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Perspective Review

Selectivity and stability reshaping high-sensitivity detection boundaries: A technical leap and paradigm shift in semiconductor surface-enhanced Raman scattering

Jie Lin[3,§] ✉, Xiangyu Meng[14,§], Yujiao Xie[3,§], Yusi Peng[2,§], Xingce Fan[6] ✉, Kun Liang[3] ✉, Fugang Xu[13] ✉, Aiguo Shen[12] ✉, Libin Yang[11] ✉, Lei Chen[10] ✉, Wei Ji[9] ✉, Tingting Zheng[8] ✉, Teng Qiu[6] ✉, Shan Cong[5] ✉, Zhigang Zhao[5] ✉, Xiaotian Wang[4] ✉, Yong Yang[2] ✉, Aiguo Wu[3] ✉, Guangcheng Xi[7] ✉, and Bing Zhao[1] ✉

[1] *State Key Laboratory of Supramolecular Structure and Materials, Jilin University, Changchun 130012, China*
[2] *State Key Laboratory of High-Performance Ceramics and Superfine Microstructures, Shanghai Institute of Ceramics, Chinese Academy of Sciences, Shanghai 200050, China*
[3] *Laboratory of Advanced Theranostic Materials and Technology, Ningbo Institute of Materials Technology and Engineering, Chinese Academy of Sciences, Ningbo 315201, China*
[4] *School of Chemistry, Beihang University, Beijing 100191, China*
[5] *Key Lab of Nanodevices and Applications, Suzhou Institute of Nano-Tech and Nano-Bionics, Chinese Academy of Sciences, Suzhou 215123, China*
[6] *Key Laboratory of Quantum Materials and Devices of Ministry of Education, School of Physics, Southeast University, Nanjing 211189, China*
[7] *Key Laboratory of Consumer Product Quality Safety Inspection and Risk Assessment for State Market Regulation, Chinese Academy of Quality and Inspection & Testing, Beijing 100176, China*
[8] *Shanghai Key Laboratory of Green Chemistry and Chemical Processes, School of Chemistry and Molecular Engineering, East China Normal University, Shanghai 200241, China*
[9] *College of Chemistry, Chemical Engineering and Resource Utilization, Northeast Forestry University, Harbin 150040, China*
[10] *School of Materials Science and Engineering, Jilin Jianzhu University, Changchun 130118, China*
[11] *College of Chemistry and Chemical Engineering, Qiqihar University, Qiqihar 161006, China*
[12] *School of Bioengineering and Health, Wuhan Textile University, Wuhan 430200, China*
[13] *College of Chemistry and Materials, Jiangxi Normal University, Nanchang 330022, China*
[14] *School of Basic Medical Sciences, Hebei University, Baoding 071002, China*
[§] *Jie Lin, Xiangyu Meng, Yujiao Xie, and Yusi Peng contributed equally to this work.*

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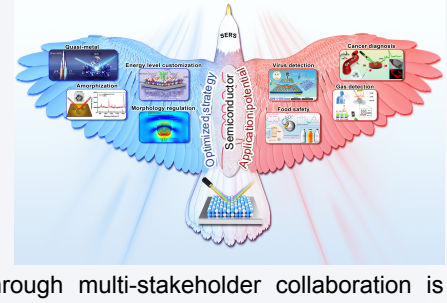
ABSTRACT: This perspective commemorates 50 years of surface-enhanced Raman scattering (SERS) by highlighting the paradigm shift toward rationally designed semiconductor substrates, enabling ultrasensitive and molecule-selective detection. Several enhancement strategies have been developed to effectively modulate the electronic band structure and charge transfer (CT) processes, such as energy level customization, amorphization, quasi-metallization, and morphology control, achieving high enhancement factors with good selectivity and stability. Moreover, semiconductor SERS substrates show broad prospects in the fields of bio-sensing and cancer diagnosis. Nevertheless, standardization gaps in substrate reproducibility and data comparability hinder its widespread adoption. Resolving these challenges through multi-stakeholder collaboration is essential to bridge the technology transfer gap and establish SERS as a core platform for next-generation inspection.



KEYWORDS: surface-enhanced Raman scattering, semiconductor, enhancement strategies, bio-sensing, cancer diagnosis

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✉Address correspondence to Jie Lin, linjie@nimte.ac.cn; Xingce Fan, x_fan@seu.edu.cn; Kun Liang, kliang@nimte.ac.cn; Fugang Xu, fgxu@jxnu.edu.cn; Aiguo Shen, agshen@wtu.edu.cn; Libin Yang, yanglibin@qqhru.edu.cn; Lei Chen, chenlei82@jlju.edu.cn; Wei Ji, jiwei@nefu.edu.cn; Tingting Zheng, ttzheng@chem.ecnu.edu.cn; Teng Qiu, tqiu@seu.edu.cn; Shan Cong, scong2012@sinano.ac.cn; Zhigang Zhao, zgzhao2011@sinano.ac.cn; Xiaotian Wang, wangxt@buaa.edu.cn; Yong Yang, yangyong@mail.sic.ac.cn; Aiguo Wu, aiguo@nimte.ac.cn; Guangcheng Xi, xiguangcheng@caiq.org.cn; Bing Zhao, zhaob@jlu.edu.cn

1 Introduction

Since the discovery of surface-enhanced Raman scattering (SERS) phenomenon in 1974, it has spurred extensive research enthusiasm in the fields of physics, chemistry, biology, and materials science [1, 2]. With the development of nanotechnology, SERS substrates have evolved from rough metal electrodes (Au, Ag, and Cu) to nanostructured materials, including noble metal nanostructures and semiconductors (both inorganic and organic). In the recent two decades, the development of semiconductor SERS systems has reshaped the SERS field by providing an alternative platform to realize dramatic Raman enhancement with intrinsic selective detection behavior towards different molecules [3].

Lombardi et al. proposed the "Unified View" of SERS in 2009, which illustrates the contribution of different resonances to SERS, providing the theoretical foundation for the materials design and development of semiconductor SERS substrates [4]. The SERS activity of semiconductor materials is mainly derived from chemical mechanisms, such as the formation of new surface complexes, chemical bonding, and photo-induced charge transfer (PICT) between substrate and molecules [5–7]. New surface complexes exhibit stronger polarization or visible-range resonances, while chemical bonding to the substrate induces static CT and enlarges the molecular Raman cross-section. When the excitation wavelength is resonant with molecule-semiconductor CT transitions, PICT becomes the dominant chemical enhancement mechanism. In addition, other resonances of the system are also implicated in the Raman enhancement, namely Mie resonance, and molecular and/or exciton resonances. These multi-resonance terms can cooperate to generate dramatic Raman enhancement [8, 9].

Over the past years, semiconductor SERS substrates have evolved from simple exploratory systems into platforms with refined optimization strategies and practical applications (Fig. 1). Several enhancement approaches, including energy level customization, amorphization, quasi-metallization, and morphology control, have been developed to modulate the electronic band structure and CT processes, thereby achieving high enhancement factors, good selectivity, and stability. Benefiting from these advances, semiconductor SERS substrates now exhibit broad application potential in bio-sensing, such as virus detection, food safety monitoring, gas detection, and cancer diagnosis.

On the occasion of the 50th anniversary of the discovery of SERS, we join hands with researchers in this field to present this perspective, aiming to provide a comprehensive view of past achievements, energize ongoing research efforts, and establish a clear roadmap for future development of semiconductor SERS.

2 Enhancement strategies

2.1 Energy level customization

The fixed electronic band structures of SERS materials restrict the energy-level alignment with target molecules and limit PICT efficiency. Customized energy level engineering is essential to achieve efficient PICT resonance and selective SERS detection. Zhao's group proved that defect engineering is an effective way to enhance the SERS activity of semiconductors [10, 11]. The introduction of oxygen vacancy and extrinsic doping modulates the electronic band structure, density of states (DOS), and surface properties of semiconductors, consequently promoting the PICT efficiency. For wide-bandgap semiconductors, their SERS performance can be further optimized by doping with appropriate transition metals, which can generate numerous surface defect states, thereby enhancing the interfacial CT efficiency for SERS. Notably, this regulation method is significantly influenced by both the doping concentration and the ionic radius of transition metals.

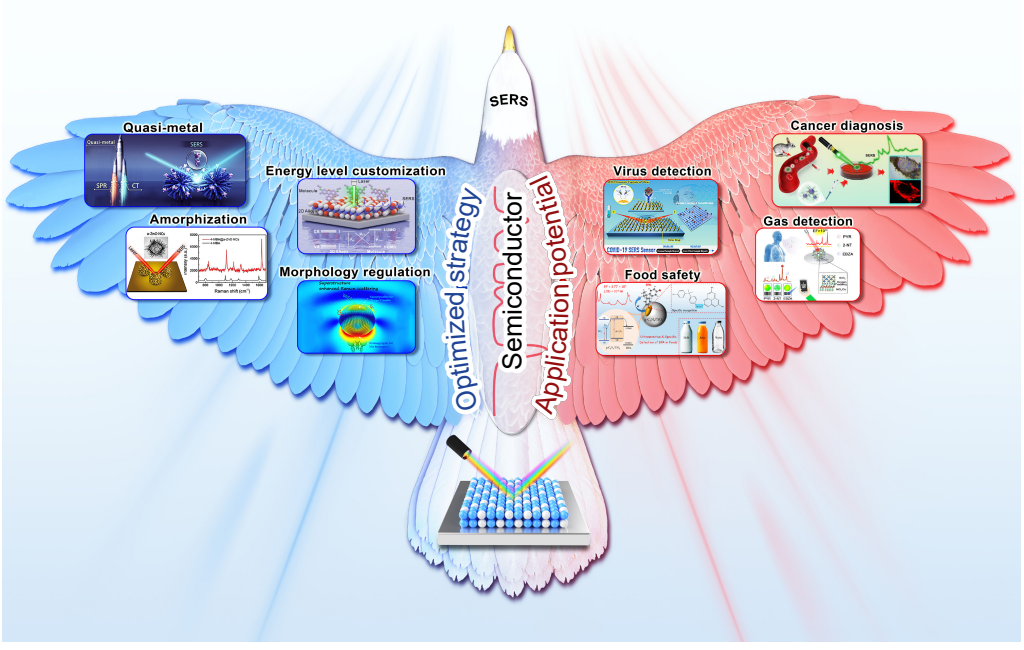

Figure 1 Enhancement strategies and application development of semiconductor SERS substrates. Reproduced with permission from Ref. [5], © Peng, Y. S. et al. 2021. Reproduced with permission from Ref. [12], © American Chemical Society 2023. Reproduced with permission from Ref. [16], © Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim 2017. Reproduced with permission from Ref. [21], © Ye, Y. T. et al. 2020. Reproduced with permission from Ref. [23], © Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim 2019. Reproduced with permission from Ref. [27], © Elsevier Ltd. 2024. Reproduced with permission from Ref. [31], © Wiley-VCH GmbH 2021. Reproduced with permission from Ref. [33], © Lin, J. et al. 2022.

Furthermore, Qiu's group developed an alloy engineering strategy to maximize the SERS intensity of a specific molecule by precisely customizing the band edges and bandgaps of two-dimensional (2D) alloy materials [12]. The band edges of monolayer $WS_{2(1-x)}Se_{2x}$ and $MoS_{2(1-x)}Se_{2x}$ ($0 \le x \le 1$) alloys can be shifted up or down with varying alloy compositions, enabling alignment of molecular energy levels with alloy band edges to boost PICT efficiency. The 2D alloy bandgap can also be tailored by adjusting compositions, allowing the design of exciton resonance term for boosting the PICT efficiency. Meanwhile, they tailored the bandgap of $MnPS_{3-x}Se_x$ ($0 \le x \le 3$) by adjusting compositions, allowing the design of exciton resonance term for boosting the PICT efficiency [13]. Theoretical studies suggest that numerous 2D materials with similar stoichiometries and crystal structures offer versatile options for designing tunable band edges and bandgaps, allowing customized energy level engineering for specific molecular detections.

Except for inorganic semiconductors, the π-conjugated framework of organic semiconductors provides an exceptional platform that allows for nearly limitless structural modifications. Ivy-like organic semiconductor, α,ω-diperfluorohexylquaterthiophene (DFH-4T) films function as metal-free SERS substrates, delivering Raman enhancements of 10^2–10^3 through a resonance CT mechanism between the analyte and the organic semiconductor. Comparative morphology studies and electronic structure calculations confirm that both the energetic alignment of the CT state and the film's textured architecture are essential to the SERS response [14]. In another work, the SERS response arises almost entirely from chemical enhancement driven by CT between poly(3,4-ethylenedioxythiophene):polystyrenesulfonic acid (PEDOT:PSS) and the probe molecule. Thermal annealing converts PEDOT from benzenoid to quinoid structures, increasing π-electron delocalization and improving orbital alignment for CT efficiency. This delocalization strengthens CT resonances, boosts vibrational polarizability, and produces the observed SERS enhancement, independent of electromagnetic effects [15].

2.2 Amorphization

Compared with crystalline semiconductors, the atomic arrangement of amorphous semiconductors is disordered, which brings about a wide variety of surface defect states and endows unique optical properties. Wang et al. proposed that the abundant dangling bonds and extended band tails in the amorphous ZnO structure facilitate the formation of metastable state electrons on the surface, thus promoting the interfacial CT. The effective molecular-semiconductor interactions can even generate π bonds in Zn–S bonds, which can further promote the charge transfer between the substrate and the molecules [16]. Furthermore, the smaller band gap and higher DOS of amorphous TiO_2 effectively enhance the resonant vibrational coupling in the substrate–molecular system, thus presenting higher SERS activity than the crystalline counterpart [17]. These studies lay a theoretical foundation for the development of amorphous semiconductor SERS substrates. Amorphous semiconductors have the following three advantages in the SERS field. Firstly, the abundant surface defect states provide more channels and active sites for charge transfer, which is conducive to the formation of more stable surface complexes, thereby significantly improving the efficiency and stability of charge transfer. Secondly, the long-range disordered structure of amorphous semiconductor materials makes the surface rich in metastable electrons, which significantly reduces the potential barrier for interface charge transfer. Thirdly, the narrow band gap of amorphous semiconductor materials can significantly enhance the energy level coupling in the base molecular system.

2.3 Quasi-metallization

Benefiting from elevated charge carrier density and high DOS near the Fermi level, quasi-metal materials have emerged as SERS platforms due to their synergistic Raman enhancement by plasmonic and CT mechanisms. Xi's group highlighted the potential of certain quasi-metallic metal oxides and nitrides as high-performance and stable SERS substrates. They developed several synthetic routes, including low-temperature, microwave, and molten-salt methods, to make porous or nanosheet forms of transition metal nitrides (TMNs), such as MoN, Mo_2N, WN, VN, and TiN [18–20]. Furthermore, transition metal oxides, such as WO_2, possess a dual enhancement mechanism of electromagnetic field enhancement and chemical enhancement due to the strong surface plasmon resonance (SPR) of the semimetals [21]. Their high-density nanopores create abundant "plasmonic hot spots", while the large surface area provides exceptional molecular adsorption capacity for optimized CT efficiency, demonstrating excellent enhancement through a synergistic effect.

2.4 Morphology regulation

Morphology regulation enhances SERS performance through two approaches: (1) increasing molecular enrichment via surface area modification and (2) achieving Mie resonance. For example, Yang and co-workers developed micrometer-sized spherical SnS_2 structures with a hierarchical "nano-canyon" morphology as SERS substrates. By controlling the growth-driving force, they achieved an extremely low detection limit (LOD) for target molecules. This ultra-high sensitivity came from two effects working together: molecular enrichment from capillary action and charge-transfer-based chemical enhancement [22]. Moreover, Mie resonance can confine free-space light into nanoscale volumes, exhibiting intriguing optical, magnetic, and electric resonance modes within the visible and near-infrared spectral regions. The rational design of micro- and nanometer-scale semiconductor superstructures is feasible for facilitating Mie resonance. Ji et al. induced Mie resonance by designing the superstructure dimensions of ZnO, leading to the contribution of the electric field to the SERS enhancement [23]. Besides, constructing semiconductor microcavities, such as whispering gallery modes (WGM) cavity and Fabry–Pérot (FP) cavity, can also regulate SERS sensitivity of the adsorbed molecules by optimizing the cavity configurations [24, 25].

3 Applications and standardization

3.1 SERS bio-sensing

With advances in semiconductor SERS materials and enhancement mechanisms, detection sensitivity has reached femtomolar levels [26]. Yang and co-workers realized the quantitative detection of bisphenol A (BPA) with low molecular affinity by a graphitic carbon nitride ($g\text{-}C_3N_4$)/TiO_2 heterojunction [27]. Furthermore, to address false epidemic alarms caused by the coronavirus infectiousness, Yang and co-workers developed a two-step SERS diagnostic route based on the SnS_2 microsphere substrates.

Through identifying SERS signals from SARS-CoV-2 spike (S) protein and ribonucleic acid (RNA) by the principal component analysis (PCA) and support vector machine (SVM) models, they accurately identify the non-infectious lysed SARS-CoV-2 virus in actual environments, overcoming limitations of the current polymerase chain reaction (PCR) method [22]. Meanwhile, Zheng and co-workers developed a self-assembled Cu_2O nanoarray, which allows rapid and sensitive detection of SARS-CoV-2 RNA down to 60 copies/mL within 5 min [28].

3.2 SERS cancer diagnosis

Semiconductor SERS substrates enable ultrasensitive detection of cancer biomarkers and circulating tumor cells (CTCs) through molecule-selective enhancement, offering unprecedented specificity for early diagnosis [29, 30]. Zheng and co-workers developed a Cu-doped SnO_2-NiO heterojunction SERS platform, enabling ultrasensitive detection of volatile biomarkers for early-stage lung cancer at ppb levels [31]. Wang et al. reported a highly sensitive non-SPR SERS substrate composed of UiO-66 metal–organic framework (MOF) loaded with copper monatomic species (Cu_1/UiO-66), with a LOD of 10 ppb for benzene and toluene, achieving the best LOD among the reported plasmon-free SERS substrates. Importantly, due to the excellent adsorption performance of Cu_1/UiO-66, rapid detection of volatile organic compounds (VOCs) gases can be achieved within 2 min [32]. Furthermore, the multiple electronic energy levels formed by the Fe^{3+}/Fe^{2+} multivalence in Fe_3O_4 nanoparticles enable effective recognition of CTCs and stable SERS imaging [33]. CTCs detection kits leveraging SERS technology have been commercialized for clinical testing, demonstrating unique selective enhancement advantages with significant potential for precise tumor diagnostics [33].

3.3 SERS standardization

SERS technology has made remarkable progress recently in the fields of detection sensitivity, expansion of application scenarios, and instrument development. However, the lack of a standardized system remains the core bottleneck that hinders its extensive application. International Organization for Standardization (ISO) and the American Society for Testing and Materials (ASTM) have initiated standardization projects for SERS terminology and testing methods. As for the standardization of SERS substrates, the Surface Chemical Analysis Technical Committee (ISO/TC 201) has established the measurement methods and parameter standards for characterizing the performance of SERS substrates. In terms of method calibration, the National Institute of Standards and Technology (NIST) has published a technical report entitled "Reference Materials and Protocols for Quantitative SERS Analysis", which proposed the standardized SERS measurement methods for reference molecules (e.g., 4-mercaptobenzoic acid (4-MBA)) and protocols for data comparability. As for instrument standardization, ASTM has formulated a standard testing guideline for Raman spectrometer performance. As for the standardization pathway, consensus has been reached for establishing interdisciplinary working groups and developing national/industry/group standards.

4 Summary

This perspective marks 50 years of SERS by showcasing the paradigm shift toward tunable semiconductor substrates, whose rational design enables ultrasensitive molecular detection and clinical diagnostics. Nevertheless, some issues for semiconductor SERS should be focused on and solved, including: reproducibility for substrate preparation, quantification standard of signal enhancement, standardization of detection procedure, and data comparability and sharing. Multi-stakeholders' cooperation is the only way to fulfill the wide lab-to-market gap of SERS technology and emerge as the core platform of the next-generation inspection.

Data availability

Not applicable.

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Declaration of competing interest

All the contributing authors report no conflict of interests in this work.

Author contribution statement

J. L., X. Y. M., Y. J. X., Y. S. P., and X. C. F.: Writing – original draft. K. L., F. G. X., A. G. S., L. B. Y., L. C., W. J., T. T. Z., T. Q., S. C., Z. G. Z., Y. Y., A. G. W., G. C. X., and B. Z.: Writing – review & editing. J. L., X. T. W., and X. Y. M.: Funding acquisition. All the authors have approved the final manuscript.

Use of AI statement

None.

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