

Controllable Growth of Hexagonal BN Monolayer Sheets on Cu Foil by LPCVD

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Abstract

Hexagonal boron nitride (*h*-BN) has 2-dimensional honey comb structure similar to graphene and it is a wide band-gap material. Although, *h*-BN sheets has been achieved on various metal substrates. Here, we demonstrate the growth of *h*-BN monolayer sheets on *Cu* foil by low pressure CVD method. Two heating zones were used to control the precursor generation and main reaction, respectively. Wafer-scale *h*-BN monolayer film was synthesized uniformly on *Cu* foil. The monolayer thickness is determined to be about 0.46 nm and the optical band-gap is ~ 6.0 eV. Extended applications of *h*-BN layer in conjunction with graphene could now be available for electronic devices.

Key words:

Chemical Vapor Deposition method (CVD), Copper Foil, Hexagonal Boron Nitride (*h*-BN), Monolayer Sheets, Selected Area Electron Diffraction (SAED);

1. Introduction

The *h*-BN has 2-dimensional honey comb structure similar to graphene with only $\sim 1.7\%$ mismatch [1][2]. In *h*-BN lattice, nitrogen and boron atoms are alternatively bonded with sp^2 hybridization, which provides an extremely robust mechanical strength and hardness [3]. In contrast, the weak sp^3 bonding between stacking *h*-BN layers brings the possibility of fast separation. Besides, unique physical and chemical properties of *h*-BN, such as high chemical inertness, high temperature stability [4], flat surface without dangling bonds, charged impurities [5][6] and wide bandgap ($E_g = \sim 6$ eV) [7][8][9] makes it one of the most promising 2D material for broad applications. For example, it can be fabricated into a solid lubricant at extreme temperature, UV-light emitters, insulating filler in composite materials, and even substrate or dielectric material for 2D electronic devices, especially for those which based upon the use of graphene as field effect transistors (FETs) for high performance and low power consumption [10][11][12][13][14][15]. So, it requires a controllable growth of the scalable size of *h*-BN nanosheet on different substrates for specified combination with various devices. Recently, *h*-BN has been reported to be grown on a variety of supporting substrates such as *Cu* [16][17][18], *Pt* [19][20], *Ni* [21][22][23] and *Si/SiO₂* [24][25][26][27][28][29][30].

A largest area ($35 \mu m^2$) hexagonal domain in a single crystal of *h*-BN is reported for electro polished *Cu* foil [31]. Freshly, various techniques are in practice for automatic preparation of thin nano-sheets of *h*-BN, in which (1) The ion beam sputtering deposition method, by this method, both triangular and polygonal *h*-BN domains were achieved on *Cu* foils [32][41], (2) The “pulsed CO_2 laser deposition” (PLD) technique was used to deposit *h*-BN film on Silicon wafers of size ($1 \times 1 cm^2$) or Molybdenum [33][41] and (3) The small to large-area *h*-BN thin films were developed by “chemical vapor deposition” (CVD) method [34][41]. In this work, the growth of *h*-BN monolayer is systematically studied on copper (*Cu*) substrate by “Low Pressure” CVD (LPCVD) method. Specified growth condition was optimized for copper foil. Wafer-scale *h*-BN monolayer synthesized uniformly on the copper *Cu* foil.

2. Experimental Details

The LPCVD system in our work includes two individual heating zones, as shown in Fig.1. Ammonia borane (borazane) powder was used as precursor source, for this source we used a quartz boat and placed it, at precursor heating zone. The substrate was placed downstream in the reaction zone, which usually could be heated up to high temperature i.e. greater than $1000^\circ C$. Before the BN growth, the substrate was annealed at around $800^\circ C$ with the flow of mixed hydrogen and argon gases for 10~20 min in order to remove the oxides on the substrate surface. Then, the temperature of the reaction zone was increased up to $1050^\circ C$ for the *h*-BN formation. During the growth, the borazane powder was heated at $60\sim 90^\circ C$ to convert it into the vapour phase. A mixture of *Ar* and H_2 were used as a carrier gas to transfer the borazane vapour into the reaction zone and pass through the substrate surface. The *Cu* foil observed by optical microscope, which shows the surface of *Cu* is smooth and clean also grains are clear as shown in Fig.2. The decomposition range of borazane is $77\sim 137^\circ C$, which is around the melting point of $106^\circ C$ at room temperature [35]. While under vacuum condition, borazane can completely decompose below its melting point.

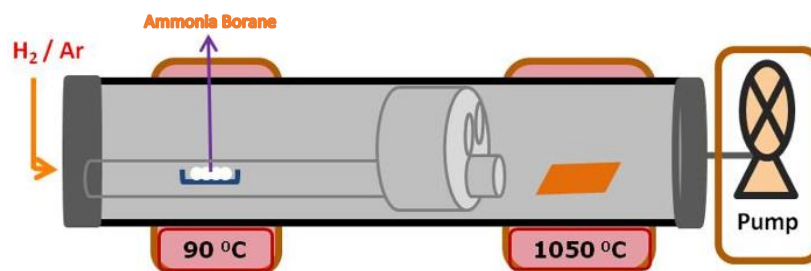


Fig.1. Schematic diagram of LPCVD designed for growth of *h*-BN.

The decomposition is accompanied by hydrogen release and heat evolution, producing hydrogen, monomeric aminoborane (BH_2NH_2) and borazine [$(HBNH)_3$], as illustrated in Fig.4. Then the nucleation and formation of *h*-BN may occur on the substrate surface. After the growth for 5~20 min, fast cooling of the reaction zone was carried out for the improvement of *h*-BN crystalline quality. For the clean monolayer *h*-BN grown on *Cu* foil, PMMA assisted method was used to transfer it onto new substrates such as *Si/SiO₂* or TEM grids for further characterizations or special demands.

The “Normarski” optical microscopy (Olympus BX51M) became handy for the observation recording of the *Cu* surface morphology prior and later growth of *h*-BN. The “Scanning Electron Microscope (Hitachi S-4800)” SEM was used to characterize the structure morphology and *h*-BN films coverage [41]. The Raman spectra were recorded using a “Renishaw InVia Raman Microprobe” equipped with a 532 nm laser for monolayer *h*-BN. The “Transmission Electron Microscope, TEM” (Philips Tecnai F30) investigation was carried for a crystalline structure. The hexagonal structure of *h*-BN film was confirmed using “Selected Area Electron Diffraction (SAED)” pattern. The “Atomic Force Microscopy (AFM)” was employed to measure the thickness and morphology of *h*-BN flakes [41].

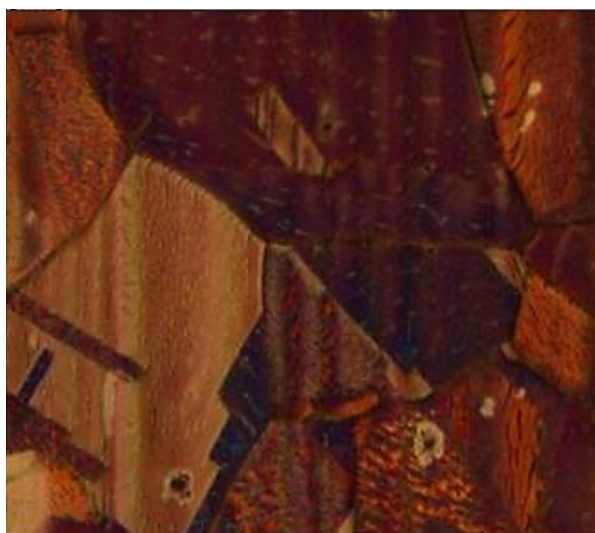


Fig.2. The optical image shows the surface of *Cu* is smooth and clean also grains are clear.

3. Growth of *h*-BN Monolayer on *Cu* Foil

Firstly, we used *Cu* as a substrate and put directly in the reaction zone to grow *h*-BN layers as shown in Fig.1. *h*-BN growth starts when the reaction zone reaches at 1050°C and the precursor zone at 90°C. Fig.2 is the optical image, which reveals the smoothness and cleanness of the *h*-BN/*Cu* surface.

After the 5 minutes of growth, the SEM image in Fig.3 shows that uniquely triangular-shape *h*-BN flakes appear on the surface uniformly. In the heating zone, borazane powder decomposes into three products, aminoborane, hydrogen and borazine and *h*-BN can grow from borazine which indicated in the schematic diagram of Fig.4. Now, we have increased the *h*-BN growth time to 20 minutes to cover the whole surface of *Cu* foil by *h*-BN layer.

In Fig.5, again SEM image shows the surface morphology of *h*-BN growth on *Cu* foil, here in this image green arrow indicates the wrinkle on the surface which is the evidence of fully covered *Cu* foil with the *h*-BN layer [42]. To confirm the structure and thickness, the *h*-BN nano-sheets were transferred on to *Si / SiO₂* substrate and measurement of AFM and Raman spectroscopy were carried out.

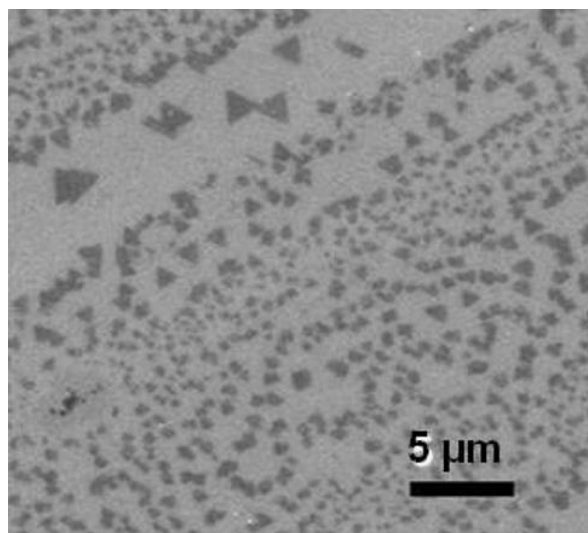


Fig.3. SEM image shows that triangular shaped *h*-BN flakes appear in 5 min.

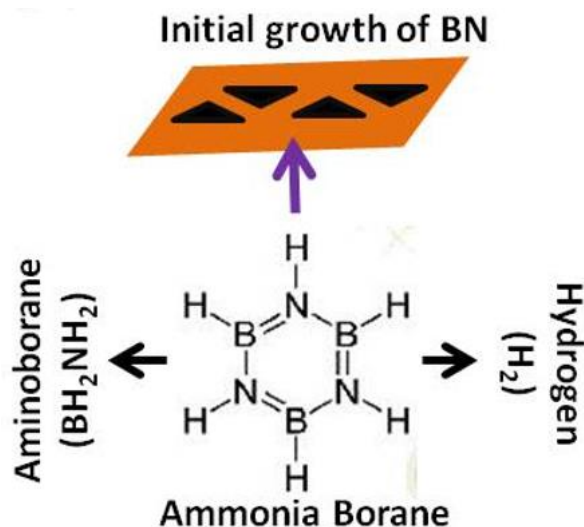


Fig.4. Schematic diagram shows ammonia borane decomposes into three products (aminoborane, hydrogen & borazine), *h*-BN nanosheets grows from borazine.

In Fig.6, we can observe the thickness of *h*-BN measured by AFM and the value indicates 0.45 nm , which shows the growth of monolayer *h*-BN on *Cu* foil [42]. As shown in Fig.7, the characteristic Raman peak at $\sim 1371 \text{ cm}^{-1}$ could be clearly seen, which is consistent well with the reported value for monolayer *h*-BN film. To further evaluate the crystalline structure of the *h*-BN film, the sample was characterized by HRTEM imaging combined with SAED, as shown in Fig.8. One can clearly see that the six-fold diffraction pattern demonstrates the hexagonal structure of the *h*-BN film.

Although, the CVD method is required to be followed to grow *h*-BN on *Cu* foil, but as per observation, the diameter is found inadequate in size for the chamber or quartz tube. So, the proposed method of “deforming copper foil” into “Quasi-3D” shape is adopted to gain result in *h*-BN wide area growth. According to the general practice, the copper foil is winded as cylindrical form fitting into the quartz

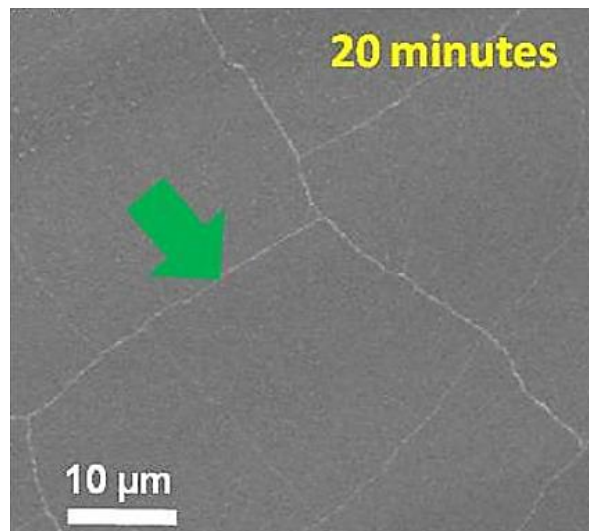


Fig.5. The green arrow shows the wrinkle which means *h*-BN fully covered on *Cu* foil in 20 min.

tube's inner surface wall [41]. The inner small tube for carrying precursor was set aligned with the central axis of the cylinder. Because of this arrangement, the precursor is dragged uniformly towards the inner surface wall of the cylindrical copper foil. Taken 1.2 inches as radius “*r*” of the quartz tube, the cylinder's circumference is obtained by $2\pi r$ formula. Hence the inner surface now provide a large area [41]. The XPS measurement was further taken to characterize the chemical composition of BN film, as shown in Fig.9(a) and Fig.9(b). The binding energies of the clear B 1s and N 1s electrons could be identified at 190.5 eV and 398.1 eV , respectively. This confirms the formation of *h*-BN layers and the stoichiometric ratio equals to 1, consistent with previous reports [36][37][38][39][40][41].

When the precursor amount increases, the growth rate of *h*-BN flakes increases accordingly and the second layer of *h*-BN begins to grow. As a result, triangular flakes form on the beneath first complete *h*-BN nanosheet. In this way, a multilayer *h*-BN nanosheet could be grown.

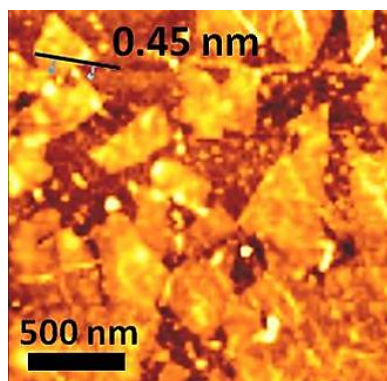


Fig.6. AFM image indicates a height of monolayer *h*-BN which is 0.45 nm .

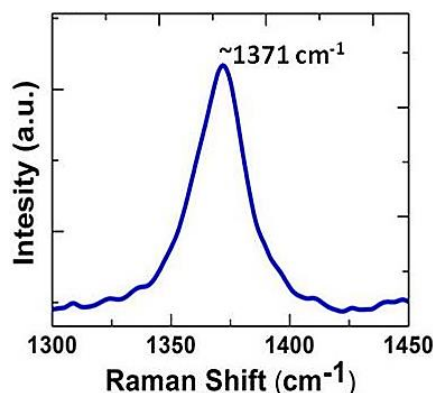


Fig.7. Raman spectroscopy peak at $\sim 1371 \text{ cm}^{-1}$, which also indicates of monolayer *h*-BN.

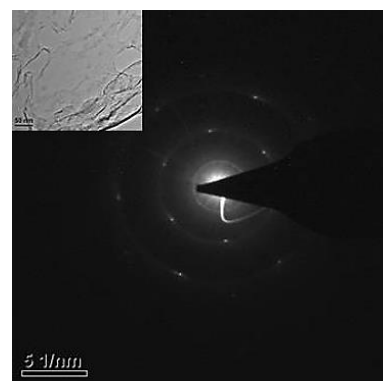


Fig.8. HRTEM image shows a hexagonal structure of BN, inset shows thin layers of *h*-BN.

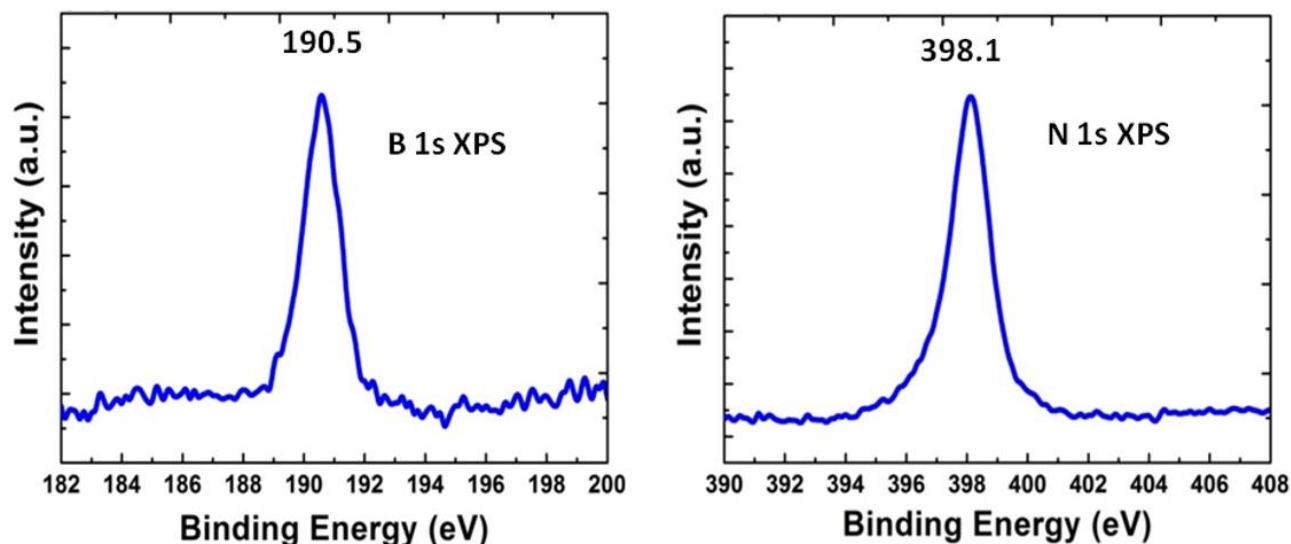


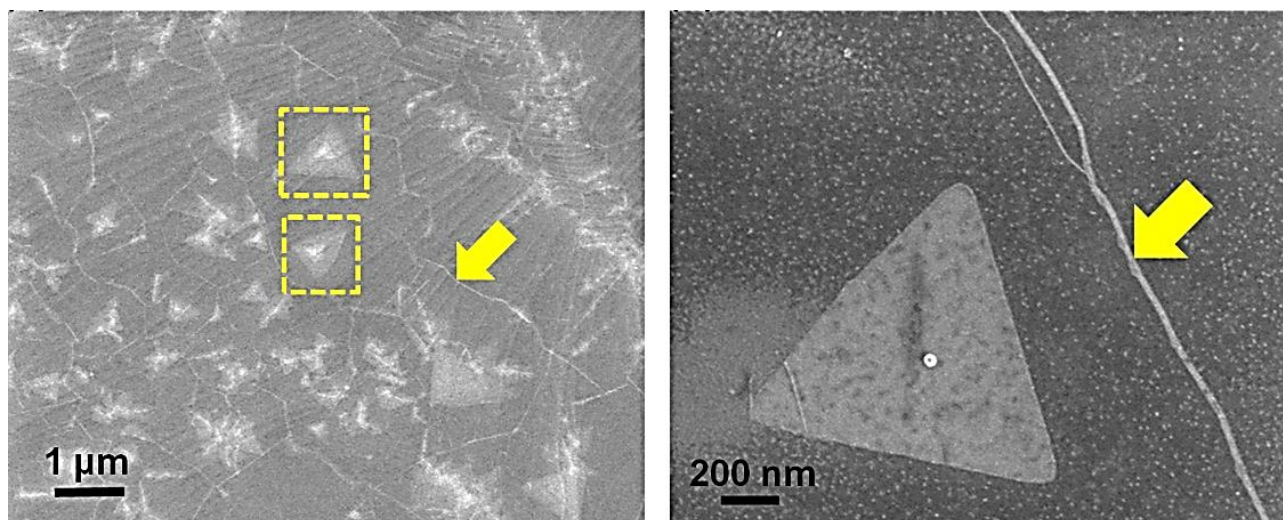
Fig.9. XPS spectra of B 1s and N 1s, is shown. The binding energy peaks for (a) B 1s is 190.5 eV and (b) N 1s is 398.1 eV.

In Fig.10, SEM images show multiple layers of *h*-BN on *Cu*, yellow squares show the second and third (white part) layer of *h*-BN and the yellow arrow indicates the wrinkle of the first layer.

In Fig.11, layers are transferred on *Si* by PMMA method, the yellow arrow indicates wrinkle of first layer and triangle shows the second layer. It means that we can transfer the *h*-BN layer from *Cu* foil to an external substrate as we transferred to the *Si* substrate. Now, *h*-BN layer on *Cu* foil as well as transferred on *Si* substrate is available for further electronic applications.

4. Conclusion

In conclusion, we successfully synthesized triangular *h*-BN nano-sheets on *Cu* foil by LPCVD method. For the synthesis of *h*-BN on *Cu* foil, monolayer/multilayer *h*-BN nanosheets in a triangular shape, as well as fully covered *Cu* foil, were grown, it means from small to the large size of the growth. After growth, *h*-BN layers transferred to the *Si* substrate by PMMA method. Now, the applications of *h*-BN layer separately as well as with conjunction of graphene are available for electronic devices.



(a) On *Cu*, yellow squares shows the second and third (white part) layer of *h*-BN and yellow arrow indicates the wrinkle of first layer.

(b) Layers are transferred on *Si* by PMMA method, yellow arrow indicates wrinkle of first layer and triangle shows the second layer.

Fig.10. SEM images show multiple layers of *h*-BN

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