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Gate voltage and temperature dependent Ti-graphene junction resistance toward straightforward p-n junction formation

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High-quality metal-graphene contact is crucial for the fabrication of high-performance graphene transistors. Although Ti has been widely used as metal electrodes in graphene-based devices owing to its excellent adhesive capability, contact resistance (R_c) for Ti/graphene (Ti/Gr) is typically high and varies largely by three orders of magnitude from $\sim 10^3$ to $10^6 \Omega \mu\text{m}$. Here, we have systematically investigated the effects of gate voltage (V_G) and temperature (T) on R_c in the Ti/Gr interface. Besides significant V_G dependence, R_c in the n branch is always larger than that in the p branch, indicating a Ti induced n-doping in graphene. In addition, R_c exhibits an anomalous temperature dependence and drops significantly as the temperature decreases, reaching $\sim 234 \Omega \mu\text{m}$ at 20 K. Such Ti/Gr contact can adjust the Fermi energy of up to 0.15 eV and can also directly form a well-defined sharp p-n junction without extra gates or chemical doping. These findings pave the way to develop the next generation of graphene-based electronic and optoelectronic devices. *Published by AIP Publishing.* <https://doi.org/10.1063/1.5052589>

I. INTRODUCTION

Graphene has emerged as one of the most promising candidates for next generation nanoelectronics due to its high mechanical strength,¹ ultrahigh thermal conductivity,² and excellent electrical conductivity with high carrier mobility.³ It has been used for a wide variety of multifunctional applications, such as high-speed flexible electrodes,^{4,5} field effect transistors (FETs),^{6–8} and ultrastrong membranes.^{9,10} However, many of the applications in electronic devices are limited by the high contact resistance (R_c) between graphene and metals. To date, there have been numerous papers addressing the experimental and theoretical studies on the impact of metals on graphene.^{11–13} Meanwhile, the reported R_c of graphene-based field effect transistors (FETs) is varied in $\sim 10^2$ to $10^6 \Omega \mu\text{m}$,^{11,14} which is considerably larger than the channel resistance of graphene and also several times larger than the contact resistance required in FET devices. For example, R_c as low as around $80 \Omega \mu\text{m}$ is needed in the commercial silicon metal-oxide-semiconductor field effect transistor (SMOSFET).¹⁵ Or worst of all, the performance of graphene-based FETs can be degraded by the formation of metal-carbide at the contact area.^{16,17} Therefore, understanding and realizing the low contact resistance of metal on graphene is crucial for the development of any high-performance graphene-based FETs.

Metals including Co, Ni, Pd, and Ti can form chemical contacts with graphene, which differs from the physical

contact of metals such as Al, Ag, Cu, and Pt due to the weak bonding of graphene to their surfaces.^{16,18} In particular, Ti has been widely used as metal electrodes in carbon-based devices because of its excellent adhesive capability in SiO_2 substrate or other insulators.¹⁶ Nagashio *et al.* compared the contact metals such as Cr, Ti, and Ni on graphene and found that R_c for Ti/Gr and Cr/Gr contact is typically high and varies largely by three orders of magnitude from $\sim 10^3$ to $10^6 \Omega \mu\text{m}$, whereas R_c for Ni/Gr can be as low as $\sim 500 \Omega \mu\text{m}$ and its variation is small as well.¹⁹ Politou *et al.* also reported that R_c of Ti/Gr is around $48\,000 \Omega \mu\text{m}$, which is about 10 times higher than that of Pd/Gr ($2800 \Omega \mu\text{m}$), Au/Gr ($3900 \Omega \mu\text{m}$), and Ag/Gr ($5000 \Omega \mu\text{m}$), and about 4 times higher than Ni/Gr ($9300 \Omega \mu\text{m}$).¹⁶ The high contact resistance of Ti/Gr was mainly attributed to the formation of Ti–C and Ti–O chemical bonds at the contact interface.¹⁷ Gong *et al.* further proved that Ti has strong chemical interactions with graphene, which resulted in Ti carbides.²⁰ Although Freedy *et al.* employed ultrahigh vacuum (UHV) electron beam evaporation and significantly improved the contact resistance, R_c still retains a high value of $\sim 2000 \Omega \mu\text{m}$,²¹ which is much higher than that of Ni/Gr contact ($349 \Omega \mu\text{m}$) and Pd/Gr contact ($230 \Omega \mu\text{m}$) reported in the literature.^{12,22} Therefore, the interaction between Ti and graphene and the associated high R_c is the bottleneck that hinders the development of graphene-based electronic devices. Moreover, measurements of contact resistance in the Ti-graphene interface have been inconsistent. In addition, the factors that determine the contact resistance remain unclear. In this letter, we have used chemical vapor deposition (CVD)-grown graphene to fabricate FETs with Ti

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contact, followed by post-annealing under a pressure of 10^{-6} Torr at 200 °C for a long time. Indeed, annealing can remove the residuals sandwiched at the metal-graphene contacts and hence improve metal-graphene contacts.²² Then the influences of back gate voltage and temperature in R_c of a Ti/Gr junction were investigated systematically. More importantly, instead of using multiple gates or typical chemical doping, a well-defined graphene p-n junction with low contact resistance was obtained. This work may lead to possible applications of Ti in graphene-based electronic and optoelectronic devices.

II. EXPERIMENTS

Single crystal graphene films were deposited on a 25- μ m Cu foil by a typical atmospheric pressure chemical vapor deposition (APCVD) process.²³ Prior to growth, the copper foil was electro-polished using a typical electrochemical workstation. The electro-polishing solution was composed of 200 ml de-ionized water, 100 ml ortho-phosphoric acid, 100 ml ethanol, 20 ml isopropyl alcohol, and 2 g urea. During the growth process, H_2 with 500 sccm flow rate and CH_4 with 4.5 sccm flow rate were purged to the chamber. The typical growth temperature and time were 1035 °C and 120 min, respectively. Subsequently, the as-prepared graphene sample on copper foil was coated one PMMA layer and the Cu foil was removed in ammonium persulfate (APS) solution. Graphene flakes were then transferred on a SiO_2/Si substrate after removing PMMA in acetone. Scanning electron microscope (SEM), Raman microscopy, and transmission electron microscopy (TEM) were performed to investigate the crystallinity and morphology of the as-grown graphene flakes. Graphene-based FET arrays with Ti/Au (5 nm/50 nm) electrodes were prepared by typical lithography and thermal

evaporation, followed by the liftoff process and post-annealing at a pressure of 10^{-6} Torr at 200 °C for 8 h.

III. RESULTS AND DISCUSSION

Figure 1(a) shows a typical SEM image of graphene grown on copper foil by the APCVD method. This large graphene domain can be intactly transferred from the copper foil to a SiO_2/Si substrate [Fig. 1(b)]. All the graphene domains shown in the SEM images and the optical images appear in an elongated hexagonal shape with smooth edges. The size of the largest graphene domain is around 50 μ m. Typical Raman spectra [Fig. 1(c)] taken on the graphene domain after being transferred on the SiO_2/Si substrate exhibit sharp G (1587 cm^{-1}) and 2D (2719 cm^{-1}) peaks with a 2D/G ratio of 2.2, which confirm that the graphene domains are single layer graphene sheets.²⁴ On the other hand, the fact that the disorder-induced D band (1350 cm^{-1}) is hardly detected on such graphene domains further indicates that the graphene domains are of high quality.²² In order to characterize the crystallinity of the single crystal graphene domains by TEM, the as-prepared graphene flakes were transferred on a holey carbon-covered TEM grid [Fig. 1(d)]. The inset of Fig. 1(d) shows one of the selected area electron diffraction (SAED) patterns taken at the area marked in Fig. 1(d). The sharp and bright spots in the SAED pattern fit the typical hexagonal structure well and are consistent with the previous reports.²⁵

A typical example of a graphene transistor array with Ti/Au electrodes is shown in Fig. 2(a), revealing three transistors designed for contact resistance measurement and electrical transport study. The electronic transports of graphene flakes were performed on a back-gated field effect transistor that was built in a graphene domain on a highly doped p-type silicon substrate with 285 nm silicon dioxide. Device A and device B

FIG. 1. (a) SEM image of CVD-grown single crystal graphene. (b) Optical image of the as-prepared graphene transferred on Si wafer with 285 nm SiO_2 . (c) Raman spectra of the graphene on three different positions (black, red, and blue). (d) TEM image of graphene on holey carbon TEM grids. The inset is the corresponding SAED pattern.

FIG. 2. (a) Optical image of fabricated FETs based on the transferred monolayer graphene. (b)–(d) are the corresponding Raman mapping at D, G, and 2D peak positions. The bar is 7 μm .

have the same width (1 μm) of the graphene channel, whereas device C is 3 μm in width. Raman spectroscopy is a powerful technique to identify the number of graphene layers and the presence of defects in graphene. The intensities of D, G, and 2D of characteristic graphene Raman peaks, D (1350 cm^{-1}), G (1580 cm^{-1}), and 2D (2690 cm^{-1}), were extracted and their spatial dependences (Raman maps) are plotted in Figs. 2(b)–2(d). Intensities of G and 2D peaks from the cleaned area were distinctly higher than the other areas, which indicated that the purity of graphene on the cleaned area was enhanced as the residues were removed during the annealing process.

Figure 3(a) demonstrates the equivalent circuit of graphene FETs. Note that the total resistivity R_{total} consisted of two parts: $2R_c$ and R_{channel} .¹¹ The former is the contact resistance in the Ti/Gr contact junction, while the latter is the intrinsic resistance of CVD-grown graphene channel. It is noteworthy that the R_{channel} is changeable according to the modulation of carrier density in the graphene channel. Figure 3(b) shows the $I_{\text{ds}}-V_{\text{ds}}$ characteristics of Ti/Gr FET devices. The gate voltage was applied through the doped Si substrate ranging from 0 to 50 V. The linearity of the $I_{\text{ds}}-V_{\text{ds}}$ curves indicates the formation of Ohmic contacts between Ti and graphene and the lack of significant Schottky barriers at the interface as well.¹⁴ Since the deposition chamber has been kept at very low vacuum ($\sim 10^{-6}$ Torr) to avoid spurious oxidation and/or catalyzed carbidization,¹⁶ we do not think that the contact resistance is due to carbide or oxide formation. In this case, contact resistance R_c can be extracted:^{11,14}

$$R_{\text{total}} = R_{\text{channel}} \frac{L}{W} + 2R_c/W_c, \quad (1)$$

where L is the channel length, W is the channel width, and W_c is the contact width. Thus, the contact resistance R_c can be extracted directly from the fitting equation (y-intercept). Figure 3(c) plots the average R_{total} as functions of the channel length with linear fitting. The values of R_c at a bias voltage of 50 and 0 V are estimated to be about 299.2 and 558.1 $\Omega\mu\text{m}$, respectively. Subsequently, R_c on the Ti-Gr junction as a function of back gate voltage was measured at room temperature and the results are plotted in Fig. 3(d). Apparently, the contact resistance R_c exhibits clear gate-voltage dependence. It peaks around V_{Dirac} and decreases as the channel is field-doped by the back gate. Moreover, the p- and n-branches always show a moderate asymmetry: R_c in the n branch is slightly larger than that in the p branch, indicating a Ti-induced n-doping of the graphene segment underneath it. By contrast, gate-dependent R_c for both Pd/Gr and Ni/Gr contacts exhibit a p-doping effect with similar asymmetry of the p- and n-branches.^{11,12} It can be attributed to the work function difference ($\Delta\Phi$) between graphene and the metals.^{26,27} The work function of Ti (4.3 eV) is slightly lower than that of graphene (4.5 eV), while the work functions of Ni (5.2 eV) and Pd (5.17 eV) are higher.^{16,19} Such work function difference should lead to the band realignment of the metal-graphene junction, resulting in a downshift or upshift of the graphene Fermi level due to the injected holes or electrons.

The transport characteristics of devices A, B, and C at 20 K are plotted in Fig. 4(a). The inset of Fig. 4(a) reveals the transfer characteristics of these devices at 300 K. Both intrinsic (right) and N-doped (left) portions exhibited separated single Dirac point, whereas the p-n junction (middle) produced a representative curve with two Dirac points.

FIG. 3. (a) The equivalent circuit of graphene FETs with Ti contact. (b) Linear I_{ds} - V_{ds} curves of the transistors as a function of back gate voltage indicating Ohmic contact. (c) Deriving contact resistance using the TLM at the gate voltage of 0 and 50 V. (d) Contact resistance R_c as a function of gate voltage.

Notably, the negative Dirac peaks derive from the n-doping induced by Ti in contact with graphene. On the contrary, the p-type graphene grown by CVD and transferred by the wet-transfer process exhibits only one positive Dirac point, which appears at the same location as the observed positive Dirac voltages. The field effect mobility μ is extracted using²⁸

$$\mu = (dI_D/dV_G)_{\text{max}} \times [L/(WC_{ox}V_D)], \quad (2)$$

where L and W are the channel length and width, respectively, C_{ox} is the gate capacitance per unit area ($1.21 \times 10^{-4} \text{ Fm}^{-2}$), and V_D is the drain source bias. As shown in Fig. 4(b), the mobilities of Devices A, B, and C at 20 K are deduced to be about 3443.6, 3411.3, and 3971.3 $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$, respectively. As the temperature reaches 300 K, the corresponding values are 3065.1, 3022.7, and 3394.8 $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$. No matter the temperature is low or high, Devices A and

FIG. 4. (a) The resistances of graphene in devices A, B, and C as a function of the back gate voltage of 50 V at 20 K. The inset is the corresponding resistances of these FETs at 300 K. (b) The electron mobilities (μ) of three FET devices at 20 K and 300 K. (c) Measured metal-graphene contact resistance R_c as a function of temperatures. (d) The R_c of graphene contact on different metals and plotted against the work function of the corresponding metal.

B exhibit almost the same values due to similar FET geometry. We have also measured the temperature dependence of the contact resistance. Figure 4(c) shows the temperature dependence of the contact resistance R_c . At $V_g = 50$ V (heavily p-doped channel), R_c is $\sim 295 \Omega \mu\text{m}$ at 300 K and then drops significantly as the temperature decreases, reaching $\sim 234 \Omega \mu\text{m}$ at 20 K. In general, the thermally averaged R_c can be extracted from the transmission T :¹¹

$$\frac{1}{R_c} = \frac{G_0}{2} \int \left[\frac{T(E)}{L} * \frac{e^{[E - (E_F + eV_g)]/k_B T}}{\{1 + e^{[E - (E_F + eV_g)]/k_B T}\}^2} \right] \frac{dE}{k_B T}, \quad (3)$$

where G_0 , $T(E)$, L , E_F , E , V_g , and T are the quantum unit of conductance, the sum of majority and minority spin transmission, the transverse length, the Fermi energy, the energy, the gate voltage, and the temperature, respectively. Based on Eq. (3), R_c largely rely on transmission values within $k_B T$ of $(E_F + eV_g)$. This equation is particularly important in low-temperature surroundings. At low temperature below tens of K, the carrier mean-free-path (λ) exceeds the metal-graphene coupling length (λ_m), resulting in nearly ballistic transport with a high transfer efficiency, which is also observed in the case of other metal contacts on graphene (e.g., Pd).¹²

Compared with Ti/Gr contact, we have expanded this analysis to other metals, including Cr, Pd, Ni, Au, and Pt, which are widely used for graphene FET fabrication, and the results are summarized in Fig. 4(d). For Cr/Gr and Ti/Gr, R_c shows a large variation ranging from 10^3 to $10^5 \Omega \mu\text{m}$,¹⁹ which is one order or two orders of magnitude larger than that for Ti/Gr contact in our work ($234 \Omega \mu\text{m}$). This can be attributed to the post-annealing treatment we have applied. Previous work has proved that annealing is an effective post-processing treatment commonly used to improve metal-graphene contacts since residue chemicals sandwiched at the metal-graphene contacts can be

removed.²² Moreover, Au/Gr and Pt/Gr demonstrate a mediate R_c of $500\text{--}3900 \Omega \mu\text{m}$.^{29,30} The lowest R_c can be reported in Pd/Gr and Ni/Gr FET devices, corresponding to 210 and $244 \Omega \mu\text{m}$.^{11,31} More importantly, R_c in this work is far superior to the minimum reported value of $\sim 2000 \Omega \mu\text{m}$ in Ti/Gr contact and also comparable to the reported values ($200\text{--}400 \Omega \mu\text{m}$) for Pd/Gr and Ni/Gr FETs.^{12,21}

The mechanism of carrier transport in a metal-graphene junction can be quantitatively described by a quantum tunneling model using Landauer's approach.^{12,18} The metal-graphene contact resistance can be expressed by $1/R_c = 2e^2 TM/h$, where e is the electron charge, T is the probability of carrier tunneling through the metal-graphene contact interface, M is the number of conduction modes in graphene, and h is the Planck constant. Generally, M is determined by the Fermi level relative to the Dirac point (E_F) $M = (E_F \hbar V_F)W$, where $E_F = \hbar V_F \sqrt{\pi \sqrt{(n^2 + n_0^2)}}$, $\hbar$ is the reduced Planck constant, V_F is the Fermi velocity ($\sim 1.0 \times 10^6 \text{ ms}^{-1}$),³² n is the carrier concentration, and n_0 is the residual concentration, resulting from the homogeneous broadening of the electronic states in graphene owing to coupling with the metal.³³ In addition, the probability of tunneling is expressed as follows:¹⁸

$$T = \left\{ 1 + U_0^2 \sinh^2 \left[W_B \sqrt{2m^* (U_0 - E_F)/\hbar} \right] / 4E_F (U_0 - E_F) \right\}^{-1}, \quad (4)$$

where U_0 and W_B are the barrier height and width, respectively, and $m^* = 0.012m_0$ (m_0 is the mass of electron) is the effective mass of electrons in graphene induced by electron-phonon interactions.³⁴ Notably, T is determined by the nature of graphene under the contact and certain parameters of the metal-graphene interface, including E_F , U_0 , and W_B . Apparently, it indicates that shifting the Fermi level away from the Dirac point and cleaning the metal-graphene

FIG. 5. (a) The band alignment illustrations of the graphene-Ti film. (b) Average effective potential in the graphene-Ti film along the graphene [0001] direction. (c) Graphene p-n junction formation owing to Ti contact.

interface are possible methods for improving the metal-graphene contact, which is consistent with former reports on the Ni-graphene contact.^{11,22}

Figure 5(a) shows the schematic band diagram of the Ti-graphene junction, with the vacuum level and the original work function (or Fermi energy) of Ti fixed as a reference. The work functions of Ti and graphene are 4.3 eV and 4.5 eV, respectively.¹⁹ Thus, the barrier height for electron tunneling through the interfacial layer is ~ 0.2 eV, leading to an upshift (ΔE_F) for the graphene Fermi energy. Generally, the carrier density (n) can be given by $n = C_{OX} V_{Dirac} / e$, where V_{Dirac} is the Dirac point shift relative to that of pristine graphene. In this device, the shift of V_{Dirac} is calculated to be about 17.1 V based on the gap between two Dirac peaks [Fig. 4(a)]. Thus, the induced electron concentration is estimated to be $\sim 1.29 \times 10^{12} \text{ cm}^{-2}$. Meanwhile, the change in Fermi energy is given by

$$\Delta E_F(n) = \hbar v_F \sqrt{\pi n}, \quad (5)$$

where v_F is the Fermi velocity ($\sim 1.0 \times 10^6 \text{ ms}^{-1}$).³² Hence, the Fermi energy in graphene with Ti contact can be adjusted to 0.146 eV, which is corresponding to the effective potential in Fig. 5(b). In this case, the effective potential is inclined in the graphene. Driven by the potential difference, the electrons can easily transfer from Ti film to graphene, thereby leading to the presence of a built-in electric field along the same direction. Then the donated electrons can raise the Fermi level of graphene, thus inducing an n-type doping, as proved by gate dependent contact resistance where R_c in the n branch is always larger than that in the p branch, indicating the presence of n-doping in the Ti-graphene interface [Fig. 3(d)]. Therefore, in the Ti/Gr contact system, a well-defined graphene p-n junction can be achieved, evidenced by the transport study in Fig. 4(a). Utilizing the intrinsic p-doping and Ti-induced n-doping, metal titanium contact can be used to build graphene p-n junctions directly [Fig. 5(c)]. The p-doping in as-prepared graphene is attributed to the residual charges created by a variety of surface treatments to Si/SiO₂ substrates.³⁵ Currently, graphene p-n junctions are typically fabricated by local chemical doping of graphene or multiple gate modulations.^{36,37} Herein, instead of using multiple gates or typical chemical doping, straightforward graphene p-n junction formation through Ti contact represents an economical, convenient, and efficient method.

IV. CONCLUSIONS

In sum, the influences of back gate voltage and testing temperature in contact resistance in the Ti/Gr interface have been investigated systematically. First, R_c exhibits distinct gate dependence and the value in the n branch is always larger than that in the p branch, indicating a Ti-induced n-doping of the graphene. Moreover, R_c in a Ti-graphene junction demonstrates an anomalous temperature dependence, dropping significantly to a value of $\sim 234 \Omega \mu\text{m}$ at 20 K as the temperature decreases. The value would be far superior to the minimum reported value for $\sim 2000 \Omega \mu\text{m}$ in the Ti/Gr contact and also comparable to those values ($210\text{--}244 \Omega \mu\text{m}$) for the Pd/Gr and Ni/Gr contacts. Finally, such Ti contact

with graphene can adjust the Fermi energy of up to 0.15 eV and hence produce a well-defined sharp p-n junction without extra gates or chemical doping.

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