Superconductivity and disorder effect in TlNi$_2$Se$_2$–$x$S$_x$ compounds

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Abstract

After our first discovery of multi-band superconductivity (SC) in the TlNi$_2$Se$_2$ crystal, we successfully grew a series of TlNi$_2$Se$_2$–$x$S$_x$ (0.0 $\leq x \leq$ 2.0) single crystals. Measurements of resistivity, specific heat, and susceptibility were carried out on these crystals. Superconductivity with $T_C$ = 2.3 K was first observed in the TlNi$_2$Se$_2$ crystal, which also appears to involve heavy electrons with an effective mass $m^* = 13$–25 $m_b$, as inferred from the normal state electronic specific heat and the upper critical field, $H_{c2}(T)$. It was found that bulk SC and heavy-electron behavior is preserved in all the studied TlNi$_2$Se$_2$–$x$S$_x$ samples. In the mixed state, a novel change of the field dependence of the residual specific heat coefficient, $\gamma_N(H)$, occurs in TlNi$_2$Se$_2$–$x$S$_x$ with increasing S content. We also found that the $T_C$ value changes with the disorder degree induced by the partial substitution of S for Se, characterized by the residual resistivity ratio (RRR). Thus, the TlNi$_2$Se$_2$–$x$S$_x$ system provides a platform to study the effect of disorder on the multi-band SC.

Keywords: Ni-based superconductor, disorder in superconductors, TlNi2S2

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

The layered compounds with ThCr$_2$Si$_2$-type structure (space group $I4/mmm$) exhibit versatile physical properties, including an antiferromagnetic (AFM) ground state in BaFe$_2$As$_2$ [1], ferromagnetic (FM) ordering in (K,Rb)Co$_2$Se$_2$ [2], and Fe-based superconductivity (SC) with $T_C = 30$–50 K in both (Ba,K)Fe$_2$As$_2$ [3] and (TL,K,Rb)Fe$_2$Se$_2$ systems [4, 5], as well as the heavy-fermion SC in CeCu$_2$Si$_2$ [6]. In particular, SC emerges in both the Ni pnictide compounds, such as BaNi$_2$As$_2$ ($T_C = 0.7$ K) [7] and SrNi$_2$P$_2$ ($T_C = 1.4$ K) [8], in which the electron effective mass is not much enhanced, and the Ni chalcogenide compounds, such as KNi$_2$Se$_2$ ($T_C = 0.8$ K) [9], KNi$_2$S$_2$ ($T_C = 0.46$ K) [10] and TlNi$_2$S$_2$ ($T_C = 3.7$ K) [11], in which SC appears to involve heavy electrons with an effective mass $m^* = 14$–20 $m_b$. Due to Ni ions in the Ni chalcogenide compounds having a mixed valence Ni$^{1.5+}$, it was suggested that the formation of a heavy-fermion state at low temperatures is driven by the hybridization of localized charges with conduction electrons, and the coherent state competes with a charge-fluctuating state [12]. Thus the question arises of whether the SC close to a charge-fluctuation state in this system is unconventional or conventional.

TlNi$_2$S$_2$ crystallizes in a tetragonal ThCr$_2$Si$_2$-type structure (space group $I4/mmm$), the same as that of TlNi$_2$Se$_2$. It can also be considered as a nonmagnetic analogue of Fe chalcogenide superconductors, i.e. TlFe$_2$Se$_2$ compounds [4, 5], recently discovered by us, but no SC emerges in TlFe$_2$Se$_2$ compounds with Fe vacancies. The TlNi$_2$S$_2$ compound is also a Pauli paramagnetic metal, similar to the TlNi$_2$Se$_2$ compound, reported first by Newmark et al [13], in 1989. They did not observe any superconducting transition above 2 K. After our discovery of multi-band SC in the TlNi$_2$Se$_2$ crystal, we successfully grew a series of TlNi$_2$Se$_2$–$x$S$_x$ (0.0 $\leq x \leq$ 2.0)
single crystals and systematically studied their structure and physical properties. SC with $T_C = 2.3$ K has been observed in TlNi$_2$S$_2$, and appears also to involve heavy electrons with an effective mass $m^* = 13-25 \, m_e$, although the $T_C$ is a little lower than that in TlNi$_2$Se$_2$ ($T_C = 3.7$ K). With the isovalent substitution of Se by S in TlNi$_2$Se$_{2-x}$S$_x$ crystals, the evolution of the superconducting properties was studied systematically. It was found that bulk SC and heavy-electron behavior are preserved in all the TlNi$_2$Se$_{2-x}$S$_x$ compounds, which was confirmed by the results of resistivity, susceptibility and specific heat. It was found that, with increasing S content $x$, the magnetic field dependence of the residual specific heat coefficient at $T = 0$ K in the mixed state, $\gamma(T)$, changes from a $\propto H^{1.5}$ relation, observed in TlNi$_2$Se$_2$, to a linear relation, consistent with the prediction for a conventional s-wave superconductor. In addition, it was found that the $T_C$ value in the TlNi$_2$Se$_{2-x}$S$_x$ crystals is related to the disorder, characterized by the RRR, which is induced by the partial substitution of S for Se.

Single crystals of TlNi$_2$Se$_{2-x}$S$_x$ ($0.0 \leq x \leq 2.0$) were grown using a self-flux method. A mixture with Tl:Ni:Se:S = 1 : (2 – $x$) : $x$ was placed in an alumina crucible sealed in an evacuated quartz tube. The mixture was heated at 950 °C for 12 h, then slowly cooled to 650 °C at a rate of 6 °C h$^{-1}$, followed by furnace cooling. Single crystals with a typical dimension of $3 \times 3 \times 0.2$ mm$^3$ were mechanically isolated from the flux, as shown on the left of figure 1 for a TlNi$_2$S$_2$ single crystal. The structure of the single crystals was characterized by x-ray diffraction (XRD). Figure 1 shows the XRD pattern at room temperature for TlNi$_2$Se$_{2-x}$S$_x$ ($x = 0.0, 1.0$ and $2.0$) powder obtained by grinding pieces of crystals; other crystals have a similar XRD pattern (not shown here), which confirms that all the crystals of TlNi$_2$Se$_{2-x}$S$_x$ ($0.0 \leq x \leq 2.0$) have the ThCr$_2$Si$_2$-type structure. With increasing S content, $x$, the lattice parameters, $a$ and $c$ values, decrease monotonically, as shown in the right-hand inset of figure 1, which is consistent with the fact that the ionic radius of S$^2_-$ ($184$ pm) is smaller than that of Se$^2_-$ ($191$ pm), indicating that S$^2_-$ can uniformly substitute for Se$^2_-$ in the TlNi$_2$Se$_{2-x}$S$_x$ system. The lattice parameters are $a = 3.87$ Å, $c = 13.43$ Å for TlNi$_2$Se$_2$, and $a = 3.79$ Å, $c = 12.77$ Å, for the other end member, TlNi$_2$S$_2$. The lattice parameter variation of the TlNi$_2$Se$_{2-x}$S$_x$ compounds as obtained from the XRD data analysis is shown in the right-hand inset of figure 1. The measurements of electrical resistivity and specific heat were carried out using a Quantum Design PPMS-9. The dc magnetic susceptibility measurements were made using a Quantum Design MPMS-SQUID.

The physical properties of TlNi$_2$Se$_2$ are summarized in figure 2. Both $\rho_{ab}$ and $\rho_{c}$ versus $T$ curves, shown in figure 2(a) and inset (I), display a metallic behavior in the normal state before dropping abruptly to zero when SC occurs at $T_C = 2.3$ K, which is also confirmed by a large diamagnetic signal (see inset (II) of figure 2) and a specific heat jump at $T_C$, as shown in inset (III). First, we discuss the resistivity in the normal state. $\rho_{ab}$ and $\rho_{c}$ at 300 K are 68 and 92 $\mu$Ω cm, respectively, and $\rho_{c}/\rho_{ab} = 1.35$, indicating that the anisotropy in TlNi$_2$S$_2$ is rather small, although the compound has a layer structure. In the normal state, no abnormal behavior related to structural or magnetic transition is observed in the resistivity curves,
as usually happens in the iron-based superconductors and the other isostructural compounds, such as BaNi$_2$As$_2$ [7] and SrNi$_2$P$_2$ [8]. The temperature dependence of magnetic susceptibility, $\chi(T)$, in the normal state was measured at 1 T field, as shown in figure 2(b). At higher temperatures, $\chi(T)$ shows almost temperature-independent Pauli paramagnetic behavior. At lower temperatures, an obvious Curie tail occurs, which likely comes from the magnetic impurity. A fit with the Curie–Weiss law below 100 K yields a temperature-independent contribution $\chi_0 = 5.3 \times 10^{-4}$ emu mol$^{-1}$ and Curie constant $C = 0.0063$, corresponding to 0.63 mol% of an $S = 1$ impurity (e.g. Ni$^{2+}$), indicating the absence of localized magnetism in the TlNi$_2$S$_2$ crystal, similar to that in TlNi$_2$Se$_2$.

Inset (iii) shows the temperature dependence of the specific heat divided by temperature, $C(T)/T$, measured at both zero field and 1 T field. At zero field, the $C(T)/T$ curve reveals a clear $\lambda$ anomaly, indicating the bulk nature of SC. The normal state specific heat $C_n(T)$ is obtained by applying a magnetic field $\mu_0 H = 1.0$ T. The $C(T)$ versus $T^2$ curve (not shown here) shows nonlinear behavior at low temperatures, so we fitted the data using a polynomial expansion, $C(T) = \gamma_T + \beta T^2 + \delta T^4$, below 6 K. $\gamma_T = 30.57$ mJ mol$^{-1}$ K$^{-2}$ and $\beta = 1.14$ mJ mol$^{-1}$ K$^{-4}$ were obtained, corresponding to a Debye temperature $\Theta = 197$ K. The value of normal state electronic specific heat coefficient $\gamma_T$ is smaller than that for TlNi$_2$Se$_2$ ($\sim 40$ mJ mol$^{-1}$ K$^{-2}$) and KNi$_2$Se$_2$ ($\sim 44$ mJ mol$^{-1}$ K$^{-2}$) superconductors. However, it is still much larger than that for LaO$_{1-x}$F$_x$NiAs ($\sim 7.3$ mJ mol$^{-1}$ K$^{-2}$) [14] and BaNi$_2$As$_2$ ($\sim 12.3$ mJ mol$^{-1}$ K$^{-2}$) [7]. Assuming 1.5 carriers/Ni and a spherical Fermi surface, it yields $m^* \sim 13 m_e$. The zero-field electronic specific heat in the superconducting state, $C_{es}$, was obtained by subtracting the phonon contribution estimated by the $C(T)$ measured at 1 T. Figure 2(c) shows the temperature dependence of $C_{es}/\gamma_T T$. The specific heat jump at $T_C$, $\Delta C / \gamma_T T_C = 1.55$, is slightly larger than the weak-coupling BCS expectation of 1.43. Then, we use two different formulae, based on the conventional BCS theory and two-gap model, respectively, to fit the data of $C_{es}(T)$ below $T_C$. Though the measured temperature is not low enough to get the best parameters, it is clear to see that the simple BCS model cannot well describe the $C_{es}(T)$ data, and the two-gap model presents the best fit, especially at low temperatures, as shown in figure 2(c). The two superconducting gaps resulting are $\Delta_1/k_B T_C = 0.95$ and $\Delta_2/k_B T_C = 1.96$, respectively, and the relative weight contributed from the first gap is about 0.18. These results are very similar to those for TlNi$_2$Se$_2$, indicating that they may contain a similar electronic structure [15].

The temperature dependence of upper critical field, $H_{c2}(T)$, obtained from $\rho(T)$ measurements at various magnetic fields, is shown in inset (iv) of figure 2. It is found that $H_{c2}$ can be well described by the Ginzburg–Landau model, $H_{c2}(T) = H_{c2}(0) \times (1 - r^2)/(1 + r^2)$, where $r$ is the reduced temperature, $T/T_C$. Then the zero temperature upper critical field $H_{c2}(0)$ is estimated to be about 0.53 T. The superconducting coherence length $\xi_0$ can be estimated to be 24.9 nm from the relation $\xi_0 = \left[\Phi_0/(2\pi H_{c2})\right]^{1/2}$. Then the Fermi velocity $v_F = 4.09 \times 10^4$ m s$^{-1}$ is obtained from $\xi_0 = 0.18h v_F / k_B T_C$.

Using a spherical Fermi surface approximation, the Fermi wave vector is given by $k_F = (3\pi^2 Z \Omega)^{1/3}$, where $Z$ is the number of electrons per unit cell and $\Omega$ is the unit cell volume. Assuming that Ni contributes 1.5 electrons ($Z = 6$), we obtain $k_F = 9.89 \times 10^3$ m$^{-1}$. Then $m^*$ and $\gamma_N$ can be estimated using the expressions $m^* = \hbar^2 k_F^2 / 2 \pi$ and $\gamma_N = \pi^2 N k_F^2 m^* / 2 \hbar^2$, which yield $m^* \sim 25.5 m_e$ and $\gamma_N \sim 73.3$ mJ mol$^{-1}$ K$^{-2}$, respectively. The values of $m^*$ and $\gamma_N$ are comparable to the values estimated from the normal state specific heat.

The effect of S substitution for Se on SC was systematically studied for the TlNi$_2$Se$_{2-x}$S$_x$ system. Figure 3(a) is the temperature dependence of resistivity for TlNi$_2$Se$_{2-x}$S$_x$ with $x = 0.0, 1.0$ and $2.0$ as typical examples. The measurement
reveals that all the samples exhibit metallic behavior at normal state, and undergo a superconducting transition at low temperatures. The superconducting transition is confirmed by both resistivity and magnetic measurement. Magnified views of the temperature dependences of resistivity and susceptibility at low temperatures of typical compositions in the TlNi$_2$Se$_{2-x}$S$_x$ series are summarized in figures 3(b) and (c), respectively. As shown in the figures, the transition temperatures yielded by resistivity and susceptibility analysis, respectively, are consistent with each other.

To obtain more information about the superconducting properties, we also carried out specific heat measurement in several typical TlNi$_2$Se$_{2-x}$S$_x$ samples with $x = 0.2, 0.8, 1.2,$ and 2.0. The results are presented in figure 4. At zero field, a superconducting transition is clearly observed at low temperatures of typical compositions in the TlNi$_2$Se$_{2-x}$S$_x$ series. The normal state specific heat $C/T$, measured at various magnetic fields, for (a) TlNi$_2$Se$_{1.8}$S$_{0.2}$, (b) TlNi$_2$Se$_{1.2}$S$_{0.8}$, (c) TlNi$_2$Se$_{0.8}$S$_{1.2}$, and (d) TlNi$_2$S$_2$. To make the figure clear, not all the data are presented.

Table 1. The fitting results of the specific heat for TlNi$_2$Se$_{2-x}$S$_x$ crystals.

|        | $\gamma$ (mJ mol$^{-1}$ K$^{-2}$) | $\beta$ (mJ mol$^{-1}$ K$^{-4}$) | $\delta$ (mJ mol$^{-1}$ K$^{-6}$) |
|--------|---------------------------------|---------------------------------|---------------------------------|
| TlNiSe$_2$ | 40                              | 1.65                            | 0.135                           |
| TlNi$_2$Se$_{1.8}$S$_{0.2}$ | 35.68                           | 1.32                            | 0.104                           |
| TlNi$_2$Se$_{1.2}$S$_{0.8}$ | 31.13                           | 0.744                           | 0.093                           |
| TlNi$_2$Se$_{0.8}$S$_{1.2}$ | 29.66                           | 0.511                           | 0.090                           |
| TlNiS$_2$ | 30.57                           | 1.14                            | 0.094                           |

Note: The data for TlNiSe$_2$ come from [11].

much larger than those of the other nickel-based superconductors, indicating that the heavy-electron property is preserved in all the TlNi$_2$Se$_{2-x}$S$_x$ samples. Here, we also carried out fitting using the two-gap model for the $C(T)$ data, which can be yielded by subtracting the phonon contribution estimated by the $C(T)$ measured at 1 T. However, the fitting results are not good, since the measured temperature is not low enough.

In our previous article [11], we reported an $H^{1/2}$ relation for the magnetic field dependence of the residual specific heat coefficient $\gamma_N$ at $T = 0$ K in the mixed state in TlNi$_2$Se$_2$, which is usually considered as a common feature of the d-wave superconductors. To figure out how the sulfur doping affects the behavior, we measured the specific heat of TlNi$_2$Se$_{2-x}$S$_x$ at various magnetic fields. The results are shown in figure 4. With increasing field, the superconducting transition is suppressed. Using the method described in [11], we obtained the residual specific heat coefficient $\gamma_N$ in the mixed state. The field dependences of $\gamma_N$ for TlNi$_2$Se$_{2-x}$S$_x$ with $x = 0.0, 0.2, 0.8, 1.2,$ and 2.0 are plotted in figure 5. From the specific heat data, the $\gamma_N$ for TlNi$_2$Se$_2$ exhibits a convex variation with field (proportional to $H^{1/2}$). With $S$ starting to substitute Se, i.e. for TlNi$_2$Se$_{1.8}$S$_{0.2}$, the $\gamma_N$–$H$ curve becomes not so convex. Then, with further increase of the $S$ content, i.e. for TlNi$_2$Se$_{2-x}$S$_x$ with $x = 0.8, 1.2,$ and 2.0, $\gamma_N$ even exhibits a linear behavior, which is consistent with that of conventional s-wave superconductors. The variation of the $\gamma_N$–$H$ curves is very interesting and exotic, since TlNi$_2$Se$_2$ and TlNi$_2$S$_2$ exhibit similar physical properties in the normal state. In the authors’ view [11], a convex behavior for the field dependence of $\gamma_N$ may be related to the strength of the vortex–vortex interactions in the mixed state [16], and not be responding to the exotic pairing state. Besides, such behavior has also been detected in some iron-based superconductors [17–19]. Bang [20] has proposed that this phenomenon may result from the variation of the superconducting gap size ratio, $\Delta_1/\Delta_2$, and is a generic feature of the multi-band superconductors. However, further investigations, such as STM and ARPES, are needed to determine the superconducting order parameter symmetry. Recently,

Figure 4. Low temperature specific heat divided by temperature, $C/T$, versus $T$, measured at various magnetic fields, for (a) TlNi$_2$Se$_{1.8}$S$_{0.2}$, (b) TlNi$_2$Se$_{1.2}$S$_{0.8}$, (c) TlNi$_2$Se$_{0.8}$S$_{1.2}$, and (d) TlNi$_2$S$_2$. To make the figure clear, not all the data are presented.

Figure 5. Magnetic field $\mu_B H$ dependence of electronic specific heat coefficient $\gamma_N$ in the mixed state for TlNi$_2$Se$_{2-x}$S$_x$ with $x = 0.0, 0.2, 0.8, 1.2,$ and 2.0, respectively.
Figure 6. The critical temperature $T_C$ and the residual resistivity ratio (RRR) as a function of S content $x$ in TINi$_2$Se$_{2-x}$S$_x$.

low-temperature thermal conductivity measurements revealed multiple nodeless superconducting gaps in the TINi$_2$Se$_2$ compound [21], which is consistent with our opinion. From this point of view, the superconducting properties in TINi$_2$Se$_2$ and TINi$_2$S$_2$ may not differ much from each other.

To clarify the superconducting properties, the critical temperature $T_C$ was depicted in figure 6 as a function of S content $x$. The critical temperature is determined by resistivity, susceptibility, and specific heat measurements. A very small difference is detected for these values, which may contribute to the disorder introduced by the S substitution effect on the superconducting properties.

For the undoped sample TlNi$_2$Se$_2$, the residual resistivity ratio (RRR) as a function of S content $x$ in figure 6. As shown in the figure, TINi$_2$Se$_2$ shows the largest RRR value. With increasing S content $x$, the RRR value decreases drastically, and achieves the smallest value (~4.1) for the sample with $x = 1.0$. With S content further increasing, the RRR value increases slightly, and achieves 8.3 for TINi$_2$S$_2$. The variation of the RRR is similar to that of $T_C$, as discussed before, suggesting that the disorder effect plays an important role in the superconducting transition temperature in this system.

In conclusion, we synthesized successfully a series of TINi$_2$Se$_{2-x}$S$_x$ (0.0 $\leq x \leq 2.0$) single crystals. The SC with $T_C \sim 2.3$ K was first detected in the TINi$_2$S$_2$ crystal, which appears to involve heavy electrons with an effective mass $m^* = 13-25 m_e$. With the S substitution, the SC and heavy-electron behavior was preserved in all the TINi$_2$Se$_{2-x}$S$_x$ samples. However, in the mixed state, a novel change of the field dependence of the residual specific heat coefficient, $\gamma_0(H)$, occurs in TINi$_2$Se$_{2-x}$S$_x$ with increasing S content $x$. On the other hand, it was found that the $T_C$ value in the TINi$_2$Se$_{2-x}$S$_x$ crystals is related to the disorder, characterized by the RRR, which is induced by the partial substitution of S for Se. We suggest that the TINi$_2$Se$_{2-x}$S$_x$ system provides an example to study the effect of disorder on multi-band SC.

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