Magnetoresistance oscillations in topological insulator microwires contacted with normal and superconducting leads

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A topological insulator (TI), which is topologically distinct from an ordinary insulator, is a material with a bulk electronic excitation gap generated by the spin-orbit interaction. This distinction, which is characterized by a $Z_2$ topological invariant, necessitates the existence of gapless electronic states on the sample boundary. In two dimensions (2D), the TI is a quantum spin Hall insulator. A strong TI is expected to have surface states whose Fermi surfaces enclose an odd number of Dirac points. This feature defines a topological metal surface phase that is predicted to have novel electronic properties.

Bismuth-based binary compounds, such as Bi$_2$Te$_3$ and Bi$_2$Se$_3$, have been long known as excellent thermoelectric materials due to their high mobility 2D carriers exist in the surface areas of the nanowires. Bi$_2$Se$_3$ and Bi$_2$Te$_3$ have a simple band structure with a single Dirac cone on the surface and a large non-trivial bulk gap of 300 meV. The semiconducting alloy In$_{0.68}$Bi$_{0.32}$ is a strong topological insulator due to the inversion symmetry of bulk crystalline Bi and Sb. To study the TI/SC interface, we prepared Bi$_2$Te$_2$Se and Bi$_{0.83}$Sb$_{0.17}$ glass-coated microwire samples using superconducting alloy In$_{0.68}$Bi$_{0.32}$ leads.

The MR oscillations equidistant in a transverse magnetic field (up to 1 T) at the TI/SC interface were observed at various temperatures (4.2 K–1.5 K) in both the Bi$_2$Te$_2$Se and Bi$_{0.83}$Sb$_{0.17}$ samples. In the Bi$_2$Te$_2$ sample with a diameter of $d = 17 \mu$m, this oscillations exist with a period of $\Delta B = 18 \text{ mT}$; in the Bi$_{0.83}$Sb$_{0.17}$ sample with $d = 1.7 \mu$m MR oscillations are characterized by a period of $\Delta B = 46 \text{ mT}$. The observed oscillations cannot be referred to the Shubnikov de Haas oscillations because they are not periodic in an inverse magnetic field and their amplitude decreases with increasing magnetic field. Most probably, transverse MR oscillations arise owing to the appearance of highly conducting edge states on the planar boundary of SC/TI.

1. Introduction

A topological insulator (TI), which is topologically distinct from an ordinary insulator, is a material with a bulk electronic excitation gap generated by the spin-orbit interaction. This distinction, which is characterized by a $Z_2$ topological invariant, necessitates the existence of gapless electronic states on the sample boundary. In two dimensions (2D), the TI is a quantum spin Hall insulator. A strong TI is expected to have surface states whose Fermi surfaces enclose an odd number of Dirac points. This feature defines a topological metal surface phase that is predicted to have novel electronic properties.

Bismuth-based binary compounds, such as Bi$_2$Te$_3$ and Bi$_2$Se$_3$, have been long known as excellent thermoelectric materials due to their unique near-gap electronic structure [1,2]. Recently, it has been shown that these materials host novel topological surface states inside the bulk energy gaps that are protected by the time reversal symmetry [3,4]. The Bi$_{1-x}$Sb$_x$ semiconducting alloy is a strong TI owing to the inversion symmetry of bulk crystalline Bi and Sb. Bismuth is a semimetal with strong spin-orbit interactions, which has an indirect negative gap between the valence band at the T point of the bulk Brillouin zone and the conduction band at the L points [5]. The substitution of bismuth with antimony changes the critical energies of the band structure. At concentrations higher than $x = 0.09$, the system develops into a direct-gap insulator, the low-energy physics of which is dominated by the spin-orbit coupled Dirac particles at L [6,7]. Here we study nanowires with $x = 0.17$ that in the bulk are semiconductors with an L-point gap of 21 meV. In our previous studies [8,9], the magnetic field dependences of the resistivity of glass-insulated Bi$_2$Te$_3$ and Bi$_{0.83}$Sb$_{0.17}$ nanowires were studied in a magnetic field range of 0–14 T. It was shown that high mobility 2D carriers exist in the surface areas of the nanowires.

The unusual metallic surfaces of these TIs can result in the development of novel spintronic or magnetoelectric devices. Furthermore, in combination with superconductors, TIs could lead to a new architecture for topological quantum bits. Following theoretical proposals [10] for an experimental setup for detecting the elusive Majorana particle, which represent a topological phase of matter that
could form the basis for quantum computation, several experiments have identified signatures of Majorana modes in topological superconducting nanowires [11–13].

2. Samples and experimental details

To prepare Bi$_2$Te$_2$Se and Bi$_{0.83}$Sb$_{0.17}$ TI microwires, we used two different technologies, namely, the Taylor technique and the Ulitovsky technique. The following materials with a high degree of purity were used as initial components: Bi (99.999%), Sb (99.999%), Te (99.999%), and Se (99.999%). Synthesis was performed at a temperature of 700–720 °C in a cylindrical furnace.

To prepare a Bi$_2$Te$_2$Se wire based on semiconductor materials with many components and volatile impurities at high temperatures, the Taylor-Ulitovsky method with thermal furnace heating has been developed (see schematic diagram in the inset of Fig. 1a). The main element of the novel method was the use of a furnace with resistive heating and stable temperature regime, which was ensured by a temperature controller with accuracy of ±0.5 °C, as a heater in the Taylor–Ulitovsky installation. The smallest and largest diameter obtained was $d = 5$ and 100 µm, respectively.

A thermal treatment of the prepared Bi$_2$Te$_2$Se glass-coated microwires at different temperatures (450–520 K) and time intervals (24–72 h) was performed to improve the microwire characteristics. Isothermal annealing of the microwires leads to an increase in the thermopower and a decrease in the resistivity. The larger the temperature and annealing time, the higher the parameters obtained. X-ray studies showed that the microwire core was in general a polycrystal consisting of big disoriented single-crystal blocks with an approximate size of 10–15 µm. At 300 K for the samples with diameter $d$ in the range 15–20 µm, the thermopower is $S = -(100–140)$ µV/K and the resistivity is $\rho = 1 \times 10^{-3}$ to $7 \times 10^{-3}$ Ω cm. A scanning electron microscope image of the cross section of the 17-µm Bi$_2$Te$_2$Se wire in a glass coating is shown in Fig. 1a.

Individual Bi$_{0.83}$Sb$_{0.17}$ nanowires were prepared using the Ulitovsky technique (see schematic diagram in the inset of Fig. 1b). This technique involves a high-frequency induction coil melting a Bi$_{0.83}$Sb$_{0.17}$ boule within a borosilicate glass (Pyrex) capsule in an argon atmosphere simultaneously softening the glass. The glass capillaries, each containing a Bi$_{0.83}$Sb$_{0.17}$ filament [14], were prepared by drawing the material from the glass. Nanowire samples with diameters ranging from 75 nm to 2 µm were prepared. The nanowires are single crystals with the (101) orientation along the wire axis. In this orientation, the wire axis makes an angle of 19.5° with the C1 bisector axis in the bisector-trigonal plane. Since it is difficult to successfully grow bulk Bi-Sb crystals, special techniques should be employed to avoid constitutional supercooling and the resulting segregation. With the Ulitovsky technique, owing to the high frequency stirring and high-speed crystallization (> $10^5$ K/s) involved, it is possible to obtain homogeneous monocrystalline Bi$_{0.83}$Sb$_{0.17}$ nanowires. Encapsulation of the Bi$_{0.83}$Sb$_{0.17}$ filament in glass protects it from oxidation and mechanical stress. A scanning electron microscope image of the cross section of the 1.0-µm Bi$_{0.83}$Sb$_{0.17}$ wire in a glass coating is shown in Fig. 1b.

The samples for measurements were cut from long microwires; the length of the samples was 2 mm. They were then mounted on special foil-clad fiberglass plastic holders. Electrical contacts between the microwire and the copper foil were made on one side with a Ga solder and on the other side using an In$_2$Bi superconducting solder with $T_c = 5.7$ K and the melting point of $T_m = 80$ °C. The wires were held in special holders and inserted in a cryostat for low temperature measurements. We carried out magnetic field-dependent resistance $R(B)$ measurements in a range of 0–1 T at the International Laboratory of High Magnetic Fields and Low Temperatures (Wroclaw, Poland) and employed a device that tilts the sample axis with respect to the magnetic field and also rotates the sample around its axis. We used the magnetic field modulation technique to measure magnetoresistance oscillations (Shubnikov–de Haas oscillations, Aharonov-Bohm oscillations (AB)). The amplitude of the oscillatory field is 45 Oe. This very sensitive technique allowed us to register the oscillation amplitude directly at the lock-in amplifier output.

3. Results and discussions

Recent efforts to detect and manipulate Majorana fermions in solid state devices have employed TI nanowires proximity-coupled to superconducting leads. This combination holds some promises for the fundamental physics and applications. The In$_2$Bi intermetallic compound with $T_c = 5.7$ K was used as a superconductor [15–17]. According to our measurements of thin (10 × 2 × 0.2 mm$^3$) polycrystalline In$_2$Bi tape in a transverse magnetic field $B_t \sim 0.25$ T at $T = 1.5$ K. Since this compound has a low melting point ($T_m = 80$ °C), it can be used for the preparation of contacts by soldering the microwire to copper leads, without damaging or modifying the investigated TI microwires. To eliminate the ambiguity of the obtained results, contacts to the TI nanowires made with In$_2$Bi were used only on one side of the samples. On the other side of the samples, the contacts were made using Ga solder ($T_c = 1$ K), which in the range of studied temperatures of 300–1.5 K was in a normal nonsuperconducting state. It should be noted that it was difficult to obtain contacts with In$_2$Bi for both the Bi$_2$Te$_2$Se and Bi-Sb microwires. As a consequence, the superconductor probably was in contact with the glass-insulated microwires not over the entire cross-sectional area. In our experiment we used 17-µm n-type Bi$_2$Te$_2$Se microwires with an outer diameter of 29 µm and 1.7-µm Bi$_{0.83}$Sb$_{0.17}$ with an outer diameter 19 µm. The $R(T)$ dependence for the Bi$_2$Te$_2$Se microwires exhibits a “metallic” behavior, whereas for the Bi$_{0.83}$Sb$_{0.17}$ microwire, it exhibits a “semiconductor” behavior. At helium temperatures in a transverse magnetic field, the resistance of these samples
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