Investigation of Nitridation on the Band Alignment at MoS$_2$/HfO$_2$ Interfaces

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Abstract

The effect of nitridation treatment on the band alignment between few-layer MoS$_2$ and HfO$_2$ has been investigated by X-ray photoelectron spectroscopy. The valence (conduction) band offsets of MoS$_2$/HfO$_2$ with and without nitridation treatment were determined to be 2.09 ± 0.1 (2.41 ± 0.1) and 2.34 ± 0.1 (2.16 ± 0.1) eV, respectively. The tunable band alignment could be attributed to the Mo-N bonding formation and surface band bending for HfO$_2$ triggered by nitridation. This study on the energy band engineering of MoS$_2$/HfO$_2$ heterojunctions may also be extended to other high-k dielectrics for integrating with two-dimensional materials to design and optimize their electronic devices.

Keywords: Nitridation treatment, Band alignment, Few-layer MoS$_2$

Background

Currently, layered transition metal dichalcogenides (TMDCs) have aroused great interest due to their fascinating properties for potential applications in modern electronics and optoelectronics [1, 2]. In particular, molybdenum disulfide (MoS$_2$) has been attracting considerable attention as a promising channel material for continuing the scaling beyond the 7-nm technology node [3, 4]. Structurally, the MoS$_2$ crystal is built up of one hexagonally arranged Mo plane, sandwiched by two hexagonally arranged S planes. A triangular prismatic arrangement was formed via the covalently bonded S-Mo-S units [5, 6]. MoS$_2$ possesses a layer-dependent bandgap, varying from a direct bandgap (1.8 eV) for single-layer (SL) MoS$_2$ to an indirect bandgap (1.2 eV) for bulk MoS$_2$ [7]. Dissimilar to graphene with a zero bandgap, the thickness-dependent modulation of bandgaps motivated the exploration of MoS$_2$ in optical and electrical devices [3, 8]. Based on the physics of MoS$_2$, the density of states of few-layer MoS$_2$ is triple that of single-layer MoS$_2$, resulting in high drive currents in the ballistic limit [8]. In this context, few-layer MoS$_2$ may deliver significant advantages for transistor applications than SL MoS$_2$ [3].

On the other hand, the electronic devices based on traditional silicon dioxide dielectrics are approaching the physical limit because of its low dielectric constant [9]. To obtain a thin equivalent oxide thickness (EOT), it is crucially important to integrate high-k dielectrics with MoS$_2$. To date, many high-k dielectrics have been investigated with MoS$_2$, including Al$_2$O$_3$, ZrO$_2$, HfO$_2$, and h-BN [10–14]. DiStefano et al. obtained the respective conduction and valence band offsets of 3.3 ± 0.2 and 1.4 ± 0.2 eV for few-layer MoS$_2$ grown by oxide vapor deposition on amorphous BN [13]. Tao et al. reported that the conduction band offset (CBO) for the monolayer MoS$_2$/Al$_2$O$_3$ (ZrO$_2$) heterojunction was deduced to be 3.56 eV (1.22 eV), while the valence band offset (VBO) was 3.31 eV (2.76 eV) [15]. And a CBO of 2.09 ± 0.35 eV and VBO of 2.67 ± 0.11 eV at the MoS$_2$/HfO$_2$ interface were reported by McDonnell et al. [12]. Among these gate dielectrics, HfO$_2$ was considered to be one of the most promising candidates owing to its high dielectric constant ($k \sim 20$), compatibility with polysilicon, TaN gates, and polycrystalline silicon gate [16]. However, HfO$_2$ has a poor thermal stability, large leakage current, high oxide trap density, interface trap density, etc. [17]. These limitations have motivated extensive investigations of searching passivation techniques, such as interface nitridation or fluorination treatment technologies [18, 19]. In this work, we studied the energy...
band alignments of few-layer MoS\textsubscript{2} on HfO\textsubscript{2} dielectrics with and without plasma nitridation, in which the effect of surface nitridation was characterized by X-ray photoelectron spectroscopy (XPS).

\textbf{Methods}

The SiO\textsubscript{2} (280 nm)/Si wafer was alternately cleaned with acetone and isopropanol by ultrasonic cleaning for each 10 min, followed by deionized water rinse and N\textsubscript{2} dry. The few-layer MoS\textsubscript{2} films were deposited on SiO\textsubscript{2}/Si substrate by chemical vapor deposition (CVD) using precursors of MoO\textsubscript{3} (0.08 mg, 99%, Alfa Aesar) and S powder (1 g, 99%) [20, 21]. After the growth procedure, the MoS\textsubscript{2} film would be transferred to HfO\textsubscript{2}/Si substrate by the poly (methyl methacrylate) (PMMA) method [22], as depicted in Fig. 1a. In this process, PMMA was first spin-coated on MoS\textsubscript{2}/SiO\textsubscript{2}/Si samples as a supporting layer. Then, the samples were immersed in KOH solution for etching away the SiO\textsubscript{2} after which the MoS\textsubscript{2} layer with PMMA would float to the top of the solution. In the end, the PMMA layer would be dissolved in acetone after the sample was transferred onto HfO\textsubscript{2}/Si substrate. The HfO\textsubscript{2} films were grown on the silicon wafer by atomic layer deposition (ALD) at a temperature of 200 °C using Hf[N(CH\textsubscript{3})(C\textsubscript{2}H\textsubscript{5})\textsubscript{4}] [tetrakis (ethylmethylamido) hafnium, TEMAH] and H\textsubscript{2}O vapor as precursors [23, 24]. During the optimization process of the plasma treatment time, it was found that the nitrogen would diffuse into the oxide greatly after 70 s nitridation treatment by SIMS measurements, which would severely deteriorate the oxide quality. While the plasma treatment time is 30 s, no obvious N peak at the oxide surface was observed from the SIMS results. For the control sample, 50 s N\textsubscript{2} plasma treatment was implemented on HfO\textsubscript{2}/Si substrate at a pressure of 3 Pa before the MoS\textsubscript{2} transfer. Under the plasma condition, the resultant N dose is about 8.4 \times 10\textsuperscript{14} atoms/cm\textsuperscript{2} estimated from the secondary mass spectrometry (SIMS) results. And the concentration of nitrogen was calculated to be about 1.5% after nitridation based on the XPS data. Four samples 1–4# were prepared for XPS measurements: 1# few-layer MoS\textsubscript{2} film on SiO\textsubscript{2}/Si substrate (few-layer MoS\textsubscript{2}), 2# thick HfO\textsubscript{2} film on Si substrate (bulk HfO\textsubscript{2}), 3# transferred MoS\textsubscript{2} film on as-grown HfO\textsubscript{2}/Si substrate (as-grown MoS\textsubscript{2}/HfO\textsubscript{2} heterojunction), and 4# transferred MoS\textsubscript{2} film on N\textsubscript{2} plasma-treated HfO\textsubscript{2}/Si substrate (nitrided MoS\textsubscript{2}/HfO\textsubscript{2} heterojunction).

\textbf{Results and Discussions}

RENIISHAW inVia Raman spectroscopy was employed to characterize the Raman spectra of few-layer MoS\textsubscript{2} film before and after transfer procedure, as illustrated in Fig. 1b. Two Raman peaks can be seen at around 382.86 cm\textsuperscript{-1} and 406.43 cm\textsuperscript{-1}, corresponding to the in-plane (\textit{E}\textsubscript{1g}) and out-of-plane (\textit{A}\textsubscript{1g}) modes, respectively [25, 26]. It was found that there is nearly no Raman shift in \textit{E}\textsubscript{1g} and \textit{A}\textsubscript{1g} mode frequencies after transfer process, indicating minimal structure modification. The frequency difference (\Delta\textit{k}) between \textit{E}\textsubscript{1g} and \textit{A}\textsubscript{1g} mode was deduced to be about 23.57 cm\textsuperscript{-1}, designating around four to five layers of MoS\textsubscript{2} film [27]. As shown in the inset of Fig. 1b, the thickness of MoS\textsubscript{2} film was verified to be approximately 2.8 nm by high-resolution transmission electron microscope (HRTEM), which is in consistent with the abovementioned Raman spectra. Moreover, we presented SIMS depth profiles of transferred MoS\textsubscript{2} film on nitrided HfO\textsubscript{2}/Si substrate. SIMS measurement was performed on a Physical Electronics ADEPT 1010 SIMS instrument with Cs primary ion beam at the energy of 1 keV, in which positive ions were collected and charge compensation was carried out. In this SIMS measurement, the nitrogen element was quantified while the other elements (Mo, Hf, and Si) are only meant as layer markers and not quantified. As illustrated in Fig. 2a, the depth profiles for transferred MoS\textsubscript{2} film on nitrided HfO\textsubscript{2}/Si substrate were determined by SIMS, in which signals of main components represented by Mo, N, Hf, and Si are plotted against the depth. The spreading of N into the HfO\textsubscript{2} layer was observed, which could be intrigued by the N injection into the underlying layer during primary beam bombardments or plasma treatments. It is also worth noting that depth profiles near the surface layer are normally complicated and meaningless because of the surface contamination and surface effects, e.g., the abnormal intensity of N element near the surface [28]. The higher signal of N profile near the HfO\textsubscript{2}/Si interface could be ascribed to that the nitrogen tends to diffuse to the HfO\textsubscript{2}/Si interface, leading to the accumulation of N near the interface [29]. The tail of Mo in HfO\textsubscript{2} film could be mainly caused by primary beam bombardments in SIMS measurements [30]. Figure 2b illustrates the respective N 1s XPS spectra for sample 3# and 4#; the high-intensity peaks for both heterojunctions were Mo 3P\textsubscript{3/2} while a low-intensity peak at ~ 395.80 eV was detected for the nitrided heterojunction, indicating the formation of Mo-N bonding [31].

To obtain the band alignments between few-layer MoS\textsubscript{2} and HfO\textsubscript{2} with and without nitridation treatment, XPS measurements with a step of 0.05 eV were carried out on VG ESCALAB 220i-XL system using a monochromatic Al Ka X-ray source (h\nu = 1486.6 eV). The constant pass energy was set at 20 eV. Additionally, the standard C 1s (284.8 eV) was used for binding energy (BE) calibration [32]. To evaluate VBO values for MoS\textsubscript{2}/HfO\textsubscript{2} heterojunctions, Mo 3d and Hf 4f core levels (CLs) were selected for sample 1–4#, respectively. Figure 3a presents the XPS narrow scan of Mo 3d and valence band spectra from sample 1# [33]. Thus, the binding energy difference (BED) between Mo 3d\textsubscript{3/2} core
level and valence band maximum (VBM) for sample 1 was calculated to be 228.49 ± 0.1 eV. Figure 3b illustrates the CLs of Hf 4f_{7/2} and VBM for sample 2; the corresponding BED was determined to be 14.10 ± 0.1 eV. Figure 3c depicts the measured XPS spectra of Mo 3d and Hf 4f CLs for MoS_2/HfO_2 heterojunctions with/without nitridation treatment. It is noted that the Mo 3d_{5/2} CL shifted from 229.45 ± 0.05 eV for sample 3 to 229.90 ± 0.05 eV for sample 4. This could be ascribed to that a nitridation interfacial layer was introduced at the MoS_2/HfO_2 interface after plasma treatment, resulting in the abovementioned Mo-N bonding. With the presence of Mo-N bonding, the consequent charge transfer between Mo and N elements contributed to the measured Mo 3d_{5/2} CL shift. Additionally, the Hf 4f_{7/2} CL of 17.40 ± 0.05 eV for sample 3 was shifted to a higher binding energy of 17.60 ± 0.05 eV for sample 4 while O 1s also showed a shift of 0.20 eV to a higher BED, as shown in Fig. 3d. These peak shifts implied the downward band bending at the HfO_2 surface, which could be interpreted as that the nitrogen plasma induced donor-like defects for HfO_2 [34]. Based on the Kraut method [35], the VBO ($\Delta E_V$) values can be calculated from the following equation:

$$\Delta E_V = (E_{MoS_2}^{Mo 3d_{5/2}} - E_{VBM}^{MoS_2}) - (E_{HfO_2}^{Hf 4f_{7/2}} - E_{VBM}^{HfO_2}) - \Delta E_{CL}$$

where $E_{MoS_2}^{Mo 3d_{5/2}}$ and $E_{MoS_2}^{VBM}$ are binding energies of Mo 3d_{5/2} CL and VBM for MoS_2, $E_{HfO_2}^{Hf 4f_{7/2}}$ and $E_{HfO_2}^{VBM}$ are

![Fig. 1](image)

**Fig. 1 a** Process flow of PMMA-assisted wet transfer method for the MoS_2/ALD-HfO_2 heterojunction formation. **b** Respective Raman spectra of as-grown and transferred MoS_2 film. The inset is the cross-section transmission electron microscopy images of as-grown MoS_2 on SiO_2/Si substrate.
Fig. 2  a) SIMS depth profiles of transferred MoS$_2$ film on nitrided HfO$_2$/Si substrate.  b) N 1s XPS spectra for MoS$_2$/HfO$_2$ heterojunctions with and without nitridation treatment, respectively.

Fig. 3  a) XPS spectra of Mo 3d CL and valence band for the few-layer MoS$_2$.  b) XPS spectra of Hf 4f CL and valence band for bulk HfO$_2$.  XPS spectra of c) Mo 3d, Hf 4f, and d) O 1s CLs for transferred MoS$_2$ film on bulk HfO$_2$ with/without nitridation treatment.
binding energies of Hf 4f 7/2 CL and VBM for ALD-HfO₂, ΔE_CL = E_{Mo 3d 5/2}^{MoS₂} − E_{Hf 4f 7/2}^{HfO₂} refers to the BED between Mo 3d 5/2 and Hf 4f 7/2 CLs for ALD-HfO₂/MoS₂ heterojunctions. Hence, the ΔE_V of MoS₂ on ALD-HfO₂ with and without nitridation treatment were calculated to be 2.09 ± 0.1 and 2.34 ± 0.1 eV, respectively.

To assess the influence of N₂ plasma treatment on the conduction band offset (CBO, ΔE_C) between ALD-HfO₂ and few-layer MoS₂, the bandgaps of 5.9 ± 0.1 eV for HfO₂ and 1.4 ± 0.1 eV for MoS₂ were used here, respectively [7, 36]. Thus, the CBO can be attained by the following equation:

$$\Delta E_C = E_{HfO₂}^g - E_{MoS₂}^g - \Delta E_V$$  \hspace{1cm} (2)

where $E_{HfO₂}^g$ and $E_{MoS₂}^g$ are the bandgaps of HfO₂ and MoS₂, respectively. According to Eq. (2), the ΔE_C between MoS₂ and ALD-HfO₂ with and without nitridation treatment were calculated to be 2.41 ± 0.1 and 2.16 ± 0.1 eV, respectively. The corresponding band diagrams are illustrated in Fig. 4. Remarkably, both VBO and CBO values of these two heterojunctions provide excellent electron and hole confinements, ensuring their suitability for MoS₂-based FETs [37]. Moreover, the nitrided heterojunction has a higher CBO compared with unnitrided heterojunction, which is better for n-channel FETs applications.

**Conclusions**

In conclusion, the XPS measurements revealed that the band alignment at the MoS₂/HfO₂ interface could be modified by introducing nitridation to HfO₂ surface prior to stacking MoS₂ film. The CBO and VBO were determined to be 2.16 ± 0.1 and 2.34 ± 0.1 eV for the unnitried MoS₂/HfO₂ heterojunction, whereas the CBO was altered up to 2.41 ± 0.1 eV and the VBO was altered down to 2.09 ± 0.1 eV for the nitrided MoS₂/HfO₂ heterojunction, respectively. A nitridation interfacial layer was introduced at the interface, which was found to result in the Mo-N bonding formation. Additionally, the nitrogen plasma could induce donor-like defects, leading to the surface band bending for HfO₂. In this way, the interfacial band alignment engineering would supply promising routes toward the flexible design and optimization of modern electronics.

**Abbreviations**

ALD: Atomic layer deposition; BE: Binding energy; BED: Binding energy difference; CBO: Conduction band offset; CL: Core level; CVD: Chemical vapor deposition; FET: Field-effect transistor; HfO₂: Hafnium oxide; HRTEM: High-resolution transmission electron microscope; MoS₂: Molybdenum disulfide; PMMA: Poly (methyl methacrylate); SIMS: Secondary ion mass spectrometry; SL: Single-layer; TEMAH: Tetrakis(ethylmethylamido) hafnium; TMDC: Transition metal dichalcogenide; VBM: Valence band maximum; VBO: Valence band offset; XPS: X-ray photoelectron spectroscopy

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**Authors’ Contributions**

WHY performed the experiment, data processing, and manuscript drafting. WJL, XYX, and SJD modified the manuscript. Other authors help review and discuss the manuscript. All authors read and approved the final manuscript.

**Availability of Data and Materials**

The datasets supporting the conclusions of this manuscript are included within the manuscript.

**Competing Interests**

The authors declare that they have no competing interests.
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