Electrostatic transfer of patterned epitaxial graphene from SiC(000\textbar) to glass

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Abstract. We report on a scalable electrostatic process to transfer epitaxial graphene onto alkali-containing glass substrates. Multilayer epitaxial graphene (MEG) was grown by heating silicon carbide (000\textbar) to high temperatures (1650–1700 °C) in an argon-mediated environment. Optical lithography was used to define patterned graphene regions, typically 20 × 20 µm², which were then transferred to Pyrex substrates. For the electrostatic transfer, a large electric potential (1.2 kV) was applied between the donor MEG sample (anode) and the heated acceptor glass substrate (cathode). Atomic force microscopy scans of the transferred graphene showed that the morphology of the transferred multilayer graphene resembles that of the donor MEG. Raman spectroscopy analysis confirmed that the graphene can be transferred without inducing defects. The sheet resistance of the transferred graphene was as low as 150 Ω/□. The transfer of small (1–2 µm wide) and large (∼ 70 × 70 µm²) graphene patterns to Zerodur demonstrates the versatility of this transfer technique.
1. Introduction

Graphene has attracted a great deal of scientific interest due to its phenomenal electrical, thermal and mechanical properties, which include the ambipolar electric field effect [1], high mobility (200 000 cm$^2$/Vs$^{-1}$ for freestanding graphene [2, 3]), long spin-coherence length (2 $\mu$m [4]), high elastic modulus (1.0 TPa [5]), excellent thermal conductivity [6] and low optical absorption (2.3% per monolayer for visible light [7]). These properties suggest that graphene is an eminently suitable material for diverse applications such as frequency multipliers [8], high-speed transistors [9]–[11], spintronics-based quantum computing devices [4], magnetometers [12], gas sensors [13] and transparent conductive electrodes [14].

Developing methods for transferring epitaxial graphene to arbitrary substrates is of great interest to help realize the above graphene-based applications. Graphene grown via chemical vapor deposition (CVD) on Ni [10, 15] or Cu [16, 17] has attracted wide interest in this regard, as these CVD-grown graphenes can be readily transferred to an arbitrary acceptor substrate. Epitaxial graphene on SiC offers the following advantages over CVD-grown graphene on Cu: higher mobilities, larger domain sizes than CVD-grown graphene on Cu [18, 19] and absence of contamination of the remaining substrate elements, which could interfere with electron transport. Published methods for transferring epitaxial graphene include mechanical exfoliation using adhesive or thermal release tape [20, 21] and peeling the graphene layers using a gold/polyimide film [22]. The adhesive tape technique transferred small, random areas of epitaxial graphene with minimal damage [20]. The gold/polyimide film technique transferred epitaxial graphene regions up to square millimeters, but induced severe damage to the epitaxial graphene, including the introduction of holes, tears and folds [22], as well as likely residual gold contamination as gold diffused into graphene [23].

Electrostatic transfer, an anodic bonding-derived technique, offers an alternative method for transferring epitaxial graphene. Random graphite flakes have been electrostatically deposited from highly orientated pyrolytic graphite (HOPG) onto Pyrex and silicon wafers [24]–[26]. A large potential (>1 kV) was applied between the HOPG anode and the insulating wafer cathode, driving many graphite flakes to the insulating substrate. The location and thickness...
Figure 1. A schematic diagram, not to scale, of the electrostatic transfer method shows the patterned epitaxial graphene pressed against the acceptor glass substrate. A flat tungsten carbide (WC) plate is used as an anode to ensure uniform voltage and pressure. The glass substrate rests on a grounded heated plate (not shown).

of the transferred graphite flakes were uncontrolled due to the random nature of the starting HOPG [24]–[26].

Similar to the traditional anodic bonding of metals and semiconductors to glass, electrostatic transfer relies on the presence of mobile alkali cations in the glass to induce the transfer of graphene. In anodic bonding, the large applied potential between the metal/semiconductor anode and the glass cathode drives the cations away from the anode/cathode interface, setting up a ∼1 µm thick region that is depleted of cations [27]. This negatively charged depletion region sets up a large electric field that pulls the surfaces into intimate contact and, in the case of anodic bonding, allows covalent bonds to bridge the interface. Optimum anodic bonding occurs for flat materials, free of any surface debris, with a minimal mismatch in the coefficient of thermal expansion (CTE) [27, 28]. A key distinction between electrostatic deposition of graphite and traditional anodic bonding is that the graphite is transferred to the glass, not covalently bonded [24].

The electric field necessary for transferring graphene can be estimated by considering the graphene and glass as parallel conductor plates. The pressure, $P$, between such plates is $P = \frac{1}{2} \epsilon_0 \epsilon_r V^2/d^2$, where $\epsilon_0$ is the permittivity of free space, $\epsilon_r$ is the dielectric constant of glass, $V$ is the applied voltage and $d$ is the distance between the plates. As the minimum stress to exfoliate a graphene monolayer from bulk graphite is $P = 0.4$ MPa [26], the magnitude of the electric field, $|E|$, needed is given by

$$|E| = \sqrt{\frac{2P}{\epsilon_0 \epsilon_r}}. \quad (1)$$

Assuming $\epsilon_r = 4.6$ for Pyrex at room temperature, the minimum $|E|$ is 140 MV m$^{-1}$. Calculations of the electric field magnitude during anodic bonding are problematic since the width of and charge density within the depletion region are not well known. Wallis and Pomerantz [29] experimentally estimated that the electric field at a metal/Pyrex interface is 300 MV m$^{-1}$ for a 800 V applied voltage, sufficient for exfoliating a graphene monolayer.

In what follows, we describe an electrostatic technique for transferring patterned multilayer epitaxial graphene (MEG) to insulating glass substrates, as indicated in figure 1. The acceptor substrates used were Pyrex, which contains Na$^+$ ions, and Zerodur, which contains Li$^+$, Na$^+$
and K\textsuperscript{+} ions and is a low CTE glass ceramic. Leveraging the potential to control the thickness and location of the transferred film via the use of MEG, thousands of pre-patterned graphene squares are reliably and reproducibly transferred in parallel from the donor SiC to the acceptor glass substrate. Electrostatic transfer, in turn, may offer a scalable process that could enable wafer-level production of graphene devices on substrates other than SiC.

2. Experimental considerations

2.1. Epitaxial graphene synthesis and lithography

MEG was grown on n-type 6H-SiC(000\textbar) (C-face, 0.069 Ω cm, Cree Inc.) using an Ar-mediated growth process \cite{30, 31}. Prior to graphene growth, the surface was hydrogen etched (45\% H\textsubscript{2} and 55\% Ar mixture) at 1425 °C for 30 min to remove the polishing damage. Graphene was grown in a high-temperature furnace operated at atmospheric pressure. The sample temperature was raised to 1200 °C in a 10\% H\textsubscript{2} and 90\% Ar mixture and then to the growth temperature, 1650–1700 °C, in an Ar atmosphere \cite{31, 32}. As frequently observed for C-face epitaxial graphene, the as-grown graphene samples were defined by smooth graphene domains bounded by ridges with height 5–80 nm and width 100–750 nm \cite{33}. An atomic force microscope (AFM) scan of a typical 20 × 20 \textmu m\textsuperscript{2} region is shown in figure 2(a). The graphene drapes continuously over the underlying SiC terraces, highlighted by dashed lines, which are a few microns wide and 5–10 nm high.

Optical lithography was then used to define patterned graphene regions, typically pillars 50 nm tall and 20 × 20 \textmu m\textsuperscript{2} across, of epitaxial graphene on top of the SiC substrate (see figure 1). An SF\textsubscript{6} reactive ion etch was used to etch both the epitaxial graphene and the underlying SiC. For the graphene that was transferred to Zerodur, a set of larger squares, 70 × 70 \textmu m\textsuperscript{2} across and 50 nm high, were defined in the same manner. Post-lithography Raman analysis showed that the graphene was undamaged, except at the perimeter of the graphene regions, and that the photoresist was removed.

Throughout the growth and transfer process, AFM and scanning Raman spectroscopy have been used to characterize the epitaxial graphene, namely the structural morphology and the presence of structural defects. Tapping-mode AFM scans were obtained using both a Nanotec Cervantes AFM and a DI 3100 AFM with MikroMasch NSC 15 cantilevers. AFM scans were analyzed using Nanotec WSxM software \cite{34}. Raman maps and spectra were obtained using a WITec alpha 300 R confocal Raman microscope with a 532 nm laser.

2.2. Electrostatic transfer of graphene

Electrostatic transfer of graphene to various alkali-containing glass substrates—a Pyrex microscope slide, a Pyrex coverslip and a Zerodur optical flat—was carried out using a custom anodic bonder\textsuperscript{1}. Prior to transfer, the glass substrate is cleaned by soaking in a 3 : 1 mixture of H\textsubscript{2}SO\textsubscript{4} and H\textsubscript{2}O\textsubscript{2} (piranha) to remove organics, followed by rinsing in DI water and drying with compressed air. Due to the fragile nature of the Pyrex coverslip, it was not wet cleaned prior to transfer.

\textsuperscript{1} The Pyrex microscope slide, R220110, and coverslip, R612012, were obtained from Esco Products. The Zerodur optical flat, NT43-331-000, was from Edmund Optics.
Figure 2. AFM and optical images of multilayer graphene transferred to Pyrex. In (a), an AFM scan shows the epitaxial graphene prior to lithography and transfer. In (b), an optical micrograph shows hundreds of $20 \times 20 \mu m^2$ squares of transferred graphene on a Pyrex coverslip. (c) An AFM scan of a transferred graphene square on Pyrex. Arrows point to ridges in the transferred graphene. (d) An AFM scan of a strip of transferred graphene surrounded by embossed Pyrex. The arrow points to the tip of the transferred graphene strip. (e, f) Phase and topography (f) AFM scans of a strip of graphene from which the thickness of the transferred graphene was measured, as shown by the profile in (g). Images (a, d–g) are of sample C; images (b) and (c) are of sample A.

To transfer the epitaxial graphene, the patterned MEG and glass substrate are pressed together with a force of 2–10 N to ensure contact, as indicated in figure 1. The top plate anode is a tungsten carbide die mount tool; the bottom cathode is a heated conductive plate (not shown). The sample stack is heated to 300 °C under pressure. When the set point temperature is reached, the potential voltage is gradually increased to the transfer voltage, 1.2 kV. At the transfer voltage, a rapidly decaying current flows. This voltage is maintained until the measured current drops from $\sim 0.3$ mA to below 0.02 mA, a time of 5–15 min. Subsequently, the sample stack is cooled to room temperature, and the samples are separated. Table 1 lists the graphene samples transferred to glass; samples A, C and D will be discussed in more detail in section 3.
Table 1. Epitaxial graphene samples transferred to Pyrex and Zerodur.

| Sample | Growth temperature (°C) | Anneal time (min) | Acceptor substrate | Pattern size (µm × µm) | Transfer efficiency (%) |
|--------|-------------------------|-------------------|--------------------|------------------------|-------------------------|
| A      | 1700                    | 5                 | Pyrex coverslip    | 20 × 20                | 63 ± 7\(^a\)           |
| B      | 1650                    | 30                | Pyrex slide        | 20 × 20                | 57 ± 3\(^a\)           |
| C      | 1650                    | 10                | Pyrex slide        | 20 × 20                | 43 ± 2\(^a\)           |
| D      | 1650                    | 10                | Zerodur            | 70 × 70                | 93 ± 5\(^b\)           |

\(^a\) Where 100% efficiency corresponds to graphene squares covering 25% of the acceptor substrate over a 0.37 mm\(^2\) region.

\(^b\) Where 100% efficiency corresponds to a 70 × 70 µm\(^2\) region entirely covered by graphene.

3. Transfer results

3.1. Optical microscopy and atomic force microscope characterization

Large areas (∼10 mm\(^2\)) of patterned MEG can be electrostatically transferred. An optical micrograph of a ∼1.0 × 0.5 mm\(^2\) area of transferred graphene squares (sample A) on Pyrex is shown in figure 2(b). To improve optical contrast, this sample was mounted on a Si wafer with a 300 nm SiO\(_2\) top oxide. The brightness of the transferred graphene varies both within individual graphene squares and from square to square. This variation in optical intensity is attributed to the variation in thickness of the transferred graphene [35]; optical images of patterned MEG samples prior to transfer show a similar contrast.

In the case of perfect transfer, graphene would cover 25% of the acceptor glass substrate. In the optical image shown in figure 2(b), 12 ± 2% of the Pyrex is covered with graphene, a transfer efficiency of ∼50%. Since the contrast between the thinnest squares of transferred graphene and the Pyrex coverslip is poor, the calculated efficiency may be an underestimate of the amount of graphene transferred\(^2\). Table 1 lists the transfer efficiencies for all samples.

An AFM scan of a transferred graphene square on a Pyrex cover slip is shown in figure 2(c). Optical microscopy and phase contrast AFM scans (not shown) confirmed that the entire graphene square was transferred to the Pyrex. Within the graphene square, roughly parallel features a few microns wide and remnants of the ridge network, indicated by the two arrows, are observed. These parallel features are due to embossing of the Pyrex by the SiC steps underlying the graphene and are indicated by dashed blue lines in figure 2(a), (c) and (d). The topography of the transferred graphene resembles that of epitaxial graphene; however, in the transferred graphene, the ridge height (5–20 nm) and density are less than in the donor MEG, such as shown in figure 2(c).

The thickness of the transferred graphene is difficult to measure since the electrostatic transfer process embosses the glass by ∼5–10 nm, preventing accurate profile measurements at the graphene/glass interface. However, in some cases within an embossed square region, only a portion of the graphene is transferred, allowing AFM height measurements of the transferred graphene. Figure 2(d) shows an AFM topography image of a strip of transferred graphene.

\(^2\) The transfer efficiency is calculated using the ‘flood’ algorithm in WSxM scanning probe microscopy software [34]. This algorithm calculates the percentage of the image with an intensity range corresponding to graphene. The error is calculated by adjusting the threshold value.
Figure 3. Reflection optical micrograph (a), Raman maps (b, c) and average spectra (d) of two squares of multilayer graphene transferred to Pyrex. The correlation with the optical image and the intensity of the 2D mode (b) indicate the consistency of the transfer. The transfer process does not significantly damage the graphene as less than 5% of the graphene, predominately near the perimeter, displays the disorder-induced D-peak (c, d). Note that all spectra are normalized to the G-peak intensity. In the Raman maps (b) and (c), yellow corresponds to the highest intensity of the analyzed spectral features.

(indicated by the blue arrow), which drapes over an embossed step edge feature, 3.5 nm high. The two bright features ~ 0.5 μm long on the sides of the graphene strip are vertical folds of graphene, similar to those occasionally observed on exfoliated graphite. A slight phase contrast between the graphene strip and the embossed Pyrex region is seen in figure 2(e). Histogram measurements of the graphene strip in figure 2(f), and the profile in figure 2(g), show that the height of the transferred graphene is 5.9 ± 0.2 nm, corresponding to ~ 18 layers of graphene.

3.2. Raman analysis

To confirm the transfer of epitaxial graphene to the Pyrex substrate and to evaluate their quality, Raman images of the samples were acquired before and after electrostatic transfer. Images were collected from a series of spectra spaced at 360 nm across regions of the sample spanning at least 1125 μm². By examining the intensity, peak position and FWHM of graphene’s D, G and 2D (G’) Raman active modes at ~ 1350 cm⁻¹, ~ 1580 cm⁻¹ and ~ 2700 cm⁻¹, respectively, the presence and uniformity of the transferred graphene were investigated [36, 37].

An optical micrograph and a Raman image of the 2D mode intensity, figures 3(a) and (b), respectively, confirm that MEG was consistently transferred to a Pyrex slide (sample C). Brighter regions correspond to thicker graphene in figure 3(a) and to a greater 2D mode intensity in figure 3(b). Additionally, the electrostatic transfer does not significantly damage the graphene,
as the D peak is observed only within \(\sim 1 \mu m\) of the perimeter of the transferred graphene, as shown in figure 3(c). Raman maps (not shown) of the epitaxial graphene pillars on SiC prior to transfer also exhibit an enhanced presence of D peaks along the perimeter of the patterned graphene, indicating that the lithography process contributes to the D peak intensity observed in figure 3(c). The representative spectra shown in figure 3(d), obtained by averaging the spectra acquired from the hundreds of acquisitions across the transferred graphene, further indicate that only the graphene is transferred, as no spectral features from the SiC are observed.

The transferred graphene is modified only marginally from its ‘as-grown’ state as the G-peak position is downshifted by \(4 \text{ cm}^{-1}\), compared to the original epitaxial graphene. The observed change is less than that for previous comparisons between exfoliated graphene on glass and epitaxial graphene on SiC(0001), in which a downshift of \(\sim 11 \text{ cm}^{-1}\) for the exfoliated graphene G-mode was reported [38]. In this instance, Wang et al attributed the higher-energy G-peak position of epitaxial graphene to greater strain in the graphene layer. As the transferred graphene samples are thick (e.g. \(\sim 18\) ML flake in figure 2(f)), substrate charges are screened and thus will not affect the G-peak position of the three samples [39, 40]. Therefore, the downshift is induced by the application of a small tensile strain that arises due to the transfer process.

3.3. Transfer of graphene to Zerodur

To demonstrate the versatility of electrostatic transfer, patterned MEG (sample D) was transferred to a Zerodur optical flat. This patterned MEG included four \(70 \times 70 \mu m^2\) squares of graphene and graphene letters reading ‘ALIGN1’. Zerodur, a low-expansion glass ceramic, was chosen since it has a lower coefficient of thermal expansion than Pyrex. Figure 4(a), an optical micrograph, shows that transferred graphene covers areas as large as \(50 \times 50 \mu m^2\). The variation in thickness of the transferred graphene, as well as the observed optical contrast, is attributed to the nonuniform thickness of the donor MEG. Within each of the four \(70 \times 70 \mu m^2\) squares shown in figure 4(a), the area of Zerodur covered by transferred graphene was 88–98%.

Average Raman spectra (figure 4(b)) and Raman maps were acquired from a portion of the transferred graphene, highlighted in the \(40 \times 40 \mu m^2\) optical micrograph in figure 4(c). The nearly uniform Raman map of the G-peak position, shown in figure 4(d), indicates that the strain minimally varies throughout the transferred graphene. The transfer to Zerodur, however, is found to be a more invasive process than that to Pyrex, as large areas of the transferred graphene exhibit a nonnegligible D-peak response (figures 4(b) and (e)). Using the relative intensities of the D and G peaks to quantify the disorder, a ratio of \(I(D)/I(G) = 0.19\) was measured for the graphene transferred to Zerodur, whereas for those transferred to Pyrex the ratio was predominately close to 0. A ratio of 0.19 for the graphene on Zerodur, meanwhile, is an order of magnitude smaller than that for graphene transferred via a gold/polyimide film \((I(D)/I(G) \sim 1.5\), extrapolated from figure 2(b) of [22], but is larger than \(I(D)/I(G) < 0.05\) for the majority of the adhesive-tape-transferred graphene flakes characterized by Lee et al [20].

Investigation of the spectra from the original and transferred graphene indicates that graphene transferred to Zerodur has a larger downshift, \(6 \text{ cm}^{-1}\), in the G-peak position than graphene transferred onto Pyrex (\(4 \text{ cm}^{-1}\)). Both values are comparable to reported

\[3\] Specifically, the as-grown epitaxial graphene has a G-peak position of \(1581 \text{ cm}^{-1}\), the graphene on Pyrex has \(1577 \text{ cm}^{-1}\) and the graphene on Zerodur has \(1575 \text{ cm}^{-1}\).
Figure 4. Reflection optical micrographs, Raman maps and average spectra of MEG transferred to Zerodur. In (a), an optical micrograph shows that both large regions, $70 \times 70 \mu m^2$ across, and detailed features, ‘ALIGN1’, (shown in the top of the image) of graphene can be transferred. In (b), the average Raman spectra acquired from a $40 \times 40 \mu m^2$ region, shown in the optical micrograph in (c), is shown. A map of the G-peak position (c) confirms that large regions of graphene were transferred. The transferred graphene film is damaged, as shown by the image acquired from the D-peak position (e) and the D-peak in the average spectra (b). An optical micrograph (f) and a Raman G-peak intensity map (g) demonstrate transfer of graphene features 1–2 $\mu m$ wide. Images (f) and (g) have been flipped vertically with respect to image (a) in order to highlight the letters of ‘ALIGN1’.

downshifts of 8 cm$^{-1}$ for the G-peak position of Si-face epitaxial graphene transferred to SiO$_2$ using adhesive tape [20]. The greater alteration in the graphene transferred onto Zerodur is likely to arise from increased strain that develops in conjunction with the more damaging transfer [41, 42].

In addition, graphene features 1–2 $\mu m$ wide can also be electrostatically transferred, as demonstrated by the transfer of ‘ALIGN1’, as shown in figure 4(f). Despite the intricate pattern, only portions of a few letters (specifically, parts of ‘A’, ‘I’ and ‘N’) failed to transfer fully, as seen in the Raman map of the G-peak intensity shown in figure 4(g).
3.4. Sheet resistance

The sheet resistance, $R_s$, of graphene transferred onto Pyrex (sample A) and Zerodur (sample D) was measured using a linear micro four-probe. This four-probe consisted of equidistant cantilevers, 3-µm wide, with a 5 µm pitch. As the glass under the graphene is nonconductive at room temperature and the thickness of the graphene is much less than the probe pitch, $R_s$ is given by

$$R_s = \frac{\pi V}{\ln 2 I},$$

where $I$ is the current sourced through the outer two probes and $V$ is the voltage drop measured between the inner two probes [43]. Equation (2) neglects the correction factors due to the nonconducting glass boundary at the perimeter of the graphene square; these correction factors are of the order of one [43]. The average $R_s$ of four transferred graphene squares on Pyrex was 320 Ω/□, where the individual $R_s$ values were 150, 180, 250 and 705 Ω/□; for graphene on Zerodur, the average $R_s$ was 250 Ω/□, where the individual values were 150, 260 and 330 Ω/□. The high variance in $R_s$ is attributed to the variation in thickness of the transferred graphene films.

Direct comparisons of sheet resistance are problematic since the thickness of graphene varies. For thermal-tape-transferred C-face epitaxial graphene on SiO$_2$, Caldwell et al reported $R_s = 175$ Ω/□ [21]. For comparison, using the same four-probe procedure, we measured $R_s = 3.3 \pm 1.1$ Ω/□ for HOPG (SPI-1 grade).

4. Conclusions

We have demonstrated the possibility of electrostatically transferring patterned MEG from SiC(0001) to Pyrex and Zerodur substrates. Optical microscopy shows that the percentage of graphene transferred to the glass varied from 63% for hundreds of 20 × 20 µm$^2$ squares transferred to Pyrex to 98% graphene coverage for a single 70 × 70 µm$^2$ region. As this transfer technique requires the donor graphene and acceptor glass substrates to be in close contact, the inherent roughness in the MEG may limit the transfer efficiency. Raman spectroscopy confirmed the graphene transfer and indicated that the electrostatic transfer to Pyrex glass introduced few defects into the graphene. The average sheet resistance of the transferred graphene is as low as 150 Ω/□, suggesting that graphene on glass is a suitable material for electronic devices.

Electrostatic transfer of graphene to glass substrates is a reliable technique that can be integrated into a large-scale fabrication of graphene devices. In comparison to other transfer methods, electrostatic transfer offers the advantage of absence of adhesive residue or contamination of the graphene film. This electrostatic transfer technique could potentially allow the integration of graphene with structured and electronically active substrates such as MEMS and CMOS.

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