Gate-tunable $h/e$-period magnetoresistance oscillations in Bi$_2$O$_2$Se nanowires

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We report on the successful synthesis and low-temperature electron transport investigations of a new form of material—Bi$_2$O$_2$Se semiconducting nanowires. Gate-tunable 0- and $\pi$-$h/e$ ($h$ is the Planck constant and $e$ the elementary charge) periodic resistance oscillations in longitudinal magnetic field were observed unexpectedly, demonstrating novel quasiballistic, phase-coherent surface states in Bi$_2$O$_2$Se nanowires. By reaching a very good agreement between the calculated density of states and the experimental data, we clarified the mechanism to be the one-dimensional subbands formed along the circumference of the nanowire rather than the usually considered Aharonov-Bohm interference. A qualitative physical picture based on downward band bending associated with the complex band structure is proposed to describe the formation of the surface states.

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I. INTRODUCTION

New-type low-dimensional materials have attracted tremendous interest due to their intriguing properties and application potentials in next-generation electronics, optoelectronics, etc. Graphene, phosphorene, and transition-metal dichalcogenide are among the star materials [1–3]. However, drawbacks also exist in these materials, either lack of a bandgap, instable in air, or with only a moderate electron mobility. Recently, a new layered semiconductor Bi$_2$O$_2$Se has drawn ever-growing attention due to its superior electronic properties such as an ultrahigh electron mobility (∼2.8 $\times$ $10^5$ cm$^2$/Vs at 2 K), a tunable bandgap (∼0.8 eV), and being stable in ambient environment [4-21]. Large-scale Bi$_2$O$_2$Se thin films from monolayer to few layer have been successfully synthesized through a chemical vapor deposition method [4,17]. The existence of strong spin-orbit interaction [21] and high-performance field-effect transistors and optoelectronics [4,11,12,15,19,22] have also been experimentally demonstrated. And the ferroelectricity/ferroelasticty is further expected [8].

Given the fascinating features in the two-dimensional (2D) form of Bi$_2$O$_2$Se, it is natural to expect that the one-dimensional (1D) nanowire form of Bi$_2$O$_2$Se, with large surface-to-volume ratio, would retain some of the unique properties of its 2D counterpart. However, investigation of Bi$_2$O$_2$Se nanowires is still a mysterious veil at present. In this work, we report on the synthesis of high-quality single-crystalline Bi$_2$O$_2$Se nanowires by means of a gold-catalyzed vapor-liquid-solid (VLS) mechanism. Low-temperature electron transport measurements reveal $h/e$-period resistance oscillations as a function of longitudinal magnetic field ($h$ is the Planck constant and $e$ the elementary charge) and a gate-tunable $\pi$-phase shift. These unexpected observations demonstrate quasiballistic, phase-coherent electron transport of surface states in Bi$_2$O$_2$Se nanowires. A very good agreement between the calculated density of states and the experimental results—the double modulation of the resistance oscillations by gate voltage and magnetic field—clarifies the mechanism to be the 1D subbands formed along the circumference of the nanowire rather than the usually considered Aharonov-Bohm interference. A qualitative picture associated with the complex band structure is further proposed to interpret the formation of surface electron accumulation based on downward band bending.

II. CHARACTERIZATION

Bi$_2$O$_2$Se nanowires were synthesized via a gold-catalyzed VLS mechanism on Si wafers in a horizontal tube furnace with Bi$_2$Se$_3$ powder as the source material. A detailed phase equilibrium process can be found in the Supplemental Material [23]. After growth, the wafer appeared to be gray as an evidence of nanowire production. Figure 1(a) shows the scanning electron microscopy (SEM) image of the as-synthesized Bi$_2$O$_2$Se nanowires, which have a length of 5–10 $\mu$m and a diameter of 80–250 nm.

To characterize the basic crystal structure, we have carried out an extensive transmission electron microscopy (TEM) study on the as-synthesized samples. Figure 1(b) shows a bright-field TEM image and the corresponding diffraction pattern taken along the [010] zone axis direction of one typical nanowire. All the main diffraction spots in these patterns can be well indexed using the expected tetragonal unit cell with lattice parameters of about $a = 0.389$ nm, $c = 1.22$ nm (space group $I4/mmm$) [4], which agree well with the x-ray powder diffraction result as shown in the Supplemental Material [23]. A better and very clear view of the atomic structure for...
FIG. 1. (a) SEM image of the as-synthesized Bi$_2$O$_2$Se nanowires on Si wafer. Scale bar 5 μm. The ball on the top of the nanowire is the Au catalyst. (b) A bright-field TEM image and the corresponding diffraction pattern taken along the [010] zone axis direction of one typical nanowire. (c), (d) The HRTEM images taken from the square-enclosed A and B regions in (b), respectively. The inset in (c) presents the FFT spectrum.

The HRTEM images in Fig. 1(b) also indicate the presence of a zigzag structure along the edge of the as-prepared nanowires. Figure 1(d) illustrates the HRTEM image taken from region B, demonstrating that there is no obvious planar defect in such areas. The layered structural morphologies are very likely caused by a layer-by-layer growth mode, in which there could be a relative shift in the $ab$ plane during the crystal growth due to the specific synthesis condition. These spectroscopic characterizations demonstrate that the as-synthesized Bi$_2$O$_2$Se nanowires are of high quality.

III. GATE TUNABLE OSCILLATIONS

For electron transport measurements, Bi$_2$O$_2$Se nanowires were mechanically transferred onto a highly doped Si substrate with a 300-nm-thick SiO$_2$ layer used for applying a back-gate voltage. Ti/Au (5 nm/150 nm) contacts were deposited using a standard lithography technique. Two-terminal measurements were carried out with a temperature down to 1.8 K and a magnetic field up to 7 T.

Figure 2(a) shows a sketch of the measurement setup. The large surface-to-volume ratio of the nanowire provides excellent geometries for probing the transport properties of surface states, if present, as indicated by the red arrows in Fig. 2(a). Figure 2(b) presents an atomic force microscopy (AFM) image of device No. 1 with two Ti/Au contacts. Figure 2(c) displays the two-terminal resistance versus magnetic field applied along the axis of the nanowire and back-gate voltage $V_g$ at 1.8 K for device No. 1. In order to identify the oscillating features more clearly, we smoothed the data of Fig. 2(c) and extracted the second-order derivatives, i.e., the phase diagram, as shown in Fig. 2(d).

One obvious characteristic of Figs. 2(c) and 2(d) is that the resistance oscillates as a function of $B$. The periodic oscillations in magnetic field along the nanowire axis [see representative curves in Figs. 3(a) and 3(e)] indicate a well-defined surface-conducting channel to pick up flux [24]. Such periodic oscillations with a period of $h/e$ or $h/2e$ have been observed in many 1D systems with a surface-conducting layer [24], such as metallic cylinders [25], carbon nanotubes [26], core/shell nanowires [27,28], semiconducting nanowires [29], topological insulator nanoribbons [30,31], Dirac semimetal nanowires [32], etc. Note that only a few small-band-gap semiconductors carry intrinsic electron-accumulated surface states, such as InN, InAs, and ZnO [33–35]. In addition, per-
In a mesoscopic ring, the mechanism of periodic resistance oscillations is relatively straightforward. The \( h/2e \)-period oscillations, named Altshuler-Aronov-Spivak oscillations \[39\], result from the interference of partial waves through two different possible paths from one terminal to the other one, associated with the interference of partial waves through two different possible paths from one terminal to the other one, called Aharonov-Bohm (AB) oscillations \[40\].

For cylindrical shell conductors, \( h/2e \) oscillations are usually observed at low magnetic fields and die out and disappear at high magnetic fields due to the requirement of time-reversal symmetry \[41\]. On the contrary, the \( h/e \) oscillations can survive at both high and low magnetic fields, without the need of time-reversal symmetry. The periodic oscillations in Figs. 2(c) and 2(d) survive at high magnetic fields \((\pm 7 \, T)\), and thus they are consistent with \( h/e \) oscillations. However, the \( h/e \) oscillations require ballistic transport \[28,29\]. In such cases, although it is still called AB oscillation \[40\], the underlying mechanism transits to the modulation of density of states by magnetic field rather than AB interference. The ballistic transport of electrons along the circumference forms 1D sub-bands with different angular momentum which is regulated by the penetrated magnetic field \[28,29,42,43\], as explained later. The competition between the \( h/e \) and \( h/2e \) components depends on the strength of the disorder and the size of the cylinder \[29,32,44\].

To analyze the oscillations as a function of longitudinal magnetic field, we take typical line cuts at \( V_g = -54 \, V \) from Figs. 2(c) and 2(d) (marked by the black arrows), as shown in Figs. 3(a) and 3(c), respectively. The periodic oscillations can be clearly recognized. Figure 3(b) shows the fast Fourier transform (FFT) spectrum of the \( R \) vs \( B \) curve in Fig. 3(a). A prominent peak can be found at a frequency of \( f = 1.46 \, T^{-1} \) as depicted by the triangle. The low-frequency peaks correspond to the large-scale background variation of Fig. 3(a), presumably due to universal conductance fluctuations (UCFs) of the bulk states. These false peaks can be readily suppressed by taking the FFT spectrum of the second-order derivative \( d^2R/dB^2 \), a method commonly applied to separate the oscillatory part from background, as shown in Fig. 3(d). The \( h/e \)-periodic oscillations are thus associated with an area of \( S = f \times h/e = 6050 \, \text{nm}^2 \), where \( S \) is the cross-section area of the nanowire. Taking the nearly elliptical cross-section dimensions, a width of 140 nm and a height of 95 nm, we need to assume a depth \( d \) of the surface states \[30\] to match the required area. \( d \sim 6 \, \text{nm} \) was deduced from \( \pi \times (65-d) \times (42.5-d) = S \) after subtracting the surface undulation of 5 nm due to zigzag structures. Note that the dimensions might be overestimated from SEM or AFM images. Nevertheless, the \( h/2e \) oscillations at a double frequency \( 2f \) were invisible. Combining the dominant \( h/e \) oscillations and the superposition of UCFs, we conclude that our \( \text{Bi}_2\text{O}_2\text{Se} \) nanowires are in the quasiballistic regime \[27,29,45\].

Another noticeable characteristic of Figs. 2(c) and 2(d) is that the resistance changes between a peak and a valley alternatively along gate voltage (even without magnetic field). Figure 3(e) depicts two curves of \( d^2R/dB^2 \) vs \( V_g \) taken out from Fig. 2(d) as marked by the black and red dashed lines, respectively. The oscillating part from background, as shown in Fig. 3(d). The \( h/e \)-periodic oscillations are thus associated with an area of \( S = f \times h/e = 6050 \, \text{nm}^2 \), where \( S \) is the cross-section area of the nanowire. Taking the nearly elliptical cross-section dimensions, a width of 140 nm and a height of 95 nm, we need to assume a depth \( d \) of the surface states \[30\] to match the required area. \( d \sim 6 \, \text{nm} \) was deduced from \( \pi \times (65-d) \times (42.5-d) = S \) after subtracting the surface undulation of 5 nm due to zigzag structures. Note that the dimensions might be overestimated from SEM or AFM images. Nevertheless, the \( h/2e \) oscillations at a double frequency \( 2f \) were invisible. Combining the dominant \( h/e \) oscillations and the superposition of UCFs, we conclude that our \( \text{Bi}_2\text{O}_2\text{Se} \) nanowires are in the quasiballistic regime \[27,29,45\].

In the following we explain the above observations consistently within the theoretical framework of ballistic electron transport through cylindrical surface states penetrated by a magnetic flux \[28,29,32,46\]. The motion of surface electrons along the cylindrical shell can be regarded as a combination

\[\text{FIG. 3}. \ \text{(a) } R \text{ vs } B \text{ curve at } V_g = -54 \, V \text{ taken out from Fig. 2(c).} \ \text{(b) FFT of } R \text{ vs } B \text{ curve in (a).} \ \text{(c) Second-order derivative } d^2R/dB^2 \text{ of the smoothed } R \text{ vs } B \text{ curve in (a) and its FFT pattern is shown in (d).} \ \text{The triangles in (b) and (d) denote the main peak at } 1.46 \, T^{-1}. \ \text{(e) Two line cuts of } d^2R/dB^2 \text{ vs } V_g \text{ taken out from Fig. 2(d) as marked by the black } (B = -1.13 \, T) \text{ and red } (B = -0.73 \, T) \text{ dashed lines, respectively.} \ \text{(f) Subbands formed at the circumference of the nanowire as a function of longitudinal magnetic flux } \Phi, \text{ without level broadening (solid gray lines) and with level broadening (DOS represented by bluish background colors).} \ \text{The green dashed line indicates } \Phi_1 \text{-period (irregular) oscillations with (without) level broadening, while the bars show } \Phi_2 \text{-period oscillations for both cases.} \ \text{A } \pi \text{-phase shift exists both in the } V_g \text{ dependence of oscillations (i.e., along the dashed black and red line cuts) and in the } \Phi \text{ dependence of oscillations (i.e., line cuts along the black and red arrows).} \]
of a circular component and a free component along the nanowire axis \( z \). The Hamiltonian can be written as

\[
\hat{H} = \frac{1}{2m^* r_0^2} \left( L_z - \frac{1}{2} e B r_0^2 \right)^2 - \frac{\hbar^2}{2m^*} \frac{\partial^2}{\partial z^2},
\]

where \( L_z \) denotes the angular momentum operator and \( r_0 \) the radius of the nanowire. With \( \pi r_0^2 B = \Phi \) as the magnetic flux through the circular loop and \( \Phi_0 = h/e \), we find the energy eigenvalues as

\[
E_l = \frac{\hbar^2 k_0^2}{2m^*} + \frac{\hbar^2}{2m^* r_0^2} \left( l - \frac{\Phi}{\Phi_0} \right)^2.
\]

The first term describes the kinetic energy for the motion along \( z \). The second term, where the angular momentum quantum number \( l = 0, \pm 1, \pm 2 \ldots \), represents a series of parabolas as a function of \( \Phi \) shifted to each other by \( \Phi_0 \). The energetic spectrum for \( k_0 = 0 (E_0) \) with each parabola belonging to a specific angular momentum state is illustrated in Fig. 3(f) by the solid gray lines. However, in contrast to the experimental observations shown in Figs. 2(c) and 2(d), \( h/e \)-periodic oscillations along \( B \) are only expected at certain discrete energies, as marked by the black bars on the right in Fig. 3(f). And irregular oscillations dominate for most of the energies, as represented by the horizontal green dashed line in Fig. 3(f). We argue that level broadening of the subbands presumably arising from thermal broadening, inelastic scattering, and/or finite thickness of the surface channel (uncertainty of flux) should be taken into account.

From the red line of Fig. 3(e), we take one full oscillation from \( V_g = -64 \) V to 12 V to estimate the Fermi-level position. Under the assumption of finite bulk contributions, this gate-voltage range corresponds to a Fermi level shift of 13.2 meV (subband separation) [23]. Using \( E_0(\Phi = 0) = \frac{h^2 k_0^2}{2m^*} \), we extract the Fermi level at \( V_g = -64 \) V to be 299.4 meV (\( l = \pm 46 \)), and at \( V_g = 12 \) V to be 312.6 meV (\( l = \pm 47 \)). The colored background of Fig. 3(f) depicts the calculated density of states (DOS) by approximating level broadening as a Lorentz shape distribution. Clearly, the double modulation by \( B \) and \( V_g \) gives rise to the \( \Phi_0 \)-period DOS (resistance) oscillations along \( B \) as well as the \( \pi \)-phase shift. When \( V_g \) tunes the Fermi level, the DOS (resistance) also oscillates at a given \( B \). The opposite phase between the black and red dashed lines can be apparently recognized, as shown by the two experimental curves in Fig. 3(e) accordingly. The \( \pi \)-phase shift, i.e., the 0- and \( \pi\cdot h/e \) oscillations, as marked by the black (DOS peak at \( \Phi = 0 \)) and red (DOS valley at \( \Phi = \pi \)) arrows in Fig. 3(f), respectively, can be immediately identified. Note that the DOS map in Fig. 3(f) represents a qualitative interpretation of the data. Details of the extraction of the DOS color map can be found in the Supplemental Material [23].

![Graph](image)

**FIG. 4.** (a) Resistance \( R \) of device No. 2 as a function of both \( B \) and \( V_g \) at 1.8 K. (b) \( R \) vs \( B \) curves at different \( V_g \) with an offset for clarity. The black dashed line guides the \( \pi \)-phase shift between different curves.

V. DISCUSSION

Next we propose a qualitative physical picture to describe the formation of surface-conducting channels on Bi\(_2\)O\(_2\)Se. For semiconductors, the surface Fermi level \( E_F \) tends to sit at a universal value of \( \sim 4.9 \) eV [47] or \( \sim 4.5 \) eV (with hydrogen adsorbed) [33] below the vacuum level to reach charge neutrality. This charge neutrality level, called branch point energy (\( E_B \)), represents the separation of donor-type (below) and acceptor-type (above) states at the surface/interface [48]. It also corresponds to the average midgap energy weighting the entire Brillouin zone in the complex band structures [49]. For a few small-band-gap semiconductors with an extremely low conduction-band minimum (\( E_C \)) and a small effective mass, the density of states around the \( \Gamma \) point is negligible compared to other band edges at the boundaries of the Brillouin zone. Therefore, the average midgap energy \( E_B \) could be higher than \( E_C \), such as in InN, InAs, and ZnO [33–35]. The direct result is the existence of donor-type states at the surface and thus the downward band bending and accumulation of electrons near the surface.

Regarding Bi\(_2\)O\(_2\)Se, the band structures indeed show a low \( E_C \) at \( \Gamma \) point, \( \sim 0.8 \) eV above the valence band maximum \( E_v \), with a relatively small effective mass of 0.14\( m_0 \) (\( m_0 \) is the free electron mass) [4]. Consequently, it is possible that \( E_B \) is higher than \( E_C \). Applying similar approximations as for small gap III-V semiconductors [49], the midgap energy at the \( X \) point, the zone face center, indeed crosses the conduction band at the \( \Gamma \) point [4,7,8,10,50]. Due to the variation between the band structures of Bi\(_2\)O\(_2\)Se obtained by different methods [4,7,8,10,15,50–52], however, we seek to propose a possible qualitative picture in the current work. Assuming that \( E_B \) is higher than \( E_C \) and a position of \( E_F \) in the gap, the band alignment near the surface is illustrated in Fig. 5. The unoccupied donor-type states at the surface are positively charged, balanced by the downward band-bending-induced space charge. Near the surface, \( E_F \) crosses \( E_C \), and thus electrons accumulate to form surface-conducting channels. Note that this mechanism originates from the intrinsic
donor-type states at the surface. Further detailed investigations are required to shed more light on the surface states of Bi$_2$O$_2$Se.

**VI. SUMMARY**

In summary, we synthesized high-quality Bi$_2$O$_2$Se nanowires by means of a gold-catalyzed VLS method. The single-crystalline nature of Bi$_2$O$_2$Se nanowire as a typical bismuth-based oxychalcogenide material was confirmed by x-ray diffraction and transmission electron microscopy. The gate-tunable $h/e$-period oscillations were observed and attributed to the quasiballistic transport through the surface states of Bi$_2$O$_2$Se nanowires. These results clearly demonstrate a new type of semiconducting nanowire with quasi-1D electron subbands of different angular momentum at the surface, which provides an ideal platform for the design of future quantum electronics. A possible intrinsic mechanism is proposed for the formation of electron accumulation, which, however, deserves further detailed theoretical and experimental inspection.

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