A pressure-induced topological phase with large Berry curvature in Pb$_{1-x}$Sn$_x$Te

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The picture of how a gap closes in a semiconductor has been radically transformed by topological concepts. Instead of the gap closing and immediately reopening, topological arguments predict that, in the absence of inversion symmetry, a metallic phase protected by Weyl nodes persists over a finite interval of the tuning parameter (for example, pressure $P$). The gap reappears when the Weyl nodes mutually annihilate. We report evidence that Pb$_{1-x}$Sn$_x$Te exhibits this topological metallic phase. Using pressure to tune the gap, we have tracked the nucleation of a Fermi surface droplet that rapidly grows in volume with $P$. In the metallic state, we observe a large Berry curvature, which dominates the Hall effect. Moreover, a giant negative magnetoresistance is observed in the insulating side of phase boundaries, in accord with ab initio calculations. The results confirm the existence of a topological metallic phase over a finite pressure interval.

INTRODUCTION

Topological concepts have greatly clarified the role of symmetry in protecting electronic states in a host of materials. In bulk semiconductors, topological insights have revised the picture of how the energy gap closes (say, under pressure $P$). In the old picture, the gap $\Delta$ closes at an “accidental” value of $P$ before reopening at higher $P$. The new view ($1-4$) predicts instead that, when inversion symmetry is broken, a gapless metallic state featuring pairs of Weyl nodes persists over a field interval ($P_1 \rightarrow P_2$). They act as sources and sinks of Berry curvature (an effective magnetic field in $k$ space). The metallic phase is protected because the nodes come in pairs with opposite chiralities ($\chi = \pm 1$). Hence, they cannot be removed except by mutual annihilation (which eventually occurs at the higher pressure $P_2$). To date, these predictions have not been tested.

Here, we show that Pb$_{1-x}$Sn$_x$Te exhibits a pressure-induced metallic phase described by the Weyl scenario. The Pb-based rock salts (5-7) have been identified as topological crystalline insulators with surface states protected by mirror symmetry (8-12). We focus on their Dirac-like bulk states (13, 14), which occur at the $L$ points of the Brillouin zone (BZ) surface. Pb$_{1-x}$Sn$_x$Te exhibits an insulator-to-metal (IM) transition at $P \sim 10$ kbar (7). However, the IM transition is little explored. We report that the metallic state appears by nucleating 12 small Fermi surface (FS) nodes. The breaking of time-reversal symmetry (TRS) in applied $B$ leads to a large Berry curvature $\Omega$. Finally, we also observe an anomalously large negative magnetoresistance (MR), which is anticipated in the Weyl scenario.

RESULTS

Phase diagram under pressure

Crystals of Pb$_{1-x}$Sn$_x$Te were grown by the vertical Bridgman technique (see Materials and Methods). Transport measurements at temperatures $T$ down to 2 K were carried out in a Be-Cu pressure cell (with a maximum pressure of $P_{\text{max}} \sim 28$ kbar) on four samples with Sn contents $x = 0.5$ (samples A1 and A2), 0.32 (Q1), and 0.25 (E1) (Table 1). In A1 and A2, indium (6%) was added to tune the chemical potential (see Materials and Methods).

Figure 1 provides an overview of the IM transitions in samples A2 and E1. As $P$ increases from 0 kbar (ambient pressure) to 25.4 kbar, the resistivity profile $\rho$ versus $T$ changes from insulating to metallic behavior in A2 (Fig. 1A). Close examination reveals a kink in $\rho$, indicating a sharp transition at $T_c$ = 62 to 70 K (arrows in inset). Figure 1B shows the resistive increase in the zero-$B$ conductivity $\sigma = \rho/\Omega$ at 5 K as $P$ exceeds the critical value $P_1 \sim 15$ kbar at the IM transition. The resistivity curves in sample E1 ($x = 0.25$) are broadly similar except that $\rho$ at ambient $P$ attains much higher values at 5 K ($3 \times 10^4 \Omega$ cm). As $P$ increases from ambient to $P_1$ (12 kbar), an IM transition occurs to a metallic state (with $\rho$ decreasing by over seven decades at 5 K). In samples E1 and Q1 (which have smaller Sn content than A1 and A2), the second critical pressure $P_2 \sim 25$ kbar is accessible in our experiment. The profile of $\sigma$ versus $P$ at 5 K (Fig. 1D) shows the metallic phase sandwiched between the two insulating phases.

The end member SnTe is known to be ferroelectric (FE) (15), but the existence of FE distortion is less obvious for finite Pb content. To establish inversion symmetry breaking, we performed dielectric measurements (see Materials and Methods) on sample E1, which has a very large $\rho$ below 10 K ($>10^3 \Omega$ cm). By varying the $E$-field (12–100 V/cm), we show that a large spontaneous dielectric response $\varepsilon_1 \sim 5 \times 10^4$ exists in the limit $E \rightarrow 0$ (Fig. 1C, inset). The spontaneous polarization $P_s$ provides direct evidence that the insulating state below $P_1$ in E1 is FE. Although dielectric measurements cannot be performed in A2 (carrier screening is too strong), the kink in $\rho$ (arrow) implies that $P_s$ appears at 62 to 70 K.

In parallel, we performed ab initio calculations (see details in the Supplementary Materials), in which the lattice parameter $a$ is varied to simulate pressure. To break inversion symmetry, we assumed a weak FE displacement $d$ $\parallel$ [111]. The calculations reveal that, above $P_1$, two pairs of Weyl nodes appear near each of the points $L_1$, $L_2$, and $L_3$ (these are equivalent in zero $B$; see Fig. 1, E and F). As $P$ increases, the 12 nodes trace out elliptical orbits (shown expanded by a factor of 10 relative to the BZ caliper) and eventually annihilate pairwise (indicated by black dots), consistent with the scenario described by Murakami (2) and Murakami and Kuga (3). The red and blue arcs refer to nodes with...
\( \chi = 1 \) and \( -1 \), respectively. The splitting of the node at \( L_0 \) occurs in a much narrower pressure interval.

### Quantum oscillations

The samples' high mobilities \( \mu (20,000 \text{ to } 4 \times 10^6 \text{ cm}^2/\text{Vs}; \text{see Table 1}) \) allow us to “count” the number of FS pockets by monitoring the Shubnikov–de Haas (SdH) oscillations. As shown in Fig. 1 (B and D), \( \sigma \) increases steeply with the reduced pressure \( \Delta P = P - P_1 \). Figure 2A shows the resistivity \( \rho_{xx} \) measured in sample A2 in a transverse magnetic field \( B (||z) \) at selected values of \( P \). From the linear variation of \( 1/B \sigma \) versus the integers \( n \) (where \( B_\sigma \) is the peak field in \( \rho_{xx} \); see Fig. 2B), we find that the FS caliper area \( S_F \) increases from 1.6 to 2.7 T between 19 and 25.4 kbar. The most prominent peak in Fig. 2A corresponds to the \( n = 1 \) Landau level (LL). The SdH-derived Fermi wave vector \( k_F \) corresponds to a hole density \( n_{11} = \frac{3}{2} \pi k_F^3 / (2\pi)^3 \) per spin (assuming a spherical FS).

The sharp increase in hole density is also evident in the Hall resistivity \( \rho_{xy} \) (which is \( B \)-linear in weak \( B \)). To highlight its behavior, we plot the ratio \( \rho_{xy}/\rho_0 \) versus \( B \) (Fig. 2C). In weak \( B \) (for example, \( |B| < 3.5 \) T in the top curve), the ratio is \( B \)-independent, which allows the ratio to be identified with the Hall density \( n_{11} \) (the abrupt increase above 3 T arises from the interesting anomalous Hall term discussed below). From \( n_{11} \), we derive \( \mu \approx 1.8 \times 10^4 \) and 2.86 \( \times 10^4 \) cm\(^2\)/Vs in A1 and A2, respectively, at 25 kbar.

Crucially, we find that \( n_{11} \) always exceeds \( n_{11}^{\text{H}} \) by an order of magnitude. This implies a large number \( N_F \) of identical pockets. The ratio \( n_{11}/n_{11}^{\text{H}} = N_F \) equals 12 \( \pm 1 \) over the whole pressure interval (Fig. 2B, inset). Because a smaller \( N_F \) (for example, 4, 6, or 8) can be excluded, the results strongly support the choice \( d || [111] \), which leads to three equivalent \( L \) points.

#### Anomalous Hall effect

We next describe the evidence for a topological metallic phase. The Hall resistivity \( \rho_{xy} \) displays a highly unusual field profile. As \( B \)
increases, the initial $B$-linear behavior abruptly changes, bending over to a nominally flat profile (Fig. 3A). At first glance, this recalls the anomalous Hall effect (AHE) in a ferromagnet (16) where the intrinsic AHE arises from a large Berry curvature rendered finite by the spontaneous breaking of TRS, but there is a subtle difference. In PbSnTe, TRS re-appears from a large Berry curvature rendered finite by the spontaneous breaking of inversion symmetry (Supplementary Materials). In terms of the integers, 1 because of inversion-symmetry breaking. With increasing $p$, the four Weyl FS near $L_1$ move apart and expand in volume [chirality $\chi = 1$ (red) and $-1$ (gray)].

Each Weyl node acts as a source ($\sigma^0_{xy}$) and Fermi energy of one pair of nodes, say $\sigma^0_{xy}$, is most prominent within the $n = 0$ LL (which is strictly chiral for Weyl fermions) suggests to us that it is intimately related to the chirality of the nodes.

Each Weyl node acts as a source (\(\chi = 1\)) or sink (\(\chi = -1\)) of \(\Omega\). As mentioned, in zero $B$, TRS requires the net sum of \(\Omega\) over each pair of Weyl nodes to vanish (Fig. 3D). The ab initio calculations (Supplementary Materials) reveal how this cancellation is spoiled when TRS is broken in finite field $B$. A finite Zeeman field $\lambda$ shifts the band energies, depending on their spin texture. This increases the $k$-space separation and Fermi energy of one pair of nodes, say $\omega^2$, while reducing them in the other $\omega^2$ (Fig. 3D). The unbalancing creates a finite $\Omega$ (hence, $\sigma^0_{xy}$) that grows with $B$ (Supplementary Materials).

**Giant MR**

Perhaps the most marked feature in Pb$_{1-x}$Sn$_x$Te is the appearance of giant negative MR at pressures just above $P_2$. In Fig. 4A, we show the
MR curves in sample Q1 for selected $T$ with $P$ fixed at 28 kbar (roughly 3 kbar above $P_1$). At 4.3 K, $\rho_{xx}$ decreases by a factor of 30 as $B$ increases to 10 T (aside from a slight dip feature below 0.5 T). In Fig. 4B, similar curves for E1 (at pressure $P = 25.4$ kbar) show an even larger negative MR (the weak-$B$ dip feature is more prominent as well). The large negative MR is steadily suppressed as we increase $P$ beyond the $P_2$ boundary. The negative MR magnitude is similar in magnitude in both the transverse MR and longitudinal MR geometries ($B \parallel z$ and $B \parallel x$, respectively). This implies a Zeeman spin mechanism. Finally, we note that, in both Q1 and E1, $\rho_{xx}$ measured at 10 T decreases as $T \to 5$ K (that is, the system is metallic).

**DISCUSSION**

The anomalously large changes in $\rho_{xx}$ imply that the insulating state (at zero $B$) is converted to a metallic state in finite $B$. This is confirmed in the ab initio calculation (Supplementary Materials). A large $\lambda$ favors the Weyl phase (the left V-shaped yellow region in Fig. 4C). As the phase boundary now tilts into the insulating side, the metallic phase is reentrant in increasing $B$. The observation of the giant negative MR provides further evidence in support of the Weyl node scenario.

As predicted in previous studies (1–4), gap closing in materials lacking inversion symmetry leads to a metallic phase that is protected by the distinct chirality of Weyl nodes. Pb$_{1-x}$Sn$_x$Te is an instructive first example. Increasing pressure $P$ drives an IM transition at $P_1$, with $\rho$ (at 5 K) falling by four to seven orders of magnitude. Above $P_1$, the growth of the FS calipers is tracked by large SdH oscillations. The number of nodes (12) is consistent with the appearance of four Weyl nodes at each of the 3 $L_1$ points on the BZ surface. The Berry curvature, rendered finite in $B$, leads to an AHE that is most prominent in the $n = 0$ LL. Finally, we find that the boundary $P_2$ is shifted in finite $B$. The reentrance of the metallic phase leads to a marked decrease in $\rho_{xx}$ by a factor of 30 to 50.

**MATERIALS AND METHODS**

**Crystal growth**

Single crystals of Pb$_{1-x}$Sn$_x$Te were grown by the conventional vertical Bridgman technique. High-purity elements (5N) with the targeted values of $x$ were sealed in carbon-coated quartz tubes under a high vacuum of $\sim 10^{-5}$ mbar. The ampoules were heated at 1050°C for 12 hours. To ensure homogeneous mixing of the melt and to avoid bubble formation in the bottom, we stirred the ampoules. The ampoules were slowly lowered through the crystallization zone of the furnace, at the rate of 1 mm/hour. High-quality single-crystal boules of length ~10 cm were obtained. The crystal boules were cut into segments of 1 cm to investigate the bulk electronic properties along the boule length. The crystals were easily cleavable along different crystallographic planes.
A major difficulty in the rock salts is having to ensure that the chemical potential of the alloy lies within the bulk gap (otherwise, the pressure-induced changes to the gap will not be observable). To achieve this goal in crystals with Sn content \( x = 0.5 \), we have found it expedient to dope the starting material with indium [at the 6% level, with composition \((\text{Pb}_{0.5}\text{Sn}_{0.5})_{1-x}\text{In}_x\text{Te}\), with \( y = 0.06 \)]. Indium doping has previously been carried out and investigated by several groups to understand the super-conducting phase in \( \text{Pb}_x\text{Sn}_y\text{Te} \) (17–19). Zhong et al. (19) have reported that In-doped \( \text{Pb}_x\text{Sn}_y\text{Te} \) (\( x = 0.5 \)) induces an insulating behavior. However, in our judgment, the precise role of In doping in the Pb-based rock salts is not well understood and merits further detailed investigation.

The x-ray diffractograms recorded for two powdered specimens of some of the parameters measured in the four samples investigated (A1, A2, E1, and Q1) are reported in Table 1. The mobilities in E1 (p-type) and Q1 (n-type) were very high (500,000 and 4.2 × 10^6 cm^2/Vs, respectively). Although the samples with \( x = 0.50 \) (A1 and A2) had lower mobilities (18,000 and 29,000 cm^2/Vs, respectively), clear SdH oscillations were observed above \( P_1 \).

**Measurement of dielectric constant**

We provide more details on the measurements of the relative dielectric constants \( e_1 \). We adopted the (modified) Sawyer-Tower method (20, 21).

The circuit of the original Sawyer-Tower method is shown in Fig. 5A. Figure 5B shows the modified method using an operational amplifier (op-amp) (21).

For the setup shown in Fig. 5A, the sample with a capacitance component \( C_0 \) and a resistance component \( R_0 \) was connected in a series with a known reference capacitance \( C_0 \geq C_0 \). The reference capacitance \( C_0 \) was connected in parallel with a fixed resistor \( R_0 \) in series with an adjustable voltage source \( V_0 \) (or, equivalently, a variable resistor \( R_0 \)) that was set such that the voltage \( V_x \) is inphase with voltage \( V_0 \), where \( V_x \) and \( V_y \) represent the voltages across the whole electrical circuit and the reference capacitor \( C_0 \), respectively. Because \( C_0 \geq C_0 \) was satisfied, the following relations hold: \( V_y \sim V_x, V_y \sim V_x/C_0 \ll V_x \). Here, \( V_x \) is the voltage applied across the sample. This means that point M in the figure can be treated as a virtual ground. This yields the condition \( V_0/R_0 \sim V_x/R_0 \).

The setup in Fig. 5B is the same as that in Fig. 5A except that the op-amp is used to provide a more stable virtual ground at point M. From point M, current \( I_0 \) was driven to the op-amp connected to \( C_0 \) and \( R_0 \) in parallel. Here, a large resistor \( R_0 (R_0C_0 \gg 1/\alpha) \) was connected in parallel as the leak resistor so that the capacitor \( C_0 \) does not become overloaded. The integration circuit yielded the equation \( V_y = C_0^{-1} \int I_x(t) dt \). If the adjustable voltage source \( V_x \) is set to cancel the current \( I_x \) flowing into the sample, that is, if \( V_x/R_0 = V_x/R_0 \) is satisfied, then the current \( I_x \) equals the current flowing into the capacitive component of the sample \( I_x = dQ/dt = d(C_0 V_x)/dt \). This gives \( V_y = V_x C_0/C_0 \), similar to the expression obtained for the case in Fig. 5A.
The advantage of using the op-amp is that one can choose any value of $C_2$ (as long the condition $R_2C_2 > \frac{1}{\alpha}$ is satisfied) to attain a larger signal $V_y$ than possible in the case in Fig. 5A. In general, the two setups in Fig. 5 (A and B) worked well. However, because the working frequency range of our op-amp (LF356N) was between 30 Hz and 30 kHz, we used setup A above 30 kHz. For frequencies below 30 kHz, both setups A and B were used. We confirmed that the two setups yielded the same results.

The measured results of the relative dielectric constants are plotted in Fig. 5C for $f = 100$ kHz and in Fig. 5D for $f = 10$ kHz. Figure 5C shows that $V_y = V_xC_2/C_1 = DS/C_2$ is proportional to $V_x = Et$. Here, $E$ is the electric field in the sample, $D$ is the dielectric displacement, and $S$ and $t$ are the area and the thickness of the sample, respectively. Using the values $S = 0.234$ mm$^2$ and $t = 0.79$ mm, we found the relative dielectric constants $\varepsilon_1 \sim 5 \times 10^4$ (shown in the inset of Fig. 5C). Unfortunately, the sample suffered dielectric breakdown above $E \sim 100$ V/cm, which prevented us from observing the saturation of dielectric displacement $D$ at higher electric fields as should be expected from the FE behavior. This large relative dielectric constant $\varepsilon_1 \sim 5 \times 10^4$ observed in the limit $E \to 0$ strongly implies that the system is in the FE state.

We remark that the nonlinear behavior shown in Fig. 5D, previously interpreted by Möllmann et al. (22) as evidence for the FE state, is actually not the manifestation of the FE state. Rather, it arises from the fact that the resistive component of the sample $R_1$ is strongly $E$-dependent, as shown in the inset. Because $R_1$ is nonlinear, it is not possible to compensate the current $I_x$ flowing through the resistive component $R_1$ because $V_1/R_1 = V_x/R_1$ cannot be satisfied for every $V_x$ unless $V_1$ is changed nonlinearly. Therefore, if $V_1$ is set to compensate the $R_1$ at some fixed value of $V_x$ (7 V in the case of Fig. 5D), then other parts of $V_x$ cannot be compensated. As a result, they produce a nonlinear behavior that looks like the saturation expected in the FE state. The way to avoid this is to use a higher frequency $f$ so that a larger portion of the current flows into the capacitive component $C_1$ rather than into the resistive component $R_1$. This is precisely the case shown in Fig. 5C ($f = 100$ kHz).

**SUPPLEMENTARY MATERIALS**

Supplementary material for this article is available at http://advances.sciencemag.org/cgi/content/full/3/5/e1602510/DC1

section S1. Ab initio band calculations

section S2. Field-induced anomalous Hall effect

fig. S1. Calculated k-space trajectories of Weyl nodes in Pb$_{1-x}$Sn$_x$Te ($x = 0.5$) under applied pressure in zero magnetic field.

fig. S2. Phase diagram of the Weyl phase in Pb$_{1-x}$Sn$_x$Te ($x = 0.25$) and orbit parameters.

fig. S3. Phase diagram of the Weyl phase in Pb$_{1-x}$Sn$_x$Te ($x = 0.25$) in the $\alpha$-$\beta$ plane with applied B $\parallel$ [112];

fig. S4. X-ray diffractograms of two powdered specimens of Pb$_{1-x}$Sn$_x$Te taken from the crystal boules.

fig. S5. Supplemental data of (Pb$_{0.5}$Sn$_{0.5}$)$_{1-x}$In$_x$Te for samples A1 and A2.

fig. S6. Supplemental data of Pb$_{0.75}$Sn$_{0.25}$Te for sample E1.

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