Deterministic patterned growth of high-mobility large-crystal graphene: a path towards wafer scale integration

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Abstract
We demonstrate rapid deterministic (seeded) growth of large single-crystals of graphene by chemical vapour deposition (CVD) utilising pre-patterned copper substrates with chromium nucleation sites. Arrays of graphene single-crystals as large as several hundred microns are grown with a periodicity of up to 1 mm. The graphene is transferred to target substrates using aligned and contamination-free semi-dry transfer. The high quality of the synthesised graphene is confirmed by Raman spectroscopy and transport measurements, demonstrating room-temperature carrier mobility of 21 000 cm² V⁻¹ s⁻¹ when transferred on top of hexagonal boron nitride. By tailoring the nucleation of large single-crystals according to the desired device geometry, it will be possible to produce complex device architectures based on single-crystal graphene, thus paving the way to the adoption of CVD graphene in wafer-scale fabrication.

Introduction
CVD synthesis of graphene on catalytically-active substrates has emerged as the most promising approach for large-area production of graphene [1]. The self-limiting nature of CVD growth on metals such as copper (Cu) and platinum allows synthesis of large-scale homogeneous films of monolayer graphene. However, electrical characterization of polycrystalline samples of CVD graphene reveals that the presence of grain boundaries causes significant degradation of the electric performance, compared to pristine material obtained by mechanical exfoliation of flakes [2]. As demonstrated initially by Petrone et al [3], samples fabricated using single-crystals of CVD graphene can have electrical performance comparable to that of exfoliated flakes [4]. Furthermore, recent reports have shown that by fully encapsulating CVD graphene with suitable materials such as hexagonal boron nitride (h-BN), low-temperature charge carrier mobility above 300 000 cm² V⁻¹ s⁻¹ [5] or even 3 000 000 cm² V⁻¹ s⁻¹ [6] can be achieved.

Over the last few years the synthesis of large-crystal graphene has attracted a huge scientific interest, with significant advances in the achievable crystal size [7–10]. Recent work has reported single-crystals of graphene measuring 1 cm [10] and, using copper/nickel alloy as the growth substrate, even 4 cm [11]. Inevitably, these approaches still produce randomly-distributed crystals of graphene, which limits their applicability to scaled production of graphene devices. Furthermore, the commonly-used transfer methods either allow scalability while introducing significant performance degradation, or are limited to transferring areas of several tens of µm² [5].

For many applications the size of individual graphene devices is limited to tens or hundreds of microns, easily achievable by the current methods of single-crystal synthesis, however, random spatial distribution of graphene crystals in such samples makes polycrystalline graphene preferable for wafer-scale integration. This issue could be mitigated by selectively pre-determining the nucleation sites for graphene crystals according to the target architecture, which could allow the fabrication of large and complex circuits utilising completely monocrystalline graphene. Patterned growth using polymer-based nucleation seeds was first reported by Wu et al [12], however, only high-density arrays of
10–20 µm crystals were demonstrated. Arrays of similar dimensions were recently presented by Song et al., using poly(methyl methacrylate) (PMMA) seeds to nucleate graphene on top of CVD-grown h-BN [13].

In this work we present a method to selectively pattern the Cu growth substrate using chromium (Cr) nucleation seeds, which allows deterministic nucleation of large-crystal graphene, measuring several hundred microns. The nucleation density is highly-controlled by the combined use of natively oxidised Cu foils, non-reducing annealing and sample enclosure [14], and measuring as low as 10 crystals per mm². We also demonstrate a clean semi-dry transfer procedure allowing aligned placement of graphene crystals on target substrates. Figures 1(a)–(d) show the proposed process flow for scalable fabrication of single-crystal devices. Initially, the device layout is designed (a), followed by the deposition of Cr seeds on copper substrate (b). Large crystals of graphene are grown in a deterministic manner (c) and are then transferred to the target substrate, followed by conventional sample fabrication (d). Combining deterministic growth of large graphene crystals and aligned transfer allows the fabrication of fully-tailored samples utilising high-quality single-crystal graphene. In principle, this technique is scalable to the size of full wafers, paving the way for wafer-scale integration of high-quality graphene.

Experimental

The initial step of seeded growth of large-crystal graphene is the preparation and patterning of the growth substrate. Natively-oxidised copper foils (Alfa-aesar, 99.8%, stock #13382) were used in this work, which were electropolished [14] in order to clean the surface and to improve the surface morphology. Nucleation sites were then patterned on the foil by optical lithography and thermal evaporation of 25 nm of chromium, followed by lift-off. The growth of large single-crystals was performed using a low-pressure (25 mbar) CVD growth procedure using a commercially-available Aixtron BM Pro cold-wall reactor. As described in our previous work [14], the substrates were annealed for 10 min in non-reducing argon atmosphere in order to preserve surface oxidation. Graphene was grown at a temperature of 1060 °C with a gas flow comprising 900 standard cubic centimetres per minute (sccm) of argon, 100 sccm of hydrogen (H₂) and 1 sccm of methane (CH₄).

To minimise the transfer-related contamination and to allow precise placement of graphene arrays on the target substrates, we developed a semi-dry aligned transfer technique which can be conveniently scaled up. A thin PMMA carrier membrane was spin-coated on the Cu foil with seeded graphene and the graphene was detached from the growth substrate using electrochemical delamination [15, 16]. As shown in the inset to Figure 3(a), a semi-rigid Kapton frame was used to handle a suspended PMMA membrane carrying the seeded array of graphene crystals, which was rinsed in deionised water, dried, and aligned to the target substrates using a micromechnical stage. The target substrates were mildly heated to improve the adhesion of graphene, and the PMMA was finally removed in acetone and isopropanol. More details about this transfer technique can be found in the supplementary information (stacks.iop.org/TDM/4/021004/mmedia).

To investigate the crystallinity of seeded graphene using electron diffraction the graphene arrays were transferred onto Quantifoil transmission electron microscopy (TEM) grids, containing a carbon film perforated with 2 µm holes on top of a Cu support with 50 µm square windows. The electron diffraction observations were performed in microdiffraction mode on a Zeiss Libra 120 microscope operating at an accelerating voltage of 120 keV. In this modality the beam is kept parallel by the Köhler illumination system and the diffracted area is selected through the size of the condenser aperture. In our case all the patterns were collected with a 19 µm condenser aperture which gives an illuminated area on the sample plane of 600 nm. The patterns were recorded with a TRS full-frame 16 bit 2k × 2k charge-coupled device (CCD) and by a Timepix single electron detection camera. A camera length of 180 mm was used.

Raman spectroscopy was performed to study the quality and homogeneity of the crystals which were transferred via aligned semi-dry transfer on top of exfoliated h-BN flakes (HQ Graphene). A Renishaw InVia system was used, equipped with a 532 nm laser and 100 × objective lens, providing a spot size of ~1 µm.

To investigate the charge carrier transport characteristics of seeded large-crystal graphene, Hall bar devices were fabricated on top of h-BN flakes by electron-beam lithography and reactive ion etching. The devices were studied at room temperature (RT) by measuring the electric-field effect using a 4-terminal configuration. The measurements were performed using a Stanford Research Systems SR830 lock-in amplifier, by passing a 1 nA current between the source–drain contacts of the Hall bar and measuring the voltage drop $V_{\text{dc}}$ along the side contacts as a function of back-gate voltage. Back-gate voltage was applied using a Keithley 2400 source-measurement unit, allowing us to ensure the lack of gate leakage.

Results and discussion

Panels (e) and (f) of figure 1 show a representative square array of large-crystal graphene produced using patterned nucleation and panel (g) displays the aligned transfer of this array to a pre-patterned wafer containing a marker grid. Clearly, at the graphene growth temperatures, the deposited Cr thin films form particles which act as effective nucleation sites for graphene, thus enabling growth of graphene patterns. The representative crystals displayed in panels (e)–(g) have an average diameter of 100 µm and a periodicity of 200 µm, which is an order of
magnitude larger than previous reports [12, 13]. As shown in the supplementary figure S3(a), such arrays present an extremely well-controlled nucleation over a large area, with non-seeded crystals comprising less than 10% of the total number. The high control on spurious nucleation is possible thanks to the fact that growth is carried on using oxidised Cu foils and a non-reducing atmosphere, as reported in [14]. It is relevant to mention that by simply varying the substrate patterning, it is possible to easily produce arrays with different periodicity (up to 1 mm), crystal size (up to 350 µm) and pattern (e.g. hexagonal, square), as shown in the supplementary information. Due to the short annealing and fast growth rate, the total synthesis time of arrays containing 350 µm crystals is notably short—i.e. around 2 h including the heating and cooling of the CVD system.

As demonstrated in figure 2, the ratio of hydrogen to methane is found to be an important parameter for regulating the growth rate of graphene as well as controlling the presence of chromium after the growth. During preliminary experiments we found that although lower H2:CH4 ratio (20:1) provided fast growth rates (~15 µm min⁻¹), the synthesised samples contained parasitic particles of Cr at the centre of each crystal (inset to figure 2(a), particle indicated by an arrow). Increasing this ratio to 100:1 caused a slight reduction to the growth rate (~12 µm min⁻¹). However, the

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**Figure 1.** (a)–(d) Proposed process flow for deterministic growth of single-crystal graphene. (e) Scanning electron microscopy (SEM) image of an array of graphene single-crystals. (f) Optical image of a graphene array on oxidised Cu foil. (g) Optical image of single-crystal array transferred on Si/SiO2 substrate with alignment markers. In figures (e)–(g) the array periodicity is 200 µm and crystal size is ~100 µm.
higher hydrogen flow had an advantageous effect of removing the chromium particles. We also note that higher hydrogen partial pressures have previously been found to improve the quality of the graphene lattice thanks to selective etching of the weak C–C bonds [17]. The patterned nucleation evident in these samples (figure 2(b)) clearly indicates that during the initial stages of the growth, Cr particles were present on the surface, successfully seeding the graphene crystals, and were then gradually removed during the growth process. The lack of chromium on the surface of the foil was confirmed using high-magnification SEM imaging (inset to figure 2(b)) as well as energy-dispersive x-ray spectroscopy. Figure 2(c) shows an EDX spectrum obtained at an accelerating voltage of 10 kV at the centre of a seeded graphene crystal grown using a high H₂:CH₄ ratio. The spectrum is clearly dominated by the L- and K-lines of Cu substrate. Additionally, the K-line of carbon can be seen at 0.28 keV. No evidence of L-line of Cr (0.57 keV) or K-line of Cr (5.41 keV) was observed.

As can be seen in figure 2, multi-layer islands were typically observed at the centre of seeded crystals, which is a phenomenon less commonly observed in non-seeded large-crystal graphene [14]. This could be explained by the fact that even after the initial nucleation of the seeded crystals, Cr particles remain as prominent surface features on the substrate. Hence, carbon radicals formed on the catalytic Cu surface are subjected to competing processes of edge-attachment to the monolayer crystals and the formation of ad-layers around the Cr seed before it is gradually removed. Graphene ad-layers grown on Cu substrates are generally thought to form at the Cu-graphene

Figure 2. Comparison of SEM images of graphene arrays synthesised using low (a) and high (b) ratio of hydrogen to methane. Insets show magnified SEM images obtained from the centers of typical crystals, as indicated by the dashed lines. (c) Energy-dispersive x-ray (EDX) spectrum obtained from the center of a crystal synthesised using high flow of hydrogen.
interface, i.e. under the initial monolayer and not on top of it [18, 19]. The multi-layer regions were typically observed to be either in alignment with the main crystal orientation (A-B stacking), or rotated by 30°. This is discussed in more detail in the supplementary information and was observed in single-crystal multi-layer graphene by other groups [17, 20]. While multi-layer regions are present, it should be noted that the large size of the seeded crystals can easily allow placement of critical device areas on the monolayer areas. Furthermore, the formation of multi-layers could potentially be suppressed by careful tuning of growth parameters.

To confirm the monocrystalline nature of seeded graphene, electron microdiffraction analysis was performed on graphene arrays transferred on top of TEM grids (figure 3). Diffraction patterns were obtained from 9 points located both at the edges and in the interior of a large seeded crystal, as indicated in figure 3(a). The diffraction patterns, displayed in panels 1–9 of figure 3(b), showed negligible misalignment, confirming that the seeded graphene is indeed monocrystalline. A sequence of patterns collected while moving the beam across one crystal and joined together in a movie shows a steady hexagonal pattern confirming the same result (see supplementary information).
Furthermore, we obtained additional diffraction patterns from neighbouring crystals to study the rotational alignment of different crystals within an array (figure 3(b), panels 10–12). As is already evident from the SEM images of seeded arrays (see supplementary figure S5), electron diffraction analysis confirmed rotational alignment between separate crystals grown on the same Cu grain (the small observed rotation of ~1 degree could be attributable to wrinkling of the PMMA support film during the transfer procedure and small deformation of the TEM grids). We note that rotational alignment of graphene single-crystals was also observed using low-energy electron diffraction (LEED) analysis in our previous work [14]. According to several recent publications [21, 22], seamless stitching of such aligned crystals could form large-area monocrystalline graphene, although rigid and highly-crystalline substrates such as Cu(1 1 1) films deposited on semiconductor wafers could provide a more suitable growth platform for such material.

It is well-known that the major limiting factor for the quality of graphene samples is the substrate [23, 24] as well as the contamination and damage caused during the transfer procedure [25]. To mitigate the negative effects of low-quality substrates and to focus on the intrinsic quality of the synthesised graphene, we used aligned semi-dry transfer to place the graphene on top of exfoliated flakes of h-BN, which is known as the optimal substrate for graphene-based devices [26]. Figure 4(a) shows a seeded crystal transferred on top of an h-BN flake with a thickness of 30 nm. With increasing scientific effort and recent progress in CVD synthesis of wafer-scale h-BN [27], this material has high potential to become widely-adopted for large-scale graphene.

Figure 4(b) shows a typical room-temperature Raman spectrum obtained from the seeded graphene transferred on top of an h-BN flake. The spectrum has 3 prominent features: the characteristic h-BN peak at 1364 cm\(^{-1}\), along with the two peaks commonly observed in high-quality graphene, the G-peak at 1582 cm\(^{-1}\) and the 2D peak at 2687 cm\(^{-1}\). No disorder-related D peak at 1350 cm\(^{-1}\) was observed, indicating an extremely low density of lattice defects [28]. The D-peak was also negligible at the centre of the crystals, further confirming the lack of negative effects from the nucleation seeds (Raman map presented in supplementary information). The intensity ratio of 2D- and G-peaks is in excess of 10 to 1. In addition, a remarkably low width of the 2D-peak \(\Gamma_{2D}\) was measured at 21 cm\(^{-1}\). The width of this peak is a reliable indicator of the sample quality, as it is highly sensitive to strain variation which is a known source of charge scattering in high-quality graphene [29, 30]. Therefore, the variation of \(\Gamma_{2D}\) over large areas of the sample was investigated via spatially-resolved Raman mapping. Figure 4(c) shows a map of the \(\Gamma_{2D}\) obtained over an area of 17 \(\times\) 20 \(\mu m^2\), whereas figure 4(d) shows the histogram of \(\Gamma_{2D}\) values obtained in the area mapped, with the majority of \(\Gamma_{2D}\) values obtained on h-BN being within 20–23 cm\(^{-1}\). This indicates the high structural quality of graphene with small strain variations [29, 30]. Lower values of \(\Gamma_{2D}\) can typically be observed only in graphene samples with full (double-sided) h-BN encapsulation [5].

To determine the carrier mobility in seeded graphene, Hall bar devices were fabricated on top of exfoliated h-BN flakes, as shown in figure 5(a). RT Electric field effect was measured in a 4-terminal
configuration, by passing a current of 1 nA across the sample and measuring the voltage drop between side contacts. Figure 5(b) reports the measured longitudinal resistance of the sample as a function of applied gate voltage (bottom axis) and gate-induced carrier density (top axis). A sharp and narrow resistivity peak was measured at 1.8 V, indicating a low residual carrier density in the sample, around $1.4 \times 10^{11}$ cm$^{-2}$. Only negligible hysteresis was observed when sweeping the gate voltage in both directions. Carrier mobility $\mu$ was estimated from room-temperature field effect measurement by fitting the data with the equation reported by Kim et al [31]. Due to the electron–hole asymmetry observed in the field effect measurement, separate fits were performed on each side of the Dirac peak, yielding hole mobility of around 21 000 cm$^2$ V$^{-1}$ s$^{-1}$, whereas electron mobility was found to be about 13 500 cm$^2$ V$^{-1}$ s$^{-1}$. These values are comparable to the RT mobility reported on high-quality non-encapsulated graphene [3, 11, 26]. Further details about the mobility estimation are given in the supplementary information.

**Conclusion**

To summarise, we have presented a method for fast and highly-controllable seeded growth of large single-crystal graphene, which can be deterministically transferred to target substrates. The periodicity and crystal size of the arrays can be flexibly varied with maximum values peaking at 1 mm and 350 $\mu$m, respectively. Both the synthetic method and the transfer approach presented in this work are scalable and lead to high quality graphene as confirmed by spectroscopic and transport measurements. Indeed, when transferred on top of h-BN, seeded graphene crystals present an ultra-low width of the Raman 2D peak $\Gamma_{2D} = 20–23$ cm$^{-1}$ and RT hole mobility above 21 000 cm$^2$ V$^{-1}$ s$^{-1}$. The quality of seeded single-crystal graphene is comparable to that of the best published results obtained on CVD graphene and exfoliated flakes. Taking into account the possibility of tailored synthesis of this material according to the target device architecture and the possibility for contamination-free
and aligned transfer, this work provides a significant advance for adoption of CVD graphene in wafer-scale fabrication.

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