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ABSTRACT

We study the origin of layer decoupling in ordered multilayer graphene grown by high temperature sublimation on C-face 4H-SiC. The mid-infrared optical Hall effect technique is used to determine the magnetic field dependence of the inter-Landau level transition energies and their optical polarization selection rules, which unambiguously show that the multilayer graphene consists of electronically decoupled layers. Transmission electron microscopy reveals no out-of-plane rotational disorder between layers in the stack, which is in contrast to what is typically observed for C-face graphene grown by low temperature sublimation. It is found that the multilayer graphene maintains AB-stacking order with increased interlayer spacing by 2.4%–8.4% as compared to highly oriented pyrolytic graphite. Electron energy loss spectroscopy mapping reveals Si atoms trapped in between layers, which are proposed to be the cause for the observed increased interlayer spacing leading to layer decoupling. Based on our results, we propose a defect-driven growth evolution mechanism for multilayer graphene on C-face SiC via high temperature sublimation.

The unique linear dispersion around the Dirac point and the free charge carriers behaving like massless Dirac fermions are key signatures of graphene that make it a very promising material for analog electronics in a postsilicon era. Graphene grown by sublimation on silicon carbide (SiC) offers a viable route toward the production of large-scale electronic grade single-crystalline material on the semi-insulating (SI) substrate without the need of transfer. In particular, sublimation of C-face 6H- or 4H-SiC(0001) and 3C-SiC(111) enables the growth of multilayer graphene (MLG), which behaves like a single layer graphene with very high charge carrier mobility parameters (>10^4 cm^2 V^{-1} s^{-1}). The latter is a result from the decoupling of the individual graphene layers in the stack.

Layer decoupling has previously been attributed to an out-of-plane rotational disorder between graphene sheets in the stack for MLG grown at temperatures below 1700 °C. More recently, we have shown that MLG grown by high temperature sublimation (1850 °C–2000 °C) on the C-face of both 4H-SiC and 3C-SiC is decoupled without an out-of-plane rotation between the layers. Different factors might be potentially invoked to explain the decoupling phenomenon in such an ordered MLG, e.g.,
intercalated species, defects, imperfections, or different ordered stackings. Understanding the origin of layer decoupling and its interrelation to stacking in MLG on SiC is decisive for future device applications, since it provides a route to the unique electronic nature of graphene. For example, AA stacked bilayer graphene (when the two layers are exactly aligned) exhibits a massless Dirac spectrum, while for minute twist between layers of the order of 0.1°–0.4°, an unusual electronic spectrum consisting of massive and massless Dirac fermions that can support valley Hall transport was observed. Layer stacking and alignment between individual layers in MLG also have impact on its radio frequency (rf) transmission characteristics (loss impedance, resistance, conductance, and capacitance), which are important for rf device applications. Decoupled MLG can be engineered for light trapping across an ultrabroad spectral range, which is relevant for a large number of energy, optoelectronic, and spectroscopic applications. However, the origin of the decoupling in ordered MLG on SiC remains unknown.

Mid-infrared-optical Hall effect (MIR-OHE) is a powerful method for studying the inter-Landau level (inter-LL) transitions in MLG and their dependence on magnetic field strength, which provide detailed information on electronic states and layer coupling. In this work, we combine MIR-OHE investigations with scanning transmission electron microscopy (STEM), electron energy loss spectroscopy (EELS), and energy dispersive x-ray (EDX) spectroscopy to reveal the origin of layer decoupling in ordered MLG on SiC and propose a model for the growth process evolution.

MLG with a thickness ranging between 0 and 25 layers was grown by high-temperature sublimation on a chemically mechanically polished 4H-SiC(0001) substrate. Before growth, the substrate was cleaned using standard RCA cleaning procedures, dipped in HF to remove surface oxides, and additionally annealed at 1200 °C for 20 min in vacuum (∼10⁻⁶ mbar) inside the growth reactor. The growth was performed in an Ar (99.9997%) pressure of 870 mbar at 1950 °C for 1 h in a furnace made of a quartz tube and stainless steel. The number of graphene layers was determined by reflectivity mapping measurements. About 94% of the sample contains between 8 and 19 layers, 4% contains 7 layers or less, and 2% contains 20 or more layers.

MIR-OHE measurements were performed in the spectral range of 1100–3500 cm⁻¹ using Mueller matrix ellipsometry. The upper 3 × 3 block of (Mij, i, j = 1, 2, 3) of the 4 × 4 Mueller matrix was measured. The measurements were performed in reflection mode at 45° angle of incidence and magnetic fields up to 8 T with the magnetic field direction parallel to the incident beam (with the magnetic field component perpendicular to the sample given as B⊥ = B/√2). The sample temperature was kept at 2 K. Analysis of the Mueller matrix spectra provides information on inter-LL transition energies and optical polarization selection rules.

The MIR-OHE measurements of our MLG reveal a number of peaks, associated with the inter-LL transitions, which only occur in the block-on-diagonal Mueller matrix element spectra (ΔMij, where ij = 12, 21, 22, 33) (see Fig. S1 of the supplementary material). No inter-LL transitions are observed in the block-off-diagonal element spectra (ΔMij, where ij = 13, 31, 23, 32). Such polarization preserving rules are characteristic for single layer graphene or for MLG consisting of decoupled layers. The inter-LL transition energies extracted from all experimental Mueller matrix spectra show a clear square root dependence on the magnetic field strength, as depicted in Fig. 1. The model for inter-LL transitions with the quantized n-th Landau level energy EN for single layer graphene is given by

\[ E_n = v_F \sqrt{2eB_n} n, \]  

where \( v_F \) is the Fermi velocity of Dirac fermions, \( e \) is the elementary charge, and \( B_n \) is the magnetic field strength. The model is fitted to the data points by varying the Fermi velocity parameter. The best fit lines of the inter-LL transition energies with this equation are also shown in Fig. 1. The obtained Fermi velocity parameter is

\[ v_F = 1.03 \times 10^6 \text{ m/s}, \]

which is in good agreement with values typically reported for single layer graphene or decoupled MLG. It is seen that all inter-LL transitions match well with the square root dependence on magnetic field strength. Transitions deviating from this dependence on magnetic field strength (such transitions appear in MLG with electronically coupled layers) were not detected. These results clearly indicate that our C-face MLG is composed of decoupled graphene sheets with linearly dispersing electronic bands (Dirac cones) at the K points of the first Brillouin zone.

Furthermore, an estimate of the free charge carrier scattering time and mobility parameters in our MLG can be obtained from the width of the peak corresponding to the B LL-transition and the lowest magnetic field at which the transition is observed following Ref. 7. A scattering time of about 100 fs and a lower limit of the free charge carrier mobility of about 20 000 cm²/Vs at 2 K were estimated. Note that the LL orbit radius is of the order of few tens of nanometers, and thus, the derived scattering time and mobility parameters should be regarded as local. We further point out that the reported value represents an ensemble average of local mobility parameters over the entire MLG area of 15 mm × 15 mm probed by the MIR-OHE.
To investigate the structural properties of our MLG and explore the origin of the observed layer decoupling, we employed high-resolution STEM. TEM specimens of the graphene samples were prepared for cross-sectional analysis by the standard sandwich method for mechanical polishing and subsequent argon ion milling. Imaging was carried out using the double aberration corrected Linköping Titan™ 60-300 (S)TEM equipped with a monochromated high brightness Schottky field emission gun (XFEG) operating at 300 kV, a FEI High Angle Annular Dark Field (HAADF) detector, and a Gatan Ultrascan 1000 CCD camera. The point resolution after correction was measured to be ∼0.7 Å. High-resolution STEM images were acquired along the [1 1 0] direction of the 4H-SiC substrate using high speed low-dose imaging acquisition (5 μs dwell time and 10 pA probe current). Knock-on damage caused by the electron beam was significantly reduced, and at the same time, a high resolution was obtained. Image postprocessing was performed using the Gatan digital micrograph with built-in routines. EELS was performed in Dual EELS mode employing the embedded GIF Quantum ERS at an energy resolution of ∼1.3 eV. EDX was acquired using a Super-X detector, and quantification was performed by employing the Cliff-Lorimer method as implemented in the ESPRIT software.

Figure 2 presents cross-sectional HAADF-STEM images of representative regions with different thicknesses: (a) thin, 7 layers (∼4%); (b) medium, 13 layers (∼94%); and (c) thick, 20+ layers (∼2%). The insets in (a) and (b) present Fourier filtered subsections highlighting the stacking sequence of MLG, and the inset in (c) shows a HRTEM image of 20+ graphene. Furthermore, an interface layer between the SiC and the graphene stack could be well resolved in the HAADF-STEM images in Fig. 2. The thickness of this interface layer increases with the increasing number of the graphene layers in our MLG.

The HAADF-STEM images in Figs. 2(a)–2(c) were acquired along the [1 1 0] direction (zigzag axis) of graphene that exhibits two different C column separations of 0.71 Å and 1.423 Å. Because of the ∼0.8 Å resolution of the probe, the C columns separated by 0.71 Å are instead depicted as a single smeared out dot feature separated by an average distance of 2.13 Å (see Fig. S2 of the supplementary material). However, the stacking sequence is still possible to determine. The AB stacking sequence with an interlayer spacing of 3.35 ± 0.02 Å, typical for the highly oriented pyrolytic graphite (HOPG), can be clearly identified for thin MLG with 7 layers [inset of Fig. 2(a)]. The lack of any rotational disorder in our MLG is confirmed by the measured average C column separation of 2.12 ± 0.12 Å for all the layers (see Fig. S2 of the supplementary material). If a mutual rotation existed, a lower spacing of the C-C distance should be observed when looking along the [1 1 0] direction (zigzag axis) of graphene, which is not the case here. For example, a rotation disorder of 30° would correspond to 1.232 Å spacing.

The MLG regions with intermediate thickness also demonstrate AB stacking order with no rotation of graphene sheets in the stack [inset of Fig. 2(b)]. In this case, however, an increased interlayer distance of either 3.43 ± 0.02 Å or 3.56 ± 0.02 Å is predominantly observed. The inset of Fig. 2(c) presents an atomically resolved cross-sectional TEM image of the MLG region with 20+ layers, which constitute of a mix of AA, AB, and ABC stacked layers with no rotation between graphene sheets in the stack. However, local bending and edge dislocations (white arrows) are observed. The average interlayer distance for the 20+ layers thick region is measured to be 3.63 ± 0.02 Å.

The TEM results show that MLG maintains AB stacking without rotational disorder. Only very thick and highly disordered regions (20+ layers), corresponding to a very small percentage of the total sample coverage (∼2%), contain stacking faults. Consequently, rotational disorder cannot be the reason for the decoupling of the layers in our MLG.

Our observation that the layers in the MLG stack are not rotated with respect to each other is in agreement with previous results from low-energy electron diffraction on similar MLG samples grown by high-temperature sublimation.2,12,13,26 On the other hand, it was shown that out-of-plane rotational disorder exists in MLG grown at lower temperatures.2,8,27,28 These findings can be understood considering the nature of the sublimation process. The higher temperature
used in our growth process enable enhanced carbon diffusion and surface restructuring, which may promote the formation of ordered graphene layers observed here.

Another important finding is the increase in interlayer spacing in the thicker MLG regions. An interlayer spacing that is 2.4%–6.3% larger as compared to HOPG is observed for the regions with intermediate thickness, which represents 94% of the MLG sample. Even a larger increase of 8.4% on average is found for the MLG regions with 20+ layers. It was previously shown via first principles calculations and quantum Monte Carlo simulations that the interaction energy between individual layers in AB stacked graphite and MLG decreases as a function of the interlayer spacing, obeying a power law with $D^{-3.29}$ and $D^{-4.2}$, respectively.

For the region with the thinnest graphene (7 layers), the STEM results reveal interlayer spacing typical for HOPG (3.35 Å), which contains AB stacked and coupled graphene layers. However, thin MLG regions ($\leq 7$ layers) correspond to a small fraction of the total surface coverage (4%). Inter-LL levels associated with coupled AB stacked MLG are not observed in the MIR-OHE data (see Fig. S1). It is therefore suggested that inter-LL transitions of these small AB stacked inclusions (at most 4%) could not be detected in the particular MIR-OHE experiment due to their minute contributions. Note that the MIR-OHE has previously been demonstrated to detect AB coupled graphene layers.

To gain further structural and chemical information, EELS and EDX mapping was performed at different sample locations. Figure 3(a) shows a representative HAADF STEM image of a MLG region with an intermediate thickness ($\sim$94% of the sample) containing 9 layers. Figure 3(b) presents the EELS spectra of C-K from the same region starting from SiC at the 0 nm indication and ending at the last graphene layer (10 nm). It is clear that the C-K shows a transition from $sp^3$ type (peak centered at $\sim 291.7$ eV) at the 4 nm indication to $sp^2$ type bonding (onsets at $\sim 284$ eV and $\sim 294$ eV) at the 7 nm indication, which are characteristic of 1s-$\pi^*$ and 1s-$\sigma^*$ transitions of C. At the interface (6 nm), a mixed bonding

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**Figure 3**

(a) STEM image showing 9 graphene layers on C-face 4H-SiC. (b) EELS spectra of C-K edges ($\sim 284$ eV) obtained at indicated distances in (a). (c) The corresponding EDX spectra showing the Si-K peak ($\sim 1.8$ keV). (d) Atomic concentrations of C, Si, and O as a function of distance, starting from SiC (blue, 0–5.5 nm) to the interface layer (orange, 5.5–7 nm) and across the graphene (green, 7–15 nm), obtained by EELS quantification, respectively.
environment is observed with a reduced intensity in the $sp^3$ edge (~291.7 eV) and a small shoulder appearing at ~287.5 eV.

Figure 3(c) presents the EDX spectra showing C-K, O-K, and Si-K peaks along the cross section of the region shown in Fig. 3(a). EELS quantification of C-K, O-K, and Si-L edges was employed to estimate the elemental concentrations of C, O, and Si as a function of distance (0–15 nm), which is presented in Fig. 3(d). Notably, the Si/C ratio decreases at a constant rate near the SiC surface (starting from the 4 nm indication), evidencing Si sublimation well before the Si surface. This is also confirmed by the reduced HAADF STEM intensity [see the top four SiC layers in Fig. 3(a)]. The Si content in the interface layer gradually decreases from ~30 at. % at the interface (~6 nm indication) to ~4 at. % at the first graphene layer (~7 nm indication). However, on average, 5 at. % Si is observed in the MLG (7–15 nm indications). O-K shows a significant increase up to ~16 at. % at the onset of the interface layer (~5.5 nm), after which it decreases to ~1 at. %. The C-K content increases in the interface layer from ~22 at. % (5.5–7 nm) to ~95 at. % at MLG (7–15 nm).

We have previously reported on the formation of an amorphous layer containing a mix of C and Si at the interface between graphene and the SiC substrate. $SiO_x$ was also observed between few layer graphene (monolayer and bilayer) and the SiC substrate. It is believed that the oxidation occurs after growth, when the sample is exposed to ambient conditions, as a result of O-diffusion via defects within the graphene sheets and binds to Si in the interface layer, with a higher concentration bordering the SiC interface. The presence of the relatively high amount of O at the interface of thicker MLG observed here confirms that this phenomenon is ubiquitous for C-face graphene independent of the number of layers in the MLG stack. Such an interface layer was shown to cause a nonideal Schottky contact between graphene and SiC, which may have critical implications for device applications.

Furthermore, the EELS results show that Si is trapped in the MLG stack (~5 at. % on average). This could explain the increased interlayer spacing of MLG observed in the TEM images. Moreover, EDX confirms that thick MLG (20+ layers) contains up to ~13 at. % of Si (see Fig. S4 of the supplementary material), while STEM analysis shows a significant increase in interlayer spacing for the thick regions as compared to HOPG. Hence, based on our results, we suggest that the observed electronic decoupling and increased interlayer spacing of MLG are likely caused by Si intercalation. This is a kinetic phenomenon where the intercalated Si atoms displace the C atoms from their equilibrium positions, which may be further affected by thermodynamic factors.

Si intercalation of MLG during growth can be explained by a lateral diffusion of the sublimed Si atoms between layers and vertical diffusion through the defects in graphene sheets. Figure 4(a) depicts the TEM image of multilayer graphene in the vicinity of a large SiC step demonstrating graphene layers bonded to the step edges. These results indicate that graphene layers grow as the SiC surface step recedes via sublimation. This is in agreement with previous reports on C-face SiC step sublimation at high temperatures in argon atmospheres. Si out-diffusion through the side-channel was proposed by Borystuk et al. because of the lack of a buffer layer formation in C-face SiC sublimation. On that account, we conclude that Si side-channel diffusion enabled by high temperature sublimation in the argon atmosphere of C-face SiC allows for pristine graphene layer growth. However, at areas where graphene is bonded to the step edges, the Si side-channel diffusion is reduced as one of the lateral directions is blocked. Arguably, new graphene layers are grown in an increased Si pressure environment causing the appeared defective graphene layers caused by Si intercalation. It is worth noting that Si atoms only penetrate the graphene sheet through grain boundaries and defects. Figure 4(b) schematically illustrates the Si diffusion process via side-channels and defects.

The presence of defects will affect the diffusion of the Si atoms during the graphene growth process and thus will also have an impact on the formation of the interface layer. We note that the thicker MLG region with 20+ layers is more defective than the thinner regions, as deduced by TEM (see Fig. 2). These findings are further supported by the larger crystallite size (~1000 nm) for MLG with the number of layers below 15 as compared to thicker regions (20 layers and above) with crystallite size around 200 nm as evaluated by micro-Raman scattering spectroscopy mapping. Based on our results, we propose a defect-driven growth evolution model for MLG grown by high temperature sublimation on the C-face of SiC. Note that this model is at this point hypothetical and will require further investigations to be fully confirmed. The model is schematically represented in Fig. 5, which depicts different growth modes of MLG governed by the Si diffusion via defects. In regions where graphene with a low density of defects initially forms, the penetration of the sublimed Si atoms through the graphene layer(s) is impeded [Fig. 5(a)]. Further growth of graphene is suppressed, and the sublimation process leads to the accumulation of Si and C atoms at the interface layer. Therefore, these regions contain thinner AB stacked MLG and no Si atoms trapped between layers. The interlayer spacing of these thin MLG regions matches the respective spacing in highly oriented pyrolytic graphite where coupling between layers occurs. In areas where more defective graphene forms at the beginning of growth, the sublimed Si atoms can penetrate the graphene

![Fig. 4.](image-url)
FIG. 5. Proposed model of high temperature sublimation growth modes of multilayer graphene on C-face SiC governed by the Si atoms diffusion via defects. (a) Thin region (≤7 layers): high quality graphene region preventing the Si diffusion through the stack, resulting in thin multilayer graphene with coupled AB-stacked layers. (b) Moderate thick region (7–19 layers): multilayer graphene of moderate quality containing Si atoms propagating through the defects, where trapped Si atoms cause increased interlayer spacing and decoupling of layers while maintaining AB stacking. (c) Thick region (20+ layers): highly defected graphene region containing the highest concentration of trapped Si between the layers and the largest interlayer spacing.

In conclusion, we have investigated epitaxial MLG graphene grown by high temperature sublimation on the C-face 4H-SiC. The MIR-OHE reveals the square root dependence of the inter-LL transition energies and polarization preserving selection rules, which clearly show that our MLG consists of electronically decoupled graphene layers. TEM measurements show that MLG layers are not rotationally disordered and maintain the AB-stacking order, while interlayer spacing is increased by 2.4%–8.4% with thicker regions showing higher interlayer spacing. EELS mapping evidences Si atoms trapped in the MLG with the increased Si content for increasing interlayer spacing in MLG. Based on our combined results from the MIR-OHE, STEM, EELS, and EDX, it is proposed that trapped Si leading to increased interlayer spacing causes layer decoupling in ordered MLG. Furthermore, our observations lead us to propose a defect-driven growth evolution mechanism for MLG on C-face SiC by high temperature sublimation in the argon atmosphere. Further investigations are needed to explain the atomistic and nanoscale processes of the growth evolution.

See the supplementary material for a description of the growth environment, Mueller matrix spectra analysis, graphene stacking analysis by TEM, interface layer thickness measurements, and EDX quantification.

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