

Superconducting Quantum Interference Devices Made of Sb-doped Bi_2Se_3 Topological Insulator Nanoribbons

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Abstract

We report the fabrication and characterization of superconducting quantum interference devices (SQUIDs) made of Sb-doped Bi_2Se_3 topological insulator (TI) nanoribbon (NR) contacted with PbIn superconducting electrodes. When an external magnetic field was applied along the NR axis, the TI NR exhibited periodic magneto-conductance oscillations, the so-called Aharonov-Bohm oscillations, owing to one-dimensional subbands. Below the superconducting transition temperature of PbIn electrodes, we observed supercurrent flow through TI NR-based SQUID. The critical current periodically modulates with a magnetic field perpendicular to the SQUID loop, revealing that the periodicity corresponds to the superconducting flux quantum. Our experimental observations can be useful to explore Majorana bound states (MBS) in TI NR, promising for developing topological quantum information devices.

Keywords: Topological insulator nanoribbon, superconducting quantum interference device, Aharonov-Bohm oscillations, Fraunhofer pattern, Majorana bound state

1. Introduction

Topological insulators (TIs) are bulk insulators having metallic surface states that are topologically protected by time-reversal symmetry [1]. The topological surface states (TSSs) are not subjected to backscattering by nonmagnetic impurities because the spin of the surface electrons are perpendicularly aligned to their translational momentum [2]. This spin-momentum locking property leads to a highly coherent quantum transport, making TIs promising candidates for quantum information devices [3]. For TI nanoribbon (NR) structure, the TSSs are modified as the one-dimensional (1D) subbands (or 1D surface modes) owing to the quantum confinement effect along the circumference of the NR, combined with the Aharonov-Bohm (A-B) phase and Berry's phase of π [4, 5]. Thus, under an applied external magnetic field along the NR axis, the magneto-conductance of TI NR oscillates with magnetic flux threading the cross-sectional area of the NR, the so-called A-B oscillations in TI NR. The A-B oscillations provide an evidence for the TSS in TI NR [6-9].

When TI materials are combined with conventional *s*-wave superconductors, they are expected to form a topological superconducting state hosting the Majorana bound state (MBS) [10], which is a building block for developing topological quantum information technology [11]. After the first observations of supercurrent through TI materials [12, 13], nonlocal Fraunhofer-like pattern [14] and macroscopic quantum tunneling behavior [15] have been observed in TI-based Josephson junctions (JJs). As an evidence for the MBS, abnormal Shapiro steps due to 4π -periodic current-phase relation (CPR) have been reported in TI-based JJs [16, 17]. It would be more plausible to directly measure such CPR using a dc superconducting quantum interference device (SQUID) consisting of two parallel JJs [18]. So far, TI-based SQUID has been studied using micro flakes or thin film of TI [19-22]. TI NR with reduced dimensions, however, is more promising for exploring MBS in TI, which results from an enhanced surface-to-volume ratio in NR.

There are three requisites for successful development of topological SQUID based on TI NR. Firstly, TSS should be guaranteed to exist in TI NR [23]. To confirm the existence of the 1D helical surface modes in TI NR, it is necessary to demonstrate A-B oscillations with the axial magnetic field in the normal state. Secondly, highly transparent contact should be formed at the interface between TI NR and the superconducting electrode to induce strong superconducting proximity effect, which is essential to form MBS. Lastly, the superconductivity of the superconducting electrode should be maintained until the magnetic flux through the cross-sectional area of TI NR reaches half flux quantum, $\Phi = h/2e$, where h is the Planck's constant, and e is the electric charge. This criterion is required to form a gapless helical 1D mode in TI NR [4].

In this work, we present an experimental study of hybrid SQUIDs made of Sb-doped Bi_2Se_3 TI NR and PbIn alloy superconductor. Our TI NR devices exhibited A-B oscillations with magnetic field along the NR axis in the normal state, which is attributed to the formation of 1D helical surface states in TI NR. Under an external magnetic field perpendicularly applied to the SQUID loop, periodic oscillation of the critical current was observed with a magnetic-flux periodicity of $h/2e$ in the loop. At high magnetic fields, the Fraunhofer pattern was superimposed on the SQUID oscillations owing to critical current modulations of individual JJs consisting of the SQUID. These results indicate that the TI NR-based SQUID can provide a promising new platform for exploring MBS and developing topological quantum computation technology.

2. Experiments

Sb-doped Bi_2Se_3 NRs [24] were synthesized via a chemical vapor deposition method in a horizontal tube furnace (Lindberg/ Blue M). Mixed Bi_2Se_3 powder (100 mg, 99.999 %, Alfa Aesar) and Sb powder (5-30 mg, 99.999 %, Johnson Matthey Inc.) were placed at the center of the tube furnace. Se pellets (200 mg, 99.999 %, Johnson Matthey Inc.) were placed upstream at a distance of 16 cm. A Si substrate with a 10 nm-thick gold film layer deposited by electron beam evaporation was

placed 7 cm downstream as the growth substrate. The tube was initially pumped down to a base pressure of about 40 mTorr and then filled with Ar gas (99.995 %) to ambient pressure. Under the flow of Ar gas at 150 sccm, the furnace was heated to 680 °C in 8 minutes and was maintained at the peak temperature for 5 hours. After that, the furnace was cooled to room temperature over approximately 3 hours. The representative scanning electron microscopy (SEM) image of the Sb-doped Bi₂Se₃ NRs after the growth process is displayed in Fig. 1a. The inset showed that the NR had a rectangular cross section. The widths (w) of the NRs used for this experiment ranged from 370 to 500 nm and the thicknesses (t) from 120 to 230 nm (see Table 1). The overall length of the TI NR was up to tens of micrometers. Energy-dispersive X-ray (EDX) spectroscopy for the NRs revealed that the atomic percentages (at. %) for Bi, Sb, and Se were 35.2 ± 1.6 , 6.5 ± 1.3 , 58.3 ± 1.1 , respectively, indicating the NR composition as (Bi_{0.84}Sb_{0.16})₂Se₃ (not shown here).

After the growth of Sb-doped Bi₂Se₃ NRs, individual NRs were mechanically transferred to a highly n -doped silicon substrate covered by a 290 nm-thick SiO₂ layer with pre-patterned Ti/Au bonding pads. Source and drain electrodes were patterned by standard electron-beam lithography followed by electron-beam evaporation of 300 nm-thick PbIn [25]. A 10 nm-thick Au film was used as a capping layer to avoid oxidation. The PbIn alloy source was made using a mixture of Pb and In pellets with a weight ratio of 1:1. Before the metal deposition, the NR surface was cleaned by oxygen plasma treatment to remove the electron-beam resist residues that could remain. Then, the NR was treated using a 6:1 buffered oxide etch (BOE) for 7 s to remove a native oxide layer that was assumed to be present. Typical SEM image of TI NR-based SQUID after completion of the device fabrication process is shown in Fig. 1b. The PbIn electrodes showed a superconducting transition at temperature $T_c = 6.8$ K, while the critical magnetic field was estimated to be $H_{c,perp} = 0.74$ T ($H_{c,para} = 1.2$ T) under a perpendicular (parallel) magnetic field [26]. The electrical transport properties of the TI NR-based SQUIDs were measured using a closed-cycle ⁴He cryostat (Seongwoo Instruments Inc.) and ³He refrigerator (Cryogenic Ltd.) which have a base temperature of 2.4 and 0.3 K, respectively.

For low-noise measurement, low-pass RC and π filters were connected in series with the measurement leads [27]. The device geometry and characteristic parameters are summarized in Table 1.

3. Results and discussion

When the magnetic field is perpendicularly applied to the surface of TI, the breaking of time-reversal symmetry results in negative magneto-conductance (MC) near the zero magnetic field, which is interpreted in the framework of the weak anti-localization (WAL) effect [28]. Since the WAL effect is due to a two-dimensional surface state, only the normal component of magnetic (B) field, $B\sin\theta$, contributes to the negative MC behavior, where θ is the angle of the magnetic field relative to the substrate. Thus, the MC curves obtained at different angles were merged into a single one when those were replotted against $B\sin\theta$ [9, 29]. Figure 1c shows the MC curves, $\Delta\sigma = \sigma(B) - \sigma(0)$, of sample **D1** contacted with Ti(10 nm)/Au(250 nm) electrodes. The width and thickness of the NR were $w = 607$ nm and $t = 90$ nm, respectively, while the channel length was $L = 4.89$ μm . All MC curves observed at different θ 's exhibited a cusp-like feature near zero magnetic field and merged into a single one when those were plotted with $B\sin\theta$, indicating the surface effect was dominant over the negative MC behavior.

According to the Hikami–Larkin–Nagaoka (HLN) theory [28], the WAL effect in two-dimensional (2D) limit can be expressed by

$$\Delta\sigma_{2D} = \alpha \frac{e^2}{2\pi^2\hbar} \left[\ln\left(\frac{\hbar}{4eB_n L_\phi^2}\right) - \Psi\left(\frac{1}{2} + \frac{\hbar}{4eB_n L_\phi^2}\right) \right], \quad (1)$$

where α is the dimensionless parameter corresponding to 0.5 for a single conducting channel, $\hbar$ is the reduced Planck's constant, B_n is the normal component of the magnetic field, Ψ is the digamma function, and L_ϕ is the phase coherence length. Our experimental MC data were fitted well by the

Eq. (1) with $\alpha = 1.35$ and $L_\varphi = 447$ nm at $T = 3.1$ K (see solid line in Fig. 1c). The obtained value of L_φ , which was similar to those previously reported using Bi_2Se_3 [30, 31], satisfied the 2D limit condition ($w > L_\varphi$). The value of α exceeded the expected value of 1, which is due to the two decoupled conducting channels at the top and bottom surfaces of TI sample. This difference can be attributed to an additional Rashba-split trivial 2D channel [32] or coherent transport in the bulk [30].

When we applied the axial magnetic field, B_{axial} , along the NR axis of device **D2**, the magnetoresistance (MR) oscillated with a period of $\Delta B_{\text{axial}} = 0.17 \pm 0.01$ T, as shown in Fig. 1d. As the dimensions of the NR were given by $w = 400$ nm and $t = 79$ nm, the magnetic-flux period corresponded to $\Phi_{\text{period}} = wt \times \Delta B_{\text{axial}} = 1.3 \hbar/e$. This Φ_{period} value was close to the magnetic flux quantum in the normal state, $\Phi_0 = \hbar/e$, as expected in the A-B oscillation theory of TI NR [4, 5]. The difference can be explained by the native oxide layer on the surface of TI NR, where the layer thickness is estimated to be about 5 nm [8, 33]. Then, we obtained $\Phi_{\text{period}} = 1.1 \hbar/e$, which was considerably closer to the theoretical expectation. Our observation of the A-B oscillations confirmed the existence of 1D surface modes [4] in our Sb-doped Bi_2Se_3 NR.

Schematic and dimensions of TI-NR SQUID's are shown in the inset of Fig. 2a and Table. 1, respectively. Two JJs, fabricated on top of the Sb-doped Bi_2Se_3 TI NR with PbIn superconducting electrodes, were connected to each other to form a superconducting loop [25, 26]. Geometrical dimensions of our devices are listed in Table 1. Figure 2a shows the current–voltage (I – V) characteristic curves of device **Sq2** measured at $T = 2.4$ K. The maximum supercurrent, so-called the critical current, was obtained as $I_C = 0.40 \mu\text{A}$ in the absence of magnetic field. When the magnetic field was perpendicularly applied to the SQUID loop, I_C gradually decreased and disappeared when the magnetic flux through the loop reached $\Phi = 0.5 \Phi_S$, where $\Phi_S = \hbar/2e$ is the superconducting flux quantum. For $0.5 < \Phi/\Phi_S < 1$, I_C increased and returned to its original value with $I_C(\Phi_S) = I_C(0)$. Figure 2b, a color plot of differential resistance dV/dI , shows the periodic modulation of I_C as a

function of B and Φ . In this figure, the dark regions corresponds to the superconducting state with $dV/dI = 0$ and oscillates with the periodicity of Φ_S and $\Delta B = 4.7$ Oe, which is a characteristic feature of the conventional SQUID [18]. The effective area for the magnetic flux was estimated to be $A_{\text{eff}} = 4.4 \mu\text{m}^2$ using a relation of $\Phi_S = \Delta B \times A_{\text{eff}}$, while the inner geometric area of the SQUID loop was $A = 1.71 \mu\text{m}^2$. This difference can be explained by the London penetration depth (λ_L), where the magnetic field penetrates the superconducting electrodes. We estimated $\lambda_L = 200$ nm for PbIn electrode from $A_{\text{eff}} = (L_S + 2\lambda_L) \times (W_S + 2\lambda_L)$, which was similar to the values obtained from previous work [34].

The periodic modulation of $I_C(\Phi)$ can be described by [35]

$$I_C(\Phi) = \left[(I_{C1} - I_{C2})^2 + 4I_{C1}I_{C2} \cos^2(\pi\Phi / \Phi_S) \right]^{1/2} \quad (2)$$

where I_{C1} and I_{C2} are the critical currents of two JJs consisting of TI-NR SQUID. The fitting results are shown in Fig. 2b (solid white lines) with parameters of $I_{C1} = 204$ nA and $I_{C2} = 198$ nA. From this, we can deduce that the device **Sq2** belongs to a symmetric case with $I_{C2}/I_{C1} = 0.97$. The self-inductance of the SQUID loop was estimated as $L_S \sim 3.5$ pH from the loop geometry, indicating that the screening parameter $\beta_L = 2\pi L_S I_0 / \Phi_S = 2.2 \times 10^{-3} \ll 1$, where $I_0 = (I_{C1} + I_{C2})/2$ is the average critical current. Thus, the self-inductance effect was negligible in our experiment.

The $I_C(\Phi)$ modulation can be reflected by voltage (V) modulation under a dc current bias I_{dc} , as shown in Fig. 2c. As the SQUID acts as a flux-to-voltage transducer, the output voltage V oscillates with the magnetic flux through the SQUID loop with a period of $\Phi_S = h/2e$ [18]. The oscillation amplitude increased with I_{dc} and became maximum near I_C . The maximum sensitivity of the TI-NR SQUID was obtained as $|\partial V / \partial \Phi| = 17 \mu\text{V} / \Phi_S$ (see Fig. 2d), which is comparable to previous studies of dc SQUID based on Bi_2Te_3 flake [19] or PbS nanowire [25].

Figure 3a displays the I - V curves obtained from the **Sq4** device at different magnetic fields and lower temperature, $T = 0.3$ K. The maximum I_C was given by $I_{C,\max} = I_C(\Phi = 0) = 7.2 \mu\text{A}$ in the absence of magnetic field, which was an order of magnitude larger than that of **Sq2**. I_C monotonously decreased with increasing Φ from 0 to $0.5 \Phi_S$. However, it was noted that a finite I_C remained even at $\Phi = 0.5 \Phi_S$, resulting in the absence of the supercurrent OFF state. The color plot of dV/dI vs. Φ , as shown in Fig. 3b, clearly displays the $I_C(\Phi)$ modulation and the existence of non-zero supercurrent at $\Phi = (n + 1/2) \Phi_S$, where n is an integer. The residual supercurrent, $I_{C,\min}$, can be attributed to a self-inductance screening effect or asymmetric JJ's in SQUID [35]. As the screening parameter was quite small ($\beta_L \ll 1$), the self-inductance effect was negligible in this experiment. Two different I_C 's for each JJ can be estimated from $I_{C1} = (I_{C,\max} + I_{C,\min})/2$ and $I_{C2} = (I_{C,\max} - I_{C,\min})/2$, respectively [35]. Thus, we obtained $I_{C1} = 4.8 \mu\text{A}$ and $I_{C2} = 2.4 \mu\text{A}$, respectively, which means $I_{C2}/I_{C1} = 0.50$. The **Sq4** device belongs to asymmetric dc-SQUID regime in contrast to **Sq2**. When plotting the Eq. (2) using the above estimates of I_{C1} and I_{C2} (see the solid white lines), it fitted well the $I_C(\Phi)$ modulation behavior in Fig. 3b.

The $V(\Phi)$ modulation of **Sq4** is shown in Fig. 3c with varying I_{dc} . The modulation periodicity was $\Phi_S = h/2e$ in terms of magnetic flux, while its amplitude was maximized near $I_{dc} \approx I_C$. Figure 3d depicts the SQUID sensitivity as a function of I_{dc} . The maximum value of $|\partial V/\partial \Phi|$ was about $152 \mu\text{V}/\Phi_0$, which is almost an order of magnitude larger than those of **Sq2** and in Ref.[19]. This difference can be explained by a larger $I_C R_N \sim 39.0 \mu\text{V}$ value of device **Sq4** in comparison with **Sq2**, where R_N means the normal-state resistance of SQUID. It should be noted that the maximum $I_C R_N$ value obtained from our TI-NR SQUID was still larger than that of TI flake-based SQUID's [21, 36], which is attributed to a relatively large superconducting gap of PbIn and formation of highly transparent contacts between TI NR and superconducting electrodes. Hence, we suggest that the superconducting proximity effect through TI NR would be more advantageous to explore MBS in TI.

When we applied higher magnetic field to **Sq2**, a beat-like interference pattern was observed in the $V(\Phi)$ modulation, as shown in Fig. 4a, where an envelope was superposed on the previous SQUID oscillations. The fast Fourier transform (FFT) analysis of the $V(\Phi)$ modulation (see the inset of Fig. 4a) revealed that there were two different frequencies centered at $\Delta B^{-1} = 0.011$ and 0.22 Oe^{-1} , corresponding to the periods of $\Delta B = 91$ and 4.6 Oe , respectively. Another device (**Sq4**) also showed similar behavior (not shown here). The latter mode with short periodicity was responsible for the SQUID oscillations in Fig. 2c, while the former one was attributed to the magnetic field-dependent I_C modulation of individual JJ's, the so-called Fraunhofer pattern. It is well known that I_C of JJ becomes minimum when the magnetic flux through the junction's lateral area reaches an integer multiple of magnetic flux quantum, i.e., $\Phi = n \Phi_S$ with an integer n [18]. Then the field interval between two successive peaks in the envelope, $\Delta B^* = 82 \text{ Oe}$, corresponds to a single flux quantum through the JJ in the SQUID. As a result, we have a relation of $\Phi_S = \Delta B^* (L_{JJ} + 2\lambda_L)w$, where $L_{JJ} = (L_1 + L_2)/2$ is the average junction length of two JJ's and w is the width of TI NR, resulting in $\lambda_L = 203 \text{ nm}$, which is consistent with previous result obtained from the low-field SQUID oscillations.

When we combine the Fraunhofer-type I_C modulation with the SQUID oscillation, Eq. (2) can be modified as follows [35]:

$$I_C(\Phi) = \left[(I_{C1} - I_{C2})^2 + 4I_{C1}I_{C2} \cos^2(\pi\Phi / \Phi_S) \right]^{1/2} \left| \sin\left(\frac{\pi\Phi_J}{\Phi_S}\right) \right| \left/ \left(\frac{\pi\Phi_J}{\Phi_S} \right) \right| \quad (3)$$

where $\Phi_J = (L_{JJ} + 2\lambda_L)wB$ is the magnetic flux through the single JJ. This new equation fits well the $I_C(B)$ modulation data obtained in the wider range of B field, as can be seen in Fig. 4b. Thus, the CPR study of TI-NR SQUID should consider the SQUID oscillations together with the I_C modulation effect of the individual junctions. Similar results can be found in other reports of TI-flake SQUID [20, 21].

In conclusion, we reported successful fabrication and characterization of SQUID's based on Sb-doped Bi₂Se₃ TI NR, contacted with PbIn superconducting electrodes. The angle-dependent WAL effect and the A-B oscillations confirm the existence of topological surface states in the TI NR. The SQUID oscillations with a periodicity of superconducting flux quantum, $h/2e$, have been investigated in terms of $I_C(B)$ and $V(B)$ modulations for both symmetric and asymmetric junction devices. Relatively large values of $I_C R_N$ product and the SQUID sensitivity indicates the formation of highly transparent superconducting contacts on TI NR. Our observations suggest that TI NR-based SQUID's are promising for exploring Majorana bound states in TI and developing topological quantum information devices.

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Figure Captions

Fig. 1. (a) SEM image of the Sb-doped Bi₂Se₃ NR. Inset: Tilted-SEM image of the NR. Scale bar: 500 nm. (b) Representative SEM image of the SQUID based on Sb-doped Bi₂Se₃ NR and PbIn superconducting electrodes. (c) MC, $\Delta\sigma = \sigma(B) - \sigma(0)$, curves at various magnetic field orientations, as a function of perpendicular magnetic field component, $B\sin\theta$ (sample **D1**). The solid line is the best fit to Eq. (1). (d) MR curve under the axial magnetic field along the NR axis, B_{axial} , measured at $T = 2.3$ K (sample **D2**). The arrows indicate resistance dips occurring at $B_{\text{axial}} = 0.08, 0.24, 0.42$ T.

Fig. 2. (a) Current–voltage (I – V) curves for different magnetic fluxes through the TI-NR SQUID loop at $T = 2.4$ K (sample **Sq2**). Inset: Schematic of TI-NR SQUID. (b) Color plot of dV/dI as a function of bias current and magnetic field (or Φ/Φ_S). The dark region indicates the supercurrent regime and the white solid line is a calculation result using Eq. (2). (c) Voltage modulation as a function of Φ/Φ_S under a dc current bias of $I_{\text{dc}} = 0.2$ μA (black), 0.4 μA (red), 0.6 μA (blue), 0.8 μA (green) and 1.0 μA (magenta). (d) Squid sensitivity as a function of I_{dc} . Error bars are indicated.

Fig. 3. (a) I – V curves of device **Sq4** at various magnetic fluxes and $T = 0.3$ K. (b) Color plot of dV/dI as a function of I_{dc} , B , and Φ/Φ_S . The white line presents calculation results using Eq. (2) considering I_C asymmetry. (c) Output voltage as a function of Φ/Φ_S with $I_{\text{dc}} = 2.8$ μA (black), 4.9 μA (red), 7.0 μA (blue), 7.7 μA (green) and 8.4 μA (magenta), respectively. (d) SQUID sensitivity as a function of I_{dc} .

Fig. 4. (a) Voltage modulation of **Sq2** as a function of B field at $T = 2.4$ K. Inset: FFT analysis of $V(B)$ modulation. (b) Magnetic field dependence of I_C of **Sq2**. The solid line is a fitting result using Eq. (3) (see text).

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Table 1. Physical parameters of the SQUIDs: w (t) is the width (thickness) of TI NR. L_1 and L_2 are the channel length of each Josephson junction. L_S and W_S are the length and width of the SQUID, respectively. T_{base} is the base temperature for I_C measurement.

Sample	w (nm)	t (nm)	L_1 (nm)	L_2 (nm)	L_S (μm)	W_S (μm)	I_C (μA)	$I_C R_N$ (μV)	T_{base} (K)
Sq1	506	200	157	121	1.34	3.43	0.45	3.2	2.4
Sq2	495	200	114	89	1.36	1.26	0.40	4.6	2.4
Sq3	414	235	290	257	1.59	1.41	2.60	14.8	0.3
Sq4	368	235	243	292	1.46	1.66	7.20	39.0	0.3
Sq5	391	121	260	260	1.30	1.70	0.10	1.6	2.5

Fig. 1

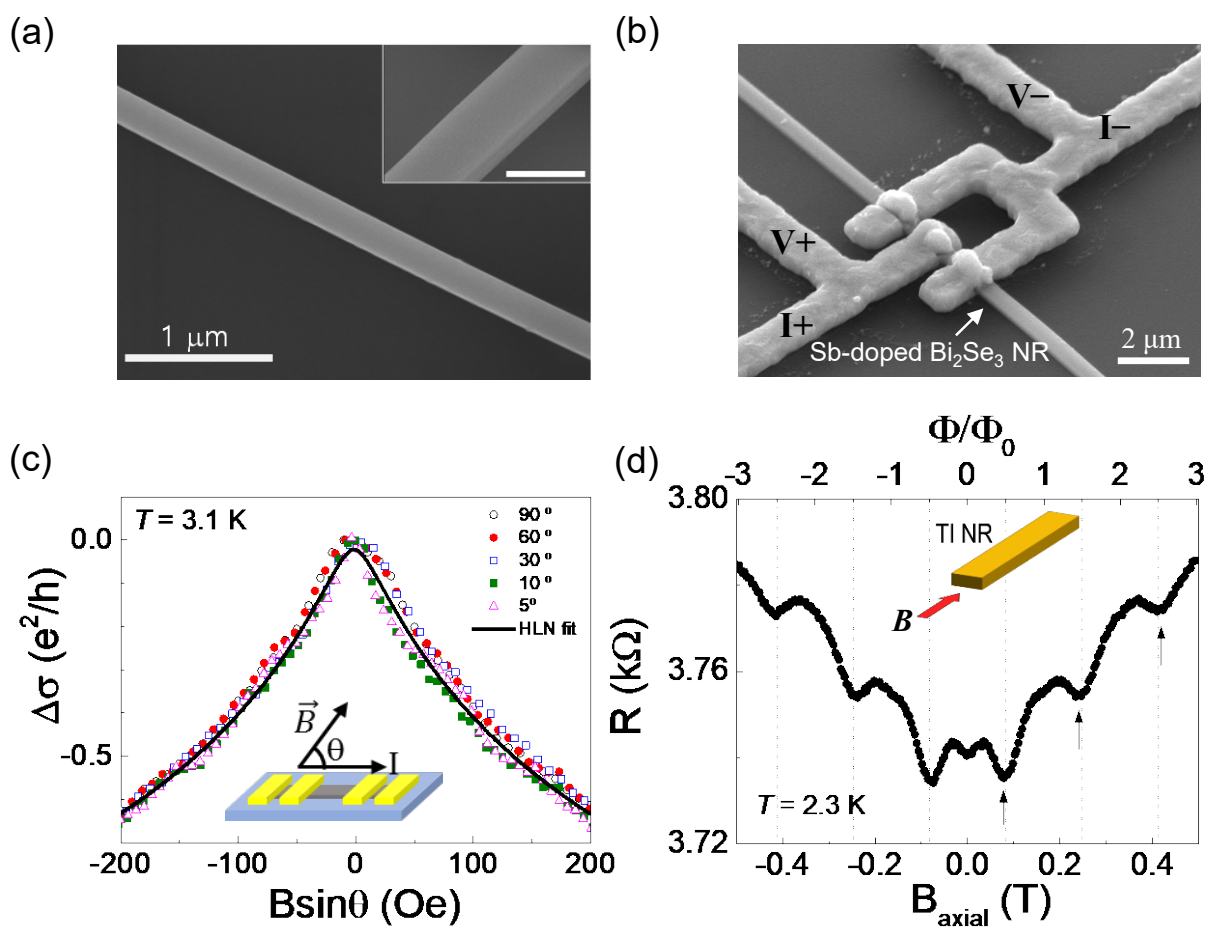


Fig. 2

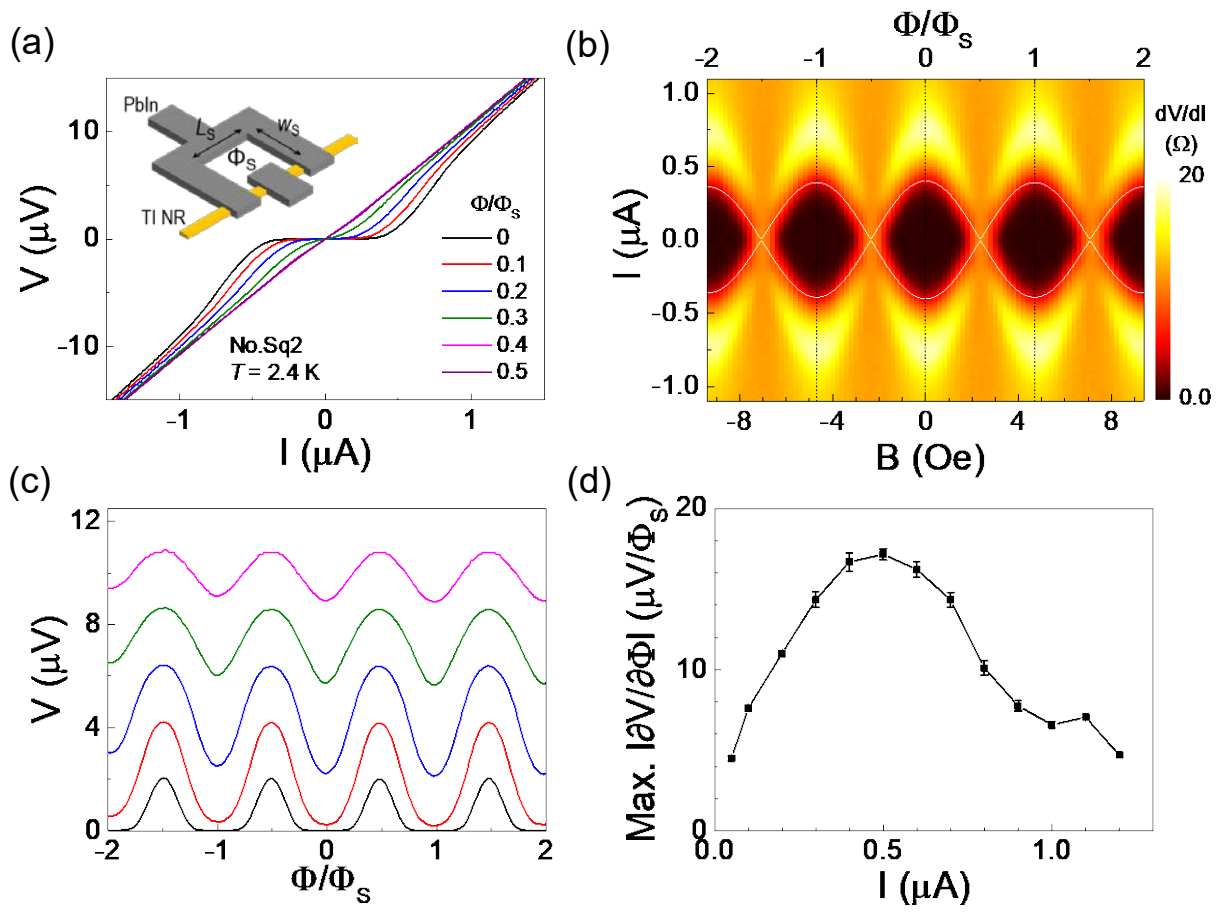


Fig. 3

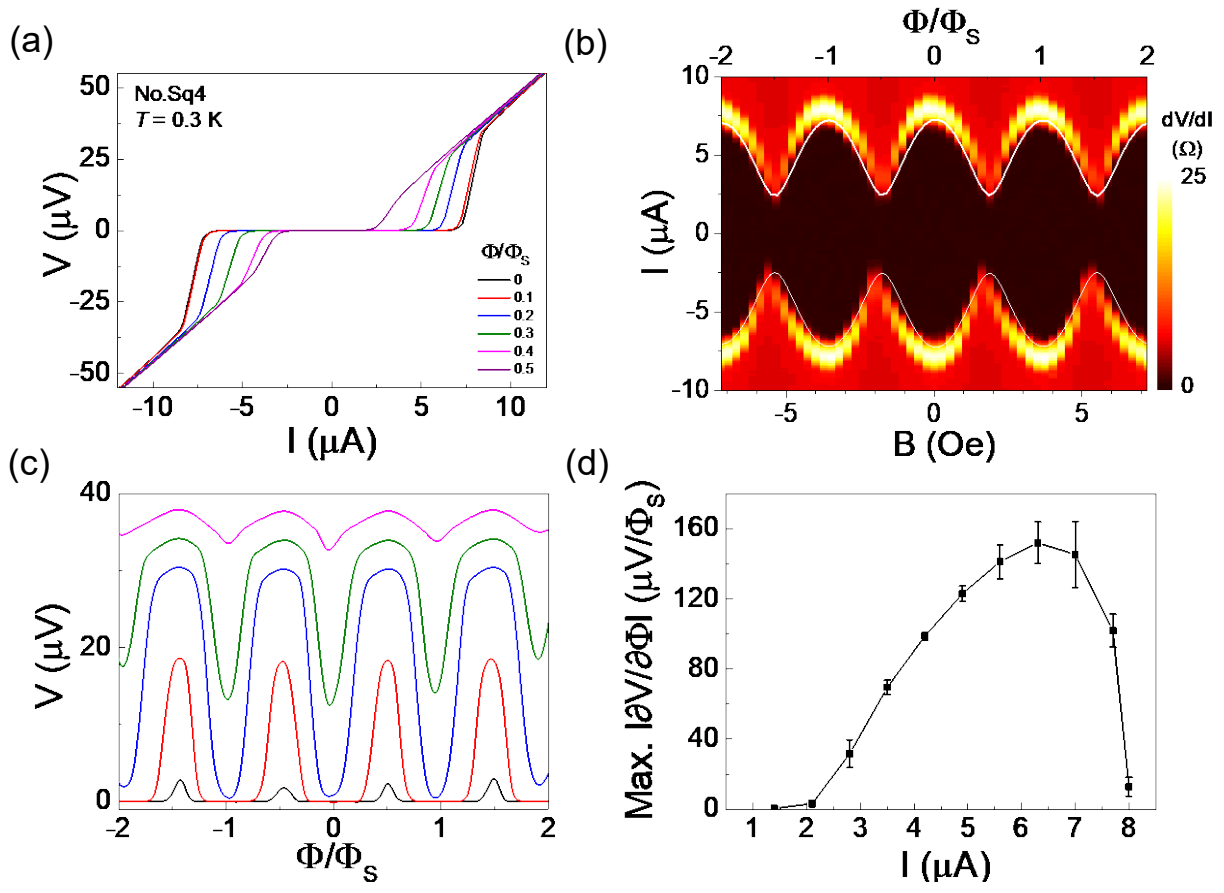


Fig. 4

