Synthesis of the Defect-Controlled Few-Layer WSe_2 via Pulsed Laser Deposition

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Abstract

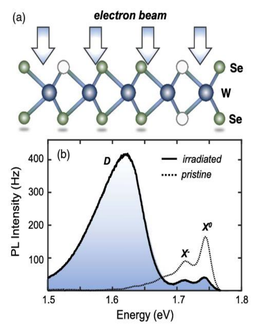
WSe_2, one of the transition metal dichalcogenide (TMD) materials, has been intensively interested owing to its fascinating properties, such as layer-dependent bandgap (~1.7 eV for monolayer and ~1.2 eV for bulk). Harnessing of layer-dependence could enable to tune not only the bandgap but also electrical and optical properties, making TMDs attractive. Therefore, properly controlling the number of layers of TMDs is beneficial for the design and optimization of novel devices.

Recently, many studies on the synthesis methods of TMD thin films have been reported. Among them, pulsed laser deposition (PLD) could achieve low growth temperature and short deposition time. The limited source transfer by PLD is advantageous for precise control of the number of layers. It is also easy for the fabrication of heterojunction with other materials through in situ deposition without the additional transfer process. However, it remains major challenges to synthesize high-quality stoichiometric thin films composed of micron-sized grains by PLD.

Our group successfully fabricated the WSe_2 thin films at low temperature via In situ chalcogen-evaporated PLD system. Using our PLD system, selenium vacancies in WSe_2 were compensated by flow control of selenium gas. Further, we obtained increased crystallinity of WSe_2 films by reducing the concentration of water and suppressing the unnecessary nucleation via the NaCl-assisted deposition process. By carrying out Raman spectral analysis and electrical measurements, we have confirmed our WSe_2 thin films show improved properties compared to those deposited in conventional PLD methods. We expect that our study will pave the way for the development of efficient TMDs synthesis method and be applied for the fabrication of next-generation electronic devices through follow-up studies.

Introduction

Defects in Transition Metal Dichalcogenides (TMDs)



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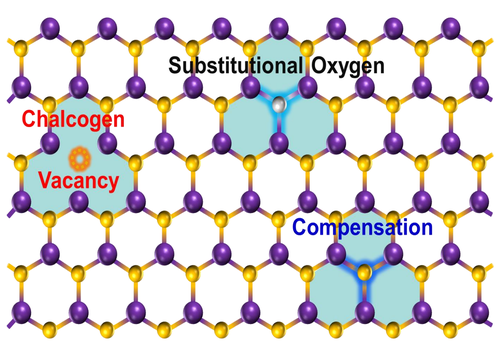

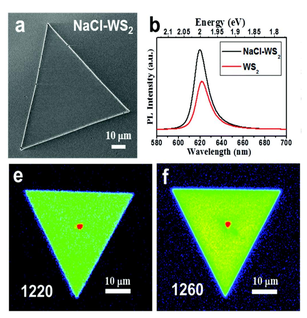

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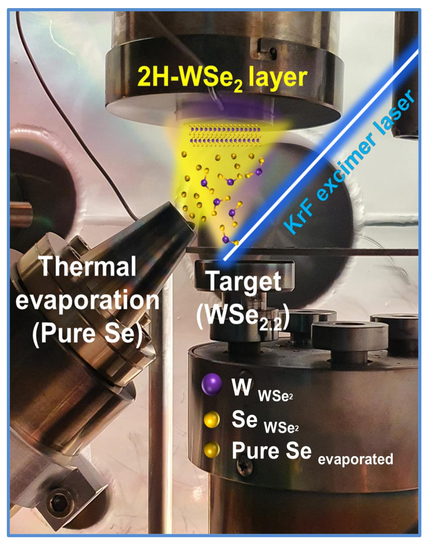
- **Chalcogen vacancy and halogen atom (ex. Cl),** as zero-dimensional quantum dot sites, can confine excitons as well as enhance the optical properties.
- **Substitutional Oxygen** can enhance the conductivity and photoconductivity of TMDs materials.

Experiments

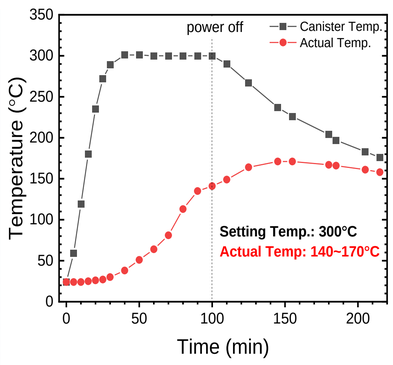
In situ Chalcogen-Evaporated Pulsed Laser Deposition (PLD)

- Challenges of injection of the pure chalcogen flux into UHV chamber
  -> Due to the solid state of chalcogen elements at room temperature,
  a gas line blockage occurred when the chalcogen gas evaporate from the source.
- To overcome this issue, we specially designed the in situ chalcogen-evaporated PLD system.

Schematic of PLD system



Selenium flux control by evaporation temperature

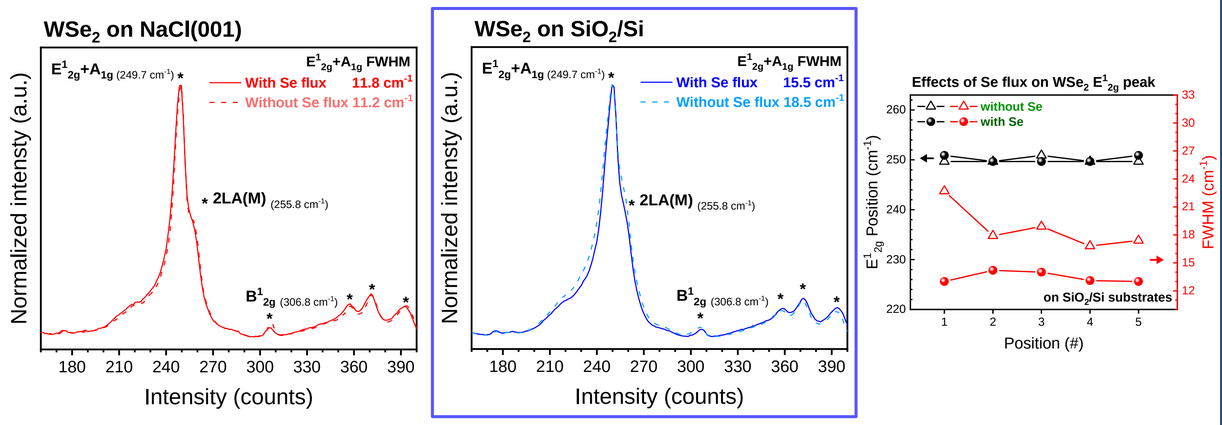


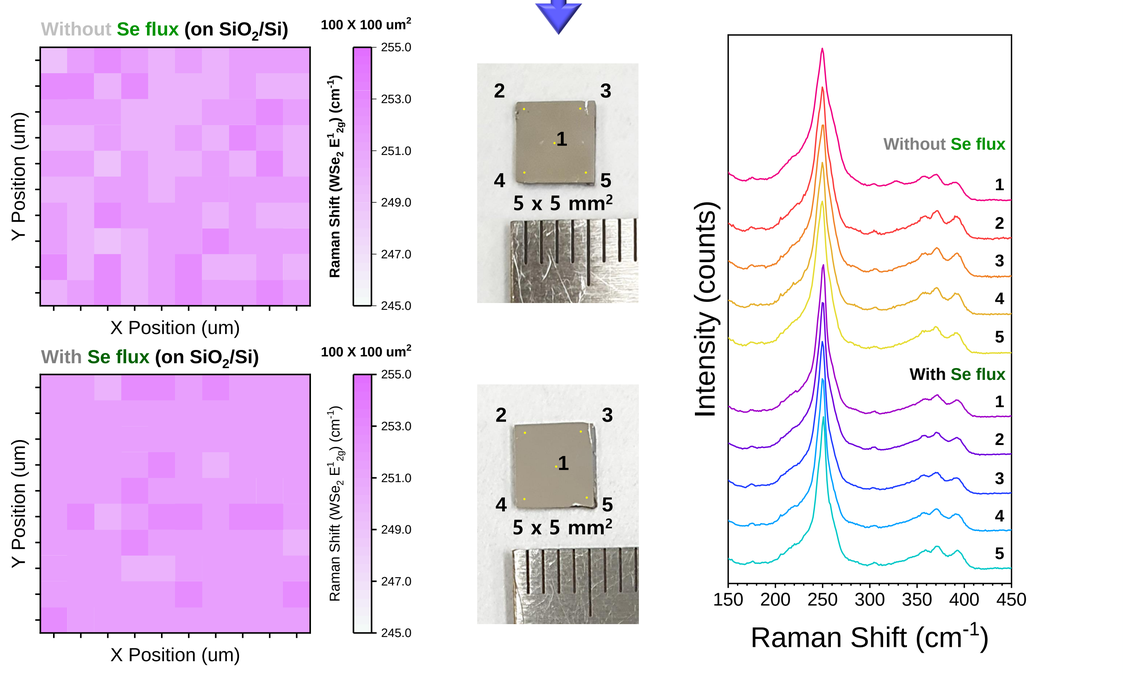
- **Setting Temp. vs. Se flux**
  exposed SiO_2/Si subs. for 20 min
  ramping time: 90 min (for heating)
  Se melting point: 220.1℃ at 760 Torr

400℃ : 1300 ~ 1000 nm/min
350℃ : 48.5 ~ 23.0 um/min
300℃ : 8.5 ~ 4.0 nm/min (In this work)
+ WSe_2 growth rate: 0.252 nm/min

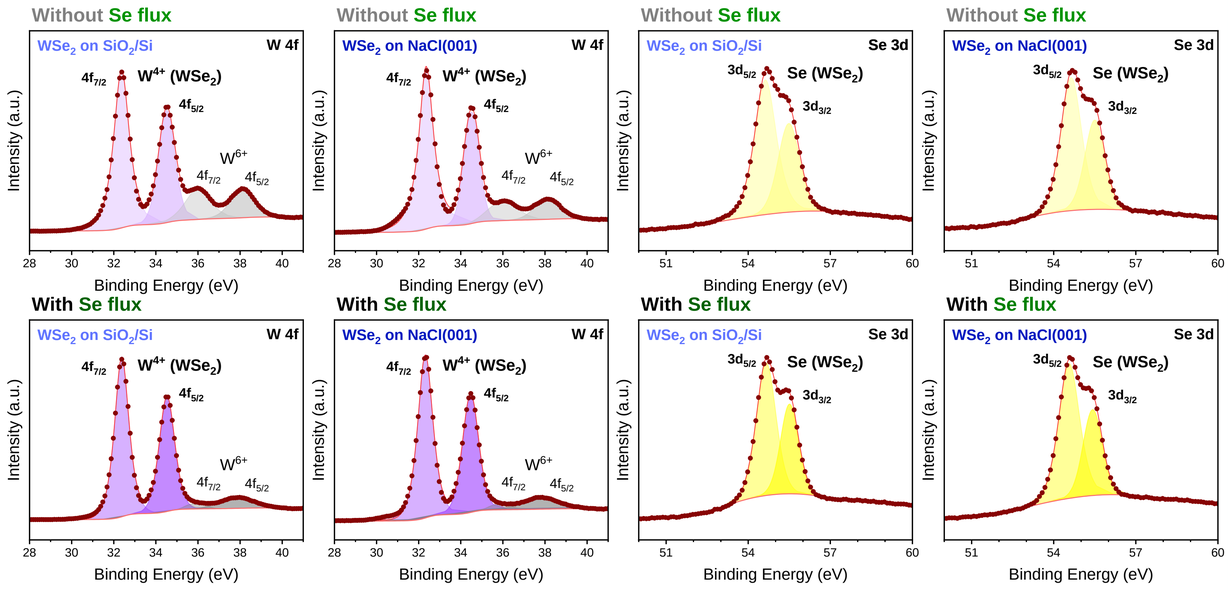
- By directly controlling the temperature of canister containing selenium, but it is slightly different from the actual selenium temperature.
- Selenium flux was estimated through the selenium growth rate as a function of temperature.
- The phases of WSe_2 on SiO_2/Si and NaCl(001) were confirmed.

Raman spectrum analysis





X-ray Photoelectron Spectroscopy (XPS)



Substrates	Se flux	Se/W Nominal ratio	W^{6+} (WO_x) ratio
SiO_2/Si	**With Se flux**	2.60	**10.54%**
	Without Se flux	2.19	24.30%
NaCl(001)	**With Se flux**	2.52	**8.22%**
	Without Se flux	2.26	19.12%

Conclusion

1. We synthesized WSe_2 films on SiO_2/Si and NaCl(001) substrates via in situ chalcogen-evaporated PLD which can compensate the selenium in WSe_2 layer.
2. More uniform WSe_2 films were obtained with additional Se flux on SiO_2/Si, while the effect of Se flux on WSe_2 films on NaCl(001) is not dominant at this stage.
3. Measuring the electrical and optical properties to evaluate the precise effect of Se flux on WSe_2 thin films is ongoing.

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