Multi-band Superconductivity in a misfit layered compound (SnSe)_{1.16}(NbSe_2)_{2}

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Abstract

We report the discovery of superconductivity with $T_c$ of about 5.3 K in a new misfit layered compound (SnSe)_{1.16}(NbSe_2)_{2}. High resolution transmission electron microscopy and selected area electron diffraction of the single-crystalline samples clearly reveal the misfit layered structure. Based on the McMillan equation, the electron-phonon coupling constant $\lambda_{e-ph}$ is estimated to be about 0.96, which is close to strong coupling range. The estimated out-of-plane and in-plane upper critical magnetic fields are 8.9 T and 15.5 T respectively. $H_{c2}(0)$ is 1.74 times the value of Pauli paramagnetic limit. Moreover, there is a positive curvature in the $H_{c2}(T)$ curves near $T_c$, indicating a feature of multi-band superconductivity. The temperature dependence of specific-heat in superconducting state can be fit by a two-band BCS model, which confirms further the multi-band superconductivity. The reduced charge transfer between the two subsystems may account for the enhanced $T_c$ comparing with (SnSe)_{1.16}(NbSe_2).

1. Introduction

The misfit layered compounds (MLCs), which consist of alternately stacked rocksalt layers $MX$ and layered transition metal chalcogenides (TMDCs) $TX_2$, can be regarded as natural van der Waals heterostructures (VDWHs). The general formula of MLCs is represented as $(MX)_{n+1}(TX_2)_n$ ($y = 0.08–0.28, n = 1, 2, 3$), where $M$ = Sn, Pb, Sb, Bi or a lanthanide; $X$ = S, Se or Te; $T$ = Ti, V, Cr, Nb or Ta. The different periodicities of the two subsystems lead to ‘misfit’ feature in structure. In the MLCs family, many superconductors have been discovered [1, 2, 3–6, 7–12, 13–15]. Their $T_c$ values are in the range of about 0.4–5.3 K. The upper critical field ($H_{c2}$) of most MLC superconductors are usually small in both in-plane and out-of-plane directions, [8, 2, 13, 15–18], and only a few MLC superconductors exhibit unusually large $H_{c2}$ [14, 19].

The physical properties of MLCs are dependent on the number of $TX_2$ layers ($n$) in one period of structure along the c-axis. For example, in (PbSe)$_{1.14}$(NbSe$_2$)$_{0}$, when $n = 1$, the compound is non-superconducting, and the two subsystems are all orthorhombic. But when $n = 2$ and 3, the compounds are both superconductors with $T_c$ of 3.4 K and 4.8 K respectively, and the subsystems are all monoclinic [5]. Similarly, (PbSe)$_{1.16}$(TiSe$_2$)$_{2}$ is not a superconductor but (PbSe)$_{1.16}$(TiSe$_2$)$_{2}$ exhibits superconductivity with $T_c$ of 2.4–3.2 K [11, 12, 20]. For more examples, $T_c$ of (LaSe)$_{1.14}$(NbSe$_2$)$_{2}$ and (LaSe)$_{1.14}$(NbSe$_2$)$_{2}$ are 1.4 K and 5.3 K respectively [6, 9]. Generally speaking, superconductivity is enhanced with the increasing number ($n$) of the $TX_2$ layers in one periodic unit. It has been proposed that the charge transfer between the layers of two subsystems could play a crucial role in determining the properties of MLCs [1, 11, 21, 22]. Charge transfer could bring the similar doping effects as chemical doping does. Angle resolved photoemission spectroscopy (ARPES) measurement of (PbSe)$_{1.16}$(TiSe$_2$)$_{n}$ proved that the transferred charge is $0.26 \epsilon^-$ and $0.074 \epsilon^-$ per TiSe$_2$ for $n = 1$ and $n = 2$ respectively [22]. In
comparison with the phase diagram of TiSe$_2$, $n = 1$ is in the overdoped regime while $n = 2$ is very close to the optimally doping point.

Although the NbSe$_2$ based-MLC (SnSe)$_{1.16}$(NbSe$_2$)$_2$ has already been reported [23] before and its superconductivity has been investigated recently [14], there is no report on (SnSe)$_{1.16}$(NbSe$_2$)$_2$ with $n = 2$ up to now. Here, we report the successful synthesis of (SnSe)$_{1.16}$(NbSe$_2$)$_2$ single crystals and the discovery of superconductivity with an onset transition temperature of 5.3 K. The misfit feature of structure is clearly revealed by high resolution transmission electron microscopy (HRTEM) and selected area electron diffraction (SAED). Similar to (SnSe)$_{1.16}$(NbSe$_2$)$_2$, (SnSe)$_{1.16}$(PbSe$_2$)$_2$ also exhibit multi-band features in both the specific-heat and $H_c2$ data. The in-plane upper critical magnetic field ($H^\text{uc}_c(0)$) exceeds the Pauli limit $H_P$ significantly, which may mainly result from the multi-band effects.

2. Experiment details

Single crystals of (SnSe)$_{1.16}$(NbSe$_2$)$_2$ were prepared via chemical vapor transport (CVT) method with bromine as the transport agent. Sn (99.999%), Nb (99.99%), Se (99.999%) and SnBr$_2$ (99.999%) powders with a total mass of 1.8 g were mixed with the molar ratio of 1.16:2.5:16:0.44 and ground adequately, then sealed into an evacuated quartz ampoule with a length of 16 cm. The ampoule was heated in a two-zone furnace for 1 week, and the temperature of source zone and growth zone was controlled to be 900 °C and 850 °C respectively. Plate-like single crystals of (SnSe)$_{1.16}$(NbSe$_2$)$_2$ with a size of 2–4 millimeters in the plane and a thickness of 0.04–0.1 millimeters were obtained.

The room temperature x-ray diffraction (XRD) data of single-crystalline samples were measured by a PANalytical x-ray diffractometer (Empyrean) with a graphite monochromator and a Cu K$_\alpha$ radiation. The chemical element molar ratio was confirmed by an energy-dispersive x-ray spectroscopy (EDX) affiliated with a GENESIS4000 EDAX spectrometer. The HRTEM and SAED images were taken at room temperature by employing an aberration corrected FEI-Titan G2 80-200 ChemiSTEM. The magnetic properties were measured on a magnetic property measurement system (MPMS-XL5, Quantum Design), and the specific-heat capacity was measured on a physical properties measurement system (PPMS-9, Quantum Design). The electrical transport properties were measured by using an Oxford Instruments cryostat with a He-3 probe and a 15T superconducting magnet.

3. Results and discussion

Figure 1 (a) shows the EDX pattern of a (SnSe)$_{1.16}$(NbSe$_2$)$_2$ single crystal. The molar ratio of SnSe and NbSe$_2$ is determined to be about 1.10(3):1, very close to the nominal composition. Moreover, it is found that about 4% Se are substituted by Br, which is unavoidably caused by the bromine transport agent. Such a halogen contamination also occurs in other MLCs such as (SnSe)$_{1.16}$(NbSe$_2$)$_2$ and (PbSe)$_{1.12}$(TaSe$_2$)$_2$ which were grown by the same CVT method [14, 15]. Figure 1 (b) shows the XRD pattern of a (SnSe)$_{1.16}$(NbSe$_2$)$_2$ single crystal at room temperature, and the inset is a photograph of the typical as-grown single crystal. All the peaks in figure 1 (b) are only (00l) peaks, indicating a single crystal feature.
Figure 2 shows the sketched structure model, HRTEM images and SAED images of a (SnSe)$_{1.16}$(NbSe$_2$)$_2$ single crystal. (a), (b) and (c) taken along the [010] direction, while (d), (e) and (f) taken along the [100] direction. The HRTEM image clearly displays the usual stacking of the SnSe layer and the double NbSe$_2$ layers, and the different features between the two directions in the $ab$ plane: incommensurate $a$-axis lattice constants between the two subsystems and the same periodicity along the $b$-axis of the two subsystems. The corresponding SAED patterns also reveal this difference: along the [010] direction, the SAED pattern contains two sets of reflections from the SnSe and NbSe$_2$ subsystems respectively, which are indicated by the red and green arrows; while along the [100] direction, the SAED pattern only contains one set of reflections due to the same periodicity of two subsystems. We calculated the lattice parameters of (SnSe)$_{1.16}$(NbSe$_2$)$_2$ from the SAED patterns and summarized in Table 1. The values of $a$ and $b$ are close to those of (SnSe)$_{1.16}$(NbSe$_2$)$_2$ for both subsystems [14], but the space group of two subsystems are both changed. We can obtained the incommensurate factor as $1+y = 2a_2/a_1 \approx 1.16$, where $a_1$ and $a_2$ represent the lattice constant along the $a$-axis for the SnSe and NbSe$_2$ subsystems respectively. In the SnSe subsystem, one unit cell contains one SnSe layer, and these layers form a monoclinic structure with a $C2/m$ space group. Meanwhile in the NbSe$_2$ subsystem, one unit cell contains two NbSe$_2$ layers, and the stack mode of the two layers is similar to $2Ha$-NbSe$_2$, but there is an offset of $b/6$ between the two layers. The double-layer NbSe$_2$ subsystem also form a monoclinic structure with a $C2/m$ space group. This structure is the same as in (PbSe)$_{1.16}$(NbSe$_2$)$_2$ [5].

Figure 3(a) shows the temperature dependence of out-of-plane ($\rho_z$) and in-plane ($\rho_{ab}$) electrical resistivity for a (SnSe)$_{1.16}$(NbSe$_2$)$_2$ single crystal. For both directions, the onset superconducting transition temperature $T_c$ is about 5.3 K (determined by 90% of normal state resistivity). Figure 3(b) shows the dc magnetic susceptibility as a function of temperature around $T_c$ under a magnetic field of 10 Oe applied along the $ab$-plane. The superconducting shielding volume fraction exceed 100% a little, which is due to the tiny influence of demagnetization factor. It should be noted that the behavior of $\rho_z$ and $\rho_{ab}$ is very different. In normal state, $\rho_{ab}(T)$ decreases monotonically with reduced temperature, i.e., a typical metallic behavior, but $\rho_z(T)$ shows a broad hump around 150 K. Similar metal-to-nonmetal crossover in $\rho_z$ has also been observed in other layered metals, such as CsCa$_2$Fe$_4$As$_4$F$_2$ [24], Li$_3$(NH$_3)_4$Fe$_2$Se$_2$ [25] and Tl$_2$Ba$_2$CaCu$_2$O$_{8+}$ [26]. The mainstream explanation for
such a metal-to-nonmetal crossover is in terms of incoherent hopping between layers because of $l_{c} \leq d_{\text{inter}}$, where $l_{c}$ is the mean free path along the $c$-axis and $d_{\text{inter}}$ is the distance between two layers.

Figure 3(c) shows the specific-heat around $T_{c}$ plotted as $C/T$ versus $T^{2}$. From the linear fit by the equation $C/T = \gamma + \beta T^{2}$, we can obtained that Sommerfeld coefficient $\gamma = 56.1 \text{ mJ mol}^{-1} \text{ K}^{-2}$ and lattice specific-heat coefficient $\beta = 6.03 \text{ mJ mol}^{-1} \text{ K}^{-4}$. The reduced Sommerfeld coefficient $\gamma/m = 6.74 \text{ mJ mol}^{-1} \text{ K}^{-2}$, where $m$ is the number of atoms per formula unit. In $2$H$\alpha$-NbSe$_{2}$, $\gamma/m = 6.33 \text{ mJ mol}^{-1} \text{ K}^{-2}$, which is close to our sample [27]. The Debye temperature is obtained, $\Theta_{D} = 138.94K$, based on the formula $\Theta_{D} = (12\pi^{4}mR/5\beta)^{1/3}$, where $R$ is the gas constant. Moreover, the electron-phonon coupling constant $\lambda_{e-\text{ph}}$ is estimated to be 0.96 from the McMillan equation [28]:

$$\lambda_{e-\text{ph}} = \frac{\mu^{*}\ln\left(\frac{\Theta_{D}}{1.457T_{c}}\right) + 1.04}{\ln\left(\frac{\Theta_{D}}{1.457T_{c}}\right)(1 - 0.62\mu^{*}) - 1.04}$$  \hspace{1cm} (1)

where $\mu^{*}$ is the Coulomb pseudopotential, and an empirical value of 0.15 is used. Comparing with (SnSe)$_{1.16}$(NbSe$_{2}$)$_{n}$ ($\lambda_{e-\text{ph}} = 0.80$) [14], the $\lambda_{e-\text{ph}}$ of (SnSe)$_{1.16}$(NbSe$_{2}$)$_{2}$ is enhanced. This value indicates that (SnSe)$_{1.16}$(NbSe$_{2}$)$_{2}$ is close to strong coupling regime. Similar phenomena also occurs in (PbSe)$_{1.16}$(TiSe$_{2}$)$_{n}$ ($n = 1, 2$). The energy distribution curves of ARPES measurements also suggest that the electron-phonon coupling of (PbSe)$_{1.16}$(TiSe$_{2}$)$_{2}$ ($n = 2$) is much larger than that of (PbSe)$_{1.16}$(TiSe$_{2}$)$_{n}$ ($n = 1$) [22].

A superconducting transition induced specific-heat jump is obvious around $T_{c}$. Figure 3(d) shows the normalized electronic specific-heat $C_{e}/\gamma T$ versus $t$, where $t = T/T_{c}$. Such a temperature dependence of specific heat cannot be fit by a model of single BCS gap. We adopted a two-gap BCS model to fit the data, as the same as in (SnSe)$_{1.16}$(NbSe$_{2}$)$_{2}$ and other multi-band superconductors [14, 27, 29–32]. From this fit, we obtained that $2\Delta_{1} = 5.66k_{B}T_{c}$ while $2\Delta_{2} = 1.59k_{B}T_{c}$, and weight $\gamma_{1}/\gamma_{2} = 52\%:48\%$. Comparing with (SnSe)$_{1.16}$(NbSe$_{2}$)$_{2}$ [14], the two gaps are both enhanced, and the weights are changed a little. These changes may be related to the change in the Fermi surface caused by the reduced charge transfer between the two subsystems. Furthermore, the normalized specific heat jump $\Delta C_{e}/\gamma T_{c}$ is estimated to be 0.85, close to some other MLCs superconductors [2, 13, 14].

In order to study the effect of charge transfer on $T_{c}$, we measured the Hall effect of (SnSe)$_{1.16}$(NbSe$_{2}$)$_{n}$ ($n = 1, 2$). The (SnSe)$_{1.16}$(NbSe$_{2}$)$_{2}$ single crystal is the same sample used in our previous work [14]. Figure 4(a)
shows the temperature dependence of Hall coefficient of (SnSe)$_{1.16}$(NbSe$_2$) (n1), (SnSe)$_{2.16}$(NbSe$_2$) (n2). In 2Ha-NbSe$_2$, the positive Hall coefficient is reduced and finally changes to be negative below 50K due to the CDW transition [33]. This phenomenon is disappeared in n1 and n2 because of the absence of CDW transition. In n1 and n2, the Hall coefficient is positive in all temperature range, indicating the predominant charge carrier is hole-type. Figure 4(b) displays corresponding carrier concentration of the two samples. Comparing with n1, the charge transfer in n2 is reduced, hence the carrier concentration is enhanced. We summarize the relationship of carrier concentration in the two MLCs. Comparing to n1, the charge transfer in n2 is reduced, hence the positive curvature close to T$_c$ are determined by the points at 50% of normal state resistivity (T$_{c0}$). In both directions, the curves exhibit a positive curvature close to T$_c$, which is usually regarded as a feature of multi-band superconductivity [14, 29, 30, 40–42]. Thus we adopt a two-band model to fit $H^c_2(T)$ [43].

$$a_0[\ln t + U(h/t)][\ln t + U(\eta h/t)]$$
$$+ a_1[\ln t + U(h/t)] + a_2[\ln t + U(\eta h/t)] = 0$$

(2)

where t = T/T$_c$ is the reduced temperature, $a_0 = 2(\lambda_{11}\lambda_{22}-\lambda_{12}\lambda_{21})/\lambda_{12}$, $a_1 = 1 + (\lambda_{11}-\lambda_{22})/\lambda_{12}$, $a_2 = 1 - (\lambda_{11}-\lambda_{22})/\lambda_{12}$, $\lambda_0 = [(\lambda_{11}-\lambda_{22})^2 + 4\lambda_{12}\lambda_{21}]^{1/2}$, $h = H^c_2 D_2/2\phi_0 T$, $\eta = D_1/D_2$, $U(x) = \Psi(x+1/2)-\Psi(x)$, the $\Psi(x)$ is
digamma function. $\lambda_{11}$ and $\lambda_{22}$ are the intraband BCS coupling constants, where $\lambda_{12}$ and $\lambda_{21}$ are the interband BCS coupling constants, and $D_1$ and $D_2$ are the in-plane diffusivity of each band. The fitting parameters are list in table 2.

The fitting parameters for the n2 sample are similar to those of n1. For one band, the intraband coupling constant is much greater than other band, but its interband coupling constant is smaller than the latter. This fitting results are further supported by the two-gap fit of the specific-heat data, indicating the multi-band superconductivity in (SnSe)$_{1.16}$ (NbSe$_2$)$_2$. Moreover, this fit yields $\mu_0H_{c2}^e(0) = 8.9$ T and $\mu_0H_{c2}^{ab}(0) = 15.5$ T. The anisotropy factor $g = H_{c2}^{ab}(0)/H_{c2}^e(0) \approx 1.74$. The Ginzburg-Landau coherence length of both directions are estimated by the two formulas: $\xi_{c}(0) \approx 3.49$ nm and $\xi_{ab}(0) \approx 6.08$ nm and $\xi_{c}(0) \approx 3.49$ nm are obtained. The Pauli paramagnetic limit ($H_P$) for the upper critical field is $\mu_0H_p = 1.84 \ T_c = 8.9$ T. Hence $H_{c2}^{ab}(0)/H_p = 1.74$, which is a large value among MLCs superconductors. As mentioned above, most MLC superconductors have small upper critical field $H_{c2}$ (compared to the Pauli limit) in both directions. This value is also larger than that in the n1 sample (about 1.25) [14]. The multi-band effects may enhance $H_{c2}^{ab}$ in both n1 and n2.

### 4. Conclusion

In summary, we synthesized a new misfit layered compound: (SnSe)$_{1.16}$(NbSe$_2$)$_2$, and discovered its superconductivity with $T_c$ of 5.3 K. The details of structure were revealed by powder HRTEM and SAED. Superconducting shielding volume fraction is close to 100%, confirming the bulk superconductivity. The reduced electron-type charge transfer could actually increase the hole-type charge carrier density in the conducting NbSe$_2$ layer and thus accounts for the enhanced $T_c$ comparing with (SnSe)$_{1.16}$(NbSe$_2$)$_2$. The Sommerfeld coefficient $\gamma$ is 56.1 mJ mol$^{-1}$ K$^{-2}$. The electron-phonon coupling constant $\lambda_{e-ph} = 0.96$. $H_{c2}(0)$ is estimated to be 8.9 T and 15.5 T for the inter-plane and in-plane directions respectively. Especially for the in-plane direction, the value of $H_{c2}(0)/H_p$ is about 1.74. Both the specific-heat and $H_{c2}$ data suggest that (SnSe)$_{1.16}$(NbSe$_2$)$_2$ is a multi-band superconductor. The multi-band effect may be the main reason for the significantly enhanced $H_{c2}^{ab}(0)$.
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