

A review of experimental advances in twisted graphene moir\'e superlattice

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TOPICAL REVIEW — Twistronics

A review of experimental advances in twisted graphene moiré superlattice*

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Twisted moiré superlattice receives tremendous interests since the discovery of correlated insulating states and superconductivity in magic angle twist bilayer graphene (MA-TBG) [*Nature* **556** 80 (2018), *Nature* **556** 43 (2018)], even gives arise to a new field "twistronics" [*Science* **361** 690 (2018)]. It is a new platform hosting strong electron correlations, providing an alternative for understanding unconventional superconductivity. In this article, we provide a review of recent experimental advances in the twisted moiré superlattice, from MA-TBG to twisted double bilayer graphene and other two-dimensional materials based moiré superlattice, covering correlated insulating states, superconductivity, magnetism, *et al*.

Keywords: twisted 2D moiré superlattice, electron correlations, superconductivity, magnetism

PACS: 81.05.U−, 73.21.Cd, 73.50.−h, 74.70.Wz **DOI:** 10.1088/1674-1056/abb221

1. Introduction

In solid-state physics, an electron in a crystal is subjected to a periodic potential from the lattice, and it obeys a dispersion from band structure in the reciprocal space.[4] The emergence of a secondary larger periodic potential, named superlattice, eventually breaks the reciprocal space by introducing miniband structures, regardless of the details about the super structure. In a common crystal, fabricating an artificial superlattice is challenging,[5] and it usually suffers from disorder and tunability. However, in a two-dimensional (2D) system such as graphene and transition metal dichalcogenide (TMD), it is easy to create such a super structure simply by twisting two layers,[6–11] resulting in twisted moiré superlattice with a long wavelength, as depicted in Figs. 1(a) and 1(b), thanks to recent developments in 2D van der Waals heterostructures.[12] Graphene/hexagonal boron nitride (hBN) moiré superlattice is a typical example, the long-periodical potential reshapes the linear band structure by producing superlattice minibands, leading to the observations of satellite secondary insulating states[6–10] and fractal Hofstadter butterfly spectrum.[7–9,13]

Compared to graphene/hBN heterostructure, twisted bilayer graphene is able to tune not only the size of moiré band, but also the electrical coupling between the two layers. The latter changes the story profoundly, bringing a single particle non-interacting picture to a many-body interacting picture.[14] In 2011, Allan H. MacDonald and Rafi Bistritzer predicted a discrete set of angles, at which the electronic Fermi velocity almost vanishes around zero Fermi energy.[14] The formation of flat band at these so called "magic angles", favors correlation effect dominated electronic behavior when the band width is smaller than the Coulomb repulsion energy.

2. The advent of the magic angle

The flat band in the magic-angle twisted double layer graphene will slows down the electrons and minimizes their kinetic energy. Considering its simple atomic structure, it is deemed as an ideal platform to study electron correlated phases and simulate Hubbard model. However, since the prediction of magic angle in 2011, such magic-angle samples were not experimentally realized until 2018.

The first and biggest challenge is the accurate control of the twist angle by transfer technique. Traditional transfer uses the edge of the exfoliated layer as an eye guide, and then stacks the other on top by aligning the edges with an angle. This technique is strongly dependent on the form of the edge structure, which could be a mix of armchair or zigzag, and it fails to meet

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the requirement of accurate angle control of crystal axis and thus brings a large uncertainty in the angle of the twisted bilayer. The breakthrough was the development of the 'tear and stack' technique by Kyounghwan Kim *et al.*[15] It starts with tearing a single flake of graphene into two by hBN stamping, and then the stamp with one graphene flake on the bottom rotates with an angle to pick up the other one. Since the two flakes are of the same lattice axis, the obtained twisted angle can be well controlled and it is only limited by the precision of the rotator stage, which is on the order of 0.01°. Besides, mechanical squeezing technique[16,17] was also developed to remove bubbles and contaminations at the interface of 2D materials, promoting the quality of sample but at a cost of less controllable twisted angle.

Another challenge comes from dynamic instability of a small angle in the twisted bilayer after transfer. The most energetically stable stacking order between the two graphene layers is Bernal stacking, which corresponds to zero-angle stacking. Since the biggest magic angle is around 1.1°,[14] such a small twisted angle will tend to decrease to zero upon the perturbation of heat, strain, or other disturbances,[18–20] while a big twisted angle leads to decoupling of the two layers due to momentum mismatch.[21] Actually, before the emergence of the magic angle sample, there have been observations of a notable renormalization of Fermi velocity,[22–24] insulating states at the superlattice band edges,[22] satellite Landau fans[11,22] by STM/STS and transport measurements. Although no extraordinary phenomena were observed in these preliminary works, they actually indicated the possibility of more exotic phenomena if the magic angle ($\sim 1.1°$) was approached, hinted by the reduced Fermi velocity.[22–24]

In 2018, Y. Cao and P. Jarillo-Herrero *et al.* finally overcame these challenges, successfully obtained magic angle twisted bilayer graphene (MA-TBG) samples, and observed the correlated insulating states[1] and superconductivity.[2]

They demonstrated a flat band nature of MA-TBG, evident from a much reduced Fermi velocity, about $1/25$ of that in the monolayer graphene, as well as a flattening of conductance minimum at the charge neutral point above 40 K.[1] They found gap opening and an insulating state at half filling of single particle moiré band,[1] with a metal–insulator transition at around 4 K. The half-filling states were suppressed by a magnetic field, regardless of the polarization. The insensitivity of gap closure to field orientation suggests a Zeeman effect rather than an orbital effect, indicative of spin singlet state origin.[1]

Even more surprisingly, the presence of superconductor states near the correlated insulating states in the temperature–carrier density (T–n) phase diagram suggests a critical temperature up to 1.7 K.[2] This is similar to phase diagrams with superconductor states flanking Mott states in unconventional superconductors, as shown in Figs. 1(k) and 1(i). The superconductor states were confirmed by a Berezinskii–Kosterlitz–Thouless (BKT) phase transition in voltage–current power law measurements,[2] which is characteristic of 2D superconductors, and further confirmed by the phase-coherent transport behavior from the SQUID-like interference pattern.[2] The relationship between the perpendicular magnetic field and the critical temperature is consistent with the Ginzburg–Landau theory, but the zero-temperature parallel critical magnetic field is slightly larger than the value given by the BCS theory, indicating the possible unconventional origin of the superconductor state in this system.[2]

However, the nature of the superconducting state is still an open question. On one side, there are a number of similarities between the superconductor state in MA-TBG and unconventional superconductors. One is the phase diagram in which superconductor accompanied by Mott insulator,[2] while the other is the observed linear-in-temperature resistance at higher temperature,[25,26] which is similar to strange metal states in unconventional superconductor systems. On the other side, recently some experimental findings strengthen the voice of conventional superconductor state in MA-TBG. By varying the distance between TBG and metallic gate in devices, the correlation strength in TBG can be qualitatively determined. With thinner hBN dielectric layer, the correlated insulator state disappears while the superconductor state remains,[27,28] and it hints that the insulating state is electron correlation dominated yet the superconductivity probably not, similar to alkali-doped C_{60} where the superconductivity and insulator states are of different origins.[28] Surprisingly, adding a tungsten-diselenide (WSe_2) on top of TBG, which induces stronger spin–orbit interaction in TBG, also stabilizes the superconducting state but closes gaps at all integer filling factors, these observations constrain the pairing mechanism to Cooper pair of time-reversed partners.[29] Meanwhile, a recent experiment was capable of *in-situ* control of electron correlation in twisted bilayer graphene, achieved by proximity Coulomb screening from a nearby Bernal bilayer graphene separated by a 3 nm hBN flake, and it demonstrated that superconducting and insulating phases in MA-TBG both become less robust as the Coulomb interaction is weakened by screening,[30] which is in contrast with works above.[27,28] It is also noted that the linear temperature dependence of the resistance can be attributed to not only the many-body Planckian dissipation in strange metal,[26,31] but also the single-particle electron–phonon scattering.[25,32] The carrier density dependence of linear slope favors electron–phonon scattering[33] but recent thermoelectricity measurement in twisted bilayer graphene shows violation of semiclassical description close to half filling, accompanied by temperature-linear resistivity,[34] indicating non-Fermi liquid in twisted bilayer graphene instead of non-interacting metallic state. As mentioned above, both mechanisms agree qualitatively with the experiments in some ways, but credible quantitative judgement needs solid

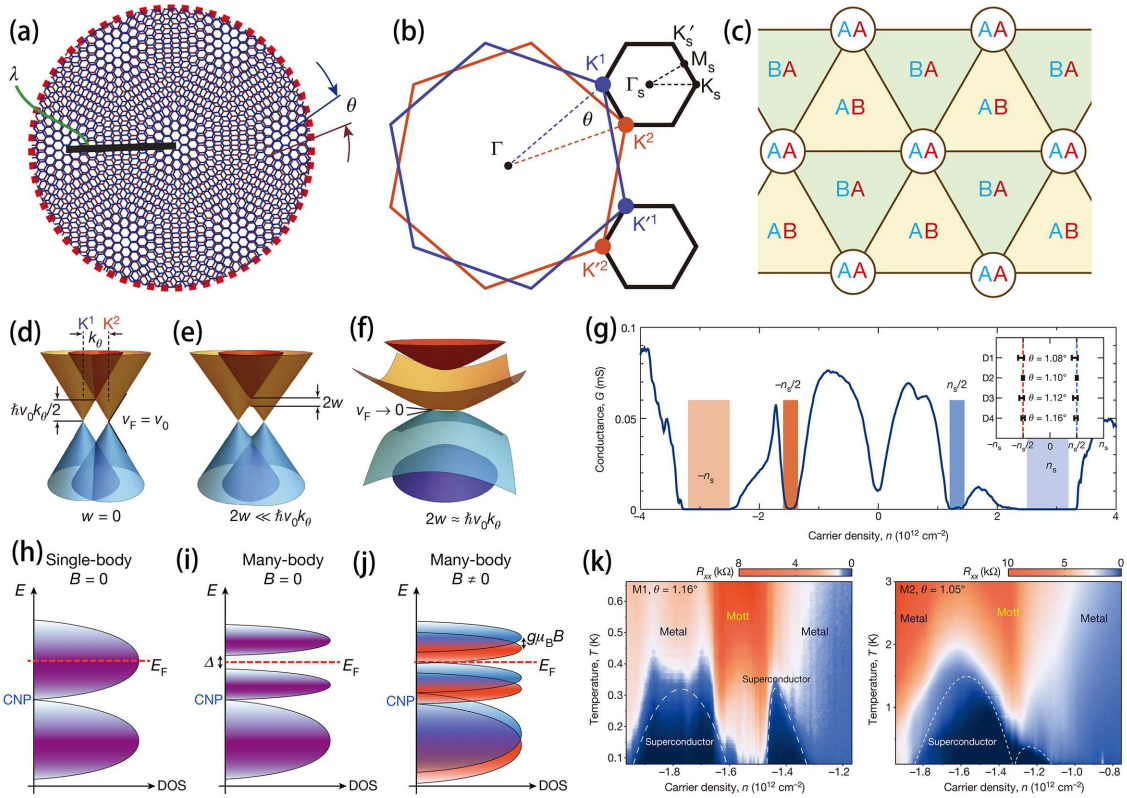


Fig. 1. (a) The moiré pattern in MA-TBG.[1] (b) The constructed moiré Brillouin zone by the difference between the two K (or K') wavevectors for the two layers.[1] (c) Top view of a simplified model of stacking order in MA-TBG.[1] (d)–(f) Effect of interlayer hybridization for twisted bilayer graphene.[1] (g) Half-filling correlated states and superconductor states in magic angle twisted bilayer graphene.[1] (h)–(j) Schematics of DOS in different scenarios,[1] (h) for single-particle flat band, (i) for many-body interaction induced band split, (j) many-body gap closed by Zeeman effect. (k), (i) Phase diagram for MA-TBG.[2] Courtesy from Refs. [1,2].

and repeatable experimental facts. The direct detail investigation into the superconducting dome is also of great significance, anisotropic behavior in the metallic phase and nematic superconducting state demonstrates richer intertwined phase diagram and votes for the unconventional nature of them.[35]

3. Big in small

Owing to the gate tunable electronic properties and the relatively simple chemical component of MA-TBG, these striking observations triggered the big gold-rush in it. Teams from Andrea F. Young and Cory R. Dean quickly followed the work, and aside from twist angle they found a new way to tune superconductivity by applying hydrostatic pressure to the sample.[36] The observation of superconductivity in non-magic angle device is due to the fact that a hydrostatic pressure could lead to a change of interlayer coupling in twisted bilayer graphene. However, details of correlated insulating states and superconducting states are slightly different from that in Cao's work,[1,36] thus demanding an improvement of the sample quality. Soon later, Xiaobo Lu and Dimitri Efetov *et al*. improved the angle uniformity by using the mechanical squeezing process.[16] In collaboration with Adrian Bachtold's group, they found that correlated states are developed at all integer filling factors, that is, 1/4, 1/2, and 3/4 fillings of valence and conduction moiré bands, which indicates a full lift of the spin and valley degeneracies.[16] Moreover, the observed superconductor domes appear not only at a doping close to half filling, but also at around a quarter filling, with a high critical temperature up to 3 K.[16]

Beside correlated insulating and superconducting states, a very interesting and surprising discovery is the observation of the ferromagnetism and quantum anomalous Hall effect (QAH). David Goldhaber-Gordon's team first observed an emergent ferromagnetism near three-quarters filling of the conduction moiré band in TBG, with a giant zero-field anomalous Hall signal and magneto hysteresis.[37] Later, well-developed quantum anomalous Hall effect with Chern number $C = 1$ was confirmed in TBG by Andrea F. Young's team.[38] The twisted bilayer graphene is free of atomic magnetic moment, so the anti-intuitive discovery of ferromagnetism[37] and QAH[38] is the result of a correlated Chern insulator. This is achieved by strong correlations of the moiré band and gap opening by aligning bottom graphene layer and the hBN substrate.

Being simple and small in material science, the effect that TBG brings is big. Superconductivity, strong correlation, topology, and magnetism are important topics in modern condensed matter physics, yet TBG has it all and tends to bring more.

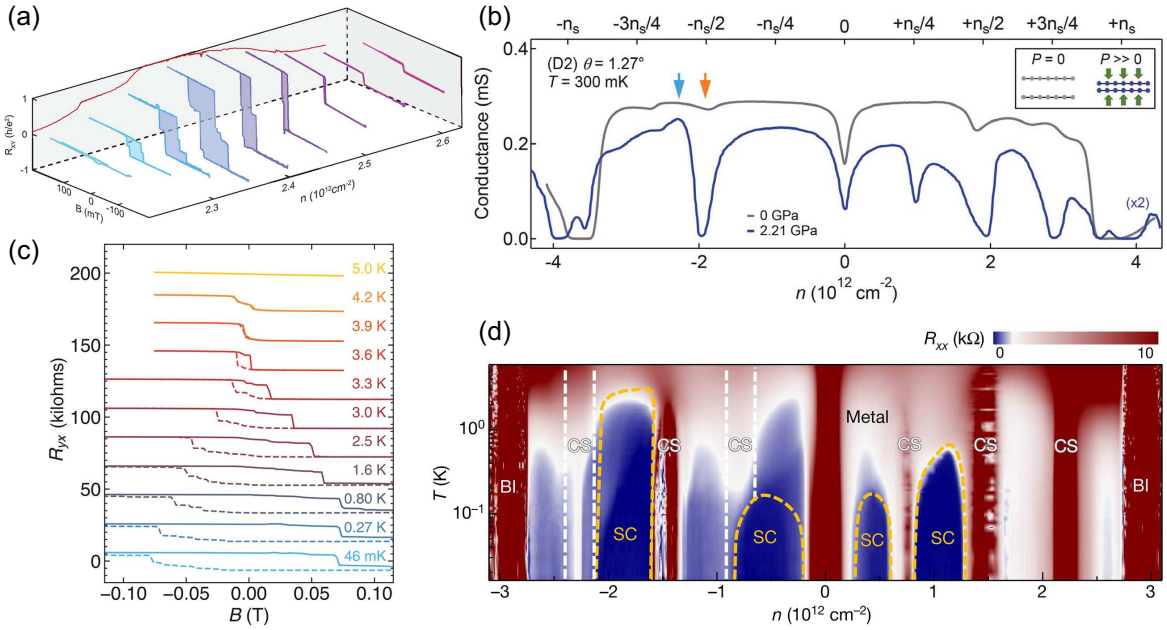


Fig. 2. (a) Quantum anomalous Hall effect[38] and (c) ferromagnetism[37] in magic angle twisted bilayer graphene. (b) Weak correlated insulating states enchanced by high pressure.[36] (d) Insulating states developed at all interger occupation of superlattice band and more superconductor domes.[16] Courtesy from Refs. [16,36–38].

4. The kinsfolk of MA-TBG

Beyond the twisted bilayer graphene, other twisted 2D moiré systems were also investigated and became a new star recently. A collaboration between Feng Wang's and Yuanbo Zhang's groups observed behaviors of correlated insulator,[39] superconductivity,[40] and even field-tunable ferromagnetism[41] in ABC-trilayer graphene/hBN moiré superlattice. The flat band in this system is achieved by tuning a vertical displacement field, which breaks the inversion symmetry and brings the moiré band isolated. At the same time, different teams including Guangyu Zhang's group, Philip Kim's group, and P. Jarillo-Herrero's group, found that twisted double Bernal bilayer graphene (TDBG) is another electric field tunable system, and they observed similar correlated insulating states[42,43,45] and possible superconducting state.[42,44] The magnetic field enhanced correlated insulating state, together with the extracted g factor being nearly 2 from the Zeeman effect, indicates its spin-polarized nature,[42–44,46] which is fundamentally different from MA-TBG. However, no solid evidence of superconductivity has been reported in TDBG, and there exists a finite residual resistance after the superconductor-like transition. Some argued that this state is an additional correlated ground state instead of the superconducting state,[47] however, more works are still needed to reveal the mystery in the future. For a larger twist angle, controlling valley polarization and interlayer coupling is achieved by dual-gated geometry,[48] and new ordered correlated states, density wave states with reconstructed Fermi surfaces emerge,[49] implying a complicated phase diagram in TDBG.

Up to now, most twisted 2D systems are stacked together with two constituent layers being the same, however, with different constituent layers, the twisted systems will have a lower crystal symmetry and thus induce some anisotropic properties. Latest experiments on twisted monolayer-bilayer graphene (TMBG) showed transport features like insulating states and van Hove singularities hosting perpendicular displacement field asymmetry[50–52] and electrically tunable ferromagnetism or Chern insulator with orbital magnetization, whose magnetization direction can be switched purely with electrostatic doping.[50,51,53] It is worth noting that TMBG is the first all-carbon ferromagnetism system without requirement of alignment to hBN. Signature of superconductivity in TMBG was also observed,[52] but experimental evidence is not solid yet. In fact, both electric field and doping can alter the stacking order in few layer twisted graphene,[54] so these correlated states may be more fragile and high-yield technique like 'cut and stack' technique[50,51] needs to be introduced in the future.

Aside from graphene-based moiré superlattice, other 2D materials like transition metal dichalcogenides (TMDs) are also promising candidates for exploring strong correlated phenomena. Twisted bilayer tungsten diselenide (WSe_2) hosts correlated states over a broader range of twisted angles, enabled by the presence of a vertical electric field.[55] WSe_2/WS_2 moiré superlattice shows signatures of Mott states and Wigner crystal states.[56,57] Moiré excitons are explored in WSe_2/WS_2,[58] $MoSe_2/WS_2$,[59] and $MoSe_2/WSe_2$[60,61] superlattices. Thus, it is reasonable to expect more and richer exotic phenomena in twisted 2D materials.

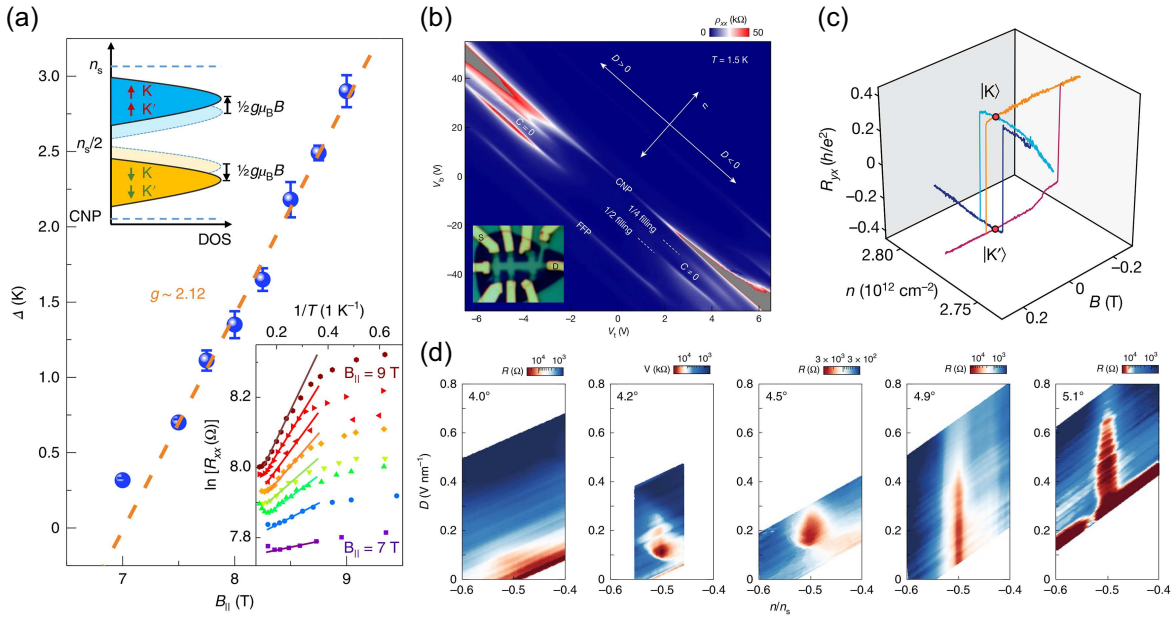


Fig. 3. (a) Spin-polarized correlated insulating state in twisted double bilayer graphene (TDBG).[44] (b) Correlated insulator states and QAHE in ABC-trilayer graphene/hBN superlattice.[41] (c) Nonvolatile switching of magnetic order by electric field in TMBG.[51] (d) Correlated states in a broad range of angle in twisted bilayer WSe_2.[55] Courtesy from Refs. [41,44,51,55].

5. Beyond transport techniques

The development of TBG and other twisted moiré superlattices brings versatile techniques beside electronic transport into the field. Nano angle-resolved photoemission spectroscopy (Nano-ARPES) was introduced to visualize and prove the flat moiré band in TBG.[62,63] Scanning tunneling microscopy/spectroscopy (STM/STS) measurements demonstrated that the density of states is mainly localized in AA stacking domain, and symmetry breaking and electronic correlated effect indeed play a leading role in TBG.[64–68] Signatures of Wigner crystal state and correlated insulator states were observed in WSe_2/WS_2 moiré superlattice by optical spectroscopy.[56,57] In addition, more techniques are joined to speed up the research of twisted 2D materials. Nano-SQUID on-tip helps to map the spatial distribution of the angle in twisted samples.[69] Electronics compressibility measurement unveils the detail about band structure transition and effect of carrier filling.[70,71] Secondary electron microscopy and near-field infrared scope provide non-destructive methods to directly image the local stacking order in fully encapsulated van der Waals heterostructures.[54,72] Advanced tip technologies like piezoresponse force microscopy (PFM) allow the imaging of moiré superlattice and fabrication of twisted 2D materials *in situ*.[73,74]

Currently, owning to the high sensitivity of the band structure to the twist angle and a limited spatial resolution of some techniques such as ARPES, a lack of large and uniform sample still stands in the way of development of twistronics. Chemical vapor deposition, by which random twist angle and 30° twisted bilayer graphene quasicrystals have been achieved,[75–78] improved mechanical exfoliation method,[79,80] direct bonding,[81] and tip-assisted twist angle manipulation[3,74] are some potential solutions, yet technical challenges like angle control remain and more efforts are expected in the future.

6. The dream-line of the magic angle

The advent of twistronics has brought a lot of surprises in this research community and definitely it will serve as an arsenal for many appealing physical issues. The priority is to construct experiments and theories to reveal the nature of superconductivity and correlated insulating states in MA-TBG, which possibly paves a way to unveil the long-standing mystery of unconventional high temperature superconductivity.[82,83] Secondly, controlling the twist angle means more in twisted 2D materials. For example, adjusting the angle will provide us more space to explore 2D quasicrystal,[76,77,84,85] marginal twisted bilayer graphene,[86–88] crystal field,[89] and so on. Besides, more and more exotic phenomena such as topological physics,[90–92] superfluid weight,[93] hydrodynamics,[94] marginal Fermi liquid[95] as well as diverse material systems (TMDs, 2D magnetic materials,[96] and 2D superconductors) are expected to be involved into the family of twistronics. Last but not least, the field is flourishing now and it is a good time to get insight of the magic of angle!

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