Postsynthesis of h-BN/Graphene Heterostructures Inside a STEM

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Combinations of 2D materials with different physical properties can form heterostructures with modified electrical, mechanical, magnetic, and optical properties. The direct observation of a lateral heterostructure synthesis is reported by epitaxial in-plane graphene growth from the step-edge of hexagonal BN (h-BN) within a scanning transmission electron microscope chamber. Residual hydrocarbon in the chamber is the carbon source. The growth interface between h-BN and graphene is atomically identified as largely N–C bonds. This postgrowth method can form graphene nanoribbons connecting two h-BN domains with different twisting angles, as well as isolated carbon islands with arbitrary shapes embedded in the h-BN layer. The electronic properties of the vertically stacked h-BN/graphene heterostructures are investigated by electron energy-loss spectroscopy (EELS). Low-loss EELS analysis of the dielectric response suggests a robust coupling effect between the graphene and h-BN layers.

1. Introduction

The electronic band gap of graphene can be tuned by compositing graphene with materials of different band gap energies. Hexagonal BN (h-BN) is suitable for this purpose, with a lattice structure similar to that of graphene and a much larger band gap. 2D heterostructures of graphene and h-BN can be formed by mechanical overlap, chemical vapor deposition (CVD), or controlled continuous growth. Vertical stacking, whether achieved mechanically or by CVD, forms h-BN/graphene (h-BN/G).1–5 Controlled in-plane growth forms laterally modulated h-BN sheets on graphene (h-BN_G).6–9 In addition to broadening the graphene band gap, the h-BN/G and h-BN_G heterostructures possess unusual physical properties, such as the fractional quantum Hall effect,10,11 Hofstadter butterfly behavior,11,12 ballistic transport,13 new Dirac point effects,14 and high ON/OFF ratios,15 which can enhance and tune the electronic, optoelectronic, and spintronic properties7,8,16,17 of such low-dimensional materials. The investigation of interactions between the constituents of such van der Waals heterostructures is strongly motivated, for the construction of functional heterostructured devices and novel hybrid 2D materials.

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Understanding edge structures at the h-BN/graphene domain boundaries at atomic levels is extremely important, as B-C terminated carbon regions tend to have larger band gaps than N-C terminated regions.\textsuperscript{[18]} Previously,\textsuperscript{[19]} we reported in-plane graphene growth at the step-edge of bilayer graphene. Here, we visualize in-plane graphene growth from the edge of an h-BN layer on a monolayer graphene substrate \((h\text{-}BN/G)/G\) using aberration-corrected scanning transmission electron microscopy (STEM) and thus identify the h-BN G grain boundaries at an atomic scale. STEM can provide chemical information at high spatial resolution.\textsuperscript{[20]} By using a postgrowth method, graphene nanoribbon can be synthesized to connect two h-BN domains with different twisting angles, forming a lateral heterostructure \((h\text{-}BN\_G\_h\text{-}BN)/G\). We also report the postsynthesis of isolated carbon islands, performed by first drilling holes on the h-BN layer and reknitting these holes by newly grown graphene. H-BN has threefold symmetry and typically forms triangular holes under the electron beam;\textsuperscript{[21]} however, under certain conditions, such as high temperatures, circular, and hexagonal holes are observed.\textsuperscript{[22]} Graphene typically has sixfold symmetry, but the newly grown graphene filling the triangular holes of h-BN shows special magnetic properties.\textsuperscript{[23–29]} Patchworks formed by such reknitting of holes\textsuperscript{[30]} can result in quantum confinement effects. We also examine the dielectric response of stacked h-BN and graphene heterostructures \((h\text{-}BN/G)/G\) by electron energy-loss spectroscopy (EELS). Pairing the results with theoretical investigation suggests that the response is not a simple overlap of the graphene and h-BN responses, but that a robust coupling effect exists between graphene and h-BN layers.

2. Results and Discussion

2.1. Lateral Heterostructure by In-Plane Graphene Growth from h-BN Step-Edge

Figure 1 depicts several postgrowth heterostructures of new graphene in-plane growth from the step-edge of h-BN on a monolayer graphene substrate (Figure 1a, vertically stacked plus lateral heterostructure of \((h\text{-}BN\_G)/G\), reknitting of triangular (Figure 1b,c) and hexagonal (Figure 1d,e) holes embedded in an h-BN layer, and a graphene interjunction between twisted h-BN layers (Figure 1f, \((h\text{-}BN\_G\_h\text{-}BN)/G\)) supported on monolayer graphene. To fabricate such lateral heterostructures of h-BN_G, monolayer graphene is overlapped with monolayer h-BN (h-BN/G) and heated within the microscope chamber to \(\approx 200–650^\circ C\), forming new post-synthesis structures. As reported previously, because the electron microscope chamber is not a perfect vacuum, residual hydrocarbon gases in the sample chamber can be used for high-temperature graphene growth because the electron beam causes hydrocarbons to agglomerate in the scanning area.\textsuperscript{[19]} A pre-existing supporting graphene layer is necessary in step-edge growth from h-BN.

Thus, we reproducibly fabricated a lateral heterostructure of \((h\text{-}BN\_G)/G\) by the postgrowth process. Figure S1a–d of the Supporting Information shows a series of annular dark-field (ADF) images of the heterostructured \((h\text{-}BN\_G)/G\), revealing the process of graphene growth from the h-BN edge. Figure S1e–h of the Supporting Information presents corresponding filtered images of Figure S1a–d in the Supporting Information, in which only B, N, and newly grown C atoms are shown. In some regions, graphene grows with the same lattice structure as the original h-BN and presents the same moiré pattern as the original h-BN/G area. Other areas of new growth show different moiré patterns, as with in-plane growth from the step-edge of bilayer graphene.\textsuperscript{[19]}

Figure 2a,b is filtered images of Figures S1a and S2b in the Supporting Information, respectively, showing only B, N, and newly grown C atoms. The intensities corresponding to graphene-substrate monolayer atoms were deleted from the fast Fourier transform (FFT) of Figure S1a,b in the Supporting Information; a further inverse fast Fourier transform (IFFT) of the remaining intensity distribution provides the contrast between BN and newly grown C atoms. Regardless of the relative positioning of h-BN or graphene with respect to the electron-beam incident direction \((h\text{-}BN/G\text{ or } G/h\text{-}BN)/G\), graphene can always grow from the h-BN edge. From
Figure 2a, h-BN usually terminates as a zigzag edge bordered with N atoms (N-terminated edge). In such cases, the growth of new graphene dominantly forms N–C bonds. This is clearly observed in Figure 2b, in which N–C bonds are the connections between the green and blue dots. In the original h-BN area, the lattice contrast shows threefold symmetry and the N atoms appear brighter than the B atoms; however, sixfold lattice symmetry in the new growth area is observed in the absence of lattice defects. Figure 2c presents an enlarged view of the square region indicated in Figure 2b. A line profile (Figure 2d) from the bottom to top of Figure 2c clearly shows the contrast differences of B, N, and C atoms. The contrast of the newly grown graphene area (indicated as G) is uniform, showing only the existence of C atoms. Core-loss electron energy-loss spectroscopy (EELS) analyses, shown in Figure S2 of the Supporting Information, demonstrate the C-only compositions of the new growth areas. Dendritic graphene growth occurs concurrently on h-BN edges, as shown in Figure S1g of the Supporting Information. Such new growth areas merge upon encountering each other, forming many defects such as pentagons, heptagons, and octagons at the domain boundaries, as shown in Figure S1h of the Supporting Information. This formation mechanism is similar to that observed in polycrystalline graphene synthesized by CVD. Graphene can grow not only from the step-edge of h-BN supported by monolayer graphene, but also from the step-edge of graphene on an h-BN substrate \(((G,G)h-BN)\), meaning that the original graphene layer can be enlarged afterward. This may occur because of the similar lattice structures of graphene and h-BN.

Such lateral postsynthesis can form a graphene nanoribbon bridge connecting two h-BN domains with different twisting angles \(((h-BN,G,h-BN)/h-BN)\), as illustrated in Figure 1f and imaged by ADF in Figure 3a–c. Figure 3a shows the initial growth of graphene (labeled G/G) from the h-BN edge enclosed. This graphene area grows to connect two h-BN domains of different twisting angles, as shown in Figure 3b. The high-magnification ADF image (Figure 3c), corresponding to the area enclosed by a green dotted line in Figure 3b, clearly shows that the new growth connection between the two h-BN domains has the same contrast as the h-BN areas, indicating the monolayer nanoribbon structure of the new connection. Some debris, mainly carbonaceous materials showing brighter contrast, can be found growing on the postgrowth nanoribbons. The formation of debris can be reduced by lowering the growth speed by increasing the sample heating temperature. Figure 3d shows the EELS analyses of the three areas labeled 1, 2, and 3 in Figure 3b. The EELS spectra in areas 1 (blue line) and 3 (green line) show both B and N K-edges together with C K-edge, illustrating the two h-BN domains supported by the graphene monolayer. The EELS spectrum of area 2 (red line), showing only the C K-edge, demonstrates that the postsynthesis connection between the two h-BN domains is a graphene nanoribbon. The intensity of the C K-edge in area 2 is almost twice that in areas 1 and 3, demonstrating the monolayer morphology of the newly grown graphene nanoribbon. The ADF image and corresponding chemical mappings of C, B, and N in the area enclosed by the magenta square in Figure 3b are shown in Figure 3e. The new postgrowth nanoribbon clearly contains only C. Figure 3f show three low-loss EEL spectra corresponding to areas 1, 2, and 3 from Figure 3b. Peaks clearly shift between the h-BN/G area 1 (blue line), 3 (green line) spectra, and the G/G area 2 (red line) spectrum. This peak drift will be discussed in Section 2.3.

2.2. Postsynthesis Carbon Islands Embedded in h-BN Layer

Isolated C islands generating quantum confinement effects can also be synthesized in an electron microscope. The STEM chamber has the ability to form well-defined holes in the
h-BN layer by the focused high-density electron beam, and then to reknit the holes by accumulating C atoms onto the electron-beam scanning area. Thus, it permits the production of designed heterostructures. A series of ADF images showing the reknitting hole processes in a h-BN layer during heating at 350 °C is shown in Figure 4, in which only the B, N, and newly grown C atoms are displayed. The intensity corresponding to C atoms in the monolayer graphene substrate were removed using the same method as in Figure 2. In Figure 4a, the terminated edges around the hole, illustrated by yellow and cyan dotted lines, are composed of zigzag and armchair edges (the yellow triangle indicates the three zigzag directions, while the cyan triangle represents the three armchair directions). Since the knock-on damage to B atoms is more severe than that to N atoms,[20] the edges around the h-BN hole are predominantly N-terminated, although a small number of B-terminated regions are observed. Although N–C and B–C bonds coexist at the edge of the reknitted hole, N–C bonds are obviously more prevalent.

The rates of drilling and reknitting of an h-BN hole compete for balance. When the electron beam density and heating temperature are sufficiently high (>700 °C here, with sample chamber pressure of ~10⁻⁵ Pa), B and N atoms along the edge of the hole are lost continuously, increasing the hole size. However, the accumulation speed of C atoms can be controlled by the local pressure and heating temperature; when this is sufficiently high, the reknitting process is favored. The h-BN hole is gradually reknitted by C atoms and finally filled (Figure 4b–h). The newly filled area is a monolayer graphene plate, with contrast showing sixfold symmetry and few defects. At lower reknitting speeds, the final defect density is lower in the newly grown graphene inside the hole. Figure 4i is an ADF image on which core-loss EELS analyses were performed. Figure 4j,k,l shows the core-loss EELS chemical mapping of C, B, and N, respectively. Figure 4m,n is EEL spectra from areas 1 and 2 in Figure 4i, respectively. The postsynthesized C island shows a hexagonal shape. Figure S3 of the
Supporting Information shows a triangular refilled C island with zigzag-direction terminated edges (the yellow triangle indicates the three zigzag directions in h-BN layer). N-terminated triangular defects with a larger number of missing B atoms have been reported to be energetically favorable; triangular C islands with zigzag terminated edges show magnetic properties.\textsuperscript{[24,25,31,32]} When a single B or N atom is substituted with a C atom, one extra electron is added or removed, respectively. If the total substituted B and N atoms are unbalanced, magnetic moments form. The aspect causing magnetic and electronic properties in hybrid nano-structures is not the shape of the isolated C islands, but the actual numbers of B and N atoms substituted by C atoms. Since graphene is a semimetal and h-BN has a band gap of $>5 \text{ eV}^{[33]}$, such reknitted graphene patches isolated within BN sheets can be regarded as quantum nano-objects.
2.3. EELS Analyses of Vertically Stacked h-BN/Graphene Heterostructures

EELS successfully demonstrated the tuning effect of vertically stacked h-BN/G heterostructures. The core-losses of B and N atoms bonded together or with C atoms at the h-BN edge do not differ significantly because of the delocalization effect. However, if only one interstitial N atom is located within graphene, the core-loss of the N peak will shift according to the valence modification. In this study, because of the delocalization effect, the core-loss peak positions of the B and N edge atoms are affected by near-edge B and N atoms within the h-BN layer; thus, only average effects are provided by EELS, which hinders the identification of B–C versus N–C bonds through EELS chemical mapping. ADF images (Figure 2) show that N atoms connect to C atoms.

Low-loss EELS yields very important information. In Figure 5a, low-loss high-resolution EEL spectra of regions of pure h-BN (red), pure graphene next to an h-BN/G bilayer, and a bilayer h-BN/G, obtained at 30 kV at −120 °C, are shown. The spectra illustrate the contributions of the graphene surface π-plasmon at ≈5.1 eV, h-BN exciton at ≈6.3 eV, and a feature at ≈7.5 eV in the h-BN spectrum associated with an interband transition. Notably, for thin materials such as the one studied here, a low-loss EEL spectrum is directly linked to the dielectric function of the material and, hence, the optical absorption thereof.

Interestingly, the low-loss EEL spectra of the h-BN/G heterostructures do not show simple overlaps of those of monolayer graphene and monolayer h-BN. The graphene surface plasmon is red-shifted by ≈220 meV. This red shift is not caused by the multilayer effect. As shown in Figure S4 of the Supporting Information, obtained at 550 °C, the graphene surface plasmon peaks experience blue shifts with increased numbers of graphene layers. Even bilayer graphene (green line) shows a blue shift relative to monolayer graphene. The observed red shift in h-BN/G heterostructures is caused by the coupling between the h-BN and monolayer graphene. The energy of a metal surface plasmon is lower for an interface with a high dielectric material than for an interface with vacuum. The h-BN exciton is blue shifted by ≈170 meV in the h-BN/G bilayer, likely because of coupling between layers.

EELS simulations were compared with the experimental results. The simulated EEL spectrum of h-BN/G shown in Figure 4b is based on a method reported by Richter and Geiger, in which the energy-loss probability for fast electrons at normal incidence was calculated for a system of m layers with different thicknesses d and dielectric functions ε(ω). In the simulation, the wavelength of the incident electrons is 0.0070 nm at 30 kV and the sheet thicknesses of graphene and h-BN were set to 0.3 nm, approximately half the c-axis lattice constants for the individual crystal structures. The dielectric functions of bulk h-BN and graphite were applied, obtained by Kramers–Kronig analyses of individual EELS spectra. The retardation effect was neglected. The calculated energy-loss probabilities were integrated by scattering angle up to 38 mrad, which corresponds to the collection semiangle for the EELS measurement system used here. The simulated spectrum of the h-BN/G bilayer shows peaks at 4.7, 6.3, and 7.5 eV. The peak at 4.7 eV is attributed to the π-plasmon of graphene, while the peaks at 6.3 and 7.5 eV are attributed to the exciton and π-plasmon, respectively, of h-BN. The graphitized π-plasmon energy at 4.7 eV of the bilayer spectrum is lower than the 5.8 eV peak energy of pure graphene. The h-BN exciton in h-BN/G does not shift significantly compared to that of pure h-BN. This tendency is consistent with the experimental results, in which the graphene surface plasmon is red-shifted. The red shift is attributed to the screening effect of plasmon oscillations in the h-BN sheet. This means that the restoring force for the plasmon oscillation frequency in h-BN significantly decreases the plasmon resonance energy of graphene. This result demonstrates how the formation of thin heterostructures can tune the optical properties of single-layered materials.

3. Conclusions

Atomic analyses of in-plane graphene growth from the step-edge of h-BN provided insight on the band gap tuning of synthesized heterostructures. We observed the lateral synthesis of heterostructured h-BN, G by in-plane epitaxial graphene growth from the step-edge of h-BN. By this post-growth method, graphene nanoribbon was synthesized to connect two h-BN domains with different twisting angles.
Isolated C islands with triangular and other arbitrary shapes embedded into the h-BN layer were produced. Low-loss EELS analyses showed that the dielectric function of vertically stacked h-BN/G heterostructures were not simple overlaps of those of monolayer graphene and h-BN, indicating electronic coupling between graphene and h-BN layers. To obtain more specific energy resolutions, a Cs-corrected electron microscope mounted with a monochromator field-emission electron gun will yield further improvements in the spectra. We expect to clarify the subtle differences in electronic properties of h-BN/G heterostructures with different rotation angles.

4. Experimental Section

Graphene Synthesis and Transfer: Monolayer h-BN was obtained commercially. Single-crystal monolayer graphene was grown by the CVD of methane (99.99%) on polycrystalline Cu foil at atmospheric pressure. For the transfer process, the as-grown graphene sheet on Cu foil was first spin-coated with a layer of poly-carbonate, then etched in HCl aqueous solution (HCl + H2O + H2O2) to remove the Cu foil. The monolayer graphene topped with the polycarbonate film was collected using monolayer h-BN grown on Cu foil. After removing the Cu foil from the stacked G/h-BN films, the G/h-BN films attached to polycarbonate were transferred onto a Mo Quantifoil grid or a SiN transmission electron microscopy (TEM) grid for TEM observations. A similar process produced h-BN/G films by scooping h-BN film with monolayer graphene grown on Cu foil. The polycarbonate was finally removed using chloroform, acetone, and 2-propanol. Energy dispersive X-ray spectroscopy (EDS) analyses demonstrated the complete removal of Cu.

STEM and EELS Observations: ADF imaging, core-loss EELS, and some low-loss EELS analyses were performed using a JEOL 2100F with a cold field-emission gun and an aberration DELTA-corrector operated at 60 kV. A Gatan GIF Quantum was used for the core-loss EELS chemical analyses. The typical resolution for STEM and ADF imaging was 0.11 nm. The inner and outer collection angles for the ADF image ($\beta_i$ and $\beta_f$) were 58 mrad and 129–140 mrad, respectively. The beam current was $\approx 10$ pA for ADF imaging, EELS chemical analyses, and low-loss EELS measurements. The images and some EELS analyses were obtained at temperatures from 200 to 550 °C using a JEOL heating holder to avoid amorphous carbon contamination. The scan rate was 8 µs per pixel for faster scans and 58 µs per pixel for atomic-resolution imaging. Images were filtered by commercial HREM-Filters Pro software.

High-spectral-resolution low-loss EELS measurements were performed in a JEOL ARM200 microscope equipped with a double-Wien filter monochromator operated at 30 keV. A Gatan Quantum spectrometer optimized for low-energy low-noise operation was used. Experiments were performed at $-120$ °C using a Gatan liquid nitrogen-cooled holder.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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