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EXPERIMENTAL TECHNIQUES AND DATA ANALYSIS FOR 2D SEMICONDUCTOR (MoS2, WS2) AND LAYERED g-C3N4

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ABSTRACT

In this chapter, the details of growth of 2D- semiconductors (MoS2, WS2) and layered g-C3N4 has been described. The working principle and method of data analysis of various characterization techniques adopted in the present work has been described. An account of the procedure followed for device fabrication. 2D- materials exhibit different physical and chemical properties relative to their bulk counterpart. To achieve the required number of layered materials, there is a need to choose a specific growth technique with defined parameters. CVD technique gives us the flexibility to fine-tune different growth parameters to achieve controlled number of layers with a large coverage area and fewer grain boundaries. An optical microscope is used to analyze the grown sample and estimate the size of crystals with their coverage area. FESEM was used to archive images with higher magnification. Raman and photoluminescence (PL) spectra were recorded using a laser Raman spectrometer from the same spot. Spectra were further analyzed to identify the number of layers. The thickness of the grown monolayer sample is obtained using Atomic force microscopy (AFM). For optoelectronic device fabrication, we used shadow masking techniques along with thermal evaporator techniques to fabricate electrodes over the grown sample. The optoelectronic response of fabricated devices is obtained using an electrical probe station attached to the source meter and different laser excitation. All the analytical techniques used has been briefly discussed with their basic features and capabilities along with their working principles.

KEYWORD: Material, (MoS2, WS2) and layered g-C3N4, Semiconductor

INTRODUCTION

Growth of 2D – Materials using Atmospheric pressure chemical vapor deposition (APCVD)

In this section, we have introduced the atmospheric pressure chemical vapor deposition system (APCVD). APCVD processes do not require a vacuum system, they commonly give us the ability to control film growth by widely varying parameters like amounts of precursors, substrate, the position of the substrate and gas flow rates, making CVD unique among other deposition techniques.

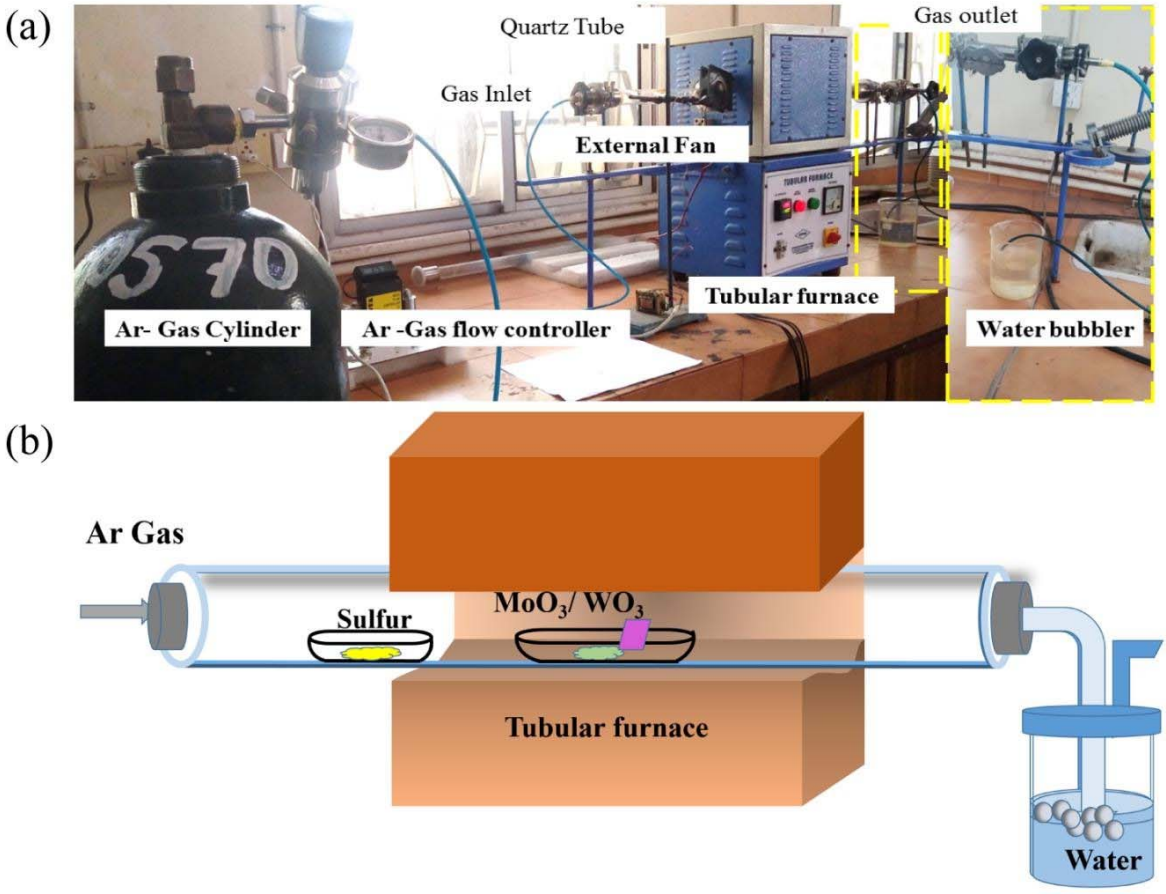
Substrate preparation and cleaning

Substrate preparation is an essential step in any growth process, such as the selection and cleaning of the substrate surface is a preliminary part of the growth process, 285 nm SiO2/Si

wafer (nanoshel) was used as a substrate for the growth of two –dimensional layered materials. The substrate was cut into the required dimensions. Substrate cleaning is necessary to avoid contamination and to obtain the desired properties in the grown sample [238]. For every set of experiments, the substrate was cleaned sequentially, using the isopropyl alcohol, deionized water and ethanol through the sonication bath process for 15 minutes each. After sonication, the substrate was washed with deionized water and then dried using an argon gas gun and was put inside the hot oven at 100 ℃ for 10 minutes.

Description of Custom made APCVD

Fig. 2.1(a) shows the single zone custom made atmospheric pressure chemical vapor deposition (APCVD) system developed at BIT Mesra, Ranchi India. It has been used for the growth of MoS2 and WS2 for the present work. In this CVD setup, the tubular furnace with a



quartz tube length of 120 cm and an outer diameter of 4.5 cm was used.

Figure 2.1: (a) Self–assembled Atmospheric pressure chemical vapor deposition setup for Two- dimensional growth. (b) Schematic diagram of APCVD with the arrangement of precursors.

Monolayer MoS2 growth parameters

Growth of MoS2 monolayer was done using highly pure molybdenum trioxide MoO3 (99.9% Sigma Aldrich) and sulfur powder (99.9 % Sigma Aldrich) was used as precursors. Fig. 2.1(b) shows schematic of the CVD setup. The temperature profile of both precursors (MoO3 and S powder) during the complete cycle of growth has been shown in Fig. 2.2(a). Fig. 2.2(b) shows gas flow rate for the entire experiment duration. The polished surface of SiO2/Si substrate faces downward to the precursor above the MoO3 powder. Precursors and substrate were placed inside the CVD furnace as schematically shown in Fig. 2.1(b) separated at 19 cm. Growth was carried out at 750 °C for 5 minutes using a single zone CVD furnace at atmospheric pressure. During growth, the amount of MoO3 was varied in the range of 10 mg to 30 mg referred as SET-I keeping the gas flow rate constant at 100 sccm and the weight of sulfur at 200 mg. In SET-II, weight of the second precursor sulfur was varied in the range of 100 mg to 500 mg keeping other conditions like weight of MoO3 (15 mg) and gas flow rate (100 sccm) fixed. The carrier gas flow rate (Argon) was varied in the range of 50 sccm to 300 sccm referred to as SET-III, while keeping the other two factors MoO3 and sulfur fixed at 15 mg and 200 mg respectively. Finally, the variation of growth time duration from 5 to 15 minutes with an interval of 5 min, keeping other optimized parameters like MoO3 (15 mg), Sulfur (200 mg) and gas flow rate (100 sccm) at 750 °C referred to as SET-IV. All the SET of experiments are listed in tabular form Table 2-1 below.

CONCLUSION

Electrical measurement of nano-scale devices and structures requires skills and hardware

to make nano-contacts. Such measurements have been difficult for number of laboratories due to cost of probe station and nano-probes. We demonstrated feasibility of assembling low cost probe station using USB microscope (US $ 30) coupled with in-house developed probe station. The effect of shape of etching electrodes on the geometry of the microprobes developed were elucidated. The variation in the geometry of copper wire electrode was found to affect the probe length (0.58 mm to 2.15 mm) and its half cone angle (1.4° to 8.8˚). As developed probes were used to make contact on micro patterned metal films and was used for electrical measurement along with semiconductor parameter analyzer. These probes show low contact resistance (~ 4 Ω) and follows ohmic behavior. These probes has potential to be used at laboratories involved in teaching and multidisciplinary research activities and scanning probe microscopy. 2D Semiconductors growth using Single zone Atmospheric pressure chemical vapor deposition (APCVD) technique for the first time, we have been able to demonstrate possibility of growth of monolayered semiconductors using single zone APCVD. Prior to this work, two-zone or, multiple zone CVD systems were used for growth of MoS2 and WS2. CVD system was custom designed and assembled making it one of the cost effective system.

C. Growth of monolayered MoS2 and WS2 with crystallite size ~ 80 □m and coverage

area > 92 % The various governing factors like metal and chalcogen weight fractions, carrier gas flow rate, growth duration and temperature affecting nucleation and growth were

systematically varied to achieve monolayered MoS2 and WS2 with large coverage area. The metal precursor concentration was found to be responsible for the self-seeding process and it decides the nucleation site and coverage area. Self-seeding nucleation and its amount was found to decide the nucleation density. The chalcogenide precursors (Sulfur) result in the increase of coverage area for 2D monolayer growth. Sulfur was found to play no role towards creation of nucleation sites, but higher concentration of Sulfur accounts towards formation of multilayered structures.

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