Effect of Ultraviolet-Ozone Treatment on MoS$_2$ Monolayers: Comparison of Chemical-Vapor-Deposited Polycrystalline Thin Films and Mechanically Exfoliated Single Crystal Flakes

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Abstract

We report the different oxidation behavior between polycrystalline chemical-vapor-deposited and mechanically exfoliated single crystal MoS$_2$ monolayers by ultraviolet-ozone treatment. As ultraviolet-ozone treatment time increased from 0 to 5 min, photoluminescence emission and Raman modes of both MoS$_2$ disappeared, suggesting structural degradation by oxidation. Analysis with optical absorbance and X-ray photoelectron spectroscopy suggested the formation of MoO$_3$ in both MoS$_2$ after ultraviolet-ozone treatment. In addition, ultraviolet-ozone treatment possibly led to the formation of oxygen vacancies, molybdenum oxysulfide, or molybdenum sulfates in chemical-vapor-deposited MoS$_2$. The measurement of electrical resistance after ultraviolet-ozone treatment suggested the transformation of chemical-vapor-deposited MoS$_2$ into doped MoO$_3$ and of mechanically exfoliated MoS$_2$ into negligibly doped MoO$_3$. These results demonstrate that the crystallinity of monolayer MoS$_2$ can strongly influence the effect of ultraviolet-ozone treatment, providing important implications on the device integration of MoS$_2$ and other two-dimensional semiconductors.

Keywords: MoS$_2$, Monolayer, Single crystal, Polycrystalline, UV-O$_3$ treatment

Introduction

There is a great interest in transition metal dichalcogenides (TMDs), such as MoS$_2$, since they offer an attractive possibility for various device applications including transistors, optoelectronic devices, heterojunction structures, sensors, and electrocatalysis [1, 2]. The existence of direct bandgaps in monolayer TMDs makes these two-dimensional semiconductors especially promising for optoelectronic devices [3, 4]. However, critical challenges to fabricate TMD-based optoelectronic devices such as phototransistors include the deposition of high-$k$ dielectrics on TMDs and the doping of TMDs. Because of the absence of dangling bonds on the surface of TMDs, it is challenging to deposit high-$k$ dielectrics on TMDs [5]. Moreover, the doping of TMDs is also challenging as the substitutional doping used for bulk semiconductors such as silicon modifies the two-dimensional structure and properties of monolayer TMDs [6].

To overcome these difficulties, surface functionalization of TMDs by O$_2$ plasma [7, 8] or ultraviolet-ozone (UV-O$_3$) [9–11] has been suggested. While these methods can functionalize the surface of MoS$_2$ by surface oxidation, they can simultaneously influence the structure and properties of monolayer MoS$_2$ [12–16]. For example, oxidation by O$_2$ plasma or UV-O$_3$ treatment altered the Raman vibration modes and photoluminescence (PL) emission of monolayer MoS$_2$ [12, 16]. However, as most studies were based on micrometer-scale monolayer MoS$_2$ flakes obtained by mechanical exfoliation from bulk single crystals, little has been known about their interaction with large-area monolayer MoS$_2$ thin films, which are typically polycrystalline. Grain boundaries in polycrystalline monolayer...
MoS₂ may allow higher reactivity with UV-O₃ than that of single crystal, resulting in different oxidation behavior. Therefore, in this study, we explore the effect of UV-O₃ treatment on MoS₂ monolayers by directly comparing the oxidation behavior of polycrystalline chemical vapor deposition (CVD) thin films and mechanically exfoliated single crystal flakes. We systematically investigate the PL and Raman spectra of both MoS₂ monolayers for different duration of UV-O₃ exposure. We also investigate the oxidation behavior of both MoS₂ monolayers during UV-O₃ treatment with X-ray photoelectron spectroscopy (XPS). We further measure electrical resistance of pristine and UV-O₃-treated MoS₂ monolayers to understand the effect of UV-O₃ treatment on MoS₂ monolayers.

**Methods**

Monolayer MoS₂ thin films were deposited on (0001)-oriented sapphire substrates (~1.5 × 1 cm²) by CVD in a two-zone tube furnace. MoO₃ (99.98%, Sigma-Aldrich) and S (99.98%, Sigma-Aldrich) powders in two separate Al₂O₃ boats were used as precursors. MoO₃ powder (14 mg) was placed upstream at zone 1 (750 °C) and S powder (1.4 g) was placed at the upstream entry of the furnace. Substrates were placed downstream at zone 2 (700 °C). MoO₃ powder was heated at a rate of 15 °C min⁻¹ and substrates were heated at 38 °C min⁻¹. After 30-min deposition, the furnace was slowly cooled down to room temperature. Ar flow of 100 sccm and a pressure of ~0.5 Torr were maintained during deposition. Monolayer MoS₂ flakes were obtained by the gold-mediated exfoliation method [17] from bulk MoS₂ crystals (2D Semiconductors) and transferred on highly-doped Si substrates with thermally grown SiO₂ (300 nm). Figure 1 shows schematic structures of both MoS₂ monolayers on substrates. The thickness of monolayer MoS₂ was measured using atomic force microscopy (AFM, Park Systems XE-100). The crystallinity of bulk MoS₂ crystals and CVD MoS₂ thin films was investigated by X-ray diffraction (XRD, Bruker D8 Discover with Cu-Kα radiation) and transmission electron microscopy (TEM, FEI Titan 80–300 at 300 kV), respectively.

MoS₂ monolayers were exposed to UV-O₃ (SEN LIGHTS PL16–110, 185 nm and 254 nm) for 0–5 min at the irradiance of 58 mW cm⁻². Optical absorbance was measured by UV-visible spectroscopy (PerkinElmer Lambda 35). Raman/PL spectroscopy (Horiba Jobin-Yvon LabRam Aramis) were measured on pristine and UV-O₃-treated MoS₂ monolayers with a 532-nm laser and a beam power of 0.5 mW. XPS (Thermo Scientific K-Alpha) was carried out using a monochromatic Al Kα x-ray source (hv = 1486.7 eV) with a take-off angle of 45°, a pass energy of 40 eV, and a spot size of 400 μm in diameter. For all samples, C 1s and O 1s were observed presumably because they are exposed to atmosphere before loaded to ultrahigh vacuum chamber for XPS analysis. Adventitious carbon (C 1s at 284.8 eV) was used as a charge correction reference for XPS spectra. The energy resolution is 0.7 eV measured using the full width at half-maximum intensity of the Ag 3d₅/₂ peak. MoS₂ samples were exposed to atmosphere while they were brought to XPS equipment. Although in situ XPS analysis could provide more accurate information, it was unavailable in this work. For peak deconvolution and background subtraction, Thermo Scientific Avantage Data System software was used. Gaussian functions were used to fit XPS spectra.

To measure the electrical resistance of MoS₂ monolayers, Au contacts (100 × 100 μm², 70 nm thick) were deposited on top of MoS₂ by electron-beam evaporation. Spin-coated photoresist on top of Au layer was then patterned by conventional photolithography to form opening areas for subsequent etching. After Au in opening areas was removed by wet etching in aqua regia, remaining photoresist was removed in acetone. Then, the devices were annealed at 200 °C for 2 h in a tube furnace (100 sccm Ar and 10 sccm H₂) to remove photoresist residue and to decrease contact resistance. Electrical resistance was calculated with current-voltage (I–V) measurement (Keithley 4200-SCS) in atmospheric environments.
Results and Discussion
Beside AFM measurement, PL and Raman spectra are measured to confirm the formation of MoS\(_2\) monolayers. Because of its direct bandgap, MoS\(_2\) monolayers allow PL emission at \(\sim 1.88\) eV \([3, 4]\). In addition, the frequency difference between the two characteristic Raman \(A_{1g}\) and \(E_{12g}\) modes of MoS\(_2\) monolayers is less than 20 cm\(^{-1}\) \([18]\). In Fig. 3, the PL emission of pristine MoS\(_2\) at \(\sim 1.88\) eV indicates that both MoS\(_2\) are monolayers. In Fig. 4, pristine MoS\(_2\) exhibits the frequency difference between 19.6 and 19.9 cm\(^{-1}\) implying monolayer MoS\(_2\). XRD and TEM analysis indicated the single crystal nature of bulk MoS\(_2\) crystals and polycrystalline nature of our monolayer MoS\(_2\) thin films (Additional file 1: Figure S1). The grain size of monolayer MoS\(_2\) thin films is \(\sim 10\) nm \([19]\).

After UV-O\(_3\) treatment, MoS\(_2\) monolayers change their color and become transparent. In Fig. 2a, b, both exfoliated flakes and CVD thin films become transparent after 5-min UV-O\(_3\) treatment. The absorbance spectrum of MoS\(_2\) thin films in Fig. 2c clearly shows the difference after UV-O\(_3\) treatment. (The absorbance of exfoliated MoS\(_2\) flakes could not be measured with UV-visible spectroscopy as the size of flakes was too small.) While pristine MoS\(_2\) thin films show absorbance peaks due to excitonic transitions (A and B) \([3, 4]\), 5-min UV-O\(_3\)-treated MoS\(_2\) thin films do not exhibit any absorbance peaks at all for the same range of wavelength. Because lightly yellow-green MoS\(_2\) thin films become transparent to visible light after 5-min UV-O\(_3\) treatment, we expect the energy bandgap of pristine monolayer MoS\(_2\) (~1.88 eV) to become wider after UV-O\(_3\) treatment (> ~3 eV). As this is in good agreement with the wide bandgap of MoO\(_3\) (>2.7 eV) \([20]\), the transparent UV-O\(_3\)-treated MoS\(_2\) suggests the formation of MoO\(_3\) after 5-min UV-O\(_3\) treatment.

We then investigate the effect of UV-O\(_3\) treatment on the PL emission of MoS\(_2\) monolayers. Figure 3 shows the PL spectra of CVD MoS\(_2\) thin films and exfoliated MoS\(_2\) single crystal flakes after UV-O\(_3\) exposure for 0, 1,
3, and 5 min, respectively. The intensity of PL emission decreases significantly with UV-O₃ treatment time and eventually PL is fully quenched for the 5-min-treated MoS₂ monolayers. These results suggest the formation of oxides or defects allowing non-radiative recombination after UV-O₃ treatment. As MoS₂ monolayers become transparent after UV-O₃ treatment, the formation of wide bandgap semiconductor MoO₃ is reasonably expected. The energy of PL emission of pristine MoS₂ is 1.88 eV for exfoliated flakes and 1.86 eV for CVD films. This slight difference is probably due to the effect of underlying substrates as substrates can strongly influence the Raman and PL emission [21]. The wider width of PL emission peak in CVD monolayers suggests higher defect density. Interestingly, further negative shift of PL emission peak is observed in single crystal MoS₂ flakes (~50 meV) than in CVD thin films (by ~10 meV) after UV-O₃ treatment. As the negative shift of PL emission is comparable with trion binding energy (10−40 meV) of MoS₂ [22], this may be due to different concentrations of trion (neutral excitons accepting an electron or a hole) formed by oxidation-induced doping [23, 24]. (In this work, single crystal MoS₂ flake is more conductive than CVD MoS₂, suggesting higher doping levels in single crystal MoS₂.) The higher doping level in single crystal MoS₂ flakes will allow high concentration of trions, of which recombination will dominate their PL emission. In contrast, the lower doping level in CVD MoS₂ thin films will allow low concentration of trions. Hence, their PL emission will be dominated by the recombination of neutral excitons. However, as the negative shift of PL emission may also be related to the effect of underlying substrates or strains, more systematic investigation is needed in the future.

Next, to investigate the structural degradation by UV-O₃ treatment, we measure the Raman spectra of MoS₂ monolayers after UV-O₃ treatment for 0, 1, 3, and 5 min, respectively (Fig. 4). The intensity of both E₁²g and A₁₈ modes decreases as the treatment time increases. While the frequency difference between E₁²g and A₁₈ modes remains unchanged for 0–5 min of UV-O₃ treatment time, the two Raman modes almost completely disappear after 5-min treatment, suggesting severe structural distortion and degradation. AFM analysis indicates an increase of surface roughness after UV-O₃ treatment (Additional file 1: Figure S2), which is consistent with the oxidation of MoS₂ [23].

To further investigate the structural degradation of MoS₂ monolayers by UV-O₃ treatment, we measure XPS spectra of MoS₂. Because the beam size of XPS is much larger than the size of single-layer MoS₂ flakes, XPS spectra for single crystal MoS₂ flakes are obtained from large-area MoS₂ single crystals (~1 cm in size and ~100 μm in thickness). Figure 5 shows the XPS spectra in Mo 3d and S 2p regions for bulk single crystal and CVD MoS₂ thin films, respectively. The existence of Mo⁴⁺ state of pristine MoS₂ can be observed from the binding energy of Mo 3d₃/₂ and Mo 3d₅/₂ orbitals. After UV-O₃ exposure, the intensity of Mo⁶⁺ state at 235.9 eV further increases with UV-O₃ treatment time indicating the expanded formation of Mo-O bonding and MoO₃. There are four distinct differences between Fig. 5a and b in Mo 3d region. (1) In Fig. 5b, Mo⁶⁺ state at 235.9 eV in pristine MoS₂ thin films is probably due to residual oxide formed during or after CVD process. (2) The intensity of Mo⁴⁺ and S 2s peaks decrease in CVD MoS₂ thin films with longer UV-O₃ exposure. However, the intensity of Mo⁴⁺ and S 2s peaks does not change with UV-O₃ treatment time in large MoS₂ single crystals as XPS can still detect Mo⁴⁺ and S 2s peaks from MoS₂ underneath the oxidized top surface. (3) In single crystal MoS₂, the binding energy of Mo⁴⁺ state shows further positive shift than that in CVD MoS₂ thin films.
suggesting higher n-type doping [25]. The peak shift after the oxidation of MoS$_2$ in this work (0.41–1.09 eV) is comparable to that in literature (0.6–1.1 eV) [23, 24]. (To prevent charging effect, which may induce similar positive shift, we used a flood gun during XPS measurement.) (4) In CVD MoS$_2$ thin films, the peaks of Mo$^{5+}$-state also appear with UV-O$_3$ treatment suggesting possibly the formation of oxygen vacancies [26] or molybdenum oxysulfide MoO$_x$S$_y$ [27]. These results can be understood by the oxidation of Mo$^{4+}$-state in MoS$_2$ into higher oxidation states (Mo$^{5+}$ and Mo$^{6+}$) with UV-O$_3$ exposure. This is also consistent with the XPS results on polycrystalline multilayer MoS$_2$ thin films after O$_2$ plasma or UV-O$_3$ treatment [26, 28, 29].

In S 2p region, the existence of S$^{2-}$-state can be observed from the binding energy of S 2p$_{1/2}$ and S 2p$_{3/2}$ orbitals in pristine MoS$_2$. The binding energy of S$^{2-}$-state in single crystal MoS$_2$ shows further positive shift than that in CVD MoS$_2$ thin films suggesting higher n-type doping [25]. Although S-O bond is observed at ~
165 eV in UV-O₃-treated single crystal MoS₂, it is below the detection limit in CVD thin films. Instead, a new doublet peak of sulfur oxidation state appears at higher binding energy (~169 eV) in CVD thin films after UV-O₃ treatment for 3 min. This new doublet corresponds to the S 2p peaks of oxidized sulfur S⁶⁺, suggesting possibly the formation of various molybdenum sulfates Mo (SO₄)ₓ [28]. While the intensity of S²⁻ doublet keeps decreasing with longer UV-O₃ exposure, the intensity of S⁶⁺ doublet further increases after 5-min UV-O₃ treatment, suggesting further conversion of S²⁻ into higher oxidation state (S⁶⁺) by oxidation. Similarly with Mo⁴⁺ peaks, the intensity of S²⁻ peaks does not change with UV-O₃ treatment time in large MoS₂ single crystals. The existence of S⁶⁺ state after O₂ plasma or UV-O₃ treatment is inconsistent in literature. Its existence was reported in polycrystalline multilayer MoS₂ thin films after O₂ plasma treatment [28]. However, it was not observed in other polycrystalline multilayer MoS₂ thin films [26, 29] or single crystals [9, 16, 30] after O₂ plasma or UV-O₃ treatment. While this inconsistency may be related to dose- and time-dependence of MoS₂ oxidation [30], more systematic investigation is needed to clarify this in the future.

The different XPS behavior may be related to the difference of composition and crystallinity between single crystals and CVD thin films. The composition of Mo:S is 1:1.97 in bulk single crystals and 1:1.5 in CVD thin films, suggesting higher concentration of S vacancies in CVD thin films. The higher concentration of S vacancies, combined with the existence of grain boundaries in CVD thin films, may allow higher reactivity to oxygen than that in single crystals.

To further understand the oxidation of MoS₂ monolayers by UV-O₃ treatment, we measure the electrical resistance of pristine and UV-O₃-treated MoS₂ monolayers. Because there is sample-to-sample variation of electrical resistance, we use relative ratio of electrical resistance \( \frac{R_{\text{After}}}{R_{\text{Before}}} \), where \( R_{\text{After}} \) and \( R_{\text{Before}} \) are electrical resistance after and before UV-O₃ treatment, respectively. Figure 6 shows \( \frac{R_{\text{After}}}{R_{\text{Before}}} \) as a function of UV-O₃ treatment time. While \( \frac{R_{\text{After}}}{R_{\text{Before}}} \) of exfoliated MoS₂ single crystal flakes significantly increases with longer treatment time, \( \frac{R_{\text{After}}}{R_{\text{Before}}} \) of CVD MoS₂ thin films decreases with longer treatment time. These results suggest that MoO₃ formed by the UV-O₃ treatment of CVD MoS₂ thin films possesses higher doping level than that of MoS₂ single crystal flakes. This is supported by XPS analysis suggesting the possible existence of oxygen vacancies, MoO₃Sₓ, or Mo (SO₄)ₓ in CVD MoS₂ monolayers. This is seemingly contradicting with the higher doping in single crystal MoS₂ suggested in Fig. 5a. However, as Fig. 5a is based on bulk single crystals, we cannot exclude the possibility that it does not provide accurate information of the top monolayer. Hence, surface oxidation of bulk MoS₂ single crystal may possibly provide doping only to MoS₂ single crystal underneath, transforming top surface region into negligibly doped MoO₃. Consistent with these results, electrical resistance also increased when monolayer

![Fig. 6 Ratio of electrical resistance of MoS₂ monolayers as a function of UV-O₃ treatment time (R_{\text{After}}, electrical resistance after UV-O₃ treatment, R_{\text{Before}}, electrical resistance before UV-O₃ treatment)](image)
MoS2 single crystal flakes were oxidized by O2 plasma [12]. As single crystal MoS2 without grain boundaries could be more tolerant to oxidation than polycrystalline MoS2, the effect of oxidation-induced doping may be stronger in polycrystalline MoS2 than in single crystal MoS2. However, further investigation is needed to understand this difference in the future.

Conclusions
In summary, we investigated the effect of UV-O3 treatment on polycrystalline CVD thin films and single crystal flakes of monolayer MoS2. Monolayer MoS2 becomes transparent after UV-O3 treatment, suggesting the formation of wide bandgap semiconductor MoO3. As UV-O3 treatment time increases, the intensity of PL and Raman spectra significantly decreased, suggesting the formation of oxides or defects. In both MoS2, XPS analysis indicated the formation of Mo-O bonds and MoO3. However, in CVD MoS2 thin films, the conversion of MoO4− and S2−-states into MoO5+ and S6−-states was also observed after UV-O3 treatment, suggesting the possible existence of oxygen vacancies, MoO2Sx, or Mo(SO4)x. As the electrical resistance of single crystal MoS2 monolayers significantly increased with longer UV-O3 treatment time, the oxidation of single crystal MoS2 into MoO3 seems to provide negligible doping. In contrast, the electrical resistance of CVD MoS2 monolayers decreased with longer UV-O3 treatment time, suggesting that the oxidation of CVD MoS2 into MoO3 provides doping. These results demonstrate the significant impact of crystallinity on the effect of UV-O3 treatment on MoS2 monolayers, providing possibly interesting implications on fabricating heterojunction structures based on two-dimensional nanomaterials.

Additional File

Additional file 1: Crystallinity and AFM analysis. (DOCX 13198 kb)

Abbreviations
CVD: Chemical vapor deposition; I-V: Current-voltage; Rres: Resistance measured after ultraviolet-ozone treatment; Rref: Resistance measured before ultraviolet-ozone treatment; TMDCs: Transition metal dichalcogenides; UV-O3: Ultraviolet-ozone; XPS: X-ray photoelectron spectroscopy

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Authors’ Contributions
CJ and HY contributed equally to this work. WC initiated the research. CJ worked on the growth and characterization of CVD thin films. HY worked on the fabrication and characterization of mechanically-exfoliated single-layer flakes. WC wrote the manuscript. All authors read and approved the manuscript.

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Availability of Data and Materials
All data and materials may be provided on a reasonable request.

Competing Interests
The authors declare that they have no competing interests.

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