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Journal

iScience, 26(1)

ISSN

2589-0042

Authors

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Publication Date

2023

DOI

10.1016/j.isci.2022.105691

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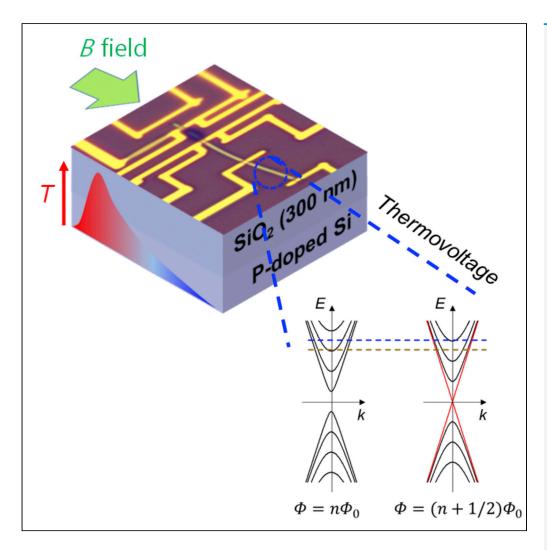
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Quantum interference probed by the thermovoltage in Sb-doped Bi₂Se₃ nanowires



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Highlights

Aharonov–Bohm interference in topological insulator nanowires probed by thermovoltage

180° out-of-phase thermovoltage oscillations with the magnetic flux and gate voltage

Thermoelectric signal as a probe of electronic structure in quantum materials

Kwon et al., iScience 26, 105691 January 20, 2023 © 2022 The Author(s). https://doi.org/10.1016/ j.isci.2022.105691





Article

Quantum interference probed by the thermovoltage in Sb-doped Bi₂Se₃ nanowires

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SUMMARY

The magnetic-flux-dependent dispersions of sub-bands in topologically protected surface states of a topological insulator nanowire manifest as Aharonov–Bohm oscillations (ABOs) observed in conductance measurements, reflecting the Berry's phase of π because of the spin-helical surface states. Here, we used thermoelectric measurements to probe a variation in the density of states at the Fermi level of the surface state of a topological insulator nanowire (Sb-doped Bi₂Se₃) under external magnetic fields and an applied gate voltage. The ABOs observed in the magnetothermovoltage showed 180° out-of-phase oscillations depending on the gate voltage values, which can be used to tune the Fermi wave number and the density of states at the Fermi level. The temperature dependence of the ABO amplitudes showed that the phase coherence was kept to T=15 K. We suggest that thermoelectric measurements could be applied for investigating the electronic structure at the Fermi level in various quantum materials.

INTRODUCTION

Thermoelectric (TE) studies of low-dimensional nanostructures have been extensively carried out to achieve a high figure of merit for the energy conversion between electricity and heat. $^{1-5}$ After the discovery of topological insulator (TI) properties of traditional TE materials such as bismuth selenide (Bi₂Se₃) and bismuth (antimony) telluride [Bi₂(Sb₂)Te₃], many theoretical and experimental approaches have been performed to improve the TE properties using the TI surface states. $^{6-11}$ However, the experimental approach to investigate the electronic structure based on the TE effect has not been intensively investigated. Because the Seebeck coefficient (S) or thermovoltage ($V_{\text{ThV}} = -S\Delta T$, where ΔT is the temperature difference between two spatially separated points) is sensitive to the electronic density of states (DOS) at the Fermi level, measurements of V_{ThV} can provide information about the electronic structure near the Fermi level. $^{12-14}$

In the present study, we report magnetothermovoltage measurements for an Sb-doped Bi $_2$ Se $_3$ (BiSe) nanowire (NW) under axial magnetic flux (Φ). Pronounced oscillations with varying Φ were observed in both the magnetothermovoltage and the magnetoconductance with the same period satisfying the Aharonov–Bohm oscillation (ABO). The thermovoltage as a function of gate voltage showed 180° out-of-phase oscillations for the two cases of $\Phi=0$ and $\pm\Phi_0/2$, where Φ_0 is the magnetic flux quantum. The magnetothermovoltage oscillations also showed the 180° out-of-phase behavior at the local maximum and minimum locations in the gate-dependent oscillation obtained at $\Phi=0$ or $\pm\Phi_0/2$. These results are consistent with the Φ -dependent sub-band dispersion model for the topologically protected surface states of a TI NW, which has been mainly demonstrated by the ABO in the magnetoconductance. The results reflect the sensitivity of the thermovoltage to the change of the DOS at the Fermi level under a varying gate voltage and varying magnetic field; thus, the thermovoltage measurement could be a useful tool to study the electronic structure of various quantum materials.

RESULTS AND DISCUSSION

Electrical characterization

Figure 1A shows an optical image of a Sb-doped BiSe NW device (BiSe#1) fabricated on a 300 nm-thick SiO₂/Si substrate using conventional microfabrication processes. The device has four metal (Ti/Au) contacts and three NW segments for the electrical and thermovoltage measurements and a heater line to

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https://doi.org/10.1016/j.isci. 2022.105691







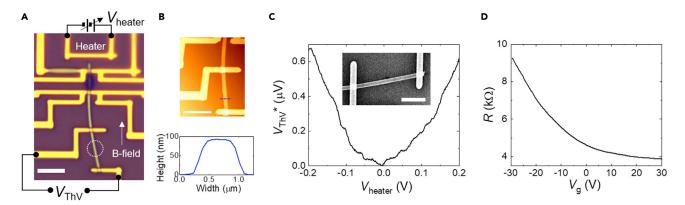


Figure 1. Device images and electrical characterization

(A) Optical image of the Sb-doped Bi_2Se_3 NW device (BiSe#1) configured for the thermovoltage measurement. Scale bar: $5 \mu m$. The bottom NW segment denoted by a dashed circle was used in the present study.

(B) Upper and lower panels: Atomic force microscopy (AFM) image and the height profile along the dashed blue line in the upper panel, respectively. Scale bar: 5 μm.

(C) Thermovoltage (V_{ThV}^*) as a function of heater voltage (V_{heater}) . Inset: Scanning electron microscope (SEM) image of the device. Scale bar: $3 \mu m$.

(D) Resistance (R) as a function of the gate voltage (V_g).

apply a temperature gradient along the NW (see STAR Methods for details of the fabrication and measurements). The highly doped Si substrate served as a gate electrode. The thermovoltage measurement configuration is also shown in the image. In the present study, we used the bottom segment of the NW [length $(L) = 6.8 \, \mu \text{m}$, width $(W) = 440 \, \text{nm}$, height $(H) = 93 \, \text{nm}$], as denoted by a dashed circle, because the topmost NW segment having two metallic thermometers was burnt during measurements and the second metal contact from the top could have been deformed by the burning. Here, the height and width of the NW were obtained from an AFM image (see Figure 1B) and SEM image (see the inset of Figure 1C), respectively. Although the third contact was as far as 13 $\, \mu \text{m}$ from the heater line, we observed a measurable V_{ThV}^* as a function of the voltage applied to the heater (V_{heater}) at $T = 4.2 \, \text{K}$ (Figure 1C), where V_{ThV}^* is a thermovoltage after subtracting a minimum voltage from the raw data in Figure S1A in the Supplementary Information. The V_{ThV}^* - V_{heater} plot shows nearly symmetric behavior for the polarity of the V_{heater} , which is evidence of a thermoelectric signal. Figure 1D displays the resistance (R) as a function of the back-gate voltage (V_g) , which shows that an n-type carrier is the dominant carrier for the examined V_g region.

Aharonov-Bohm oscillations in magnetoconductance and magnetothermovoltage

In Figure 2, we investigate the conductance (G) and thermovoltage ($-V_{ThV}$) under an applied magnetic (B) field at $V_{o} = 0$ V and T = 4.2 K with BiSe#1, where the B field was applied parallel to the axial direction of the first segment of the NW from the top in Figure 1A. The NW examined in the present study (indicated by the dotted circle in Figure 1A) was misaligned with the B-field direction by as much as ~13°. Here, we used the thermovoltage as $-V_{\text{ThV}}$ to match the polarity to the Seebeck coefficient S. The black curve in the upper panel of Figure 2A displays G as a function of the B field (top axis)-; the curve shows that the conductance decreases with increasing B field in both polarities, resulting in a sharp peak near zero B field. This result indicates the existence of weak antilocalization (WAL) because of the spin-orbit interaction in the bulk. 16,17 A relatively small magnetoconductance modulation superimposed on the WAL curve of the smooth fitting result (the red curve in the upper panel of Figure 2A) was observed. The lower panel of Figure 2A shows a pronounced oscillation in ΔG as a function of the B field, where ΔG was obtained by subtracting the smooth fitting result from the raw data (black curve). From a fast Fourier transformation (FFT) analysis (Figure 2B), we obtained the oscillation period as $\Delta B = 0.112$ T, which is consistent with that estimated from a TI NW with a measured geometry of W = 430 nm and H = 83 nm when a 5 nm-thick oxidized layer of the TI NW was assumed to be present and the angle was tilted with respect to the Bfield direction, following the ABO. 18-21 This result indicates that the magnetoconductance oscillation observed in the TINW is related to the surface state of the NW. The lower horizontal axis is Φ in the unit of Φ_0 (=h/e), where h and e are Planck's constant and the elementary charge, respectively. Although the conductance maxima in the oscillation are located at integer multiples of Φ_0 for $-5 < \Phi < 5$, the oscillation phase continuously evolves with increasing B fields for other regions. A small component corresponding to the h/2e-periodicity in Figure 2B was also observed. In ref. 18 with Sb-doped Bi₂Se₃ TI nanoribbons, the observed h/2e periodicity was

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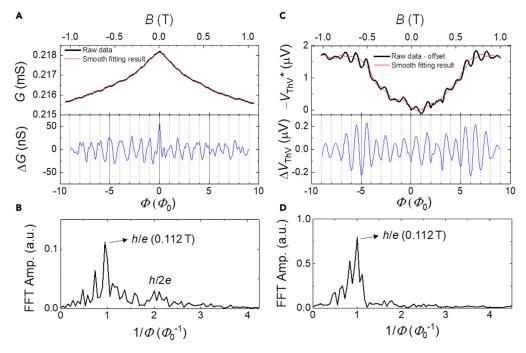


Figure 2. Magnetoconductance and magnetothermovoltage oscillations of BiSe#1

(A) Upper panel: conductance (G) as a function of magnetic flux (Φ , bottom axis) (black curve) and the smooth fitting result (red curve) at $V_g = 0$ V and T = 4.2 K. Here, Φ is the magnetic flux normalized by the magnetic flux quantum, Φ_0 . Top axis: B field. Lower panel: conductance difference between the raw data and smooth fitting result in the upper panel (ΔG) as a function of Φ .

(B) FFT amplitude of $\Delta G(\Phi)$ curve.

(C) Upper panel: $-V_{\text{ThV}}^*$ as a function of Φ (black curve) and smooth fit result (red one) at $V_g = 0$ V and T = 4.2 K. Lower panel: thermovoltage difference between the $-V_{\text{ThV}}^*$ (Φ) curve and smooth fit result in the upper panel (ΔV_{ThV}) as a function of Φ .

(D) FFT amplitude of $\Delta V_{\text{ThV}}(\Phi)$ curve. In (A) and (C), we performed the smooth fitting of the raw data by using the Savitzky-Golay method with neighbor 50 points for a point.

interpreted as the Altshuler—Aronov—Spivak (AAS) oscillations, based on the analysis of temperature and gate-voltage dependences for various channel lengths, compared to the Aharonov-Bohm (AB) h/e oscillations. In the study, it was found that the AAS oscillations were suppressed when the perimeter length (L_p) of the nanowire is shorter than the channel length (L) because a complete pair of the time-reversed paths was not formed along the perimeter of the nanowire in that case. On the other hand, in ref. 19, the observed h/2e was explained by the interference of electrons that travel around the circumference of the TI NW two times in a quasi-ballistic regime, based on the exponential decay of the interference amplitude with increasing temperature. In our case, the channel length ($L = 6.8 \ \mu m$) was much longer than the perimeter of the nanowire ($L_p \approx 1 \ \mu m$), which indicates that our nanowire is closed to a diffusive limit allowing the complete formation of the time-reversed path along the perimeter of the NW to observe the AAS oscillations. The lower panel of Figure 2A shows the h/2e oscillation only in a weak B-field region of $-\Phi_0 < \Phi < \Phi_0$. The AAS oscillations are usually suppressed with increasing field because it needs a coherent backscattering condition under the time-reversal symmetry, which is broken at a sufficiently high B field. Thus, we consider that the h/2e oscillation observed in the magnetoconductance is related to the AAS oscillations.

We here address the variation of the thermovoltage with the B field. The upper panel of Figure 2C shows $-V_{\text{ThV}}$ * as a function of the B field (black curve) at $V_{\text{heater}} = 100$ mV, $V_g = 0$ V, and T = 4.2 K (see the raw data in Figure S1B of the Supplementary Information). The magnetothermovoltage also shows oscillation behavior under a varying B field. After subtracting the smooth fitting result (red curve) from the raw data, we plotted the result [ΔV_{ThV} as a function of the B field (top axis)] in the lower panel of Figure 2C. For the magnetothermovoltage case, the FFT analysis provides an oscillation period of $\Delta B = 0.112$ T (Figure 2D), which is consistent with that for the magnetoconductance oscillation. The magnetothermovoltage shows an even function of the B field [e.g., S(B) = S(-B)], which is attributed to the invariant system when the B-field



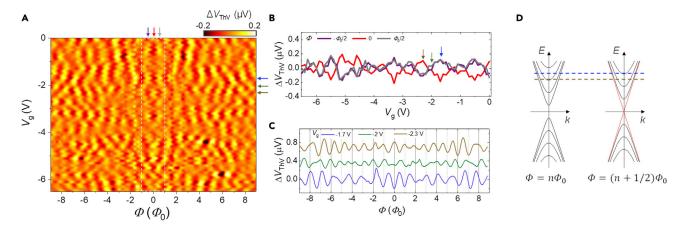


Figure 3. 180° out-of-phase oscillations depending on the magnetic flux and gate voltage

(A) ΔV_{ThV} as a function of Φ and V_{g} of BiSe#1.

(B) ΔV_{ThV} as a function of V_g at $\Phi=0$ and $\pm\Phi_0/2$, where locations are also indicated by the same-colored arrows in (A).

(C) ΔV_{ThV} as a function of Φ at $V_g = -1.7$, -2, and -2.3 V; the corresponding locations are also indicated by the same-colored arrows in (A) and (B). Curves were vertically shifted for the clarity.

(D) Electronic sub-band dispersion at $\Phi = n\Phi_0$ and $(n+1/2)\Phi_0$, where n is an integer.

direction changes.²² For the magnetothermovoltage oscillation case, the ΔV_{ThV} minima in the oscillation are located at integer multiples of Φ_0 for the examined Φ , without the FFT amplitude corresponding to the h/2e oscillation (see Figure 2D). For the topmost NW segment in Figure 1A (BiSe#2), we also obtained similar behaviors in the magnetoconductance and magnetothermovoltage with similar periodicities (see Figure S2 in the Supplementary Information).

Gate-voltage and magnetic-field dependences of the thermovoltage

Figure 3A displays ΔV_{ThV} as a function of Φ and V_{g} for BiSe#1, which shows symmetric oscillations with respect to the zero field for the examined $V_{\rm q}$ region (also see Figure S3 in the Supplementary Information for a different V_{α} region). Near $\Phi = 0$, as indicated by a dashed white box, the patterns show oscillation behaviors for varying V_g . Figure 3B shows ΔV_{ThV} as a function of V_g at $\Phi = -\Phi_0/2$, 0, and $\Phi_0/2$; these locations are indicated by vertical purple, red, and gray arrows in Figure 3A, respectively. The curve at $\Phi=0$ shows a 180° out-of-phase modulation with respect to the two curves at $\Phi=\pm\Phi_0/2$, whereas the two curves show nearly the same modulation. This result is consistent with the sub-band model for the topologically protected surface state (TPSS) for a TI NW. In Figure 3D, the sub-band structures for $\Phi = n\Phi_0$ and $(n+1/2)\Phi_0$ are plotted with a dispersion relation of $E(k,l,\Phi)=\pm\hbar v_F\sqrt{k^2+\Delta k^2(l+0.5-\Phi/\Phi_0)^2}$, where n is an integer, \hbar is the reduced Planck's constant, v_F is the Fermi velocity in the surface state, k is the NW axial wave number, and l (=0, ± 1 , ± 2 , ...) is the angular momentum quantum number; Δk (= $2\pi/$ C) is the quantized wave number along the perimeter of the TI NW, where ${\it C}$ is the circumference of the TI NW. ^{23,24} The cases of $\Phi = 0$ and $\pm \Phi_0/2$, i.e., n = 0 correspond to the left and right panels of Figure 3D, respectively. The dashed brown and blue lines indicate Fermi levels that meet the bottom of each sub-band for $\Phi=0$ and $\pm\Phi_0/2$, respectively. Whenever the Fermi level crosses the sub-band with increasing $V_{\rm q}$, the DOS shows a local maximum because of the flat bottom of each sub-band. The sub-band energy spacing at both $\Phi = 0$ and $\pm \Phi_0/2$ is determined by $\Delta E = \hbar v_F \Delta k$, where $v_F = 3 \times 10^5$ m s⁻¹ and C = 1026 nm (i.e., \sim 1.2 meV).

Because the Fermi level aligns with the bottom of sub-bands for the cases $\Phi=0$ and $\pm \Phi_0/2$ in a 180° out-of-phase manner, the local maximum in the DOS is expected to alternatively appear as the V_g is varied. The thermoelectric effect is sensitive to the change of the DOS in energy; thus, the 180° out-of-phase modulation behaviors in the thermovoltage with varying V_g for cases of $\Phi=0$ and $\pm \Phi_0/2$ in Figure 3C directly originate from the 180° out-of-phase behavior of the DOS with varying V_g for the two cases. The two locations at $V_g=-2.3$ and -1.7 V indicated by brown and blue arrows in Figure 3B indicate the local magnetothermovoltage maxima for the cases of $\Phi=0$ and $\pm \Phi_0/2$, respectively (see also the same-colored arrows in Figure 3A). In Figure 3C, the ΔV_{ThV} (Φ) curves show a local maximum and minimum at $\Phi=0$ for $V_g=-2.3$ and -1.7 V, which correspond to the Fermi energy locations, as indicated by dashed brown and





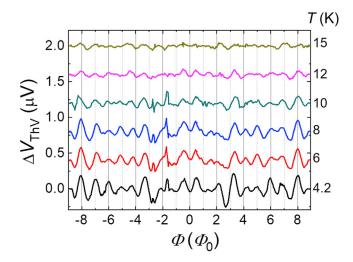


Figure 4. Temperature dependence of magnetothermovoltage oscillations ΔV_{ThV} as a function of Φ at $V_{\text{q}} = 0$ V for various temperatures (BiSe#1). Curves were vertically shifted for the clarity.

blue lines in Figure 3D, respectively. As Φ increases to $\Phi \sim \pm 2\Phi_0$, the thermovoltage modulation follows the prediction; for instance, local maxima and minima appear at even and odd multiples of \pm $\Phi_0/2$, which are known as AB oscillations with zero and π phases (0-ABO and π -ABO), respectively. Pror $2\Phi_0 < |\Phi| < 4\Phi_0$, the local maximum and minimum locations in the oscillations at $V_g = -2.3$ and -1.7 V deviate slightly from those observed in $-2\Phi_0 < \Phi < 2\Phi_0$, possibly because of an additional dephasing effect in a strong spin–orbit-coupled system. However, at $V_g = -2$ V indicated by the green arrow in Figures 3A and 3B, the oscillation pattern in Figure 3C shows a weakened oscillation amplitude with a non-regular period even at $-2\Phi_0 < \Phi < 2\Phi_0$, which is attributed to the regime being an intermediate state between the 0-ABO and π -ABO states. These behaviors were also observed in a relatively high V_g region of 27–30 V, as shown in Figure S1 of the Supplementary Information. For the direct comparison between the magnetoconductance and magnetothermovoltage as a function of V_g and Φ , we prepared another BiSe NW (BiSe#3) and displayed the corresponding datasets in Figures S4–S6 of the Supplementary Information.

Temperature dependences of magnetothermovoltage

Figure 4 shows ΔV_{ThV} as a function of Φ at various temperatures at $V_g = 0$ V with BiSe#1, where the amplitude of the ABO is suppressed with increasing temperature, whereas the oscillation behavior is maintained even at T=15 K. We note that all ΔV_{ThV} (Φ) curves showed the π -ABO with the minimum value at $\Phi = 0$, contrary to the observations at $V_q = 0$ V in Figures 2 and 3. This implies that the intrinsic doping level changed. Actually, the temperature dependences were measured after performing the $\Delta V_{\mathsf{ThV}}\left(\pmb{\varPhi} \right)$ measurements in a range of 27–30 V for \sim 15 h, as shown in Figure S1 of the Supplementary Information. We consider that such high gate biasing for a relatively long period could induce a doping effect to the NW because of charge traps in the SiO_2 oxide layer, ²⁵ resulting in a shift of the Fermi level compared to the that before applying such high $V_{\rm q}$ condition. Because the temperature difference (ΔT) between two different points in the relation of $S = -V_{ThV}/\Delta T$ changes as the base temperature is varied, the direct comparison of $-V_{ThV}$ at different temperatures could not provide a physical meaning. Nevertheless, it is clear that the phase coherence was kept to $T=15~\mathrm{K}$ without a transition between $\pi\text{-ABO}$ and 0-ABO with increasing temperature, which indicates that the surface protected surface state may not experience de-phasing by disorder up to the 15 K. 19,23 In Figure S7 of the Supplementary Information, we also direct compared the magnetoconductance and magnetothermovoltage for varying temperatures with BiSe#3.

Conclusions

We performed thermoelectric measurements for an Sb-doped Bi₂Se₃ NW to reveal the variation of the DOS at the Fermi level of topologically protected surface states when Φ , $V_{\rm g}$, and T were varied. The thermovoltage oscillations with varying $V_{\rm g}$ at $\Phi=0$ and $\pm 2\Phi_0$ showed 180° out-of-phase behavior,





which indicates that surface-state sub-bands because of the confinement along the perimeter of the TI NW exist at the NW surface. The magnetothermovoltage obtained at two $V_{\rm g}$ values showing local maximum and minimum thermovoltage values at $\Phi=0$ also showed the 180° out-of-phase modulation behavior, as expected on the basis of the Φ -dependent sub-band dispersion model. Thus, thermoelectric measurements could be used to reveal the electronic structure at the Fermi level for various quantum materials.

Limitations of the study

In previous studies, thermoelectric measurements were conducted by measurement of the Seebeck coefficient S. In the present study, however, we performed the thermoelectric measurement in terms of the thermovoltage because we did not have metallic thermometers for the NW. Consequently, to justify the T-dependence of the thermovoltage, we estimated ΔT at the examined temperatures with another device having the same geometrical conditions but with metallic thermometers.

STAR*METHODS

Detailed methods are provided in the online version of this paper and include the following:

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Supplemental information can be found online at https://doi.org/10.1016/j.isci.2022.105691.

ACKNOWLEDGMENTS

This work was supported by the National Research Foundation funded by the Korea Government (Grant Nos. 2016R1A5A1008184, 2018R1A2A1A05078440, 2018R1A3B1052827, 2021R1A2C3012612, 2020R1A6A1A030 47771, 2022M3H4A1A04074153, 2020R1A2C1011000) and the Korea Research Institute of Standards and Science (KRISS-2022-GP2022-0001). This work was also supported by Korea Institute for Advancement of Technology (KIAT) grant funded by the Korea Government (MOTIE) (P0008458, The Competency Development Program for Industry Specialist).

AUTHOR CONTRIBUTIONS

M.B. and J.S. conceived the experiments. D.K. fabricated the device and performed the measurements with B.K. D.Y. grew the Sb-doped Bi_2Se_3 nanowires and Y.D. helped with the analysis. All authors discussed the results and contributed to the writing of the manuscript.

DECLARATION OF INTERESTS

The authors declare no competing interests.

Received: August 2, 2022 Revised: November 9, 2022 Accepted: November 28, 2022 Published: January 5, 2023

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STAR*METHODS

KEY RESOURCES TABLE

REAGENT or RESOURCE	SOURCE	IDENTIFIER
Chemicals, peptides, and recombinant proteins		
Bi ₂ Se ₃ powder (99.999%)	Alfa Aesar	13126
Sb powder (99.999%)	Alfa Aesar	14640
Se powder (99.999%)	Alfa Aesar	10603
Ti pellets	ITASCO	7440-32-6
Au pellets	ITASCO	7440-57-5
Software and algorithms		
Origin 2019b	Origin Lab	https://www.originlab.com/

RESOURCE AVAILABILITY

Lead contact

Further information and requests for resources should be directed to and will be fulfilled by the lead contact, Dr. Myung-Ho Bae (mhbae@kriss.re.kr).

Material availability

This study did not generate new unique reagents.

Data and code availability

- Data reported in this paper will be shared by the lead contactupon request.
- There is no dataset or code associated with this work.
- Gate-dependent magnetoconductance (magnetothermovoltage), Temperature-dependent magneconductance (magnetothermovoltage)

EXPERIMENTAL MODEL AND SUBJECT DETAILS

This study does not use experimental methods typical in the life sciences.

METHOD DETAILS

Synthetic method

 $Bi_{2-x}Sb_xSe_3$ nanoribbons (x ranging from 0.15 to 0.4) were grown by a chemical vapor deposition (CVD) method in a Lindberg Blue M tube furnace. Bi_2Se_3 powder (99.999%, Alfa Aesar) mixed with Sb powder (99.999%, Alfa Aesar) was placed at the center of the tube, whileSe pellets (99.999%, Alfa Aesar) were placed upstream at 16 cm from the center. A silicon substrate coated with a 10 nm Au film was placed downstream 14 cm from the center. During the 5 h of growth time, the temperature of the furnace was maintained at 680 °C, Argon flow rate was kept at a 150 sccm, and the pressure was maintained at room pressure. The furnace was then cooled down to room temperature over approximately 3 h.

Device fabrication

Sb-doped Bi_2Se_3 NWs were synthesized on a Si substrate in a tube furnace via the chemical vapor deposition method. A selected Sb- Bi_2Se_3 NW was mechanically transferred using a tungsten tip onto a Si substrate covered with a 290 nm-thick SiO_2 layer. For the electrical measurement, we deposited contact metal Ti/Au (thickness: 160/50 nm) using electron (e)-beam lithography and e-beam evaporation. Before the e-beam evaporation, e-beam resist residue and the native oxide layer on the surface of the NWs were removed using an O_2 plasma asher (power: 200 W, time: 80 s) and by dipping into a 6:1 buffered oxide etchant for 10 s, respectively.

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Measurements

Electrical measurements for the NWs were performed using the two-probe scheme with a lock-in system (frequency: $21.77 \, \text{Hz}$, ac current: $50 \, \text{nA}$) with the sample in a liquid $^4 \text{He}$ system equipped with a $9 \, \text{T}$ magnet. Thermovoltage measurements were conducted with a nanovoltmeter in DC mode with a DC heater voltage.