Mechanical transfer of electrochemically grown molybdenum sulfide layers to silicon wafer

Talha Nisar · Torsten Balster · Veit Wagner

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Abstract

Large area MoS₂ ultra-thin film deposition is one of the big challenges in the recent years. Electrodeposition provides an opportunity to grow such ultra-thin films on large scale. However, the transfer of the electrochemically grown film is challenging. Standard transfer of those thin films is done by wet etching in which the underlying substrate is etched. In this work, the polymer coated electrodeposited MoS₂ films on Au are separated mechanically from the underlying substrate by using ultra-sonication. Collapse of micron-sized bubbles produced by ultra-sonication at the interface of Au and silicon substrate provides enough energy for separation due to their weak adhesion. The Au layer is then removed by standard Au-etchant (K/KI) and the polymer coated film is transferred to a desired substrate. Ammonium tetrathiomolybdate (ATTM) has been used as precursor material for the electrodeposition of the films. Initial electrochemically grown films consist of MoS₃ which is reduced to MoS₂ by a post-annealing step at 450–900 °C. Obtained films are investigated by AFM, Raman, UV–Vis and XPS. Crystal quality improves by increasing the post-annealing temperature. The thickness of the thinnest film was found to be equivalent to 2 monolayers of MoS₂, which is desirable for future electronics.
Keywords MoS$_2$ · Electrodeposition · Mechanical transfer of 2D TMD layer

1 Introduction

After discovery of graphene as an atomically thin two-dimensional (2D) material [1], a complete new research area has been opened. Recently, 2D semi-conducting materials analogue to graphene, i.e. transition metal dichalcogenides (TMDCs), have attracted great attention due to their interesting optical and electronic properties and 2D nature. Among other 2D TMDCs, molybdenum disulfide (MoS$_2$) exhibits a non-zero band-gap, unlike conducting graphene, and it has interesting properties such as a tunable bandgap [2], optoelectronics [3], and spin–orbit coupling effects [4]. It can be used in switching devices such as a field effect transistor and in hydrogen evolution reaction because of its suitable band-structure [5, 6]. TMDCs thin films have been deposited by various deposition methods such as pulsed laser deposition [7], chemical vapor deposition (CVD) [8, 9], mechanical exfoliation [5], liquid-phase exfoliation [10], dip-coating [11, 12] and spin-coating [13]. Fabrication of transistors is also reported by solution-based processes such as spin-coating and liquid-phase exfoliation [10, 14]. Each of these growth methods has advantages and disadvantages. Mechanical exfoliation method yields a good quality MoS$_2$ flakes but the lateral flake size and thickness of MoS$_2$ flake is not controllable with this method [5]. CVD also yields high quality MoS$_2$ flakes with large lateral size but it requires vacuum [9]. Similarly, ball milling can be used for large scale production of MoS$_2$ but it damages the crystal structure and causes a lot of defects [15]. MoS$_2$ and WS$_2$ monolayer flakes have been also obtained by dip-coating process but again thickness and lateral size is not controllable [11, 12].

However, a major challenge is to grow MoS$_2$ ultra-thin films on large scale and transferring those films to a desired substrate for different applications and characterization while preserving the quality of the films. In this work we present an alternative solution-based deposition methods which gives us an opportunity to control the thickness and lateral size of the grown layer. Ultra-thin films of MoS$_2$ have been...
grown electrochemically on an Au substrate and transferred onto a silicon substrate by mechanical transfer method followed by gold etching. Such mechanical transfer method while the MoS2 film is supported by a poly(methyl methacrylate) (PMMA) layer was recently reported [16], which is an alternative transfer method to chemical etching of the substrate. For chemical etching of the substrate, usually toxic etchants are used such as hydrofluoric acid (HF) for etching of SiO2 [17]. In order to avoid toxic etchants for transfer, the environmental-friendly transfer method has been used in our approach. The MoS2 films are mechanically separated by ultra-sonication from the silicon substrate along with Au layer because of its weak adhesion to the oxidized silicon surface. The Au layer is removed by a non-toxic gold-etchant such as potassium iodide. This mechanical transfer followed by gold etching doesn’t harm the MoS2 film and the underlying substrate can be reused. The obtained films have been characterized by various spectroscopic methods, i.e. Raman spectroscopy, UV–Vis and XPS before and after transfer.

2 Experiment

Ammonium tetrathiomolybdate ((NH4)2MoS4, ATTM) was dissolved in di-ionized (DI) water in order to prepare 1 mM precursor solution for the electrodeposition. The aqueous solution of the precursor was ultra-sonicated for 20 min to get an uniform solution. Silicon wafers were cleaned with acetone and isopropanol and dried with nitrogen. Afterwards the wafers were treated with UV-ozone for 10 min to remove organic contaminations. A 50 nm gold layer was deposited on the silicon wafers by e-beam evaporation. However any standard deposited method can be used to deposit Au thin film such as physical vapor deposition or sputter coating. After preparing electrolyte and substrates for electrodeposition, the electrochemical setup was prepared. The Potentiostat consists of three electrodes: counter electrode (Pt), working electrode (Au deposited on a silicon wafer) and reference electrode (calomel electrode). The reduction potential of the calomel electrode is 0.27 V, which is referenced to standard hydrogen electrode (SHE). The floating PMMA/MoS2/Au stack to separate from the silicon wafer and to float on the surface of the DI-water. The floating PMMA/MoS2/Au layer stack was then transferred from DI-water to a gold etching solution (K/KI) in order to etch the Au layer. Undiluted standard gold etchant (651,818) was used and was bought from sigma-aldrich. After etching the gold, the PMMA/MoS2 layer stack was picked up with a cleaned silicon wafer with highly hydrophilic surface and annealed at 120 °C for 30 min. Finally, the PMMA layer was removed with acetone. This way the MoS2 layer was successfully transferred of the conducting Au substrate to a silicon wafer. The transferred MoS2 sample was then annealed at target temperature (450, 500, 600, 700, 800, 900 °C) with a heating rate of 20 °C/min and with an additional sulfur source in inert 95% Ar and 5% H2 atmosphere. For this purpose, the sample was placed in the middle of a quartz tube and pure sulfur was placed in the upstream near the edge. The quartz tube was then placed in an oven. Initially the flow of Ar/H2 was kept high to remove residual air. The target temperature was maintained for 120 min. The complete sequence of the experiment is illustrated in Fig. 1.

The electrodeposited films and the films after transfer were characterized with different spectroscopic techniques to study the surface morphology, composition of the film and crystal quality. Atomic force microscope (AFM) (Nanosurf) was used in tapping mode to analyze surface morphology and uniformity of the films. The thickness of the films is measured by a Dektak profilometer. UV–Vis spectroscopy (Cary 5000 UV–Vis–NIR Spectrometer) was done in a spectral range from 200 to 1100 nm in order to observe the excitonic finger print of MoS2. For UV–Vis measurements, an electrochemically deposited film was transferred to a quartz substrate before and after post-annealing. Raman measurements (Horiba Jobin–Yvon T64000) were carried out using 514.5 nm laser excitation source in order to identify the chemical structure based on unique molecular bond vibration and to analyze the crystallinity from the width of the peaks. The Raman signal was collected by a×50 objective and dispersed with 2400 lines/mm grating and detected by a CCD (charge coupled device) detector at 110 °C (cooled by liquid nitrogen). In order to do x-ray photoelectron spectroscopy (XPS), the samples were introduced to the XPS vacuum vessel, which has a photoelectron spectrometer with a hemispherical analyzer (Specs Phoebos 100) and a Mg/Al X-ray gun (Specs XR-50). The analyzer was used in transmission mode with a pass energy of 50 eV and Mg Kα radiation (E = 1253.6 eV) was used as source of excitation. The XPS data evaluation was done with the CASA XPS™ software and Shirley method was used to subtract the
background. The minor shift in the binding energy because of charging was corrected with respect to the C\textsubscript{1s} peak.

### 3 Results and Discussion

Thermal decomposition of ATTM happens between 120 and 360 °C and converts to MoS\textsubscript{3}. Above 800 °C in inert environment it turns into MoS\textsubscript{2} [18]. It has been reported that MoS\textsubscript{2} conversion temperature can be reduced from 800 to 450 °C using H\textsubscript{2} in an inert environment [18]. ATTM can be converted to MoS\textsubscript{3} at room temperature electrochemically according to Eq. 1 and 2. MoS\textsubscript{3} can be further reduced to MoS\textsubscript{2} by annealing above 450 °C in an inert environment. The thermolysis of electrochemically deposited MoS\textsubscript{3} takes place during post-annealing step and results in MoS\textsubscript{2} as one of the final reaction products. The chemical reactions that occur during electrodeposition and post-annealing process are given below [19].

\begin{align*}
(\text{NH}_4)_2\text{MoS}_4 & \rightarrow 2\text{NH}_4^+ + \text{MoS}_4^{2-} & \text{Dissociation (1)} \\
\text{MoS}_4^{2-} & \rightarrow \text{MoS}_3 + \text{S} + 2e^- & \text{Au} – \text{electrode (2)} \\
\Delta_{\text{MoS}_3} & \rightarrow \text{MoS}_2 + \text{H}_2\text{S} & \text{(3)}
\end{align*}

The color contrast of the optical microscope images shows that obtained films are uniform and cover large area (see Supplementary Data Fig. S1). Films with different thicknesses are obtained by varying the deposition time yielding a linear correlation (see Supplementary Data Fig. S2) which correlates with the difference in the color contrast of the optical microscope images (see Supplementary Data Fig. S1). ATTM solution with low concentration (1 mM) results in uniform and closed thin films. Ultra-thin films having thickness down to 1 nm have been successfully
obtained by electrodeposition, which is equivalent to thickness of a bi-layer of MoS$_2$. Electrodeposition is one of the best ways to produce large area ultra-thin films of MoS$_2$ at room temperature and without using vacuum. The polymer coated electrodeposited film with Au layer is mechanically separated from silicon wafer by means of ultra-sonication as shown in Fig. 1b. Due to the operational principle of the ultra-sonication machine standing acoustical waves are produced. Millions of micron-sized bubbles are produced at the nodes of those standing waves. Initially, there is a negative pressure creating the bubbles. The size of these bubbles is getting bigger and bigger, until the pressure switches from negative to positive pressure. Due to this positive pressure bubbles are compressed and collapse. This implosion of bubble releases energy and transfer it to the environment. The imploding bubbles can provide enough energy to separate two layers [16]. As a result of ultra-sonication, the PMMA/MoS$_x$/Au stack is separated from the silicon substrate because of low adhesion of Au to silicon. The PMMA layer protects the MoS$_x$ layer during this procedure from breaking [16].

In Fig. 2 roughness of the electrodeposited film obtained from the AFM images before and after transfer is shown. The surface roughness of the 2.2 nm thick MoS$_2$ film is ~0.7 nm which indicates that the obtained films are uniform and closed. Furthermore, no big change in the roughness of the films with increasing thickness is observed due to the amorphous nature of the films. The obtained films are uniform and closed and can be seen from the small roughness values of the films in Fig. 2. The AFM images are given in Fig. S3.

The MoS$_2$ formation was investigated by non-destructive spectroscopic methods such as Raman, XPS and UV–Vis spectroscopy. In Fig. 3 the UV–Vis spectra of an electrodeposited film before and after post-annealing are shown. In the spectrum of the film before post-annealing a gradual increase in the absorption is observed with decreasing wavelength. It doesn’t exhibit any absorption peak which indicates the absence of MoS$_2$ content in the initial electrochemically deposited film. In contrast, the spectrum of the film after post-annealing exhibit clear characteristic peaks of MoS$_2$ at 612 nm and 674 nm, which indicates the conversion of electrochemically deposited film to MoS$_2$. The peaks at 612 and 674 nm correspond to the A and B exciton of MoS$_2$ which are attributed to the direct transition at the $K$ point of the Brillouin zone [20, 21]. In addition a broad peak is visible at around 400–450 nm which is attributed to contain two individual peaks i.e. the C and D excitons which are attributed to inter-band transitions [20].

In Fig. 4a Raman spectra of electrodeposited films post-annealed at different temperatures (450–900 °C) are shown. The Raman spectra of the annealed films exhibit two characteristic peaks at 382 cm$^{-1}$ and 407 cm$^{-1}$, which correspond to the Raman active modes of MoS$_2$, i.e. the $E'_{2g}$ (in-plane vibrational mode) and $A_{1g}$ (out of plane vibrational mode) [22]. According to group theory, there are four Raman active modes of MoS$_2$ [23] but only two of them are observed in the measured spectral range. The four Raman active mode of MoS$_2$ are $A_{1g}$ (out-of-plane vibration (c-axis) of S atoms) at 407 cm$^{-1}$, $E'_{2g}$ (in-plane vibration (basal plane) of Mo and S atoms) at 382 cm$^{-1}$, $E_{1g}$ (in-plane vibration (basal plane) of Mo and S atoms) at 32 cm$^{-1}$, $E'_{1g}$ (in-plane vibration (basal plane) of S atoms) at 286 cm$^{-1}$, and $E_{2g}$ (in-plane vibration (basal plane) of Mo and S atoms) at 32 cm$^{-1}$. $E_{2g}$ is invisible under back-scattering Raman geometry and for $E'_{2g}$ the sensitivity factor is relatively small [24]. Among all Raman active modes of
MoS$_2$, $E_{2g}^1$ and $A_{1g}$ are widely studied and they also depend on the thickness of the MoS$_2$ films [22]. The Raman measurements allow to study the crystallinity of the MoS$_2$ films post-annealed at different temperatures. In Fig. 4b the full width at half maximum (FWHM) of both modes ($E_{2g}^1$ and $A_{1g}$) is plotted against the post-annealing temperature. The FWHM for both Raman active modes is large for low temperature and small for high temperature [25]. Decreasing FWHM with increasing temperature indicates that the crystalline quality of the MoS$_2$ improves for higher post-annealing temperatures. For comparison the Raman spectrum for geologically found bulk MoS$_2$ is also measured. It is found that the FWHM of the MoS$_2$ film annealed at 900 °C is essentially equal to the value of the reference MoS$_2$ crystal ($FWHM_{A_{1g}} = 1.94$ cm$^{-1}$ and $FWHM_{E_{2g}^1} = 1.71$ cm$^{-1}$). The minor difference in FWHM between electrochemically obtained MoS$_2$ and the geological MoS$_2$ crystal is attributed to the polycrystalline nature of the layers, which has more grain boundaries and related imperfections. A reported study shows that crystal grain size of MoS$_2$ increases with increasing post-annealing temperature [13]. Similarly, HR-TEM also shows high crystal quality for samples annealed at 900 °C [13] which is consistent with our measured Raman results.

In Fig. 5 Raman spectra of MoS$_2$ films with different thicknesses (Bi-monolayer to bulk) are shown. A red-shift is observed for the peak position of the $E_{2g}^1$ mode and a blue-shift is observed for the peak position of the $A_{1g}$ mode with increasing number of MoS$_2$ monolayers. Both peaks are moving away from each other with increasing number of MoS$_2$ monolayer. According to literature the difference between $A_{1g}$ and $E_{2g}^1$ peak positions is 20 cm$^{-1}$ for MoS$_2$ monolayer and 27 cm$^{-1}$ for MoS$_2$ bulk [22, 26]. So in Fig. 5 the Raman spectrum with a difference of 22 cm$^{-1}$ between peak positions is attributed to MoS$_2$ bi-monolayer while the Raman spectrum with 27 cm$^{-1}$ difference between peak positions is attributed to bulk-like MoS$_2$, i.e $>6$ML. These results agree with the expected layer thickness from profilometer measurements. In Fig. 5, AFM image of transferred MoS$_2$ layer is shown. The thickness of the film is 1.9 nm which is equivalent to three monolayers of MoS$_2$ as thickness of the MoS$_2$ monolayer is 0.65 nm [27].

In Fig. 6 x-ray photoelectron spectra of the Mo$_3$d doublet and the S$_{2s}$ singlet with corresponding peaks are shown. The spectrum in the upper panel is of a film after the
electrochemical deposition while the lower panel contains the spectrum of this film transferred to Si substrate and after annealing at 800 °C. The spectrum of the electrochemically deposited film exhibits a Mo3d doublet with binding energy of 232.7 eV and 229.5 eV for the Mo3d3/2 and 3d5/2, respectively, which is attributed to MoS3 [28]. The atomic ratio between Mo3d and S2s peaks is found to be 1:2.85. It can also be deduced from the atomic ratio that the electrodeposited film contains MoS3. In the lower panel, the spectrum of the transferred film annealed at 800 °C consists of a Mo3d doublet with a binding energy of 232.2 eV and 229 eV for the Mo3d3/2 and 3d5/2, respectively. The red-shift in the binding energy of the 3d state of Mo with respect to the MoS3 counterpart is 0.5 eV, which is attributed to the formation of MoS2 [29]. The peak intensity of the S2s peak reduces when the film is annealed at 800 °C because the MoS2 reduces to MoS2. The Atomic ratio between Mo3d and S2s peaks is found to be 1:2.08, which is close to the ideal MoS2 ratio. Hence the formation of MoS2 is confirmed after annealing which is also verified by Raman and UV–Vis spectroscopy.

![XPS spectrum: Mo3d and S2s peaks of the electrodeposited film before and after conversion to MoS2](image)

**Fig. 6** XPS spectrum: Mo3d and S2s peaks of the electrodeposited film before and after conversion to MoS2

4 Conclusion

Ultra-thin films of MoSx were successfully deposited by means of electrochemical deposition at room temperature. The film thickness was controlled by varying the electrodeposition time. A film thickness down to 1 nm was successfully achieved. For film thickness beyond a thickness of 10 nm the roughness stayed around 1 nm which is comparable to the thickness of monolayer of MoS2. The roughness of the films was found to be essentially thickness independent as expected from smooth, uniform films covering the surface completely. The electrochemically deposited films were successfully transferred from the initial Au substrate to a bare silicon substrate. The mechanical transfer method followed by environmental-friendly standard Au etching is a novel green method for thin film transfer without using toxic chemicals. It doesn’t harm the MoSx film during transfer process. Furthermore, the underlying growth substrate can be reused. The MoSx films were successfully converted to MoS2 by post-annealing with an additional sulfur source in Ar/H2 environment. The reduction of the electrodeposited films to MoS2 was confirmed by Raman, XPS, and UV–Vis spectroscopy. The crystallinity of the MoS2 films was improved with higher post-annealing temperatures as confirmed by Raman measurements. The successful preparation and transfer of Bi-monolayer of MoS2 was as well confirmed by Raman spectroscopy.

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