

Full Length Article

Growth and microstructural evolution of WS₂ nanostructures with tunable field and light modulated electrical transport

Pawan Kumar, Viswanath Balakrishnan*

School of Engineering, Indian Institute of Technology Mandi, Kamand, Himachal Pradesh, 175005, India



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ABSTRACT

We report CVD growth of WS₂ nanostructures with the ability to control the evolution of 1D to 2D microstructural changes for light and field effect transistor applications. Detailed mechanistic growth sequences from WO₃ nanorod to nanotube, monolayer and pyramidal structures of WS₂ has been achieved using atmospheric pressure chemical vapor deposition (APCVD). Electron microscopy and Raman spectroscopy analysis showed the growth evolution of different nanostructures and their formation mechanism. Location specific growth of different WS₂ nanostructures can be achieved by drop casting dispersed WO₃ nanorods on required substrate. Layer dependent photoluminescence (PL) properties of WS₂ indicate the effect of quantum confinement induced radiative recombination and enhanced PL intensity in monolayer WS₂ provides suitability for nanoscale photodetector application. The fabricated device shows light as well as field modulated switching at ultra-low biased voltage in hybrid WS₂ nanostructure that contains 1D (nanotube)-2D (flake) interface. The demonstrated aspects of CVD growth and hybrid device characteristics provide opportunities to tune electrical transport of WS₂ nanostructures at low active power.

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1. Introduction

Single and few-layer two-dimensional (2D) material of transition metal dichalcogenides (TMDCs) display a wide variety of interesting and potentially useful thickness-dependent phenomena including band-gap tunability [1,2], large direction-dependent variations in intrinsic optical [3] and electrical properties [4–6]. Much of the earlier work in the field of TMDCs based 2D materials are focused on MoS₂ with aim to make prototype devices for applications in optoelectronics [7], nanoelectronics [8] and various sensor [9–11] and energy devices [12] by considering suitable direct optical band gap transition in monolayer variant. WS₂ shows band gap transition with decreasing number of layers from bulk, indirect band gap of 1.3 eV to monolayer, direct band gap of ~1.95 eV [1]. Direct band gap semiconductor, 2D WS₂ have attracted much attention due to high electron mobility [13,14], optical responsivity [15] and robust device oriented results [16]. Monolayer WS₂ constituted a sandwich of one atomic layer of W between two atomic layers of S through covalent W–S bonds is also have hexagonal symmetry with tremendous increment in electrical capabilities. The remark-

able luminescence enhancement of monolayer WS₂ also make it strong candidate for photo-diode and solar based devices [17,18].

Monolayer 2D material, especially TMDCs are being derived by various methods from mechanical and chemical exfoliation [19] to physical [20] (Thermal evaporation, Sputtering, MBE deposition) and chemical [[19],20,21] (ALD, CVD) deposition techniques. All the adopted techniques have several different process windows to achieve multilayer to single atomic thin layer. Most of the optimized process recipe [21,22] to make 2D materials rely on their oxide precursors especially in the case of chemical vapor deposition (CVD) technique. The raw oxide precursor having several micron order of particle size is usually taken for sulfurization/selenization to form MX₂ (M=Mo, W; X=S, Se) based 2D material. Typically, amount of precursor and process temperature are known to affect CVD growth of 2D materials, while how the microstructure of starting oxide precursor influence the growth is not reported in literature. Our hypothesis is that the microstructure of as utilized precursor such as WO₃ could be in such form having very high aspect ratio such that higher availability of surface area may aid efficient conversion into 2D sheet with larger domain size after sulfurization. In addition, if we can achieve efficient sulfurization of WO₃ precursor, then it might be possible to spatially control the WS₂ growth by introducing the WO₃ precursor in desired location. The key difference between direct conversion of precursor

* Corresponding author.

E-mail address: viswa@iitmandi.ac.in (V. Balakrishnan).

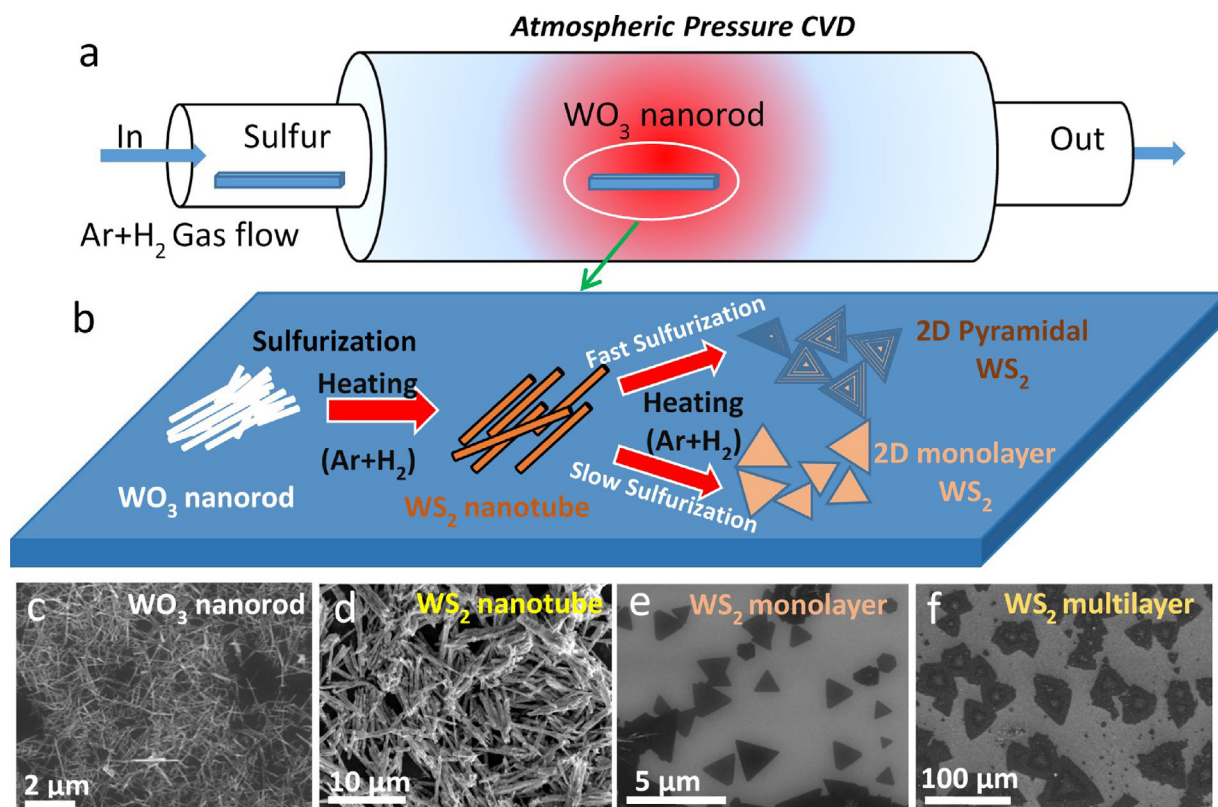


Fig. 1. Structural and phase evolution of WS_2 from 1D WO_3 nanorod by controlled sulfurization; (a) Schematic representation for CVD growth set up and used precursors and environment and corresponding (b) conversion reaction schematics; Secondary electron morphologies of WO_3 nanorod being used as precursor source and after conversion to WS_2 are (c) WO_3 nanorod, (d) WS_2 nanotube, (e) 2D monolayer WS_2 nanosheet and (f) 2D pyramidal multilayered WS_2 nanosheet.

with high aspect ratio and conventional vapor transport method utilizing bulk WO_3 powder is the energy involved in the growth process. For example, bulk powder of WO_3 has been employed as a main precursor with various methods [22,23] to grow 2D WS_2 using CVD method. Also, direct vapor phase deposition using WS_2 powder results in formation of few layer WS_2 nanostructures [24]. Moreover the conventional vapor transport methods needed complex conditions to sulfurize precursor material in presence of higher concentration of hydrogen gas with Ar carrier gas which complicate the precise control of WS_2 monolayer due to availability of limited process window.

In this report we have developed a facile route to grow large size single crystalline 2D monolayer WS_2 by efficiently converting hydrothermally synthesized WO_3 nanorods using modified CVD technique. We have first time optimized the process window to form 1D, 2D and hybrid nanostructures of WS_2 by controlling temperature and supersaturation conditions. The facile technique having location specific growth is helpful to achieve growth of 2D monolayer WS_2 flake with large domain size on Si_3N_4 membrane TEM grid by directly converting WO_3 nanorod. As grown monolayer 2D WS_2 flake shows strong PL for highly sensitive photodiode applications in nanoscale regime. Transient response in photoconductivity as well as back gated electrical characteristics reveal excellent performance in hybrid (1D+2D) WS_2 devices compared to WS_2 nanotube based only.

2. Results & discussion

Growth of WS_2 nanostructures from WO_3 nanorods in required location of the substrate has been carried out using home built atmospheric pressure CVD system as shown in schematic (Fig. 1a). Zoomed picture of central zone of CVD system have been pre-

sented using another schematic to indicate the process flow for the formation of different nanostructures of WS_2 in Fig. 1b. The as shown secondary electron image of WO_3 nanorod in Fig. 1c has been used as primary oxide precursor material which converted into WS_2 with different microstructures, 1D nanotube (Fig. 1d), 2D monolayer (Fig. 1e) and 2D pyramidal nanosheet WS_2 (Fig. 1f) by hydrogen reduction and simultaneous sulfurization. We drop casted WO_3 nanorods that are dispersed in water on desired substrate location and placed it in high temperature zone of CVD furnace. The optimized conditions has been achieved for the growth of different confined structures by varying the parameters like gas ($\text{Ar}+\text{H}_2$) mixture, flow rate as well reaction temperature. Along with the growth of 2D monolayer WS_2 , we have also increased the domain size of WS_2 to around $50\text{ }\mu\text{m}$ by allowing more growth time. In addition, supersaturation has been varied by changing sulfur evaporation rate to achieve layered, pyramidal growth of WS_2 nanosheet (Fig. 1f).

To control the WS_2 growth with the understanding of phase and microstructural evolution, we have performed numbers of growth by varying the final reaction temperature from 550°C to 900°C . Role of temperature demonstrates the sequential conversion of WO_3 nanorod to monolayer and pyramidal WS_2 nanostructures along-with hybrid morphologies at intermediate temperature and constant ($\text{Ar}+\text{H}_2$) environment. Fig. 2a–f show the sequential conversion of WO_3 nanorod to WS_2 nanotube, formation of 2D monolayer and layered pyramidal WS_2 nanostructure. The length and width of as synthesized WO_3 nanorods are found to vary from $1\text{ }\mu\text{m}$ – $10\text{ }\mu\text{m}$ and 10 nm – 50 nm respectively. Typically at around 550°C , WO_3 nanorods undergo phase transition leading to formation of WS_2 nanotubes while at 650°C , the overall microstructure consists of WS_2 nanotubes decorated with 2D flakes. With further increase of temperature, WS_2 nanotubes mostly get converted into

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