Structure of epitaxial graphene on Ir(111)

Alpha T N’Diaye¹, Johann Coraux, Tim N Plasa, Carsten Busse and Thomas Michely
II. Physikalisches Institut, Universität zu Köln, Zülpicher Straße 77, 50937 Köln, Germany
E-mail: ndiaye@ph2.uni-koeln.de

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Abstract. A graphene monolayer has been prepared on an Ir(111) single crystal via pyrolytic cleavage of ethylene (C₂H₄). The resulting superstructure has been examined with scanning tunneling microscopy (STM) and low energy electron diffraction. It has been identified as a well aligned, incommensurate (9.32 × 9.32) pattern, which is described as a moiré. This pattern shows three distinct regions resulting from different local configurations of the carbon adlayer with respect to the Ir-substrate. These regions are imaged differently by STM and differ strongly in their ability to bind metal deposits.

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¹ Author to whom any correspondence should be addressed.
1. Introduction

Graphene, the two dimensional (2D) sp² hybridized carbon crystal, has received increasing attention in the last 4 years [1]. Although theoretical studies date back to the 1950s [2], the interest in graphene has been very much stimulated by experimental studies of its electronic features, especially electronic transport mimicking quantum relativistic charge carriers [3]. Graphene’s unique band structure, room temperature ballistic electron transport, and the possibility to manufacture graphene transistors make it a promising candidate for future applications.

Today, there are three major methods of graphene preparation: (i) mechanical exfoliation from a bulk graphite crystal [4], (ii) thermally induced surface segregation from a carbon containing substrate-like ruthenium [5] or carbides (SiC) [6] and (iii) chemical decomposition of a carbon containing gas on a metal surface [7, 8].

Mechanical exfoliation gives very large flakes of high quality, but it is a very sophisticated and time-consuming process. Preparing graphene through segregation produces large graphene flakes, but their defect density and the typical structure sizes are rather undefined. The chemical vapor deposition (CVD) of graphene may be one route to grow high quality epitaxial graphene in a controlled manner. This path, however, is currently only viable on metal surfaces.

The chemical deposition of carbon on metal substrates has been extensively studied from the 1970s to 1990s, often because carbon films act as a poison for catalysts but the properties of graphene have not been investigated in detail. Due to the increasing importance of graphene, it is desirable to obtain a thorough understanding of the structure of graphene on metals and of its interplay with the underlying substrate.

Moirés and moiré-like superstructures are a widespread phenomenon in epitaxial growth [9, 10]. It is not always clear whether the meshes are to be considered incommensurate or commensurate [11], whether a mismatch in lattice parameter is accommodated by strain relief [12, 13] or whether strain only plays a negligible role. Such superstructures have relevancy for nanopatterning, as in some cases they enable templated growth of clusters [14]–[16].

The aim of this paper is to clarify the structure of graphene on Ir(111) on an atomic level. We will briefly address the preparation of epitaxial graphene on Ir(111), describe the resulting superstructure and present a model for the superstructure unit cell. Three different regions are found in this cell which are also visible in scanning tunneling microscopy (STM) topography data. We show the chemical inhomogeneity of the moiré cell by post-decoration experiments. Finally, STM imaging issues will be addressed.

2. Experimental

Experiments were carried out in an ultra high vacuum variable temperature STM apparatus with a base pressure in the 10⁻¹¹ mbar range equipped with a mass separated ion source, an Ir evaporator, a low energy electron diffraction (LEED) system, and a gas doser for ethylene approximately 2 cm above the sample surface. Due to the gas doser the ethylene pressure at the substrate surface exceeds the ethylene-induced pressure rise in the vacuum system by a factor of ≈170. The substrate was cleaned by repeated cycles of annealing to 1500 K and sputtering with a beam of 1.5 keV Xe⁺ ions at 1100 K. Graphene was prepared through exposure of the substrate to >99.95% pure ethylene. For post deposition experiments iridium was evaporated from a current heated >99.9% pure Ir-wire with a standard deposition rate of 3.0 × 10⁻³ ML s⁻¹,
where 1 ML (monolayer) is the areal density of the Ir(111) surface (1.57 × 10^{19} \text{ m}^{-2}). Precise deposition rate calibration was performed by STM image analysis of the fractional area of Ir islands on the clean Ir(111) surface after a defined deposition time. Besides the usual topographic gray scale images we also show in a few cases (figures 1(a) and 7) differentiated topographs which appear as if illuminated from the left.

3. Graphene preparation

For the data presented in this paper, we mainly used the preadsorption method of graphene preparation.

Ethylene is known to have a sticking coefficient near unity on the Ir(111) surface. At room temperature it partly dissociates there and decomposes entirely upon annealing. This behavior has been studied by thermal desorption spectroscopy, electron spectroscopy methods and alkali ion flashing [17]–[20]. Annealing the substrate covered to saturation in UHV leads to complete decomposition of the hydrocarbon. Decomposition is completed at 820 K [17].

We have exposed the Ir(111) surface to the ethylene doser for 30 s at a chamber pressure of 2 × 10^{-7} \text{ mbar} to ensure saturation coverage. After annealing to 1470 K for 40 s STM imaging displays graphene flakes covering about 22% of the substrate surface with a typical lateral dimension of the order of 1000 Å. The simultaneous presence of Ir and graphene on the surface is of great help for the structure determination (see below).

A second, from an application oriented point of view, more viable route to graphene is CVD via the exposure of the hot iridium surface to ethylene. For experiments using the CVD growth method presented here, the chamber pressure was set to 2 × 10^{-9} \text{ mbar} with the hot sample exposed to the gas inlet for 20 s at 1320 K. The ethylene decomposes through pyrolysis at the hot Ir(111) surface, and the resulting carbon forms a graphene layer. Since the decomposition can only take place on the uncovered Ir(111) surface, the process is self-limiting and multiple layer regions are avoided. The resulting graphene sheets cover large areas of the surface and the structures found are typically too extended to assess their size with STM (larger than 1 \mu m).

These sheets are coherent over step edges and domain boundaries [21]. STM topographs of a sample prepared with the CVD method are shown in figures 8(a) and (b).

The kinetics of graphene formation through the two methods will be discussed in detail in a forthcoming paper.

4. LEED and STM results

Graphene on Ir(111) was examined by LEED and STM. Figure 1(a) shows a differentiated STM topograph of the sample prepared with the preadsorption method. Graphene flakes together with uncovered areas are visible on the Ir(111) surface. A superstructure is clearly visible on the flakes. The atomically resolved STM topograph in figure 1(b) shows an iridium terrace on the left to which a graphene flake is attached from the right. It is a detailed view of a situation present in many places in figure 1(a), where graphene flakes are attached to iridium steps. In figure 1(b), the superstructure unit cell is marked with a white rhombus on the atomically resolved graphene flake.

The STM approach allows us to measure the superstructure and carbon lattice periodicity in direct comparison with the atomic nearest-neighbor distance of 2.715 Å [22] in the Ir(111) surface. The carbon lattice has a periodicity of 2.45 ± 0.04 Å as measured with STM.
Figure 1. (a) Graphene on Ir(111). The moiré with its 25.3 Å periodicity is clearly visible (1300 Å × 1300 Å, $U_T = 1.5$ V and $I_T = 0.5$ nA). (b) Attached to an iridium step edge (left) lies a graphene flake (100 Å × 100 Å, $U_T = -0.17$ V and $I_T = 21$ nA). The superstructure unit cell is marked as a white rhombus. (c) LEED pattern of the partially graphene covered Ir(111) surface (inverted, 80.4 eV). The spots consist of two main spots (inner spot due to iridium (111) and outer spot due to graphene) which are surrounded by smaller satellite spots reflecting the periodicity of the moiré. Inset: the center spot (46.7 eV) is also surrounded by moiré-induced spots. (d) Graphene on Ir(111). An atomic carbon row has been highlighted by white dots. The row is in parallel with the superstructure (154 Å × 54 Å).

the limits of error this agrees with the periodicity of a single carbon layer in graphite (2.4612 Å) [22]. The superstructure unit cell repeat vector has a length of 25.2 ± 0.4 Å.

A symmetric LEED pattern of this surface can be observed (figure 1(c)) [17]. It consists of six pairs of spots. The inner spot of each pair originates from the Ir(111) surface, the outer spot is due to the carbon overlayer which has a smaller lattice repeat vector than the iridium surface.
Figure 2. A moiré is a superposition of two lattices generating a third one. On the left, there are two misaligned stripe patterns with slightly different stripe separations. The moiré appears as a striped diagonal pattern with a much larger periodicity. One may assign reciprocal lattice vectors to these patterns that are normal to the stripes. The reciprocal lattice vector of the moiré is then given by the difference of the reciprocal lattice vectors of the two constituting vectors. This is visualized on the right side, where the corresponding lattice vectors are shown. The direction of the difference vector is aligned with the moiré.

Each pair of iridium and carbon spots is surrounded by a group of satellite spots, signifying a superstructure with a large periodicity. For the inset of figure 1(c), the sample has been tilted by a few degrees making the central spot of the LEED image visible on the screen. Also this central spot is surrounded by six satellite spots in line with the underlying iridium substrate and the carbon spots. The fact that only first-order satellite spots occur and that they do not fill out the complete surface brillouin zone indicates that the superstructure is incommensurate.

The lattice constant of the carbon overlayer is also determined by LEED with the Ir(111) surface as a reference. The resulting repeat vector of the graphene layer amounts to $2.47 \pm 0.02$ Å again in agreement with the literature value for a single carbon layer in graphite. The LEED measurement delivers a value of $25.8 \pm 2.0$ Å for the periodicity of the superstructure. As shown in the inset of figure 1(c) the superstructure spots are quite close to the specular beam, so that the error of the reciprocal space measurement becomes fairly large.

STM and LEED also show that the dense packed [110]-direction of the iridium substrate and the [1120]-direction of the graphene layer are parallel.

5. The moiré of graphene on Ir(111)

The graphene superstructure can be viewed as a moiré. This regular pattern, resulting from the superposition of two regular lattices—just like the beat that emerges from the superposition of two sound waves—has a reciprocal lattice vector, which is the difference of the constituting reciprocal lattice vectors.

Figure 2 shows this for a 1D example. The patterns on the left have reciprocal lattice vectors $\mathbf{k}_1$ and $\mathbf{k}_2$, corresponding to the vectors drawn on the right. The moiré effect produces a
Table 1. The crystallographic parameters according to the three methods described in the text.

| Parameter                      | Direct STM | LEED    | Moiré analysis |
|--------------------------------|------------|---------|----------------|
| Graphene repeat distance $d_C$ | (2.45 ± 0.04) Å | (2.47 ± 0.02) Å | (2.452 ± 0.004) Å |
| Moiré repeat distance $d_{\text{moiré}}$ | (25.2 ± 0.4) Å   | (25.8 ± 2) Å    | (25.3 ± 0.4) Å    |
The superstructures on different graphene flakes in figure 1(a) are slightly misaligned with respect to each other. Following the atomic carbon rows in figure 1(b) confirms that in contrast to the situation described for measuring the lattice parameter of the graphene (figure 1(d)), the orientation of the superstructure deviates slightly from the atomic rows. According to equation (1) this can only be due to a misalignment between the iridium substrate’s and the graphene’s dense packed rows. A direct STM measurement of this misalignment gives values below 0.5° which are in the range of the error. As for the lattice constant of graphene, we can take advantage of the moiré’s magnification effect. The rotation angle is magnified by the superstructure. Let φ_{C,Ir} be the angle between the two original lattices and φ_{C,moiré} the angle between graphene lattice and the moiré. It can be derived from equation (1) that
\[
\sin(\phi_{C,Ir}) = \left(\cos(\phi_{C,Ir}) - \frac{k_C}{k_{Ir}}\right) \tan(\phi_{C,moiré}).
\]
(4)
For small angles φ_{C,Ir} and φ_{C,moiré}, the equation simplifies to
\[
\phi_{C,Ir} = \frac{k_{Ir} - k_C}{k_{Ir}} \cdot \phi_{C,moiré}
\]
(5)
with a constant factor of \left(\frac{k_{Ir} - k_C}{k_{Ir}}\right)^{-1} \approx 10.6 for the angular magnification.

With knowledge of the lattice parameters and the angle between the moiré and the atomic rows of one lattice, the tilt of the two lattices with respect to each other can be determined. The moiré amplifies angular misorientations of the graphene layer with respect to the substrate into its own misorientation with respect to the substrate.

We examined the angular scatter of the moiré on the flakes from a number of experiments prepared with the preadsorption method. In some the flakes were decorated with iridium clusters, as described below. A histogram is displayed in figure 3. The scatter of the moiré translates into a measure for the angular imperfections of the carbon rows with respect to the substrate. Formula (5) reveals the measured moiré scatter of ±2.6° to originate from an angular spread of ±0.25° on the atomic level. Note that this spread is not an uncertainty, but a measure for the epitaxial perfection of the graphene flakes on the iridium substrate. The uncertainty for each measurement of the angle between the dense packed \{1\bar{1}0\}-direction of the iridium substrate and the \{11\bar{2}0\}-direction of the graphene layer is about 0.04°.

The LEED image and the non-integer value of atomic iridium and graphene sites per moiré cell show that graphene on Ir(111) is an incommensurate system. Nevertheless, the crystallographic \{1\bar{1}0\}-directions of the Ir substrate are well aligned with \{11\bar{2}0\}-directions of the deposited graphene and so epitaxial relations are fulfilled. Graphene on Ir(111) is thus an example of incommensurate epitaxy.

6. Moiré unit cell

After discussing the lattice of the moiré, we will now address the anatomy of the single moiré cell which is the base of the surface structure. For simplicity, we will only discuss the case of perfect alignment. All following considerations also hold for slightly rotated meshes, as they are present in the experiments. Both constituting lattices are known. First there is the unreconstructed (111)-oriented iridium fcc crystal substrate. On this surface, the sites with the highest symmetry are the atop sites, directly above the iridium atoms and the two kinds of
threefold coordinated adsorption sites, one of which corresponds to regular fcc stacking and the other to faulted hcp stacking. Note that hcp and fcc sites are very similar. Both are local energy minima for iridium adatom adsorption and their differences arise only from discrepancies in the second layer of the iridium surface.

The counterpart of the iridium lattice in the moiré is the graphene structure, which consists of the well-known hexagonal honeycomb. A model can easily be generated by geometric superposition of one onto the other. Such a model is shown in figure 4.

An inspection of this model shows three prominent regions, marked by white circles. The regions differ by the arrangement of the carbon atoms with respect to the underlying iridium surface sites. The labeling corresponds to the type of iridium surface site, which is located below the center of the carbon rings in this region. The fcc region (hcp region) centers an iridium fcc site (hcp site) in the honeycomb while every second carbon atom is positioned in the other threefold coordinated site, namely the hcp site (fcc site) and every second atom is on top of an iridium substrate atom. In contrast, in the atop region, the iridium substrate atom lies right in the center of the ring, while the six carbon atoms occupy all threefold coordinated sites.

The geometrical model shows that the fcc and hcp regions have similar atomic configurations—one carbon atom above an iridium atop site, and the other above an hcp or an fcc site, respectively—while the atop region differs from those, because no carbon atom is poised over an iridium surface atom and all carbon atoms are located at the threefold coordinated hollow sites. To examine the properties of the different regions, it is necessary to link them to their appearance in the STM topographs.

Figure 5 shows a magnification of the topograph from figure 1(b). The iridium terrace is partly shown on the left, the carbon flake is attached to it from the right. A mesh has been adjusted to match the positions of the iridium atoms on the terrace with the intersections of the lines. (We assume the bright spots on the iridium terrace to correspond to the iridium atoms...
Figure 4. The superstructure unit cell is constructed by superposition of the graphene lattice (yellow balls) onto the iridium (111) surface (gray balls). The darker shades of gray represent the second and third layers of the iridium substrate. In the atop regions (white arcs in the corners of the cell), an iridium atom of the topmost layer is centered in the graphene honeycomb. The carbon atoms cover the threefold coordinated hollow sites. In the fcc region (dashed circle) and the hcp region (dotted circle) there are threefold coordinated hollow sites centered under the carbon ring’s center: either an fcc site (fcc region) or an hcp site (hcp region). Every second atom is located at the other hollow site and every second above an iridium surface atom.

and the dark spots on the graphene layer to correspond to the center of the carbon rings, where the electron density is lowest [24]. Prior to the magnification, the mesh given by the iridium surface atoms of the terrace was extended to also span over the carbon covered part.

The sketch on the right helps to interpret the lines. It depicts a small part of the iridium substrate, which is oriented in the same way as the sample. There is an upper terrace on the left, corresponding to the uncovered iridium in figures 1(b) and 5. The lower terrace on the right corresponds to the carbon covered part of the substrate. The carbon is not shown in the sketch, so that the arrangement of iridium substrate atoms can be seen.

Since the crystal is fcc stacked, an intersection of the lines above the lower terrace marks an fcc adsorption site. The lower terrace’s hcp sites are centered in triangles pointing to the left, while atop sites are found on rightwards pointing triangles of the mesh. This way a glimpse under the adsorbed graphene layer is possible.

Although STM distortions introduce some uncertainty, the regions where the carbon ring centers are in a rightwards pointing triangle of iridium substrate atoms (red triangle), can be found in or close to the dark regions of the moiré. This is the atop type configuration as marked by solid white circles in the corners of the unit cell shown in figure 4. Accordingly, fcc (green dots) and hcp (blue triangle) regions are located in the corresponding positions of the moiré as expected from figure 4 (dashed and dotted circles). This way it is possible to relate the
Figure 5. STM topograph (zoom of figure 1(b)) showing an iridium terrace on the left with an attached graphene flake, both atomically resolved (image width 100 Å). An Ir-grid was extrapolated over the whole of the image derived from the maxima of the Fourier transform of the area inside the white box to the left. The intersections of the mesh correspond to atom position on the step as shown in the sketch. In the same way, from the Fourier transform of the area inside the box to the right, a C-grid was determined. The positions of the dark spots derived in this way are marked by the colored symbols. Finally, each Ir unit cell was divided into three regions of equal size (fcc, hcc and on-top) and the points of the C-grid were thus assigned to the different stacking types (fcc: green circle, hcp: blue triangle and on-top: red triangle). It is obvious that for the part of the graphene closest to the atomically resolved Ir (i.e. where the extrapolation is most reliable), the prominent moiré sites correspond to on-top regions.

local stacking to STM topography data, which is the basis of attributing specific properties to an atomic configuration. The dark spots of the moiré in figure 5 can thus be identified as the atop regions, where all carbon atoms occupy the threefold coordinated sites and the atop site is centered in the carbon ring. The hcp and the fcc regions are in downward and upward pointing triangles of atop regions. This assignment will be backed up by the results concerning the chemical inhomogeneity of the moiré.

7. Chemical inhomogenity

Along with the change in appearance goes a change in chemical properties. The inhomogeneity in binding can be probed by postdecoration with iridium as described in the experimental section. Figure 6(a) shows an STM topograph of a graphene flake on the Ir(111) surface. The flake is attached to a step edge. This surface has been decorated with 0.10 ML of iridium [16]. On the uncovered part of the substrate well-known island growth takes place [25]. On the graphene covered part of the substrate, iridium clusters nucleate in the pattern precoined by the moiré. So the binding energy for iridium is obviously lower in some regions than in others.
Figure 6. (a) STM topograph (1000 Å × 1000 Å, $U_T = 0.6$ V and $I_T = 0.3$ nA) of postdecorated graphene on Ir(111). The flake in the middle is attached to a step edge. The step edge is deformed, where it nestles to the graphene. Evaporated iridium (0.10 ML) nucleates islands on the bare substrate while there is a formation of regularly ordered clusters on the moiré of the flake. (b) Deposition of small amounts (0.01 ML) leads to the nucleation of few clusters only. The moiré is atomically resolved and the clusters appear as large bright spots. According to the determination in figure 5 the adsorption sites of the clusters are identified as the hcp regions (90 Å × 90 Å, $U_T = 0.20$ V and $I_T = 9.7$ nA).

To assess which region of the moiré is involved in cluster binding, we have deposited a small amount (0.01 ML) of iridium on the moiré leading to only a fraction of moiré cells being populated with clusters. Figure 6(b) shows an STM topograph with atomically resolved moiré and some clusters. The clusters are not adsorbed on the dark atop regions, but in downward pointing triangles of those regions. A comparison with figures 4 and 5 shows that these are hcp regions. Experiments with higher coverages [16] confirm that clusters exclusively bind on hcp regions if grown at 350 K.

At lower growth temperatures, however, cluster binding also takes place at fcc regions, as demonstrated in figure 7. In this experiment 0.04 ML have been deposited at a temperature of 160 K instead of 350 K. In the right part, the hcp sites have been blanked out with black circles to facilitate recognition of the periodicity. This blanks out the majority of the clusters. The remaining clusters are adsorbed in downward pointing triangles of hcp regions. The white rhombus in figure 7 illustrates that these clusters are adsorbed in the fcc regions as defined in the unit cell of figure 4. This second energy minimum at the fcc regions is no surprise at all, because as discussed above, hcp and fcc regions realize very similar local atomic configurations.

Based on local density approximation calculations, cluster binding can be attributed to a local sp$^2$ to sp$^3$ rehybridization of the graphene layer [26].
Figure 7. At 160 K the clusters nucleate at hcp and fcc regions. Differentiated topograph (420 Å × 420 Å, $U_T = 1.6$ V and $I_T = 0.4$ nA) of graphene on Ir(111) which has been postdecorated with 0.04 ML of iridium at 160 K. Although the moiré defines the registry, the order is less pronounced than for deposition at higher temperature as in figure 6. Blanking out the regular cluster positions with black circles (right part) reveals that most of the clusters are within that lattice, while some are located in downward pointing triangles of those regular cluster nucleation sites. The white rhombus corresponds to the unit cell of figure 4. A comparison shows that these are fcc regions.

8. Contrast inversion

Tunneling parameters and tip states play a major role in imaging the moiré. Figures 8(a) and (b) show two STM topographs of the same sample position. They were recorded subsequently under stable conditions without any tip switch. Figure 8(a) was imaged with a low tunneling voltage (0.32 V) while a high voltage (1.5 V) was chosen for figure 8(b). The insets (recorded at different positions) show the moiré with atomic resolution. The moiré is imaged in two different ways: in part (a) of the figure, the atop regions appear as dark depressions in a bright surrounding (dark-atop contrast), just as shown throughout the preceding paragraphs. In part (b), the moiré contrast has inverted. Now the atop regions are imaged as bright protrusions in a dark surrounding (bright-atop contrast). One can easily confirm that the contrast has inverted and that not just another region of the moiré is emphasized by the changed tunneling conditions: the position of the brightness extrema with respect to the kinks in the preexisting step edge does not shift. The observation of the voltage-dependent contrast inversion requires a sharp tip and stable conditions. Low tunneling currents ($< 0.5$ V) favor the bright-atop contrast, higher...
voltages favor the dark-atop contrast. Such a contrast inversion is reversible without any sign of a tip change.

In figure 8(c), there are two profiles from the inset of figures 8(a) (red) and (b) (blue). These profiles represent the corrugation across the moiré cell. The position of the individual
carbon atoms is modulated on the moiré-induced long-range brightness oscillation which has been scaled to ±1 au. In both cases, the atop region and the fcc region mark the extrema and the hcp region’s brightness is closer to that of fcc regions than that of atop regions. This is in accordance with the fact that hcp and fcc regions are based on similar local configurations.

The contrast can not only be inverted by varying the tunneling parameters, but it is also affected by tip switches. Figure 9 shows an STM topograph which was measured with an unstable tip. The upper part is imaged in dark-atop contrast and the lower part—after a tip switch—is imaged with bright-atop contrast. We expect this switch to happen due to a contamination of the tip with a foreign molecule or atom-like CO, ethylene, or carbon, which changes its density of states. So, other states of the graphene layer are predominantly probed, as the overlapping states of the tip and the sample determine the tunneling current. In contrast to our previous publication [16], where only bright-atop contrast images were used, in the current paper we show both kinds of topographs.

These rich electronic features demand further investigation with scanning tunneling spectroscopy, which is currently being undertaken.

9. Summary

Graphene can be epitaxially grown on Ir(111) with the Ir [110]-direction parallel to the graphene [1120]-direction despite their incommensurate lattices. The mismatch of the lattices gives rise to long scale moiré with a periodicity of 25.3 Å. The moiré can be used to magnify atomic misorientations. The orientational scatter of the atomic rows of graphene flakes grown by thermally decomposing an ethylene layer at 1470 K for 20 s is 0.25°.
The fcc, hcp and atop regions in the moiré differ in their local atomic arrangement. Nevertheless the hcp and fcc regions behave similarly in several ways: both can harbor iridium clusters, as grown in post decoration experiments. The local binding energy minimum for iridium clusters is deepest in the hcp regions. Clusters nucleate there even above room temperature. Hcp and fcc regions are imaged with comparable corrugations, while the atop region is imaged either much brighter or much darker than the other regions, whenever there is moiré contrast.

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