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Stoichiometric edges during the intrinsic growth of hexagonal boron nitride on Ir(111)

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Abstract

The growth of hexagonal boron nitride, hBN, on Ir(111) at 1150–1200 K by thermal decomposition of borazine has been monitored in situ by means of low energy electron microscopy, LEEM. A major and unexpected hBN growth induced transformation of the step morphology on Ir(111) is observed. The dominant orientation of the steps evolves persistently from ⟨1–10⟩ during the initial experiment(s), towards ⟨11–2⟩ after numerous experiments. The latter orientation was established directly from an involved analysis of the profile of the specular beam in a selected area diffraction pattern. Images of the hBN islands grown on surface regions with dominant ⟨1–10⟩ steps reveal that the hBN edges are oriented along ⟨1–10⟩ and ⟨11–2⟩ directions in about equal abundance. Using the direct ascertainment of the step orientations we are able to identify unequivocally the nature of the edges of individual hBN islands on areas with predominant ⟨11–2⟩ steps as solely of the armchair type. This assessment consolidates both the major evolution of the step morphology from ⟨1–10⟩ into dominant ⟨11–2⟩ steps during hBN growth on Ir(111) and the earlier reported alignment of the moiré pattern with the substrate.

Introduction

The interest in the structure and physical properties of two-dimensional materials has risen to an immense summit. It has been boosted by the discovery of spectacular properties of graphene [1, 2] and is followed by intense interest in the allotropes of the group IV elements Si, Ge and Sn [3–6]. More recently, transition metal dichalcogenides, as for instance MoS2, [7, 8] are of high actual interest. Many interesting applications, including electronic, opto-electronic, magnetic and chemical sensor devices [e.g. 9–12] have been suggested. However, the coupling to (metallic) substrates forms a severe road block to realize some promising applications. The deposition of graphene structures on top of a hexagonal boron nitride (hBN) covered metal substrate is commonly viewed as a viable solution to overcome this problem. Note that hBN is an insulator with a band gap of about 6 eV [13]. The strong structural similarity between hBN and graphene is considered to be an important enabler. We refer to a recent review by Auwärter, one of the founding fathers of hBN research, for a detailed and impressive survey of the properties and diverse applications of hBN [14]. Since detailed in situ information on the growth is still scarce we have embarked on such a study using LEEM.

Here, we restrict ourselves to the growth and structure of hBN films on Ir(111) substrates. The current results are believed representative of hBN flakes on medium strongly reactive substrates. Recently, together with a few collaborators, we have reported that, unlike in the graphene case, the edges of hBN have a (di)polar character. This has important consequences with respect to the shape of hBN flakes on Ir(111) [15]. We have identified BN-dimers acting as the basic building blocks for the growth of the hBN flakes. Earlier, we have combined successfully cook-and-look scanning tunneling microscopy, STM, with in situ low energy electron microscopy, LEEM, and spot profile low energy electron diffraction, SPALEED, for a detailed understanding of the growth and the structure of graphene on Ir(111) [16–20]. Guided by this success, we take the seminal STM
study of hBN on Ir(111) by Farwick zum Hagen et al [21] as a bench-mark for our current LEEM studies on the same system. In brief, they report a coincident moiré unit mesh of (12 × 12) hBN cells on (11 × 11) Ir(111) cells with two oppositely oriented hBN phases. Locally about 1.5 Å deep pits anchor the moiré pattern to Ir(111) by means of chemical binding. Finally, the orientation of the hBN flakes is aligned with that of the hosting Ir(111) surface, i.e. the lines connecting adjacent chemically bound pits line up with the close packed ⟨10−10⟩ directions on Ir(111).

Recently, Petrović et al [22] have used selected area LEED (μLEED) to obtain information on the orientation of the edges of hBN flakes on Ir(111). The reasoning behind this approach is that the base vectors in reciprocal space are perpendicular to the base vectors in real space. For a hexagonal lattice this implies that the lines connecting the specular beam with the first order diffraction beams run along ⟨11−2⟩ directions in real space. Comparing those directions with the directions of the edges of the hBN flakes does then contain unique information on the orientation of their edges in real space. This is straightforward in the integral LEED mode.

Below we introduce a direct and precise approach to obtain information on the morphology of atomic steps at the local scale using μLEED. It provides a powerful and elegant method to pinpoint the local orientation of atomic steps on the surface and to unequivocally link directions in real and reciprocal space. This enabled us to solidify and consolidate the preference for armchair edges in hBN grown on Ir(111) as reported in [15]. It leads to a global view on the distribution of hBN edges, which is by its nature not readily available in STM.

An Elmitec LEEM III with a base pressure of 1 × 10⁻¹⁰ mbar was used to study the growth of hBN on Ir(111). Ir(111) single crystals, purchased from Surface Preparation Laboratory, were cleaned by alternating cycles of Argon ion sputtering and annealing in oxygen at 1300 K, followed by flash annealing to 1600 K before each measurement. HBN was removed by annealing at 1300 K and subsequently the sample was cleaned as described above involving sputter cleaning. The hBN was grown by thermal decomposition of ultraclean borazine purchased from Chemos GmbH.

Results

We now discuss the results obtained from the first hBN growth experiment on the freshly prepared, newly purchased Ir(111) substrate shown in figure 1(a) representing a frame from a LEEM movie. The slightly curved stripes reveal atomic steps in a region flatter than nominal (m miscut ~0.1°–0.2°) [23]. The dark objects in figure 1(b), (e) and (f) show the growing hBN islands. Note that the microscope settings for optimal imaging of steps and islands differ slightly. This renders simultaneous mapping of steps and hBN edges a bit troublesome. Without exception the islands nucleate at step edges. Figure 1(c) shows a μLEED pattern taken from the area encircled by the solid blue circle in figure 1(b) and represents the structure of the right angled trapezoids. The μLEED pattern in figure 1(d) is taken from the area limited by the red dashed circle in figure 1(b) and represents the structure of the triangular hBN island. Figure 1(e) shows the evolution of the growing islands. The central bright features show the hBN islands at 202 s and the inner dark area shows what has grown between 202 and 404 s. The same holds for the outer bright area for growth between 404 and 606 s, while the outer dark areas represent the growth between 606 and 808 s. In the frame displayed in figure 1(f) the solid blue lines and the dashed red lines indicate the ⟨1−10⟩ and the ⟨11−2⟩ azimuths obtained from figures 1(c) and (d). (The quantitative assessment of this identification is discussed in detail below.) Acentric nucleation is clearly visible in figure 1(e). This is in line with the earlier noted exclusive nucleation at steps [15]. We emphasize that the internal hBN structure, illustrated in figure 1(c), is identical for all islands, including the trapezoids and except for a mirrored one in the triangle as illustrated in 1d. The three-fold symmetry of the substrate is clearly visible and the satellite spots originating from the moiré pattern of hBN on Ir(111) are most marked around the specular and the first order substrate beam. The moiré pattern reveals that the hBN is nicely aligned with the substrate structure. This finding agrees with STM data reported in [21]. Therefore, zigzag edges run along ⟨1−10⟩ directions and armchair edges run along ⟨11−2⟩ directions of Ir(111) in real space. (See figure 2 for a sketch. For more detailed information we refer to [15].)

The red lines in figure 1 connect the first order beams through the specular beam and since the base vectors in reciprocal space are oriented perpendicular to the base vectors in real space they unequivocally mark ⟨11−2⟩ directions in real space. Assuming that the real space image follows truly the directions given by the red dashed lines a transfer of the hexagonal solid and dashed lines in figures 1(c) and (d) to the real space image in figure 1(a) allows one to check the orientation of the atomic steps. They appear to run predominantly along the ⟨1−10⟩ direction given by the solid blue lines. This fact was expected as the minimum energy orientation of monatomic steps on surfaces of face centered cubic crystals as Ir along ⟨1−10⟩ [24–26].

A closer inspection of figure 1(b) leads to a general conclusion that the most frequent hBN island shape resembles a right angled trapezoid. Irrespective of the exact orientation coupling between figures 1(b)–(d) it is obvious that the edges of the individual islands make edges of about 30°, 90° and 150°. This immediately implies solidly that we observe a coincidence of zigzag (or dimer terminated ones (see figure 6 of reference [15])) and
Figure 1. The images in (a), (b), (e) and (f) are taken at 1200 K, field of view (FoV) 20 \( \mu m \) and electron energy 16.3 eV. (c) and (d) show \( \mu \)LEED patterns from the areas indicated by the blue (solid) and the red (dashed) circles in (b), respectively using a 1.4 \( \mu m \) aperture. They were measured at 1200 K with 45 eV electrons. The solid blue and dashed red lines in (c), and (d) indicate the \( \langle 1–10 \rangle \) and the \( \langle 11–2 \rangle \) azimuths, respectively. The central bright features show the hBN islands at 202 s and the inner dark areas indicate what has grown between 202 and 404 s, the same for the outer bright areas between 404 and 606 s and the outer dark areas between 606 and 808 s. (f), Solid blue and dashed red lines indicate the \( \langle 1–10 \rangle \) and the \( \langle 11–2 \rangle \) directions from the \( \mu \)LEED patterns in (c) and (d), respectively.

Figure 2. Sketch of the Ir(111) substrate (top view). The atoms are situated on the crossing points of the hexagonal lattice for the first, second and third layer, represented by solid, dashed and dotted lines (A), (B) and (C), respectively. The hBN film is sketched by N-atoms (red) and B-atoms (blue). The N-atoms are located on top of first layer Ir atoms and the B-atoms are either above second layer Ir-atoms (hcp-hBN; left) or above third layer Ir-atoms (fcc-hBN; right). The fcc phase is slightly preferred \[21\] and oriented oppositely to the hcp one. The sketch applies for the minima in the moiré pattern, where the hBN is chemically bound to the substrate \[21\]. The close packed directions of the substrate and the hBN run parallel. This alignment implies that zigzag hBN edges run parallel to \( \langle 1–10 \rangle \) and consequently armchair edges parallel to \( \langle 11–2 \rangle \). An example for a zigzag and for an armchair edge is indicated by green lines.
armchair edges in about equal abundance in this image. This observation is in contrast with the suggestion in [21] that zigzag edges prevail for hBN islands on Ir(111). Encouraged by the success of finding the predicted and previously found preference for (1–10) steps as discussed above we now go one step ahead and can actually assess the orientations of the edges. To this goal we directly transfer the prominent real space (1–10) and (11–2) directions as obtained from the diffraction pattern in figures 1(c) and (d) as indicated by solid blue and dashed red lines, respectively to the real space image in figure 1(f). It is then obvious that the parallel sides of the trapezoid are formed by (11–2) edges and thus are of armchair type, while the sides indicated by the solid blue lines are (either of the zigzag type or of the dimer terminated type (see figure 6(d) of reference [15])). We note that the nominal 30° angle in all right angled trapezoids tends to be rather more blunt. This is attributed to the system’s way of dealing with the energetically very unfavorable sharp corners.

A triangular hBN island is imaged too near the center of figure 1(b). Although this island is not perfectly equilateral it is obvious that the by far dominant orientation of the edges is along (11–2). The edges therefore have a predominant armchair character. As elaborated in [15], these armchair edge orientations are energetically favored when compared to zigzag edges. This is in contrast to graphene flakes for which zigzag edges are thermodynamically favored by a factor $2/\sqrt{3}$. The difference between graphene and hBN is attributed to polar contributions to the zigzag edge energy resulting from the mixed ionic and $sp^2$ nature of the binding in hBN islands. This very nature is also responsible for the exclusive nucleation on step edges due to Smoluchowski type charge redistribution associated with steps at metallic surfaces.

The exact determination of the real space directions in figure 1 is crucial to settle the issue of armchair versus zigzag edges [15, 21, 22]. Therefore, we will discuss in detail an elegant method of getting exact, direct and quantitative access to this question by means of a brief excursion. Figure 3(a) shows a measured LEEM image of clean Ir(111) after numerous growth experiments at 1150–1200 K. The field of view is 20 µm. The wavy stripes show step edges on Ir (111) and we distinguish two regions (labeled I and II) with preferred step orientations, which differ by 90°, which is a consequence of a change in the preferred step orientation after numerous growth experiments [15]. The border between both areas is a quite straight line at an angle of about 45° with the preferred step directions in both areas, i.e. the fault is oriented at about [3–1–2]. The result is similar to that reported in [15] except for the orientation of the fault line. Figure 3(b) shows an image of the same area, but with a different magnification (field of view is 50 µm). The image is rotated by 90° with respect to the one in figure 3(a). The rotation is attributed to the rotation due to magnetic lenses in our instrument. This illustrates that unknown rotations may affect a comparison of the orientations of images in real space w.r.t. to the orientation of the measured integral LEED patterns.

In principle, the presence of steps leads to a broadening of the specular peak in the direction perpendicular to the steps and quantitative information on the step distribution is accessible via a careful analysis of the profile of, e.g. the specular beam by means of SPALEED for destructive interference conditions for adjacent terraces [27, 28]. The sensitivity is then determined by the angular spread and the transfer width of a typical SPALEED instrument is about 150 nm. Because the integral information is useless in this case we apply $\mu$LEED and have to accept a loss of resolution. To counteract this drawback we use the lowest energy data since the transfer width is proportional to the applied wave length of the probing electrons [29]. The procedure is elaborated using the data shown in figure 4. Figure 4(a) shows a 30 × 30 µm² area taken from figure 3. The $\mu$LEED pattern obtained from the encircled area in figure 4(a) is exhibited in figure 4(b). The used aperture has a diameter of 1.4 µm. We attempt to obtain directly exact information on the step orientation in region II via measurements at $\lambda = 4 \cdot \Delta h / n$, with $\lambda$ is the electron wave and $\Delta h = 2.22$ Å is the atomic step height on Ir(111). Destructive interference conditions hold for odd values of the integer $n$, while constructive interference occurs for even
values of \(n\). The first destructive interference condition occurs at \(\lambda = 8.88\,\text{Å}\), or \(E = 1.9\,\text{eV}\), etc. Figure 4(c) shows profiles of the specular spot along \([1\,\text{–}\,10]\). A broadening is just visible at 2.0 eV \((n = 1\), blue pluses\) when compared to the profile obtained under constructive interference conditions at 8 eV \((n = 2\), red diamonds\) as shown in figure 4(c). This is more clearly visualized by calculating the specular intensity measured at 2 eV divided by the one measured at 8 eV as illustrated in figure 4(d). We observe a split structure. Two features are observed which gradually disappear, i.e. are less and less well resolved, with increasing energy from 2 to 8 eV, see figures 4(d)–(f). The splitting occurs only along the \([1\,\text{–}\,10]\) direction and broadening is totally absent along \([1\,\text{–}\,12]\). This is exactly what is expected for steps running along \([1\,\text{–}\,12]\) with a finite terrace width distribution \([27, 28]\). This is further reinforced by figures 4(e), (f), taken at 3 and 4 eV. The splitting becomes less prominent and this is precisely expected when moving from destructive towards constructive diffraction conditions. The features in figures 4(g)–(i) show profiles obtained for, respectively, \(n = 3\), \(n = 4\) and \(n = 5\) divided by the one for \(n = 2\). The splitting does occur for odd \(n\) values and is absent for \(n = 4\). Its azimuthal orientation remains identical and the lesser prominent splitting with increasing energy is a direct consequence of the accordingly decreasing transfer width \([29]\). These observations unequivocally proof that elastic scattering effects govern the acting physics. We have now demonstrated directly, without any possible doubt and independent from image rotation by magnetic lenses, that the steps on region II areas are oriented along \([1\,\text{–}\,12]\). The preferred step orientations on region I areas are directed along \([1\,\text{–}\,10]\), see figure 5. By comparing the edge directions of the hBN islands with the preferred step directions we can now immediately derive the nature of the hBN edges as being oriented along \((1\,\text{–}\,10)\) or along \((11\,\text{–}\,2)\), the latter corresponding to the armchair type in both surface regions.

Before carrying on, we care to make a few general comments with respect to the discussed step assessment. The just introduced method to connect the crystallographic information from LEED to real-space directions on a local scale is applicable to any material with a high enough step density and parallel steps. The minimum required step density depends on the transfer width of the instrument, which is of the order of a few hundred Å. A conservative estimate of the required step density is then of the order of 1%. The accuracy of the obtained step direction depends also on the width of the step density distribution and the degree of orientation anisotropy.
These factors need to be considered in any specific situation in order to estimate the uncertainty in the obtained step direction. A conservative estimate for the error bar leads us to ±3° in the current case. A distinction between zigzag and armchair edges, which are 30° apart, is thus straightforward.

After this excursion we identify unequivocally the orientation of the hBN edges, grown on type I regions as displayed in figure 1. The parallel edges of the right angled trapezoid are of the armchair type, while the ones at relative angles of 30° and 90° run along ⟨11–2⟩ and are of the zigzag type, or see [15] of the dimer terminated zigzag type. Note that the edges of the triangle in the center of figure 1(a) are predominantly of the armchair type.

Now we address the shape and edge orientations of the hBN islands on type II regions, i.e. with steps running predominantly along ⟨11–2⟩. By applying the results outlined above, we can now identify in a straightforward and direct fashion the nature of the hBN edges grown on type II areas. The relevant data are exhibited in figure 6. It shows a frame taken from a LEEM movie during growth of hBN at 1150 K on a type II region. The arrow indicates the [-1-12] direction in a direct derivation from the μLEED patterns shown in figure 4 as described above. We conclude that all hBN edges nicely run along ⟨11–2⟩ and are thus overwhelmingly of the armchair type.

Figure 6. (a) Frame from a LEEM movie during growth of hBN (black features) on type II regions at 1150 K. FoV = 20 μm, electron energy 17.0 eV. The red arrow marks the [-1-12] direction as was obtained directly from μLEED pattern in figure 4 as described above. All edges all nicely aligned along ⟨11–2⟩ and thus of the armchair type. Figure 4(b) shows growing hBN islands. See for the information on the alternating bright and dark areas figure 1. Here the temporal increment is 275 s.

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As explained in [15] the islands which point in the direction from top left to the bottom right have nucleated in the inner corner of the steps and do not expand as readily across their parent step, while the islands pointing towards the upper left have nucleated on the upper side of the step and they are free to expand equally well in all directions.
Discussion and conclusions

The current finding is in contrast with the conclusion drawn in [22] that \(\langle 1–10 \rangle\), or zigzag, edges prevail. Their conclusion was based on images also taken with an Elmitec LEEM III. We assume that the authors of [22] may not have realized that a direct connection of the orientations of the \(\mu\)LEED pattern to those in the real space images is not as straightforward as in conventional LEED, or in SPALEED since the used magnetic lenses cause rotation of the images, depending e.g. on the chosen field of view (see figure 2 for an illustration). In any case, an explicit verification of the orientation of the LEEM images with respect to the \(\mu\)LEED pattern, as demonstrated above, is lacking.

It has been suggested that the edges of hBN are of the zigzag type and thus oriented along \(\langle 1–10 \rangle\) [21]. This information is obtained by means of STM from a vacancy hole in a closed hBN layer, i.e. at a relatively high coverage (see figure 1, [21]). The assumption that these edges represent the thermodynamic equilibrium structure was the basis for applying density functional theory calculations also for presumed small hBN ad-islands as opposed to small vacancy islands. I.e. the basic underlying assumption is that the equilibrium edges are of the zigzag type and that zigzag island edges govern the morphology. There are several problems with this approach, which we want to address below.

- First, the dominant and crucial role of steps in the island nucleation and morphology as revealed in this study is neglected completely.

- Second, depending on the prevailing local step direction we have identified right angled trapezoids as the equilibrium shape on type I (\(\langle 1–10 \rangle\) steps) regions. Irrespective of the attribution of edge directions it is obvious that both zigzag and armchair edges occur in equilibrium configurations with about 30° (150°) and 90° angles between them. Consequently, armchair edges are observed for relatively small hBN adatoms and steps. Note that these islands are still extremely large when compared to the calculated ones [21].

 Third, the triangular islands bordered by zigzag edges have to be either B- or N- terminated. This was realized early by Auwärter and colleagues [31]. The B-terminated ones were shown to be favored [21]. There is a severe problem with that result. In a growth situation this implies that the borazine decomposition even has to occur at the level of breaking B–N dimer bonds. This bond is the strongest bond in the system: and leads to boron films due to the preferential associative desorption of \(\text{N}_2\) [22, 32, 33]. This does occur at an about 200 K higher temperature, but involves a non-repairable loss of stoichiometry and the further growth of hBN becomes impossible. This holds in particular and critically for the small nuclei and the heavy relative loss of N simply cannot lead to the evolution of stoichiometric hBN films. This is in obvious contrast with our \textit{in situ} growth observations.

For completeness we note that zigzag edges, or better edges along \(\langle 1–10 \rangle\) azimuth, have been observed beyond any doubt for relatively small hBN islands on Ir(111) [34, 35]. The transformation into type II regions at the cost of type I regions is persistent during growth experiments of hBN on Ir(111). A quantitative analysis is unfortunately out of reach since the regions are macroscopic and step resolution is required. The relative increase of type II regions, i.e. areas with Ir-steps along \(\langle 11–2 \rangle\) is best illustrated by the gradually increasing ease of finding type II areas during the experimental series.

The massive transition of the step morphology, even resulting in rotation of the preferred direction is unusual in surface science. To the best of our knowledge this hBN induced change of the step morphology is quite unique and it must be attributed to a preference for armchair edges in combined with the ideal parallel alignment of hBN and Ir(111) and the exclusive nucleation of hBN on step edges in an intrinsic growth experiment.

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