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Electrical Properties and Thermal Annealing Effects of Polycrystalline MoS₂-MoS_x Nanowalls Grown by Sputtering Deposition Method

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Abstract: Straightforward growth of nanostructured low-bandgap materials is a key issue in mass production for electronic device applications. We report here facile nanowall growth of MoS₂-MoS_x using sputter deposition and investigate the electronic properties of the nanowalls. MoS₂-MoS_x nanowalls become gradually thicker and taller, with primarily (100)-plane growth directions, with increasing deposition time. Nanowalls combine with nearby walls when a rapid thermal annealing (RTA, 200 °C–500 °C) process is applied. All samples have conventional low-bandgap semiconductor behavior with exponential resistance increase as measurement temperature decreases. The 750 nm-thick MoS₂-MoS_x nanowalls have a sheet carrier mobility of up to 2 cm²·V^{−1}·s^{−1} and bulk carrier concentration of ~10¹⁷–10¹⁹ cm^{−3} range depending on RTA temperature. Furthermore, perpendicular field-dependent magnetoresistance at 300 K shows negative magnetoresistance behavior, which displays resistance decay by applying a magnetic field (MR ratio in the −1 % range at 5 T). Interestingly, 400 °C RTA treated samples show a resistance upturn when applying an external magnetic field of more than 3 T. Our research suggests tuneability of MoS₂ nanowall size and mesoscopic electronic transport properties.

Keywords: MoS₂; nanowall; crystal growth; sputter deposition; MoS₂ polycrystal characterization

1. Introduction

Transition metal dichalcogenides (TMDs), including MoS₂, WS₂, NbSe₂, and VS₂, have been intensely researched because they exhibit bandgaps ranging from 0 to 2 eV. TMDs can be metals, semimetals, semiconductors, insulators, or superconductors based on the tuning of elemental composition and/or their structural parameters, including defects [1–4]. Recently, growth of TMDs, especially MoS₂, with successful wafer scale production has gained attention for electronic device applications [5–14]. Two-dimensional systems and nanostructured mass production are an important topic for high performance and functional device applications [7,11,15–21].

MoS₂, which consists of weakly bonded S-M-S sandwiched layers, is an interesting material for electronic devices [22–27], optical sensors [10,13,18,28–31], spintronic devices [32–35], and energy storage [15,17,19]. MoS₂ nanostructures are attractive due to their unique properties and structural characteristics for use in multiple applications, such

as sensors, biomedical probes, optical devices, energy conversion, and storage devices. However, large-scale growth while tuning their size, shape, and composition is still a challenge and few research groups have reported large-scale nanostructure growth methods. Tao et al. and Hussain et al. reported methods for an atomic layer and a few layers of MoS₂ large-area growth for application to electronic devices, respectively [6,26]. Wang et al. reported the development of a simple “bottom-up” method for directly grown and well-dispersed ultra-small MoS₂ nanodots [7]. Sari et al. reported direct growth of MoS₂ nanowalls on carbon nanofibers for supercapacitor applications [15]. Kim et al. reported a large area growth method of MoS₂ nanowalls for efficient photoelectric application [31]. Additionally, Alexaki et al. illustrated a different MoS_x nanocrystal production method [16]. Thus, MoS₂ nanostructured large-scale growth methods need to be widely developed and improved for future device applications.

In this study, we report direct growth of MoS₂ nanowalls via sputter deposition and show the possibility of tunable electronic transport through electromagnetic property analysis. The growth behavior of MoS₂-MoS_x nanowalls was studied by changing growth time and annealing temperature. All samples show exponentially increasing resistance depending on the annealing temperature decrease, which corresponds with conventional low bandgap semiconductor behavior. The 750 nm-thick MoS₂-MoS_x nanowalls have a sheet carrier mobility of up to $2\text{ cm}^2\cdot\text{V}^{-1}\cdot\text{s}^{-1}$ and bulk carrier concentration ranging from $\sim 10^{17}\text{--}10^{19}\text{ cm}^{-3}$, depending on the annealing temperature. Moreover, perpendicular field dependent magnetoresistance at 300 K, which displays resistance decay by applying a magnetic field, shows negative magnetoresistance behavior. Interestingly, 400 °C rapid thermal annealing (RTA)-treated samples show resistance upturn when applying an external magnetic field of more than 3 T. These results show the feasibility of electromagnetic property tuning through growth conditions and post treatment, such as an annealing process.

2. Materials and Methods

2.1. MoS₂ Nanowall Growth

MoS₂ nanowalls were grown on soda-lime glass substrates and SiO₂(400 nm)/Si wafers using a sputtering system. In our sputtering system, the distance between the substrate and the target is 95 mm. The base pressure of the sputter chamber was 8×10^{-6} mTorr. Deposition was carried out at a pressure of 10 mTorr by flowing Ar gas (25 sccm) with 100 W RF power and using a MoS₂ target (99.5 % purity, 4-inch diameter, 1/8-inch thickness) with a Cu backing plate while the substrate was rotated at a speed of 5 rpm and was at room temperature. MoS₂ nanowalls were grown on glass substrate or Si wafers with maximum diameters of 6 inches. For the investigation of the annealing effect, the as-grown MoS₂ nanowall samples underwent an RTA (RTP600, LAT Co. Ltd., Osan-si, Korea) process for 10 min at temperatures ranging from 200 to 500 °C in a vacuum.

2.2. Characterization of the MoS₂ Nanowalls

The size and thickness of the nanowalls were confirmed using field emission scanning electron microscopy (FE-SEM, Sirion 400, FEI, Waltham, MA, USA) and atomic force microscopy (AFM, DI-3100, Veeco, Plainview, NY, USA). The change in crystallinity of annealed samples was characterized using X-ray diffraction (XRD, D/MAX-2500, RIGAKU, Tokyo, Japan) and Raman spectroscopy (alpha 300R, WiTec, Ulm, Germany). All electrical measurements were performed at low pressure (below 0.01 Torr) in a PPMS (Physical Property Measurement System; Quantum Design, San Diego, CA, USA) for temperatures ranging from 100 to 300 K and with an external perpendicular magnetic field of up to 5 T. The Van der Pauw (VDP) geometry was used to connect the sample to the sample puck through copper wire bonded by Ag or paste. A Keithley 2636 source meter and Keithley 2182 nanovoltmeter were used to apply source current and to detect voltage via programmed LabVIEW software.

3. Results and Discussion

Figure 1a shows brief illustrates the MoS₂-MoS_x nanowall growth sequence. During sputter deposition on glass substrates, polycrystals of MoS₂-MoS_x formed as a thin film and nanostructured islands. Then, the nanostructured islands increase in size and height as the deposition progresses. As a result, high-density nanowalls are grown after a total sputtering deposition time of 3000 s, as shown in Figure 1. The average thickness of the MoS₂-MoS_x nanowalls are confirmed to be approximately 52 nm by the SEM and AFM analysis shown in Figure 1b,c (Figure 1b inset shows approximately 750 nm-tall nanowalls). In our system, nanowalls could be grown on 6-inch wafers. Supplementary Materials Figure S1 shows MoS₂-MoS_x nanowalls grown on a 6-inch SiO₂/Si wafer with a total thickness deviation across the wafer within 10 nm.

Figure 1. (a) Schematic illustration of the nanowall growth sequence. (b) Scanning electron microscopy (SEM) images and (c) atomic force microscopy (AFM) profile of the as-grown MoS₂-MoS_x nanowall surface after 3000 s of sputter deposition.

Images in Figure 2 indicate that step-by-step growth depends on deposition time. As illustrated in Figure 1a, a film with islands growing on the surface, with preferred growth direction, appears for a deposition time of 250 s (Figure 2a,b). Then, the nanowalls appear at deposition time of 500 s (Figure 2c,d). This suggests that the islands that are initially grown mainly grow in the vertical direction, rather than the horizontal direction, as the deposition time increases. For a deposition time of 1000 s, the thickness and height of the nanowalls were ~24 nm and 290 nm, respectively (Figure 2e,f). When deposition time was further increased to 3000 s, the thickness and height of the nanowalls increased to ~52 nm and 730 nm, respectively (Figure 2g,h).

To confirm the annealing effect of the MoS₂-MoS_x nanowalls, RTA treatment with different temperatures, ranging from 200 °C to 500 °C, was performed for 10 min each on fully grown nanowalls (3000 s deposition time). Figure 3 shows the RTA treatment dependency as a function of RTA temperatures. Figure 3a–d correspond to visual identification of RTA temperatures ranging from 200 to 400 °C by FE-SEM images. At an annealing temperature of 200 °C, it is seen that the spacing of the nanowalls slightly increases, as shown in Figure 3b. It seems that the nanowalls are partially recrystallized. The nanowalls not only combine with the nearest walls but begin to re-crystallize at an annealing temperature of 300 °C, as shown in Figure 3c. When the annealing temperature is 400 °C, nanowalls appear to fully re-crystallize and combine with each other to create a thin film, as shown in

Figure 3d. When the annealing temperature is increased further to 500 °C, the nanowalls fully melt and collapse, as shown in Figure S2b.

Figure 2. SEM images of step-by-step nanowall growth. Four different sputtering deposition times, (a,b) 250 s (c,d) 500 s (e,f) 1000 s, and (g,h) 3000 s, show sequential growth of nanowalls from islands to thicker and taller nanowalls. (a,c,e,g) are cross section images and (b,d,f,h) are top-view images.

Figure 3. Deformations of nanowalls according to the annealing temperature. SEM images of (a) as grown, (b) 200 °C rapid thermal annealing (RTA) treatment, (c) 300 °C RTA treatment, and (d) 400 °C RTA treatment samples.

To investigate the annealing effect of MoS₂-MoS_x nanowalls, we performed XRD and XPS measurements shown in Figure 4a and Figure S3. In the as-grown MoS₂-MoS_x nanowall sample, a strong peak in the (100)-plane is observed and a weak and broad peak is also observed corresponding to the (008)-plane, which indicates nanowall growth preference in the (100) direction (JCPDS Card No. 75-1539). As the annealing temperature increases to 400 °C, the peak in the (100)-plane is more enhanced and a new peak appears at the (002)-plane. This new peak is remarkably enhanced with increasing annealing temperature. When the temperature is further increased to 500 °C, a new peak occurs

again around 26.5° , which is assumed to be the (040)-peak of MoO_3 [6,36,37]. This can also be confirmed by XPS analysis. Figure S3 shows XPS narrow-scan spectra of the Mo 3d and S 2p before and after the annealing process. The MoO_x peak rapidly increases after 500°C annealing process. This result supports that the newly formed XRD peak after 500°C annealing is MoO_3 . We estimate that the sulfur atoms on the surface of the MoS_2 nanowalls evaporate in the RTA chamber during the high-temperature annealing process, resulting on sulfur vacancies on the MoS_2 - MoS_x nanowalls [38,39]. When the annealed sample is placed in atmosphere after the RTA process, the (040) peak of MoO_3 is generated by reacting with oxygen.

Figure 4. (a) X-ray diffraction (XRD) spectra and (b) Raman shift as a function of annealing temperatures.

For a better understanding of the annealing effect, we also performed Raman spectra measurements, as shown in Figure 4b. The two main Raman peaks corresponding to E_{2g}^1 and A_{1g} are clearly observed in all cases, depending on the annealing temperature. In the as-grown sample, two peaks are observed at 374.4 cm^{-1} and 411.2 cm^{-1} ; the frequency difference of these two Raman peaks is 36.9 cm^{-1} . After annealing at 200°C and 300°C , a slight blue shift is observed in the E_{2g}^1 peak. When the annealing temperature was increased to 400°C , the intensity of the peak is increased; the E_{2g}^1 peak is red-shifted (374.5 cm^{-1}) and the A_{1g} peak is blue-shifted (408.4 cm^{-1}). The frequency difference of the two Raman peaks is 33.9 cm^{-1} for this RTA temperature, which is less than that of the as-grown sample. This suggests that the crystallinity of annealed nanowalls is improved, which is in agreement with the XRD results in Figure 4a. Compared with single crystal monolayer MoS_2 peaks [32,40], the observed Raman peaks show a blue shift for E_{2g}^1 peaks and broadening at both peaks. The E_{2g}^1 peak is associated with in-plane motion of Mo and S, in opposite directions, and the A_{1g} peak is associated with out-of-plane motion of S atoms [16]. Similar shifts and broadening results have been reported in previous papers [16,17,20,21,34]. The blue shift and asymmetric broadening can be associated with several effects from structural parameters, defects, size, crystallinity, elemental composition, etc [16,21,40]. Our nanowalls have a polycrystal structure, different wall sizes, and possibly contain different elemental compositions, such as MoS_2 , MoS_x and MoO_x .

Next, the response of electrical properties to a perpendicular magnetic field were studied. Figure 5a shows sheet resistance trends as a function of measurement temperature. All resistance was measured while decreasing temperature and using the VDP geometry (5 mm-square samples). The data is displayed in log scale for both the temperature (x -axis) and sheet resistance (y -axis). As the sample temperature decreases, the sheet resistance behavior shows a higher-order trend, which has quadratic-like behavior in agreement with conventional semiconductor performance, for all samples. As shown in Figure 5a, the slope of the temperature dependent sheet resistance has the lowest value for the as-grown sample, which has the maximum value of residual resistance ratio (RRR) [$R(300\text{ K})/R(200\text{ K}) = 0.24$] among the four samples. The other RRR values were 0.18 [$R(300\text{ K})/R(200\text{ K})$] at 200°C

RTA and $0.18 [R(300 \text{ K})/R(200 \text{ K})]$ at 300°C RTA. The 400°C RTA sample already reached the voltage detection limit at $\sim 250 \text{ K}$.

The RRR can be strongly affected by the amount of impurities and other crystallographic defects. It indirectly represents the purity and overall quality of a sample [41,42]. The RRR value for 200°C and 300°C RTA samples indicate that they have better crystallinity than the as-grown sample. The 400°C RTA sample has different phases of crystallinity and conductance due to the melt-and-recrystallization procedure. Furthermore, the obtained sheet mobility (μ) and carrier concentration (n) show reasonable behavior of the higher mobility but lower carrier concentration at as-grown sample and lower mobility but higher carrier concentration at 200°C and 300°C RTA samples (Figure 5b). However, the 400°C RTA sample has both low mobility and carrier concentration, which may be due to changes in elemental composition and nanowalls structure by the recrystallization process. In this study, all samples show the independently grown nanostructures, which are not connected to each other, not the form of a film in which the grains are connected. This structural effect makes the long and complex pathway for carrier transport, which is the reason that all samples show lower mobility values compared to previous reports [6,26].

Figure 5. (a) Temperature dependent sheet resistance and (b) Hall mobility and carrier concentrations at 300 K for four nanowall samples (as grown and RTA at 200°C , 300°C , and 400°C for 10 min).

Furthermore, we studied magnetoresistance behavior as a function of perpendicular magnetic field sweep. All data were obtained using samples with a 5 mm-square geometry and four-probe measurements. Note that all magnetoresistance behavior is not smooth and shows less-quadratic trends because of the geometrical effect during the electromagnetic response measurement, which is caused by side carrier transport. As plotted in Figure 6, the magnetoresistance behavior mostly exhibits negative magnetoresistance behavior except for the 400°C RTA sample. Negative magnetoresistance represents higher defects, disorder, and complicated diffusive carrier transport in crystalline materials [43–46]. This negative magnetoresistance behavior observed in three samples, from as-grown to 300°C RTA, corresponds with high defect density caused by the nanowall structures. The 400°C RTA annealed sample shows decreased negative magnetoresistance and close to conventional magnetoresistance (quadratic behavior with increasing magnetic field originated by cyclotron motion) at the higher magnetic field regime (approximately more than 3 T). This dramatic change for the 400°C RTA sample is also in agreement with its film-like structure and phase transform due to the melt-and-recrystallization procedure. Thus, we can control electromagnetic response by tuning the post-annealing temperature and/or conditions.

Figure 6. Magnetoresistance behavior of samples (as grown, 200 °C, 300 °C, and 400 °C RTA for 10 min) at 300 K. Magnetoresistance values are normalized with ΔR divided by R (at 5 T).

4. Conclusions

We have shown the direct growth of MoS₂ nanowalls via a sputtering deposition method and indicated the possibility of electronic transport tuning by investigating MoS₂ nanowall electromagnetic properties. The MoS₂-MoS_x nanowall growth behavior was studied by changing growth time and the RTA process. All samples show exponential resistance increase with decreasing temperature, which corresponds to conventional low bandgap semiconductor behavior. The ~750 nm-thick MoS₂-MoS_x nanowalls have sheet carrier mobilities up to 2 cm²·V⁻¹·s⁻¹ and bulk carrier concentrations ranging from ~10¹⁷–10¹⁹ cm⁻³ depending on the RTA temperature. Moreover, perpendicular field dependent magnetoresistance at 300 K shows negative magnetoresistance behavior, which indicates resistance decay by an applied magnetic field. Interestingly, the 400 °C RTA samples show resistance upturn when applying an external magnetic field of more than 3 T. These results suggest the possible tuneability of MoS₂ nanowall size and mesoscopic electronic transport properties.

Supplementary Materials: The following are available online at <https://www.mdpi.com/article/10.3390/cryst11040351/s1>. Figure S1: Large area growth of MoS₂-MoS_x nano-walls on 6-inch wafer. Figure S2: Fully Melted and collapsed MoS₂-MoS_x nano-walls at an annealing temperature of 500 °C. Figure S3: XPS narrow scan spectra of MoS₂-MoS_x nanowalls before and after annealing at 500 °C.

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