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Electrical transport properties and morphology of topological insulator Bi_2Se_3 thin films with different thickness prepared by magnetron sputtering

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ABSTRACT

Topological insulator Bi_2Se_3 thin films were grown by magnetron sputtering on Si (1 0 0) substrate and their phase structures and electrical properties were studied. The films have good crystalline quality, and their surfaces exhibited terrace-like quintuple layers. In the high temperature region, contribution from surface carrier and bulk conduction band electrons excited from an impurity band; in the low temperature region, surface electron became dominant. The weak antilocalization (WAL) cusp was observed in the magneto-transport measurement at low temperature under low magnetic field. The linear magnetoresistance (MR) under high-field was found, which was associated with the gapless topological surface states and of quantum origin. Insulating tendency was much stronger in thinner films. The WAL effect became weaker in thicker films. The top surface was only partially decoupled.

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1. Introduction

Recently, research on three-dimensional (3D) topological insulators (TIs) such as Bi_2Te_3, Bi_2Se_3, and Sb_2Te_3 has attracted much interest as they have great potential applications in spintronics and thermoelectrical devices. Topological insulator is a new material with strong spin-orbit coupling (SOC) and time reversal invariant symmetry [1–5]. Some quantum transport phenomena in 3D TIs have been observed via magneto-transport studies, which is associated with the gapless surface state. Aharonov–Bohm (AB) oscillations were discovered in Bi_2Se_3 nanoribbons, proving the existence of a coherent surface conducting channel [5]. Several groups reported the weak antilocalization (WAL) effect in 3D TI nanoribbons [6] and thin films [7–9]. Non-saturating linear magneto-resistance (LMR) under high-field was recently observed in Bi_2Se_3 nanoribbons [10] and films [11,12], which suggested that the linear signatures arouse from the linear Dirac surface dispersion.

Bi_2Se_3 is an ideal candidate 3D TIs because it has a gapless single Dirac cone surface states and semiconductor gap in the bulk (approximately 0.3 eV) which has been experimentally confirmed by angle-resolved photoelectron spectroscopy (ARPES) [3]. The research work on Bi_2Se_3 has been extended from bulk material to thin film, as the bulk carriers' contribution can be minimized. Therefore, it is important for the films fabrication in both fundamental research and the practical application.

Several techniques have effectively prepared Bi_2Se_3 thin films, including pulsed laser deposition [13,14], thermal evaporation deposition [11], and molecular beam epitaxial growth [7,15]. Compared with the above technologies, magnetron sputtering deposition is rarely used for depositing Bi_2Se_3 films. Magnetron sputtering has become a useful technique for depositing films as it has many advantages, such as high deposition rate, good film-forming uniformity and a relatively high adhesion. In addition, this technique can precisely control the thickness of the coating, has a better repeatability. According to our previous work, topological insulator Bi_2Se_3 films could be grown by magnetron sputtering deposition [16]. In that work, we mainly studied the effect of annealing temperature on the structure and electrical properties. More importantly, detailed physical mechanism was not given. As it is known that the thickness is one key issue for films especially for topological insulator films.

In this work, the electrical and magnetic transport properties of Bi_2Se_3 films on single silicon substrates (Si 100) with different thickness grown by magnetron sputtering method were studied. At low magnetic fields, the MR exhibits a weak antilocalization (WAL) cusp which was suppressed when the films thickness increased. In addition, the MR linearly increased with increasing field and non-saturating at high fields ($B > 7$ T). The magnetoconductance can be fitted at low field data ($|B| < 1$ T) at 10 K by Hikami–Larkin–Nagaoka (HLN) quantum interference model.

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2. Experimental

The Bi_2Se_3 films were grown using magnetron sputtering method. Bi_2Se_3 alloy target of 99.999% purity was used as the source material. The target-to-substrate distance ($d_{T–S}$) was 60 mm. The base pressure remained less than 2×10^{-4} Pa. The (100) oriented silicon wafers were used as the substrates. The silicon substrates were cleaned by the standard cleaning procedure before they were loaded into the deposition chamber. Bi_2Se_3 thin films were deposited at substrate temperature (T_S) of 220 °C and the deposition time was kept at 1–10 min. The thickness of all the films was varied from 30 to 300 nm confirmed by SEM. After deposition, the as-grown films were post-annealed in Se-rich environment at 300 °C.

The crystal structure of the films was characterized by a powder X-ray diffraction (XRD, X'Pert PANlytical) with Cu Kα emission (λ = 1.5418 Å). Transport properties were examined using a physical properties measurements system (PPMS, Quantum Design) with the standard four-probe method at the temperature ranging from 2 to 300 K and magnetic field ranging from 0 to 9 T. Microstructure was performed with field emission scanning electron microscopy (FESEM, JSM-7001F). The compositions were examined by energy dispersive X-ray analysis (EDX) equipped with FESEM.

3. Results and discussions

All diffraction peaks correspond to (003n) reflections without second phase, confirming that the films with different thickness are highly textured, with *c*-axis perpendicular to the surface plane, as shown in Fig. 1(a). The deposited Bi_2Se_3 films show a pure layered rhombohedral crystal structure in the space group$R\bar{3}m$. The details of the XRD peaks reflect that all the thin films have the same structure. The diffraction peak of the thickest films ($d = 300$ nm) has the highest relative intensity, which is consistent with the results reported by Bansal et al. [17] The deposited Bi_2Se_3 films exhibit clear mirror-like surface with metallic luster, shown in Fig. 1(b).

SEM images for Bi_2Se_3 films with thickness of from 30 to 300 nm are shown in Fig. 2. The Bi_2Se_3 films are roughly equal in large dimensions. In a magnified SEM image, the multi-layered nanostructured with the step bunching features could be observed rated. The SEM images show hexagonal and triangle nanoplates with an edge length of about 200 nm. The surface consists of flat and ordered terraces and steps, reflecting the hexagonal crystal structure inside of the (0001) plane [18]. There are two types of ordered structures, one is hexagonal shape and another is truncated trigonal morphology. The triangular features indicate that the products have the three-fold symmetry [19]. The XRD patterns indicate that the samples shown in Fig. 1(a) have a rhombohedral phase with good crystallinity, in agreement with SEM analysis. The surface was dominated by triangular and hexagonal mounds conforming to the fcc stacking of the rhombohedral crystal structure of Bi_2Se_3 [20]. This result supports that Bi_2Se_3 films obtained via van der Waals epitaxy mechanism.

Fig. 3(a) shows the electrical resistivity as a function of temperature (ρ–T) for Bi_2Se_3 thin films with thickness $d = 30$ to 300 nm from 300 to 2 K. In the high temperature range above 183 K, the resistivity collapses onto one curve, indicating the resistivity is dominated by the bulk conductance. [21]

In the whole temperature range, the thickest film ($d = 300$ nm) shows weak metallic resistivity. However, others exhibit different characteristics. The conductivity at high- and low-temperature regions via different mechanisms, which can be explained by the fact that bulk carriers freeze out and surface state carriers dominate the conductivity at low-temperature regions, while bulk conductance dominates the at high-temperature regions [21].

For the thinner films ($d < 300$ nm), the resistivity starts to drop down monotonically by cooling from room temperature. The metallic behavior indicates phonon scattering dominants in the high temperature region. After lower than minimum temperature between 72 and 183 K, depending on the thickness of films, the resistivity exhibits an increase. We fitted the curves by an activation type, giving the activation energy E_a listed in the Table 1. He et al. [21] found that is about 37 meV for the films with thicknesses above 10 nm and 90 meV for 7 nm film. The large discrepancy is assumed to be associated with the Se vacancies density and defects. The above results indicate that a shallow impurity band from Se vacancies is lower than the bulk conduction band. The resistivity increases below T_{min} where the resistivity reaches the minimum, indicating the occurrence of insulator behavior. As d increases further, T_{min} shifts to low temperature, shown in of Fig. 3(b). This suggests that thickness of the films has significant effect on the total conductance. Both observations indicate that the insulating tendency is much stronger in thinner films.

When the temperature decreases below the activation region, the thermally excited electrons in the bulk conduction band are frozen out. As a result, the resistivity of the thinner films increases abruptly, then saturates at a constant value in the low temperature region. This temperature-dependent resistance shows a nonmetallic behavior in this region [22], which implies that the Fermi energy sits within the gap between the bulk conduction band and valence band. The similar temperature dependence of resistivity was reported in Sb doped Bi_2Se_3 nanoribbons [23] and Bi_2Se_3 topological insulator thin films on Si (111) [21].

Below 20 K, the resistivity of the thinner films increases slightly, as the conductance provides other electron channels, which reveals an insulating ground state [24]. The fitting data of resistivity follows a typical Mott $T^{-1/4}$ law, implying a 3D variable range hopping (VRH) transport mechanism [25].

Hall measurements were also performed. The sheet carrier density is plotted as a function of the temperature for the thin film with the thickness of 30 nm as shown in Fig. 3(d). The sheet carrier density n of all

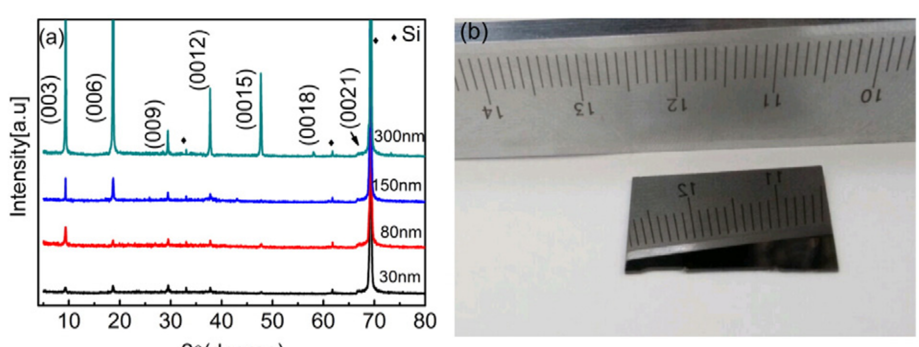

Fig. 1. (a) X-ray diffraction patterns of Bi_2Se_3 films with different thickness; (b) photo images for Bi_2Se_3 films ($d = 300$ nm), revealing a mirror-like surface.

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