Research article

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Surface plasmon polariton–enhanced photoluminescence of monolayer MoS₂ on suspended periodic metallic structures

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Abstract: Plasmonic nanostructures have garnered tremendous interest in enhanced light–matter interaction because of their unique capability of extreme field confinement in nanoscale, especially beneficial for boosting the photoluminescence (PL) signals of weak light–matter interaction materials such as transition metal dichalcogenides atomic crystals. Here we report the surface plasmon polariton (SPP)-assisted PL enhancement of MoS₂ monolayer via a suspended periodic metallic (SPM) structure. Without involving metallic nanoparticle–based plasmonic geometries, the SPM structure can enable more than two orders of magnitude PL enhancement. Systematic analysis unravels the underlying physics of the pronounced enhancement to two primary plasmonic effects: concentrated local field of SPP enabled excitation rate

increment (45.2) as well as the quantum yield amplification (5.4 times) by the SPM nanostructure, overwhelming most of the nanoparticle-based geometries reported thus far. Our results provide a powerful way to boost two-dimensional exciton emission by plasmonic effects which may shed light on the on-chip photonic integration of 2D materials.

Keywords: photoluminescence; surface plasmon polariton (SPP); transition metal dichalcogenides (TMDs).

Transition metal dichalcogenides (TMDs) such as molybdenum disulfide (MoS₂) have attracted massive research interest in optics [1, 2] because of their phase transition from indirect to direct band gap as the thickness decreases down to monolayer, beneficial for pronounced photoluminescence (PL) [3]. However, the limited interaction distance (~0.62 nm) makes the overall PL intensity and quantum yield of monolayer MoS₂ far from conventional spontaneous emitters [4, 5]. To boost the PL intensity, potential strategies ranging from optical cavities [6, 7], chemical, or electrical doping [8–10], to mechanical engineering [11], etc., have been proposed to manipulate the exciton emission. Especially, plasmonic nanocavities are regarded as ideal candidates for both PL enhancement and subwavelength integration of light sources because of the unique capability of pronounced local resonances and extreme field concentration at nanoscales [12–15]. Thus far, localized surface plasmons of metallic nanoparticles, gap surface plasmons, and surface plasmon polaritons (SPPs) along structured metallic films have been intensively used to enhance PL of the two-dimensional (2D) exciton systems [16–31]. However, most of the TMD/metal hybrid structures are suffering from limited active material areas with enhanced radiative and nonradiative decay rate [32], which severely hinders the applications of plasmon enhanced PL of TMD monolayers. Here we demonstrate the SPP-enhanced exciton emission of MoS₂ monolayer through a suspended periodic metallic (SPM) structure by visible laser excitations. Thanks to the coherent propagating mode of the SPP that is in resonance with

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the excitation lasers, two orders of magnitude amplification of PL signals are enabled in the experiment, overwhelming most of plasmon-enhanced PL systems thus far [16–31]. The underlying physics of such pronounced PL amplification is theoretically explored, which can be ascribed to the excitation rate increment from the boosted electric field of SPP as well as quantum yield enhancement of monolayer MoS₂ modulated by the suspended metallic nanostructures. Our findings may pave a pathway toward highly efficient atomic layer light source for next-generation on-chip integration optoelectronic devices.

The schematic of the monolayer MoS₂ loaded SPM structure, together with the fabrication process, is illustrated in Figure 1a. Basically, to maximize the PL enhancement of the exciton emission of monolayer MoS₂, there are at least three crucial issues that should be carefully designed. 1) The intrinsic exciton of the transferred MoS₂ monolayer should be as bright as possible without destruction or contamination during the fabrication. 2) The metallic structure should be of ultraflat as well as low optical loss so that the transferred hybrid structure is conformal and of high quality. 3) The metallic structure can provide extra resonance mechanism to enhance the exciton emission rate, for example, the plasmon mode is in resonance with the pump laser. In the experiment, to minimize the destruction and contamination of the exciton system, an ultrathin suspended SiN film on Si was used as the substrate, beneficial for decoupling the transfer process of monolayer MoS₂ and microfabrication of metallic

nanostructures on different sides of the substrate. During the process, a 2D periodic circular hole arrays (radius $R = 85$ nm, period $P = 440$ nm for 633 nm laser) are fabricated using the focused ion beam (FIB) system (strata FIB 201, 30 KeV Ga ions, FEI Company) in a 20-nm-thick suspended SiN film. Then, a 60-nm-thick silver film is sputtered on the lower surface of SiN film. The optically thin silver film combined with the periodic SiN pattern can give rise to high quality of SPP mode, ensuring atomic flatness and low loss. As the final step, the monolayer MoS₂ synthesized by chemical vapor deposition (CVD) is transferred onto the upper surface using a wetting transfer process (see Methods for more details). A representative top view of SEM image of the transferred monolayer MoS₂-loaded SPM structure is depicted in Figure 1b. The crack edge of the monolayer flake can be clearly observed, indicating the successful transfer process.

To further identify the quality of the as-prepared monolayer-loaded SPM structure, we first measured the Raman spectra of the monolayer MoS₂ on the SPM structure as well as on the SiN/Si substrate. MoS₂ monolayer can be clearly identified by the frequency difference between two strong Raman peaks at 384.9 cm⁻¹ (in-plane E_{2g}^1) and 404.6 cm⁻¹ (out-of-plane A_{1g}) being 19.7 cm⁻¹ (Figure 1c) [33, 34]. The frequency difference of all the Raman peaks of the transferred monolayer MoS₂ on the SPM structure is in good accordance with that on the SiN film, which suggests that the MoS₂/SPM hybrid structure is possible of high sample quality [35]. In addition, to precisely identify the wavelength of the SPP mode as designed, we conducted

Figure 1: (a) Scheme of the sample preparation process. (b) Top-view scanning electron micrograph of the hybrid structure with period of 260 nm. That pointed by purple arrow is a crack of monolayer MoS₂. (c) Raman spectra of monolayer MoS₂ on the perforated metallic structure (black solid line) and SiN/Si substrate (red dash line). A characteristic peak of silicon (520.7 cm⁻¹) appears in Raman spectrum on the substrate.

the reflection spectra both experimentally and numerically. Figure 2a shows the measured reflection spectra of monolayer MoS₂ loaded metallic structures of different lateral period, which are well reproduced by the calculated ones (Figure 2b, see Methods for more details). One may find that a distinct reflection dip (pointed by black arrows) almost linearly redshifted with the lateral period, indicating the diffraction wave on SPM structure coupled to the propagating plasmon [36]. To further identify its underlying mechanism, the electric field E_z distributions with $P = 440$ nm were calculated (Figure 2c), unraveling the unique opposite sign on the upper and lower surfaces of the sample, which indicates that it is stemmed from the symmetric (or long range) SPP [37, 38]. It is worth noting there are at least two roles that the SPM structures can play for the PL manipulation. One is fine tuning the excitation wavelength in resonance with the SPP mode of the metallic film to increase the excitation rate. The other is quantum yield amplification of excitons of monolayer MoS₂ originating from the enhanced scattering effect from the metallic structure, in which behind clue can be found in absorption enhancement of excitons (Figure 2, Supplementary material, Note S1).

To demonstrate the plasmon-enhanced A and B exciton emission, we then measured the period dependence of the PL spectra of the monolayer MoS₂ under 532 and 633 nm cw laser excitation, respectively. Two MoS₂-loaded SPM structures ($P = 260$ nm, 440 nm) are fabricated with symmetric SPPs at wavelength 526 and 630 nm, respectively, quite approaching the excitation laser wavelengths. Figure 3a and b illustrate the measure PL spectra with A and B exciton peaks around 677 and 637 nm under

the 532 and 633 nm laser excitations, respectively. It is demonstrated that both PL peaks reach the strongest when the plasmonic mode is resonant with the laser excitation, which clearly reveals that the SPP excited in the SPM structures can efficiently enhance the PL intensity of monolayer MoS₂. In addition, apart from the effect due to resonantly enhancing the excitation fields, a supplementary PL enhancement mechanism can be ascribed to the changed surroundings in the form of a structured silver film (Supplementary material, Note S2). As the reference, the PL of monolayer MoS₂ located on the SiN/Si substrate for both cases are illustrated as well. The PL enhancement factor (EF) can thus be obtained by normalizing the measured plasmon-enhanced PL spectra with the reference ones, respectively, as depicted in the insets of Figure 3. The measured SPP-based EF s of A exciton (677 nm) are ~ 77 and 104 due to the in resonance excitation via 532 and 633 nm pump lasers, respectively. Note that A-exciton peak of the monolayer on the metallic structure has a red shift of about 5 nm with respect to the reference sample probably due to dielectric screening of the SPM structures [39], the measured maximal PL enhancement is a little bit deviating from the A-exciton (677 nm).

To unravel the underlying mechanisms of the PL enhancement, the EF averaged over area of P^2 is used due to the position-dependent plasmonic effects, which is defined as [22, 34, 40]

$$EF = \frac{I_{str}}{I_{sub}} = \frac{|E_{str}|^2}{|E_{sub}|^2} \frac{Q_{str}}{Q_{sub}}, \quad (1)$$

where I_{str} (Q_{str}) and I_{sub} (Q_{sub}) are the PL emission intensity (quantum yield) of monolayer MoS₂ on the perforated metallic

Figure 2: Measured (a) and calculated (b) reflection spectra with different periods. The black dash arrows represent the evolutions of the symmetric surface plasmon polaritons (SPPs) resonant dips of the metallic perforated structures with periods. Blue and red dash line exhibit the absorption of A- and B-exciton of monolayer MoS₂, respectively. Green and red arrows point to the 532 and 633 nm excitation laser positions, respectively. (c) The electric field E_z distributions at wavelength 633 nm for the perforated metallic structures with $P = 440$ nm.

Figure 3: The photoluminescence (PL) spectra of monolayer MoS₂ on the metallic perforated structures with the different periods excited by a 532 nm with power of 0.4 mW (a) and 633 nm (b) laser, respectively. As a reference, the PL spectra of monolayer MoS₂ on the SiN/Si substrate are also plotted in the figures. The corresponding enhancement factors are shown in the insets of (a) and (b), respectively. A Raman peak of Si appearing in PL spectrum on SiN/Si substrate (632.8 nm excitation laser) results in a dip in enhancement factor curve.

structures and SiN/Si substrate, respectively. The quantum yield is defined as the ratio between the radiative decay rate and the total decay rate of excitons, reading as [12, 14, 41]

$$Q = \frac{\Gamma_i + \Gamma_m}{\Gamma_i + \Gamma_m + \gamma_i + \gamma_m}, \quad (2)$$

here Γ_i (γ_i) and Γ_m (γ_m) refer to intrinsic and plasmon-assisted radiative (nonradiative) decay rates, respectively (Supplementary material, Note S3). Equation 1 clearly reveals that the PL EF is determined by both the enhanced electric field factors $|E_{str}|^2/|E_{sub}|^2$ in monolayer MoS₂, and the enhanced quantum yield Q_{str}/Q_{sub} of the exciton emission. Thus, the enhancement of quantum yield can be evaluated by the PL EF and the enhanced electric field. Figure 4 demonstrates the calculated normalized electric field distributions $|E_{abs}|^2/|E_0|^2$ at the 532 nm ($P = 260$ nm) and 633 nm ($P = 440$ nm) wavelengths along the z -axis on the perforated structures (at the center of circular hole) and on the SiN/Si substrate, respectively, where E_{abs} and E_0 are the total electric field and electric field of incident light, respectively. For the metallic perforated structures, the symmetric SPPs are excited by the coupling between the SPPs along the upper and lower surface of metallic film. There are strong electric fields in close proximity of both metallic surfaces. The electric field $|E_{abs}|^2/|E_0|^2$ inside monolayer MoS₂ for the wavelengths of 532 nm ($P = 260$ nm) and 632.8 nm ($P = 440$ nm) are 18.2 and 4.9, respectively. For the SiN/Si substrate, the coherent superposition of the incident wave and the reflected wave of the Si surface results in the standing wave. While the out-of-phase reflected and incident light results in a node with much weaker

amplitude on Si surface. Therefore, the electric field $|E_{abs}|^2/|E_0|^2$ inside monolayer MoS₂ for the wavelengths of 532 and 632.8 nm are 0.3 and 0.27, respectively. Furthermore, owing to a large amount of the induced charges accumulated nearby the nanohole edges, the strong electric field mainly concentrates in the close vicinity of the metallic nanoholes (Figures 4c, d, 5c). It indicates that, only monolayer MoS₂ located on the nanohole can get the maximal PL enhancement.

To further explore the position-dependent plasmon-assisted PL enhancement, we further measured the PL images for 633 nm excited cases, as shown in Figure 5a. One may find that, the PL intensity of both monolayers loaded SPM structures are not uniformly across the sample plane (xy plane), which agree well with the calculated in-plane electric field profile for the resonant excitation wavelength (Figure 5b). Furthermore, one can easily obtain the quantitative spatial dependent plasmonic enhancement by extracting the measured and calculated data of the A exciton from above, as shown in Figure 5c. One can observe that, for the 532 nm ($P = 260$ nm) and 633 nm ($P = 440$ nm) in resonance excitations, the enhanced electric field factors $|E_{str}|^2/|E_{sub}|^2$ in monolayer MoS₂ averaged over the unit cell area are 14.2 and 45.2, whose enhanced quantum yield Q_{str}/Q_{sub} is 5.4 and 2.3, respectively (Supplementary material, Note S4). Note that, as the existence of the discrepancy between patterned structures and measurement uncertainty while there are no better simulation strategies, the measured Q factors could be lower than the simulation ones. Finally, as a more straightforward demonstration, the representative

Figure 4: The total electric field enhancement $|E_{abs}|^2/|E_0|^2$ along z axis in the metallic perforated structures (a) and SiN substrate (b) at wavelengths 532 nm ($P = 260$ nm) and 633 nm ($P = 440$ nm). (c) The electric field E_{abs} distributions at wavelength 633 nm for the SiN/Si substrate and perforated metallic structures with $P = 440$ nm, respectively. (d) The enhanced electric field factors $|E_{str}|^2/|E_{sub}|^2$ curves in monolayer MoS₂ along x-axes at wavelengths 532 (black solid line) and 633 nm (red dash line) in the structures with $P = 260$ and 440 nm, respectively.

Figure 5: The measured A-exciton photoluminescence (PL) intensity mapping (a) and calculated total electric field distributions (b) of the monolayer MoS₂ loaded suspended periodic metallic (SPM) structure with $P = 440$ nm, respectively. (c) The A-exciton PL peak intensity (black) and electric field intensity (red) profiles along the indicated white line in (a). (d) Comparison of PL enhancement factors of different plasmon-based PL enhancement strategies in the past 5 years. Note that all enhancement factors shown in (d) are actual results of experimental measurement (not be normalized by the area of single nanocavity).

plasmon-based PL enhancement structures reported in the past few years are shown in Figure 5d for comparison. It suggests that, our SPM structure surpasses most of the

nanoparticle-based systems because of the SPP enhancement mechanism as well as the much larger effective active material area.

In summary, we experimentally demonstrate two orders of magnitude enhancement of PL intensity of MoS₂ monolayer by precisely designed suspended SPM structure which can enable low loss SPP excitation in resonant with the pump lasers. Theoretical analysis reveals the underlying mechanisms of the plasmonic PL enhancement, which involves both increment of excitation rate stemmed from electric field of SPPs and enhancement of quantum yield modulated by the SPM structure. Most strikingly, more than five times quantum yield enhancement of exciton emission by the SPM structure is evaluated, indicating the much higher radiative decay process amplification with respect to the nonradiative decay process. Our findings may gain a novel insight in the quantitative understanding on the plasmonic PL enhancement and pave the way toward high-performing 2D light sources for on-chip optoelectronic integration.

1 Materials and methods

1.1 Growth of monolayer MoS₂

The monolayer MoS₂ was synthesized by an improved ambient pressure CVD system with three furnaces. The sulfur (S) and molybdenum oxide (MoO₃) powders were utilized as the S and Mo precursor, respectively. Two coaxial quartz tubes were used to separate S from MoO₃ for avoiding cross contamination during the reaction. The S and MoO₃ powers were loaded at the central of furnace 1 and 2 in the outer and inner quartz tube, respectively. The SiO₂ (285 nm)/Si (n-doped) substrate was placed in the upstream of furnace 3 and the distance from the outlet of the inner tube is 12–20 mm. Before the growth, the system was pumped down to 5 Pa and then injected Ar to atmospheric pressure. This process was repeated three times to remove oxygen in the system. The Ar flow rates of inner and outer tube were maintained at 6 sccm and 60 sccm, respectively. The temperature of furnace 1 was raised up to 160 °C in 15 min and was held for 25 min. At the same time, the temperature of furnace 2 and 3 were raised up to 630 °C and 750 °C in 25 min and were held for 15 min, respectively. After the growth, furnace 2 and 3 were opened to rapidly cooled down to room temperature.

1.2 Device fabrication

The device was fabricated on a commercially available TEM grid with suspended silicon nitride (SiN) membrane (thickness of 20 nm). A square circular hole (radius of 85 nm and period of P) array was fabricated using the FIB system (strata FIB 201, FEI Company, 30 KeV Ga ions) in the suspended silicon nitride film. A 60 nm thickness Ag film is sputtered on the bottom side of the SiN film, forming a suspended perforated Ag film (Figure 1a). Then, monolayer MoS₂ was transferred onto the SiN substrate by a wetting transfer process. A layer of PMMA (5%, 996k, Sigma-Aldrich) was spin-coated onto the grown MoS₂ sample at 1500 rpm for 10 s and 4000 rpm for 50 s. The sample was baked on a hot plate at 160 °C for 10 min. Subsequently,

the sample was cut into appropriate size and floated it on the surface of NaOH solutions. After dozens of minutes, the PMMA/MoS₂ film was separated from the SiO₂/Si substrate, and then picked up with a PET sheet and put it in deionized water to clean three times. Finally, the PMMA/MoS₂ film was scoop up with the suspended SiN/Ag structures and was totally dried. To make the sample and substrate bond more firmly and avoid oxidation, the PMMA/MoS₂/SiN needed to be heat at 150 °C for 10 min in the glove box. The sample was put into acetone about 5 min to remove the PMMA.

1.3 Spectroscopy

All the experimental spectra were measured by using a WITec alpha 300R micro-Raman confocal system. In Raman and PL spectra measurement, the 532 and 633 nm CW laser was focused on the sample by a 100× objective lens with numerical aperture of 0.9. The lasers power was controlled at 0.4 mW to avoid laser-induced sample damage. The spot sizes of the lasers are less than 500 nm. The grating groove density of reflection, PL and Raman spectra are 150 and 600 g/mm, respectively. In reflection measurement, an (x-linearly polarization) light from the halogen lamp was incident on the sample by a 50× objective lens with numerical aperture of 0.75. For obtaining the reflectance of the samples, reflection from the Ag mirror was used as a reference. PL intensity mapping was carried out by using the 633 nm laser with power of 0.5 mW. Integration time of every spot is 0.5 s. And the step size is 150 nm (less than half of the structural period). The PL spectra from every spot of the sample can be recorded. The peak intensity is obtained by using Gauss lineshape to fit the A-exciton of PL spectra.

1.4 Simulation

Unit cell with area of P^2 is used to calculate the optical properties of the designed samples. Thicknesses of SiN and silver films with a circular hole of radius 85 nm are set to 20 and 60 nm, respectively. A 0.615-nm-thick monolayer MoS₂ is covered on SiN film. The dielectric constant of SiN and Si are set as 4.21 and 11.67, respectively. The dielectric constant of silver was determined from the experimental data by Palik. The relative permittivity of monolayer MoS₂ was obtained from Li et al. [42]. Background environment is assumed to be air with a permittivity of unity. A linear polarized light along x-axes propagates in the -z direction. The reflection spectra and the electric field distributions were calculated by the frequency-domain finite-element method. Bloch boundary condition was applied to mimic the two-dimensional nature of the geometry, and adaptive tetrahedral meshing was used in the simulations. All the simulations reached proper convergence.

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