

# Synthesis and characterization of 2D NbSe<sub>2</sub>

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**Abstract:** Two-dimensional van der Waals (vdW) materials possess novel physical properties and promising applications. A wide range of 2D vdW materials having been obtained via the chemical vapor transport (CVT) method. In this work, we develop the controllable growth method of 2H-NbSe<sub>2</sub> single crystals via the CVT method. The quality of fabricated crystals was characterized by X-ray diffraction, and electron dispersive spectrometry (EDS) measurements. Crystals of the best quality were successfully grown under selected temperature/time schedule.

**Keywords:** TDMC, CVT (CHEMICAL VAPOR TRANSPORT), NBSE2, SINGLE CRYSTAL, STRUCTURE

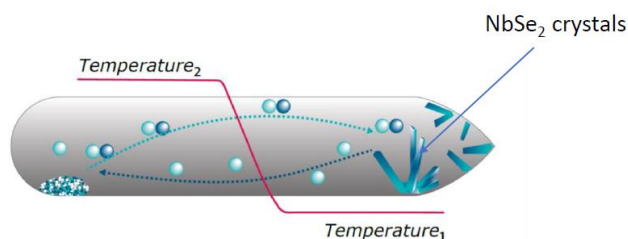
## 1. Introduction

In recent years, interest in the synthesis and applications of transition metal dichalcogenides MX<sub>2</sub> (M: Mo, W, X = S, Se, Te) nanomaterials has steadily grown because of their unique structure and superior properties [1]. Transition metal dichalcogenides have a sandwich interlayer structure formed by the stacking of the X–M–X layers, which are loosely bound to each other only by van der Waals forces and are easily cleaved. Moreover, MX<sub>2</sub> exhibits unique physical, optical and electrical properties correlated with their layered structure. In addition, their electronic structure is such that band-edge excitation corresponds largely to a metal centered d-d transition. Owing to these features, TMDCs materials have numerous applications such as sensors, detectors, solid lubricants, catalysis, electrocatalysis, high-density batteries and optoelectronic devices [2].

Niobium diselenide (NbSe<sub>2</sub>) is transition metal dichalcogenide layered compound, and it is exceptionally attractive because of its super conductivity and the formation of a charge-density-wave (CDW) state [3]. 2H-phase niobium diselenide (2H-NbSe<sub>2</sub>) is a superconducting vdW (Van der Waals) crystal with charge density wave (CDW) and Weyl semimetal properties. It has a superconducting critical temperature of ~7.8K and charge density wave behavior at ~34K. It has layered structure (lamellar) with weak interlayer coupling. NbSe<sub>2</sub> displays metallic and superconducting behavior.

## 2. Experimental details

Chemical vapour transport (CVT), a technique / process where a condensed phase, typically a solid is volatilized in the presence of a gaseous reactant (transport agent) and deposited elsewhere in the form of crystals. Typical transport agents include halogens and halogen compounds. The setup consists of a 2-zone furnace, the reactant and transport agent sealed in an ampoule. The various parameters that must be optimized for a successful CVT are growth temperature, transport direction, rate of the mass transport, choice of the transport agent and the free energy of the reaction.



**Fig. 1** Schematic of the experimental setup.

Single crystals of NbSe<sub>2</sub> were grown by a chemical vapour transport technique using iodine as transporting agent. A 5 gm mixture of Nb (purity: 99.95 %, Alfa Aesar, Germany) and Se (purity: 99.99 %, Alfa Aesar, Germany) was filled in a dried ampoule. Iodine of the quantity 5-6 mg/cc of the ampoule volume was sealed in the thin capillary and placed in the ampoule as transporting agent. Then the ampoule was sealed at the pressure of 10<sup>-5</sup> torr. The sealed ampoule

was introduced into a two-zone furnace at constant reaction temperatures to obtain the charge of NbSe<sub>2</sub>. The charge so prepared was rigorously shaken to ensure proper mixing of the constituents and kept in the furnace again, under appropriate condition to obtain single crystals of NbSe<sub>2</sub>. The experimental conditions for crystals growth are shown in Table 1.

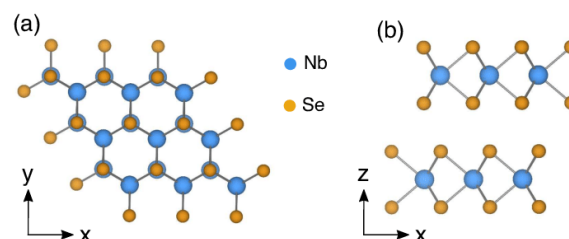
**Table 1:** Growth parameters for NbSe<sub>2</sub> single crystals using chemical vapor transport technique.

	Length (mm)	ID (mm)
Ampoule dimensions	300	25
	Hot zone (K)	Cold zone (K)
Temperatures	1123	1053
	Time (hours)	
Growth time	240	

Powder X-ray diffraction (XRD) patterns were gathered within the 2 $\theta$  range from 20 to 80° with a constant step 0.02° on a Bruker D8 Advance diffractometer with a Cu K $\alpha$  radiation and LynxEye detector. Phase identification was performed with the Diffracplus EVA using the ICDD-PDF2 Database.

## 3. Results and discussions

NbSe<sub>2</sub> is Molybdenite-like structured and crystallizes in the hexagonal P6<sub>3</sub>/mmc space group. The structure is two-dimensional and consists of two NbSe<sub>2</sub> sheets oriented in the (0, 0, 1) direction. Nb<sup>4+</sup> is bonded to six equivalent Se<sup>2-</sup> atoms to form distorted edge-sharing NbSe<sub>6</sub> pentagonal pyramids. All Nb–Se bond lengths are 2.62 Å. Se<sup>2-</sup> is bonded in a 3-coordinate geometry to three equivalent Nb<sup>4+</sup> atoms.

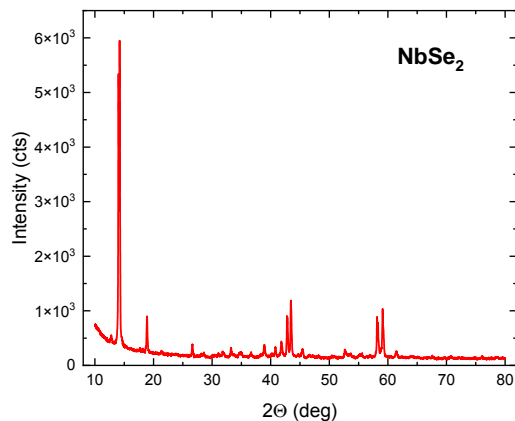


**Fig.2** (a) Crystal structure of bulk NbSe<sub>2</sub> illustrating the (a) top view and (b) side view of the structure. The x, y and z axes denote the cartesian axes

Many methods have been developed to improve the crystalline quality of superconducting NbSe<sub>2</sub>, for example: (a) exfoliation from bulk single-crystal NbSe<sub>2</sub> by the electrochemical exfoliation method [4]; (b) growth by salt-assisted CVD [5]; (c) growth by molecular beam epitaxy (MBE) under ultra-high vacuum [6]; and (d) growth of a wafer-scale NbSe<sub>2</sub> film in oxygen-free conditions by a two-step vapor deposition method [7]. However, most of the above preparations of NbSe<sub>2</sub> have either a lot of point defects or a small grain size, which reduces its environmental stability or T<sub>c</sub>. Therefore, it is still challenging to develop a reliable strategy to

grow 2D NbSe<sub>2</sub> with a large area, high crystalline quality, and high repeatability.

The CVT single crystals growth was chosen due to the advantages of bulk single crystals as large crystals to measure basic properties: structural, optical, electrical; nearly-perfect state of material: purity, no grain boundaries, less structural defects, stress, etc. The tunability of bulk properties: control of phase stoichiometry, doping level, alloy composition is readily obtained. The layered crystals as NbSe<sub>2</sub> are easily of exfoliation and stacking of heterostructures.



**Fig.3** XRD patterns of NbSe<sub>2</sub> single crystal

The crystallinity, structure, and phase purity of the prepared samples were confirmed by XRD and EDS. As shown in Fig. 1a, all observed diffraction peaks can be exactly indexed to the hexagonal phase of NbSe<sub>2</sub> with lattice constants  $a = 3.445 \text{ \AA}$  and  $c = 12.55 \text{ \AA}$  (PDF No. 65-7464). No characteristic peaks were observed, and the sharp diffraction peaks imply a good crystallinity of the obtained NbSe<sub>2</sub> products under current synthesis conditions. The Energy-dispersive X-ray Spectrometry (EDS) result demonstrates that the samples are consisted of elements Nb and Se, while no other elements were observed.

#### 4. Acknowledgements

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