Superconductivity and weak anti-localization in nodal-line semimetal SnTaS$_2$

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Received 22 February 2022, revised 21 May 2022
Accepted for publication 1 June 2022
Published 17 June 2022

Abstract
Topological semimetals with superconducting properties provide an emergent platform to explore the properties of topological superconductors. We report magnetization, and magneto-transport measurements on high quality single crystals of transition metal dichalcogenide SnTaS$_2$. It is a nodal line semimetal with superconducting transition below $T_c = 2.9$ K. Moderate anisotropy ($\gamma = 3.1$) is observed in upper critical fields along $H//c$ and $H//ab$ plane. In the normal state we observe large magneto-resistance and weak anti-localization effect that provide unambiguous confirmation of topological features in SnTaS$_2$. Therefore, genuine topological characteristics can be studied in this material, particularly with regard to microscopic origin of order parameter symmetry.

Keywords: superconductivity, single crystal, nodal-line semimetal

(Some figures may appear in colour only in the online journal)

1. Introduction

The role of electronic band topology in the condensed matter systems, particularly in topological superconductors, has attracted considerable attention in the recent past [1–3]. While the theoretical predictions on the characteristic signatures of such systems are well documented [3–6], their experimental manifestations continue to be under persistent debate. In analogy with topological insulators, topological superconductors are predicted to be gapped superconductors in the bulk-interior along with Majorana fermions at the surface states [1, 2, 4, 7]. Realization of superconducting transition in such prototype systems via doping or external pressure has been reported [5, 8, 9]. Early topological superconductors were analyzed to possess topological surface states which was either due to proximity effects [6, 10] or due to intercalation unto topological insulators [5, 11–13]. However, unequivocal confirmation of the predicted p-wave singlet order parameter is still awaited [3, 4]. Furthermore, detection of Majorana fermions has remained an open question. In this paper we report synthesis of high-quality single crystals of SnTaS$_2$ with superconducting $T_c$ of 2.9 K along with clear evidence for topological characteristics in the normal state.

The clearest signature of non-trivial topological band structure is the weak-anti localization (WAL) effect that can be studied using magneto-resistance measurements. This is primarily a quantum interference effect in the quantum diffusive regime [14, 15]. In fact, this is frequently used as an alternative definition of topological insulators because of their delocalized surface states [14]. Over the years, several topological semimetals such as Dirac semimetal, Weyl semimetal, nodal-line semimetal have shown superconductivity with such non-trivial band structure. Some of them exhibit superconductivity in high pressure regime (Cd$_3$As$_2$, WTe$_2$) [8, 9] while some others exhibit superconductivity at ambient pressures (PbTaSe$_2$, PbTaS$_2$). Moreover, superconductors having layered crystal structure are of interest due to their
anisotropic properties and high critical fields that provide them similar with the cuprates [16]. SnTaS$_2$ is one such layered transition-metal dichalcogenide where 2H–TaS$_2$ layers [17] are connected by intercalation of Sn atoms that makes a linear array of Sn–S–Sn units [18, 19]. It is reported to be a nodal line semi-metal [20] and superconducting below the critical temperature $T_c \approx 2.8$ K [21, 22]. It is isoelectronic to PbTaSe$_2$ [23] but it has centrosymmetric structure while PbTaSe$_2$ is non-centrosymmetric crystal. First principle calculations have shown that SnTaS$_2$ exhibits nodal-line band structure and drumhead like states when spin-orbit coupling (SOC) is excluded [23, 24]. There have been some studies [23, 25, 26] on superconducting properties of SnTaS$_2$ but topological aspect related to non-trivial band structure is an open question. In specific, the experimental manifestations of non-trivial band structure in the normal and superconducting states are yet to be unambiguously ascertained. In such scenario the condensate pairing is theoretically predicted to be a single-let p-wave state [3, 4]. In the normal state, the nodal-line fermions and drumhead like surface states [23, 25] are expected to affect the WAL phenomenon. Moreover, the critical question would be to correlate the nodal-line fermionic state to the Majorana zero modes in low dimensional topological superconductor [2, 3, 24]. Here we attempt to address these issues through normal state magneto-transport measurements on high purity single crystals of SnTaS$_2$.

2. Experimental technique

The single crystals of SnTaS$_2$ were synthesized using the chemical vapor transport method that consists of two parts. First, polycrystalline Sn$_{0.33}$TaS$_2$ was synthesized using the conventional solid-state reaction method by putting the stoichiometric mixture of Sn (Sigma Aldrich, 99.8% pure), Tantalum (Alpha Aesar, 99.9% pure) and Sulphur (Sigma Aldrich, 99.98% pure) in an evacuated quartz tube. This was placed in a furnace at 850 °C for two days. Then the polycrystalline sample was mixed with excess Sn powder to achieve elemental ratio of 1:2:2. It was vacuum sealed with iodine (3.5 mg cm$^{-1}$) in a quartz tube. Iodine was used as a transport agent. Excess Sn was mixed to rule out the reappearance of the Sn$_{0.33}$TaS$_2$ phase in the final product [23]. The tube was then put into a two-zone furnace (hot zone at 1000 °C and cold zone at 960 °C) for two weeks. Normal cooling to room temperature yielded shiny flake like single crystals of typical dimension $\sim 2 \times 2 \times 0.02$ mm$^3$. The structural characterization was done at room temperature using Rigaku x-ray diffractometer (XRD, MiniFlex-600 with Cu–Kα radiation). The single crystal XRD was done using Bruker XRD with Mo as x-ray source. The magneto-transport measurements were performed using a Cryogenic Cryogen Free Magnet (8 T, 1.6 K) and a separate 14 T Cryogenic physical property measurement systems. Energy dispersive x-ray spectroscopy (EDAX) and scanning electron microscopy (SEM) imaging were done using Bruker AXS microanalyzer along with a Zeiss EVO40 SEM analyzer, respectively.

3. Results and discussion

SnTaS$_2$ has a layered hexagonal structure that belongs to the space group (p6$_{3}$/mmc). The layered structure consists of alternating stacks of TaS$_2$ and Sn layers [18, 19]. Like 2H phase of TaS$_2$, SnTaS$_2$ is a centrosymmetric compound with the Sn layer held together between TaS$_2$ layers through van der Waals bonding. XRD measurements on as grown crystals were carried out via two methods; on a flake-like thin crystal using a powder diffractometer as well as by collecting Laue data from a single crystal diffractometer. All the reflection peaks resemble with the underlying hexagonal symmetry that can be indexed in (0 0 l) direction (figure 1(a)). This is in agreement with c-axis oriented layered growth of SnTaS$_2$ single crystals [25]. Further, Laue pattern (shown in inset (ii) of figure 1(a)) shows the concentric spot-like circles that reconfirms crystalline structure of single phase in the sample. The obtained XRD data were refined using Fullprof software to deduce the lattice parameters that come out to be $a = b = 3.336$ Å, $c = 17.447$ Å.

This is in agreement with reported data [23, 25, 26]. The schematic unit cell of SnTaS$_2$ is shown in inset (i) of figure 1(a)). A quantitative analysis of stoichiometric ratio was done using EDAX that was performed at several points on sample surface (figure 1(b)). The atomic percent acquired through EDAX is close to the intended stoichiometric values; that Sn:Ta:S comes out to be 30%, 23%, 47% (Sn:Ta:S). The scanning electron microscope image (inset (ii) of figure 1(b)) clearly shows the layered morphology of the specimen. The measurements of temperature dependent electrical resistivity were carried out using linear four probe method. Figure 2 shows the zero field resistivity data from 1.6 K to 300 K. Inset (i) of figure 2 shows the evidence for superconducting transition at $\sim 2.9$ K.

The transition is quite sharp and its transition width is ($\Delta T_c = T_{c\text{onset}} - T_{c\text{zero}} = 0.24$ K). The RRR (residual resistivity ratio $= \rho(300 \text{K})/\rho(4 \text{K})$) is estimated to be 530, which is the highest RRR reported in this system [23, 25, 26]. It reflects high quality of the single crystal that is devoid of defects and impurities. Furthermore, a strikingly linear behavior, with intercept passing through the origin, is seen in the normal state temperature dependence of electrical resistivity. This is akin to optimally doped cuprates [16, 27]. In general terms, electrical resistivity of metals exhibits such linear behavior when the excitonic (phononic or spin waves etc) energy scale is lower than $k_B T$. At lower temperatures, still in the normal state, there would be a crossover to a region where excitonic scale would be higher than thermal energy. This is reflected as a power law behavior of resistivity. The normal state resistivity data therefore could be divided into three parts. First part is from just above the $T_c$ to around 40 K, where resistivity curve shows power law behavior. Second region is from 40 K to 140 K with a slope of 1.83 $\mu \Omega \text{cm} \text{K}^{-1}$ is seen. The third region shows linear behavior with a slope 1.55 $\mu \Omega \text{cm} \text{K}^{-1}$ from 140 K up to 300 K with intercept passing through the origin (inset (ii) of figure 2).

Next we discuss the dc magnetization measurement that was performed in H//lab and H//c directions of the crystal.
Figure 1. (a) X-ray diffraction data of single crystal SnTaS$_2$. Inset (i) of (a) shows the schematic unit cell of SnTaS$_2$. Inset (ii) shows Laue pattern. (b) EDX mapping of SnTaS$_2$ crystal. Inset (i) shows the optical micrograph of single crystal and inset (ii) of (b) shows SEM image of crystal which confirms the layered morphology.

Figure 2. Temperature dependent resistivity from 1.6 K to 300 K for single crystal SnTaS$_2$. Inset (i) shows the superconducting transition in a broader scale. Inset (ii) shows the linear fit to the normal state resistivity (dashed black line) in the range 140 K–300 K.

Zero field cooled (ZFC) and field cooled data were taken along $H_{//ab}$ direction using field of 1 mT which is shown in inset (i) of figure 3 (a).

The critical temperature obtained from magnetization measurements (2.9 ± 0.1 K) matches well with the $T_c$ obtained from resistivity measurements. Superconducting shielding volume fraction of the sample deduced from ZFC susceptibility comes out to be (99%). The magnetization versus magnetic field (M–H) loop for both orientation ($H_{//c}$ and $H_{//ab}$) is shown in figure 3 (a) and inset (ii) of figure 3 (a) respectively. For the determination of lower critical field $H_{c1}$ in $H_{//ab}$ direction, the M–H data at different temperature were taken in the fourth quadrant. This is shown in figure 3 (b). Deviation from the linear diamagnetic behavior extrapolates to the lower critical field at each temperature. Inset (i) of figure 3 (b) shows fitting between $H_{c1}$ as a function of temperature using the parabolic relation $H_{c1}(T) = H_{c1}(0) (1 – t^2)$, where $t$ denotes reduced temperature $T/T_c$. Since the demagnetization effects are negligible in $H_{//ab}$ direction (due to thin platelike sample) lower critical field can be calculated with sufficient accuracy.
The extrapolation of the curve leads to determination of $H_{c1,ab}(0)$ as $4.7 \pm 0.2$ mT.

From the M–H along the $H//c$ direction, taken at 1.6 K (figure 3(a)), the obtained value for $H_{c1,c}$ comes to be 0.58 mT. Using similar extrapolations, $H_{c1,c}(0)$ is estimated to be 1.88 mT. Since demagnetization factor in $H//c$ orientation loop cannot be neglected, we calculated the actual value after demagnetization factor correction by using the Brandt’s formula $[28]$, $H'_{c1,c}(0) = H_{c1,c}(0)/(\tanh(0.67, c a^{-1})^{5/2})$. Here $c$ and $a$ are the thickness of the sample and length perpendicular to the field direction respectively. The demagnetization corrected $H_{c1,c}$ comes out be 20.5 mT.

Next, we discuss the onset of magneto-resistance in the presence of different magnetic fields both in $H//c$ and $H//ab$ directions (figures 4(a) and (b)). The superconducting transition gets broadened when applied field is increased, more so when the field is applied parallel to $c$-axis of the crystal. Critical temperature for various fields is obtained from the mid-point of in-field transition criterion in both the orientations. In $H//c$ direction the $H_\kappa$ versus temperature curve (inset (i) of figure 4(a)) can be fitted using the Ginzburg–Landau equation $H_{c2,c}(T) = H_{c2,c}(0)\left(1 - T^2/T^2_c\right)$, where $t = T/T_c$. The data fit with the GL equation very well and the intercept of the fit at $y$-axis gives the value of upper critical field $H_{c2,c}(0)$. This is estimated to be $25.2 \pm 0.9$ mT. The Pauli limit of upper critical field in weak coupling limit is given by $1.84 T_c = 5.3$ T. This indicates orbital effects limit the upper critical field magnitude in SnTaS$_2$. The temperature dependence of upper critical field in $H//ab$ direction, shows upward behavior which results in deviation from the GL fit (inset (i) of figure 4(b)). In such cases $H_{c2,ab}$ can be fitted using the equation $H_{c2,ab}(T) = H_{c2,ab}(0)\left(1 - T^2/T^2_c\right)$ [25]. The upward curvature in upper critical field (near $T_c$) seen here has been previously reported in SnTaS$_2$ [23, 25, 26], PbTaSe$_2$ [29, 30] and other intercalated chalcogenides [9, 31]. There are several possible origin for this upward curvature in upper critical field in $H//ab$ direction. The primary cause could be the effect of impurities and disorders [32, 33], but this could be discarded on the basis of high RRR of single crystals reported in this study. Other reasons are dimensional crossover [34], multiband effect and non-local effects in clean limit [35]. Previous reports in organic molecules [31, 36] and tantalum based intercalated dichalcogenides [37, 38] have explained this upward behavior with dimensionality crossover model [34]. Further, Pauli paramagnetic limit is also exceeded in such cases. But as the coherence length $\xi_c$ in the case of SnTaS$_2$ is larger than the interlayer spacing ($\sim 8.72$ Å), the bulk behavior is indicated. Further, we note that $H_{c2,ab}(79.1 \pm 0.016$ mT) is less than the Pauli limit. Hence the dimensional crossover model is not suitable to explain upward curvature in upper critical field. By deduction, this implies that possible non-local effects may be the dominant reason for the departure from GL behavior. Furthermore, the anisotropy parameter calculated as the ratio of two upper critical fields i.e. $\gamma = H_{c2,ab}(0)/H_{c2,c}(0)$ yields the value of 3.1. This is close to the value obtained from $H_{c1}$ measurements and is substantially lower than previous reports [23, 25]. The Ginzburg–Landau (GL) coherence lengths in both directions $\xi_{ab}$ and $\xi_c$ are calculated using the formulas $\xi_{ab} = (\Phi_0/2\pi H_{c2,ab})^{1/2}$ and $\xi_c = \Phi_0/2\pi H_{c2,ab}$ respectively [39, 40] with $\Phi_0$ being the magnetic flux quantum. These calculations lead to estimation of coherence length as $\xi_{ab} = 114.3$ nm and $\xi_c = 36.4$ nm. The GL parameters $\kappa_{ab}(0)$ and $\kappa_c(0)$ can be calculated by using the formula $\kappa_{ab}(0) = (\lambda_{ab}/\lambda_c)^{1/2}$ and $\kappa_c(0) = \lambda_{ab}/\kappa_{ab} \kappa_c(0) = \lambda_c/\kappa_c$. From the ratio of upper critical field and lower critical field, $\kappa_{ab}$ can be obtained, because $H_{c2,ab}/H_{c1,ab} = 2\kappa_{ab}^2/(\ln(\kappa_{ab}))$ [13, 23]. Therefore $\kappa_{ab} = 3.039$. This further leads to determination of penetration depth $\lambda_{ab}(0) = 110.6$ nm and $\lambda_c(0) as 347.3 nm.

Next, we turn to the question as to whether the superconducting state in SnTaS$_2$ is derived from non-trivial topological states as indicated in the reported electronic band structure [24, 26]. As we know, WAL and nodal lines are normal state properties. Current evidence shows that SnTaS$_2$ is a fully gapped superconductor [24, 25]. However, it is not clear if in these experiments surface states in the superconducting regime were studied at all. It is possible that the current data on gap estimation can only be ascribed to bulk interior. If the surface states are assessed, then there is possibility that drumhead like states may give rise to correlation with Majorana zero modes.
in vortices of a topological superconductor. SnTaS$_2$ is a nodal line semimetal in the normal state and it is worthwhile to study its topological properties. Such materials have the unique surface states that make them different from trivial class of materials [1–3]. In topological insulators the surface states are helical Dirac states and the upspin and downspin components are delocalized on opposite surfaces intertwined by the bulk states. From transport measurements this is ascertained from weak anti-localization (WAL) theories [14, 15, 41, 42]. The experimental manifestation of this is the negative magnetoconductivity that is the decrease in electrical conductivity in the presence of external magnetic field. This is because in the quantum diffusion regime, the time reversed inverted scattering trajectories interfere destructively, giving rise to enhanced conductivity [14]. External magnetic field can destroy these interference effect which can give rise to negative magnetoconductivity. Although nodal-line semimetals in general do not possess surface states, the trajectory along the nodal line under adiabatic limit is such that it picks a π Berry phase [20]. By definition, there is a minimal overlap of conduction and valence band in a semimetal where the 1D loop constitute the nodal line (Inset (i), figure 5(b)). If the Fermi level is not coincident with the crossover loop then the Fermi surface becomes toroidal. In single crystals, the short range disorder driven scattering will be relatively negligible compared to long range scattering mechanism such as Coulombic scattering etc. Since the scattering momentum along the circumferential direction would be negligible, the cross-sectional loop would be dominant with π Berry phase. Recent theoretical reports have claimed weak localization (WL) and WAL feature in nodal-line semimetals depending on the length scale of mechanism responsible for scattering [43, 44]. Inset (i) of figure 5(a) shows magneto transport measurement up to ±5 T at 5 K in the normal state of single crystal of SnTaS$_2$. A large non-saturating magneto-resistance of 320% is estimated by using the formula \[ \frac{|R(H) - R(0)|}{R(0)} \times 100\% \]. This non-saturating MR can be attributed to topological feature because classical MR saturates at higher fields [38, 45]. A special feature observed is the V-shape cusp like behavior at low fields up to ±1.2 T (figure 5(a)) [46, 47]. This cusp like behavior confirms the WAL effect in SnTaS$_2$. We took MR upto ±1.2 T at different temperatures (15 K, 20 K, 30 K, 50 K, 100 K) to study temperature dependence of MR, which is shown in figure 5(a). Further, to study WAL in magneto-conductivity, we plotted the magnetoconductivity at different temperatures (figure 5(b)). There is the predicted increase in conductivity at lower temperatures (inset (ii) figure 5(b)) and negative magneto-conductivity [14, 15, 41] (figure 5(b)). In the quantum diffusive regime, at low fields, the conductivity can be well described by Hikami–Larkin–Nagaoka (HLN) equation [48]. This equation plays a very important role in determining the values of physical parameters (i.e. phase coherence length, number of conduction channels) which characterize the WAL and WL phenomena in topological materials [41]. We fitted the magnetoconductivity using HLN [48] model (figure 5(b)). HLN equation in strong SOC limit is given as

\[
\delta \sigma (H) = \sigma (H) - \sigma (0)
\]

\[
= - \left( \frac{\alpha e^2}{\pi \hbar} \right) \ln \left( \frac{B_\phi}{H} \right) - \Psi \left( \frac{1}{2} + \frac{B_\phi}{H} \right)
\]

where \(\Psi\) is digamma function, \(B_\phi = h/8\pi e l_\phi^2\) is characteristic field and \(l_\phi\) is the phase coherence length, which is defined as the length travelled by an electron before loosing its phase. It usually depends on the inelastic scatterings in the system. The factor \(\alpha\) represents the number of conduction channels and type of localization present in the material. By fitting magneto-conductivity we obtained the value of \(l_\phi\) at different temperatures which are plotted in inset (iii) of figure 5(b). Values of \(l_\phi\) are 195.2 nm, 30.5 nm, 17.9 nm, 11.4 nm, 7.5 nm at 5 K, 15 K, 20 K, 30 K and 50 K respectively. Clearly, the phase coherence

\[
\delta \sigma (H) = \sigma (H) - \sigma (0)
\]

\[
= - \left( \frac{\alpha e^2}{\pi \hbar} \right) \ln \left( \frac{B_\phi}{H} \right) - \Psi \left( \frac{1}{2} + \frac{B_\phi}{H} \right)
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length shows decrease with increasing temperature. It can be understood qualitatively as increasing phononic contribution at higher temperatures. We fitted the temperature dependence of phase coherence length ($I_\phi$) using the formula [20]

$$I_\phi (T) = A T^{-3/4} + B T^{-3/2}.$$  

Here the first term corresponds to decoherence resulting from electron–electron interaction and the second term is due to electron–photon interaction. The fitted curve matches with experimental data very well (inset (iii) of figure 5(b)). The values of $A$ and $B$ comes out to be $-65.2$ nm K$^{3/4}$ and $2396.1$ nm K$^{3/2}$.

Values of $\alpha$ were obtained from the HLN fitting itself. Value of $\alpha$ at 5 K comes out 3.2 $\times$ 10$^7$ $\mu$m$^{-1}$. To calculate total number of conduction channels we multiply it by sample thickness $\sim$20 $\mu$m which gives the value 6.30 $\times$ 10$^8$. Such a large value indicates towards the bulk conduction channels responsible for the WAL. The value of $\alpha$ also substantially decreases with increasing temperature and becomes 3.1 $\times$ 10$^6$ $\mu$m$^{-1}$ at 30 K. There are reports on several other semimetallic samples CaCuSb [49], Bi$_2$Te$_3$ [50], LuPdBi [51], where such large number of conduction channels are observed.

The large WAL effect in SnTaS$_2$ can be understood qualitatively as follows. In weakly disordered nodal-line semimetals, the screening effect becomes unconventional. This means the scattering potentials becomes long ranged [44]. In such limit, motion of quasiparticles can be confined to 2D planes perpendicular to the nodal line, and backscattering is dominated by those loops which encircle the nodal line [43]. In essence, the additional $\pi$ Berry phase of nodal line makes the interference destructive leading to WAL. This effective 2D diffusion is therefore the reason which makes the WAL correction so large since there are a large number of such 2D subsystems [43]. Conductivity of each subsystem adds up to give a large conductivity. Further if the Fermi energy above the nodal line is nearby, WAL effect become favorable [43]. Previous first principle calculations [23, 24, 26] as well as ARPES studies [24] have reported that the nodal lines in SnTaS$_2$ is in the vicinity of Fermi level. So SnTaS$_2$ satisfies all the requirements for large WAL effect confirming dominance of topological features in the normal state.

4. Conclusion

In summary, we report the synthesis of high-quality single crystal of nodal line semimetal SnTaS$_2$ which is an intercalated transition metal dichalcogenide. The crystals were grown using chemical vapor transport method. The residual resistivity ratio is found to be $\sim$530 with no sign of structural phase transition. The resistive transition temperature is found to be 2.9 $\pm$ 0.1 K. The upper critical field $H_{c2,\infty}$ fits well with GL fit while $H_{c2,ab}$ shows upward curvature near $T_c$. Anisotropy factor is estimated to be 3.1. Magneto-transport measurements show a non-saturating MR of 320% at 5 K up to $\pm$5 T. We report first experimental confirmation of WAL effect in normal state of SnTaS$_2$ which is in support of the ARPES studies. In essence, SnTaS$_2$ provides a platform for understanding the superconducting transition with nodal-line fermions. A lot about the pairing symmetry as well as the topological features need to be unraveled in this rekindled superconducting system.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Acknowledgments

M Singh acknowledges CSIR for support through CSIR-JRF fellowship. P Saha, V Nagpal thank UGC for providing JRF. We thank FIST program of Department of Science and Technology, Government of India for low temperature high magnetic field facility at JNU. We thank Advanced Instrumentation Research Facility (AIRF), JNU for technical support.

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