Crystal and magnetic structure of magnetic topological insulators MnBi$_{2n}$Te$_{3n+1}$

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Using single crystal neutron diffraction, we present a systematic investigation of the crystal structure and magnetism of van der Waals topological insulators MnBi$_{2n}$Te$_{3n+1}$ ($n = 1$, 2), where emergent quantum phenomena have been recently observed. We show unambiguously that MnBi$_2$Te$_4$ orders antiferromagnetically below 24 K featured by a magnetic symmetry $R_2$-3c while MnBi$_4$Te$_7$ is antiferromagnetic below 13 K with a magnetic space group $Pc$-3c1. They both present antiferromagnetically coupled ferromagnetic layers with spins along the $c$-axis. We put forward a stacking rule for the crystal structure of an infinitely adaptive series MnBi$_{2n}$Te$_{3n+1}$ with the building unit of [Bi$_3$Te$_3$]. A comparison of magnetic properties between MnBi$_2$Te$_4$ and MnBi$_4$Te$_7$, together with the recent density-functional theory calculations, enables us to draw that the interlayer exchange interactions between Mn atoms are significantly suppressed with the increase of the value of $n$ and a two-dimensional magnetism limit might be realized in the derivatives. Our work may promote the theoretical prediction of exotic quantum states in the series of MnBi$_{2n}$Te$_{3n+1}$.

Introduction. In condensed matter physics, van der Waals (vdW) magnetic heterostructures stacked layer-by-layer in a controlled sequence have attracted a great deal of interest as they have been found to show exotic physical properties and emergent phenomena [1–4]. Novel properties in these materials can be controlled by adjusting the stacked atomic layers, paving the way for designing new quantum materials [1]. VdW magnetic topological insulators have been suggested as a promising material platform for the exploration of exotic topological quantum phenomena such as the quantum anomalous Hall effect (QAHE), Majorana fermions as well as topological magnetoelectric effect [4–7]. However, a homogenous heterostructure with intrinsic magnetism, an ideal platform for studying such topological quantum effects, is experimentally elusive [4].

Very recently, MnBi$_2$Te$_4$ was proposed to be the first intrinsic antiferromagnetic (AFM) topological insulator [7–17]. It has been shown that below 24 K, MnBi$_2$Te$_4$ orders into an A-type magnetic structure based on magnetic properties, density-functional theory calculations and powder neutron diffraction measurement [7, 12, 14, 18]. Since such a spin configuration breaks the product ($S$) of the time-reversal symmetry and the primitive-lattice translational symmetry at the (001) surface, it is expected that a gapped surface Dirac cone can be observed by the angle-resolved photoemission spectroscopy (ARPES) made on the cleaved (001) surface [5, 12]. However, both gapped and gapless [8, 11, 13, 15] Dirac cones have been observed in ARPES measurements. This casts doubt on the magnetic configuration determined using the powder neutron diffraction data [7]. Furthermore, a new family of MnBi$_{2n}$Te$_{3n+1}$ ($n = 2$, 3) were later discovered [19]. Among them, MnBi$_4$Te$_7$ [19–21] and MnBi$_6$Te$_{10}$ [19] have been suggested to be new magnetic topological insulators with weak interlayer magnetic coupling through a combined ARPES and first-principles calculations [20]. Although the A-type AFM was suggested for MnBi$_4$Te$_7$ by its anisotropic magnetic properties, no neutron experiment has been reported to prove it. Therefore, to unambiguously determine the magnetic structures of MnBi$_2$Te$_4$ and MnBi$_4$Te$_7$, single crystal neutron diffraction experiment, which can map out the complete magnetic reflections with a better resolution, is urged.

Beyond the investigation of the magnetic structures, motivated by the above mentioned discoveries, our work is devoted to setting up the relationship between crystal structure and magnetic properties by examining the simple lattice-stacking rule starting from the prototype topological insulator Bi$_2$Te$_3$ [23]. Bi$_2$Te$_3$ is a strong topological insulator with a single surface Dirac cone [24]. Conventionally, its structure is described by the stacking of three “quintuple layer” building blocks [22, 25]. The bond coupling is rather strong between two atomic layers within one quintuple layer but much weaker, predominantly of the vdW type, between neighboring quintuple layers. Yet, “quintuple layer” stacking description does not reflect the structural symmetry of Bi$_2$Te$_3$ that may be important in understanding the topological properties in MnBi$_{2n}$Te$_{3n+1}$. The proposed stacking rule for building magnetic topological insulator Mn-Bi-Te series in this work practically captures the structural symmetry, reflects the precise layer stacking sequence, and reveals an infinitely adaptive series, stimulating theoretical calculations of exotic quantum states in the series of MnBi$_{2n}$Te$_{3n+1}$.

Stacking rule and crystal structures of vdW MnBi$_{2n}$Te$_{3n+1}$. We examined the crystal structure of both powder and single crystalline MnBi$_2$Te$_4$ and MnBi$_4$Te$_7$ samples using x-ray and neutron diffraction techniques [26]. For MnBi$_2$Te$_4$, the slice view of neutron diffraction data at 30 K is shown in Fig.3(a) while the powder x-ray diffraction pattern is presented in [27]. For MnBi$_4$Te$_7$, Fig. 4 shows the contour map of neutron diffraction at 7 K, where the presence of sharp reflections indicates the high quality of the crystal. We have refined both the single crystal neutron diffraction data and x-ray powder diffraction data using Fullprof software [28]. The refined results as well as structural parameters are shown in Fig. 3(b) and Table S1-S2 [27], respectively. They are in good agreement with the previous report [29]. The refined lattice parameters are presented in Table I.
TABLE I. Derivatives of the vdW MnBi$_{2n}$Te$_{3n+1}$ topological insulators. Lattice parameters (i: experiment, ii: prediction) are shown at room temperature. The lattice parameters for each sequence of the stacking units are based on Bi$_2$Te$_3$: \(a=b=4.3896(2)\ \text{Å}, \ c=30.5019(10)\ \text{Å}\) [22]. When \(n=2+3m\) \((m = 0, 1, 2...)\), the lattice of derivatives is primitive. The predicted magnetic space groups (MSG) are made based on a symmetry analysis using the \(k\) vectors of \((0, 0, 3/2)\) for the rhombohedral lattice, \((0, 0, 1/2)\) for the hexagonal lattice, \((0, 0, 0)\) for the cases with ferromagnetic (FM) order.

| \(n\) | Sequence | SG     | Lattice parameter | Magnetic properties and MSG |
|------|----------|--------|-------------------|----------------------------|
| 1    | M-C-M-B-M-A-M | R-3m   | 4.336(2) Å 40.926(3) Å | AFM, \(R_t\)-3c | i, this work |
| 2    | A-M-C     | P-3m1  | 4.345(5) Å 23.705(3) Å | AFM, \(P_t\)-3c1 | i, this work |
| 3    | M-C-A-B-M-A-B-C-M-B-C-A-M | R-3m | 4.3745(3) Å 101.985(8) Å | AFM, \(R_t\)-3c or FM, R-3m | ii |
| 4    | M-C-A-B-M-B-C-A-M | R-3m | 101.94 Å | AFM, \(R_t\)-3c or FM, R-3m | ii |
| 5    | M-C-A-B-C-A-M | P-3m1 | 54.32 Å | AFM, \(P_t\)-3c1 or FM P-3m1 | ii |
| 6    | M-C-A-B-C-A-M-B-C-A-B-M-B-C-A-B-C-A-M | R-3m | 193.47 Å | AFM, \(R_t\)-3c or FM, R-3m | ii |

FIG. 1. Schematic drawings of the crystal structures of Bi$_2$Te$_3$ and MnBi$_{2n}$Te$_{3n+1}$ \((n = 1, 2)\). Note that for the latter cases, spin arrangements are denoted by red arrows.

Based on the crystal structure of MnBi$_2$Te$_4$, MnBi$_4$Te$_7$, as shown in Fig. 1, we can put forward a stacking rule of the crystal structure of this family that is based on the rhombohedral lattice as seen in the topological insulator Bi$_2$Te$_3$. The aforementioned structural description of the Bi$_2$Te$_3$ is based on the stacking of three “quintuple layer” building blocks. This description has been used to predict the infinitely adaptive series of thermoelectric materials [25, 30]. However, it seemingly does not capture the essential structural symmetry but rather simply depicts the stacking lattice. As shown in Fig. 1, here the crystal structure of Bi$_2$Te$_3$ is designed as A-B-C stacking sequence with A, B and C represent distinct [Bi$_2$Te$_3$] units. Imposed by the inversion and rhombohedral centering translation, the A unit can be progressed into B, and subsequently C configuration. Simply, we introduce magnetic atoms by inserting M (M denotes MT$_6$ octahedra layer) layers into this A-B-C stacking sequence, giving rise to an infinitely adaptive series MnBi$_{2n}$Te$_{3n+1}$. Accordingly, we can immediately name the sequence M-C-M-B-M-A-M with \(n = 1\) which should be represented by the crystal symmetry R-3m. This yields the compound MnBi$_2$Te$_4$, as illustrated in Fig. 1. When \(n = 2\), in other words, one more [Bi$_2$Te$_3$] unit is added, we get the stacking sequence A-M-C with the space group P-3m1. It corresponds to the compound MnBi$_4$Te$_7$, indicating a great compatibility between these structural units. It occurs that the B-type unit occurring in MnBi$_2$Te$_4$ disappears in MnBi$_4$Te$_7$. Consequently, this delivers a general stacking rule for a vast number of vdW magnets in this family: the magnetic Mn-layer can replace one of A, B or C-type unit but still leaves the -A-B-C- stacking sequence unchanged. Indeed, with the increment of the value of \(n \geq 1\), the sequence of the building units, crystal symmetry and lattice parameters of the corresponding vdW magnet can be generated and listed in Table I. Following this stacking rule, one can make an infinitely adaptive series as those listed in Table I (we only list up to \(n = 6\)).

**Thermodynamic properties of MnBi$_2$Te$_4$ and MnBi$_4$Te$_7$.** The ZFC temperature dependent magnetic susceptibilities of
MnBi$_2$Te$_4$ and MnBi$_4$Te$_7$ measured with the field parallel to the c-axis are shown in Fig. 2. For the MnBi$_2$Te$_4$ case, the susceptibility first increases with decreasing temperature and exhibits a sharp cusp at $T_N=24\,\text{K}$, signaling the AFM ordering. In order to further characterize the magnetic phase transition, we measured the specific heat of MnBi$_2$Te$_4$, shown in Fig. 2(b). The cusp at 24 K is indicative of the AFM transition, in good agreement with the magnetic susceptibility data. Thus MnBi$_2$Te$_4$ only undergoes one AFM transition at 24 K. The magnetic susceptibility of MnBi$_4$Te$_7$ shows a sharp peak at $T_N=13\,\text{K}$ (Fig. 2(c)), indicating an AFM transition. The nature of the transition can be crosschecked by the specific heat data of MnBi$_4$Te$_7$. As shown in Fig. 2(d), the specific heat shows a small anomaly at 13 K, consistent with the magnetic susceptibility results and the previous results.[20, 21] The Neél temperature of MnBi$_4$Te$_7$ is about two times smaller than that of MnBi$_2$Te$_4$.

**A-type magnetic structure of MnBi$_2$Te$_4$ and MnBi$_4$Te$_7**. The magnetic structure of MnBi$_2$Te$_4$ has been investigated previously using powder neutron diffraction [18]. They found that the AFM structure has the magnetic symmetry $P_2$-3c1 (BNS symbol) propagated by a vector $\mathbf{k}=(0, 0, 1/2)$. Our neutron diffraction experiment on the crystal from 7 K to 30 K reveals a different magnetic symmetry characterized by a different propagation vector $\mathbf{k}'=(0, 0, 3/2)$. As shown in Fig. 3(a), comparing the neutron diffraction data at 7 K and 30 K, a set of magnetic reflections appears at 7 K. They can be indexed by the vector we found. As a matter of fact, the magnetic reflections shown in Ref. [18] should be reasonably indexed by $\mathbf{k}'=(0, 0, 3/2)$, implying that the proposed magnetic space group should be reconsidered here. To solve the magnetic structure of MnBi$_2$Te$_4$, we have carried out the magnetic symmetry analysis by considering the $\mathbf{k}$ vector we found and the parent space group $R-3m1'$ with the help of the ISODISTORT software [31]. There are two active magnetic irreducible representations, $\text{mT}2^+$ and $\text{mT}3^+$. After testing these candidates, we found the irrep $\text{mT}2^+$, corresponding to the magnetic space group $Rf$-3c, is the best solution. The Rietveld refinement of the magnetic neutron diffraction data at 30 K provides the scale factor that was taken in the magnetic structure refinement. Magnetic neutron diffraction data were then refined using the magnetic symmetry $Rf$-3c and the results of the refinement are shown in Fig. 3(b). We selected four magnetic reflections that are quite distinguishable from each other for the final magnetic structure refinement. This should not cast any doubt as only one parameter was refined for the basis vector. We found that spins align ferromagnetically in the $ab$ plane below 24 K whereas between layers, magnetic moments are antiparallel. Note the spin arrangement is consistent with the reported one despite different propagation vectors [18]. The refined magnetic moment of Mn$^{2+}$ at 7 K is 4.2(5)$\mu_B$, relatively smaller than the expected totally ordered moment 5$\mu_B$ for Mn$^{2+}$ ion.

Single crystal neutron diffraction data of MnBi$_4$Te$_7$ show clearly the appearance of magnetic reflections at the (H 0 L+0.5) positions (H, L denote the Miller index) upon cooling below 13 K, confirming the formation of a long-range AFM order. As shown in Fig. 4 all magnetic reflections can be indexed by the propagation vector $\mathbf{k}=(0, 0, 1/2)$. Starting with the parent space-group $P-3m1'$ and the propagation vector $\mathbf{k}$ in A point in the Brillouin zone, through ISODISTORT, two active magnetic irreducible representations, mAl- and mA3- were obtained. We found that magnetic space group $P_r$-3c1 (BNS symbol, basis=(1,0,0),(0,1,0),(0,0,2), origin=(0,0,1/2)), generated from the single active mA1- irreducible representation, can be adopted to describe the magnetic structure. The scale factor was refined by taking a structural model from the x-ray diffraction. Ultimately, the refined magnetic structure

![FIG. 3. (a) Contour map of neutron intensity of MnBi$_2$Te$_4$ in the (H0L) reciprocal plane at 30 K and 7 K. (b) The results for the nuclear and magnetic structure refinements. $\chi^2_{\text{nucl}}=0.85, \chi^2_{\text{mag}}=3.51$.](image-url)
demonstrates an AFM arrangement between the adjacent FM layers, as shown in Fig. 1. The refined total magnetic moment at 7 K is 4.3(1)μB along the c-axis, slightly smaller than the value of 5 μB expected for S = 5/2 of Mn⁵⁺. It appears that MnBi₄Te₇ bears a similar spin arrangement between layers (different magnetic symmetry) to MnBi₃Te₄ even though it orders at a significantly lower temperature.

Having known the magnetic structures of the vdW magnets with n = 1, 2, we now examine the temperature dependence of the strongest magnetic reflections for each case. The temperature-dependent intensity of the magnetic reflections (0 -1 -0.5) and (1 0 0.5) for MnBi₂Te₄ (upper panel) and MnBi₄Te₇ (lower panel), respectively are shown in Fig. 5. They may be fit to an empirical power-law behavior [32, 33],

$$I = A \left( \frac{T_M - T}{T_M} \right)^{2\beta} + B$$  \hspace{1cm} (1)

gives Neél temperatures of T₈=24.2(1)K and 13.08(6)K for MnBi₂Te₄ and MnBi₄Te₇. This estimate is in good agreement with the thermodynamic results shown in the previous section. The fit for MnBi₂Te₄ yields the critical exponent β=0.31(3), a value relatively smaller than the results in Ref.[18]. This β value is very close to the value, 0.325, expected for a universality class of the three-dimensional Ising model[32]. Unexpectedly, we found that the β=0.34(2) of MnBi₄Te₇ is slightly larger than that of MnBi₂Te₄. This seems to be contradictory to the less three-dimensional magnetism in MnBi₄Te₇ caused by the much larger interlayer Mn-Mn distance. The reason may lie in the fact that the fitting for the MnBi₂Te₇ case was done in a temperature range that is relatively close to the critical region which often comes with a crossover from three-dimensional to two-dimensional behavior upon cooling [33, 34].

Magnetism of MnBi₂₃Te₃ₙ₋₁ derivatives. Recently, theoretical calculations on MnBi₂Te₄ [12] have shown that in the (ab) plane the FM interaction between the first nearest neighbors J₁=1.693 meV strongly dominates over all others. By considering all the exchange interactions in the layer and magnetic anisotropy energy, they suggest the FM ordering temperature of T₈=12(1)K in a single free-standing MnBi₂Te₄ septuple layer [12]. Interestingly, MnBi₄Te₇ orders antiferromagnetically at 13 K, very close to the aforementioned 12(1) K. This indicates that in MnBi₄Te₇ intralayer exchange interaction is robust whereas the interlayer one is minimal. Indeed, recent calculations on MnBi₂Te₄ yielded that J₁ and J₂, (a summed exchange coupling between the adjacent layers) [20] are 1.704 meV and -0.150 meV, respectively. With the increase of non-magnetic building units, FM could be realized by quenching all interlayer exchange interactions. Therefore, for MnBi₂₃Te₃ₙ₋₁ with n>2, if we assume their magnetic ground state to be either A-type AFM or FM with all magnetic moments being aligned along the c-axis, the magnetic space group for each compound is predicted and presented in Table I. Experiments to characterize these derivatives and to ascertain the predicted magnetic space groups are called.

Conclusion. In summary, we have investigated systematically the crystal structure and magnetism of vdW topological insulators MnBi₂₃Te₃ₙ₋₁. We have revealed a simple lattice-stacking rule that can lead to an infinitely adaptive series of MnBi₂₃Te₃ₙ₋₁. Although different magnetic symmetry exists in MnBi₂Te₄ and MnBi₄Te₇ with the former being R₂₃c and the latter being P₂₋₃c₁, our single crystal neutron diffraction measurements have unambiguously established that in the ordered state, both compounds show the A-type AFM structure with all spins parallel in the ab plane but antiparallel along the c axis which is critical for the observation of quantized anomalous Hall effect when they are exfoliated down to a few layers.

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