Control of Radiative Exciton Recombination by Charge Transfer Induced Surface Dipoles in MoS$_2$ and WS$_2$ Monolayers

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Due to the two dimensional confinement of electrons in a monolayer of 2D materials, the properties of monolayer can be controlled by electrical field formed on the monolayer surface. F$_4$TCNQ was evaporated on MoS$_2$ and WS$_2$ monolayer forming dipoles between strong acceptor, F$_4$TCNQ, and monolayers of MoS$_2$ or WS$_2$. The strong acceptor attracts electrons (charge transfer) and decreases the number of the ionized excitons. Free excitons undergo radiative recombination in both MoS$_2$ and WS$_2$. Moreover, the photoluminescence enhancement is stronger in WS$_2$ where the exciton-phonon coupling is weaker. The theoretical model indicates that the surface dipole controls the radiative exciton recombination and enhances photoluminescence radiation. Deposition of F$_4$TCNQ on the 2D monolayers enables a convenient control of the radiative exciton recombination and leads to the applications of these materials in lasers or LEDs.

When an exciton, a quasiparticle consisting of an electron and a hole bound together by simple Coulomb interaction, recombines, i) photoluminescence occurs in the case of weak exciton-phonon coupling or ii) exciton recombines radiation-less increasing the phonon energy if this coupling is strong. In a monolayer of transition metal dichalcogenides (TMD), or a two dimensional electron gas, excitons can interact with free electrons forming charged excitons also known as trions, quasiparticles composed of two electrons and a hole. Due to the presence of these tightly bound negative trions, the number of non-bounded excitons available for radiative recombination is limited and the photoluminescence is suppressed. In other words, the non-bounded excitons cannot radiative recombine producing photon (light) because they are bonded with free electrons forming trions. Therefore, to increase the photoluminescence, the concentration of trions needs to be reduced. A strong electrical field formed by gate electrode on the two-dimensional (2D) layer of TMD or dipoles on the surface of TMD monolayer can reduce the trion concentration.

In previous studies, a chemical doping method was used to enhance the photoluminescence by interaction of TMD monolayer with acceptor in solvent. Furthermore, some reports studied PL and optical properties controlled by the charge transfer between MoS$_2$ and metal nanoparticle or graphene quantum dots. It was shown that not only the PL intensity has been changed, but also the phase transition in MoS$_2$ monolayer is caused by charge transfer. In this work, we evaporate 2,3,5,6-tetralluoro-7,7,8,8-tetracyanoquinodimethane (F$_4$TCNQ) on TMD monolayer forming dipoles between strong acceptor, F$_4$TCNQ and monolayers of MoS$_2$ or WS$_2$. The strong acceptor attracts electrons (charge transfer) and decreases the number of the ionized excitons. Free excitons undergo radiative recombination in both MoS$_2$ and WS$_2$. Moreover, the photoluminescence enhancement is stronger in WS$_2$ where the exciton-phonon coupling is weaker. No solvent was used, which provide a clean system to compare to theoretical calculations. The theoretical model indicates that the surface dipole is controlling the radiative exciton recombination, which further increases the photoluminescence.

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Results

MoS$_2$ and WS$_2$ monolayer growth and characterization. A triangle monolayer of WS$_2$ and MoS$_2$ was grown with the chemical vapor deposition (CVD) method onto a SiO$_2$/Si wafer. The monolayer growth apparatus is shown in Fig. 1(a). Triangular WS$_2$ and MoS$_2$ were grown at random locations on the substrate. Figure 1(b,d) show the optical images of the WS$_2$ and MoS$_2$, respectively. The thickness of the WS$_2$ and MoS$_2$ was determined by atomic force microscopy (AFM), as shown in Fig. 1(c,e). The AFM images indicate that both the WS$_2$ and MoS$_2$ have a smooth surface. The cross section height of the WS$_2$ and MoS$_2$ is approximately 0.70 nm and 0.76 nm, respectively, which corresponds to the monolayers of WS$_2$ and MoS$_2$.

The monolayer structure of WS$_2$ and MoS$_2$ is further confirmed by the Raman spectrum shown in Fig. 2. The $E_{2g}^1$ and $A_{1g}$ modes of monolayer WS$_2$ are located at approximately 355 and 417 cm$^{-1}$, respectively. With the number of layers increased, the in-plane vibrational $E_{2g}^1$ is slightly red-shifted, and the out-of-plane $A_{1g}$ mode is blue-shifted. The energy difference between the Raman $E_{2g}^1$ and $A_{1g}$ modes increased with the layer number. Thus, the energy difference can be used to identify the number of layers of WS$_2$. The energy difference shown in Fig. 2(a) is 62.5 cm$^{-1}$, which coincides with previous reports for monolayer WS$_2$. The same phenomenon is also observed in monolayer MoS$_2$. The in-plane vibrational $E_{2g}^1$ phonon mode is ~385 cm$^{-1}$, and the out-of-plane $A_{1g}$ mode is ~404 cm$^{-1}$. The energy difference between the two modes is also dependent on the number of layers of MoS$_2$. The energy difference between the two modes is 18.2 cm$^{-1}$, as shown in Fig. 2(b), indicating that the MoS$_2$ is a monolayer.

Photoluminescence intensity after F$_4$TCNQ was deposited onto monolayer MoS$_2$/WS$_2$. Figure 3(a) shows that the PL intensity before and after F$_4$TCNQ was deposited onto monolayer WS$_2$. The PL intensity is approximately fifty times higher after the F$_4$TCNQ deposition. The position of the PL peak of monolayer WS$_2$ is slightly blue-shifted, while the peak shape did not change, as shown in Fig. 3(b). The PL intensity is also increased by approximately ten times after the F$_4$TCNQ is deposited on MoS$_2$ monolayer. The position of the peak is also slightly blue-shifted, but the shape is not changed, as is shown in Fig. 3(c,d).
Discussion

To understand the charge transfer from MoS$_2$/WS$_2$ to F$_4$TCNQ, we performed density functional (DFT) calculations\(^2\) on the model systems shown in Fig. 4. In both the F$_4$TCNQ-doped MoS$_2$ and WS$_2$ cases, electron density depletion (as indicated by the white isosurfaces) were found in the interface regions where the nitrogen atoms in the F$_4$TCNQ molecules are closest to the surface sulfur atoms in MoS$_2$ and WS$_2$, as shown in Fig. 4(a,c), respectively. The electron density depletion in the MoS$_2$ layer is slightly greater than that in the WS$_2$ layer according to the electron density difference plots. An electron density increase (red isosurfaces) is observed on the F$_4$TCNQ molecules in both cases, as shown in Fig. 4(a,c). Charge transfer occurs around the interface regions in both cases. The energy level of F$_4$TCNQ and MoS$_2$/WS$_2$ are shown in Supporting Information - Figure S1. In addition, the barycenters of the holes (white isosurfaces) shown in Fig. 4(b,d) clearly suggest that the holes are close to the MoS$_2$ and WS$_2$ surfaces, indicating the charge transfer from MoS$_2$ or WS$_2$ to F$_4$TCNQ. The charge transfer distance (D$_{CT}$) between MoS$_2$ and F$_4$TCNQ (calculated D$_{CT}$ = 1.299 Å) is shorter than that between WS$_2$ and F$_4$TCNQ (calculated D$_{CT}$ = 1.391 Å). However the charge transfer direction, indicated by arrows on the Fig. 4(b,d) is determined by the orientation of F$_4$TCNQ molecule relative to the surface of the TMD monolayer.

According to our discussion in introduction, the charge transfer between MoS$_2$ or WS$_2$ monolayer and acceptor, F$_4$TCNQ forms dipole layers at interface and reduces the ratio of charged exciton to neutral excitons. Therefore, the photoluminescence (PL) of both materials was enhanced due to the charge transfer.

The experimental results for PL enhancement for both MoS$_2$ and WS$_2$ are similar to the earlier reported photoluminescence of MoS$_2$ and WS$_2$ doped with F$_4$TCNQ from solution\(^2\,^5\). In previous studies\(^2\,^5\), mechanical exfoliated MoS$_2$ and WS$_2$ were used. Mechanical exfoliation is the easiest and the fastest method to obtain monolayers of MoS$_2$ and WS$_2$. However, only a small portion of MoS$_2$ and WS$_2$ crystals are exfoliate to monolayers, leaving a majority of samples as thicker flakes. In this study, we used the chemical vapor deposition to obtain large-area, high-quality monolayers of MoS$_2$ and WS$_2$. Therefore, after the F$_4$TCNQ deposition, our photoluminescence intensity of WS$_2$ and MoS$_2$ is approximately fifty times and ten times higher, respectively. Compared to

Figure 2. Raman spectra of a CVD-grown WS$_2$ monolayer (a) and MoS$_2$ monolayer (b). The inset shows the energy difference between the Raman $E_{1g}$ and $A_{1g}$ modes.
the solution-based chemical doping on MoS$_2$ monolayer$^2$, the PL increases approximately three times. During the vacuum deposition of F$_4$TCNQ on MoS$_2$ monolayer, there is no solvent contamination and interaction between MoS$_2$ and F$_4$TCNQ and therefore the PL increases stronger.

The optical properties of MoS$_2$ and WS$_2$, especially the photoluminescence, are affected by the number of layers. Few-layered MoS$_2$ and WS$_2$ have an indirect band gap and show low photoluminescence, while monolayers of MoS$_2$ and WS$_2$ have a direct band gap and strong photoluminescence. To understand the effects of charge transfer, the photoluminescence peaks, which are due to the direct band gap transition, have been analyzed by fitting them with photoluminescence from trions and photoluminescence from neutral excitons represented by two Lorentzian functions, as shown in Fig. 5. For the all cases studied, the photoluminescence signal can be decomposed as A and B peaks, but the intensity of the B peak is negligible. The A peak can be further decomposed to trion (X$^-\ $) and exciton (X) components. Peak positions from the fitting can be found in Table 1. The exciton binding energy of MoS$_2$ and WS$_2$ (1.85 eV and 1.985 eV) was determined in our work. The trion spectral weight $I_{X^-}/I_{total}$ was also calculated and listed in Table 1.

For both WS$_2$ and MoS$_2$, the trion spectral weight $I_{X^-}/I_{total}$ decreases after charge transfer, as shown in Table 1. This indicates that the charge transfer significantly decreases the concentration of trions by transferring electrons from the trions into acceptors, thereby enhancing the photoluminescence.

Upon the deposition of F$_4$TCNQ on the monolayers, the charge transfer reaches a maximum because the trion spectral weight reaches the saturation region at approximately 0.2. We observe that the peaks for the corresponding X$^-\ $ and X of PL are sharper for WS$_2$ than for MoS$_2$. The wider peak width is associated with a stronger coupling strength or a larger Huang-Rhys factor $S$ for a typical semiconductor, so that we may ascribe the narrower PL peaks for WS$_2$ samples compared to MoS$_2$ as indicative of slightly weaker exciton-phonon coupling. After charge transfer to the F$_4$TCNQ molecules, the peak width change is almost the same. The weaker exciton-phonon scattering of WS$_2$ results in narrower PL peaks with a larger amplitude.

The DFT calculated electron transferred from MoS$_2$ and WS$_2$ to F$_4$TCNQ was 0.271 and 0.237, respectively. These data are in good agreement with the trion spectral weight data (Table 1). Larger amount of charge transferred causes more trions to be dissociated to excitons, thereby leading to a lower trion spectral weight. The surface dipole is formed due to the charge transferred from MoS$_2$/WS$_2$ to F$_4$TCNQ. The amount of transferred charge can control the intensity and position of PL. The adding electrons to or withdrawing electrons from the 2D monolayer decreases or increases (this work) the intensity of PL.

It is worth to notice that a dipoles formed by charge transfer to acceptor deposited directly on 2D semiconductor is comparable to dipoles formed by Helmholtz double layer in a electrolyte double layer transistor (EDLT), where gate is a reference electrode in an ionic organic liquid. In an EDLT, the number of induced charges is in the range of $10^{14}$ 1/cm$^2$. It is almost one order of magnitude larger than the charge induced by the layer of dipoles in our experiment, but two orders of magnitude larger than the charge induced

![Figure 3. PL spectra of monolayer WS$_2$ (a) and monolayer MoS$_2$ (c) before and after F$_4$TCNQ doping. PL peak shift of monolayer WS$_2$ (b) and monolayer MoS$_2$ (d) before and after F$_4$TCNQ doping.](#)
in MoS₂ transistor with 280 nm SiO₂ and gate voltage of −70 V. Such high concentration of charge induced in a monolayer of TMD semiconductors should lead to correlated effects like ferromagnetism or to superconductivity in EDLT MoS₂ system. Additionally acceptor layer deposited on the surface on 2D semiconductors can be considered as a stable gate that doesn’t require additional connector for gate voltage.

**Conclusion**

In summary, triangle monolayer WS₂ and MoS₂ were grown using the chemical vapor deposition (CVD) method. The formation of the monolayers was confirmed by both AFM and Raman spectra. The PL increased after a thin layer of F₄TCNQ was deposited on the surface of the WS₂ and MoS₂ monolayers. The ratio of charged excitons, trions, to neutral excitons decreases due to the charge transfer from monolayer WS₂ and MoS₂ to strong acceptor, F₄TCNQ. The weaker exciton-phonon interaction of WS₂ results in narrower PL peaks with larger amplitudes than in MoS₂, while this interaction is strong. Acceptors or donators deposited on the surface of MoS₂ or WS₂ and also on other 2D monolayers provide an effective mechanism for controlling the electron distribution in such heterojunctions. In this way, it is a convenient method of tuning the optoelectronic properties of 2D materials and leads to the application of these materials in lasers or LEDs.

**Methods**

**Chemicals and materials.** WO₃ (>99.5%), MoO₃ (>99.5%) and sulfur (>99.95%) powders were purchased from Sigma-Aldrich and used without any purification. F₄TCNQ (>99.5%) was purchased from Jilin OLED Materials Tech. Co. Ltd. and purified at 220 °C via physical vapor transport (PVT).

**Preparation of MoS₂ and WS₂ monolayers.** For both the triangular shaped MoS₂ and WS₂ monolayers, we used the same method of chemical vapor deposition (CVD). The growth process for the two materials is almost the same, with the only difference being the precursor. Commercially available SiO₂/Si substrates were used in this study. All the substrates were successively cleaned with acetone, methanol, H₂O₂/H₂SO₄ (1 volume/4 volume) and distilled water in an ultrasonic bath for 5 min and then dried in ambient N₂. First, fine WO₃ or MoO₃ powder was spread on the bottom of the crucible. One piece of SiO₂/Si substrate (1 × 1 cm) was
placed face-down on the crucible, and the crucible was put in the center of the growth furnace. Another small crucible with approximately 50 mg sulfur powder was put in another part of the furnace near the gas input side at a temperature of 200 °C. The furnace was heated to 750 °C at 25 °C/min and then maintained at that temperature for 20 min before naturally being cooled down to room temperature. Argon gas was provided during the whole growth process at 60 sccm.

Table 1. Peak position and width for Lorentzian functions used to fit PL peak A in Fig. 5. (The values in brackets for peak positions were previously reported2,5).

| Sample                  | Peak name | Peak Position (eV) | FWHM (meV) | IX−/I_{total} |
|-------------------------|-----------|--------------------|------------|---------------|
| 1L WS₂ as-prepared      | X trion   | 1.972 (1.96)       | 35         | 0.63          |
|                         | X exciton | 1.985              | 35         |               |
| F₄TCNQ doped 1L WS₂     | X trion   | 1.973 (1.98)       | 42         | 0.25          |
|                         | X exciton | 1.991 (2.02)       | 33         |               |
| 1L MoS₂ as-prepared     | X trion   | 1.83 (1.84)        | 48         | 0.70          |
|                         | X exciton | 1.85 (1.88)        | 40         |               |
| F₄TCNQ doped 1L MoS₂    | X trion   | 1.83 (1.84)        | 70         | 0.20          |
|                         | X exciton | 1.865 (1.88)       | 60         |               |

Figure 5. Fitted PL spectra of monolayer WS₂ (a) before and (b) after F₄TCNQ doping. Fitted PL spectra of monolayer MoS₂ (c) before and after F₄TCNQ doping (d). Lorentzian functions were used to fit the A and B peaks, with A peaks assumed to be composed of trions (X−) and excitons (X).
Preparation of F$_4$TCNQ layers on WS$_2$ and MoS$_2$ monolayers. 2-nm F$_4$TCNQ was deposited on the WS$_2$ and MoS$_2$ monolayers by the evaporation of F$_4$TCNQ in a Tectra mini-coater (Germany) with a deposition rate of 0.1 Ångstrom per second.

Characterization. Photoluminescence was measured at the same area before and after F$_4$TCNQ deposition. Both the laser beams (solid-state laser, 473 nm and Nd:YAG solid-state laser, 532 nm) were collimated and focused through a × 100 objective onto the sample surface. All the spectra were collected using a confocal triple-grating spectrometer (Horiba-JY T64000). Raman spectra were recorded using a Renishaw Raman microscope configured with a charge-coupled device array detector with the excitation laser line of 532 nm. Atomic force microscopy was performed on a Digital Instruments 3100.

Density functional theory calculations. The geometry of the F$_4$TCNQ on the surface of the MoS$_2$/WS$_2$ was optimized using the Dmol$^3$ [25,34] with the dispersion-corrected (OBS) PW91 (GGA) functional at the level of the DNP basis set. The geometry of the models is regarded as converged when the total energy difference is less than $1 \times 10^{-4}$ Ha, the total force difference is less than $4 \times 10^{-3}$ Ha/Å, and the maximum displacement of atoms is less than $5 \times 10^{-3}$ Å during the optimization. The optimized geometries of the models were subsequently fed into the MultiWFN 3.3.7 package [10] to perform single-point energy calculations (with SCF convergence criteria set as $1 \times 10^{-6}$ Ha) at the level of B3LYP/6-31G(d,p) (with Mo and W atoms treated using SDD effective core potentials). To facilitate the charge transfer analysis, the MultiWFN 3.3.7 package was used to calculate the charge transfer based on electron density difference.

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Author Contributions

P.H., X.H., H.J., Z.L. and C.K. conceived the experiments. P.H. and X.H. performed the experiments including monolayers growth, characterization and data analysis. K.D., K.K.Z., X.W. and Q.X. performed the PL measurement. J.Y. performed all theoretical calculations and wrote respective discussions. J.Y. also prepared Fig. 4 and assist fitting of Fig. 5 and preparation of Table 1. P.H., X.H., H.J. and C.K. co-wrote the manuscript. Z.L., H.J. and C.K. supervised the project. All of the authors reviewed the manuscript, discussed the data and gave profound suggestions.

Additional Information

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This article contains an error in Figure 2, where the same image was inadvertently shown in both panel (a) and (b). The correct Figure 2 appears below:
Figure 2.