Evolution of the Fermi surface of a doped topological insulator with carrier concentration


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I. INTRODUCTION

A topological superconductor is a state of matter in which the bulk is fully gapped, but gapless surface states host Bogoliubov quasiparticles. Point-contact experiments have shown the existence of zero bias conductance peaks (ZBCP) in Cu$_2$Bi$_2$Se$_3$, and these were interpreted as a signature of Andreev surface bound states that were theoretically predicted to exist in certain classes of topological superconductor (TSCs). While the topological properties of topological insulator (TIs) are set by the band structure and should not depend on the chemical potential, the properties of the superconducting samples are sensitive to the chemical potential and to the shape of the Fermi surface (FS). Bi$_2$Se$_3$ has carriers in the conduction band even when carefully prepared. These carriers are believed to be the result of Se vacancies which are always present in the material. The carrier concentration can be increased further by Cu intercalation. The band structure of Bi$_2$Se$_3$ is three dimensional (3D), i.e., there is substantial electronic dispersion in the $k_z$ direction. On the other hand, the material is layered, cleaves easily, and its resistivity is anisotropic with $\rho_{xx}/\rho_{xx} \approx 10$. Band structure calculations (BSC) indicate that the $\Gamma Z$ dispersion is weaker than the $\Gamma L$ dispersion. Early angle-resolved photoemission spectroscopy (ARPES) experiments have shown that the dispersion can be even weaker than the BSC predictions. It is therefore plausible that, on adding charge carriers, the FS will grow in an anisotropic fashion, where $k_F$ along the $k_z$ direction becomes considerably larger than $k_F$ in the $k_xk_y$ plane. The FS therefore can change from being a closed spherical FS at low carrier densities in an open cylinder-like FS at high carrier densities. An important question is whether Cu$_2$Bi$_2$Se$_3$ has a closed or open FS at carrier densities in which the system is superconducting, $n \simeq 10^{20}$ cm$^{-3}$.

The Shubnikov-de Haas (SdH) effect probes extrema in the cross section of the FS. According to the Onsager relation, the frequency of the magnetoresistance oscillation as a function of inverse magnetic field is $F = \frac{2}{\pi^2} A(\epsilon_F)$, with $A(\epsilon_F)$ being the maximal cross-sectional area of the FS in a plane perpendicular to the magnetic field. By rotating the field with respect to the crystal, one can map the full momentum dependence of the FS.

Another common method for studying the FS is ARPES, which was found to be an ideal tool for studying the topological insulators. ARPES allows one to observe directly the surface states even in samples where the transport is dominated by the bulk. On the other hand, the surface of a topological-insulator is a very complicated environment for photoemission; the ARPES spectrum consists of contributions from the bulk bands and surface states and possibly from a confined 2D electron gas. One way to disentangle these contributions is to scan the photon energy used in the experiment. Different photon-energies provide information about the dispersion at different $k_z$ values and allow one to distinguish 2D-like bands from the 3D bulk band.

In this work we use the two powerful experimental tools, SdH and ARPES; their combination allows us to determine the evolution of the FS as a function of carrier concentration. Using ARPES we show that the Dirac surface states exist throughout the carrier concentration range under study. However, only parts of the bulk-band FS can be clearly seen using ARPES due to the photon-energy dependence of the matrix elements. The SdH effect, in principle, allows a direct mapping of the entire FS but its amplitude depends exponentially on the effective mass. Consequently, it is less sensitive to high-band-mass regions on the FS. Despite the limitations of both probes, together they bring deep insight into the shape and properties of the FS.

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FIG. 1. (Color online) Transport and ARPES characterization. (a) Longitudinal resistivity versus temperature for Cu-doped Bi$_2$Se$_3$ with $n \approx 10^{20}$ cm$^{-3}$. Metallic type behavior as well as superconductivity below $\approx 3$ K is observed. Different samples may exhibit various superconducting volume fractions and $T_c$ variations. (b) Transverse resistivity versus magnetic field at 2 K for three samples. The solid lines are linear fits from which we extract the carrier concentration $n$. Inset: The same for $n \approx 10^{19}$ cm$^{-3}$. (c) Typical ARPES data from a highly doped sample measured with 20-eV photon energy. The detector image shows the dispersion along the $\Gamma$-K direction. One can see that both the surface-state and the bulk-band dispersion coexist. (d) Surface-state dispersion as measured using ARPES for eight different samples with carrier concentrations ranging from $4 \times 10^{17}$ cm$^{-3}$ (green curve) to $4 \times 10^{20}$ cm$^{-3}$ (gray curve).

II. METHODS

For this experiment, we prepared a series of Bi$_2$Se$_3$ samples with different carrier concentrations. We used two kinds of samples: off-stoichiometric Bi$_2-x$Se$_{3+x}$ with low carrier density ($n \approx 10^{17}$–$10^{19}$ cm$^{-3}$), and Cu intercalated Bi$_2$Se$_3$ with high carrier density ($n \approx 10^{20}$ cm$^{-3}$). All the samples were prepared using the modified Bridgeman method as described in Refs. 4 and 17. For transport measurements, flakes $\sim$1–30 $\mu$m thick were freshly cleaved perpendicular to the C3 axis in a nitrogen environment. Gold contact wires were attached to the samples using silver paint. Hall measurements up to
FIG. 2. (Color online) SdH data and analysis. (a) Longitudinal resistance versus magnetic field applied parallel to the C3 axis ($\theta = 0$) at 4.2 K for $n \approx 10^{20}$ cm$^{-3}$ (sample A). Inset: Fast Fourier transform (FFT) of these data plotted versus $1/\mu_0 H$ after subtracting a smooth polynomial background. The sharpness of the FFT peak indicates a well-defined frequency. Its full width at half maximum is used as an upper limit for the uncertainty in determining the frequency. A drawing of the sample configuration used in this experiment is also shown. (b) Resistance versus $1/\mu_0 H$ after subtraction of a smooth polynomial background at various temperatures [4.2 K data are taken from Fig. 2(a)]. The field is applied parallel to the C3 axis. Inset: Effective mass is extracted by following the oscillation amplitude at high field as a function of the temperature. The solid line is a fit to the Dingle formula, yielding $m^* \approx 0.24 m_e$ for this sample ($n \approx 10^{20}$ cm$^{-3}$). (c) The frequency as determined from the FFT versus tilt angle $\theta$ between the magnetic field and the C3 axis for three carrier concentrations. Solid lines are fits for an ellipsoidal FS ($n \approx 10^{17}, 10^{19}$ cm$^{-3}$) and for a cylindrical FS [$F \propto \cos(\theta)$] for $n \approx 10^{20}$ cm$^{-3}$ (sample B).

III. RESULTS AND DISCUSSION

Typical resistivity versus temperature and low temperature Hall measurements are shown in Figs. 1(a) and 1(b), respectively. Typical ARPES data from a Cu intercalated sample with $n \approx 4 \times 10^{20}$ cm$^{-3}$ are shown in Fig. 1(c). A well-defined surface state can be seen, with a Dirac point at about 500 meV below the Fermi level. The two linearly dispersive surface-state branches enclose the parabolic bulk band whose FS is the subject of this paper. Figure 1(d) shows the dispersion of the surface states for various samples with different carrier concentrations $n$. Upon changing $n$, the Dirac

FIG. 3. (Color online) Evolution of the Fermi surface with carrier concentration. [(a) and (b)] Calculated ellipsoidal FS from the SdH data in Fig. 2(c), for $n \approx 10^{17}, 10^{19}$ cm$^{-3}$, respectively. Detailed profile view of the Fermi surfaces is shown. (c) Calculated FS using tight binding corrugated cylinder model fit to the SdH data in Fig. 2(c) (sample B, $n \approx 10^{20}$ cm$^{-3}$) (see Appendix E for more information). (D) The Brillouin-zone momenta axes. [(e)–(g)] The Fermi surfaces of (a)–(c), respectively, plotted to scale with respect to the Brillouin zone.
that the bulk band is visible at the entire photon-energy range measured, which covers a momentum range larger than the
visible at the entire photon-energy range measured. In panel (b) we show low-photon-energy data, believed to be more bulk sensitive. We find
the low-carrier-density sample the bulk band is seen only around 19 eV (a).

The resistance slope) remains unchanged.

from Fig. 2(c). The first two samples

The FS of the n \approx 10^{20} cm^{-3} is clearly more elongated than the FS of n \approx 10^{17} cm^{-3}. For n \approx 10^{20} cm^{-3} the oscillation amplitude decreases with increasing angle and cannot be observed beyond an angle of 55°. This angular dependence of the SdH frequency follows almost perfectly \( F \propto \frac{1}{\cos(\theta)} \), which is the dependence expected for a cylindrical FS.22

In Fig. 2 we show SdH data [see lower inset of Fig. 2(a) for the configuration used in this experiment]. The resistance as a function of magnetic field for a highly doped sample (n \approx 10^{20} cm^{-3}) is shown in Fig. 2(a). Clear SdH oscillations can be seen. The Fourier transform of these oscillations is shown in the upper inset. A single, well-defined frequency can be seen. The Fourier transform of these oscillations is

In Fig. 4(b) and 4(c) for two highly doped samples, the 2D surface states are insensitive to the photon energy used.

[Fig. 4. (Color online) Photon energy dependence of the ARPES data. We show normal emission data for three different samples: (a) n = 4 \times 10^{17} cm^{-3}, (b) n = 4 \times 10^{18} cm^{-3}, and (c) n = 2 \times 10^{19} cm^{-3}. The white dots represent the bottom of the bulk band. For the low-carrier-density sample the bulk band is seen only around 19 eV (\Gamma point), and for the high-carrier-density samples the bulk band is visible at the entire photon-energy range measured. In panel (b) we show low-photon-energy data, believed to be more bulk sensitive. We find that the bulk band is visible at the entire photon-energy range measured, which covers a momentum range larger than the \Gamma-Z separation.]

dispersion remains intact with a rigid shift of the Dirac point towards lower energies, while the Fermi velocity (i.e., the slope) remains unchanged.

In Fig. 2 we show SdH data [see lower inset of Fig. 2(a) for the configuration used in this experiment]. The resistance as a function of magnetic field for a highly doped sample (n \approx 10^{20} cm^{-3}) is shown in Fig. 2(a). Clear SdH oscillations can be seen. The Fourier transform of these oscillations is shown in the upper inset. A single, well-defined frequency is observed. The oscillations persist up to surprisingly high temperatures [see Fig. 2(b)]. From the temperature dependence of the amplitude, the effective mass m^* \approx 0.24m_e is extracted [see the inset in Fig. 2(b)]. In Fig. 2(c) we show the angular dependence of the SdH frequency for three samples. For the low-carrier-concentration samples the oscillations persist up to a tilt angle of 90° [see Fig. 2(c)], indicative of a closed ellipsoidal FS. These results are in agreement with previous observations.15,18–20 The FS of the n \approx 10^{19} cm^{-3} is clearly more elongated than the FS of n \approx 10^{17} cm^{-3}. For n \approx 10^{20} cm^{-3} the oscillation amplitude decreases with increasing angle and cannot be observed beyond an angle of 55°. This angular dependence of the SdH frequency follows almost perfectly \( F \propto \frac{1}{\cos(\theta)} \), which is the dependence expected for a cylindrical FS.22

In Fig. 3 we show the FS of these samples as reconstructed using the SdH data from Fig. 2(c). The first two samples with n \approx 10^{17}, 10^{19} cm^{-3}, have an ellipsoidal FS. For the third sample (n \approx 10^{20} cm^{-3}) we fit our data to a simplified corrugated-cylinder model (see Appendix E for more information). The resulting FS is shown in Figs. 3(c) and 3(g). Our SdH data suggest a transition in the shape of the FS from a closed ellipsoid to an open FS as n increases. Below we show that the ARPES data verify this effect.

In an ARPES experiment the signal intensity allows a direct mapping of the electronic dispersion along momentum directions which are parallel to the sample surface. This is because only the in-plane momentum is conserved. To map the dispersion along k_Z, one needs to scan the photon energy. We used the free-electron final-state approximation21 to find the correspondence between the photon energy and k_Z (see Appendix B for more information).

In order to map the dispersion along the k_z direction, we performed ARPES measurements at normal emission over a wide range of photon energies in steps of 0.5 eV. This was done for various samples with different carrier concentrations. A set of scans is shown in Fig. 4(a) for a n \approx 10^{17} cm^{-3} sample and in Fig. 4(b) and 4(c) for two highly doped samples, n \approx 10^{20} cm^{-3}. One can see in Fig. 4 that, as expected, the 2D surface states are insensitive to the photon energy used.

For the low-n sample shown, the bulk band is visible only in a narrow range of photon-energies around 20 eV, which corresponds to the \Gamma point, and completely vanishes as the photon energy is changed. This indicates that on going along the \Gamma-Z direction the dispersion crosses the chemical potential and that the FS is closed. On the other hand, for the high-n samples, the bulk band remains visible for all photon energies. The maximal width of the bulk band is obtained at about 20 eV (\Gamma point); the band then disperses upward towards a minimum at both zone boundaries located at photon energies of about 14.5 eV and 23.5 eV. This is a clear indication of an open FS at high n.

Next, we look in more detail at the band structure of a n \approx 10^{20} cm^{-3} sample. Around the \Gamma point the parabolic bulk
band is very clear; this can be seen in Figs. 5(a) and 5(b). By following the peaks in the momentum distribution curves (MDCs), we extract the band dispersion [Fig. 5(b)]. Fitting the data to a simple parabolic dispersion model, we can find $k_F$ and the effective mass at different photon energies. The parabolic best fits are shown as dashed lines in Fig. 5(a). The effective masses resulting from these fits are shown in Fig. 5(c) together with $k_F$. We find that when moving away from the $\Gamma$ point towards the zone-boundary $k_F$ decreases and the effective mass increases substantially.

In Fig. 5(d) we show the same FS shown in Fig. 3(c) with a color code representing the effective mass, which is measured by ARPES [using the parabolic fit in Fig. 5(c)] for various $k_z$. Naively, for this type of FS one would expect two frequencies: the first from the maximal cross section, at the plane going through the $\Gamma$ point, and the second from the minimal cross section at zone boundary (the $Z$ points). The $k_z$ dependence of the effective mass extracted from the ARPES data suggests that the SdH signal arising from the minimal cross section at the zone boundary will be very weak, as the SdH intensity depends exponentially on the effective mass. This, together with our finding that the area of the cross section perpendicular to $k_z$ changes by merely 5%, explains why the second frequency is absent in our measurements. Furthermore, as the angle $\theta$ with respect to the C3 axis increases, the SdH is probing parts of the FS at which the electron’s effective mass is larger, so we expect the signal to become weaker, as observed.

The ARPES data suggests a slightly larger corrugation ratio compared to the SdH results, but overall the agreement between the ARPES and the SdH results is impressive. The two probes yield similar values of $k_F$ for the various samples (see Appendix D for more information), and both techniques show clearly a transition from a closed FS at low carrier concentrations to an open FS at high carrier density. In particular, all superconducting samples have an open FS.

IV. CONCLUSION

In order for a time-reversal-invariant superconductor with odd-parity pairing to be a 3D topological superconductor, it must have a Fermi surface that encloses an odd number of time-reversal-invariant (TRI) momenta in the Brillouin zone. We show here that the FS of Cu$_x$Bi$_2$Se$_3$ encloses two TRI points, $\Gamma$ and $Z$. Our results cast doubt on Cu-doped Bi$_2$Se$_3$ as a possible realization of a TRI 3D topological superconductor. Interestingly, this material can be a realization of a 2D-like weak topological SC. Such a system is predicted to have counterpropagating edge states that can produce Andreev bound states but not on the (001) surface. If this is the case, the observed ZBCPs in recent point contact experiments can be a result of tunneling into crystalline facets exposing surfaces other than the (001) one.

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APPENDIX A: RIGID SHIFT

The data presented in Fig. 1(d) of the main text are cuts of the surface state in the ΓK direction passing through the Γ point. The dispersion was obtained by tracking the peak in the MDCs in the ARPES spectra. The energy at which we find the Dirac point and the Fermi momentum (of the surface state) for each of these samples is plotted in Fig. 6. There is a linear relation between these two quantities, which is expected in the case of a rigid shift of the chemical potential in a linearly dispersing band, where the chemical potential is determined by the carrier density. The scatter in the plot is a result of slight misalignment of the different samples.

APPENDIX B: INNER POTENTIAL

We relate the value of \( k_z \) for electrons inside the crystals to their kinetic energy after photoemission using the free-electron final-state approximation

\[
\frac{\text{\textit{k}}_z}{\text{\textit{Å}}^{-1}} = \sqrt{\frac{2m}{\hbar^2} (E_{\text{kin}} + V_0)}
\]

The mapping of band dispersion in the \( k_z \) direction is then carried out by changing the photon energy used in the photoemission process, thus changing the value of \( E_{\text{kin}} \) and obtaining a different value for \( k_z \). The constant \( V_0 \) is specific to the material and is called the inner potential; formally it is given by

\[
V_0 = \mu + \phi,
\]

where \( \mu \) is the chemical potential measured from the bottom of the band and \( \phi \) is the work function. We can determine the value of \( V_0 \) experimentally from the ARPES data, similarly to what appears in Fig. 4 of the main text, by identifying the high-symmetry points in the dispersion along \( k_z \). In Fig. 7 we plot the position of the high-symmetry points as a function of photon energy and obtain a value of approximately \( V_0 \sim 10.3 \text{ eV} \).

APPENDIX C: BAND DISPERSION ALONG \( k_z \)

To make a more quantitative analysis, we need to find the bottom of the band for each \( k_z \) cut. This is found to be a tricky task, and for some photon energies, a clear parabolic dispersion is seen in the data but for other photon energies we find a parabolic region "filled" with almost constant intensity. We look for the energy for which the intensity drops to half its maximal value; this criterion is used for the entire data set. The \( k_z \) values were calculated using the free-electron final-states assumption with an inner potential \( V_0 = 10.3 \text{ eV} \).

In Fig. 8, we plot the dispersion of the bulk band along \( k_z \), for a few samples with different chemical potential values. The data for the different samples are plotted relative to the bottom of the band for each sample. The horizontal solid lines represent the chemical potential for each sample measured.

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**FIG. 6.** (Color online) The energy position of the Dirac point and the value of \( k_F \) for the different samples appearing in Fig. 1(d) of the main text. Error bars are 95% confidence levels.

**FIG. 7.** (Color online) Finding the inner potential \( V_0 \): The high symmetry points are identified from the photon energy scans, similarly to what is shown in Fig. 4 of the main text. The \( k_z \) axis separation between the data points is taken to be the Γ − Z distance, and the error bars show the uncertainty in the position. The dashed line is the equation of free-electron final-state approximation (see text) plotted with \( V_0 = 10.3 \text{ eV} \).

**FIG. 8.** (Color online) Dispersion of the bulk band along the \( k_z \) axis. The plot contains data from five different samples with varying Fermi energies (noted by the horizontal dash lines) as measured from the bottom of the bulk band at the Γ point.
relative to the bottom of the band. One can see that the band shifts rigidly with the increasing doping level; this is apparent in the way the data for different samples coincide. The dispersion of the different samples is identical going from the $\Gamma$ point toward the upper $Z$ point. Going from the $\Gamma$ point down, the agreement is not as good. This might be a result of the way we define the bottom of the band. In addition, the free-electron final-state approximation can lead to errors.

The dispersion of the sample with the low chemical potential (36 meV) crosses the Fermi level, an indication of a closed FS. For the other samples, we do not find a crossing of the Fermi level, an indication of an open FS. Furthermore, for the highly doped samples we find a saturation of the occupied carrier density relative to the bottom of the band. One can see that the band shifts rigidly with the increasing doping level; this is apparent in the way the data for different samples coincide. The dispersion of the different samples is identical going from the $\Gamma$ point toward the upper $Z$ point. Going from the $\Gamma$ point down, the agreement is not as good. This might be a result of the way we define the bottom of the band. In addition, the free-electron final-state approximation can lead to errors.

The data points for samples with low carrier density lie slightly below the line, as expected, whereas the points representing high density samples are found above the line. Furthermore, the values we get for $k_F$ in the $k_y$ direction for the samples with $n \sim 10^{20}$ cm$^{-3}$ are larger than the $\Gamma-Z$ distance, indicating that the FS crosses the top zone boundary, meaning that the FS is open. The blue dots in Fig. 9 are the $k_F$ from the SdH data, one can clearly see the good agreement between the values obtained using ARPES and SdH.

APPENDIX E: CORRUGATED CYLINDER MODEL

We use a simple tight-binding model to fit the SdH data. Expanding around $k_x \simeq k_y \simeq 0$, we get the equation describing the entire Fermi surface:

$$\frac{1}{2} t_L (k_x^2 + k_y^2) a^2 + t_z [1 - \cos(k_c c)] = \epsilon_F,$$

(E1)

where $a$ and $c$ denote the crystals constants in the $xy$ plane and $z$ axis, respectively, and $t_L$ and $t_z$ denote the transfer integrals in the $xy$ plane and $z$ axis, respectively, where $t_L > t_z > 0$.

Applying a magnetic field directing at an angle $\theta$ relative to the $z$ axis due to symmetry in the $xy$ plane (taking $\phi = 0$, where $\phi$ is the angle in the $xy$ plane), the equation for the Fermi surface boundary of the cross sectional region is

$$\frac{1}{2} t_L (k_x^2 + k_y^2) a^2 + t_z [1 - \cos(k_c c \tan \theta)] = \epsilon_F.$$  

(E2)

Measuring wave numbers in units of $a^{-1}$ and energies in units of $t_L$ this equation becomes

$$k_x^2 + k_y^2 + 2\eta [1 - \cos(k_c c \tan \theta)] = 2\epsilon_F,$$

(E3)

$$\eta \equiv \frac{t_z}{t_L} < 1, \gamma \equiv \frac{c}{a} > 1.$$  

The solutions for the cross sectional curve is

$$k_y = \pm \sqrt{2\epsilon_F - k_x^2 - 2\eta [1 - \cos(k_c c \tan \theta)]}.$$  

(E4)

Finally, we calculate the cross-sectional area perpendicular to the magnetic field. Using the Onsager relation, the corresponding frequency is given by

$$F(\theta, \eta) = \frac{\hbar}{2\pi ea^2} \int k_x d(k_x / \cos \theta)$$

$$= \frac{1}{\cos \theta} \frac{\hbar}{2\pi ea^2}$$

$$\times \left\{ \int 2(\epsilon_F - \eta) - k_x^2 + 2\eta \cos(k_c c \tan \theta) dk_x \right\}.$$  

(E5)

Expressing the Fermi energy using the zero angle frequency (where the magnetic field is perpendicular to the $xy$ plane),

$$F_0 \equiv F(\theta = 0, \eta) = \frac{\hbar \epsilon_F}{ea^2}.$$  

The final equation for the angular dependence of the SdH frequency is

$$F(\theta, \eta) = \frac{\Delta(\theta, \eta)}{\cos \theta},$$

$$\Delta(\theta, \eta) = \frac{\hbar}{2\pi ea^2} \int \left\{ 2(F_0 a^2 / \hbar - \eta) - k_x^2 + 2\eta \cos(k_c c \tan \theta) \right\}^{1/2} dk_x.$$  

(E6)
Performing a numerical integration (on $k_z$) combined with a numerical fit (frequency versus magnetic field tilt angle), one can extract the desired parameter $\eta$ (which defines the corrugation of the cylinder). Our experimental data (the frequency $F$ at different magnetic field tilt angles $\theta$) enables us to perform this analysis. We note that this simple theoretical model does not allow us to find the dependence of the cyclotron mass on $k_z$ as experimentally determined in Fig. 5 in the paper.

APPENDIX F: ADDITIONAL SDH DATA AND ANALYSIS

In Figures 10, 11, and 12 we present the SdH measurement of three different samples with various carrier concentrations (presented in the paper itself). The measurements are at various tilt angles between the C3 axis and the magnetic field. For these measurements we present FFT after background subtraction for each tilt angle and a fit for each samples Fermi surface.
FIG. 12. (Color online) Sample with $n3D \simeq 10^{20}$. (a) Longitudinal resistance versus magnetic field at various tilt angles. (b) Frequency versus tilt angle. The solid line is a fit for a cylindrical Fermi surface model $[F \propto \frac{1}{\cos(\theta)}]$. Inset: FFT analysis for each tilt angle.