Utilizing complex oxide substrates to control carrier concentration in large-area monolayer MoS$_2$ films

Cite as: Appl. Phys. Lett. 118, 093103 (2021); https://doi.org/10.1063/5.0038383
Submitted: 21 November 2020 • Accepted: 18 February 2021 • Published Online: 03 March 2021


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Utilizing complex oxide substrates to control carrier concentration in large-area monolayer MoS2 films

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ABSTRACT

Bandgap engineering is central to the design of heterojunction devices. For heterojunctions involving monolayer-thick materials like MoS2, the carrier concentration of the atomically thin film can vary significantly depending on the amount of charge transfer between MoS2 and the substrate. This makes substrates with a range of charge neutrality levels—as is the case for complex oxide substrates—a powerful addition to electrostatic gating or chemical doping to control the doping of overlying MoS2 layers. We demonstrate this approach by growing mono-layer MoS2 on perovskite (SrTiO3 and LaAlO3), spinel (MgAl2O4), and SiO2 substrates with multi-inch uniformity. The as-grown MoS2 films on these substrates exhibit a controlled, reproducible, and uniform carrier concentration ranging from (1–4) x 1013 cm−2, depending on the oxide substrate employed. The observed carrier concentrations are further confirmed by our density-functional theory calculations based on ab initio mismatched interface theory (MINT). This approach is relevant to large-scale heterostructures involving monolayer-thick materials in which it is desired to precisely control carrier concentrations for applications.

Interfaces between dissimilar materials have enabled a variety of key technologies including high-efficiency light-emitting diodes,2 solar cells,3 and quantum electronics.4 Two-dimensional transition metal dichalcogenides (TMDs), with a wide range of band structures and direct bandgaps at monolayer thicknesses, provide a unique material platform for interface-engineered electronics.5 Due to their atomic thinness, the electronic properties of monolayer TMDs can be widely tuned by the substrate. For instance, monolayer molybdenum disulfide (MoS2), one of the most studied TMDs, exhibits a higher electron concentration on SiO2 substrates than when prepared on SrTiO3, h-BN, or Gel-Film.6–9 Understanding and further controlling the substrate-dependent carrier concentration are, thus, important for developing TMD-based electronics.

Unlike three-dimensional semiconductor heterostructures, the atomic thinness of monolayer TMDs (< 1 nm) limits the formation of a depletion or accumulation region.10 As a result, the difference between the charge neutrality level of a TMD and the underlying substrate leads to a change in the carrier concentration of TMDs through a charge transfer process to ensure an aligned Fermi level.7,11 This enables direct control of the carrier concentration in monolayer TMDs by selecting different substrates. Complex oxides, with a range of charge neutrality levels, provide a means for tuning the carrier concentration in TMDs that can supplement electrostatic gating or chemical doping,7,12–14 which are crucial for device applications (e.g., p–n junctions and transistors). For example, a complex oxide substrate can be used to uniformly shift the threshold voltage in a TMD-based transistor. The atomically smooth surface of complex oxide substrates further promotes the formation of a high-quality interface to TMDs with minimal charged impurities.15 Although charge transfer has been widely studied between graphene and oxide substrates,16–18 semiconducting TMDs have been
synthesized mainly on SiO$_2$ and interfacing TMDs with functional complex oxides remains a largely untapped avenue for altering the electronic properties of the TMDs. While exfoliated MoS$_2$ on LaAlO$_3$ and SrTiO$_3$ has been shown to have different carrier concentrations, large-scale growth of MoS$_2$ on complex oxide substrates with a controlled carrier concentration remains an unsolved challenge, as existing solid-precursor-based CVD only produces monolayer MoS$_2$ with limited spatial uniformity.

Here, we report the large-scale growth of monolayer MoS$_2$ with reproducible and controlled carrier concentrations on a series of complex oxides including perovskites (SrTiO$_3$ and LaAlO$_3$) and spinel (MgAl$_2$O$_4$). This monolayer MoS$_2$ is synthesized on complex oxides with spatial uniformity across three inches, taking advantage of a metal-organic chemical vapor deposition (MOCVD) method that uses precisely controlled gas-phase precursors to improve uniformity. The electron concentrations of MoS$_2$ on complex oxides are substrate dependent and are all lower than those of MoS$_2$ on SiO$_2$, as revealed by our Raman and photoluminescence spectroscopy (PL) measurements. The Raman and PL measurements are all performed at room temperature. To understand the observed substrate-dependent electron concentration of MoS$_2$, we performed density-functional theory (DFT) calculations using \textit{ab initio} mismatched interface theory.

FIG. 1. Uniform growth of monolayer-thick MoS$_2$ on (100) LaAlO$_3$, (100) SrTiO$_3$, (100) MgAl$_2$O$_4$, and amorphous SiO$_2$. (a) Schematic of the MOCVD growth process. Precursors are independently introduced into the furnace. Yellow, sulfur atom; red, molybdenum atom; white, carbonyl or ethyl ligand. (b) Photograph of monolayer MoS$_2$ grown on 1 in. diameter fused silica substrates. A bare substrate is shown on the left for comparison. (c) Optical micrographs of monolayer MoS$_2$ with partial coverage and (d) full coverage on LaAlO$_3$, SrTiO$_3$, and MgAl$_2$O$_4$, respectively. (e) Raman and (f) photoluminescence spectra of MoS$_2$ on LaAlO$_3$, SrTiO$_3$, and MgAl$_2$O$_4$. (g) Photograph of monolayer MoS$_2$ grown on a 2 in. diameter fused silica substrate. A bare substrate is shown on the left for comparison. (h) Raman spectra for MoS$_2$ taken at different locations marked on the corresponding fused silica wafer from (g). (i) AFM image of monolayer MoS$_2$ grown on LaAlO$_3$. 

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Figure 1 shows uniform, monolayer MoS2 films deposited on (100) SrTiO3, (100) LaAlO3, (100) MgAl2O4 (denoted as STO, LAO, and MAO, respectively), and amorphous SiO2 substrates. The growth was carried out in a home-built MOCVD system following the design of Kang et al. [Fig. 1(a)], using gas-phase precursors of Mo(CO)6 and (C2H5)2S (see the experimental methods in the supplementary material for details). The as-grown films are homogeneously yellow in appearance and are clearly distinguishable from the bare substrates [Figs. 1(b) and 1(g)]. We assessed the quality of the as-grown monolayer MoS2 using optical imaging, Raman, PL, and atomic force microscopy (AFM). Figures 1(c) and 1(d) show the optical micrographs of MoS2 with partial coverage and full coverage, where no multilayer region was observed before the completion of the growth of the first layer. Raman spectroscopy reveals characteristic E2g and A1g peaks of MoS2 at 384.1 and 403.6 cm−1 for MoS2/SiO2 [Fig. 1(b)], 384.4 and 405.2 cm−1 for MoS2/LaAlO3, 384.5 and 405.6 cm−1 for MoS2/SrTiO3, and 383.8 and 404.9 cm−1 for MoS2/MgAl2O4, respectively [Fig. 1(e)] (see Fig. S1 in the supplementary material for background subtraction), suggesting the high structural quality of our monolayer MoS2. The PL spectra show a sharp A exciton peak for MoS2/LaAlO3 and MoS2/SrTiO3 (centered at 1.86 eV), as well as for MoS2/MgAl2O4 (centered at 1.84 eV) [Fig. 1(f)], consistent with a previous report on an exfoliated sample. The full width at half maximum (FWHM) of the PL peaks on all substrates was smaller than 70 meV. The as-grown films are homogenously yellow in color, as seen by the narrow distribution of the A1g peak position across the entire film, as seen by the narrow distribution of the A1g peak position across the entire film. The position of the A1g peak is sensitive to the substrate upon which it is grown. Figure 2(a) compares the Raman spectra of MoS2 grown on SiO2, MgAl2O4, LaAlO3, and SrTiO3. The E2g mode (in-plane vibration) shows little dependence on the substrate. Specifically, only small shifts in the E2g peak position of MoS2/SiO2, MoS2/MgAl2O4, MoS2/LaAlO3, and MoS2/SrTiO3 are observed relative to those of MoS2/SiO2 [Fig. 2(b)]. The E2g peak position is known to depend linearly on the magnitude of the strain. From previous studies, E2g peak shifts by 2.1 cm−1 per % of uniaxial strain and by 5.2 cm−1 per % of biaxial strain. Thus, the strain in our MoS2 films on complex oxides does not exceed 0.21% (uniaxial) or 0.09% (biaxial). The minimal magnitude of strain suggests that the intrinsic lattice constant of MoS2 is not being perturbed by the underlying substrate.

In contrast to the E2g peak, the A1g peak exhibits a sizeable blue shift as well as a narrower FWHM [Fig. 2(b)], when the substrate is changed from SiO2 to complex oxides. The blue shift is uniform across the entire film, as seen by the narrow distribution of the A1g peak position measured at ten locations on two substrates placed 8 cm apart during growth [Figs. 2(c) and S2]. The distributions of the A1g peak position display blue shifts of 1.28±0.04 cm−1 (for MgAl2O4), 1.66±0.06 cm−1 (for LaAlO3), and 1.98±0.06 cm−1 (for SrTiO3) relative to MoS2 grown on SiO2. The position of the A1g peak is sensitive to the substrate upon which it is grown.
to strain\textsuperscript{30,31} and to electron doping due to the much stronger electron-phonon coupling of the A\textsubscript{1g} mode compared to the E\textsubscript{2g} mode.\textsuperscript{32,33} Since the Raman measurements show the strain effect to be minimal, we attribute these sizable blue shifts in the A\textsubscript{1g} peak to different amounts of charge transferred from the substrates. Based on the reported relationship between the electron density and the A\textsubscript{1g} peak position,\textsuperscript{34} the electron densities of monolayer MoS\textsubscript{2} grown on MgAl\textsubscript{2}O\textsubscript{4}, LaAlO\textsubscript{3}, and SrTiO\textsubscript{3} are estimated to be 0.30 ± 0.03 \times 10\textsuperscript{13} \text{ cm}\textsuperscript{2}, 0.48 ± 0.01 \times 10\textsuperscript{13} \text{ cm}\textsuperscript{2}, and 0.65 ± 0.02 \times 10\textsuperscript{13} \text{ cm}\textsuperscript{2} lower than when MoS\textsubscript{2} is deposited on SiO\textsubscript{2}, respectively.

The difference in electron density for MoS\textsubscript{2} grown on different substrates is further confirmed by PL spectroscopy. As a direct bandgap semiconductor, MoS\textsubscript{2} shows strong PL emission at room temperature,\textsuperscript{35} and the prominent A exciton peak can evolve into multiple peaks corresponding to a neutral exciton (A\textsuperscript{0}), a negatively charged trion consisting of one electron and two holes (A\textsuperscript{−}), or a positively charged trion consisting of two electrons and one hole (A\textsuperscript{+}).\textsuperscript{12} We note that the A\textsuperscript{−} and A\textsuperscript{+} peaks show blue shifts when the substrate is varied from SiO\textsubscript{2} to MgAl\textsubscript{2}O\textsubscript{4}, LaAlO\textsubscript{3}, and SrTiO\textsubscript{3}, which is caused by the dielectric screening of the Coulomb interactions.\textsuperscript{34} As the dielectric constant increases from SiO\textsubscript{2} (κ ≈ 4) to MgAl\textsubscript{2}O\textsubscript{4} (κ ≈ 8), LaAlO\textsubscript{3} (κ ≈ 24), and SrTiO\textsubscript{3} (κ ≈ 300),\textsuperscript{36–38} both the electronic bandgap and the exciton binding energies of MoS\textsubscript{2} decrease, leading to a small blue shift of A\textsuperscript{−} and A\textsuperscript{+} PL peaks. We note that the large κ of SrTiO\textsubscript{3} does not result in a substantial blue shift, which is caused by the PL peak energy approaching the electronic bandgap. This observation is consistent with previous report by Lin et al.\textsuperscript{35} that A\textsuperscript{−} and A\textsuperscript{+} peak positions start to saturate (approaching the electronic bandgap) as the relative dielectric constant of the environment exceeds 18.

Figure 3(d) compares the intensity ratio between the trion peak and the neutral exciton peak (I\textsubscript{A\textsuperscript{+}}/I\textsubscript{A\textsuperscript{0}}). MoS\textsubscript{2}/SiO\textsubscript{2} has the highest I\textsubscript{A\textsuperscript{+}}/I\textsubscript{A\textsuperscript{0}} intensity ratio of 1.62 ± 0.12, while this value drastically decreases to 0.60 ± 0.03 for MoS\textsubscript{2}/MgAl\textsubscript{2}O\textsubscript{4}, 0.52 ± 0.03 for MoS\textsubscript{2}/LaAlO\textsubscript{3}, and 0.46 ± 0.06 for MoS\textsubscript{2}/SrTiO\textsubscript{3}. Based on the quantitative relation from the mass-action model, which estimates the exciton and trion population by assuming dynamic equilibrium (A\textsuperscript{0} + e\textsuperscript{−} ↔ A\textsuperscript{−}),\textsuperscript{13} the differences in the electron concentration are estimated to be Δn (MoS\textsubscript{2}/SiO\textsubscript{2} - MoS\textsubscript{2}/MgAl\textsubscript{2}O\textsubscript{4}) ≈ 2.53 \times 10\textsuperscript{13} \text{ cm}\textsuperscript{2}, Δn (MoS\textsubscript{2}/SiO\textsubscript{2} - MoS\textsubscript{2}/LaAlO\textsubscript{3}) ≈ 2.70 \times 10\textsuperscript{13} \text{ cm}\textsuperscript{2}, and Δn (MoS\textsubscript{2}/SiO\textsubscript{2} - MoS\textsubscript{2}/SrTiO\textsubscript{3}) ≈ 2.89 \times 10\textsuperscript{13} \text{ cm}\textsuperscript{2} [Fig. 3(e)]. The lower, and closer to intrinsic, electron density of MoS\textsubscript{2} on complex oxides than on SiO\textsubscript{2} might be enabled by the atomically smooth surfaces of complex oxide substrates with reduced interfacial impurities.\textsuperscript{35,38}

The large-scale uniformity of the electron concentration in our MoS\textsubscript{2} films is corroborated by the narrow distributions in the histogram of the trion spectral weight.
predicted electron concentrations of 2.49 
oxide substrate layer. The finite-size scaling of the cluster calculations 
the interface of MoS2/SiO2, which are not considered in MINT calcu-
ments [Fig. 4(b)]. Specifically, the MINT predicted values for MoS2 on 
corner to low electron transfer from the substrate to MoS2. (b) Comparison of the 
strates exhibits a controlled, reproducible, and uniform carrier concen-
tration depending on the oxide substrate employed. This large-scale 
strates is not limited to MoS2 on MgAl2O4, LaAlO3, and SrTiO3. It can be readily applied to 
create interfaces between semiconducting TMDs and a myriad of 
complex oxides. Indeed, our preliminary attempts show that MoS2 
growths are also possible on gadolinium gallium garnet (GGG), 
YSiO3, and sapphire (Fig. S5 in the supplementary material). Our 
results demonstrate a means to provide uniform and reproducible 
electron transfer over large areas. Thus, complex oxide substrates pos-
possess significant potential for creating wafer-scale TMD-based devices 
with various electronic and optoelectronic properties.

See the supplementary material for experimental methods, 
detailed optical characterization of MoS2/oxides, MINT calculations, 
growth results on more complex oxides, fast growth of MoS2 on SiO2, 
and charge transfer between MoS2 and Nb-doped SrTiO3.

This work was supported by the National Science Foundation 
[Platform for the Accelerated Realization, Analysis, and Discovery 
of Interface Materials (PARADIM)] under Cooperative Agreement 
No. DMR-1539918. This work made use of the Cornell Center for 
Materials Research (CCMR) Shared Facilities, which are supported 
through the NSF MRSEC program (No. DMR-1719875). This work 
also used the CESI Shared Facilities partly sponsored by the NSF 
(Grant No. DMR-1338010) and the Kavli Institute at Cornell. The 
authors would like to thank Matthew R. Barone, Dr. Felix Hensling, 
Dr. Hanjong Paik, and Jiaxin Sun for helpful discussions.

data availability 
The data supporting the findings of this study are available within 
the paper. Additional data related to the growth and structural character-
arization are available at https://doi.org/10.34863/083j-x818. Any 
additional data connected to the study are available from the corre-
spending author upon reasonable request.

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SUPPLEMENTARY MATERIAL

Utilizing Complex Oxide Substrates to Control Carrier Concentration in Large-Area Monolayer MoS$_2$ Films

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Contents

- Section S1 - Experimental methods
- Section S2 - Raman and PL spectra of MoS$_2$ on complex oxides compared to bare substrates
- Section S3 - Growth uniformity of MoS$_2$ on complex oxide substrates
- Section S4 - Optical microscope images of uniform MoS$_2$ growth on a 2-inch wafer
- Section S5 - MINT calculations of electron transfer from oxide substrates to MoS$_2$
- Section S6 - Growth of MoS$_2$ on other oxide substrates
- Section S7 - Fast growth of MoS$_2$ on SiO$_2$ substrates
- Section S8 – Charge transfer between MoS$_2$ and Nb-doped SrTiO$_3$
Section 1 - Experimental methods

**MOCVD growth:** The growth of monolayer MoS$_2$ was carried out in a 95 mm (inner diameter) hot-wall quartz tube furnace. Molybdenum hexacarbonyl (Mo(CO)$_6$, MHC, Sigma Aldrich, 99.9% purity) and diethyl sulfide ((C$_2$H$_5$)$_2$S, DES, Sigma Aldrich, 98 % purity) were used as chemical precursors for molybdenum and sulfur. They entered the furnace in the gas phase due to their high equilibrium vapor pressure near room temperature. SAES in-line gas purifiers were used to remove residual water vapor and oxygen from ultra-high purity H$_2$ and Ar. The growth was performed at 600 °C with a total pressure of 5.98 Torr. The flow rates were 6 sccm for MHC, 0.3 sccm for DES, 1 sccm for H$_2$, and 1200 sccm for Ar, which were regulated by mass flow controllers (MFC). The growth time for continuous monolayer MoS$_2$ was 2.25 hours on SiO$_2$, 2.5 hours on SrTiO$_3$, 2.8 hours on LaAlO$_3$, and 3.3 hours on MgAl$_2$O$_4$. The growth time can be further reduced to 0.83 hours on SiO$_2$ by increasing the growth pressure and flow rates while still achieving uniformity over large-area substrates (see Figure S6). NaCl was loaded in the upstream region to act as a nucleation suppressant during the growth.

**Raman and Photoluminescence Spectroscopy:** The Raman and PL spectra were excited with a 532 nm laser under ambient conditions in a confocal Raman spectroscopy system (WITec-Alpha300 R). A 100× objective was used with a numerical aperture of 0.9 and a working distance of 1 mm. The laser power on the sample was 1 mW. Multiple spectroscopy measurements were taken across the substrates. The background signal measured on bare substrates under the same measurement conditions were subtracted for Raman and PL analysis.

**MINT calculations:** To calculate the charge transfer between MoS$_2$ and the substrates, we use *ab initio* mismatched interface theory (MINT) and perform density-functional theory (DFT) calculations of increasingly large MoS$_2$ clusters consisting of 3-, 6-, 12-, and 18-Mo atoms above a substrate layer. From finite-size scaling of the cluster calculations we predict charge transfer to MoS$_2$ of 3.30, 1.38, 1.15, and 0.97 (in units of 10$^{13}$ cm$^{-2}$) from SiO$_2$, MAO, LAO, and STO, respectively. The *ab initio* MINT calculations were carried out within the total-energy plane wave density-functional pseudopotential approach, using Perdew-Burke-Ernzerhof generalized gradient approximation functionals and optimized norm-conserving Vanderbilt pseudopotentials. Plane wave basis sets with energy cutoffs of 30 Hartree were used to expand the electronic wave functions. We used fully periodic boundary conditions and a $9 \times 9 \times 1$ $k$-point mesh to sample the Brillouin zone. Electronic minimizations were carried out using the analytically continued functional approach starting with an LCAO initial guess within the DFT++ formalism, as implemented in the open-source code JDFTx using direct minimization via the conjugate gradients algorithm. All unit cells were constructed to be inversion symmetric.
about \( z = 0 \) with a distance of \( \approx 60 \) Bohr between periodic images of the substrate, using Coulomb truncation to prevent image interaction.

Section S2 - Raman and PL spectra of MoS\(_2\) on complex oxides compared to bare substrates

**FIG. S1.** (a) Raman and (b) photoluminescence spectra of monolayer MoS\(_2\) grown on LaAlO\(_3\), SrTiO\(_3\), and MgAl\(_2\)O\(_4\), respectively, with comparison to signals from bare substrates.
FIG. S2. (a) Optical micrographs of MoS$_2$ grown on two identical substrates placed 8 cm apart in the MOCVD furnace. (b) Raman and photoluminescence spectra of MoS$_2$ on complex oxides measured from ten different locations at the positions marked in the schematic diagram.
Section S4 - Optical microscope images of uniform MoS$_2$ growth on a 2-inch SiO$_2$ wafer

FIG. S3. (a) Optical micrographs of MoS$_2$/SiO$_2$ taken at the locations marked on the schematic of the corresponding wafer.
Section S5 – MINT calculations of electron transfer from oxide substrates to MoS$_2$.

**FIG. S4.** Convergence of calculated electron doping $n_e$ of MoS$_2$ based on MoS$_2$ cluster-oxide heterostructures with increasing cluster size on (a) SiO$_2$, (b) MgAl$_2$O$_4$, (c) LaAlO$_3$ and (d) SrTiO$_3$. In each plot, the value of electron concentration of MoS$_2$ (marked by the red dashed line) is extrapolated from the power law fitting (black dashed line) of the calculated values for different MoS$_2$ cluster sizes (blue circles).
Section S6 - Growth of MoS$_2$ on other oxide substrates

FIG. S5. (a) Optical micrographs and (b) Raman spectra of monolayer MoS$_2$ grown on GGG, sapphire, and YAIO$_3$ substrates, respectively.

Section S7 - Fast growth of MoS$_2$ on SiO$_2$ substrates

FIG. S6. Scanning electron microscopy (SEM) images of MoS$_2$ grown on SiO$_2$ in different growth times. A continuous monolayer is reached at a growth time of 50 min.
Section S8 – Charge transfer between MoS$_2$ and Nb-doped SrTiO$_3$

We have grown MoS$_2$ on 0.05 wt.% Nb-doped (100) SrTiO$_3$ (Nb: SrTiO$_3$) and the charge transfer observed in Nb: SrTiO$_3$, which is electron doped that elevate its fermi level, is larger than undoped SrTiO$_3$. This is expected because due to the heavy electron doping, the Fermi level of the Nb: SrTiO$_3$ is set to be very close to the conduction band, far above the charge neutrality level of undoped SrTiO$_3$. Raman spectra [Fig. S6(a)] reveal that MoS$_2$ grown on Nb: SrTiO$_3$ shows a $A_{1g}$ peak position of 404.9 ± 0.04 cm$^{-1}$, which is 0.71 ± 0.06 cm$^{-1}$ smaller than that of MoS$_2$ grown on undoped SrTiO$_3$ [Fig. S6(b)], suggesting a greater electron transfer from Nb: SrTiO$_3$ to MoS$_2$.

FIG. S7. Raman of monolayer MoS$_2$ deposited on SiO$_2$, Nb-doped SrTiO$_3$ and undoped SrTiO$_3$ substrates. (a) Normalized Raman spectra of monolayer MoS$_2$. (b) Histograms and their Gaussian fittings of $A_{1g}$ peak position of MoS$_2$ on different substrates.

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