Anisotropic Terahertz Emission from Bi$_2$Se$_3$ Thin Films with Inclined Crystal Planes

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Abstract

We investigate the surface states of topological insulator (TI) Bi$_2$Se$_3$ thin films grown on Si nanocrystals and Al$_2$O$_3$ substrates by using terahertz (THz) emission spectroscopy. Compared to bulk crystalline Bi$_2$Te$_2$Se, film TIs exhibit distinct behaviors in the phase and amplitude of emitted THz radiation. In particular, Bi$_2$Se$_3$ grown on Al$_2$O$_3$ shows an anisotropic response with a strong modulation of the THz signal in its phase. From x-ray diffraction, we find that the crystal plane of the Bi$_2$Se$_3$ films is inclined with respect to the plane of the Al$_2$O$_3$ substrate by about 0.27°. This structural anisotropy affects the dynamics of photocarriers and hence leads to the observed anisotropic response in the THz emission. Such relevance demonstrates that THz emission spectroscopy can be a sensitive tool to investigate the fine details of the surface crystallography and electrostatics of thin film TIs.

Keywords: Topological insulator, Bi$_2$Se$_3$, Thin film, Terahertz emission

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Background

Topological insulators (TIs) behave as a charge-gapped insulator in their interior but hosting a spin-momentum-locked Dirac state at the surface. When the Fermi level crosses over a conduction/valence band, a bulk charge transport can overwhelm the surface contribution, so that thin film TIs have been highlighted as a method to reduce bulk carrier effects due to a large surface to bulk volume ratio [1–6]. During extensive research activities on thin film TIs, several experimental techniques have been employed to characterize thin film properties; for example, atomic force microscopy and transmission electron microscopy could reveal the existence of twin domains with a triangular shape and an inversion symmetry breaking even in the bulk state of film TIs [7–11]. Also, surface-sensitive techniques can be utilized to characterize a Dirac dispersion of the surface state; Fermi surface information can be extracted from Shubnikov-de Haas oscillations, and in particular, the interface state formed at the junction between TI and conventional semiconductors can be studied by using tunneling spectroscopy under magnetic field [12–17].

Terahertz (THz) spectroscopy also can provide useful information about the surface state of the TIs. From conventional THz time-domain spectroscopy, Aguilar et al. could retrieve optical response functions, such as optical conductivity, of the TI surface state, and determine electrodynamic parameters of Dirac fermions [18, 19]. Similar information could be obtained from THz emission spectroscopy [20]. Whereas the THz wave can be emitted from the acceleration of photocarriers generated by an illumination of a pulsed laser onto TIs, a change in THz intensity with a variation of the bulk carrier density could be satisfactorily explained by considering the contribution of Dirac fermions together with bulk charge carriers, which then provided useful information about the mobility of surface carriers.

In this paper, we demonstrate that time-domain THz emission measurement can be a sensitive method to investigate the details of the surface crystallography and electrostatics of thin film TIs. We examine an azimuth-dependent THz radiation emitted from Bi$_2$Se$_3$ thin films grown on Si nanocrystals (NCs) and Al$_2$O$_3$ and also...
from the bulk Bi$_2$Te$_2$Se. Whereas the bulk crystalline Bi$_2$Te$_2$Se shows an isotropic behavior of the THz emission, film TIs exhibit contrasting behaviors in the phase and amplitude of emitted THz radiation. In particular, the Bi$_2$Se$_3$ film grown on the Al$_2$O$_3$ substrate exhibits a strong modulation of the THz electric field profiles upon the variation of the sample azimuth. We will discuss the details of such emitted THz waves from the surface of Bi$_2$Se$_3$ films by considering the anisotropic dynamics of the photocarriers under the structural anisotropy in the thin film TI.

**Methods**

High-quality Bi$_2$Se$_3$ films with 30–33-nm thickness were grown on Si NCs [9, 21–24] and Al$_2$O$_3$ by a molecular beam epitaxy (MBE) system. Si NCs were fabricated by annealing SiO$_2$/Si$_2$ multilayers grown on Si wafers by ion beam sputtering, whose detailed processes were described in previous reports [21]. The average size of Si NCs used in this work was estimated to be ~3.1 nm by transmission electron microscopy [22, 24]. As a control, we prepared a single crystalline Bi$_2$Te$_2$Se sample using the self-flux method with a stoichiometry of chunks (Bi:Te:Se = 2:1.95:1.05) [25, 26].

For THz emission spectroscopy, we use 70-fs laser pulses with a center wavelength of 800 nm generated from a Ti:sapphire oscillator at a repetition rate of 80 MHz. The beam is focused onto the sample with a 300-μm spot size at an incidence angle of 45° (inset of Fig. 1a) and a power density of about 0.6 kW/cm$^2$, which is below the damage threshold [14]. Because the penetration depth of incident light is about 20 nm [14], THz wave generation takes place near the surface of the sample. Emitted THz radiation is guided by a pair of parabolic mirrors in a specular reflection geometry. The transient electric field of the THz wave is then detected by a photoconductive antenna made of the low-temperature-grown GaAs [27].

Although thin films were grown along the $c$-axis in a hexagonal setting of the crystal, we perform the x-ray diffraction experiment with three degrees of freedom, i.e., θ, 2θ, and Φ motion (Fig. 3a) to characterize the orientation of the crystal plane more accurately. In the geometry of an asymmetric reflection, we tilt the sample using the θ motion with respect to the surface normal until we find a maximum intensity of the Bragg peak (006) of Bi$_2$Se$_3$ with the 2θ motion fixed to $\theta_B$ as indicated in Fig. 3a. The same procedure is taken for the substrate diffraction peak. The difference in the tilting angle $\theta$ between the film and substrate at the same azimuth angle $\Phi$ corresponds to the inclination angle ($\Delta \varphi$) of the film Bragg plane with respect to the crystal plane of the substrate.

**Results and Discussion**

We examine first the THz emission from the bulk crystalline Bi$_2$Te$_2$Se of which results are displayed in Fig. 1a, b. Time profiles of the THz electric field (Fig. 1a) are obtained by varying the sample azimuth Φ, and they commonly show a single-cycle oscillation with a well-defined crest and trough. The THz signals at these points marked by an open square and closed circle are plotted as a function of the sample azimuth Φ for 0° to 270°. The inset shows a schematic of the THz generation experiment. a Time profile of the THz electric field emitted from Bi$_2$Te$_2$Se obtained with a variation of the sample azimuth Φ from 0° to 270°. The inset shows a schematic of maximum (filled circle) and minimum (open square) amplitudes indicated in (a), c, d THz generation mechanism by two surge current contributions, i.e., the photo-Dember effect and photocarrier acceleration by surface band bending, respectively. The horizontal axis means the depth d from the surface. The vertical axes in (c) and (d) indicate a lateral dimension in the sample and an electron energy $E_e$, respectively. $e$ (h) represents electron (hole). $E_F$ and $E_C$ ($E_V$) lines denote a Fermi energy and conduction band minimum (valence band maximum) of the bulk state.
absorption will raise the carrier temperature enhancing this effect [28]. Also, it is well known that a charge distribution is inhomogeneous at the surface of TIs resulting from the surface band bending [29]. Therefore, although we could exclude the optical rectification as a mechanism of THz emission in Bi₂Te₂Se, it is not clear yet which has a more dominant contribution to the observed THz emission signals between the photo-Dember effect and surface field acceleration.

Figure 2a shows a THz time profile emitted from the thin film Bi₂Se₃ grown on Si NCs. Compared to the case of the bulk sample, the amplitude of the THz wave is comparable and the azimuth dependence is similarly isotropic (Fig. 2b). On the other hand, a phase of the emitted THz wave is turned out to be opposite. When the THz wave is generated by photo-Dember effect, its phase is expected to remain the same in a similar kind to the materials where the relative difference in the electron and hole mobilities remains unchanged under the variations of the chemical composition or the carrier concentration. On the other hand, the direction of the built-in electric field or the surface band bending can be developed differently depending on the major carrier type which can change the direction of the photocarrier acceleration and accordingly the phase of emitted THz wave. Therefore, the surface field acceleration rather than the photo-Dember effect would be a more dominant mechanism of THz emission in the Bi₂Se₃ thin film and possibly in the Bi₂Te₂Se single crystal as well.

Accordingly, these two systems can have different situations related to the surface band bending.

Interestingly, the Bi₂Se₃ film grown on the Al₂O₃ substrate shows more distinctive behaviors in its THz emission response. Differently from the former two cases, the phase of the THz electric field profile is not well defined under the variation of the sample azimuth Φ (Fig. 2c) We pick up the amplitudes around 3 ps (filled circle) and 4.5 ps (open square) and display them in Fig. 2d as a function of Φ. Although they have a circular shape, the circles themselves are displaced from the center of the coordinates. The off-centering of two circles signifies the existence of a Φ-dependent onefold anisotropy affecting THz wave radiation. We can exclude a possible contribution of the optical rectification from the surface state of the 3m point group as it should exhibit a sixfold (or threefold) symmetry. Rather than such an intrinsic inversion symmetry breaking at the surface of Bi₂Se₃, we pay attention to an additional symmetry breaking possibly induced during the growth process of Bi₂Se₃ thin films on the substrate.

Using the x-ray diffractometer, we examine the relative crystalline directions of the Bi₂Se₃ films and the substrate. Figure 3c displays Bragg peaks near 2θ = 18.6° corresponding to a (006) peak of Bi₂Se₃ grown on Si NCs and near 2θ = 69.2° for the (004) peak of a Si substrate beneath the Si NC layer. The sample azimuth Φ is set to 225°. It should be noted that the maximum intensity of each peak cannot be obtained simultaneously in a single diffraction geometry. When φ is optimized to Si (004), the diffraction peak for Bi₂Se₃ (006) (line) has a smaller intensity (by about 20 %) than that of the peak obtained after the optimization to itself (open circle). φ is obtained by varying Φ for both the film and the substrate, and their difference Δφ is plotted in Fig. 3d. It shows a slightly off-centered circle in the Φ variation where the largest deviation from zero value (gray line) appears near Φ = 45° and 225° with about Δφ = ±0.1° and +0.1°, respectively. This angle corresponds to the degree of inclination of the Bragg plane in the Bi₂Se₃ thin film with respect to the substrate Bragg plane.

We employ the same procedures to Bi₂Se₃ films grown on an Al₂O₃ substrate where the angle φ for the Al₂O₃ substrate is optimized to its (006) diffraction peak around 2θ = 41.7°. At the sample azimuth Φ = 90°, the Al₂O₃ (006) peak can be optimized to have a maximum intensity such as a line curve in Fig. 3e, but it shifts rightwards with a large reduction of its intensity when φ is optimized to the Bi₂Se₃ (006) peak. Note that the estimated inclination angle from the azimuth-dependent Δφ shown in Fig. 3f is about 0.27°, and this is about 2.5 times larger than for the film grown on the Si NCs. Typically, Al₂O₃ substrates can have a larger vicinal angle than that of a Si wafer. When Bi₂Se₃ films are grown on...
such vicinal substrates, the crystallographic orientation of the films may not follow that of the substrate, and the tilting between the film and the substrate in their c-axis orientations can be proportional to the substrate vicinality.

It is intriguing to note that such an inclination of the crystal plane of the film is intimately connected to the aforementioned modulation in the THz electric field. We estimate the modulation amplitude of the THz electric field $\Delta E_{\text{THz}}$ with a variation of $\Phi$ from the curves given in Figs. 1b and 2b, d. Certainly, $\Delta E_{\text{THz}}$ is negligible for the bulk $\text{Bi}_2\text{Te}_2\text{Se}$ and thin film $\text{Bi}_2\text{Se}_3$ on Si NCs whereas it is relatively large for the film on $\text{Al}_2\text{O}_3$. This THz-response $\Delta E_{\text{THz}}$ is plotted versus $\Delta \phi$ in Fig. 4 where the $\Delta \phi$ of the bulk $\text{Bi}_2\text{Te}_2\text{Se}$ is set to zero. From this plot, we can reasonably expect a possible relationship between these two quantities. To support this hypothesis, it is worthy to note a similar phase relationship between the THz amplitude and $\Delta \phi$ in their $\Phi$ dependences shown in Figs. 2d and 3f, respectively; they both have a onefold symmetry, and the maximum deviations of each quantity from an isotropic response occur near the same azimuth.

Actually, when the crystal plane is inclined with respect to the sample surface (Fig. 3a), the photocarrier dynamics and consequently the characteristics of the emitted THz waves will be influenced by such a onefold in-planar anisotropy. As a quasi-two-dimensional system, $\text{Bi}_2\text{Se}_3$ can
have an anisotropic behavior along the c-axis with respect to an ab-plane, such as a dc-conductivity ratio $\sigma_{ab}/\sigma_{c}$ of about 3–4 [30]. When the crystalline ab-plane is inclined from the sample surface, the surface-normal direction will be defined with contributions from both the crystallographic c-axis and the a- (or b-) axis even though the latter will have a minor contribution. Then the photocarriers generated at the surface, which otherwise are accelerated simply along the surface-normal direction, will experience an anisotropic electrostatic potential or anisotropic transport behavior as they travel inside of the film. Consequently, THz light emitted through a surge current can have the finite anisotropic response on the sample azimuth. Since x-ray diffraction results revealed the relative tilting angle between diffraction planes of the TI film and the substrate, crystallographic planes can be drawn differently as in Fig. 3b where the crystal plane of the substrate is inclined with respect to the sample surface and the TI crystal plane is in parallel with the sample surface. We consider that the configuration in Fig. 3a instead of that in Fig. 3b is more preferable to explain the anisotropic THz emission results since the THz emission in the case of Fig. 3b should appear isotropically as a function of $\Phi$. Although the anisotropic THz responses in the variation of $\Phi$ were discussed in terms of the surge current THz emission process, it should be noted that the results would be explained also by the optical rectification process by considering the lowered surface symmetry.

Conclusions

We demonstrate that the terahertz (THz) emission spectroscopy can be a sensitive tool to investigate the fine details of the electrostatics and surface crystallography of topological insulator thin films. For the Bi$_2$Se$_3$ thin film grown on Si nanocrystals, the emitted THz electric field has an opposite phase to that from the bulk Bi$_2$Te$_2$Se crystal, and this can be attributed to different electrostatics related to the surface band bending of two samples. For the Bi$_2$Se$_3$ thin film grown on Al$_2$O$_3$, the THz emission occurs with a strong modulation particularly in its phase upon the variation of the sample azimuth. Through x-ray diffraction experiment, we found that the crystal plane of the film is inclined with respect to the substrate crystal plane, and the resultant planar anisotropy is responsible for the onefold symmetry in the anisotropic THz response. As topological insulators in the form of the thin film have been attracting much attention these days to exploit the Dirac fermionic surface state with less contribution of conventional bulk carriers, we expect that phase-sensitive THz emission spectroscopy can have a clear contribution in such researches by elucidating the lateral and longitudinal electrostatics related to the band bending as well as the structural symmetry at the surface and/or interface of the film.

Abbreviations

THz: Terahertz, TI: Topological insulator.

Competing Interests

The authors declare that they have no competing interests.

Authors’ Contributions

SYH, SHP, and JWH carried out the terahertz experiment and analyzed the results. SYH drafted the manuscript. JHJ and SJK fabricated the Bi$_2$Se$_3$ films on Al$_2$O$_3$. SK and SHC designed the structure of the Bi$_2$Se$_3$/Si nanocrystals (NCs) and prepared the Si NCs on Si wafers, and then NB and SO made the Bi$_2$Se$_3$ films on Si NCs. JH and JSK prepared the Bi$_2$Te$_2$Se single crystals. JMK and DYN carried out the x-ray diffraction experiment. JSL supervised the research and revised the manuscript. All authors read and approved the final manuscript.

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