Manipulating surface states in topological insulator nanoribbons

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Topological insulators display unique properties, such as the quantum spin Hall effect, because time-reversal symmetry allows charges and spins to propagate along the edge or surface of the topological insulator without scattering¹⁻¹⁴. However, the direct manipulation of these edge/surface states is difficult because they are significantly outnumbered by bulk carriers^{9,15,16}. Here, we report experimental evidence for the modulation of these surface states by using a gate voltage to control quantum oscillations in Bi2Te3 nanoribbons. Surface conduction can be significantly enhanced by the gate voltage, with the mobility and Fermi velocity reaching values as high as ${\sim}5{,}800~\text{cm}^2~\text{V}^{-1}~\text{s}^{-1}$ and ${\sim}3.7\times10^5~\text{m}~\text{s}^{-1}{,}$ respectively, with up to ${\sim}51\%$ of the total conductance being due to the surface states. We also report the first observation of h/2e periodic oscillations, suggesting the presence of timereversed paths with the same relative zero phase at the interference point¹⁶. The high surface conduction and ability to manipulate the surface states demonstrated here could lead to new applications in nanoelectronics and spintronics.

Bi₂Te₂ is well known as a thermoelectric material. It has also been predicted to be a three-dimensional topological insulator with robust and unique surface states consisting of a single Dirac cone at the Γ point^{3,11}. Theorists have also predicted that it should be possible to observe a number of exotic particles (such as dyons, axions and majorana fermions) in topological insulators, suggesting a fertile field for the exploration of fundamental physics^{1,11,17}. Angleresolved photoemission spectroscopy (ARPES) experiments have now confirmed that the surface states of Bi₂Te₃ have a single non-degenerate Dirac cone with a linear dispersion in momentum space18,19. Recent magnetotransport experiments in exfoliated Bi2Te3 bulk materials have also suggested two-dimensional conduction channels originating from the surface states⁶. However, despite significant efforts in material doping¹⁸⁻²⁰ and electric gating^{9,15}, it has been a challenge to modulate surface conduction because of the dominant bulk contribution due to impurities and thermal excitations in small-bandgap semiconductors^{6,9,21,22}. With their large surface-to-volume ratios, topological insulator nanoribbons are expected to significantly enhance surface conduction²², and also enable surface manipulation by external means. Here, we have used thin Bi2Te3 nanoribbons as conducting channels in field-effect transistor (FET) structures, and we demonstrate for the first time the possibility of surface state control in topological insulator nanostructures.

Transmission electron microscopy (TEM) was carried out to determine the structural characteristics of the ${\rm Bi_2Te_3}$ nanoribbons. Low-magnification TEM revealed a typical width of ${\sim}50{-}230$ nm and

length in micrometres (Fig. 1a; Supplementary Fig. S1). Selectedarea electron diffraction patterns revealed a perfect hexagonal structure with a single-crystalline rhombohedral phase (Fig. 1b). A typical high-resolution TEM image further confirmed the single crystallinity. The atomic plane spacings, marked by two pairs of parallel lines in Fig. 1c, were determined as 0.22 nm, which is consistent with those of the (11 $\overline{2}0$) atomic planes in Bi₂Te₃ nanoplates²³.

Back-gate FET devices with a standard four-terminal geometry were fabricated for transport measurements (shown schematically in Fig. 2a). The temperature dependence of the resistance is shown in Fig. 2b in linear (inset) and logarithmic scales. Channel resistance initially decreases with temperature, showing a typical metallic behaviour in the temperature range \sim 50–300 K (refs 6, 15, 24 and 25), in which phonon scattering dominates (Fig. 2b, inset). When the temperature drops below 50 K, the resistance increases, primarily due to the carrier freeze-out effect, similar to that observed in lightly doped Bi₂Se₃ (ref. 25). We have attempted to extract the activation energy \tilde{E}_a in the temperature range ~20–50 K, where a nearly linear relation exists for $R \approx e^{E_a/k_BT}$ (Fig. 2b, logarithmic scale), where R is the channel resistance, $k_{\rm B}$ is the Boltzmann constant, and T is the measurement temperature. Note that our fitting result yields only a small activation energy, less than 1 meV, which cannot be explained simply by the impurity levels in the Bi₂Te₂ (refs 18 and 19). In this temperature range, surface conduction can be enhanced compared to that at higher temperatures, therefore becoming a non-negligible component. The multiple-channel conduction⁶ makes the equation $R \approx e^{E_a/k_BT}$ invalid for extracting the activation energy (see also the band diagram analysis in Supplementary Fig. S8). As will be discussed later, the surface conduction has approximately the same order of magnitude as bulk conduction in our nanoribbons. Further decreasing the temperature below 20 K does not lead to a significant change in resistance, as can be seen in the logarithmic plot (Fig. 2b). Although a clear R-Tplateau was not identified in a large temperature range, the small variation of resistance below 4 K (Fig. 2b, logarithmic plot) suggests the presence of surface conduction, which can be further supported by the observation of Shubnikov-de Haas (SdH) and Aharonov-Bohm (AB) oscillations^{6,22}.

Figure 2c shows the channel conductance *G* as a function of both gate voltage $V_{\rm g}$ and *T*. Under zero gate bias, the total conduction at $T \approx 1.4$ –10 K falls in the range ~6.3–6.4 mS. As the gate voltage increases, the conductance decreases, exhibiting a clear *p*-type semiconductor behaviour, which is in a good agreement with typical *p*-type characteristics from undoped Bi₂Te₃ crystals⁶. It is believed that the general change in conductance as a function of gate bias suggests bulk hole conduction as the dominant component during

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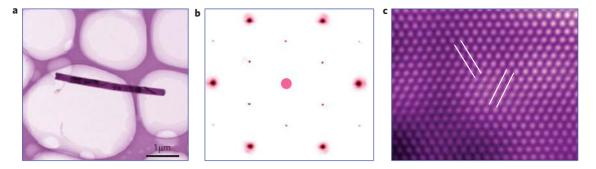


Figure 1 | Structural characterizations of a Bi₂Te₃ nanoribbon. a, Low-magnification TEM image of a Bi₂Te₃ nanoribbon on a holey carbon grid. The nanoribbon in this image is \sim 230 nm wide and \sim 3.5 µm long. **b**, Selected area diffraction pattern taken along the Bi₂Te₃ [0001] direction. Sharp diffraction spots indicate high-quality single crystals. **c**, High-resolution TEM image taken along the Bi₂Te₃ [0001] direction, revealing a perfect crystalline structure. The spacing between the (1120) atomic planes is measured to be 0.22 nm (marked by two pairs of parallel lines in c).

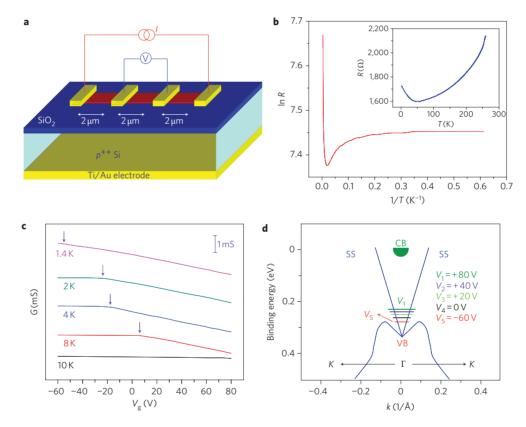


Figure 2 | Electrical transport measurements of a nanoribbon FET. a, Schematic of a typical back-gate FET device. The nanoribbon has a width of ~185 nm, thickness of ~30 nm and channel length of ~2 μ m. A constant current geometry was used during transport measurements. **b**, Channel resistance *R* on a logarithmic scale versus the inverse of temperature, 1/*T*. Inset: *R* versus *T*. **c**, Channel conductance *G* versus gate voltage V_g at five different temperatures; the magnetic field is zero. Arrows indicate a systematic shift of the Fermi level when applying gate biases at different temperatures. All curves are vertically shifted for clarification. **d**, Sketch of surface-state dispersion near the Γ point showing surface states (SS), valence band (VB) and conduction band (CB) of the bulk states (from refs. 6 and 18). The Fermi level (horizontal lines) shifts towards the middle of the bandgap as the gate voltage is increased from -60 V to +80 V, resulting in the bulk making a smaller contribution to the overall conduction of the system.

transport. Note that there are singularity points when the temperature falls to ~2–8 K (arrows in Fig. 2c), where the Fermi level is speculated to lift up from the valence band edge, leading to a sudden change in the $G-V_{\rm g}$ slope. Figure 2d presents a sketch of the band structure and surface state dispersion near the Γ point. The Fermi levels indicated in this sketch are confirmed by the observed voltage-controlled SdH oscillations, as will be described below and are qualitatively consistent with the aforementioned $G-V_{\sigma}$ plots.

One of the most intriguing challenges in topological insulators is identification of the surface states. Quantum oscillations such as SdH and AB interference currently comprise the most convincing evidence of surface electronic states^{16,26}. In parallel with the few experimental efforts regarding transport measurements^{6,24–27}, we have carried out extensive investigations into voltage-controlled SdH oscillations and AB interference, aiming to understanding the controllability of the quantum behaviour of these surface states. In the following, we present dual evidence that both quantum oscillations can be enhanced when the bulk contribution is reduced by an external gate bias.

Magnetotransport measurements in a transverse device geometry were carried out at low temperatures. The magnetic field H is

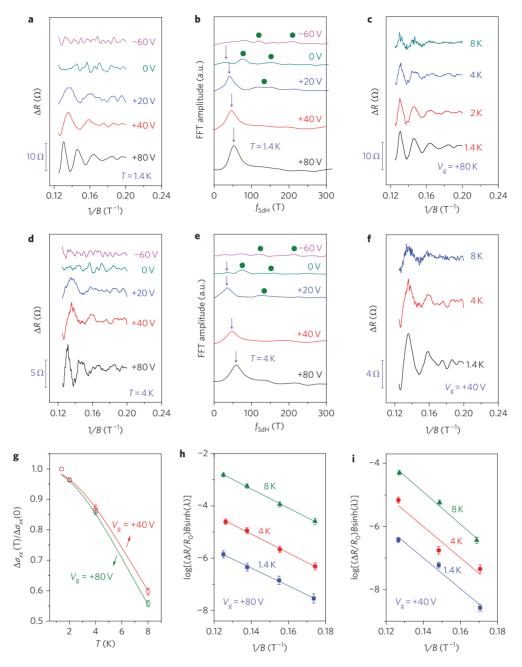


Figure 3 | **SdH oscillations in a nanoribbon FET. a-f**, Shubnikov-de Haas oscillations at different temperatures and gate voltages. The oscillations become more pronounced as the gate voltage increases from -60 V to +80 V at T = 1.4 K (**a**) and 4 K (**d**). Purple arrows in the corresponding FFT spectra (**b,e**) indicate the frequency of the SdH oscillations, which represent the surface states. Green solid dots suggest the presence of other oscillation frequencies, which are developed when the bulk carrier concentration increases under negative gate voltages. The oscillations also become more pronounced as the temperature decreases from 8 K to 1.4 K at gate voltages of +80 V (**c**) and +40 V (**f**). **g**, Normalized conductivity amplitude versus temperature at gate voltages of +80 V and +40 V (**f**). **g**, Normalized conductivity amplitude versus temperature at gate voltages of +80 V and +40 V (**f**). Transport lifetime, mean free path and mobility can be extracted from the best fit to log [$(\Delta R/R_{\alpha})Bsinh \lambda$].

perpendicular to both current flow and the nanoribbon surface. Under zero gate bias at 1.4 K, the magnetoresistance does not show an evident SdH oscillation (Fig. 3a, dark cyan); instead, the spectrum exhibits a nearly linear magnetoresistance with super-imposed universal conductance fluctuations (UCFs; Supplementary Fig. S2b,c; $V_g = 0$ V). By applying a positive gate voltage (20–80 V), however, the hole carriers in the bulk material are partially depleted, leading to reduced bulk conduction. The surface states in this scenario manifest themselves through progressively growing SdH oscillations, as shown in Fig. 3a. Correspondingly, fast

Fourier transform (FFT) spectra show dominant oscillation frequencies ($f_{\rm SdH}$ (T), Fig. 3b), in accordance with the SdH periodicities (Fig. 3a). We also note that the frequency peaks shift steadily towards higher magnetic fields, suggesting an increase in the two-dimensional carrier density of the surface. In contrast, a negative voltage on the gate induces holes into the bulk material, leading to diminished SdH oscillations and pronounced UCFs (Fig. 3a; $V_{\rm g} = -60$ V). To further verify the genuine nature of this controllability, we performed the same experiments at 4 K (Fig. 3d,e), where the oscillation amplitudes apparently decrease compared to those at

Table 1 Estimated parameters from the SdH oscillations at $T = 1.4$ K.												
V _g (V)	f _{sdн} (Т)	$n_{\rm 2D}$ (10 ¹² cm ⁻²)	m _{cycl} (m _o)	k _F (Å ^{−1})	V _F (10 ⁵ ms ⁻¹)	E _F (meV)	T (10 ⁻¹³ s)	ℓ (nm)	μ (cm $^2/V^{-1}s^{-1}$)			
+20	40.3	0.97	0.111 ±0.002	0.035	3.68	~85	_	_	_			
+40	47.5	1.1	0.119 <u>+</u> 0.003	0.038	3.67	~92	~3.11	~ 114	~4,560			
+80	58.1	1.4	0.131±0.002	0.042	3.74	~103	~4.28	~ 160	~5,790			

1.4 K (Fig. 3a,b). It is well known that SdH oscillation is a characteristic of the two-dimensional Fermi surface⁶, where magnetoresistance varies periodically with the inverse of the magnetic field $\Delta(1/B)$ through the following equations^{22,27}: $\Delta(1/B) = e/(hn_{2D})$ and $\Delta(1/B) = 4\pi e/k_{\rm F}^2 h$, where *B* is magnetic flux density, n_{2D} is the two-dimensional carrier density, $k_{\rm F}$ is the Fermi vector, *e* is the electron charge, and *h* is Planck's constant. By using the obtained FFT peak values with $V_{\rm g} = +20$, +40 and +80 V (as shown in Fig. 3b,e), $n_{\rm 2D}$ and $k_{\rm F}$ can be extracted. The calculated results are summarized in Table 1. Alternatively, these parameters can also be calculated based on the fitting of 1/B versus the Landau level *n* (Supplementary Fig. S3).

The SdH amplitudes as a function of temperature were analysed to obtain more information about the surface states under constant voltages of +80 and +40 V (Fig. 3c,f, respectively). Oscillation amplitudes at both voltages decrease rapidly with a temperature increase from 1.4 to 8 K. The temperature-dependent amplitude $\Delta \sigma_{xx}$ of the SdH oscillations is described by $\Delta \sigma_{xx}(T)/\Delta \sigma_{xx}(0) = \lambda(T)/\sinh(\lambda(T))$ (ref. 6). The thermal factor is given by $\lambda(T) = 2\pi^2 k_{\rm B} T m_{\rm cvcl} / (\hbar eB)$ (refs 6 and 25), where $m_{\rm cvcl}$ is cyclotron mass and \hbar is the reduced Planck constant. By taking the conductivity oscillation amplitude and performing the best fit to the $\Delta \sigma_{\rm xx}(T)/\Delta \sigma_{\rm xx}(0)$ equation, $m_{\rm cycl}$ is extracted as ~0.131 m_0 at $V_g = +80$ V and $\sim 0.119 m_0$ at $V_g = +40$ V, as shown in Fig. 3g. Here, m_0 is the electron rest mass. Because the cyclotron mass is given by $m_{\rm cycl} = E_{\rm F}/V_{\rm F}^2$ (ref. 6), where $E_{\rm F}$ and $V_{\rm F}$ are the Fermi level and Fermi velocity, respectively, both $E_{\rm F}$ and $V_{\rm F}$ can be precisely obtained provided $m_{\text{cycl}}V_{\text{F}} = \hbar k_{\text{F}}$ (ref. 26). Note that the Fermi level moves from \sim 85 meV towards the middle of the bandgap, ~ 103 meV, when increasing the gate voltage from +20 V to +80 V (Table 1, Fig. 2d). The Fermi velocity, however, remains constant, saturating at $\sim 3.7 \times 10^5$ m s⁻¹. This saturation behaviour can be explained by the linear dispersion relation of the surface states^{6,18} (Fig. 2d) described by $E_{\rm F} = (\hbar V_{\rm F})k_{\rm F}$, where $\hbar V_{\rm F}$ is the slope of the energy E versus the vector k in momentum space. The systematic development of SdH oscillations under external gate biases clearly presents strong experimental evidence of the tunability of the surface states, which are, in essence, induced by shifting of the Fermi level (Table 1, Fig. 2d), together with partial depletion of bulk carriers.

We can also estimate the transport lifetime of the surface states (τ) by considering the Dingle factor e^{-D} , where $D = 2\pi^2 E_{\rm F}/(\tau eBV_{\rm F}^2)$ (refs 6, 24 and 26). Note that $\Delta R/R$ is proportional to $[\lambda (T)/\sinh(\lambda(T))]e^{-D}$, and the lifetime can be inferred from the slope in the logarithmic plot of $\log[(\Delta R/R_0)B\sinh(\lambda(T))] \approx [2\pi^2 E_{\rm F}/(\tau eV_{\rm F}^2)] \times (1/B)$ (Fig. 3h,i). By using extracted cyclotron masses at $V_{\rm g} = +40$ and +80 V, the surface state lifetime can be estimated to be $\sim 3.1 \times 10^{-13}$ and $\sim 4.3 \times 10^{-13}$ s, respectively.

Next we will acquire important parameters such as mean free path $\ell = V_F \tau$ and the surface mobility^{6,24,25} $\mu = e\tau/m_{cycl} = e\ell/\hbar k_F$. Our calculation yields estimated mean free paths of 114 and 160 nm, and mobilities of ~4,560 and ~5,790 cm² V⁻¹ s⁻¹, for $V_g = +40$ and +80 V, respectively. Such a long mean free path and such a high mobility re-confirm the existence of surface states, which are also consistent with those reported in Bi₂Te₃, Bi₂Se₃ and Bi_{1-x}Sb_x bulk materials^{6,22,26}. The rich information obtained from the gate-modulated and temperature-dependent SdH oscillations allows the possibility of estimating the percentage of total conduction that is surface conduction. Table 2 provides two typical conditions under which the surface $[G(surface) = (e^2/h)k_F\ell]$ and bulk conductance [G(total)] are provided. It is found that surface conduction can be dramatically enhanced up to ~51% under a gate voltage of +80 V. Compared to their bulk counterpart, these Bi₂Te₃ nanoribbons have great advantages, with their large surface-to-volume ratio being crucial to enabling voltage-controlled quantum oscillations as well as high surface conduction.

We next analyse AB oscillations in the Bi₂Te₃ nanoribbons and explore possible voltage-controlled phase interference. An external magnetic field was applied along the length of the nanoribbon, parallel to the direction of the electric current; conduction carriers remain phase-coherent after completing closed trajectories. Indeed, our low-temperature magnetoresistance measurements along this direction provided periodic resistance oscillations, even without the gate voltage (Fig. 4a). After subtraction of the smooth background (Supplementary Fig. S4a), periodic AB oscillations could be clearly resolved with a period of \sim 0.75 T, corresponding to a sample cross-sectional area of $5.54 \times 10^{-15} \text{ m}^2$ (Fig. 4b; Supplementary Fig. S4b); this value is consistent with sample dimensions of ~ 185 nm (width) and ~ 30 nm (thickness), as determined by atomic force microscopy. When the temperature was raised to 8 K, the amplitude of the AB oscillation diminished rapidly (Fig. 4b), suggesting that the bulk contribution affects the carriers' trajectories, and phase coherence can be jeopardized by the increased phonon scattering at elevated temperatures²².

To explore the possibility of gate modulation on these AB oscillations, both positive and negative gate biases were applied, with the aim of tuning the Fermi level. As expected, a positive gate voltage induces pronounced AB oscillations, but a negative voltage diminishes quantum interference (Fig. 4c,d), reminiscent of the results observed for the SdH oscillations. The same trend was also confirmed at T = 2, 4 and 6 K (Supplementary Fig. S4c-h). To understand the origin of these AB oscillations, a FFT was performed (plotted in Fig. 4e). The prominent h/e oscillation frequency indicates that the surface carriers travel the perimeter of the nanoribbon without confining themselves to the top and bottom surfaces. This behaviour is in agreement with the scenario for Bi₂Se₃ nanoribbons²², where electrons propagate coherently through the sidewalls to produce the quantum interference effect. The presence of the h/2e frequency, however, suggests that there is the possibility that carriers could propagate in complete circles, clockwise or anticlockwise, all with the same relative zero phase at the point of interference, and thus being robust against averaging^{16,28}. The FFT amplitude of the AB oscillations was also plotted as a function of temperature (Fig. 4f). The $T^{-1/2}$ fit indicates the absence of inelastic phonon scattering²². The phase-coherent diffusion length is described by $L_{\phi} = (\ell V_{\rm F} \tau_{\phi})^{1/2}$, where τ_{ϕ} is the mean time between inelastic events ($\tau_{\phi} \approx \hbar/k_{\rm B}T$, ref. 22). At $V_{\rm g} = +80$ V, L_{ϕ} can be estimated to be ~ 0.57 µm. Such a long phase-coherent diffusion length

Table 2 | Estimated surface conduction percentage with zero magnetic field and at T = 1.4 K.

V _g (V)	G(surface) (mS)	R(total) (Ω)	$R_{ m sheet}$ (total) (Ω \Box^{-1})	G _{sheet} (<i>total</i>) (mS)	G(surface)/ G _{sheet} (total)
+40	1.68	1,911.3	176.8	5.66	~30%
+80	2.60	2,138.3	197.8	5.06	~51%

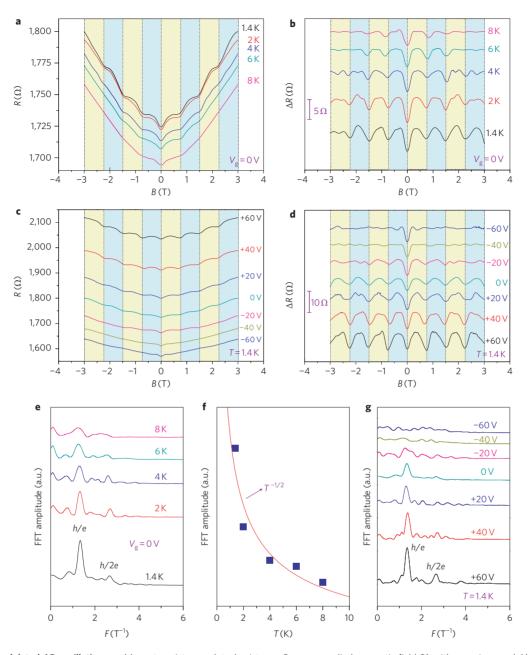


Figure 4 | Gate-modulated AB oscillations. a, Magnetoresistance plots (resistance *R* versus applied magnetic field *B*) with superimposed Aharonov-Bohm oscillations at five different temperatures between 1.4 and 8 K, and no gate voltage. **b**, AB oscillations can be clearly seen when the smooth magnetoresistance background is subtracted from the plots in **a**. A detailed description of the subtraction method can be found in Supplementary Fig. S2a. **c**, Magnetoresistance plots at seven different gate voltages between -60 and 60 V, at 1.4 K. **d**, Significant enhancement of the AB oscillation for positive gate voltages can be clearly seen when the smooth magnetoresistance background is subtracted from the plots in **c**. **e**, FFT of the magnetoresistance plots in **a** reveals AB oscillations with periods of *h/e* and *h/2e*, suggesting different interference paths during in-plane transport. The peaks at *h/e* and *h/2e* are most pronounced at 1.4 K. **f**, Amplitude of the FFT spectra in **e** versus temperature: the amplitude, fitted to $T^{-1/2}$ (red line), suggests the absence of inelastic phonon scattering. **g**, AB oscillations with periods of *h/e* and *h/2e* can also be seen in the FFT of the magnetoresistance plots in **c**. The peaks at *h/e* and *h/2e* are most pronounced at a gate voltage of +60 V.

would enable both h/e and h/2e oscillations, given the fact that the surface carriers could travel a long distance before randomly changing their wavefunction phase.

Finally, a FFT was also carried out for voltage-controlled AB interference at T = 1.4 K (Fig. 4g). The dramatic development of the amplitude further verifies the tunability of phase interference on the surface using gate voltage. Both the voltage-controlled SdH and the AB oscillations suggest that by applying an external gate voltage, the Fermi level can be tuned away from the valence band, leading to the depressed

bulk conduction and the enhanced surface states. The successful modulation of the surface states in our study offers a ready pathway towards the practical applications of topological insulators.

Methods

Synthesis. High-purity polyvinylpyrrolidone (PVP) was dissolved in ethylene glycol (36 ml), followed by the additions of Bi_2O_3 powder (1 mmol), tellurium powder (3 mmol) and ethylenediamine tetraacetic acid (EDTA) powder. The resulting suspension was stirred vigorously and subsequently sealed in a steel autoclave. The autoclave was then heated to a temperature in the range 180–220 °C and maintained

over 4 h. The synthesized products were collected by high-speed centrifugation, washed several times with distilled water and absolute ethanol, and finally dried at 50 °C for 48 h in an oven. Single-crystalline nanoribbons were formed during the synthesis process and were typically several micrometres in length and several hundreds of nanometres in width. The crystalline orientation of the as-grown nanoribbons was along [1120] direction.

Device fabrication and characterization. Back-gate FET devices were fabricated by dispersing Bi₂Te₃ nanoribbons on 300 nm SiO₂/silicon substrates and subsequently using electron-beam lithography to fabricate electrodes (10 nm titanium and 90 nm gold). A typical FET device as used for the voltage-controlled experiments comprised a narrow Bi₂Te₃ nanoribbon as a conducting channel, with a width of ~185 nm and a channel length of ~2 μ m. Standard four-terminal electrodes were fabricated to eliminate contact resistance. A constant a.c. current of ~0.05–0.1 μ A with a frequency of 130 Hz was fed through two outer contacts, and the voltage drop across inner pads was measured to determine resistance. The structural characteristics of the Bi₂Te₃ nanoribbons were investigated using a FEI Tecnai F20 TEM. The bismuth/tellurium composition was analysed using an energy-dispersive spectroscopy technique. Transport measurements were carried out with a superconducting magnet system (American Magnetics). The lowest temperature used was 1.4 K, and the magnetic field could be up to \pm 8 T.

AB interference. It is widely considered that the AB interference of conductance is one of the most remarkable manifestations of electron phase coherence in mesoscopic samples. When two possible paths of an electron enclose magnetic flux, an observable interference effect occurs due to the phase difference, $\Delta \phi_{AB}$, between the two paths: $\Delta \phi_{AB} = \oint e(S \cdot dI)/\hbar = e\Phi/\hbar = 2\pi\Phi/\phi_0$. Here Φ represents the magnetic flux enclosed by the paths, S is the cross-sectional area projected normal to the field, and $\phi_0 = h/e$ is the flux quantum^{16,29}. The topological insulator nanoribbons can be envisioned as hollow metallic cylinders with surface transport channels. The quantum interference of surface carriers with different paths in this hollow structure could make the conductance an oscillatory function of magnetic flux^{16,30}.

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Author contributions

F.X. and L.H. designed and fabricated the devices. F.X., L-T.C., M.L. and A.S. carried out the measurements. L-N.C., Y.W., Z.G.C. and J.Z. synthesized the Bi₂Te₃ nanoribbons and performed structural analysis. Y.W., G.H., X.K., X.J. and Y.Z. contributed to the measurements and analysis. K.W. supervised the research. F.X., Y.W., L.H., J.Z. and K.W. wrote the paper, with help from all other co-authors.

Additional information

The authors declare no competing financial interests. Supplementary information accompanies this paper at www.nature.com/naturenanotechnology. Reprints and permission information is available online at http://npg.nature.com/reprintsandpermissions/. Correspondence and requests for materials should be addressed to F.X. and K.L.W.