Abstract: Electronic devices based on two-dimensional (2D) MoS₂ show great promise as future building blocks in electronic circuits due to their outstanding electrical, optical, and mechanical properties. Despite the high importance of doping of these 2D materials for designing field-effect transistors (FETs) and logic circuits, a simple and controllable doping methodology still needs to be developed in order to tailor their device properties. Here, we found a simple and effective chemical doping strategy for MoS₂ monolayers using CuCl₂ solution. The CuCl₂ solution was simply spin-coated on MoS₂ with different concentrations under ambient conditions for effectively p-doping the MoS₂ monolayers. This was systematically analyzed using various spectroscopic measurements using Raman, photoluminescence, and X-ray photoelectron and electrical measurements by observing the change in transfer and output characteristics of MoS₂ FETs before and after CuCl₂ doping, showing effective p-type doping behaviors as observed through the shift of threshold voltages (Vth) and reducing the ON and OFF current level. Our results open the possibility of providing effective and simple doping strategies for 2D materials and other nanomaterials without causing any detrimental damage.

Keywords: MoS₂ monolayer; copper chloride; transition metal chloride; p-type doping; spin coating

1. Introduction

Monolayer transition metal dichalcogenides (TMDCs) have been considered to be the next-generation semiconducting channel materials because of their incredible electronic and mechanical properties that make them suitable for flexible, wearable, and transparent devices [1–5]. In addition, their ideally dangling bond-free surface and atomic thickness show promise for van der Waals integration on various substrates/materials and in reducing short channel effect, thus becoming candidates for the semiconducting channel materials in nano-scaled electronics and optoelectronics devices [2,6–8]. Especially, the field-effect transistors (FETs) composed of TMDC monolayers show high carrier mobility, large On/Off ratio (>10⁸), and low power consumption, which have inspired experimental research in advancing FET performance of these devices [5,9–14].

To implement the TMDC monolayers for practical electronic device applications, the device properties need to be tailored to show the desired output characteristics of electronic devices. One way to achieve the desired device characteristics is through doping [3,15–19]. The doping of 2D materials is recognized to be the key to precisely controlling their fundamental properties, based on the history of the contemporary Si or III–V-based semiconductors. Ion implantation is one of the possible doping techniques. However, this uses high energy and can be detrimental to atomically thin 2D crystals. On the other hand, chemical doping is potentially more advantageous compared to the ion implantation method, as the chemical doping is generally based on a charge transfer by chemical potential of adsorbed organic molecules and leads to less damage in 2D crystal structures [18,20–23]. The chemical doping of 2D TMDCs have been mostly relied on employing self-assemble monolayer (SAM) techniques [3,9], substitutional doping [17,24], and passivation of sulfur vacancy defects [5,25,26]. However, using such techniques, it is difficult to tune the amount...
of doping, and they generally require a controlled doping environment. Therefore, it is required to find a convenient method to modulate electrical/optical properties, as well as the electronic device properties.

Herein, we report a simple and controllable doping method for 2D MoS$_2$ using copper (II) chloride (CuCl$_2$) performed at ambient conditions. In this process, the CuCl$_2$ is dissolved in ethanol, and the solution is simply spin-coated onto 2D MoS$_2$ to effectively modulate charge carrier densities without any damage to MoS$_2$ crystals and their devices. The change in doping was analytically confirmed through Raman, photoluminescence (PL), and X-ray photoelectron spectroscopy (XPS), showing the p-type doping effect on 2D MoS$_2$. We further confirmed the feasibility of this doping process by simply coating the different concentrations of CuCl$_2$ solution onto the back-gated MoS$_2$ transistors, showing effective p-type doping behaviors as observed through the shift of threshold voltages (Vth) and reducing the ON and OFF current levels. These findings pave an important pathway toward modulating 2D materials and devices and designing logic devices based on 2D materials.

2. Materials and Methods

Synthesis of monolayer MoS$_2$: Monolayer MoS$_2$ was synthesized on a SiO$_2$ (300 nm)/Si substrate using the previously reported thermal chemical vapor deposition (CVD) method [27,28]. Here, 0.05 mg of MoO$_3$ precursors were prepared by dissolving the MoO$_3$ powders into ammonium hydroxide (NH$_4$OH) solution and loading the MoO$_3$ onto alumina boats using a micropipette. The SiO$_2$/Si substrate was placed above the alumina boat with the substrate placed faced down. The growth was carried in a 2-inch quartz tube, an alumina boat containing 100 mg of sulfur powder was placed upstream, while the alumina boat containing 0.05 mg of MoO$_3$ and substrate was placed downstream in the middle of the CVD furnace. The growth was carried out at 750 °C for 10 min, and the furnace was naturally cooled down to room temperature. MoS$_2$ crystal sizes around 30–50 µm were obtained from the CVD synthesis.

Fabrication and measurement of MoS$_2$ transistors: The synthesized MoS$_2$ monolayers were transferred onto HfO$_2$/Si substrate using polystyrene (PS) film as the transferring medium. The PS film (M$_W$~192,000) was spin-coated onto MoS$_2$/SiO$_2$/Si substrate and the film was detached during the transfer process while the film was floated on DI water. The detached PS film with MoS$_2$ was dried in air for 2 h and transferred onto HfO$_2$/Si substrate for device fabrication. The source and drain electrode pads were patterned using photolithography, and 5 nm Ti/40 nm Au electrodes were deposited using a thermal evaporator. The devices were annealed at 150 °C for 1 h under vacuum conditions. The electrical properties were measured using semiconductor parameter analyzer (Keithley 4200A-SCS) and MS Tech probe station.

Characterization of MoS$_2$: The Raman and PL measurements were carried out using Alpha 300 R confocal Raman spectroscopy with 532 nm laser. AFM measurement was carried out using XE7 (Park Systems, Suwon, Korea). XPS measurement was performed using NEXSA (Thermofisher Scientific, Waltham, MA, USA).

3. Results

MoS$_2$ monolayers were synthesized on a SiO$_2$ (300 nm)/Si substrate using a chemical vapor deposition (CVD). Figure 1a shows the CVD-grown monolayered MoS$_2$ profiled by atomic force microscopy (AFM) height measurement. The morphology and thickness of the as-grown MoS$_2$ show its thickness around 0.7 nm, confirming the single-layered thickness. For doping of MoS$_2$, CuCl$_2$ was employed in this study as the metal chlorides offer a wide range of doping molecules, have been frequently employed to modulate electrical properties of graphene, and are known to be strong electron acceptors [29–31]. The metal chloride generally acts as a strong electron acceptor due to the high electronegativity of chlorine compared to molybdenum or sulfur [31]. It should be noted that CuCl$_2$ has never been used for doping 2D MoS$_2$. Figure 1b illustrates our simple CuCl$_2$ doping process. CuCl$_2$ was firstly dissolved in ethanol at different molar concentrations (0.5 M and 1 M).
The as-grown MoS$_2$ layer on SiO$_2$ was then placed on a spin coater, and CuCl$_2$ solution was dropped onto the as-grown MoS$_2$, followed by the spin coating at 3000 RPM. The doped MoS$_2$ samples were then dried on a hot plate at a mild temperature below 90 °C. All of the doping processes were performed in ambient conditions.

In order to understand the effect of CuCl$_2$ doping on MoS$_2$ monolayers, we first performed Raman and PL analysis of the pristine MoS$_2$ and CuCl$_2$-doped MoS$_2$ as shown in Figure 2a, b. Figure 2a shows the Raman spectrum of pristine MoS$_2$ (dotted grey line), 0.5 M CuCl$_2$-doped MoS$_2$ (purple line), and 1 M CuCl$_2$-doped MoS$_2$ (magenta). The Raman spectrum of pristine MoS$_2$ shows two characteristic peaks located at around 381 cm$^{-1}$ and 400 cm$^{-1}$, which correspond to the in-plane E$_{12g}$ and out-of-plane A$_{1g}$ vibrational modes, respectively. As the CuCl$_2$ is doped onto MoS$_2$, the Raman peaks of MoS$_2$ were monotonically blue shifted with increasing the CuCl$_2$ doping concentrations. Such a trend of shifting in Raman peaks is a clear signature that carrier concentrations were changed without damaging the crystal structure, and can be understood as a CuCl$_2$-induced p-type doping effect, which is in agreement with the previous studies [9,32]. Figure 2b shows PL spectra measured for the pristine MoS$_2$ and CuCl$_2$-doped MoS$_2$. The pristine MoS$_2$ shows direct bandgap PL emission at around 1.82 eV. As the MoS$_2$ monolayers were doped with CuCl$_2$, the PL intensity was largely increased. It has been widely accepted that the PL intensity of 2D MoS$_2$ monolayer can be due to the reduced trion formation and strongly increased exciton radiative recombination rates through decreasing the carrier concentrations of CuCl$_2$-doped MoS$_2$ monolayers [33]. Therefore, CuCl$_2$ doping of MoS$_2$ monolayer decreases carrier concentrations due to the strong electron accepting nature of CuCl$_2$, which was observed through Raman and PL measurements.

To further confirm the effect of CuCl$_2$ doping and the chemical state of CuCl$_2$ molecules, we performed X-ray photoelectron (XPS) analysis as shown in Figure 3a–d. XPS analysis was performed for both pristine MoS$_2$ and CuCl$_2$-doped MoS$_2$, and we compared any change in the binding energies. Figure 3a, b show the main binding energy of MoS$_2$, Mo 3d and S 2p peaks, and we compared the change of binding energies before and after CuCl$_2$ doping. It is clearly observable that the binding energies of both Mo 3d and S 2p peaks shifted toward lower binding energy by about 0.3 eV. The shift to a lower binding energy in semiconducting MoS$_2$ can be attributed to the shift of Fermi-level energy toward the valence band, which results in the change of binding energies in MoS$_2$, and the results agree with the Raman and PL analysis that show the p-type doping effect of CuCl$_2$ on MoS$_2$. 

**Figure 1.** CuCl$_2$ doping process for MoS$_2$ monolayers. (a) AFM topography image of MoS$_2$ monolayers showing its atomic thickness around 0.7 nm. Scale bar: 5 μm. (b) Schematic illustration of CuCl$_2$ doping process. The as-synthesized MoS$_2$ monolayers on SiO$_2$/Si substrate were placed on a spin coater, and CuCl$_2$ solution was dropped onto the substrate for spin coating at 3000 RPM.
peaks, respectively, which were recorded from the CuCl2-doped MoS2 sample. As shown in Figure 3c,d, Figure 3c and d show the high-resolution XPS peaks of Cu 2p and Cl 2p peaks, respectively, which were recorded from the CuCl2-doped MoS2 sample. As shown

Figure 2. (a) Raman and (b) PL measurements before and after CuCl2 doping. 0.5 M and 1 M CuCl2 solutions were employed for doping. After CuCl2 doping, the Raman spectrum of MoS2 was blue-shifted and PL intensity was largely increased, showing p-type doping effect on MoS2.

Figure 3. X-ray photoelectron spectroscopy (XPS) measurements before and after CuCl2 doping. XPS spectrum of (a) Mo 3d and (b) S 2p before and after CuCl2 doping. The shift of the binding energies to lower energy indicates lowered Fermi level in MoS2. XPS spectrum of (c) Cu 2p and (d) Cl 2p was found in CuCl2 doped MoS2 film, demonstrating the CuCl2 is doped onto MoS2.

XPS analysis on CuCl2-doped MoS2 also showed the chemical state of CuCl2 as shown in Figure 3c,d. Figure 3c and d show the high-resolution XPS peaks of Cu 2p and Cl 2p peaks, respectively, which were recorded from the CuCl2-doped MoS2 sample. As shown
in Figure 3c, it can be seen that the binding energies of Cu 2p are composed of the main characteristic doublet peaks centered at around 953 eV and 933.3 eV, which correspond to Cu 2p1/2 and Cu 2p3/2, respectively, and other satellite peaks [34]. The difference between the two peaks is around 19 eV, which is in good agreement with the value reported in the literature [35]. Cl 2p peaks can be deconvoluted into two main doublets, which are found at 200.6 eV and 198.9 eV and correspond to Cl 2p1/2 and Cl 2p3/2, respectively. The peak difference of the two peaks is around 1.7 eV, which is in good agreement with the value reported in the literature [36]. The XPS results and the presence of Cu 2p and Cl 2p binding energies confirm the presence of CuCl2 and the effective p-type doping on MoS2.

To understand the effect of CuCl2 doping on the electrical properties of FETs based on a 2D MoS2 channel, the electrical properties were measured before and after doping the MoS2 FETs with CuCl2. The FET devices were fabricated on a HfO2/Si substrate using photolithography and metal deposition using a thermal evaporator. Figure 4a shows the schematic description of the doping process of MoS2 FETs. The as-fabricated MoS2 FETs were spin-coated with CuCl2 solution. Figure 4b shows the representative transfer curve, drain-source current, $I_{DS}$, as a function of the gate voltage, $V_G$, which is plotted on a logarithmic scale at the applied drain voltage of $V_{DS} = 0.1$ V. The inset of Figure 4b shows the transfer curve in a linear scale. The as-fabricated MoS2 FET showed n-type transfer characteristics and a large ON/OFF ratio above $10^7$. From the transfer characteristics, we have estimated a field effect mobility using $\mu_{FE} = \frac{L}{W C_{ox}} \frac{dI_{ds}}{dV_{gs}}$, where $L$ is channel length, $W$ is channel width, and $C_{ox}$ is the gate capacitance of 309.9 nF cm$^{-2}$. The field effect mobility was measured to be 12.3 cm$^2$/Vs, which is in good agreement with reported values for a back-gated transistor using CVD-grown MoS2 monolayers. It can be observed from Figure 4b that the CuCl2-doped MoS2 FETs shows a gradual decrease in both ON and OFF current as the CuCl2 is doped onto the MoS2 FETs.

![Figure 4](https://example.com/figure4.png)

**Figure 4.** (a) Schematic description of CuCl2 doping on MoS2 FETs. (b) The transfer characteristics, (c) output characteristics, (d) threshold voltages of MoS2 FETs before and after CuCl2 doping.
Such behavior was also found when an output curve, the drain-source current versus drain-source voltage on a linear scale, was measured as shown in Figure 4c (inset image shows the output curve of 1 M CuCl$_2$ doped MoS$_2$ FET). The output curve of as-fabricated MoS$_2$ FET shows a high ON current and a linearly dependent drain current showing good Ohmic contact between MoS$_2$ and electrodes. As the CuCl$_2$ is doped onto MoS$_2$ FETs, the channel conductance is largely decreased, showing a p-type doping effect of CuCl$_2$ on MoS$_2$, in accordance with transfer curve measured in Figure 4b.

Following the reduced ON current and channel conductance, it was also shown that as CuCl$_2$ was doped onto MoS$_2$ FETs, threshold voltage ($V_{th}$) was shifted towards positive voltages from -2.85 V to -1.45 V and -0.9 V as 0.5 M and 1 M CuCl$_2$ was doped onto MoS$_2$ FETs as shown in Figure 4d. Using the changes in the $V_{th}$, the change in carrier concentrations upon CuCl$_2$ doping can be calculated using the parallel-plate capacitor model \cite{3,9}, $N_{doping} = \frac{C \Delta V_{th}}{e}$, where $C$ is the gate capacitance, $\Delta V_{th}$ is the change in the $V_{th}$ after the CuCl$_2$ is doped onto the device compared to the as-fabricated MoS$_2$ FET, and $e$ is the elementary charge. The amount of doping concentration was estimated to be $2.72 \times 10^{12}$ cm$^{-2}$ and $3.77 \times 10^{12}$ cm$^{-2}$ when 0.5 M CuCl$_2$ and 1 M CuCl$_2$ were doped onto MoS$_2$ FETs, respectively. The electrical analysis of MoS$_2$ FETs before and after CuCl$_2$ doping show that CuCl$_2$ is an effective p-type dopant for MoS$_2$ that can be easily employed using simple spin coating of different concentrations of CuCl$_2$ solution.

4. Conclusions

To conclude, we have demonstrated simple and effective p-type doping on the 2D MoS$_2$ FETs by simply spin coating the device with CuCl$_2$ solution at ambient conditions. The effect of CuCl$_2$ doping was confirmed analytically through Raman, PL, and XPS measurements. The p-type doping on the MoS$_2$ channel showed largely decreased channel conductance and the shift in threshold voltages towards positive gate voltages in back-gated MoS$_2$ transistors. It was also shown that the amount of doping can be simply controlled by the CuCl$_2$ concentrations in a solution containing ethanol. The results and findings present an important pathway towards designing a CMOS circuit based on 2D FETs and other nanomaterials.

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