Synthesis of Epitaxial MoS$_2$/MoO$_2$ Core–Shell Nanowires by Two-Step Chemical Vapor Deposition with Turbulent Flow and Their Physical Properties

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ABSTRACT: MoO$_2$ nanowires (NWs), MoO$_2$/MoS$_2$ core–shell NWs, and MoS$_2$ nanotubes (NTs) were synthesized by the turbulent flow chemical vapor deposition of MoO$_2$ using MoO$_3$, followed by sulfurization in the sulfur gas flow. The involvement of MoO$_2$ suboxide is suggested by density functional theory (DFT) calculations of the surface energies of MoO$_2$. The thickness of the MoS$_2$ layers can be controlled by precise tuning of sulfur vapor flow and temperatures. MoS$_2$ had an armchair-type winding topology due to the epitaxial relation with the MoO$_2$ NW surface. A single ~ few-layer MoO$_2$/MoS$_2$ core–shell structure showed photoluminescence after the treatment with a superacid. The resistivities of an individual MoO$_2$ NW and a MoS$_2$ NT were measured, and they showed metallic and semiconducting resistivity–temperature relationships, respectively.

1. INTRODUCTION

One-dimensional nanomaterials (1D) have been synthesized from various atomic layer materials as nanotubes (NTs), such as carbon NTs (CNTs) from graphene and inorganic NTs (INTs) from transition metal dichalcogenides. They exhibit various functions due to their unique electronic structures arising from reduced dimensionality and symmetry. CNTs exhibit a distinct difference in their electronic structures depending upon the chirality of the winding patterns. Similarly, different electronic structures in the INTs are predicted by theoretical calculations. The scalable synthesis of the INTs is limited to MoS$_2$ and WX$_2$ (X = S, Se, Te) because Mo and W have volatile trioxides, which are essential to the formation of nanowires (NWs) of the suboxides that work as precursors of the INTs. INT formation has been reported using various methods such as the thermal reaction of metal oxide precursors with H$_2$S, thermal decomposition of (NH$_4$)$_2$MoS$_4$ chemical vapor transport (CVT), CVT with C$_60$, the reaction in a nanoporous Al$_2$O$_3$ template, heating of MoS$_2$ with a nucleation reagent (Mo$_2$S$_3$, SiO$_2$), and chemical vapor deposition (CVD) methods with a flowing carrier gas. We have reported the CVD synthesis of MoS$_2$ NTs using iron nanoparticles as catalysts. However, the synthesis of MoS$_2$ NTs remains a major challenge in terms of NT purity, uniformity, and mass synthesis. A recent report has solved the mystery of the poor yield of MoS$_2$ NTs without catalyst nanoparticles, in which the precursory suboxide NW was identified as less stable than the W counterparts. This is probably the reason why the study of the physical properties of the MoS$_2$ NTs is very limited compared to the WS$_2$ counterparts, which includes high current field effect transistors (FETs), superconductivity, intrinsic photovoltaic effects, and second harmonic generation. The applications of well-defined MoS$_2$ NTs in the literature are only FETs with limited mobility, optical absorption with structural resonance, and NO$_2$ sensors. There are few photoluminescence studies on MoS$_2$ NT despite that it is a unique probe and important application of MoS$_2$ monolayers, which have a direct band gap, distinctly different from the indirect band gap of the bulk MoS$_2$. Very recently, a PL of single-layer MoS$_2$NT was reported.

In this paper, we report the scalable synthesis of MoO$_2$ NWs by turbulent flow CVD, sulfurization of MoO$_2$ NWs by elemental S to form the MoO$_2$/MoS$_2$ core–shell structures with various thicknesses, and measurement of their physical properties, including the resistivity of the MoO$_2$ NWs and MoS$_2$ NTs and PL of the MoO$_2$/MoS$_2$ core–shell structures. A distinct difference from previous reports in terms of the synthesis is in not using toxic H$_2$S as the sulfur source and the use of the two-step reaction—formation of MoO$_2$ NWs and sulfurization for the thickness control of the MoS$_2$ shells. The surface energies of MoO$_2$ were calculated by density functional theory (DFT) to examine the involvement of the suboxides in the formation of the MoO$_2$ NWs.

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2. EXPERIMENTAL SECTION

2.1. CVD. A specially designed CVD apparatus was used to grow the MoO$_3$ and MoS$_2$ nanostructures, as shown in Figure 1: a three-zone tube furnace with a 28.6 mm diameter outer quartz tube, two 4.0 mm diameter inner quartz tubes with MoO$_3$ and/or sulfur, respectively, and a turbulence grid. This tube furnace allows independent control of the temperatures of the material sublimation site (zone A) and the reaction sites (zones B and C). Ar was used as the carrier gas, and two flows were employed to independently control the raw material flow and the main flow. Al$_2$O$_3$ and Si wafers with thermal oxide films were placed in the reaction zones (zones B and C) as substrates for the nanostructure growth. The most distinctive feature of this CVD system is the turbulence grating inserted in the main flow. The turbulence grid consists of about 10 stainless meshes (wire diameter 0.1 mm with 0.15 mm opening) (see Figure 1b), which were placed in the stainless holder, as shown in Figure 1c. The turbulence grid has been used in hydrodynamics to produce turbulence flow, which can effectively change the morphology of the products in CVD.

Turbulence flow is supposed to enhance the nucleation in the carrier gas because turbulence increases the collision rate of molecules to the substrate and produces enough energy for nucleation as a result.

After optimizing the conditions of the temperature, flow rate, and reaction time, the following condition was adopted for the MoO$_3$ NWs with a high aspect ratio. After setting the source material and substrates, the CVD system was thoroughly evacuated by a rotary pump. After the air was purged to about 5 Pa, the system was filled with Ar gas. The CVD experiment was then performed at atmospheric pressure.

The temperatures of the MoO$_3$ source (zone A) and reaction zone (zones B and C) were ramped from room temperature to 640 and 1000 °C, respectively, for 2.5 h. During this ramping period, there was no carrier gas flow through the MoO$_3$ source, while that of the main flow was 100 sccm. After the designed temperatures were reached, the flow rate through the MoO$_3$ source and the main flow was set to 10 and 500 sccm, respectively, for 2 h. The furnace was then naturally cooled to room temperature, and the carrier gas flow rates of MoO$_3$ and main flow were reduced to 0 and 100 sccm, respectively. As a result, the MoO$_3$ NWs were obtained. Next, to obtain MoS$_2$ NT, the sulfurization of the MoO$_3$ NWs was performed by setting the reaction temperature in zones B and C to 400–900 °C and the sulfur source temperature in the other furnace at 260 °C, with the duration time as a variable parameter.

MoO$_3$ (Kanto Chemical, >99.5%) and S (Kanto Chemical, >99.5%) powders were utilized as the source materials without further purification. Al$_2$O$_3$ (5 × 5 mm$^2$) (c-plane) and 1 × 1 cm$^2$ SiO$_2$ (285 nm)/Si wafers were used as the substrates. The substrates were cleaned by the RCA (Radio Corporation of America) cleaning method to remove any metal and organic contaminants. Sapphire was annealed in the air for 1 h at 1000 °C to obtain a surface with atomic steps.

2.2. Characterization. The products were characterized by an optical microscope, a SEM (JEOL JSM-6500F), Raman spectroscopy (Renishaw Invia with 532 nm excitation), and XPS (JEOL JPS-9200). The morphology and crystal orientations of the NWs were determined by a TEM (JEOL JEM-2010, 200 kV). Atomic-resolution images were obtained by an aberration-correction TEM (FEI Titan 300). Cross-section samples of the NWs were prepared by FIB-SEM (JEOL JSM-6500F). For the Raman and TEM measurements, we selected one NW from the original substrate and put it on a fresh SiO$_2$/Si substrate using a Micro Pickup System (MicroSupport Axis Pro) to remove possible molybdenum compound peaks not from the NW on the original substrate.

To fabricate electrical contacts for the transport measurements, we prepared the samples as follows: the samples were dispersed in ethanol and drop-coated on SiO$_2$/Si (semi-insulating, resistivity > 1 MΩ cm$^{-1}$) substrates. Poly(methyl methacrylate) (PMMA) on the substrates was used as an electron-beam (e-beam) resist. The 50-nm-thick gold electrical contacts were then fabricated by e-beam lithography and thermal evaporation techniques. The electrical resistance was measured using the standard four-point probe lock-in method, while the sample temperature was controlled from 300 to 2 K in a variable temperature insert, which was immersed in a liquid $^4$He bath.

2.3. DFT Calculations. DFT calculations were employed to estimate the surface energies of the various faces of a MoO$_3$ crystal to understand the growth mechanism of the MoO$_3$ NWs. We used the Vienna ab initio simulation package (VASP), which uses plane wave basis functions and pseudopotential with projector augmented waves (PAW). The Perdew–Burke–Ernzerhof (PBE) functional, the 8 × 8 × 1 k-point mesh, and a cutoff energy of 700 eV were used to calculate the (110), (011), (111), (111), (101), and (010) faces of the P21/n symmetry MoO$_2$. A slab model was used, in which a >10 Å thick vacuum was inserted between the slab, and two atomic layers near the surface were optimized. The surface energies $\sigma$ were obtained as

$$\sigma = \frac{1}{A} (E_{\text{slab}} - nE_{\text{bulk}})$$

where $A$, $n$, $E_{\text{bulk}}$, and $E_{\text{slab}}$ are the surface area of the slab, number of atoms in the slab, energy of the bulk, and energy of the slab, respectively.
3. RESULTS AND DISCUSSION

3.1. MoO₂ NWs. The MoO₂ NWs were selectively obtained by CVD with turbulence grating. MoO₃ was sublimated by heating to 640 °C, and then transported to the reaction site at 1000 °C by Ar gas, an inert gas, where it was reduced from MoO₃ to MoO₂ to earn entropy. The NWs typically had the dimensions of a 150 nm diameter and a 40 μm length (Figure 2a). Comparing the dimensions of the obtained NWs with those previously reported, although the diameter of the obtained NWs is larger, the length is so long that an unprecedented high aspect ratio of up to 270 was achieved. In contrast, the CVD without turbulence grating yielded only plate-like MoO₂ crystals, suggesting that turbulence grating is a powerful tool for controlling the morphology of the CVD products. The density of the MoO₂ NWs was higher at the edge of the substrate, indicating that local turbulence at the edge could also help to synthesize the NWs. The Raman spectra (Figure 2b) of the NWs were in good agreement with those of the reported bulk MoO₂ crystals, demonstrating the formation of MoO₂ NWs by this method.

The MoO₂ NWs were structurally characterized by TEM and selected area electron diffraction (SAED) measurements. The SAED pattern, obtained from the NW (Figure 3a,b), confirms the monoclinic MoO₃ phase and is consistent with previous reports. The spot spacing perpendicular to the growth direction was 2.8 Å, which corresponds to the (101) plane spacing of MoO₂ with space group P2₁/n. The spot spacing perpendicular to the growth direction was 3.4 Å in Figure 3b. The TEM image and the SAED diffraction pattern of a cross section of the MoO₂ NW are shown in Figure 3c,d. The SAED diffraction pattern of the cross section shows a face spacing of 3.4 Å along the crystal facets, suggesting that the NWs were surrounded by (111) or (111). It should be noted that MoO₂ has a unit cell similar to a hexagonal lattice (β ≈ 120°, a ≈ c), and the a:b ratio is special to give various orthogonal indices in the reciprocal space, including [101], [111], and [111] and [101], [111], and [010].

To examine the growth mechanism of the MoO₂ NWs, the surface energies of the various faces were calculated using DFT of the slab models. Table 1 summarizes the result. The details of the calculation and the optimized surface structures are explained in the Supporting Information, including Figure S1. Since a crystal face tends to grow larger when the surface energy is lower, we expected that the surface energy of (101), which is the cross section of the NW, would be high and the surface energies of (111) and (111), which form the sides of the NW, would be low. Table 1 does not show this tendency, resulting in a discrepancy between the calculated and experimental results. Therefore, it is considered that the 1D NW structure of MoO₂ was not formed based on thermodynamics governed by the surface energy. The feasible explanation is that the 1D NW structure was the result of the growth of excess oxide (such as the recently reported MoO₂-x), and it turned into MoO₂ NW. The temperature dependence of the resistivity, which will be discussed below, supports the existence of defects as expected from the stoichiometry conversion of the Mo oxides.

The electric conductivity of MoO₂ has not been measured on single crystalline samples, although the theoretical prediction and measurement on polycrystalline films were reported. Figure 4 shows the temperature dependence of the resistance of a MoO₂ NW measured in the temperature range from 300 to 2 K using the four-terminal method. In the temperature range from 300 to 50 K, the resistance showed a metallic temperature dependence, decreasing with decreasing temperature, whereas the resistance showed almost no temperature dependence in the temperature range below 50 K. This result is attributed to the dominance of thermal phonon scattering and impurity scattering at high and low temperatures, respectively. The metallic conduction behavior at high temperatures is consistent with theoretical calculations. Ideally, the sample should show a metallic conduction behavior down to very low temperatures, but this sample probably contains impurities. In previous studies, it has been reported that the metallic electronic structure of MoO₂ is sensitive to its stoichiometry and lattice strain and that thin MoO₂ films show a thermally activated temperature dependence even at around room temperature. Metallic conduction observed down to 50 K in the present study indicates that the MoO₂ NWs have excellent stoichiometry and alleviate strain.

It should be noted that the MoO₂ NW formation was greatly enhanced by introducing the turbulence grid and that the MoO₂ NWs are mostly found on the edge of the substrate (Figure 2a). This result shows that the nucleation of the NW precursor (such as MoO₃) is strongly affected by the carrier...
gas flow. The difficulty in the growth of the MoS$_2$ NTs in the previous reports probably comes from this strong influence of the hydrodynamic effects.

### 3.2. Formation of Armchair MoS$_2$ NT Shells from MoO$_2$ NWs

MoO$_2$/MoS$_2$ core–shell NTs were synthesized by a two-step CVD in which a sulfur stream was passed through the MoO$_2$ NW obtained by the above method. The conditions for the NT fabrication were optimized by fixing the sulfur heating temperature at 260 °C and the reaction time at 2 h and varying the reaction temperature from 400 to 1000 °C. In the Raman spectra of the NWs obtained at each reaction temperature (Figure 5), a peak characteristic of MoS$_2$ at around 382 cm$^{-1}$($E^\parallel_{2g}$) and 408 cm$^{-1}$ ($A_{1g}$) was observed, suggesting the formation of MoS$_2$ at all reaction temperatures.

In the Raman spectra of the products at temperatures below 600 °C, peaks derived from both MoS$_2$ and MoO$_2$ were observed, whereas only peaks derived from MoS$_2$ were observed for the products formed at higher temperatures above 600 °C. The region characteristic to MoO$_2$ (600–900 cm$^{-1}$) only showed a 736 cm$^{-1}$ peak corresponding to MoO$_2$ below 600 °C. The EDS spectrum also shows residual O below 600 °C (Supporting Information Figure S2), which is consistent with the Raman spectra. These results suggest the formation of NWs with a MoO$_2$-MoS$_2$ core–shell structure at lower temperatures and completely sulfurized MoS$_2$ NTs at higher temperatures. Nevertheless, we found that the sulfurization process is not only governed by the reaction temperature, time (2 h), and S source temperature, but other factors such as room temperature, relative humidity of the environment, and the time to dry the reaction system by flowing Ar gas. We suspect a great impact of residual water molecules in the reaction system on the sulfurization reaction. Currently, we cannot control these factors, and the degree of the reaction deviates among the experiments. In the following, we show the results after confirming the reaction degree by TEM observations.

The TEM and SAED results of the NW structure proved this prediction. In Supporting Information Figure S3, the TEM image of the NW produced at 400 °C is shown. A layer structure surrounding the NW was confirmed. A few layers of the MoS$_2$ layered structure are formed surrounding the MoO$_2$ NWs. In addition, the diffraction pattern of MoO$_2$-derived P21/n was obtained in the SAED image, indicating that the crystal structure of the MoO$_2$ NW core is retained. Since MoO$_2$ is unlikely to evaporate at a reaction temperature of 400 °C, the formation of MoS$_2$ NT would proceed via a solid-gas phase reaction, i.e., MoS$_2$ is epitaxially formed after S gas is adsorbed on the MoO$_2$ NW surface.

NWs formed at 700 °C showed both the MoO$_2$/MoS$_2$ core–shell structure and MoS$_2$ NT formation deviating among experimental batches. The HRTEM image of the MoO$_2$/MoS$_2$ core–shell structure formed at 700 °C is shown in Figure 6.

The HRTEM image shows two to three MoS$_2$ layers surrounding the MoO$_2$ NW. The lattice spacing of the inner and outer portions of the NW corresponds to MoO$_2$ (2.8 Å, (101)) and MoS$_2$ (6.5 Å, (001)), respectively.

The distribution of Mo, O, and S was examined by EDS (Supporting Informan Figure S4). Mo was distributed throughout the structure, whereas O was mostly distributed in the center and S in the periphery. These results suggested the formation of a core–shell structure in which MoO$_2$ with a diameter of about 50 nm is covered by MoS$_2$ with a thickness of about 100 nm. The thicker MoS$_2$ in this sample compared to the core–shell structure formed at 400 °C suggests that the MoS$_2$ formation proceeds from the outside to the inside, using Mo oxide (in this case, MoO$_2$) as the raw material and the outer MoS$_2$ as a template. The synthesis at high temperatures

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**Table 1. Surface Energies of Various Faces of MoO$_2$**

| faces  | (011) | (101) | (110) | (111) | (001) | (001) |
|--------|-------|-------|-------|-------|-------|-------|
| surface energy/µm$^{-2}$ | 2.25  | 1.84  | 2.36  | 2.55  | 2.55  | 1.30  | 1.75  | 1.10  |

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**Figure 4.** Temperature dependence of resistance measured for MoO$_2$ NW. The inset shows the image of the sample.
allows S to easily penetrate between the MoO$_2$ and MoS$_2$, which probably results in the effective formation of multilayers of MoS$_2$.

We found that all samples showed SAED patterns corresponding to the armchair chirality of the MoS$_2$. Figure 7 shows the TEM and SAED images of the MoO$_2$/MoS$_2$ core–shell NW formed at 700 °C. The diffraction from the core is very weak, and the pattern shows a sixfold symmetry originating from the honeycomb structure of MoS$_2$. The observed electron diffraction spot spacing (2.7 Å) is in good agreement with the value estimated from the lattice constant of the MoS$_2$ crystal (2.74 Å). Considering the relative relationship between the NT growth direction and the electron diffraction pattern, the MoS$_2$ NTs have an armchair-type chirality, as we have reported for the MoS$_2$ NTs by FeO-catalyzed growth.

As for the mechanism of formation of the armchair-type NTs, we considered the epitaxial relationship between the MoO$_2$ NW surface and MoS$_2$ essential. MoO$_2$ has a pseudohexagonal lattice ($a_{MoO_2} = 5.55$ Å ≅ $a_{MoS_2} = 5.62$ Å and $\beta = 119.69 \sim 120^\circ$). The lattice matching of $(a + c)/2$ (one lattice constant of (101) surface) and $a_{MoS_2} = 3.16$ Å is +2%. Although the length of $b_{MoO_2}$ does not match with the lattice constant of MoS$_2$, this one-dimensional matching will stabilize the interface more than a mismatch in two directions in the van der Waals epitaxy. There are some reports regarding the MoS$_2$ NTs or MoO$_2$/MoS$_2$ core–shell structures (including nanoplates or nanoclusters) showing electron diffraction patterns. Various chiralities and orientations were reported, including zigzag, armchair, random, and vertical alignment of the MoS$_2$ layers on MoO$_2$(110). We consider that there are several reasons for the observations of armchair chirality in the present result. One is that the wide faces of the MoO$_2$ NWs are suitable for the planar growth of the MoS$_2$ layers, and van der Waals epitaxy comes into play. Another is that our experiment employs well-defined MoO$_2$ NWs prepared during the first step and very gentle sulfurization conditions with precautions to obtain reproducibility of the MoS$_2$ thickness as much as possible.

### 3.3. Structure and Stoichiometry of MoS$_2$ NTs after Complete Sulfurization

The cross-sectional TEM of MoS$_2$ NT formed at 700 °C is shown in Figure 8, where a cavity-like structure was observed; the EDS results confirmed the formation of the MoS$_2$NT with a hollow center, as no Mo, O, or S atoms were distributed in that region. Based on the HRTEM results, the face spacing of the formed MoS$_2$ layered structure was estimated to be 6.5 Å. This value is somewhat wider than the interlayer distance (face spacing (001)) of the bulk MoS$_2$ crystal (6.2 Å). We do not know the reason for this discrepancy, and the possible models include the strain of the folded layers and the adventitious intercalation of S or O atoms between the layers. A boundary structure with a different layer orientation was formed in the NT. The boundary line is formed toward the center of the NT, suggesting that the multilayered tube structure of MoS$_2$ progresses inward from the outer periphery. The volumes per Mo atom are 32.86 and 52.85 Å$^3$ for MoO$_2$ and MoS$_2$, respectively. This increased volume will pull out the materials and make the center void of MoS$_2$ NT. The stoichiometry of the aggregated MoS$_2$ NTs was evaluated by XPS (Figure S5 and Table S1 in the Supporting Information). By integrating the peaks and dividing them by the sensitivity factors, Mo:S = 1:1.83, indicating a sulfur deficiency.

At higher temperatures (800–1000 °C), the sulfurization reaction was complete. It was observed that a MoS$_2$ NT was surrounded by SiO$_2$, which probably came from the quartz reaction tube (Figure S6 in the Supporting Information). The chirality of the MoS$_2$ NTs was armchair.

### 3.4. Photoluminescence

It is expected that the low temperature and short reaction time enables the synthesis of the MoO$_2$ core–monolayer MoS$_2$ shell structures. Since the photoluminescence of MoS$_2$ is an indication of a monolayer or a few-layer thickness, we examined the photoluminescence of the samples fabricated under various sulfurization conditions. It was difficult to make MoS$_2$ layers thin enough for this purpose at a temperature equal to or higher than 700 °C. It was reported that the photoluminescence of the monolayer MoS$_2$ is significantly weakened by electron doping and it can be improved by depriving excess electrons by the treatment with superacid such as bis(trifluoromethanesulfonyl)imide (or trifluorosulfuric imide, TFSI, C$_3$H$_7$F$_4$NO$_2$S$_2$). Figure 9 shows the photoluminescence from the NW sample sulfurized at 600 °C. Before the TFSI treatment, no PL was observed. After the TFSI treatment (drop casting 0.02 M TFSI in 1,2-dichloroethane in an Ar glovebox and dried), a strong PL signal was observed. By peak decomposition by least-squares fitting, the peak areas of the PL A-exciton ($X_a$, 663 nm):B-exciton ($X_b$, 618 nm):trion (678 nm):bound exciton due to a defect site ($X_{bb}$, 698 nm) = 5:74:0:21. The negligible intensity of the trion peak is consistent with previous reports on the superacid
treatment of monolayer MoS$_2$ which eliminates the trion peak and increases the total photoluminescence intensity. Therefore, it is proved that the “MoO$_x$ core”-“a monolayer ~ a few-layer MoS$_2$ shell” structure was successfully fabricated by the present method, although defect sites ($X_{Mo}$) and PL-quenching electron doping existed without the TFSI treatment.

3.5. Conductivity Measurement. The temperature dependence of the resistance of a MoS$_2$ NW fully sulfurized at 900 °C was measured in the temperature range from 300 to 2 K by the four-terminal method (Figure 10). The NW sample was covered by a single crystalline BN sheet to prevent contamination after the contact fabrication. In the temperature range from 300 to 200 K, the resistance exponentially increased, typical semiconducting behavior, whereas below 200 K, the resistance increased almost linearly with the temperature. The apparent temperature dependence of the resistance is thought to be reduced due to carrier doping caused by the $S$ defects. The previous reports regarding MoS$_2$ NT FETs showed limited carrier mobilities ($<10^{-1}$ cm$^2$ V$^{-1}$ s$^{-1}$) when grown by sulfurization of the MoO$_x$, whereas rather high mobilities ($\sim$ 40 cm$^2$ V$^{-1}$ s$^{-1}$) were observed when grown by CVT. Reducing the defect concentration is an issue to be addressed in the future for high-performance semiconductor MoS$_2$NTs.

4. CONCLUSIONS

The MoO$_x$ NWs, MoO$_x$/MoS$_2$ core–shell NWs, and MoS$_2$ NTs were synthesized by turbulent flow chemical vapor deposition of MoO$_x$ using MoO$_3$ followed by sulfurization in sulfur gas flow. The involvement of MoO$_x$ suboxide is suggested by DFT calculations of the surface energies of MoO$_x$. The thickness of the MoS$_2$ layers can be controlled by the precise control of the sulfur vapor flow and temperatures. MoS$_2$ had an armchair-type winding topology due to the epitaxial relation with the MoO$_x$ NW surface. A single ~ few-layer MoO$_x$/MoS$_2$ core–shell structure showed photoluminescence after treatment with a superacid. The resistivities of an individual MoO$_x$ NW and a MoS$_2$ NT were measured and showed metallic and semiconducting resistivity–temperature relationships, respectively. The cross section of a MoS$_2$ NT showed a noncylindrical layer orientation, which might be responsible for the poor performance of the reported MoS$_2$ FETs. The improvement of the process parameters for the ideal structures will open the way for the quantum-mechanical properties arising from the NT structures.

■ ASSOCIATED CONTENT

* Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.2c05586. Optimized surface structure; EDS spectrum; EDS mapping; and XPS, TEM, and SAED measurements (PDF)

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Author Contributions

M.G. performed the growth and structural characterization of the samples (TEM, Raman) and wrote the first draft of this paper. I.Y. performed the DFT calculations. S.A. contributed to the photoluminescence measurement and data analysis. Y.S. performed the electrical measurement of the single NWs. Y.-L.C. contributed to the STEM characterization of the core−shell structures. T.Y. and T.S. designed present research and S.Y., T.Y., T.N., and T.S. prepared the samples (TEM, Raman) and wrote the first draft of this paper.

Notes

The authors declare no competing financial interest.

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