Low Partial Pressure Chemical Vapor Deposition of Graphene on Copper

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***Abstract*—A systematic study of the Cu-catalyzed chemical vapor deposition of graphene under extremely low partial pressure is carried out. A carbon precursor supply of just P_{CH_4} ∼0.009 mbar during the deposition favors the formation of large-area uniform monolayer graphene verified by Raman spectra. A diluted HNO_3 solution is used to remove Cu before transferring graphene onto SiO_2/Si substrates or carbon grids. The graphene can be made suspended over a ∼12 μm distance, indicating its good mechanical properties. Electron transport measurements show the graphene sheet resistance of ∼0.6 k$\Omega/\square$ at zero gate voltage. The mobilities of electrons and holes are ∼1800 cm^2/Vs at 4.2 K and ∼1200 cm^2/Vs at room temperature.**

***Index Terms*—Chemical vapor deposition, graphene, low partial pressure, nanoelectronics, wet transfer.**

I. INTRODUCTION

GRAPHENE is an atomically thin network of carbon atoms arranged in a hexagonal lattice; it is being intensively studied because of its fascinating electrical and optical properties [1]. Graphene electronic band structure can be engineered by making nanoribbons of various widths [2]. By exploiting the ambipolar transport properties of graphene, full-wave rectification and frequency doubling can be realized at high frequency [3]. Graphene has a high charge-carrier mobility that is promising for making ultrafast transistors. Graphene is believed to play an important role in future nanoelectronics [4].

Mechanical exfoliation of graphite is the traditional technique to prepare graphene [5]. However, the method is not scalable and the resulting graphene flakes are small. Nowadays, there are two main strategies for fabricating high-quality graphene in large scale. The first is the high-temperature decomposition of silicon carbide where Si is evaporated leaving behind free carbon atoms that subsequently form graphene layers at the surface [6]. However, this method is expensive and is not suitable for graphene transfer onto other substrates. On the other hand, catalytic chemical vapor deposition (CVD) of graphene on the surfaces of several transition metals [7], [8] is a scalable technique which is compatible with the existing semiconductor-industry processing. Recently, 30-inch graphene films for making transparent electrodes have been realized by CVD [9], making the technique very promising for future applications.

Ni [7] or Cu [8] are typical catalysts used in CVD of graphene. However, the growth mechanisms associated with each catalyst are different. In the former case, carbon dissolves in Ni at high temperature and then precipitates forming graphene before and during the cool down. The method is sensitive to process timing that is not optimal for getting a monolayer of graphene in every sample. In contrast, the solubility of C in Cu is small; the formation of graphene largely results from the surface chemistry limiting the number of graphene layers to a minimum. The Cu-catalyzed graphene CVD is, however, subject to surface kinetics [10] that allows for multilayer graphene as well, depending on the processing parameters [10]–[13].

In this paper, we provide a summary of our recipe of large-area graphene produced on Cu by CVD. One of the unique features of our experiments is that graphene is grown under one to four orders of magnitude lower partial pressure of hydrocarbon than reported earlier in the literature. The low carbon concentration 1) lowers the deposition rate that 2) decreases the density of nucleation centers [14], [15] promoting large graphene domains, and 3) suppresses formation of multilayer graphene. Although carbon does not have high chemical affinity to Cu [16], our results suggest that it is possible to achieve high-quality graphene using even extremely small amount of carbon atoms in the precursor gas. This work can help a better understanding of the CVD of graphene, important for its future large-scale production.

II. MATERIAL GROWTH

The graphene is produced in a cold-wall low-pressure CVD system specially designed for carbon nanomaterial deposition

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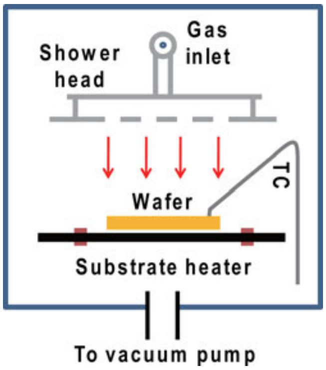


Fig. 1. Schematic of the cold-wall CVD system used in this study. The specimen is placed on a current-heated graphite holder situated at about 10 cm below the gas inlet shower head.

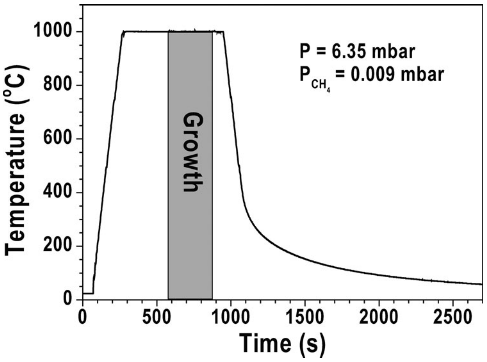


Fig. 2. Temperature profile of a typical low partial pressure graphene deposition process. Very fast (300 °C/min) heating and cooling ramp rate is used. The carbon precursor is evacuated immediately after the growth, preventing any possible deposition during chamber cooling.

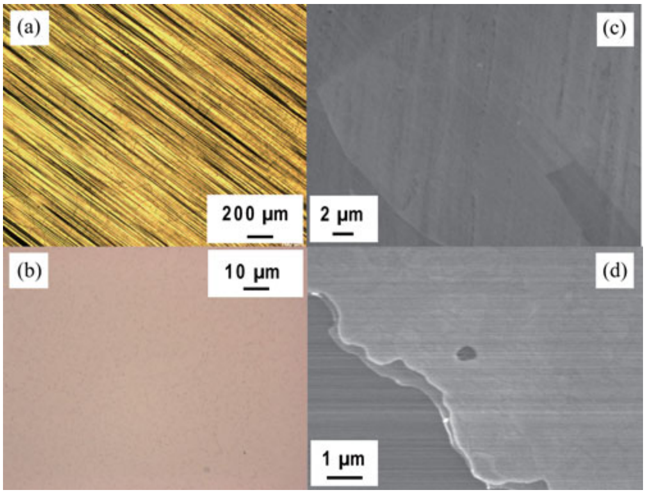


Fig. 3. Optical and SEM images showing the morphology of the Cu foils (a) and (c) and Cu thin films (b) and (d) after the graphene CVD. The sample shown in (d) is grown by low partial pressure CVD with C_2H_2 precursor to enhance the observed graphene overgrowth.

(Black Magic, AIXTRON Nanoinstruments Ltd.), as illustrated in Fig. 1. The samples are directly put onto a low-mass Joule heater which is capable of rapid thermal ramp rates. The temperature is measured by a thermocouple (TC) contacting the sample. A quartz shower head is used to premix and introduce the gas mixture uniformly across the substrate surface. The catalysts used in this study are 25-μm-thick Cu foils (99.9%, GoodFellow) and 300 nm Cu thin films evaporated on thermally oxidized silicon wafers (400 nm SiO_2). The Cu thin film thickness is crucial for the high-temperature graphene growth. If the film is too thin (e.g., 100 nm), Cu partially evaporates/sublimates at high temperature leading to a holey structure [17]. On the other hand, too thick a film (e.g., 2 μm) accumulates large strain due to the variation in thermal expansion coefficients between the Cu and the substrate, resulting in film delamination. 200–400 nm is the optimal thickness in our experiments.

The main precursor gas is a high purity methane (99.9995%) prediluted with Ar to 5%. While methane generally gives the best graphene quality, other hydrocarbons such as acetylene or ethylene can be used as well. The advantage of CH_4 is that it has only one C atom per molecule and a high decomposition temperature [18]. The samples are first cleaned in acetic acid, acetone, and isopropanol to remove the native oxides and organic contaminations. Then, they are heated to 1000 °C at 300 °C/min and annealed for 5 min, under a flow of 20 sccm H_2 and 1000 sccm Ar. Subsequently, 30 sccm prediluted CH_4 is introduced into the chamber to initiate graphene growth. The partial pressure P_{CH_4} is 9×10^{-3} mbar at the total pressure P of 6.35 mbar (Fig. 2). Our P_{CH_4} value is one to four orders of magnitude lower than used elsewhere [19]–[21], in spite of some groups reporting that the graphene growth under such an extreme condition is impossible [22]. This partial pressure corresponds to $\sim$150 graphene layers grown per second assuming the 100% efficiency of CH_4 decomposition and C sticking to the surface. After 5 min of deposition, the three gasses are stopped and the system is evacuated to $<$0.1 mbar. Finally, the samples are cooled at 300 °C/min to room temperature under 20 sccm H_2 and 1000 sccm Ar.

Fig. 3 shows optical images of the Cu foil (a) and thin film (b), respectively. No apparent changes in the morphology are observed after the graphene growth except that the grain boundaries of the poly-crystalline Cu become visible. The grains in the Cu foil are up to several 100 μm large to be compared to much smaller grains in the Cu thin film (a few μm). The bigger grain size can account for the improved quality of the graphene produced on the Cu foil than on the Cu thin film. Fig. 3(c) and (d) is scanning electron microscopy (SEM) images of the Cu foil- and thin-film samples, respectively. The grains are more clearly visible, but no graphene wrinkles or islands are observed [8], indicating continuous graphene sheets conformally attached to the copper surfaces. The Cu thin film has a weak adhesion to the SiO_2/Si substrate and can therefore be detached by simply using a sticky tape. Fig. 3(d) is taken at the wafer edge, where some parts of the Cu thin film are intentionally peeled off prior to the graphene growth. Interestingly, overgrowth of the graphene can be directly observed. The graphene is sticking out several hundred nm from the Cu thin film edge. This implies that graphene can smoothly grow over Cu grain boundaries to form a continuous film. By properly designing the geometry of the holes or trenches, suspended graphene for microelectromechanical system (MEMS) applications should be possible to realize directly on prepatterned Cu thin films.

III. FILM TRANSFER

The as-prepared graphene can be transferred to other substrates by a wet-etching technique. We use spin-coated copolymer MMA(8.5)MAA (EL10) to mechanically support graphene after copper is etched away. Graphene grows also at the bottom side of the Cu foil because the gases easily penetrate the small gap between the heater and the foil. This graphene layer is removed by oxygen plasma while the top-side graphene is protected by the copolymer. For the Cu thin film samples on SiO_2/Si, the plasma etching is not needed, but it is necessary to scratch the sample edges to remove the resist to ease the separation of the copolymer from the substrate following the Cu etching. $FeCl_3 \cdot 6H_2O$ or $Fe(NO_3)_3 \cdot 9H_2O$ can be used as etchants. However, we find that some impurities (possibly Fe) in commercial Cu foils cannot be completely etched leaving contaminating residues at the graphene surface. Nitric acid etches almost all metals with few exceptions like Au and Ta. Therefore, we use HNO_3 to etch Cu to obtain clean graphene sheets without extra cation contaminations. We dilute available 70% HNO_3 by H_2O (1:7) to minimize possible oxidation of graphene. The etching time is typically several hours. The graphene/copolymer samples are then rinsed in water and aligned to the target substrates. As the copolymer is only a few hundred nm thick, adhesive tapes are attached to the resist edge before etching for easy handling. After the transfer, the samples are dried at ambient temperature. Post-baking (160 $^{\circ}$C) is performed to remove residual H_2O. Finally, the copolymer is removed either by hot acetone or by baking samples at 450 $^{\circ}$C in H_2 and Ar during 10 min.

Fig. 4(a) and (b) shows optical micrographs of the graphene transferred from the Cu foil and thin film, respectively, onto 300-nm-thick SiO_2 thermally grown on Si wafers; this thickness gives maximum optical contrast. Bi-layer or multilayer graphene flakes (as in [10]–[13]) are not observed, suggesting that the graphene is highly uniform over large areas. In Fig. 4(a), the (almost) vertical lines are replicas of the microstructure at the unpolished surface of the mechanically processed Cu foils [see Fig. 3(a)]. The graphene transferred from Cu thin films is smoother. Shown in Fig. 4(b) image is intentionally captured at the place with a few wrinkles in the graphene and some uncovered areas of the substrate to guide the eye and visually set off the graphene layer.

The large-area graphene can be suspended by the transfer to transmission electron microscopy (TEM) grids. Fig. 5(a) demonstrates that a $\sim\o 12$ μm hole in the a-C network within a Cu TEM grid is completely covered by graphene with no visible ruptures and other defects. This indicates excellent mechanical properties of the graphene. Some wrinkles yet present at several places allow for the direct counting of graphene layers [see Fig. 5(b)]. It is seen that the as-produced graphene is a monolayer in this case.

Further proof can be found in Fig. 6 where two Raman spectra are shown, corresponding to graphene deposited on the Cu foil or thin film, and subsequently transferred to SiO_2/Si. In both cases, the G band and 2-D band peaks at $\sim$1594 cm^{-1} and $\sim$2690 cm^{-1} are clearly observed. The G and 2-D bands are signatures of sp^2 hybridized carbon [23]. In Fig. 6, the 2-D peak intensity is much higher than the G peak intensity. The 2-D peak is symmetric

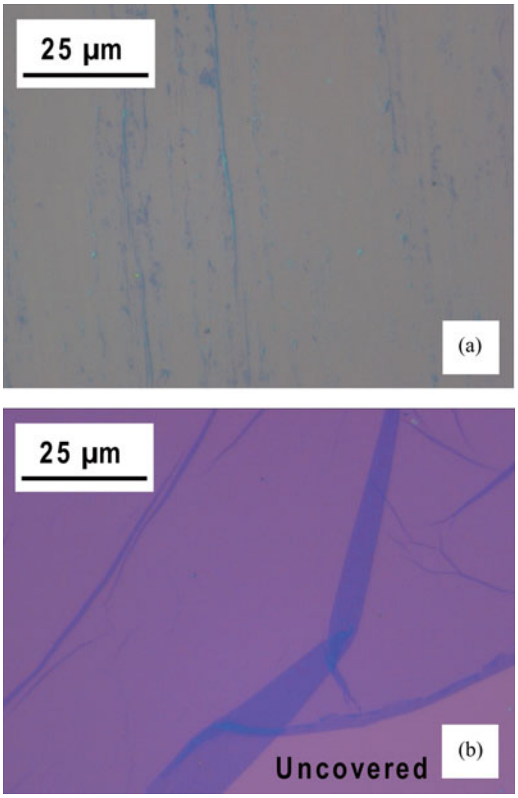


Fig. 4. Optical micrographs of the graphene produced by (a) Cu foils and (b) Cu thin films after being transferred to 300 nm SiO_2/Si.

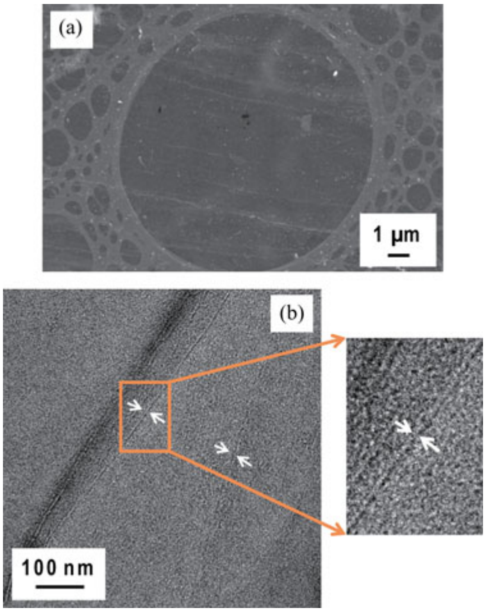
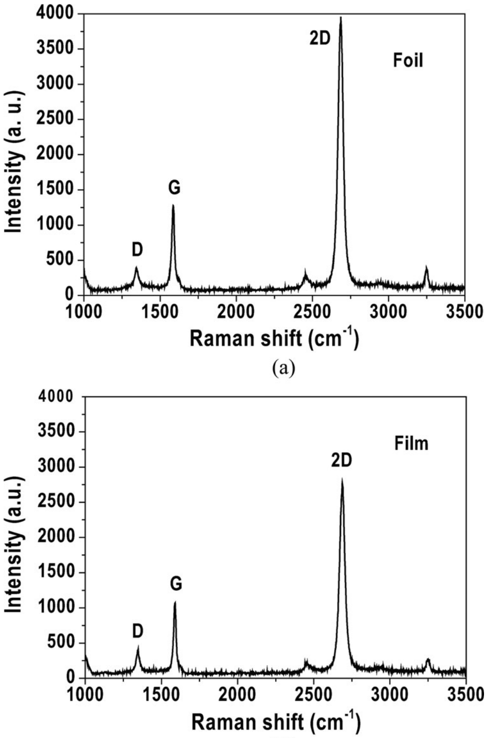


Fig. 5. (a) SEM image of the Cu-foil-grown graphene transferred to a standard TEM carbon grid. (b) Plan-view TEM image of the graphene. The enlarged micrograph indicates that the film is monolayer.



(a)

(b)

Fig. 6. Representative Raman spectra (514 nm) of the graphene grown on (a) Cu foils and (b) Cu thin films, and subsequently transferred to SiO_2/Si.

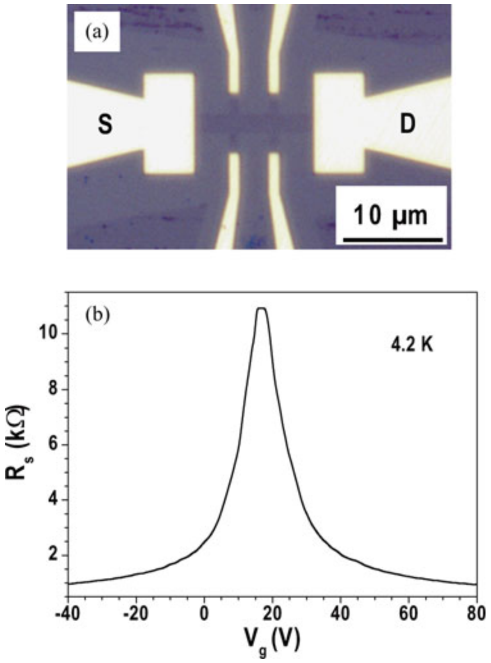


Fig. 7. (a) An optical image of a Hall device fabricated by EBL. The current-bias source and drain contacts are denoted by S and D, respectively. The other four electrodes allow for four-probe resistance measurements. (b) A typical R_s-V_g curve of the device shown in (a), measured with the four-probe configuration at liquid-helium temperature. The symmetric bell-shape of the curve suggests a nearly equal mobility of the electrons and holes.

with a full width at half maximum (FWHM) of ~37 cm^{-1} for (a) and ~42 cm^{-1} for (b), which is typical of CVD graphene [8], but larger than that of exfoliated graphene (28–30 cm^{-1}) [24]. A small D peak at ~1345 cm^{-1} is detected, implying a number of defects in the carbon lattice. The 2G band [23] and the 2450 cm^{-1} band [25] are attributed to high-order Raman signals.

IV. DEVICE CHARACTERIZATION

The Hall-bar structures are fabricated by electron beam lithography (EBL) or, in some cases, conventional photolithography. First, the Hall-bar patterns are made by EBL using ZEP 520A resist and oxygen plasma etching. Then, the electrodes connecting to the graphene structure are defined by the lift-off method using the double-layer resist MMA(8.5)MAA (EL10)/PMMA 950A2 and 3 nm Ti and 77 nm Au thin films deposited by electron beam evaporation.

Electron transport measurements are carried out in air (room temperature) and liquid helium (4.2 K) without prior sample annealing. Typically, the transport characteristics of the as-grown graphene is ohmic; the sheet resistance R_s of ~0.6 kΩ/□ (V_g = 0 V) at room temperature is suitable for applications like transparent conducting electrodes. An optical micrograph of a device made of graphene transferred from Cu foil is displayed in Fig. 7(a) with S and D denoting the source and drain electrodes, respectively. There are four more (potential) electrodes in this device allowing for Hall-effect- and four-probe resistance measurements. The active area of this device enclosed between the four probes is 2×4 $\mu\mathrm{m}^2$ (two squares). Fig. 7(b) shows the electrical-field effect, $R_s(V_g)$, in the graphene device [Fig. 7(a)] measured at 4.2 K. The gate voltage V_g is applied to the doped Si substrate relative to the graphene structure—a back-gate configuration. Calculated from the slope of the symmetric bell-shaped $R_s(V_g)$ curve, the mobilities for electrons and holes are both ~1800 cm^2/Vs while decreasing to $\mu_e \approx \mu_h \approx$ 1200 cm^2/Vs at room temperature. The former value is confirmed by the Hall-effect measurements (data not shown) at 4.2 K. The Dirac point of graphene is at ~16.5 V, implying a minor charge doping. On the other hand, devices made by photolithography generally show a pronounced p-doping, presumably due to the residues of Shipley S1813 resist at the surface of graphene [26]. Finally, we note that some of our devices show carrier mobilities up to ~2700 cm^2/Vs at room temperature, calculated by a two-terminal device model [27]. Nevertheless, both the transport measurements and the Raman spectra suggest that further improvements, such as a more accurate temperature control during the CVD and a more reproducible transfer process, are necessary to enhance the quality of graphene to a level which is comparable to the mechanically exfoliated graphene.

V. CONCLUSION

In summary, we have carried out a systematic study of very low partial pressure CVD of graphene using Cu foils or thin films as catalysts. As-synthesized large-area monolayer graphene is uniform, with no bilayer or multilayer parts detected. We use diluted HNO_3 to etch copper and metal impurities, giving a cleaner graphene surface compared to the case of ferric saline as etchant. Adequate mechanical, optical, and electrical properties of the graphene are revealed by SEM, TEM, Raman spectra, and transport measurements. It is hoped that the results presented here will aid in understanding of the Cu-catalyzed graphene CVD process under these extreme process conditions.

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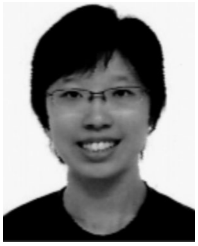

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