Glassy magnetic ground state in layered compound MnSb_2Te_4

Hao Li^{1,6}, Yaixin Li³, Yukun Lian⁴, Weiwei Xie⁵, Ling Chen⁴, Jinsong Zhang^{3,7}, Yang Wu^{2,6*} and Shoushan Fan^{3,6}

ABSTRACT As a sister compound of MnBi_2Te_4 , the high-quality MnSb_2Te_4 single crystals are grown *via* solid-state reaction where prolonged annealing and narrow temperature window play critical roles on account of its thermal metastability. Single-crystal X-ray diffraction (SCXRD) analysis on MnSb_2Te_4 illustrates a crystal model that is isostructural to MnBi_2Te_4 , consisting of Te-Sb-Te-Mn-Te-Sb-Te septuple layers (SLs) stacking in an ABC sequence. However, MnSb_2Te_4 reveals a more pronounced cation intermixing in comparison with MnBi_2Te_4 , comprising 28.9(7)% Sb antisite defects on the Mn (3a) site and 19.3(6)% Mn antisite defects on the Sb (6c) site, which may give rise to novel magnetic properties in emerging layered MnBi_2Te_4 -family materials. Unlike the antiferromagnetic (AFM) nature in MnBi_2Te_4 , MnSb_2Te_4 exhibits a glassy magnetic ground state below 24 K and can be easily tuned to a ferromagnetic state under a weak applied magnetic field. Its magnetic hysteresis, anisotropy, and relaxation process are investigated in detail *via* static and dynamic magnetization measurements. Moreover, anomalous Hall effect as a p-type conductor is demonstrated with transport measurements. This work grants MnSb_2Te_4 a possible access to the future exploration of exotic quantum physics by removing the odd/even layer number restraint in realizing quantum transport phenomena in intrinsic AFM MnBi_2Te_4 -family materials, as a result of the crossover between its magnetism and potential topology arising from the Sb-Te layer.

Keywords: MnSb_2Te_4 , crystal growth, glassy magnetic ground state, anomalous Hall effect

INTRODUCTION

The pursuit of emerging topological materials that cultivate exotic quantum phenomena, for instance, dissipationless electronics transport and topological quantum computation, has been a research frontier in condensed matter physics and materials science [1–3]. In this regard, magnetic topological quantum materials (MTQMs) can be one of the most fascinating examples, owing to their interplay of nontrivial topology and

magnetism that could give rise to numerous transport phenomena, such as the quantum anomalous Hall (QAH) effect and the axion insulator state [3–11]. One of the important examples of MTQMs is the Cr-doped $(\text{Bi}_x\text{Sb}_{1-x})_2\text{Te}_3$ thin film fabricated in a molecular epitaxy process that hosts the QAH effect [7]. However, subsequent studies on magnetic topological insulators (TIs) revealed that doping with magnetic impurity was a fairly challenging strategy in realizing homogeneous distribution in materials, restricting significant improvement in observing quantized conductance in high temperatures, and hindering the experimental progress and further application [8–13]. Therefore, intrinsic MTQMs with naturally ordered magnetic-topological interplays could serve as a cornerstone for the future discovery of numerous emerging topological properties in materials [14].

Recently, MnBi_2Te_4 , a representative ternary chalcogenide with a general formula of MB_2T_4 (M = transition-metal or rare-earth element, B = Bi or Sb, T = Te, Se, or S), has been identified to be the first intrinsic magnetic TI by both theoretical and experimental efforts [14–32]. As a van der Waals layered compound with a tetradymite-derived structure, MnBi_2Te_4 (space group $R\bar{3}m$) consists of Te-Bi-Te-Mn-Te-Bi-Te septuple layers (SLs) stacking in an ABC sequence. Appealingly, magnetic order and nontrivial topology inherently coexist in MnBi_2Te_4 and it belongs to MTQMs. On the one hand, each Mn^{2+} offers a magnetic moment of $5 \mu_B$ according to the Hund's rule in high-spin configuration. The intralayer exchange coupling between Mn-Mn is ferromagnetic (FM) with an out-of-plane easy axis, while the interlayer coupling between neighboring SLs is antiferromagnetic (AFM), corresponding to an A-type AFM order. On the other hand, the Bi-Te layers could generate topological states by their strong spin-orbit coupling. The magnetic and topological properties of MnBi_2Te_4 together generate a gapped surface state by symmetry-breaking on the (0001) surface, confirmed experimentally on the high-quality MnBi_2Te_4 single crystal [21]. Moreover, MnBi_2Te_4 revealed layer-number-dependent topological phase evolution, where the long-sought axion insulator phase and QAH effect have been realized in mechanically exfoliated six-SL and five-SL MnBi_2Te_4 films, respectively [31,32]. The thriving research on MnBi_2Te_4 not only contributes to the emerging topological quantum states but also

¹ School of Materials Science and Engineering, Tsinghua University, Beijing 100084, China

² Department of Mechanical Engineering, Tsinghua University, Beijing 100084, China

³ State Key Laboratory of Low Dimensional Quantum Physics and Department of Physics, Tsinghua University, Beijing 100084, China

⁴ Beijing Key Laboratory of Energy Conversion and Storage Materials, College of Chemistry, Beijing Normal University, Beijing 100875, China

⁵ Department of Chemistry and Chemical Biology, Rutgers University, Piscataway 08854, USA

⁶ Tsinghua-Foxconn Nanotechnology Research Center, Tsinghua University, Beijing 100084, China

⁷ Frontier Science Center for Quantum Information, Beijing 100084, China

* Corresponding author (email: wuyangthu@mail.tsinghua.edu.cn)

motivates the synthesis of potential MTQMs. The great variety of unexplored MB_2T_4 family is highly possible to generate tremendous novel topological quantum phases and thus arouse further interests in quantum physics.

Herein, we report the growth of MnSb_2Te_4 single crystals, a typical member of the MB_2T_4 -family materials, and establish its property in terms of thorough characterizations of the structure, composition, thermal and magnetic analysis. Interestingly, MnSb_2Te_4 single crystal exhibits glassy behaviors below the freezing temperature $T_f \sim 24$ K, different from the predicted and experimentally revealed A-type AFM state MnBi_2Te_4 , or FM interaction reported in polycrystalline MnSb_2Te_4 [33–35]. In fact, the considerable net magnetic moment is observed in MnSb_2Te_4 single crystals below its magnetic transition temperature, which makes it a promising candidate to realize intriguing quantized conductance in zero-magnetic-field, without the restraint of layer number dependence in AFM MnBi_2Te_4 [14].

EXPERIMENTAL SECTION

Materials

Sb shot (99.99%, Alfa Aesar), Mn pieces (99.95%, Alfa Aesar), and Te lump (99.999%, Aladdin) were used as received.

Synthesis

MnTe precursor was synthesized by directly heating a stoichiometric mixture of high-purity Mn (99.95%, Alfa Aesar) and Te (99.999%, Aladdin) at 1273 K in a vacuum-sealed silica ampoule for three days. The ampoule was quenched in the air to obtain the MnTe precursor (Fig. S1b). Sb_2Te_3 precursor was synthesized by a solid-state reaction of a stoichiometric mixture of high-purity Sb (99.99%, Alfa Aesar) and Te (99.999%, Aladdin). The reaction mixture was vacuum-sealed in a silica ampoule, and heated to 1073 K and held for 24 h. After being slowly cooled to 863 K at a rate of 0.1 K min^{-1} and held for 48 h, the ampoule was quenched in the air to obtain the Sb_2Te_3 precursor (Fig. S1a).

Characterization

Morphology and element analyses were carried out using an FEI NOVA SEM450 scanning electron microscope (SEM) equipped with an energy dispersive X-ray (EDX) detector. X-ray photoelectron spectroscopy (XPS) measurements were conducted using an Ulvac-Phi Quantera II X-ray photoelectron spectrometer with monochromatic Al K α radiation (1486.6 eV). The C 1s peak at 284.8 eV was used as the reference. Single-crystal X-ray diffraction (SCXRD) data were collected at 298 K on a Bruker D8 Quest diffractometer using monochromatic Mo K α radiation ($\lambda = 0.71073 \text{ \AA}$). The absorption correction was done by the multiscan method. The structure was solved by SHELXT and refined by SHELXL in OLEX2 [36–38]. Powder XRD (PXRD) patterns were collected on a Rigaku D/max-2500/PC X-ray diffractometer, using Cu K α radiation and operating at 40 kV and 200 mA. Raman spectra were collected on a Horiba Jobin Yvon LabRam-HR/VV Spectrometer with a 514-nm laser source and an 1800-line grating. Differential thermal analysis (DTA) and thermogravimetric (TG) analysis were carried out on a NETZSCH STA449 F3 simultaneous thermal analyzer under N_2 atmosphere. The MnSb_2Te_4 sample was enclosed in an Al_2O_3 crucible, and heated from room temperature to 973 K at a rate of

10 K min^{-1} and then cooled to 673 K at a rate of 10 K min^{-1} .

Magnetic measurement

Magnetic property measurements were carried out using a magnetic property measurement system (MPMS SQUID VSM Quantum Design). Dynamic (AC) magnetization measurements were performed with an oscillating field, which can be written as

$$H(t) = H_{\text{DC}} + H_{\text{AC}} \cos(\omega t),$$

where t is the time, ω is the angular frequency, H_{DC} is the magnitude of static (DC) field, and H_{AC} is the amplitude of oscillating field [39,40]. The magnetization response of a measured material can be expressed as

$$M(t) = M_{\text{DC}} + M_{\text{AC}} \cos(\omega t - \phi),$$

where ϕ is a phase shift due to the magnetic relaxation. AC susceptibility can be defined as

$$\chi = \chi' - i\chi'',$$

$$\chi' = M_{\text{AC}} \cos(\phi) / H_{\text{DC}},$$

$$\chi'' = M_{\text{AC}} \sin(\phi) / H_{\text{DC}},$$

where the real term (χ') keeps in-phase with the AC field, and the imaginary term (χ'') is out-of-phase with the AC field and is a measure of the energy losses due to magnetic relaxation.

For a single relaxation time (τ), AC susceptibility can be described by the Debye model as

$$\chi(\omega) = \chi + (\chi_0 - \chi_\infty) / (1 + i\omega\tau),$$

where χ_0 and χ_∞ are the susceptibility at zero frequency and extremely large frequency, respectively. In actual magnetic materials, there is a distribution for τ , and a generalized Debye model:

$$\chi(\omega) = \chi + (\chi_0 - \chi_\infty) / (1 + i\omega\tau)^{(1-\alpha)},$$

where α is the distribution parameter for τ , is used to describe the χ'' - χ' plot (Cole-Cole plot). The derived parameters α and τ provide specific features of a magnetic material [39–42].

Transport measurement

Transport property measurements were carried out using a cryostat (Oxford Instruments) with a base temperature of ~ 1.6 K and a magnetic field up to 2 T. The longitudinal and Hall voltages were detected simultaneously by using Stanford Research Instrument SR830 lock-in amplifiers with an AC current generated with a Keithley 6221 current source. The MnSb_2Te_4 single crystal sample with a thickness of $\sim 35 \mu\text{m}$ was obtained by mechanical exfoliation from a raw MnSb_2Te_4 single crystal by Scotch tape.

RESULTS AND DISCUSSION

Crystal growth

Learning from the thermal stability and synthetic strategy of MnBi_2Te_4 crystal [29], we adopted a similar growth strategy with extended annealing for the crystal growth of MnSb_2Te_4 , predicted to be isostructural to MnBi_2Te_4 . Firstly, Sb_2Te_3 and MnTe binary used as precursors were prepared through direct reactions of high-purity Sb and Te, and Mn and Te in stoichiometric mixtures, respectively (EXPERIMENTAL SECTION). Hereafter, high-quality MnSb_2Te_4 single crystals well grew from a 1:1 mixture of Sb_2Te_3 and MnTe . The vacuum-sealed sample was

heated to 1173 K in 9 h and slowly cooled to 893 K at a rate of 1 K min^{-1} , followed by prolonged annealing at 893 K for at least 14 days. Finally, the sample was quenched in the air and cracked to pick shiny crystals by cautious selection. To remove impurities, coarse parts of crystals were carefully cut off using a scalpel or cleaved off by scotch tape under an optical microscope. The growth route can successfully afford millimeter-size MnSb_2Te_4 single crystals (Figs 1a and 2b).

Composition and oxidation state analysis

To get rid of the influence from ambient contamination, we prepared a fresh surface of MnSb_2Te_4 crystals by cleaving them with scotch tape. The SEM image of the exfoliated MnSb_2Te_4 crystal shows a smooth surface (Fig. 1a). Distinctive edges between MnSb_2Te_4 layers of different thicknesses can be observed, confirming it is a van der Waals layered compound. Therefore, it is reasonable to obtain high-quality few-SL MnSb_2Te_4 flakes *via* mechanical exfoliation. EDX spectroscopy was further adopted to analyze the composition. The EDX spectrum (Fig. S2a) displays no peaks other than Sb, Mn, Te, and C (from carbon tape). In addition, the corresponding EDX elemental mapping images in Fig. 1b–d clearly show a uniform elemental distribution of Sb, Mn, and Te. Quantification based on the EDX spectrum using Sb L, Te L, and Mn K edges gives the composition as Mn 15.6, Sb 26.9, Te 57.4 at%, corresponding to the chemical formula of $\text{Mn}_{1.1}\text{Sb}_{1.9}\text{Te}_{4.0}$.

XPS measurements were carried out on the exfoliated fresh surface of MnSb_2Te_4 crystal to determine the oxidation states. The XPS survey spectrum (Fig. S2b) consolidates the constituent elements as Sb, Mn, and Te. In the high-resolution Sb 3d spectrum in Fig. 1e, two major peaks at 537.7 eV (Sb 3d_{3/2}) and 528.3 eV (Sb 3d_{5/2}) can be assigned to Sb³⁺ in Sb–Te bonds [43,44]. Two shoulder peaks at 539.0 and 530.0 eV corresponding to Sb–O bonds imply the slight oxidation due to the short exposure to air [43,44]. Fig. 1f shows the deconvoluted Mn 2p spectrum that consists of two sets of four sub peaks (Mn1('), Mn2('), Mn3('), and Mn4(')). Mn1 and Mn1' peaks located at 639.9 and 651.4 eV could be attributed to broken Mn–Te bonds by Te deficiency. The strongest Mn2 and Mn2' peaks at 641.0 and 653.1 eV define the major oxidation state of Mn as 2+. Mn3

and Mn3' peaks at 642.5 and 654.8 eV, and Mn4 and Mn4' peaks at 645.0 and 657.9 eV are satellite peaks of Mn1 and Mn1', and Mn2 and Mn2', respectively, which result from the charge transfer between the unfilled 3d shell of Mn and the outer shell of Te in the photoelectron process, similar to MnBi_2Te_4 [45–48]. For the Te 3d spectrum in Fig. 1g, it contains two peaks at 572.0 eV (Te 3d_{5/2}) and 582.4 eV (Te 3d_{3/2}) that can be assigned to Te^{2–} in Mn–Te and Sb–Te (Mn/Sb–Te) bonds [43,44]. To gain insight into its oxidation behavior, the MnSb_2Te_4 crystal was exposed to air for more than a week and investigated using XPS. As shown in Fig. S3, the Sb–O and Te–O signals turn out to be evidently higher than Sb–Te and Mn/Sb–Te peaks in the high-resolution Sb 3d and Te 3d spectra, indicating serious surface oxidation that MnSb_2Te_4 suffers in air condition. On this account, it is necessary to keep MnSb_2Te_4 samples, especially the few-layer ones, in an inert atmosphere to avoid damage by oxygen.

Crystal structure and refinement

The crystal structure of the as-grown MnSb_2Te_4 crystal was elucidated *via* SCXRD measurement. As an isostructural compound to MnBi_2Te_4 , MnSb_2Te_4 also crystallizes in the centrosymmetric space group $R\bar{3}m$ (No. 166) featuring the Te–Sb–Te–Mn–Te–Sb–Te SLs stacking in an ABC sequence along the *c*-axis (Fig. 2a and Fig. S4). Table 1 lists the lattice parameters of MnSb_2Te_4 , $a = 4.2284(5)\text{ \AA}$ and $c = 40.705(1)\text{ \AA}$, slightly smaller than those of MnBi_2Te_4 . This is reasonable taking the smaller ionic radius of Sb than that of Bi into account. Besides, cation antisite disorder is ubiquitous in MB_2T_4 -type compounds [15,35,49]. For this reason, antisite mixing of Sb and Mn was taken into account in the structural refinement by allowing Sb (6c) to occupy Mn (3a) site and vice versa. Te vacancies were only allowed in Te2 position that occupied the outermost layer in the Te–Sb–Te–Mn–Te–Sb–Te SLs, because those Te atoms are easier to lose considering the weak van der Waals bonding in MnSb_2Te_4 . Charge neutrality was kept in the structure refinement under the restraint of eight positive charges per formula unit by considering $\text{Mn}^{\text{II}}(\text{Sb}^{\text{III}})_2(\text{Te}^{\text{II}})_4\text{T}$ oxidation states according to XPS results.

The refinement generated a nonstoichiometric model con-

Figure 1 Morphology and elemental analyses of the exfoliated MnSb_2Te_4 single crystals. (a) SEM and (b–d) EDX elemental mapping images (Sb, Mn, and Te). (e) Sb 3d, (f) Mn 2p, and (g) Te 3d high-resolution XPS spectra.

Figure 2 (a) Crystal structure of the $\text{Mn}_{1.09(7)}\text{Sb}_{1.90(3)}\text{Te}_{3.95(1)}$ model with cation intermixing. The unit cell is marked with black lines. (b) PXRD patterns of MnSb_2Te_4 single crystal (top) and powder (middle). Inset: An optical image of the as-grown MnSb_2Te_4 single crystal, and the scale bar is 2 mm. (c) Raman spectra of MnSb_2Te_4 and Sb_2Te_3 . (d) DTA and TG analysis curves of MnSb_2Te_4 .

Table 1 Crystallographic data of $\text{Mn}_{1.09(7)}\text{Sb}_{1.90(3)}\text{Te}_{3.95(1)}$, refined with cation intermixing and Te vacancies from SCXRD

Empirical formula	$\text{Mn}_{1.09(7)}\text{Sb}_{1.90(3)}\text{Te}_{3.95(1)}$
Formula units	$Z = 3$
Crystal system, space group	Trigonal, $R\bar{3}m$ (no. 166)
Lattice parameters	$a = 4.2284(5) \text{ \AA}$ $c = 40.705(10) \text{ \AA}$ $V = 630.3(2) \text{ \AA}^3$
Temperature for SCXRD	304.36 K
The range for data collection	$6.006^\circ \leq 2\theta \leq 60.982^\circ$ ($\lambda = 0.71073 \text{ \AA}$)
Index ranges	$-5 \leq h \leq 6, -6 \leq k \leq 6,$ $-57 \leq l \leq 57$
Collected reflections	3406 measured, 286 unique
R indexes of merging	$R_{\text{int}} = 0.1012, R_\sigma = 0.0364$
Data/restraints/parameters	286/1/15
Final R indexes [$I > 2\sigma(I)$]	$R_1 = 0.0526, wR_2 = 0.1346$
Final R indexes (all data)	$R_1 = 0.0564, wR_2 = 0.1386$
Goodness-of-fit on F^2	1.130
Min./max. residual electron density	$-1.96/1.99 \text{ e \AA}^{-3}$

sisting of 28.9(7)% Sb occupancy on the Mn (3a) site, 19.3(6)% Mn occupancy on the Sb (6c) site, and 2.4(5)% void defects on the Te2 (6c) site (Table 2). The composition that amounts to $\text{Mn}_{1.09(7)}\text{Sb}_{1.90(3)}\text{Te}_{3.95(1)}$ is in good consistent with the SEM-EDX quantification result of $\text{Mn}_{1.1}\text{Sb}_{1.9}\text{Te}_{4.0}$. The full structural information is shown in Tables 1 and 2, and Tables S1 and S2 in the Supplementary information (SI). For the sake of convenience, the formula MnSb_2Te_4 instead of $\text{Mn}_{1.09(7)}\text{Sb}_{1.90(3)}\text{Te}_{3.95(1)}$ is still used throughout the article. For

Table 2 Crystallographic data of $\text{Mn}_{1.09(7)}\text{Sb}_{1.90(3)}\text{Te}_{3.95(1)}$, refined with a cation intermixing model from an SCXRD experiment

Atom	x	y	z	SOF	U_{eq} (pm^2)
Te1 6c	2/3	1/3	0.04089(3)	1	189(4)
Te2 6c	0	0	0.13122(3)	0.975(5)	188(4)
Sb1 6c	1/3	2/3	0.09149(4)	0.806(4)	216(5)
Sb2 3a	0	0	0	0.289(7)	220(5)
Mn1 6c	1/3	2/3	0.09149(4)	0.193(6)	216(5)
Mn2 3a	0	0	0	0.710(3)	220(8)

comparison, the cation intermixing is in a much lighter extent in MnBi_2Te_4 prepared using the similar prolonged annealing method, which just comprises 16.0(2)% Bi in the Mn (3a) site (see Tables S3–S5 in the SI). Please note that MnBi_2Te_4 crystals grown by slow cooling within a narrow range exhibit similar antisite defects on the Mn 3a site (21.5(1)% Bi, 73.6(1)% Mn, and 4.9(1)% voids) and 5.7(1)% of Mn mixing on the Bi (6c) site [15]. Therefore, MnSb_2Te_4 affords much more pronounced cation antisite disordering than MnBi_2Te_4 , and this may induce new magnetism distinct to AFM coupling.

The PXRD pattern of raw MnSb_2Te_4 single crystal in Fig. 2a displays sharp and intense peaks that follow the (0 0 l), $l = 3n$, diffraction rule, indicating the rhombohedral symmetry and a strongly preferred orientation along the [0 0 1] direction. As displayed in Fig. 2b, the PXRD pattern of ground MnSb_2Te_4 powder is in good agreement with the one calculated from the structure model of $\text{Mn}_{1.09(7)}\text{Sb}_{1.90(3)}\text{Te}_{3.95(1)}$. No impurity peaks are observed and sharp and intense peaks are displayed, confirming the high purity and crystallinity of the as-grown MnSb_2Te_4 single crystal. The Raman spectrum of MnSb_2Te_4 crystal in Fig. 2c shows an appreciable blue shift and weaker A_{1g} peaks associated with Sb_2Te_3 , which might indicate some impurities that cannot be detected by PXRD [50,51].

Thermal stability

The thermal property of MnSb_2Te_4 was revealed using DTA. As shown in Fig. 2d, the DTA curves display an intense exothermic peak at 918 K on the heating trace and an endothermic peak at 907 K on the cooling trace, corresponding to the melting and crystallization of MnSb_2Te_4 respectively. The lower intensity of the endothermic peak at 918 K compared with that of the exothermic peak at 907 K implies that the crystallization of MnSb_2Te_4 is slow and difficult. An exothermic peak at 892 K on the heating trace and an endothermic peak at 878 K on the cooling trace can be ascribed to the melting and crystallization of Sb_2Te_3 respectively, revealing that MnSb_2Te_4 might be partly decomposed to Sb_2Te_3 during the heating. The exothermic peak at 892 K is considerably low while the endothermic peak at 878 K is stronger, suggesting that the decomposition of MnSb_2Te_4 proceeds notably faster upon the melting of Sb_2Te_3 and MnSb_2Te_4 . The weight loss in the TG curve in Fig. 2d could arise from the volatilization of Sb or Te. Accordingly, the synthesis of MnSb_2Te_4 single crystals requires strict control of the annealing temperature, balancing adequate energy for the reacting MnTe and Sb_2Te_3 and avoiding the severe decomposition of MnSb_2Te_4 .

Magnetic and transport properties

To check the magnetic characteristics of the as-grown MnSb_2Te_4 single crystal, DC magnetization measurements were performed. Fig. 3a and b show the temperature dependence of magnetic susceptibility (χ) for the MnSb_2Te_4 single crystal in out-of-plane ($H//c$) and in-plane ($H//ab$) magnetic fields, respectively. As shown in Fig. 3a, the paramagnetic (PM) regime follows Curie-Weiss law:

$$\chi(T) = \chi_0 + C / (T - \theta_{\text{CW}}),$$

where χ_0 includes core diamagnetism and Pauli paramagnetism. The Curie-Weiss fitting (70–250 K region) yields the effective magnetic moment of μ_{eff} of 5.4 μ_{B} by taking the relationship $C = N_A \mu_{\text{eff}}^2 / 3k_{\text{B}}$ into account, in good consistency with the value for the high-spin state of Mn^{2+} (5.92 μ_{B}). At a small field of 0.01 T, a large bifurcation between zero-field-cooled (ZFC) and field-cooled (FC) curves and lack of a cusp in the ZFC curve in both $H//c$ and $H//ab$ are indicative of a disordered glassy magnetic ground state below a freezing temperature $T_f \sim 24$ K [41,52,53]. At higher fields of 0.1 T for $H//c$ and 2 T for $H//ab$, ZFC and FC curves coincide, indicating it enters an FM state. The larger magnetic susceptibility in the low temperature region (5–30 K) in $H//c$ than that in $H//ab$ suggests a strong magnetic anisotropy and the magnetization-easy axis along the c direction in MnSb_2Te_4 . The magnetic anisotropy is further revealed in the field dependence of magnetization in Fig. 3c–d. The M - H curves in $H//c$ (Fig. 3c) display obvious hysteresis loop below 20 K featured by a remanent magnetization of about 0.001 μ_{B}/Mn and a coercive field of about 0.01 T at 5 K, consistent with the magnetic transition below ~ 24 K. Nevertheless, the linear M - H curves in $H//c$ above 30 K indicate the PM state. The M - H curves at low fields in $H//ab$ (Fig. 3d) display linear dependence with no hysteresis, suggesting that the magnetization along the ab plane is merely due to the field-bent magnetic moments originally sited along the c direction. Since DC magnetization data indicate the glassy spin system of MnSb_2Te_4 , the magnetic relaxation was investigated to further reveal the magnetic properties of MnSb_2Te_4 . Time-dependent magnetization curves in $H//c$ are

Figure 3 DC magnetization of MnSb_2Te_4 single crystal. (a, b) Temperature dependence of magnetic susceptibility measured at different fields following ZFC and FC processes, and inverse magnetic susceptibility as a function of temperature (top x axis and right y axis). The yellow line represents the Curie-Weiss fitting. The magnetic field is applied in out-of-plane (a) and in-plane (b) directions, respectively. (c, d) Field dependence of magnetization measured at different temperatures, and χ' and χ'' correspond to the real and imaginary part of AC magnetic susceptibility. The magnetic field is out-of-plane (c) and in-plane (d), respectively.

shown in Fig. 4a and b. In both ZFC and FC processes, magnetization continues to vary over minute-long periods. However, when the field is just increased to 0.1 T, magnetization in ZFC process shows no relaxation, demonstrating that MnSb_2Te_4 transforms into a field-induced FM state. Fig. 4c and d display the variation of the real (χ') and imaginary (χ'') part of dynamic (AC) magnetic susceptibility at different frequencies as a function of temperature in $H//c$ and $H//ab$, respectively. For $H//c$, the appearance of χ'' is a clear indication of the magnetic relaxation, and the peaks of $\chi'(T)$ and $\chi''(T)$ shift toward higher temperature as the frequency (f) increases, similar to the typical behavior of a classical spin glass state [39–41,52]. Fig. S5a displays the shift of χ'' peaks toward lower temperatures at a larger AC driving field of 10 Oe with respect to those at an AC driving field of 5 Oe (Fig. 3c), illustrating that the magnetic relaxation becomes faster at a larger AC driving field and is sensitive to the amplitude of AC driving field. As shown in Fig. S5b–d, χ'' is around zero and χ' becomes frequency-independent at a DC driving field higher than 600 Oe, indicating the disappearance of magnetic relaxation. As a result, the magnetism in MnSb_2Te_4 can be turned into a field-induced FM state with a DC driving field up to 600 Oe as affirmed by peaks in χ' , consistent with the DC magnetization results (Figs 2a and 3a). Furthermore, as shown in Fig. S6, by fitting the frequency (f) dependence of the peak shift in χ' using $K = \Delta T_f / (T_f \Delta \log f)$, K is calculated to be 0.005 at $H_{AC} = 5$ Oe and 0.007 at $H_{AC} = 10$ Oe, in good agreement with the values ($0.0045 \leq K \leq 0.08$) found in canonical spin glass systems [52]. For $H//ab$, $\chi''(T)$ approximates zero and peaks of $\chi'(T)$ show no frequency-dependence, illustrating that the magnetic relaxation is weak in the in-plane direction. That is to say, the magnetization along the ab plane is mainly from the field-bent

magnetic moments, in good accordance with the DC magnetization results in Fig. 3.

The distribution of magnetic relaxation time was further investigated by the Cole-Cole plot for χ'' and χ' (Fig. S7) using a generalized Debye model (EXPERIMENTAL SECTION) to obtain the τ -distribution parameter α ($0 \leq \alpha \leq 1$). α is 0 for a single τ , while α becomes greater for a broader distribution of τ . The small derived α of MnSb_2Te_4 implies a rather simple magnetic relaxation process. The α value (0.09 and 0.41 at 24 and 20 K, respectively) becomes greater with decreasing temperature, suggesting that the magnetic relaxation is largely affected by thermal energy. At a higher temperature, thermal activation could stimulate the transition between different magnetic states, generating a simpler magnetic relaxation scenario. When the temperature becomes lower, such transition needs to overcome a higher energy barrier with a more complicated magnetic relaxation process. The thermal activation of the glassy magnetic relaxation can be derived from the frequency-dependent behavior of χ'' . As shown in Fig. S8, the data (black dots) of $\ln(1/\tau)$ vs. T^{-1} obtained at $H_{DC} = 0$ and $H_{AC} = 5, 10$ Oe follow the Arrhenius law:

$$\ln(1/\tau) = -E_a/k_B T + \ln(1/\tau_0),$$

where τ is calculated from $\tau = 1/2\pi f$, and T is derived from peaks in χ'' [39–41]. Accordingly, the effective thermal activation energy is calculated to be 579 meV at $H_{AC} = 5$ Oe, and 324 meV at $H_{AC} = 10$ Oe, suggesting that the faster and easier magnetic relaxation with a larger AC driving field.

Different from the AFM nature of MnBi_2Te_4 [15,29], MnSb_2Te_4 exhibits a glassy magnetic ground state below $T_f \sim 24$ K, which results from the much more pronounced cation

Figure 4 Magnetic relaxation of MnSb_2Te_4 single crystal. (a) Time-dependent magnetic susceptibility at 5 K measured at different fields following a ZFC process; (b) time-dependent magnetization at 5 K at zero field following an FC process at a field of 1 kOe; (c, d) AC magnetic susceptibility is measured at different frequencies. The magnetic field is out-of-plane (c) and in-plane (d), respectively.

Figure 5 Transport properties of MnSb_2Te_4 single crystal. (a) Temperature dependence of longitudinal resistivity (ρ_{xx}) measured from 1.5 K to room temperature; (b) MR curves in an out-of-plane magnetic field at varied temperatures from 1.5 to 40 K; (c) magnetic field dependence of Hall resistivity (ρ_{yx}) traces measured at the same condition as (b).

intermixing in MnSb_2Te_4 relative to that in MnBi_2Te_4 . The cation intermixing as high as 28.9(7)% Sb on the Mn (3a) site and 19.3(6)% Mn on the Sb (6c) site may significantly alter the Mn spin correlation in intra- or inter-SL layers, and lead to a frustrated ground magnetic state due to the random AFM-FM competition. Indeed, MnSb_2Te_4 can maintain considerable magnetic moment at low temperatures with no applied field as illustrated in Figs 3c and 4b. The interplay between the intrinsic magnetism and topology arising from the Sb-Te layer may lead to the realization of the zero-magnetic-field QAH effect in MnSb_2Te_4 , breaking the odd-layer restraint in the AFM-MB₂T₄ [14].

It is noteworthy that the magnetic ground state of MnSb_2Te_4 could be influenced by the synthesis conditions. It has been reported that the MnSb_2Te_4 polycrystalline sample synthesized *via* direct melting of Mn, Sb, and Te shows field-induced FM interaction [35], which is consistent with our work. In comparison, the MnSb_2Te_4 crystal grown *via* the Sb-Te flux method displays the AFM ground state [34], and the composition and magnetism are highly dependent on the growth condition. For instance, the MnSb_2Te_4 single crystals grown at 620 and 640°C exhibit AFM and FM state with an applied magnetic field of 1 kOe, respectively [54].

Furthermore, electrical transport measurements were carried out on a MnSb_2Te_4 single crystal with a thickness of $\sim 35\ \mu\text{m}$ to probe the transport property. Fig. 5a shows the metallic temperature dependence of longitudinal resistivity ρ_{xx} in the absence of an external magnetic field from 1.5 K to room temperature. At high temperatures, ρ_{xx} rises with increasing temperature, showing a metallic behavior. At low temperatures, an upturn of ρ_{xx} takes place at $\sim 26.7\ \text{K}$, which can be attributed to the change in a spin-flip scattering of charge carriers. Fig. 5b displays the magnetoresistivity (MR) curves measured in $H//c$ at varied temperatures. The drop of MR with the increasing magnetic field is indicative of the alignment of magnetic domains. In the presence of a magnetic field, the localized magnetic moments become more aligned, reducing the scattering of charge carriers and leading to the drop of MR. Fig. 5c presents the magnetic field dependence of Hall resistivity measured in $H//c$ at varied temperatures. The positive slopes of the Hall curves demonstrate a p-type conductor with a hole density of $1.8 \times 10^{20}\ \text{cm}^{-3}$, and the mobility of $45\ \text{cm}^2\ \text{V}^{-1}\ \text{s}^{-1}$ at 1.5 K. The high carrier concentration is probably caused by the cation anti defects and Te vacancies. An anomalous Hall effect featured a hysteresis loop is observed, consistent with the magnetic behavior in M - H curves

(Fig. 3c) and recent reports on few-layer MnSb_2Te_4 [55]. Furthermore, the demagnetization steps in the Hall curves at 1.5 K are a signal of relatively complicated local magnetic structures transforming from one to another, which is distinct from those of FM metals, again confirming the glassy magnetic ground state [56–58]. The demagnetization steps disappear above 5 K, implying that the thermal energy could overcome the energy barrier of the transforming process, which is in good accordance with the magnetic measurements.

CONCLUSIONS

We developed a growth route for MnSb_2Te_4 single crystal, allowing the experimental exploration of a new member of MnBi_2Te_4 -family material [29]. Similar to MnBi_2Te_4 , the growth of high-quality MnSb_2Te_4 single crystals requires prolonged annealing in a narrow temperature range near 893 K due to its thermal metastability. The crystal structure analysis based on SCXRD data revealed the pronounced cation intermixing between Mn and Sb and led to an unexpected glassy magnetic ground state below $T_f \sim 24\ \text{K}$, accompanied with strong magnetic anisotropy with an easy axis along the c direction. This magnetic behavior makes MnSb_2Te_4 quite different from MnBi_2Te_4 with a well-established AFM state. Instead, it presents a frustrated spin system owing to the FM-AFM competitions. Furthermore, MnSb_2Te_4 displays considerable magnetic moment at zero applied field, as evidenced in the hysteresis loop of M - H and Hall curves in $H//c$. The interaction of net magnetism and possible nontrivial topology in MnSb_2Te_4 enables an attractive testbed for the discovery and realization of exotic quantum phenomena without the restraint of layer number in the AFM-type MnBi_2Te_4 -family materials.

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Conflict of interest The authors declare that they have no conflict of interest.

Supplementary information Supporting data are available in the online version of the paper.

Hao Li received her BSc from the University of Science and Technology of China in 2016. She is now a doctoral candidate of materials science and engineering at Tsinghua University. Her research includes exploratory synthesis and physical properties of low-dimensional topological materials.

Yang Wu received his BSc from Tsinghua University in 2002 and PhD from Arizona State University in 2011. He is an associate professor at the Department of Mechanical Engineering and Tsinghua-Foxconn Nanotechnology Research Center of Tsinghua University. His research interests focus on the synthesis of energy storage materials and topological quantum materials.

层状化合物MnSb₂Te₄中的玻璃磁基态

李昊^{1,6}, 李耀鑫³, 连宇坤⁴, 谢伟伟⁵, 陈玲⁴, 张金松^{3,7}, 吴扬^{2,6*}, 范守善^{3,6}

摘要 作为MnBi₂Te₄的姊妹化合物, 高质量的MnSb₂Te₄单晶可以通过固态反应法合成; 而由于MnSb₂Te₄的热亚稳性, 长时间的退火和狭窄的退火温区是合成的关键. 单晶X射线衍射分析结果说明MnSb₂Te₄具有与MnBi₂Te₄类似的晶体结构, 由七个原子层的Te-Sb-Te-Mn-Te-Sb-Te的层状结构单元沿面外方向以ABC顺序堆叠而成. 但是, 与MnBi₂Te₄相比, MnSb₂Te₄显示出更为严重的阳离子互占位, 表现为Mn (3a)位上的28.9(7)%Sb反位缺陷和Sb (6c)位上的19.3(6)%的Mn反位缺陷, 这种原子分布的变化能够为MnBi₂Te₄家族材料带来新的磁学特性. 与MnBi₂Te₄的反铁磁性质不同, MnSb₂Te₄在低于24 K的温度下展现出玻璃磁基态, 并且在弱的外加磁场下很容易被诱导至铁磁态. 通过静态和动态磁学测量, 我们详细研究了MnSb₂Te₄的磁滞行为、磁各向异性和磁弛豫过程. 此外, 通过输运测量, 我们证明了合成的MnSb₂Te₄晶体是p型导体, 并且能够呈现出反常霍尔效应. MnSb₂Te₄的不同于MnBi₂Te₄的磁学特性与Sb-Te层产生的潜在拓扑特性相互作用, 有望突破反铁磁MnBi₂Te₄家族材料中实现量子输运现象的奇数或者偶数的层数限制, 为新奇的量子物理学的探索带来新的可能.