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IMPACT OF REDUCED GRAPHENE OXIDE ON MOS2 GROWN BY SULFURIZATION OF SPUTTERED MOO3 AND MO PRECURSOR FILMS (POSTPRINT)

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Interim Report

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14. ABSTRACT *(Maximum 200 words)*
Monolayer molybdenum disulfide (MoS2), a two dimensional semiconducting dichalcogenide material with a bandgap of 1.8–1.9 eV, has demonstrated promise for future use in field effect transistors and optoelectronics. Various approaches have been used for MoS2 processing, the most common being chemical vapor deposition. During chemical vapor deposition, precursors such as Mo, MoO3, and MoCl5 have been used to form a vapor reaction with sulfur, resulting in thin films of MoS2. Currently, MoO3 ribbons and powder, and MoCl5 powder have been used. However, the use of ribbons and powder makes it difficult to grow large area-continuous films. Sputtering of Mo is an approach that has demonstrated continuous MoS2 film growth. In this paper, the authors compare the structural properties of MoS2 grown by sulfurization of pulse vapor deposited MoO3 and Mo precursor films. In addition, they have studied the effects that reduced graphene oxide (rGO) has on MoS2 structure. Reports show that rGO increases MoS2 grain growth during powder vaporization.

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Impact of reduced graphene oxide on MoS_2 grown by sulfurization of sputtered MoO_3 and Mo precursor films

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I. INTRODUCTION

Molybdenum disulfide (MoS_2) has attracted much attention due to its direct bandgap of 1.8–1.9 eV observed in monolayer thick MoS_2 films,[1,2] making MoS_2 an attractive two dimensional (2D) semiconducting material for low power electronic and optoelectronic devices.[3,4] Growth processes of 2D MoS_2 include mechanical exfoliation,[5–7] chemical vapor deposition (CVD),[1,2] intercalation assisted exfoliation,[8–11] physical vapor deposition (PVD),[12,13] and a wet chemistry approach involving thermal decomposition of a precursor containing Mo and S.[14] Among these, mechanical exfoliation and CVD are the most common procedures currently used to produce mono- to few layers of MoS_2. In contrast to mechanical exfoliation, the growth of MoS_2 by CVD can enable large area films for device fabrication. Molybdenum disulphide films grown using CVD has demonstrated promising results for semiconductor grade material properties, with observed FET mobilities around 500 $cm^2/V\,s$.[15] During CVD growth, sulfurization of molybdenum containing precursors such as Mo, MoO_3, and $MoCl_5$ is usually performed. In the case of MoO_3 (Ref. 2) and $MoCl_5$,[16] powders or ribbons are the precursors, whereas Mo has been prepared by e-beam evaporation[1] or sputtering.[17] While the use of these powder and ribbon precursors have demonstrated large crystal growth,[15] it is difficult to continuously grow these large domains over an entire wafer. In addition, the presence of grain boundaries prevents optimum device

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TABLE I. List of thickness used for Mo and MoO_3 precursor films. *r* indicated rGO was used during experiments.

Sample (*r*-rGO)	Precursor	Precursor thickness (nm)
S1, S1*r*	Mo	3
S2, S2*r*	MoO_3	3

performance. Lee et al.[18] reported that seed promoters such as reduced graphene oxide (rGO), perylene-3,4,9,10-tetracarboxylic acid tetrapotassium salt (PTAS), and 3,4,9,10-perylene-tetracarboxylicacid-dianhydride improved layer growth of CVD grown MoS_2.[1] Ling *et al.*[19] furthers the investigation and demonstrates an increase in domain size when seed promoters are used during MoS_2 synthesis. In this work, we investigate the impact of rGO on MoS_2 grown by sulfurization of MoO_3 and Mo precursor films at room temperature. While CVD growth of MoS_2 films using RF sputtered MoO_3 precursors has been reported,[20] our work consists of using DC magnetron sputtered Mo and MoO_3 films. Sputtering of precursor films, followed by sulfurizing, has demonstrated continuous film growth of MoS_2.[17] Sputtering and other PVD approaches for precursor film preparations have advantages in uniformly covering large substrate areas, which can lead to large area 2D film synthesis, an advantage over using powder and ribbon precursors. X-ray photoelectron spectroscopy (XPS) was performed to confirm the chemical composition, and transmission electron microscopy (TEM) was used to examine the MoS_2 structure. Finally, surface morphology of the MoS_2 film was investigated with atomic force microscopy (AFM).

II. EXPERIMENT

Thin films of metallic Mo and MoO_3 were sputtered on c-face sapphire substrates (diameter of 25.4 mm) using a DC magnetron sputtering system (500 V at 100 mA) at room temperature, with an argon pressure of 0.92 Pa. The thickness of the precursor (3 nm for both Mo and MoO_3) was controlled by manipulating the sputtering so that there were equal amounts of Mo sputtered in the MoO_3 and Mo films. Table I lists the precursors and names of the sample. The substrates were ultrasonically cleaned in acetone for 5 min prior to deposition of Mo and MoO_3. Following sputtering of Mo and MoO_3 onto the substrates, the precursors were separately placed at the center of the quartz tube (Fig. 1). Sulfur powder (2 g) was placed in a ceramic boat, upstream from the Mo and MoO_3 films. Reduced graphene oxide (Sigma Aldrich) was dispersed in isopropyl alcohol and drop cast on separate sapphire substrates. The rGO samples were air dried before they were placed in the furnace next to the sputtered precursor films of Mo and MoO_3 (a distance of 5 mm between the precursor and rGO samples). After pumping the furnace down to a vacuum pressure of 667 Pa, the samples were heated to 300 °C at 20 °C/min and held there for 15 min. This enabled the removal of any residual water molecules. Next, the furnace was heated to 850 °C at a rate of 20 °C/min. As the temperature of the furnace approached 850 °C (around 830 °C), the boat with sulfur was heated to 125 °C using a heating tape. The samples and the sulfur were held at their temperatures for 1 h, followed by cooling to room temperature. All experiments were performed in an Ar/H_2 environment, with a flow rate of 75 sccm.

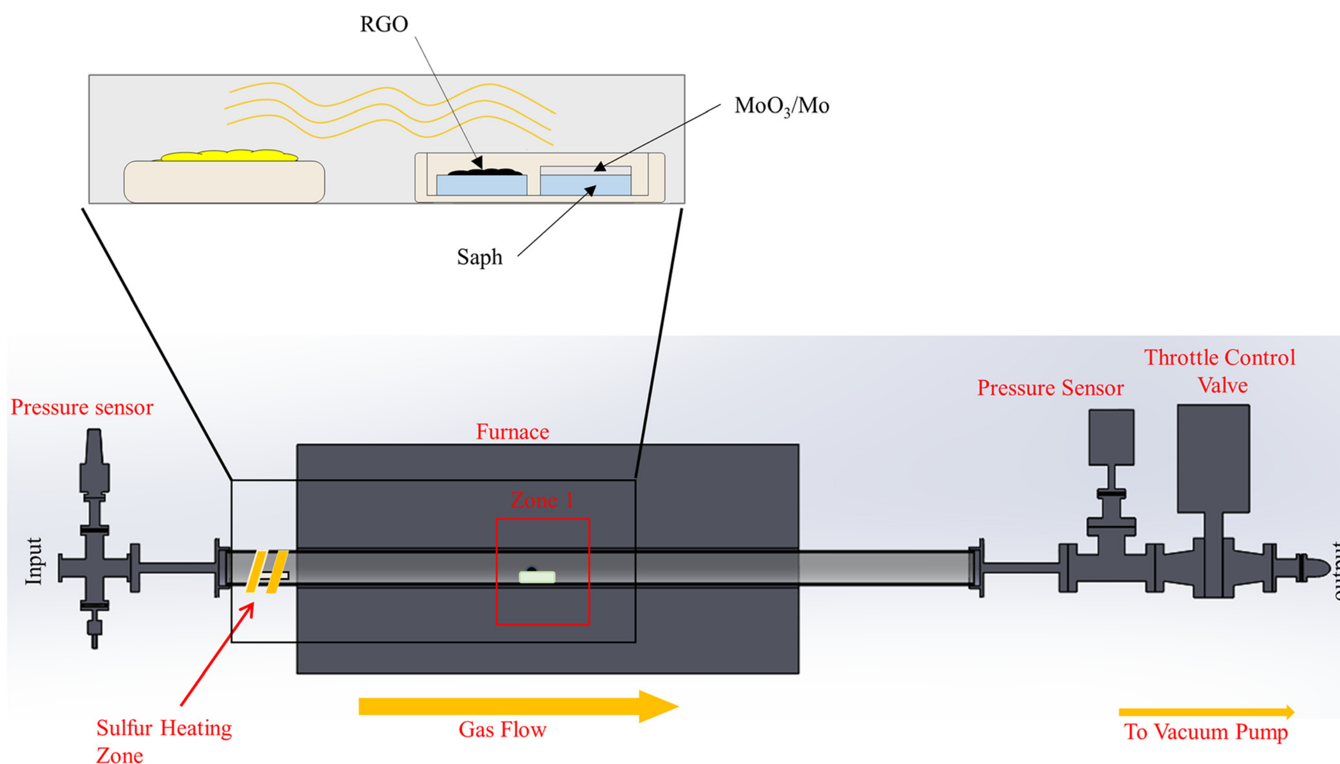

FIG. 1. (Color online) Chemical vapor deposition setup for MoS_2 growth on Al_2O_3 substrates.

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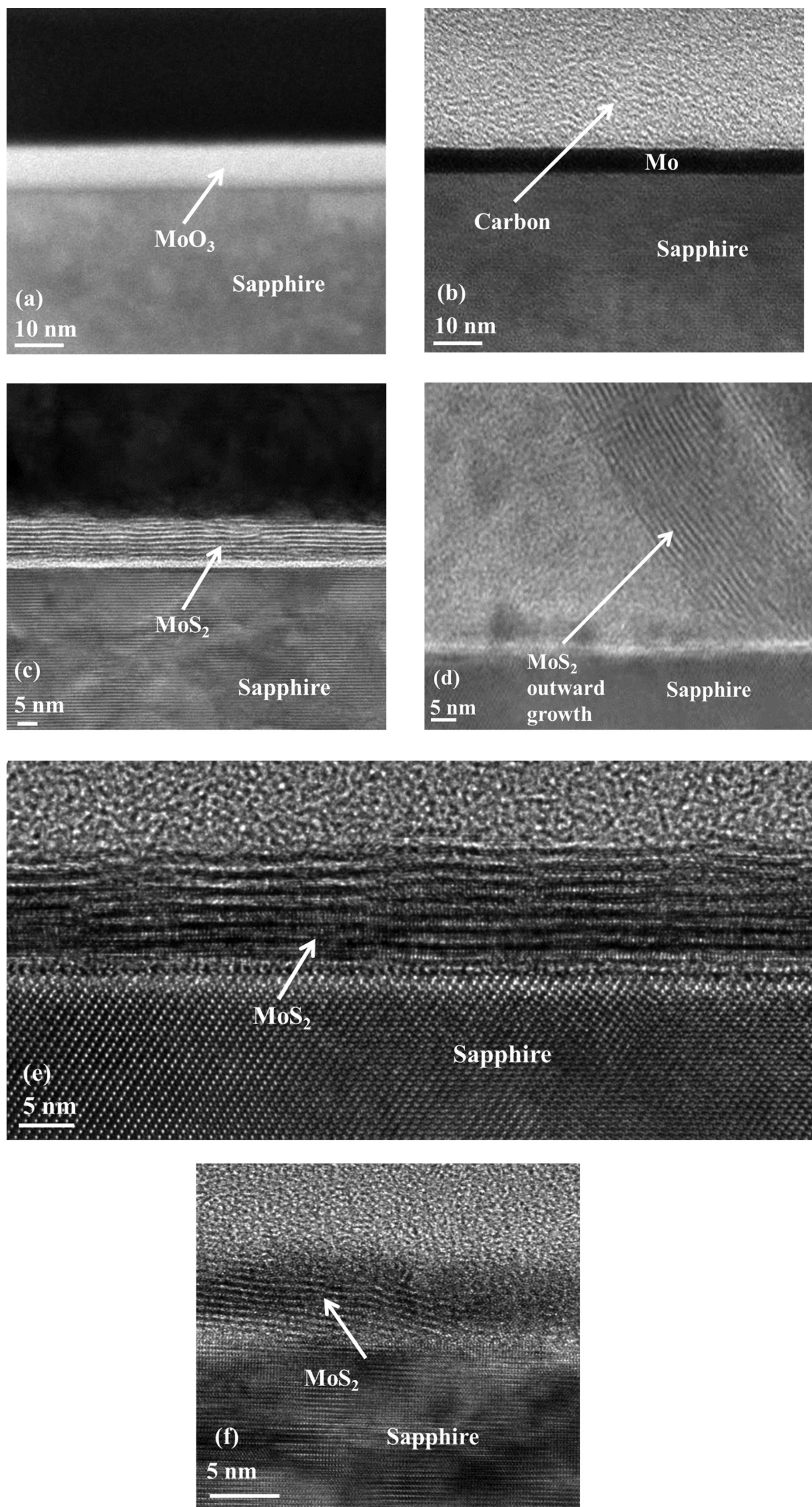

FIG. 2. TEM image of (a) MoO_3 precursor film used for MoS_2 growth, (b) Mo precursor film for MoS_2 growth, (c) sample S1 (MoS_2 on sapphire using Mo precursor) showing a layer thickness of 7 nm, (d) sample S2 (MoS_2 on sapphire using a MoO_3 precursor) showing an outward growth of MoS_2, with a thickness of 15 nm, (e) sample S1*r* (Mo precursor) using rGO with a measured thickness of 7–8 nm, and (f) sample S2*r* (MoO_3) using rGO with a thickness of 7 nm.

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A. Characterization

AFM images were taken in an Asylum MFP-3D system in the tapping mode. Images were 512 × 512 pixels, and the scan speed was 1 Hz (1 line per second). The nominal radius of curvature for the probe tips was 9 nm. The structure of the MoS_2 film was observed in a high-resolution TEM. Images were taken using a FEI Titan 80-300 S/TEM, which was operated at 300 kV. A FEI Nova focused ion beam (FIB) microscope equipped with

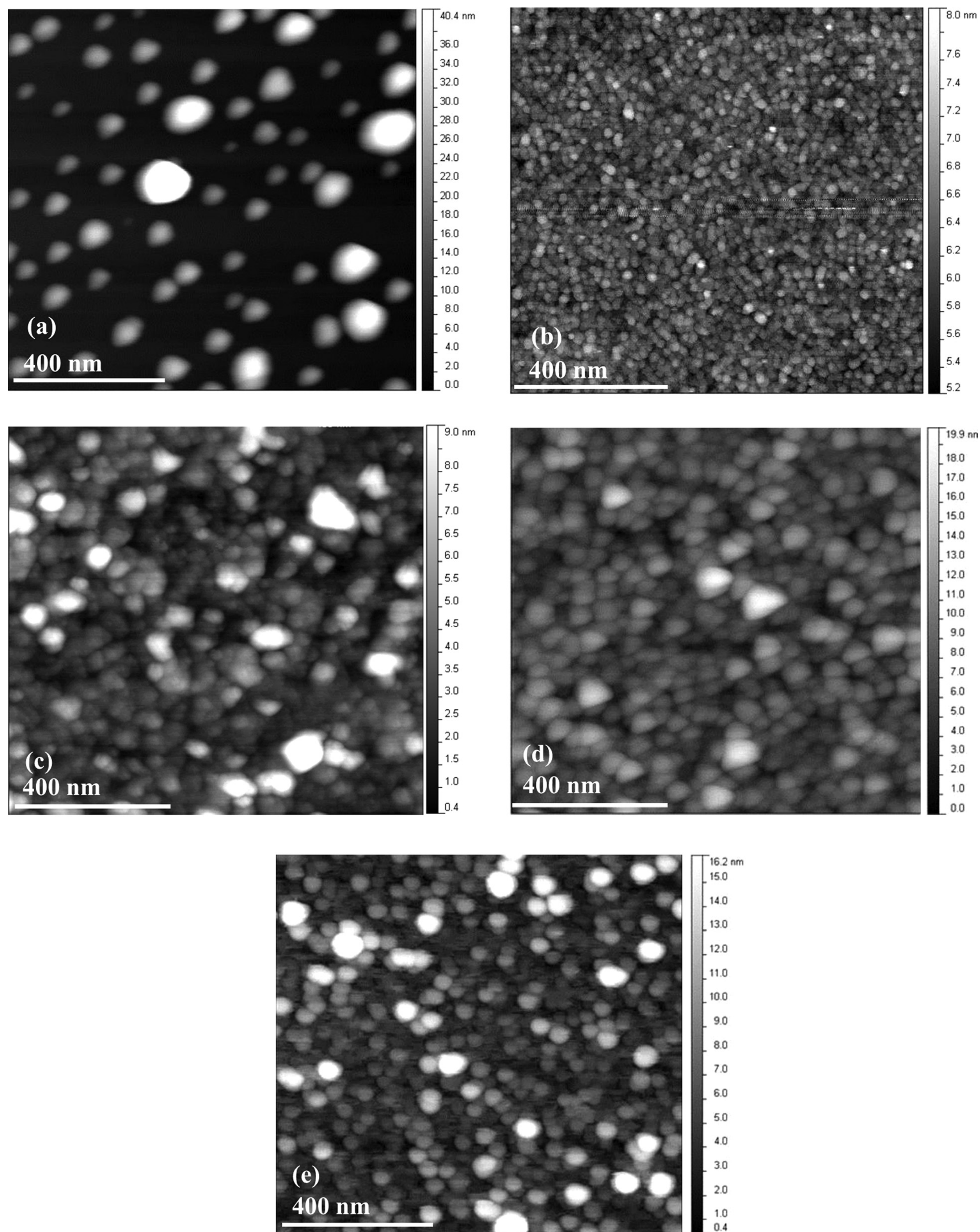

FIG. 3. AFM surface topography (1 × 1 μm) for (a) MoO_3 precursor that was heated to 850 °C forming MoO_2 islands, (b) sample S1 (Mo precursor) showing a dense film of MoS_2 with a grain size of 4.4 nm, (c) MoS_2 sample S2 (MoO_3 precursor) with a grain size of 7.9 nm, (d) MoS_2 sample S1*r* (Mo precursor with rGO) with a grain size of 17.7 nm, and (e) MoS_2 sample S2*r* (MoO_3 precursor with rGO) with grain size of 12.2 nm.

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an Omniprobe manipulator was employed to prepare the cross-sectional specimens. X-ray photoelectron spectroscopic analysis was carried out using a Phi Model 5700 spectrometer with a monochromatic Al Kα x-ray source (1486.6 eV) operated at 75 W (15 kV, 5 mA) and at approximately 1.5×10^{-12} Torr.

III. RESULTS AND DISCUSSION

A. MoS_2 film structure and grain size

TEM imaging of the MoS_2 film cross-sections for samples S1 (MoS_2 grown from Mo precursor) and S1*r* (MoS_2 film grown using Mo precursor with rGO seed) is shown in Fig. 2. The precursor films, MoO_3 and Mo, are both shown in Figs. 2(a) and 2(b). Samples S1 and S1*r* [Figs. 2(c) and 2(e)] show uniform and continuous layer growth of MoS_2. Both samples have a thickness of 7–8 nm, indicating that rGO used during the CVD growth of sample S1*r* had no effect on the film thickness. AFM showed samples S1 and S1*r* had an RMS of.360 and 2.43 nm (respectively), and the grain size increased from 4.5 to 17.7 nm, respectively [see Figs. 3(a) and 3(b)]. This increase in the grain size indicated that the

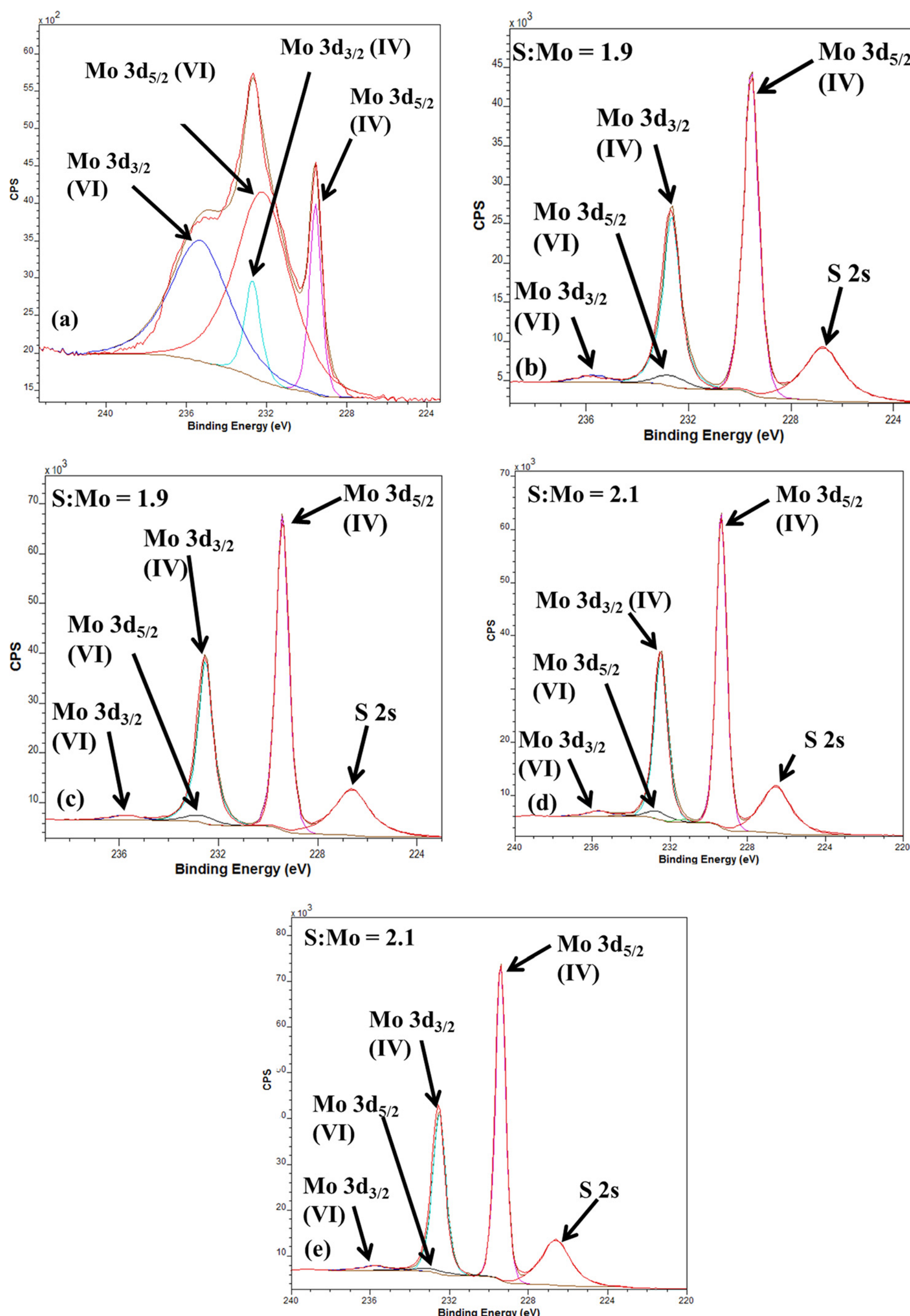

FIG. 4. (Color online) XPS spectra of annealed MoO_3, MoS_2 films S1 (MoS_2 using Mo precursor), S2 (MoS_2 using MoO_3 precursor), S1*r* (MoS_2 using Mo precursor and rGO), and S2*r* (MoS_2 using MoO_3 precursor and rGO). The annealed MoO_3 (a) shows Mo(IV) peaks which are indicative of MoO_2, and Mo(VI) peaks that occur when MoO_3 is present. Both (b) and (c) shows spectra for samples S1 and S2 (respectively) having a stoichiometric structure. In (d) and (e), the stoichiometry has been increased for samples S1*r* and S2*r* (respectively).

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rGO played a role in grain growth of the MoS_2. In contrast to the uniform and continuous film growth of samples S1 and S1*r*, samples S2 (MoO_3 precursor) and S2*r* (MoO_3 precursor with rGO seed) demonstrated a nonuniform, outward growth of MoS_2 [Figs. 2(d) and 2(f)]. It is reported that at 600 °C, MoO_3 reduces to MoO_2 under an H_2 environment.[21] In this research, it was indicated that MoO_2 had formed after annealing MoO_3 at 850 °C. X-ray photoelectron spectra [Fig. 4(a)] showed peaks at 229.57 and 232.7 for Mo(IV) (typical of MoS_2 and MoO_2) and 232.19 and 235.32 for Mo(VI) (typical of MoO_3). AFM was performed on the same annealed sample [Fig. 3(c)], and we noticed small islands across the substrate. We believe that these islands were formed when the sputtered MoO_3 film reduced to MoO_2 during annealing at 850 °C. Consequently, sulfurization of MoO_2 islands causes MoS_2 growth in a Volmer–Weber growth mechanism, which is a result of the film not wetting the substrate.[22] Moser and Levy reported similar growth patterns using sputtering technique to deposit thick MoS_2 films.[23]

Figures 3(d) and 3(e) show the grain structures of MoS_2 grown using the sputtered MoO_3 films (S2 and S2*r*). The RMS values for these samples were 2.00 nm (S2) and 3.66 nm (S2*r*), and the grain size increased from 7.9 nm (S2) to 12.2 nm (S2*r*) when rGO was used during the growth. This correlates well with the data from samples S1 and S1*r* that suggest that rGO promotes grain growth when using sputtered precursor films. There was also a decrease in the film thickness, from 15 nm (S2) to 7 nm (S2*r*) when rGO was used during the sulfurization process [Figs. 2(d) and 2(f)]. Ling *et al.*[19] reports that organic seed promoters (such as PTAS) enable heterogeneous nucleation sites and that the size of the MoS_2 domains is dependent upon the distance of the seed promoter from the precursor. We believe this is what occurred when rGO was used in our experiments involving sputtered Mo and MoO_3 films. However, further investigation needs to be conducted to better understand the kinetics, and mechanism of increasing grain size, when using rGO during the sulfurization sputtered films.

B. MoS_2 chemical composition

XPS analysis was performed in order to assess the composition and chemistry of the films. The survey spectra (not shown) from sulfurized thin films of samples S1/S1*r* and S2/S2*r* showed peaks from Mo and S, as expected. As mentioned previously, Fig. 4(a) shows the spectra for annealed MoO_3, which was discussed in Sec. III A. The influence of the rGO on MoS_2 stoichiometry was analyzed by comparing the S:Mo ratios obtained from the XPS spectra. The MoS_2 films grown from samples S1 and S1*r* had S:Mo ratios of 1.9 and 2.1, respectively [see Figs. 4(b) and 4(d)]. Samples S2 and S2*r* demonstrated the same respective S:Mo ratios of 1.9 and 2.1 [see Figs. 4(c) and 4(e)]. The apparent improvement in the film stoichiometry is most likely a result of the Mo:S averaging over large spot size analysis area in XPS, which is orders of magnitude larger when compared to the average grain sizes of synthesized MoS_2 films. The presence of the rGO helped to increase the MoS_2 grain size areas and correspondingly decrease contributions of photoelectrons escaped from the grain boundary areas.

Curve fits to the Mo 3d doublets for all of the samples revealed two populations of Mo atoms. The Mo $3d_{5/2}$ peak at 229.8 eV and Mo $3d_{3/2}$ peak at 232.9 eV reveal the presence of Mo(IV), with a binding energy typical of MoS_2 or MoO_2.[24] The Mo $3d_{5/2}$ peak at 232.7 eV and Mo $3d_{3/2}$ peak at 235.8 eV indicates the presence of Mo(VI), with a binding energy typical of MoO_3.[25] This suggests that while nearly all of the Mo(IV) is present in the form of MoS_2, there is likely some MoO_2 present at the surface or at grain boundaries within the films. However, the intensity of both the Mo(IV) and Mo(VI) peaks are very low that the presence of MoO_2 and MoO_3 is negligible.

IV. SUMMARY AND CONCLUSIONS

The influence of rGO on the sulfurization of metallic Mo and MoO_3 thin film precursors for MoS_2 growth has been investigated. TEM established that rGO did not have an impact on the MoS_2 films thickness for sputtered Mo, but that it was responsible for the increase in the grain size. We also observed an increase in the grain size when rGO was used during sulfurization of sputtered MoO_3. Reports demonstrate that seed promoters diffuse onto growth substrates, acting as nucleation sites for MoS_2 growth.[19] In addition, the size of the MoS_2 domains are dependent upon the distance between the seed promoter and the growth substrate. Further investigations are being conducted to explain the kinetics involved when rGO is used during sulfurization of sputtered Mo and MoO_3. Nevertheless, the nanocrystalline size of our MoS_2 films would not make them suitable candidates for electronic devices. However, these nanocrystalline films are potentially applicable for optoelectronics.

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