Exploration of terahertz from time-resolved ultrafast spectroscopy in single-crystal Bi$_2$Se$_3$ topological insulator

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Abstract
In this article, we reconnoiter the differential reflection signal of a Bi$_2$Se$_3$ single-crystal flake, using ultrafast transient absorption spectroscopy in the femtosecond time domain and thereby explore the experimental data in terms of terahertz frequency generated in the sample. An exfoliated flake of a well-characterized self-flux grown bulk Bi$_2$Se$_3$ single crystal having rhombohedral structure and layered morphology is used in the present study. The kinetic profile of the same being generated through a reflection signal by a pump laser of 650 nm at an average power of 0.5 mW is studied utilizing time-resolved ultrafast technique. The silhouette as a function of probe delay predicting the capability of the terahertz generation is estimated. Here, two methods FFT (fast Fourier transformation) and FFD (filtering high-frequency component followed by fitting data) are performed to estimate the value of terahertz generated in the system. While comparing the two (FFT & FFD), it is found that a large amount of magnitude difference occurs in the prediction of terahertz frequency. Summarily, we not only report the generation of terahertz in Bi$_2$Se$_3$ flake, also but points out that the exact order of magnitude and the capability of the same depends upon the method of analysis. It is important to extract the vibration signal from the background one so that to find the exact order of magnitude and capability of terahertz generation by any quantum material.

1 Introduction
A new quantum state of matter having conducting surface state and an insulating bulk energy band structure has attained a lot of interest of the research community [1–5]. These fascinating compounds are called topological insulators (TI), of whom astonishing surface state (SS) are protected by time-reversal symmetry (TRS) and spin–orbit coupling (SOC) [1–7]. This non-trivial conducting state has a one-to-one corresponding relation between energy and momentum in a low-energy region, which is termed as Dirac cone [1, 2, 8, 9]. The spin-polarized characteristics of the surface states of the TIs do have a large potential in the field of quantum computation, spintronics as well as in the field of nonlinear optics (NLO) [4, 10–12]. Further, TIs had gained tremendous momentum in the field of optical nonlinearity in terms of their intriguing applications such as terahertz generation/detection, frequency shifting, optical signal processing, optical switching, optical power limiters, and optical data storage for information processing [13–23]. The exploration of the optical properties, electron–phonon interaction, carrier dynamics, low-energy electronic responses, etc., of TIs, has aggravated to exploit these materials for optimizing as high-frequency and high-intensity terahertz generators and optoelectronic device performance [14, 17, 24, 25]. Ultrafast statistics of carriers and phonons in TI were studied by time-resolved ultrafast spectroscopy (TRUS). It is one of the standard tools for studying the excited state dynamics of the system.

Further, the dynamic properties of hot carriers in TI that are excited by pump laser have engrossed a great deal of attention as the three-dimensional single crystal of topological insulator generates terahertz frequency [17, 26–30]. The terahertz frequency has an effervescent role in numerous applications such as bio-imaging, medical diagnoses, T-bit communications, security cameras, explosive detection, and industrial inspections [26, 31–33]. Terahertz frequency is generated by the hot carriers in a single crystal of TI (Bi$_2$Se$_3$) as the system is pumped by the laser in its bulk conduction band. These hot carriers while
relaxing cause phonon oscillations in optical as well as the acoustic regime. The order of terahertz frequency can be experimentally calculated using the differential reflection signal that is generated in TRUS. The differential reflection signal as a function of probe delay is then theoretically analyzed by various approaches. A detailed study of terahertz generation of single-crystal Bi$_2$Se$_3$ is taken up, and its theoretical interpretation by two different fitting models of experimental data is reported in this article. First, by FFT (fast Fourier transformation) and another by FFD (filtering high-frequency component followed by fitting data) [14, 17, 24, 25]. The two distinct theoretical approaches (FFT & FFD) resulted in elucidation of terahertz frequency differing by an order of magnitude which has also been reported for perovskite, such as, two-dimensional lead-free hybrid halide perovskite using super atom anions with tunable electronic properties; stable dynamics performance and high efficiency of ABX$_3$-type super-alkali perovskites first obtained by introducing H$_2$O$_2$ cation; tunable electronic structures and high efficiency obtained by introducing super alkali and super halogen into AMX$_3$-type perovskites and in Bi$_2$Te$_3$ topological insulator [14, 17, 24–26, 34–37]. The current article not only substantiates terahertz generation from flakes of bulk single-crystal Bi$_2$Se$_3$, but also points out an important fact that various fitting models may lead to different results. Caution is required before using available fitting models.

2 Experimental details

Single crystal of Bi$_2$Se$_3$ TI is grown by a solid-state reaction route. The purest form (> 4 N) of Bi (Bismuth) and Selenium (Se) was taken as precursor to the grow the crystal via the self-flux method [38, 39]. The encapsulated weighed and inert atmosphere well ground rectangular bar sample was kept in a fully automated tube furnace under a well-optimized heat treatment as described in Fig. 1a [34, 35]. After this vigorous heating process, a silver-shiny 2-cm-long single crystal is grown as shown in Fig. 1b. In order to confirm the structural characteristics of single crystal, room-temperature X-ray diffraction (XRD) using Rigaku made Mini Flex II of Cu–K$_{α}$ radiation ($λ$ = 1.5418 Å) was performed. SEM and EDS are performed on Bruker made scanning electron microscope. The analysis of vibration modes in Bi$_2$Se$_3$ crystal has been done using the Renishaw Raman spectrometer. The ultrafast transient absorption spectroscopy system was used for the measurement of differential reflection signals. This system consists of a Ti: Sapphire-based femtosecond mode-locked laser (MICRA by coherent), amplifier (legend by Coherent), and spectrometer (Helios by ultrafast systems). The laser beams of 800 nm coming from amplifier are a Gaussian pulse with a spectral width of 60 nm and a repetition rate of 1 kHz. This amplified beam splits into 70:30 proportions and the large one is used as a pump beam fed to the operational parametric amplifier (OPA from light conversion). In the Helios spectrometer, a probe beam is steered through an 8-ns-long delay stage and, thereafter, strikes on sapphire/CaF$_2$ crystal to generate a white light continuum. This provides a wide range of spectral profiles from 320 to 1600 nm.

3 Theoretical details

The TRUS system is used for the measurement of differential reflection signals as a function of the probe beam. When Bi$_2$Se$_3$ single crystal is pumped by the laser, the hot carriers in bulk conduction band are excited and thereby thermalization between these hot carriers and phonons
takes place. One of them is thermalization between these carriers and optical phonons in a sub-picosecond timescale, and the other one is in between carriers and acoustic phonons in subsequently slow component of timescale than the optical phonons [25]. The focus of this article is the generation of terahertz frequency being induced from pump laser-created optical phonon vibrations in the system. The visualization of the vibration of phonons can be easily done by Fourier analysis. But the fact is that the analysis can be done by numerous approaches and different theoretical analysis gives different terahertz frequency. The two approaches that have been taken here are FFT and FFD [13, 14, 16, 17, 22, 24, 25]. In FFT, the oscillations in the sub-picosecond regime (1 ps–8 ps) are analyzed by transforming the time function to the frequency domain using Origin software version 9.1. The Origin software uses the FFTW library (Fastest Fourier Transform in the West) to compute the Fourier transformation. FFT is the fastest as well as a convenient way to compute DFT (discrete Fourier transform). A DFT converts a frequency signal to its frequency counterpart, if \( x_i \) be the sequence with a length of \( N \). The DFT function is

\[
F_n = \sum_{i=0}^{N-1} x_i e^{-2\pi i n_i/n}
\]

The second approach to attribute the terahertz frequency generation is to perform FFD. In FFD, the high-frequency component is extracted out from the raw data generated by TRUS followed by its damped sinusoidal fitting. The abstraction is done by filtering out the oscillations from the curve by high-pass Fourier filter with a cutoff frequency of 2.32 THz in Origin software. The process of selecting the frequency from the signal using Fourier transforms to analyze the input is known as filtering. After filtering out the signal, the high-frequency components are fit by using damped sinusoidal function,

\[
Y = Ae^{-(r-t_0)/\tau}\sin[2\pi f(t - t_f)]
\]

where \( A \) is the amplitude, \( r \) is the decay time, \( f \) is the frequency, and \( Y \) is corresponding to \( \Delta R/R \) [25].

The fitting equation gives out the approximate frequency generated in the system. These two methods include various steps and are also described in the flowchart as shown in Fig. 2a, b. These two approaches are used for the exploration of the experimental measurements by TRUS.

4 Result and discussion

The XRD pattern of crystal flake on its surface and in its powder form (PXRD) is shown in Fig. 3a, b, respectively. The peaks at (00L) in the XRD pattern of crystal surface confirm the formation of the single crystalline structure. Further, the Rietveld refinement of the PXRD pattern of \( \text{Bi}_2\text{Se}_3 \) is done using the FullProf Suite toolbar, confirming the rhombohedral crystal structure with R-3 m (D5) space group. The Rietveld refinement gives the atom positions to be Bi (0,0,0,3999(5)), Se1(0,0,0), and Se2(0,0,0,207(4)) with lattice parameters \( a = b = 4.14(2) \text{ Å} \) and \( c = 28.7(1) \text{ Å} \). The full description of the crystal structure and its Rietveld refinement is described in our previous report [40]. Using these parameters and lattice constant, the unit cell structure of \( \text{Bi}_2\text{Se}_3 \) single crystal is described with the help of VESTA software and is shown in Fig. 3c. It can be seen in Fig. 3c that this TI contains three bi-layers of Bi and Se stacked monolayers of either Bi or Se in a closely packed FCC lattice structure [38, 39]. The vibration modes of \( \text{Bi}_2\text{Se}_3 \) single crystal are recorded using Raman spectroscopy at room temperature. The peaks around 72.1, 131.2, and 177.1 cm\(^{-1}\) correspond to \( A_{1g}^1, E_g^2, \) and \( A_{1g}^2 \), respectively, as shown in Fig. 3d. These three distinct Raman active modes were in good agreement with the earlier reported results [38–41]. The experimental evidence of \( \text{Bi}_2\text{Se}_3 \) single crystal shows a layered crystalline structure as depicted by SEM studies. Through EDS analysis confirmed the quantitative amounts of constituent atoms in the studied single crystal close to the stoichiometric ratio and is depicted in Fig. 4b.

A flake of bulk \( \text{Bi}_2\text{Se}_3 \) crystal being grown through self-flux method is used for excited state dynamics studies. The TRUS measurements are done using 650 nm as a pump beam (400-micron spot size and a pulse duration of 70 fs with a repetition rate of 500 Hz which give a peak power of 22.71 GW/cm\(^2\)) with probing in the NIR region. The time-domain data has been collected from few femtoseconds to 6 ns. The kinetic profile as shown in Fig. 5a has been obtained at an average power of 0.5 mW as measured by power meter at point of interaction. This silhouette is being fitted using the surface Xplorer software as shown in Fig. 5b, elucidating the optical excitation of charge carriers of \( \text{Bi}_2\text{Se}_3 \) single-crystal flake. The pump energy lies in the higher regime than the envisaged bulk electronic energy band gap of \( \text{Bi}_2\text{Se}_3 \). The exited electron hastily decays to its lower-lying energy state via inter and intraband phonon-mediated scattering processes, which is probed by the NIR continuum as described in kinetic profile. A metastable population of electrons occur within 1.2 ps, filling the surface state with a steady supply of charge carriers by the agitated beam and then exponential decay up to 8 ps. This occupation of a metallic state is in fact a unique situation. The decay kinetic from 1.2 to 8 ps
consists some sort of oscillations in the signal which corresponding to the optical phonons vibration of the flake. As we engrossed the kinetic profile in this smaller timescale, the oscillations are relatively faster than the other vibrations in higher timescale [13, 14, 18, 19, 22, 25]. It is clear from Fig. 5a that there are oscillations in the kinetic profiles.
Fig. 3  a X-ray diffraction pattern for Bi$_2$Se$_3$ single crystals.  b Rietveld fitted room-temperature X-ray diffraction pattern for powder Bi$_2$Se$_3$ crystals.  c Unit cell structure of Bi$_2$Se$_3$ single crystals.  d Raman spectra of Bi$_2$Se$_3$ single crystals at room temperature

Fig. 4  a Show layered morphology of Bi$_2$Se$_3$ through Scanning electron microscopie.  b Elemental analysis of Bi$_2$Se$_3$ single crystals by Energy-dispersive X-ray spectroscopy
from 1 to 10 ps and 10–50 ps, which happens due to the vibrations of lattice phonons. The thermalization between hot electrons and optical phonons gives upsurge to the fast component of the sub-picosecond timescale. On the other hand thermalization between acoustic phonons and electrons gives rise to a slow component of several picoseconds [14, 16, 17, 20]. The slow oscillations components between 10 and 50 ps correspond to the acoustic phonons vibration in the lattice and the fast oscillations between 1 and 10 ps are attributed to optical phonons as shown in Fig. 5a [24, 25, 40]. The frequency of the fast component can be assigned as the $A_{1g}^1$ COP (coherent optical phonons) mode of Bi$_2$Se$_3$ as prescribed in Raman spectroscopy and the slow component is attributed to the CAP (coherent acoustic phonons). The order of magnitude of terahertz in COP is higher than CAP [24]. Here, we focus on the fast components (COP mode, Fig. 6a and thereby its interpretation in terms of terahertz frequency generation. The exploration of the oscillations is being done by two different methods, i.e., (a) FFT and (b) FFD. These two methods include various steps as described in the flowchart in Fig. 2a, b.

The FFT analysis of the fast oscillations component being described in Fig. 6b shows the generation of 0.1 THz of frequency with a magnitude of 0.3 by the studied Bi$_2$Se$_3$ crystal. This analysis is done by following the flowchart described in Fig. 2a, where the kinetic signal of the flake is cropped from 1 to 10 ps in order to only considering the optical phonon vibration of the flake. These induced oscillations are generated in the flake through the high pulsed laser of order of few gigawatts per cm$^2$. The cropped oscillations are then plotted in the origin software and using the FFT command in it, a complete description of the pulsations

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**Fig. 5**  
(a) Differential reflection signal of the Bi$_2$Se$_3$ single crystals by TRUS.  
(b) Fitted kinetic profile of the Bi$_2$Se$_3$ single crystals by Surface Xplorer

**Fig. 6**  
(a) Cropped reflection signal as a function of probe delay.  
(b) Fast Fourier transformation (FFT) of cropped experimental data
in the experimental data is taken out. It is quite clear that
the highest terahertz frequency generated in the system is
0.1 THz. This much low frequency subsequently is not in
accordance to the capability of the crystal as the data that
is analyzed through this kind of interpretation include both
the pulsations as well as exponential decay profile due to
thermalization of the charge carriers. This convolution of
the different effect in one data reduces the accuracy of the
analysis. Thereby, a secondary analysis of the data is carried
out which is termed as FFD.

The second analysis is FFD, in which the cropped data is
firstly filter out from the exponential decay profile and there-
after fitted using damped sinusoidal function as described
in flowchart of Fig. 2b. The fast oscillations extracted data
shown in Fig. 6a is first filtered out from the decay profile.
The slow varying components are filtered out from the vibra-
tions using high-pass Fourier filter with a cutoff frequency
of 2.32 THz as shown in Fig. 7a using the origin software.
This filtered data is then fitted using a damped sinusoidal
equation as depicted in Fig. 7b for finding out the value of
terahertz frequency generated in the flake. Figure 7c shows a
picture of the fitting data depicting the accuracy of the over-
lapping in the fitting equations and experimental data. Vari-
os parameters that come out from this fitting are described
in Table 1. The value of frequency is easily calculated from
the fitting parameters. While comparing the fitting equation
as described in Table 1 with the Eq. (2), it is established that
frequency in terms of ‘w’ is given by

$$ f = \frac{1}{2w} $$

After calculating the frequency from the formula, the
value of terahertz frequency generated by the single crystal
is $2.69 \pm 0.01$ THz. The terahertz frequency being gener-
at in the crystal is analyzed using FFD, but still, it is in
time-domain scale. Further, these FFD fitted data are Fourier
transformed into the frequency domain to verify the tera-
hertz frequency that is governed by the FFD analysis. Fig-
ure 8, shows the Fourier transform of the FFD fitted data and
it is concluded that the crystal is capable of generating the

![Fig. 7](image_url)

**Fig. 7**  
(a) Optical phonons oscillations by filtering the high-pass Fourier filter with a cutoff frequency of 2.32 THz.  
(b) Damped sinusoidal fitting of the optical phonons vibrations.  
(c) Skyrocketed out experimental fitting data
terahertz frequency. Interestingly, this is almost equivalent to that one as determined by the time-domain FFD analysis. The value of frequency after the Fourier transform of the FFD fitted data is 2.64 ± 0.01 THz. It is clear by the analysis of the TRUS kinetic profile data of the studied Bi$_2$Se$_3$ single crystal, that although the generation of terahertz frequency is evidenced via both models, the relative amount is different.

### 5 Conclusion

We presented the TRUS of an exfoliated flake of bulk Bi$_2$Se$_3$ single crystal grown and studied the nonlinear optical response of the same in the terahertz domain. The two different approaches, i.e., FFT & FFD were used to extract the terahertz frequency from the experimental data and it is found that the former (FFT) and later (FFD) analysis results into 0.1 THz and 2.69 ± 0.01THz, respectively, at a same fluence of 0.5 mW (average power). The FFD data is obtained by using a high-frequency filter, which removes the noise along with the background incident signal, and thus results in better and improved values of the THz oscillations frequency. It is concluded that it is the necessity to remove the background signal from the output signal in order to get the precise and more accurate value of the frequency generated in TI crystals. Further, it is proposed that real time-domain terahertz spectroscopy has to be performed in order to further use this study for terahertz detector applications.

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