

# Wafer-scale epitaxial graphene growth on the Si-face of hexagonal SiC (0001) for high frequency transistors

Christos Dimitrakopoulos,<sup>a)</sup> Yu-Ming Lin, Alfred Grill, Damon B. Farmer, Marcus Freitag, Yanning Sun, Shu-Jen Han, Zhihong Chen, Keith A. Jenkins, Yu Zhu, Zihong Liu, Timothy J. McArdle, John A. Ott, Robert Wisnieff, and Phaedon Avouris  
*IBM T. J. Watson Research Center, Yorktown Heights, New York 10598*

(Received 14 May 2010; accepted 26 July 2010; published 7 September 2010)

Up to two layers of epitaxial graphene have been grown on the Si-face of 2 in. SiC wafers exhibiting room-temperature Hall mobilities up to  $2750 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ , measured from ungated, large,  $160 \times 200 \mu\text{m}^2$  Hall bars, and up to  $4000 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ , from top-gated, small,  $1 \times 1.5 \mu\text{m}^2$  Hall bars. The growth process involved a combination of a cleaning step of the SiC in a Si-containing gas, followed by an annealing step in argon for epitaxial graphene formation. The structure and morphology of this graphene has been characterized using atomic force microscopy, high resolution transmission electron microscopy, and Raman spectroscopy. Furthermore, top-gated radio frequency field-effect transistors (rf-FETs) with a peak cutoff frequency  $f_T$  of 100 GHz for a gate length of 240 nm were fabricated using epitaxial graphene grown on the Si-face of SiC that exhibited Hall mobilities up to  $1450 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$  from ungated Hall bars and  $1575 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$  from top-gated ones. This is by far the highest cutoff frequency measured from any kind of graphene. © 2010 American Vacuum Society. [DOI: 10.1116/1.3480961]

## I. INTRODUCTION

Graphene is an atomically thin layer comprising carbon atoms arranged in a two-dimensional (2D) hexagonal lattice. It is a 2D semiconductor that exhibits a linear relationship between electronic energy and 2D momentum, and the carriers in it are modeled as relativistic Dirac fermions.<sup>1,2</sup> Graphene exhibits remarkable transport properties<sup>3-5</sup> including carrier mobilities on the order of  $200\,000 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$  at cryogenic temperatures in suspended graphene.<sup>6</sup> As a result, it is a good candidate for future high frequency field-effect transistor (FET) applications. Although a macroscopic, 2D graphene sheet is generally a zero-band-gap semiconductor, it has potential to be used as the active channel in FETs for analog, rf applications such as low-noise amplifiers.

The 2D nature of graphene facilitates the fabrication of planar graphene devices and integrated circuits using standard semiconductor industry processes, a major advantage over one-dimensional structures such as carbon nanotubes. Graphene has been produced by exfoliation of graphite,<sup>7</sup> epitaxially on SiC by high temperature decomposition of the latter,<sup>8</sup> or on metals.<sup>9-11</sup>

Although high quality graphene flakes can be obtained by repetitive exfoliation of graphite, this is not a process that can be used in large scale fabrication, mainly due to the fact that no suitable method has been demonstrated yet that enables the formation of structurally coherent graphene over wafer-scale areas, or even the formation of large arrays of small-size graphene flakes arranged with a unique and predictable azimuthal orientation on a substrate. Growing graphene by subtractive epitaxy on semi-insulating SiC substrates offers the advantage of structural coherence over large

areas, because the azimuthal orientation of graphene is governed, to a large degree, by the crystal structure of the substrate surface. This is especially true in the case of the Si-face surface of SiC.<sup>12,13</sup>

In this paper, we report the growth of one to two layers of epitaxial graphene on the Si-face of 2 in. SiC wafers, its structure and morphology, and its room-temperature Hall mobility measured on large ungated Hall bars and small gated Hall bars. Furthermore, we provide for the first time a detailed account on the graphene growth process and characterization, device fabrication, and device characteristics for the recently reported record-breaking radio frequency field-effect transistors (rf-FETs) comprising such epitaxial graphene on SiC and exhibiting record cutoff frequency  $f_T$  up to 100 GHz for a gate length of 240 nm.<sup>14</sup>

## II. EXPERIMENT

We grew graphene on the Si-face of either high purity semi-insulating (HPSI) 4H(0001) 2 in. SiC wafers or semi-insulating 6H(0001) 2 in. SiC wafers. The wafers of both polytypes had a chemical mechanical polished epitaxy-ready surface on their Si polar face.

Graphene was grown in an UHV chamber (base pressure of  $\sim 3 \times 10^{-10}$  Torr) equipped with a custom-designed, inductively heated graphite susceptor hot zone that can accommodate 2 in. wafers. The shape, dimensions, shielding, and materials of the hot zone were optimized to produce uniform heating of 2 in. wafers up to the required process temperatures. The temperature was measured with a two-color pyrometer, which is connected in a feedback loop comprising a programmable temperature controller and a 12 kW rf power supply.

For the samples that we used in the demonstration of rf-FET devices, we grew graphene on 4H(0001) SiC wafers by

<sup>a)</sup>Electronic mail: dimitrak@us.ibm.com

combining for the first time a cleaning step under a Si-containing gas followed by annealing/graphenization at a higher temperature in argon. The SiC wafer was cleaned mainly from oxide contamination by annealing at 810 °C under disilane flow (20% disilane in He). Chemical vapor cleaning processes involving Si-containing gas molecules have been shown in the past to effectively remove oxides from the surface of SiC,<sup>15,16</sup> mainly by converting SiO<sub>2</sub> to SiO, the latter being a more volatile oxide. After the cleaning step, the SiC wafer was annealed at 1450 °C for 2 min under Ar flow at a pressure of  $3.5 \times 10^{-4}$  Torr and then was allowed to cool down in Ar.

The sample that showed the highest gated Hall mobility in Table III was grown on the Si-face of a semi-insulating 6H(0001) SiC wafer. The process sequence used for this sample comprised an extra annealing step (compared to the original process described above) in 20% disilane in He at  $T=1140$  °C at pressure  $P=8 \times 10^{-7}$  Torr, i.e., under conditions that favor the  $\sqrt{3} \times \sqrt{3}$  surface reconstruction of SiC.<sup>16</sup> After the cleaning step at 810 °C and the 1140 °C anneal (both under disilane flow), the SiC wafer was annealed at 1450 °C for 10 min under Ar flow at a pressure of  $3.5 \times 10^{-4}$  Torr and then was allowed to cool down in Ar.

Ungated Hall bar devices ( $200 \times 160 \mu\text{m}^2$ ) were fabricated by depositing Ti/Pd/Au contacts on top of blanket graphene using optical lithography and lift-off, followed by graphene patterning to the appropriate Hall bar shape using another optical lithography step, oxygen reactive ion etching, and wet stripping of the resist. For the smaller Hall devices ( $1.5 \times 1.0 \mu\text{m}^2$ ) e-beam lithography was used instead of optical lithography.

Atomic layer deposition (ALD) was used for the deposition of the high- $k$  gate dielectric used in the fabrication of top-gated graphene Hall bars and rf-FETs. In order to enable uniform oxide coverage, a 10-nm-thick layer of polyhydroxystyrene-based polymer was spun first on the graphene surface to act as a nucleation layer, followed by ALD of a 10-nm-thick layer of HfO<sub>2</sub>.<sup>17</sup> Finally, a Pd/Au (20 nm/40 nm) top-gate electrode was deposited and patterned by lift-off.

The samples were characterized by micro-Raman spectroscopy, tapping mode atomic force microscopy (AFM), and high resolution transmission electron microscopy (HRTEM). Micro-Raman spectroscopy was performed at room temperature with a Triax 322 spectrometer (Horiba Jobin Yvon) at 514.5 nm, using a 1200 line, 500 nm blaze grating. The light was sent through a 100 $\times$  objective with 0.8 numerical aperture to form a  $\sim 500$  nm focal spot in the middle of the Hall bars. Measurements were performed with submilliwatt incident power to avoid heating. A SiC background, recorded on the same wafer in an area where the graphene had been removed, was subtracted from the raw spectrum, leaving behind only peaks due to the graphene overgrowth.

Transmission electron microscopy (TEM) samples were prepared using a dual-beam focused ion beam *in situ* lift-out technique using 30 keV Ga ions. Prior to ion milling, a sacrificial film was deposited to preserve the ion damage from

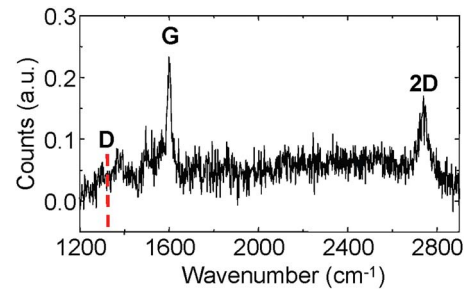


FIG. 1. (Color online) Raman spectrum from a HPSI 4H(0001) SiC wafer after graphene formation and patterning. The spectrum was taken in the middle of a graphene Hall bar. The SiC background, recorded on the same wafer in an area where the graphene had been removed, was subtracted. The dashed line indicates the expected position of the D peak.

the focused ion beam. The samples were cleaned by lower energy (a few keV) Ga ions. Bright-field HRTEM images of the interface between graphene and SiC were taken using a JEOL-3000F TEM with an accelerating voltage of 300 kV.

### III. RESULTS AND DISCUSSION

Figure 1 shows the Raman spectrum of a graphene on 4H(0001) SiC sample prepared as described above, after subtraction of the SiC background. The main features observed in this spectrum are the peaks labeled G and 2D. We also label the expected position of the D peak (at about  $1325 \text{ cm}^{-1}$ , marked with a dashed line), which is attributed to defects in graphene (e.g., edges or disordered regions).<sup>18</sup> This band is forbidden in defect-free graphene and graphite, and its absence is an indication of high quality graphene and minimal contribution from domain edges. The G peak centered at  $1600 \text{ cm}^{-1}$  is a first order band due to the doubly degenerate zone center  $E_{2g}$  optical phonon.<sup>19</sup> The 2D-phonon band at  $2739 \text{ cm}^{-1}$  is a second order peak due to two zone boundary optical phonons. The relative intensities of the G and 2D peaks and the weak absorption of the SiC Raman signal (not shown here) due to the graphene overlayer indicates one to two layer graphene.

Figure 2(a) shows a tapping mode AFM height image taken *ex situ* from this sample. The surface morphology is uniform over the whole wafer. The wafer surface is vicinal but pits appear on the terraces. Nevertheless, long continuous strips of unpitted material remain on each of the terraces. Figure 2(b) shows the corresponding AFM phase image. AFM phase image contrast has been shown to be created between the  $\sqrt{3} \times \sqrt{3}$  surface reconstruction of SiC and the buffer layer (the  $6\sqrt{3} \times 6\sqrt{3} \cdot R30$  C-rich reconstruction of hexagonal SiC) as well as between the buffer layer and graphene.<sup>16</sup> However, we have obtained evidence that AFM phase image contrast is also created when different phases exist immediately below the topmost graphene layer (e.g., graphene on buffer versus graphene on graphene) due to different rates of energy dissipation between the tip and the sample for the different layer stacks (phase contrast arises from differences in the energy dissipation between the tip and the sample<sup>20</sup>). The dark gray (brown) regions in Fig. 2(b)

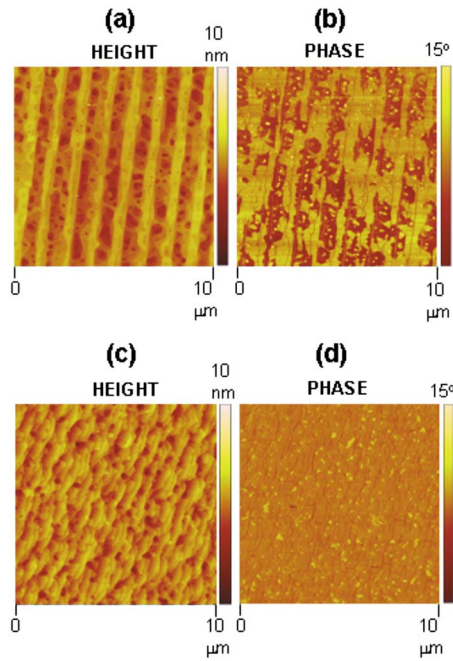


FIG. 2. (Color online) [(a) and (b)] Tapping mode AFM images from 4H(0001) SiC wafer after graphene formation using the original growth process. (a) Height image. (b) Phase image from the same area of graphene as in (a) light gray (yellow) vs dark gray (brown) regions: 1 ML graphene vs 2 ML graphene (see discussion in text). One continuous graphene layer grown conformally over steps and pits. [(c) and (d)] Tapping mode AFM images from 6H(0001) SiC wafer after graphene formation using the modified growth process. (c) Height image. (d) Phase image from the same area of graphene as in (c) light gray (yellow) vs dark gray (brown) regions are observed that as in (b) they should correspond to 1 ML graphene vs 2 ML, respectively; however, in this sample, the dark gray (brown) region covers the majority of the surface.

are mostly linked to surface pits created by SiC decomposition, thus are expected to have more graphene than the light gray (yellow) regions. If the light gray (yellow) regions corresponded to buffer layer and dark gray (brown) regions to graphene regions, there would be no electrical conduction, as the dark gray (brown) regions are discontinuous and the buffer layer is not conductive. Thus, based on the excellent sheet conductivity and mobility properties measured from this sample (see Table I below), the light gray (yellow) regions should correspond to 1 monolayer (ML) of graphene and the dark gray (brown) regions to 2 ML of graphene. Based on this, we can conclude that one continuous graphene layer has been grown conformally over steps and pits on the SiC surface of this sample, and in addition, a second, discon-

TABLE I. Room-temperature Hall measurement results from as-prepared graphene on HPSI 4H(0001) SiC (ungated Hall bars with size  $160 \times 200 \mu\text{m}^2$ ).

Device (row, column)	4,1	5,1	1,3
Sheet resistance ( $\Omega/\text{sq}$ )	1541	1624	1550
Carrier density ( $\text{cm}^{-2}$ )	$2.8 \times 10^{12}$	$3.6 \times 10^{12}$	$3.0 \times 10^{12}$
Hall mobility ( $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$ )	1450	1070	1330

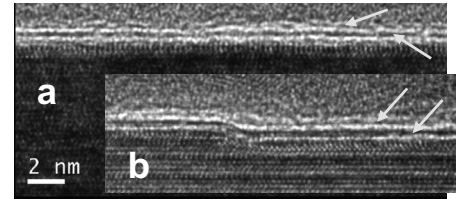


FIG. 3. HRTEM cross sections. (a) Graphene is the dark stripe between the thick bright stripes, which are attributed to the spacing between graphene and adjacent layers in this focus condition. One continuous graphene layer and possibly a second graphene layer can be observed (arrows). (b) Graphene layer conformally covering a two-bilayer step on the SiC surface.

tinuous graphene layer has been grown [dark gray (brown) regions] under the top graphene layer.

Figures 3(a) and 3(b) show HRTEM cross sections taken from the ungated Hall bar device area of the same sample. The position of the graphene layers is indicated by a dark band sandwiched between two bright bands, due to the slightly underfocused conditions used.<sup>21</sup> Up to a maximum of two layers of graphene (one plus a second possibly incomplete layer) appear to have been grown over SiC. Interestingly, the graphene layers seem to have been grown conformally over a two-bilayer step ( $\sim 0.5 \text{ nm}$  high) of the SiC vicinal surface [Fig. 3(b)]. This is a direct confirmation of previous evidence provided by scanning tunneling microscopy that graphene layers are continuous over SiC steps.<sup>8,22</sup> It also explains why the existence of step edges had a minor effect on the mobility of multilayer graphene FETs having their current flowing parallel or perpendicular to the steps.<sup>23</sup>

Standard, ungated Hall bar devices were used to measure the Hall mobility of graphene. The basic assumptions of the standard Hall measurement method (one kind of dominant charge carrier, the sheet resistance parallel to the constant current flow being independent of applied magnetic field) are valid for one to two layers of epitaxial graphene grown on the Si-face of SiC. Sheet resistance,  $R_s$ , was calculated from Hall bar devices built on as-prepared graphene using the equation

$$R_s = \frac{(V_{X_2} - V_{X_1})w}{Id}, \quad (1)$$

where  $w$  is the Hall bar channel width ( $200 \mu\text{m}$ ),  $d$  is the distance between voltage leads  $X_2$  and  $X_1$  ( $160 \mu\text{m}$ ), and  $I$  is a known current flowing between leads 1 and 2 (Fig. 4 shows a micrograph of the Hall bar device used for these measurements).

The 2D carrier density ( $n_s$ ) is derived by the equation

$$n_s = -\frac{1}{eR_H}, \quad (2)$$

where  $e$  is the electron charge,  $R_H$  is the Hall coefficient given by



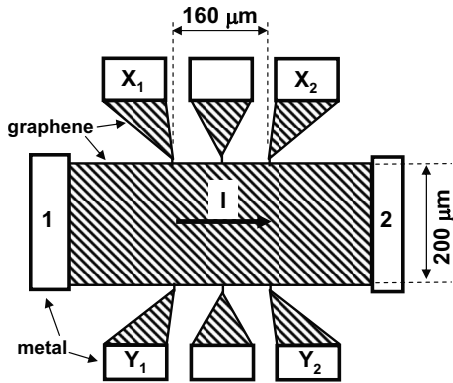


FIG. 4. Drawing of photolithographically patterned graphene Hall bar device.

$$R_H = \frac{V_{Y_1} - V_{X_1}}{IB}, \quad (3)$$

and  $B$  is the magnetic field applied perpendicular to the Hall bar plane.<sup>24</sup> The Hall mobility ( $\mu_H$ ) is calculated by

$$\mu = \frac{1}{n_s e R_s}. \quad (4)$$

Measurements from ungated Hall bar devices were performed on various devices built over one-quarter of the same 2 in. wafer used in the characterization above. Mobilities varied from 1070 to 1450  $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$  with carrier densities between  $3.6 \times 10^{12}$  and  $2.8 \times 10^{12} \text{ cm}^{-2}$ , respectively. The majority carriers were electrons as shown by the positive slope of the plot of the voltage difference between electrodes  $X_2$  and  $Y_2$  of the Hall bar (indicated by  $V_{xy}$ ) versus the magnetic field  $B$  (in Fig. 5) that corresponds to Hall bar (4,1) whose electrical properties are described in Table I. A perfectly linear plot corroborates the validity of the assumptions employed in the standard Hall mobility measurement for one to two layer graphene grown on the Si-face of SiC, which were described above.

Table I shows the properties of three such devices. It should be stressed that the size of the Hall bar devices

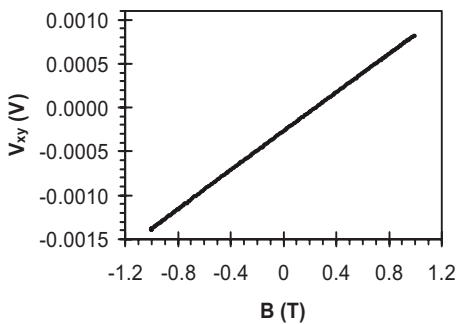


FIG. 5. Plot of the voltage difference  $V_{xy}$  between electrodes  $X_2$  and  $Y_2$  of the Hall bar vs the magnetic field  $B$ . A perfectly linear plot proves the validity of the assumptions used in the standard Hall mobility measurement method for one to two layer graphene grown on the Si-face of SiC (e.g., dominance of a single type of majority carriers, and constant  $R_s$  with varying magnetic field).

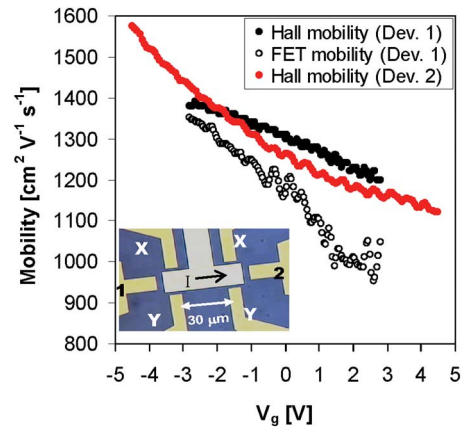


FIG. 6. (Color online) Hall and field-effect mobility vs gate voltage measured from two top-gated Hall bar devices.

( $200 \times 160 \mu\text{m}^2$ ) is large and that it contains approximately 100 or more vicinal terraces of SiC. This means that either a single graphene domain larger than these dimensions occupies the entire Hall bar area or that graphene domain boundaries, if existing, do not substantially affect the effective mobility of a multidomain graphene device, which would explain the high mobility measured.

Top-gated rf-FETs were fabricated on the same SiC/graphene wafer to study the high frequency response of epitaxial graphene transistors. Adjacent to the rf-FETs, top-gated Hall bar devices (inset of Fig. 6) were built to study the effect of gate-modulated charge carrier concentration on Hall mobility. The Hall mobility was measured at discrete gate voltage  $V_G$  values, as described above. Figure 6 shows Hall mobility versus gate voltage  $V_G$  for two devices. A maximum Hall mobility of  $1575 \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}$  was measured at  $V_G = -4.5 \text{ V}$ .

Field-effect mobility,  $\mu_{\text{eff}}$ , was measured from the top-gated Hall bar device using the relation

$$\mu_{\text{eff}} = \frac{\Delta G_s}{e \cdot \Delta n_s}, \quad (5)$$

where  $\Delta G_s$  is the change in the sheet conductance,  $\Delta n_s$  is the 2D carrier density modulation due to the change in  $V_G$ , because

$$\Delta G_s = \frac{\Delta I_{ds} \cdot L}{V_{ds} \cdot W} \quad (6)$$

and

$$\Delta n_s \cdot e = C_i \cdot \Delta V_g. \quad (7)$$

The capacitance per unit area  $C_i$  was measured to be  $1.3 \times 10^{-7} \text{ F cm}^{-2}$ . It is important to note that using the four-probe setup of the Hall bar device to measure  $G_s = 1/R_s$ , we avoided making any assumptions about the contact resistance of the FET, which would have introduced errors in the estimation of effective mobility. The distance between the voltage leads  $X_2$  and  $X_1$  of the top-gated Hall bar was  $L = 30 \mu\text{m}$ , while the graphene bar width was  $W = 4 \mu\text{m}$ .

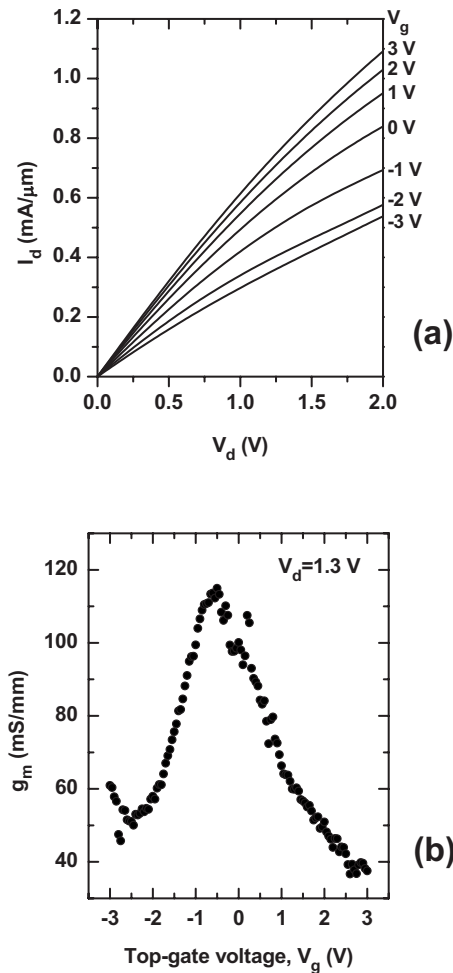


FIG. 7. (a) FET output characteristics for a device with gate length  $L_G = 550$  nm and channel width  $W = 30$   $\mu\text{m}$ . The drain current  $I_d$  is normalized to channel width. (b) Plot of transconductance  $g_m$ , normalized to channel width, as a function of top-gate voltage  $V_g$  at  $V_d = 1.3$  V for this rf-FET.

Based on the variation of the measured sheet conductance and the 2D charge density as a function of  $V_g$  and using the equations shown above, the field-effect mobility versus  $V_g$  plot was derived and is shown in Fig. 6 (device 1, open circles). Good agreement between Hall and field-effect mobilities is observed (within 10%). The highest field-effect mobility measured in this way was  $1400$   $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$ . The agreement between our Hall mobilities measured before and after the deposition of the gate dielectric stack and the field-effect mobilities is quite remarkable and unusual based on previous literature results.

In order to study the high frequency response of epitaxial graphene top-gated rf-FETs, we used the methodology described by Lin *et al.*<sup>25</sup> The high frequency performance of a transistor for small-signal response is measured by S-parameter measurements up to 30 GHz, and it is mainly determined by the transconductance  $g_m = dI_d/dV_g$ .<sup>25</sup>

Figure 7(a) shows the FET output characteristics for a device with  $L_G = 550$  nm and channel width  $W = 30$   $\mu\text{m}$ . The maximum on-current (normalized to channel width) measured from this *n*-type epitaxial graphene FET was

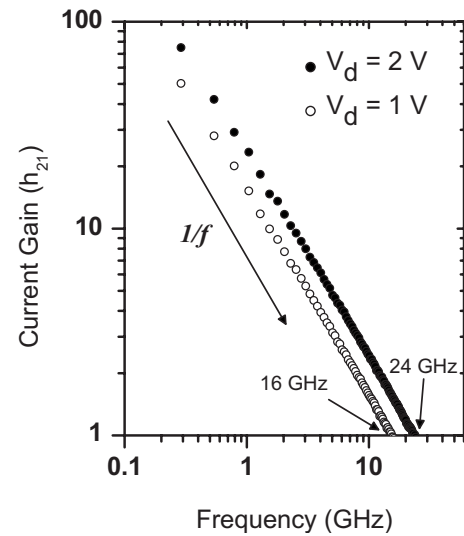


FIG. 8. De-embedded current gain  $|h_{21}|$  vs frequency from the same rf-FET as in Fig. 7 ( $L_G = 550$  nm). Cutoff frequency  $f_T$  is determined to be 16 and 24 GHz for  $V_d$  of 1.0 and 2.0 V, respectively. The current gain exhibits the  $1/f$  frequency dependence expected for a well-behaved FET.

1.1  $\text{mA}/\mu\text{m}$  at top-gate voltage  $V_g = 3$  V and  $V_d = 2$  V. Figure 7(b) shows a plot of transconductance  $g_m$ , also normalized to channel width, as a function of top-gate voltage at  $V_d = 1.3$  V for this rf-FET. The magnitude of  $g_m$  is strongly dependent on  $V_g$ , but its sign is positive throughout the  $V_g$  range used, showing that transport is dominated by electrons, in agreement with Hall measurements. For  $V_d = 1.3$  V, the peak  $g_m$  is measured to be  $115$   $\text{mS}/\text{mm}$  at  $V_g = -0.5$  V. Figure 8, which corresponds to one of the first devices fabricated and tested on this graphene wafer, shows the current gain  $h_{21}$  as a function of frequency, after de-embedding the pad parasitics using specific short and open structures. The current gain exhibits the  $1/f$  frequency dependence expected for a well-behaved FET. The cutoff frequency, defined as the frequency of unity current gain, is measured to be 16 and 24 GHz (Ref. 31) for  $V_d$  of 1.0 and 2.0 V, respectively, for the 550-nm-gate-length graphene FET. The highest  $f_T$  we measured for devices built on the same wafer and having the same gate length  $L_G = 550$  nm was 53 GHz, as shown in Fig. 9. The variation in  $f_T$  for several devices built on the same wafer and having the same gate length of 550 nm was between 20 and 53 GHz.

As expected from previous results on exfoliated graphene FETs showing that  $f_T$  is proportional to  $(1/L_G)^2$ ,<sup>25</sup> FETs built on the same epitaxial graphene on SiC wafer and having a shorter gate length ( $L_G = 240$  nm) produced higher peak cutoff frequencies as shown in Fig. 10 (some of the data in Fig. 10 were first reported in Ref. 14 recently). The maximum  $f_T$  we measured from these devices was 100 GHz.<sup>14</sup> This is the highest cutoff frequency reported from any kind of graphene-based FETs. The highest  $f_T$  value reported previously for epitaxial graphene FETs on SiC was 4.4 GHz,<sup>28</sup> while the highest  $f_T$  value reported previously for exfoliated graphene-flake-based FETs was 50 GHz.<sup>26</sup>

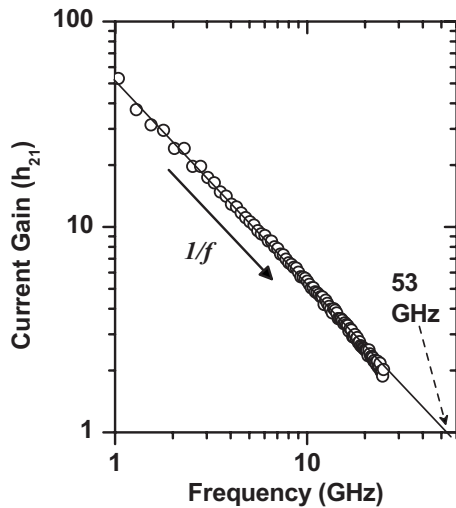


FIG. 9. De-embedded current gain  $|h_{21}|$  vs frequency for a rf-FET with  $L_G = 550$  nm, exhibiting the maximum cutoff frequency  $f_T = 53$  GHz that we measured from devices of this gate length. The current gain exhibits the  $1/f$  frequency dependence expected for a well-behaved FET.

Since the fabrication and testing of these rf devices, we have improved the Hall mobility of epitaxial graphene on the Si-face of SiC wafers by improving the process and materials combination that we used (see Sec. II). While we are still optimizing the process, Hall mobilities up to  $2750 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$  have been measured at room temperature from large, ungated  $160 \times 200 \text{ }\mu\text{m}^2$  Hall bar devices fabricated with epitaxial graphene grown on the Si-face of SiC, as shown in Table II. This epitaxial graphene was grown on the Si-face of a semi-insulating 6H(0001) SiC and the process sequence comprised an extra annealing step in 20% disilane in He at  $T = 1140 \text{ }^\circ\text{C}$ . Then, the SiC wafer was annealed at  $1450 \text{ }^\circ\text{C}$  for 10 min under Ar flow at a pressure of  $3.5 \times 10^{-4}$  Torr. The first three devices listed in Table II, which had mobilities of 1765, 1775, and  $1804 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ , were measured approximately two weeks after graphene formation and soon after the Hall bar device fabrication. The last three devices listed in Table II, with mobilities of 1580, 2640, and  $2750 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ , were measured after six months of exposure to ambient atmosphere. An interesting trend in the last three devices is that they exhibit four to five times lower carrier density than the first three devices that were measured originally. While it is expected that a lower carrier density could lead to higher carrier mobility in graphene, the cause for this difference needs to be investigated further.

Figure 2(c) shows tapping mode AFM height image from epitaxial graphene grown on the Si-face of a semi-insulating 6H(0001) substrate with the aforementioned process. There is less pitting than that shown in Fig. 2(a), corresponding to graphene on 4H(0001) SiC grown without the extra annealing step in 20% disilane in He at  $T = 1140 \text{ }^\circ\text{C}$ . The phase image in Fig. 2(d) is from the same area of graphene. Light gray (yellow) versus dark gray (brown) regions, like in Fig. 2(b), should correspond to 1 ML graphene vs 2 ML, respectively. However, in this sample, the dark gray (brown) region covers the majority of the surface. The better coverage of

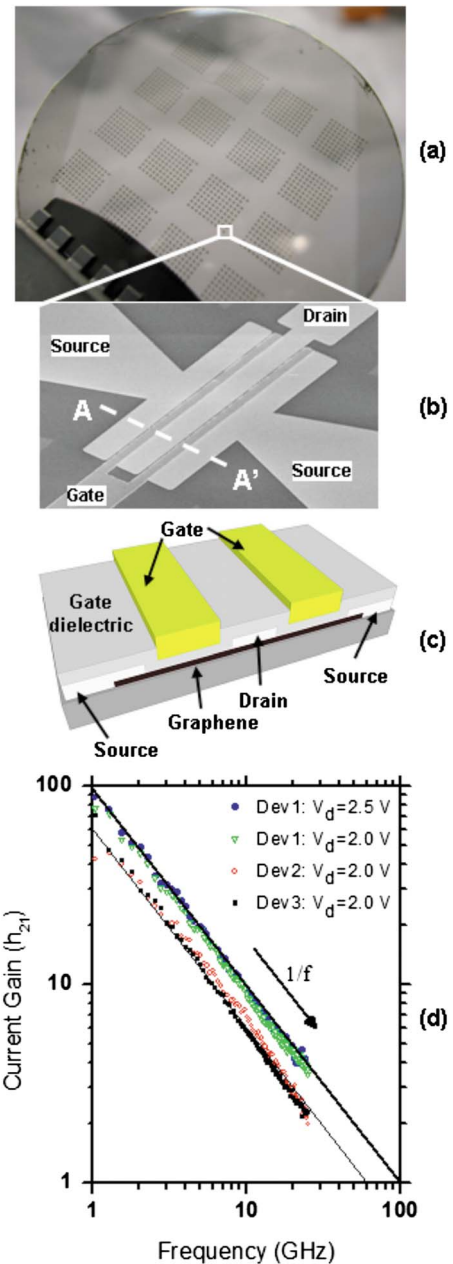


FIG. 10. (Color online) (a) Devices fabricated on epitaxial graphene on the Si-face of a 2 in. HPSI SiC 4H(0001) wafer. (b) SEM micrograph of a rf-FET device. AA' line denotes the direction of the cross-section drawing depicted in (c). (c) Three-dimensional cross-sectional slice drawing of epitaxial graphene RF-FET device. (d) De-embedded current gain  $|h_{21}|$  vs frequency for three rf-FETs with  $L_G = 240$  nm exhibiting a range of cutoff frequencies  $f_T$  between 60 and 100 GHz. All three devices were tested at a drain bias  $V_d$  of 2 V, and the device that showed the highest  $f_T$  at 2 V was also tested at  $V_d = 2.5$  V resulting in  $f_T = 100$  GHz. (Some of these data were reported in Ref. 14.) The current gain exhibits the  $1/f$  frequency dependence expected for a well-behaved FET.

two graphene layers leads to a higher Hall mobility ( $2750 \text{ cm}^2/\text{V s}$ ) on this sample than that measured from the sample shown in Figs. 2(a) and 2(b) ( $\sim 1450 \text{ cm}^2/\text{V s}$ ).

Figure 11(a) shows the Raman spectrum of graphene on SiC 6H(0001) [same sample corresponding to Table II and Figs. 2(c) and 2(d)] after SiC background subtraction. Apply-

TABLE II. Room-temperature Hall measurement results from graphene on semi-insulating 6H(0001) SiC (ungated Hall bars with size  $160 \times 200 \mu\text{m}^2$ ). Devices (4,3), (5,3), and (5,5) were measured 6 months after the first three devices.

Device (row, column)	(2,1)	(3,2)	(4,1)	(4,3)	(5,3)	(5,5)
Sheet resistance ( $\Omega/\text{sq}$ )	1451	1692	1715	5820	5950	6300
Carrier density ( $\text{cm}^{-2}$ )	$2.4 \times 10^{12}$	$2.1 \times 10^{12}$	$2.1 \times 10^{12}$	$3.7 \times 10^{11}$	$3.9 \times 10^{11}$	$6.2 \times 10^{11}$
Hall mobility ( $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$ )	1804	1765	1775	2750	2640	1580

ing the same analysis as for the spectrum in Fig. 1, we conclude that the graphene coverage is about one to two layers (but less than three) from the relative intensities of the G and 2D peaks and the weak absorption of the SiC Raman signal (not shown here) due to the graphene overlayer. We took Raman spectra in three positions from the center to the edge of the wafer and plotted the intensity of the 2D peak (characteristic of graphene) for the three positions [Fig. 11(b)]. Very good thickness uniformity from center to edge can be deduced.

To test the effect of pits on the measured Hall mobility at room temperature, we fabricated small, gated Hall bars (size  $1 \times 1.5 \mu\text{m}^2$ ) in a targeted, pit-free area of graphene on SiC (same sample corresponding to Table II). Figure 12 shows

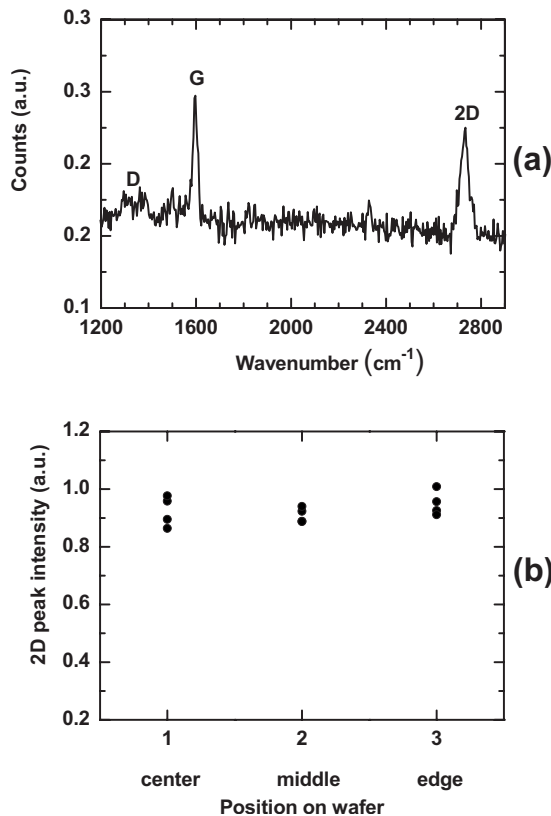


FIG. 11. (a) Raman spectrum of as-grown graphene on SiC 6H(0001) [sample corresponding to Table II and Figs. 2(c) and 2(d)] after SiC background subtraction. (b) Plot of the 2D peak intensity of the Raman spectrum of the same sample for three different positions on a quarter of a 2 in. wafer (close to the center, intermediate position between center and edge, and close to the edge). For each position, several spectra separated by about 10  $\mu\text{m}$  from each other were recorded.

the room-temperature Hall mobility measured from such a Hall bar as a function of top-gate voltage. The mobility increases as the concentration of electrons in graphene is reduced, up to a value  $\mu_{\text{Hall}}$  just above  $4000 \text{ cm}^2/\text{Vs}$  at  $V_{\text{TG}} = -7.2 \text{ V}$ , where the electron density is calculated to be just above  $1.0 \times 10^{11} \text{ cm}^{-2}$  [dark gray (red) symbols]. At more negative top-gate voltages (lower electron concentrations) and as the Dirac point is approached, ambipolar transport starts becoming important and the equations used to model the device behavior are not expected to be valid (light gray symbols). The mobility measured at an electron density of  $2.27 \times 10^{12} \text{ cm}^{-2}$  (similar to the electron density values reported in Table II from large Hall bars) is  $1750 \text{ cm}^2/\text{Vs}$ . This mobility is similar to the Hall mobility measured from the large Hall bars (Table II), the latter being an average (effective) mobility for graphene on flat and pitted areas, and hundreds of vicinal steps and terraces. This confirms that pits on the SiC surface do not have a serious effect on the Hall mobility of the graphene grown over that surface.

Table III lists the room-temperature Hall mobilities (published<sup>27-32</sup> and from this work) from graphene grown on the Si-face of hexagonal SiC. The ungated Hall mobility we report is higher than previously published results, while the gated Hall mobility reported here is by far the highest Hall mobility (gated or ungated bars) reported from graphene grown on the Si-face of SiC.

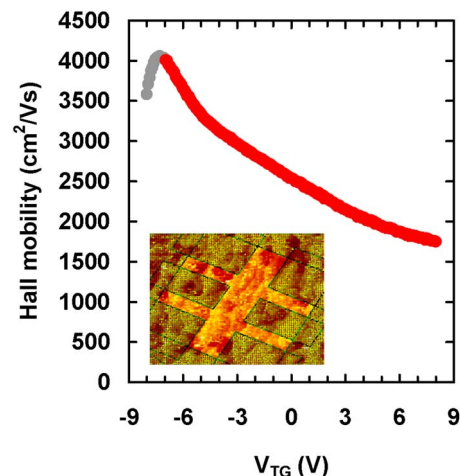


FIG. 12. (Color online) Plot of room-temperature Hall mobility vs top-gate voltage from a device built on the sample corresponding to Fig. 11.



TABLE III. Room-temperature Hall mobilities (published and from this work) from graphene grown on the Si-face of hexagonal SiC

Hall mobility ( $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$ )	Reference
2750 <sup>a</sup>	This work
4000 <sup>b,c</sup>	This work
2400	32
1640	33
1600 <sup>c</sup>	30
~1600	34
1450 <sup>a</sup>	31 and this work
1575 <sup>c</sup>	31 and this work
1150	35
1000	28
900	27
860	29

<sup>a</sup>Hall bar size  $160 \times 200 \mu\text{m}^2$ .

<sup>b</sup>Hall bar size  $1 \times 1.5 \mu\text{m}^2$ .

<sup>c</sup>From gated Hall bar.

#### IV. CONCLUSION

In conclusion, we have grown up to two layer epitaxial graphene on the Si-face of 2-in.-diameter semi-insulating 4H(0001) SiC wafers, and fabricated and tested ungated and gated Hall bar devices as well as rf-FETs using such graphene as the active channel. Room-temperature Hall mobilities up to  $2750 \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}$  were measured from ungated, large,  $160 \times 200 \mu\text{m}^2$  Hall bars, and up to  $4000 \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}$  from top-gated, small,  $1 \times 1.5 \mu\text{m}^2$  Hall bars. Record peak cutoff frequency,  $f_T$ , up to 100 GHz was measured from top-gated epitaxial graphene FETs with gate length  $L_G=240 \text{ nm}$ , which is the highest cutoff frequency reported from FETs with any kind of graphene channels. The graphene used for the latter rf-FETs had room-temperature Hall mobilities up to  $1450 \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}$ . Thus, based on the higher mobilities demonstrated by our most recent epitaxial graphene samples grown with our modified graphene growth process, there is considerable potential for achieving even higher rf-FET performance in the near future.

#### ACKNOWLEDGMENTS

This work was supported by DARPA under Contract No. FA8650-08-C-7838 (CERA program). The authors are grateful to Chun-Yung Sung for the guidance and administration of the CERA project at IBM.

- <sup>1</sup>A. H. Castro Neto, F. Guinea, N. M. R. Peres, K. S. Novoselov, and A. K. Geim, *Rev. Mod. Phys.* **81**, 109 (2009).
- <sup>2</sup>A. K. Geim and A. H. MacDonald, *Phys. Today* **60**(8), 35 (2007).
- <sup>3</sup>K. S. Novoselov, A. K. Geim, S. V. Morozov, D. Jiang, M. I. Katsnelson, I. V. Grigorieva, S. V. Dubonos, and A. A. Firsov, *Nature (London)* **438**, 197 (2005).
- <sup>4</sup>Y. Zhang, Y. W. Tan, H. L. Stormer, and P. Kim, *Nature (London)* **438**, 201 (2005).
- <sup>5</sup>C. Berger *et al.*, *Science* **312**, 1191 (2006).
- <sup>6</sup>K. I. Bolotin, K. J. Sikes, J. Hone, H. L. Stormer, and P. Kim, *Phys. Rev. Lett.* **101**, 096802 (2008).
- <sup>7</sup>K. S. Novoselov, A. K. Geim, S. V. Morozov, D. Jiang, Y. Zhang, S. V. Dubonos, I. V. Grigorieva, and A. A. Firsov, *Science* **306**, 666 (2004).
- <sup>8</sup>C. Berger *et al.*, *J. Phys. Chem. B* **108**, 19912 (2004).
- <sup>9</sup>Q. Yu, J. Lian, S. Siriponglert, H. Li, Y. P. Chen, and S.-S. Pei, *Appl. Phys. Lett.* **93**, 113103 (2008).
- <sup>10</sup>A. Reina, X. Jia, J. Ho, D. Nezich, H. Son, V. Bulovic, M. S. Dresselhaus, and J. Kong, *Nano Lett.* **9**, 30 (2009).
- <sup>11</sup>X. S. Li *et al.*, *Science* **324**, 1312 (2009).
- <sup>12</sup>A. J. Van Bommel, J. E. Crombeen, and A. Van Tooren, *Surf. Sci.* **48**, 463 (1975).
- <sup>13</sup>I. Forbeaux, J.-M. Themlin, A. Charrier, F. Thibaudau, and J.-M. Debever, *Appl. Surf. Sci.* **162–163**, 406 (2000).
- <sup>14</sup>Y.-M. Lin, C. Dimitrakopoulos, K. A. Jenkins, D. B. Farmer, H.-Y. Chiu, A. Grill, and Ph. Avouris, *Science* **327**, 662 (2010).
- <sup>15</sup>S. W. King, R. Kern, M. Benjamin, J. Barnak, R. Nemanich, and R. Davis, *J. Electrochem. Soc.* **146**, 3448 (1999).
- <sup>16</sup>J. B. Hannon and R. M. Tromp, *Phys. Rev. B* **77**, 241404(R) (2008).
- <sup>17</sup>D. B. Farmer, H.-Y. Chiu, Y.-M. Lin, K. A. Jenkins, F. Xia, and Ph. Avouris, *Nano Lett.* **9**, 4474 (2009).
- <sup>18</sup>C. Faugeras, A. Nèrrière, M. Potemski, A. Mahmood, E. Dujardin, C. Berger, and W. A. de Heer, *Appl. Phys. Lett.* **92**, 011914 (2008).
- <sup>19</sup>A. C. Ferrari *et al.*, *Phys. Rev. Lett.* **97**, 187401 (2006).
- <sup>20</sup>P. J. James, M. Antognozzi, J. Tamayo, T. J. McMaster, J. M. Newton, and M. J. Miles, *Langmuir* **17**, 349 (2001).
- <sup>21</sup>W. Norimatsu and M. Kusunoki, *Chem. Phys. Lett.* **468**, 52 (2009).
- <sup>22</sup>T. Seyller *et al.*, *Surf. Sci.* **600**, 3906 (2006).
- <sup>23</sup>J. Kedzierski, P.-L. Hsu, P. Healey, P. W. Wyatt, C. L. Keast, M. Sprinkle, C. Berger, and W. A. de Heer, *IEEE Trans. Electron Devices* **55**, 2078 (2008).
- <sup>24</sup>D. K. Schroder, *Semiconductor Material and Device Characterization* (Wiley-Interscience, New York, 1990), Chap. 5.
- <sup>25</sup>Y. M. Lin, K. A. Jenkins, A. Valdes-Garcia, J. P. Small, D. B. Farmer, and Ph. Avouris, *Nano Lett.* **9**, 422 (2009).
- <sup>26</sup>Y.-M. Lin, H.-Y. Chiu, K. A. Jenkins, D. B. Farmer, and P. Avouris, *IEEE Electron Device Lett.* **31**, 68 (2010).
- <sup>27</sup>K. V. Emtsev *et al.*, *Nature Mater.* **8**, 203 (2009).
- <sup>28</sup>J. S. Moon *et al.*, *IEEE Electron Device Lett.* **30**, 650 (2009).
- <sup>29</sup>B. L. VanMil *et al.*, *European Conference on Silicon Carbide and Related Materials*, 2008 (unpublished); *Mater. Sci. Forum* **211**, 615 (2009).
- <sup>30</sup>T. Shen, J. J. Gu, M. Xu, Y. Q. Wu, M. L. Bolen, M. A. Capano, L. W. Engel, and P. D. Ye, *Appl. Phys. Lett.* **95**, 172105 (2009).
- <sup>31</sup>C. Dimitrakopoulos *et al.*, 2009 Fall MRS Meeting, Boston, MA, 30 November–4 December 2009 (unpublished), Abstract No. L7.7.
- <sup>32</sup>A. Tzalenchuk *et al.*, *Nat. Nanotechnol.* **5**, 186 (2010).
- <sup>33</sup>S. Weingart, C. Bock, U. Kunze, K. V. Emtsev, Th. Seyller, and L. Ley, *Physica E* **42**, 687 (2010).
- <sup>34</sup>X. Li, X. Wu, M. Sprinkle, F. Ming, M. Ruan, Y. Hu, C. Berger, and W. A. de Heer, *Phys. Status Solidi A* **207**, 286 (2010).
- <sup>35</sup>J. A. Robinson *et al.*, *Nano Lett.* **9**, 2873 (2009).