaction. Consequently, this tunability facilitates the adjustment of the SERS intensity on MoS₂ (Quan et al., 2022), as shown in Figure 1E.

The introduction of defects on the one hand acts as an active site, which is favorable for the adsorption of probe molecules. On the other hand, the introduction of defect energy levels is more favorable to the charge transfer between the substrate and the molecules, leading to the improved SERS effect (Song et al., 2021). Overall, these

findings underscore the significance of defect engineering and its potential in tailoring the SERS properties of 2D materials, enhancing their applicability as versatile substrates for molecular sensing and analysis.

Interface spacing engineering

The unique layered structure of 2D materials grants them distinctive interlayer coupling freedom. In the case of MoS₂ with small interlayer spacing (Figure 1F), it achieves a remarkable SERS enhancement factor of up to 5.31×10^5 (Li et al., 2020). The Raman signal of the probe molecules increases with a reduction in the interlayer spacing. This exceptional enhancement can be primarily ascribed to the efficient charge transfer between the substrate and probe molecules, facilitated by the reduced interlayer spacing in the material.

The interface spacing between 2D SERS materials and the probe molecules also generates considerable impact on the SERS performance. The CM-based SERS intensity can be substantially reduced with increasing substrate-molecule spacing, since the CM is a short-range effect (Ling et al., 2010). Comparative investigations have shown that placing an ultrathin Al₂O₃ dielectric layer (~5 nm) between 2D SERS materials (such as ReS₂ and PdSe₂) and dye molecules can mostly vanish the SERS signals, by efficiently blocking the interface charge transfer (Miao et al., 2018; Lei et al., 2022). This dielectric spacing technique provides a facile method for verifying the CM enhancement in SERS.

Building van der Waals heterostructures

The van der Waals interaction between 2D materials enables the facile stacking of 2D materials to form Lego-like heterostructures. The stacking configuration of different 2D materials also influences their SERS effects. When WSe₂ is stacked with graphene using different configurations, it was found that the graphene/WSe₂ (Figure 1G) stacking configuration produces higher SERS enhancement on probe molecules (Tan et al., 2017). This enhancement is mainly attributed to the increased electronic density of states on the material surface, which arises from the interlayer coupling in the heterostructure.

The interlayer spacing in van der Waals heterostructures plays another important role on the SERS effects. In the investigation of graphene/WSe₂ heterostructures for SERS, a crucial finding reveals that an interlayer spacing of less than 0.6 nm enhances interlayer coupling, resulting in a significant influence of the interlayer spacing variation on the SERS effect (Chen et al., 2023).

Additionally, the surface deposition of ReO_xS_y on graphene has been demonstrated as an effective approach to enhance the SERS sensitivity of graphene. Precise control over the oxygen concentration in ReO_xS_y can modulate the electronic structure of ReO_xS_y, allowing for the modulation of the Raman signal (Seo et al., 2020). The enhanced exciton and charge transfer resonances allows an ultrasensitive limit of detection of SERS to the femtomolar level.

In addition to 2D heterostructures, the fabrication of WO_{3-x}/WSe₂ mixed-dimensional heterostructures through oxygen plasma

treatment on the WSe₂ surface has facilitated the SERS molecular detection at the femtomolar level (Lv et al., 2023). The remarkable SERS sensitivity stems from the effective charge transfer facilitated by the unique structure of WO_{3-x} nanowires, in combination with the efficient interlayer coupling within the heterostructure. Notably, the duration of oxygen plasma treatment can efficiently modulate the SERS signal. The incorporation of mixed-dimensional heterostructures represents a novel avenue for advancing research and development in multifunctional tunable SERS devices.

Concluding remarks

This mini review provides an overview of recent research on tuning the SERS effects of 2D materials through various means, including electrical methods, surface doping, alloy engineering, phase engineering, defect introduction, interlayer coupling modulation, and stacking configurations. By summarizing recent studies on the utilization of 2D materials as tunable SERS-active substrates, we underscore the vast potential of these materials as adaptable SERS substrates. Central to these modulation strategies is the adjustment of the energy levels and band structures of 2D materials. By aligning the energy levels of 2D materials with those of probe molecules, efficient charge transfer between the substrate and the probe molecules is achieved, leading to notable enhancements in the Raman signal of the probe molecules. The large surface-to-volume ratio and flexible tunable properties of 2D materials contribute to their tremendous potential as tunable SERS substrates. In the future, leveraging the tunable properties of 2D materials can lead to the development of multifunctional tunable optoelectronic devices based on these materials. This promising avenue of research has the potential to revolutionize various fields, including molecular sensing, analytical chemistry, and semiconductor devices, paving the way for advanced and adaptable sensing technologies in the future.

Author contributions

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