Transport evidence of second-order Dirac cones in graphene monolayer on twisted boron nitride substrate

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Strong band engineering can be achieved by twisting successive layers of two-dimensional (2D) materials with a small angle, where rich physics and new applications emerge. Even though controlling the twist angle between bilayer channel materials or between the channel material and the substrate has been intensively studied, the effects of twisted bilayer substrates on the unaligned channel material are much less explored. In this work, we report the realization of second-order multi-Dirac cones with the coexistence of the main Dirac cone in a monolayer graphene (MLG) on a 1° twisted double-layer boron nitride (tBN) substrate. Transport measurement reveals the emergence of multiple metallic or insulating states around the pristine Dirac cone, featuring three pairs of prominent second-order Dirac points. From the insulating states we find that the second-order Dirac cones have gapless spectra; from the metallic states we extract displacement field tunable, electron-hole asymmetric Fermi velocities. The experimental observation of multi Dirac cones in MLG/tBN heterostructure is supported by our band structure calculations employing a periodic potential. Our results unveil the potential of “twisted-substrate” as a universal band engineering technique for 2D materials regardless of lattice matching and crystal orientation, which might pave the way for a new branch of twistronics.

The recently developed technique that enables the fabrication of van der Waals heterostructures with highly controlled orientations initiated a substantial surge in the research of moiré physics in these systems\cite{1,2}. The first widely investigated system is twisted bilayer graphene (tBLG)\cite{1-10}. Flat energy bands form near charge neutrality when the twist angle between two graphene monolayers is close to 1.08°, i.e. the so-called “first magic angle”. The formation of flat bands plus the energy gap that isolates the flat bands from dispersive bands, lead to a clear manifestation of electron correlation and many-body ground states in this system. Phase diagram of tBLG near
magic angle consists of a correlated insulating state surrounded by two superconducting domes[2,10], resembling high $T_c$ superconductors such as cuprates[11-13] and iron pnictides[14,15]. Besides tBLG, there are plenty of other moiré structures including homobilayers, heterobilayers and multilayers, with constituents being graphene, boron nitride (BN)[16-18], transition metal dichalcogenides[19,20], 2D magnets[21], layered superconductors[22] and so on. Numerous exotic physical phenomena have been discovered, such as orbital magnetism, quantum anomalous Hall states and Chern insulators[3,4,10].

In this paper, we study the effect of a much less explored type of moiré material, twisted boron nitride (tBN) homobilayer, as a “twisted-substrate”. The effect of the twisted-substrate is probed via transport measurement of the unaligned monolayer graphene which is in contact with it. Boron nitride homobilayers are twisted by a small angle of $1^\circ$, forming moiré supercells with a periodic potential[23]. We found that the transport behavior of graphene is strongly modified when it is placed 2nm from the twisted BN-BN interface; in contrast, transport behavior of graphene seems unaffected when it is placed 8nm from the twisted BN-BN interface, which agrees with predicted exponential decay of the tBN moiré potential with distance[23]. Such control in distance between graphene and the twisted BN interface is achieved via the use of a thin flake of BN with both 2nm-thick portion as well as 8nm-thick portion in the twist-and-stack device fabrication process. Graphene on 2nm-tBN shows multiple emerging insulating and metallic states, which are strongly tuned by the displacement field applied perpendicular to the heterostructure. We observed three pairs of prominent insulating states, each pair located at both the electron side and the hole side of with respect to the pristine Dirac point of the graphene sample. The temperature-dependent resistance of these insulating states does not fit to the thermal activation behavior, indicating the absence of a global gap. This is in agreement with DFT calculations that predict the emergence of three pairs of resonant second-order Dirac points with gapless spectra. On the other hand, the metallic states show linear temperature-dependent resistance, which agrees with expectations of electron-phonon scattering in a Dirac band[24,25]. Interestingly, such linear temperature dependence reveals Fermi velocities that are strongly tunable by displacement field and are electron-hole asymmetric. Thus, tBN-assisted band engineering shows strong tunability on the graphene density of states (DOS) and could be readily generalized to other 2D materials.

Figure 1(a) illustrates the device structure in the experiment. A thin BN flake is torn, twisted by
and stacked on part of itself to form tBN homobilayer structure; such a structure is then placed on top of a monolayer graphene. This tBN-graphene heterostructure is encapsulated by two additional flakes of hBN (~50 nm in thickness), with graphite bottom gate and Cr/Au top gate. The thin BN includes a 2nm-thick portion and an 8nm-thick portion (see Supplementary Information I for details), so that we can simultaneously examine the band tuning effects of different tBN interface-to-graphene distances using a single piece of graphene. The optical micrograph of the device is shown in Figure 1(a), where the tBN region with two different BN thicknesses is marked with color shadings. In particular, the blue shading area indicates the location of twisted 2nm BN-graphene heterostructure (G-2nm-tBN) while the brown shading area indicates the location of twisted 8nm BN-graphene heterostructure (G-8nm-tBN).

Dual gate transport curves are obtained at 2 K for both G-2nm-tBN and G-8nm-tBN, as shown in Figure 1(b) and (c), respectively. In sample G-2nm-tBN, multiple resistance peaks near the main Dirac point appear and the relative amplitudes of these peaks vary with the bottom gate, indicating that they can be tuned by a changing displacement field. For comparison, in sample G-8nm-tBN, all the transport curves with top gate swept at different bottom gate voltages are very similar to that of a normal hBN encapsulated graphene. Since the same piece of hBN is interfaced with the same piece of graphene during the fabrication of both G-2nm-tBN and G-8nm-tBN, the difference between the behavior of these two samples helps us rule out the possibility of unintentional alignment of graphene and tBN, which could form new superlattice and could obscure the effect of the twisted substrate. From the $R_{xx}$-$V_{tg}$ curves of G-8nm-tBN we obtain a mobility of $3 \times 10^4$ cm$^2$V$^{-1}$s$^{-1}$ at 300K and a mobility exceeding $2 \times 10^5$ cm$^2$V$^{-1}$s$^{-1}$ at 2 K, indicating the high quality of our sample. Therefore, the relatively broad resistivity peak in G-2nm-tBN shall be attributed to the effects of the moiré supercells of the 2nm tBN substrate. Later in this paper, we will focus on sample G-2nm-tBN where interesting new physics is observed.

Figure 2(a) shows the carrier density $n$ and displacement field $D$ dependent resistance $R_{xx}$ of G-2nm-tBN. One can see an insulating state exists near $n = 0$ cm$^{-2}$ which is slightly tuned by $D$. This state is at the peak of the bell-shaped transport curve, therefore recognized as the main Dirac point, as marked by dotted line in Figure 2(a). This assignment of the main Dirac point is also confirmed by Hall measurement as shown in Figure 2(b). The $n$ and $D$ dependent $R_{xx}$ map also
reveals a series of resistance peaks around the main Dirac point, with the most prominent six marked by black arrows in Figure 2(a). It is clear in Figure 2(a) that these resistance peaks are highly dispersive with respect to $D$. In order to better understand the resistance features in Figure 2(a), the carrier density dependent resistance change ($\Delta R_{xx} = R_{xx}(T) - R_{xx}(T = 32 \text{ K})$) for $T$ ranging from 0.25 K to 32 K is shown for three different $D$ values: $D = +0.18 \text{ V/nm (Fig.2(c))}$, 0 V/nm (Fig.2(d)) and -0.18 V/nm (Fig.2(e)), respectively. The main Dirac point is marked by red triangles in Figure 2(c)-(e), while the six prominent insulating states around the main Dirac point for the above mentioned three different $D$ values are marked by black triangles (two full triangles and four empty triangles). The two insulating states marked by full triangles will be discussed in detail in the main text, while the other four insulating states marked by empty triangles have very similar behaviors and are discussed in the Supplementary Information IV. On the electron and hole sides at the immediate vicinity of the main Dirac point, two prominent metallic states are also visible (marked by blue arrows in Figure 2(c)-(e)), and we are going to analyze them further in this article.

In Figure 2(b), the $n$ and $D$ dependence of the Hall conductance $1/R_H$ is shown. Ripples appear in the Hall conductance, yet there is no global deviation from the straight lines, nor any additional sign reversal behaviors like those in tBLG[7], twisted double bilayer graphene (tDBG)[26], graphene under supercell[27] or artificial periodic potential. Thus, although tBN provides a periodic potential, it does not fold the Dirac band completely and form global higher order Dirac points, nor does it open a band gap. The tBN moiré only creates “second-order” Dirac points together with an uneven DOS in the graphene energy band. This assertion is also supported by the investigation of the temperature-dependent resistance of the insulating states around the main Dirac point, as discussed below in Figure 3.

It is worth noting that a similar work on graphene/tBN structure has been done recently[18], where monolayer hBN is fabricated into parallel or small angle (0.6°) twisted homobilayers structure. Large ferroelectricity switching behavior is observed in parallel (0.6° twisted) BN bilayer structure, with the coercive displacement field $\Delta V_g/t_{BN} \approx 0.13 \text{ V/nm (0.03 V/nm)}$, see Fig.3c in Ref.[18]. In our experiment, no sign of ferroelectricity is observed for sweeping both top and bottom gate in two directions, see Supplementary Information Figure S2, down to a hysteresis level of $\Delta V_g/t_{BN} \approx 0.0002 \text{ V/nm}$, since we are in a different regime. Detailed discussions on the
absence of ferroelectricity in our devices can be found in Supplementary Information II.

Next, we turn to the temperature dependent longitudinal resistance of two insulating states and two metallic states, which are marked by black full triangles and blue arrows in Figure 2 (c)-(e) respectively. For the insulating states, the corresponding Arrhenius plots are shown in Figure 3 (a) and (b). The temperature dependence of the insulating states does not fit to the thermal activation behavior, therefore the graphene spectrum under tBN is indeed gapless even at the second-order Dirac points, consistent with Hall measurement results. $R_{xx}$ vs. $T$ curves of the metallic states are shown in Figure 3 (c) and (d). For both electron and hole sides, $R_{xx}$ is linear with $T$, which is a common behavior in similar graphene moiré systems[25,26,28]. Such a linear dependence is a consequence of acoustic phonon scattering of charge carriers[25,29]. Theoretically, resistivity due to quasi-elastic scattering of acoustic phonons in a graphitic system can be expressed as:

$$\rho = \frac{\pi F D A^2 g e^2}{6h \rho_m v_F^2 v_{ph}^2} k_B T \quad \text{Eq.(1)}$$

where $F$ is the form factor accounting for the scattering matrix elements of differing electron-phonon processes. In the case of monolayer graphene, $F = 1$. $D_A = 18\text{eV}$ is the deformation potential describing the electron-phonon scattering strength. $g = 4$ is the degree of degeneracy of spin and valley for monolayer graphene, and $\rho_m = 7.6 \times 10^{-7} \text{kg/m}^2$ is the mass density of graphene. $v_F$ and $v_{ph} = 2 \times 10^4 \text{m/s}$ are Fermi velocity and phonon group velocity in graphene, respectively[30]. Since $D_A$ and $v_{ph}$ are properties of the atomic lattice of graphene, it is reasonable to assume both parameters are invariant with the applied displacement field $D$ or carrier density $n$. Therefore, the slope of the linear $R_{xx}$ vs. $T$ curves $S = dR/dT \propto 1/v_F^2$ gives us an estimation of Fermi velocity of this system. The $D$ dependence of such slopes, $S_E$ for electrons and $S_H$ for holes, are plotted in Figure 3 (e). As $|D|$ increases, both $S_H$ and $S_E$ clearly drop, indicating enhanced $v_F$ at large $|D|$. Meanwhile, the ratio of $S_H$ to $S_E$ is shown in Figure 3 (f), which shows a monotonic dependence on $D$. From Eq.(1) we are able to extract the Fermi velocities on both the hole ($v_{F,H}$) and electron side ($v_{F,E}$). It is clear that $v_{F,H}$ is larger (smaller) than $v_{F,E}$ for $D < 0$ ($D > 0$). In other words, as $D$ varies from -0.35 V/nm to 0.35 V/nm, the ratio of $v_{F,E}$ to $v_{F,H}$ increases monotonically and reaches 1 near zero displacement field (Figure 3(h)), revealing an electrical switchable electron-hole asymmetry in the tBN modulated monolayer graphene.
To understand the physics behind the insulating states and metallic states which are highly dispersive in $D$, we employ band calculation of the MLG/tBN heterostructure based on density functional theory (DFT). It has been showed that twisted bilayer hBN forms a moiré pattern, which acts as periodic potential and can be used to engineer the electronic properties of the materials close to it\cite{18,23,31}. Here we consider the model Hamiltonian\cite{32,33}:

$$H = t \sum_{\langle i,j \rangle} a_i^\dagger a_j + V \sum_{\alpha,i} \cos(G_\alpha x_i) n_i$$  \hspace{1cm} \text{Eq. (2)}$$

where $t$ is the magnitude of the nearest neighbor hopping on the graphene honeycomb lattice, $V$ is the magnitude of the periodic potential, and the reciprocal vectors $G_\alpha (\alpha=1,2,3)$ are determined by the moiré pattern of the twisted hBN layers. $G_1 = 4\pi/\sqrt{3}\lambda (\cos\theta, \sin\theta)$, where $\lambda$ is the wavelength of the moiré superlattice, and $\theta$ is the angle between the moiré pattern and the graphene lattice. $G_{2,3}$ are the other two vectors generated by rotating $G_1$ by $60^\circ$ and $120^\circ$ respectively. Adapting the typical values $t = 2.7$ eV and $V = 30$ meV, we calculated the low energy band structures of tBN supported MLG as shown in Figure 4. Indeed, in addition to the pristine Dirac cone (D0), multiple second-order Dirac cones emerge in the electronic band of a graphene monolayer on the tBN substrate (labeled as D1, D2, D3), and there is no global band gap. Thus, there are dips in the DOS around these second-order Dirac cones which are mainly responsible for the insulating behavior discussed in Figure 2. There are three pairs of prominent second-order Dirac cones due to resonance effects\cite{33,34} from a perturbative viewpoint in the band structures at each side of the pristine Dirac cone and thus three pairs of corresponding dips in the DOS (see Supplementary Information III), which are in accord with the three pairs of insulating resistance peaks experimentally measured as shown in Figure 2(a). Combining the experimental results and theoretical calculations, we conclude that the moiré potential in the MLG/tBN heterostructure could induce multiple second-order Dirac cones near the major Dirac point, and could lead to electric displacement field tunable electron-hole asymmetry in graphene, revealed by the adjustable Fermi velocities in Figure 3(e)-(h).

In conclusion, we have fabricated the heterostructures of an unaligned monolayer graphene with $1^\circ$ twisted double layer BN as substrate. We experimentally ensured that such an unaligned graphene-BN interface by itself does not result in any peculiar transport properties in graphene, while the twisted BN interface creates a moiré potential that would strongly modify the graphene
band structure. Gapless second-order Dirac cones are observed around the pristine Dirac point in the modified band structure of the MLG/tBN heterostructure when the graphene is 2 nm away from tBN interface, which is corroborated by DFT calculations. Fermi velocities near the main Dirac point are highly tunable by the applied displacement field and are electron-hole asymmetric. Comparing to the widely studied “twisted channel” and “twisted channel-substrate” method which created static Fermi velocities, the “twisted substrate” method provides highly tunable Fermi velocities, which adds more control parameters in the search for exotic emerging physics. Furthermore, our result unveils the potential of twisted thin double layers of BN as a universal provider of a moiré potential that does not require specific alignment between the two-dimensional conductive channels and the encapsulated BN crystal, which may pave the way for a new branch of twistronics.

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**Contributions**

J.-H.C. and S.C. conceived the experiment; S.C. fabricated the device and performed most of the measurement; N.M. and M.C. aided in device fabrication; R.Z., J.L., Y.Z. and S.Y. aided in transport measurement; J.Z. and X.C.X. provided theoretical analysis; K.W. and T.T. grown high quality boron nitride bulk crystals; S.C., J.Z., M.C. and J.-H.C. wrote the manuscript and all authors commented and modified the manuscript.

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Transport evidence of second-order Dirac cones in graphene monolayer on twisted boron nitride substrate

Figure 1. Longitudinal resistance of twisted boron nitride (tBN) decorated monolayer graphene. (a) Optical microscopic picture of the tBN-graphene heterostructure device with twisting angle ~ 1°. Blue shading indicates twisted 2nm hBN double layer area (G-2nm-tBN), and brown shading indicates twisted 8nm tBN double layer area (G-8nm-tBN). Scale bar is 5μm. (b-c) Transport curves of (b) G-2nm-tBN and (c) G-8nm-tBN at $T = 2$ K. Curves from right to left correspond to bottom gating voltage varying from -2.5 V to +2.5 V, in 0.5 V steps. Insets: schematic illustration of tBN and monolayer graphene heterostructure.
Figure 2. Carrier density and displacement field dependent transport of the G-2nm-tBN sample. (a) The resistance at $T=250\text{mK}$ as a function of displacement field $D = \varepsilon_{\text{BN}}\left(V_{tg}/d_{tg} - V_{bg}/d_{bg}\right)/2$ and carrier density $n = \varepsilon_0\varepsilon_{\text{BN}}\left(V_{tg}/d_{tg} + V_{bg}/d_{bg}\right)/\varepsilon$, where $\varepsilon_{\text{BN}} = 3.5$ is the relative permittivity of hBN, $V_{tg}$ ($V_{bg}$) and $d_{tg}$ ($d_{bg}$) are gate voltage and thickness of the hBN on top (bottom), respectively. The dotted line denotes the Dirac point, black arrows mark strong insulating states around the Dirac point, as guide for the eye. (b) Carrier density and displacement field dependence of the Hall conductance. (c-e) The carrier density dependence of $\Delta R = R(T) - R(32\text{K})$ at (c) $D = +0.18\text{ V/nm}$, (d) $D = 0\text{ V/nm}$ and (e) $D = -0.18\text{ V/nm}$. Different curves correspond to temperature from 0.25K (purple) to 32K (red), in approximately 4K steps. $\Delta n$ is the carrier density with respect to the Dirac point where $n$ is taken to be zero. Red triangles denote the main Dirac point, black triangles denote the position of side Dirac points, among them the full ones and empty ones will be discussed in the main text and Supplementary Information respectively. Blue arrows indicate the metallic states in the vicinity of the main Dirac point, which will be addressed in Figure 3.
Figure 3. Temperature dependent behavior of the metallic and insulating states in the G-2nm-tBN sample. (a-b) Arrhenius plot of the second insulating states from the main Dirac point at (a) the hole side and (b) the electron side, marked by red arrows in Figure 2 (c)-(e). (c-d) linear $R-T$ relationship of the metallic states at (c) the hole side and (d) the electron side near the Dirac point, marked by blue arrows in Figure 2 (c)-(e). Dashed lines are guide for eye. (e) $dR/dT$ as a function of displacement field $D$ for the metallic states at the hole and electron sides and (f) their ratio. (g) Experimentally extracted Fermi velocities at the electron and hole sides and (h) their ratio.
Figure 4. Multi-Dirac cones in a tBN decorated graphene monolayer. Calculated energy spectrum and density of states (DOS) of MLG/tBN with $V = 30$ meV for an 81×81 graphene supercell. The original Dirac cone of the graphene and three additional Dirac cones above it are labeled as D0, D1, D2, and D3, respectively. The two black arrows pointing D0 cone indicate the metallic states analyzed in Figure 3. The right panels show the zoom-in spectra.