Hydrothermal Synthesis and Transport Properties of FeS$_{1-x}$Te$_x$ (0 ≤ x ≤ 0.15) Single Crystals

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Abstract
In this work, a series of FeS$_{1-x}$Te$_x$ (0 ≤ x ≤ 0.15) single crystals were successfully synthesized by a hydrothermal method for the first time. According to the measurement of in-plane resistivity, Hall effect, and magnetoresistance (MR), we find that the superconducting transition temperature $T_c$ is rapidly suppressed with the increasing Te substitution, and finally the superconductivity disappears when x > 0.05. With the substitution of Te for S, the residual resistivity $\rho_0$ increases while the residual resistivity ratio (RRR) decreases monotonously. Meanwhile, the MR of FeS$_{1-x}$Te$_x$ is also reduced by Te doping. All these results reveal that the Te substitution introduces more impurity scattering. In consequence, the non-linear field-dependent of Hall resistivity $\rho_{xy}$ at low temperature region is suppressed and a linear behavior is restored upon Te doping. The negative Hall coefficients $R_H$ for all the FeS$_{1-x}$Te$_x$ samples suggest that the electron-type carrier dominates the electrical conduction. Moreover, the MR of FeS$_{1-x}$Te$_x$ obviously follows Kohler’s law, indicating the isotropic scattering rates in the Fermi surface.

Keywords FeS$_{1-x}$Te$_x$ · Hydrothermal synthesis · Impurity scattering · Hall effect · Magnetoresistance

1 Introduction
The discovery of superconductivity in LaFeAsO$_{1-x}$F$_x$ with superconducting transition temperature ($T_c$) about 26 K opens up a new route for both searching high-temperature superconductor and probing their mechanism [1]. Tetragonal FeSe is an iron-based superconductor with the simplest layered structure. Its $T_c$ ~ 9 K [2] can be obviously enhanced to 37 K under high pressure [3], and over 40 K by intercalating space layers between the FeSe layers [4, 5]. Meanwhile, bulk FeSe exhibits a structural transition at 87 K without a long-range magnetic order at ambient pressure [6]. The physical properties of FeS and FeTe are significantly different from FeSe, although they display the same anti-PbO structure. FeS was synthesized by hydrothermal method with $T_c$ ~ 4.5 K [7] and remained its tetragonal structure from room temperature to $T_c$ [8]. High pressure or chemical intercalation provides limited contribution to superconductivity, probably due to its peculiar structural characteristics and topology of Fermi surface [9–14]. Pure FeTe is non-superconducting, but exhibits antiferromagnetic ordering around 70 K [15]. However, the doping among them can obviously regulate $T_c$, and shows rich physical properties.

A lot of previous work has been focused on the isovalent S doping in FeSe, which is equivalent to internal positive chemical pressure. The nematic order is gradually suppressed by S doping in FeSe$_{1-x}$S$_x$ and a non-magnetic nematic quantum critical point (QCP) appears at $x \sim 0.17$. However, these samples synthesized by chemical vapor transport (CVT) [16, 17] or solid state reaction [18, 19] were limited to the low sulfur content of $x < 0.29$, which hindered the study of high sulfur doping region. The successful synthesis of FeS single crystal by the hydrothermal method can provide reference for the growth of FeSe$_{1-x}$S$_x$ [7]. Recently, a series of FeSe$_{1-x}$S$_x$ superconducting single crystals were prepared by the same method, especially the high-S doping region (0.3 ≤ x ≤ 1) for the first time [20]. The complete phase diagram exhibits presents a dome-shaped behavior of anomalously resistive characteristic temperature $T^*$ competing with superconductivity in the moderate-S doping region, resulting from the weak localization effect or possible magnetic transition.
According to isovalent substitution of Se, accompanying with the suppression of the antiferromagnetic ordering, non-superconducting FeTe could generate superconductivity under internal positive chemical pressure. The maximum value of $T_c$ can reach up to 14 K when $x = 0.6$ in FeSe$_1$-$_x$Te$_x$ [21]. Recently, the high-quality FeSe$_{1-x}$Te$_x\ (0 \leq x \leq 0.5)$ single crystals were grown by the self-flux method, which addresses the lack of single crystals in the phase separation region, and $T_c$ exhibits a minimum value at $x = 0.2$ [22]. Thus, a complete doping phase diagram of FeSe$_{1-x}$Te$_x\ (0 \leq x \leq 1)$ was established. Similarly, a partial substitution of S by Te can also induce superconductivity with $T_c$ around 10 K. However, the superconducting volume fraction of FeS$_{1-x}$Te$_x\ (x = 0.8, 0.9)$ was less than 20% [23]. Meanwhile, it is difficult to synthesize FeS$_{1-x}$Te$_x$ single crystals with higher sulfur content by solid state reaction due to the low solubility limit of S for the Te [24]. The preparation of high-quality FeS$_{1-x}$Te$_x$ single crystals with S-rich part is still a great challenge until now. Therefore, it is desirable to investigate how the transport properties of FeS evolve with the larger ion radius of Te substitutes the S site.

In this work, a series of FeS$_{1-x}$Te$_x\ (0 \leq x \leq 0.15)$ single crystals were successfully synthesized by a hydrothermal method for the first time. We find that the superconductivity of FeS is continuously suppressed upon Te doping. Furthermore, the field-dependent of Hall resistivity $\rho_{xy}$ evolves from non-linear to linear behavior at low-temperature region with Te substitution, and accompanied by an obvious reduction of magnetoresistance (MR). All these results can be explained by introducing more impurity scattering. The negative values of $R_H$ for FeS$_{1-x}$Te$_x$ reveal that the conduction is dominated by electron-like charge carriers.

### 2 Experimental Section

The single crystals of FeS$_{1-x}$Te$_x$ were synthesized by de-intercalation of K$^+\$ from K$_{0.8}$Fe$_{1.6}$(S$_{1-x}$Te$_x$)$_2$ using a hydrothermal method. The precursors of K$_{0.8}$Fe$_{1.6}$(S$_{1-x}$Te$_x$)$_2$ single crystals with $x = 0, 0.06,$ and 0.13 were grown by a self-flux method. Stoichiometric mixtures of K, Fe, S, and Te were mixed by elements of Na and Te in a molar ratio of 2:1, loaded into an aluminum crucible, sealed in an evacuated quartz tube, and heated at 700 °C for 24 h, a certain amount of NaOH (Aladdin Industrial, 99% purity), and several pieces of K$_{0.8}$Fe$_{1.6}$(S$_{1-x}$Te$_x$)$_2\ (x = 0, 0.06, 0.13)$ crystals were added into 10 mL de-ionized water. The autoclave loaded with the mixture solution was tightly sealed and heated at 130 °C for 3 days to ensure the K release and Fe introduction. Silver colored FeS$_{1-x}$Te$_x$ single crystals were obtained by washing the powder with de-ionized water.

The single crystal X-ray diffraction (XRD) data were performed using Rigaku diffractometer with Cu-Kα radiation. The average stoichiometries of Fe, S, and Te of the single crystal samples were determined from energy-dispersive X-ray spectroscopy (EDX) analysis. The actual Te content $x$ was obtained by EDX to be 0, 0.02, 0.03, 0.04, 0.05, 0.11, and 0.15, respectively. No trace of K element is detected for all single crystals, indicating the interlayer K ions are completely released from the K$_{0.8}$Fe$_{1.6}$(S$_{1-x}$Te$_x$)$_2$ substrates. Electrical resistivity and magnetoresistance were measured on a Quantum Design Physical Property Measurement System (PPMS) and the equipped VSM option. The Hall resistivity and magnetoresistance were measured on the same single crystal using the six-lead method with the applied field parallel to the c-axis and perpendicular to the applied current.

### 3 Results and Discussion

Figure 1 a shows the XRD patterns of FeS$_{1-x}$Te$_x\ (0 \leq x \leq 0.15)$ single crystals. Only (00$l$) diffraction peaks can be detected, indicating good c-axis orientation. All (00$l$) peaks can be well indexed by the tetragonal cell structure with P4/nmm space group [25, 26]. As expected, all of the diffraction peaks shift systematically to lower degrees with the increasing of Te concentration due to the larger ionic radius of Te$^{2+}$ than S$^{2-}$. The corresponding lattice constant $c$ as a function of Te content $x$ is plotted in Fig. 1 b, and it is found that the lattice constant $c$ increases monotonically with the increasing of Te content, indicating that the Te atom is successfully incorporated into the crystal lattice.

Figure 2 a presents the temperature dependence of the normalized in-plane resistivity $\rho(T)/\rho(300$ K) of FeS$_{1-x}$Te$_x$ single crystals for different doping levels. All of the resistivity reduce monotonically with lowering down of temperature, which exhibits a strongly metallic conductivity. For undoped FeS single crystal, an abrupt resistivity drop and zero resistivity can be observed obviously at $T_c = 4.6$ K. Upon Te doping, $T_c$ is gradually suppressed from 4.6 K for $x = 0$, to 4 K for $x = 0.02$, 3.3 K for $x = 0.03$, 2.8 K for $x = 0.04$, and 2.5 K for $x = 0.05$. The enlarged view of the resistivity in the region of superconducting state is shown in Fig. 2 b. The small superconducting transition width, $\Delta T_c < 0.5$ K, shows the excellent quality of our samples. With the further doping, superconductivity is finally killed when $x > 0.05$, and it do
not appear again until $x = 0.15$. The normal-state resistivity in the vicinity of $T_c$ provides important information on the superconducting properties. In order to gain more insight into the doping effect, we analyze all the normal-state resistivity $\rho(T)$ in low-temperature region by fitting with a power law $\rho(T) = \rho_0 + AT^n$, where $\rho_0$ is the residual resistivity, and $A$ and $n$ are the temperature coefficient and the exponent, respectively. In addition, the value of RRR, which is defined as $\rho(300\, \text{K})/\rho_0$ and describes the strength of disorder scattering in samples. The RRR values and residual resistivity $\rho_0$ of superconductivity region are plotted in Fig. 2c. By fitting the resistivity curve, we find that the value of exponent $n$ fluctuates around 2 for all the samples, which is expected for a conventional metal described by Landau Fermi-liquid theory.

Fig. 1  a The typical XRD patterns for FeS$_{1-x}$Te$_x$ ($0 \leq x \leq 0.15$) single crystals. b The lattice parameter $c$ plotted as a function of the doping concentration $x$.

Fig. 2  a Temperature dependence of in-plane resistivity (normalized by corresponding values at $T = 300$ K) for FeS$_{1-x}$Te$_x$ ($x = 0, 0.02, 0.03, 0.04, 0.05$, and $0.11$) single crystal samples. b An enlarged view of the resistivity at low-temperature range for FeS$_{1-x}$Te$_x$ with $x = 0, 0.02, 0.03, 0.04$, and $0.05$. c The residual resistivity ($\rho_0$) and the residual resistance ratio (RRR) as a function of Te content $x$ in superconductivity region. d DC magnetization of FeS$_{1-x}$Te$_x$ ($x = 0, 0.02, 0.03$) single crystals under 5 Oe.
similar with FeS single crystal. Nevertheless, RRR decreases from 48 in undoped FeS single crystal to 11 at \( x = 0.05 \), while residual resistivity \( \rho_0 \) shows the opposite behavior with increasing Te doping. All the characteristics show that the doping of Te leads to more disorder that acts as the impurity scattering center. The superconducting transitions can also be seen in the temperature dependence of the magnetic susceptibility measurements with the zero-field-cooled (ZFC) and field-cooled (FC) modes, as shown in the Fig. 2 d. The onset of the diamagnetism is almost consistent with \( T_c \) determined from resistivity measurement. The superconducting shielding fractions are close to 100%, indicating the bulk superconductivity and the high quality.

Based on the resistivity and magnetization measurement, we establish the doping phase diagram of FeS\(_{1-x}\)Te\(_x\) single crystals, as shown in Fig. 3. For comparison, the corresponding data of FeS\(_{1-x}\)Te\(_x\) (1 \( \leq x \leq 0.88 \)) single crystals taken from Ref [27] are also included. For the rich-Te region, the long-ranged AFM phase \( T_N \) gradually decreases from 70 K with increasing of S doping, and it coexists with the region of the superconducting phase (SC-II) between 0.95 and 0.89 [27], exhibiting a common property of the iron-based superconductors [22, 28]. However, the phase diagram for \( x < 0.88 \) is restricted because of the solubility limitation of S. For the rich-S region on the right, superconducting (SC-I) is gradually suppressed from 4.6 K, exhibiting an approximate linear dependence on the Te content. Considering the whole phase diagram of FeS\(_{1-x}\)Te\(_x\), we are surprised to find that the second superconducting phase (SC-II) appears in the high Te-doped
region after the SC-I is suppressed until disappears. The similar phenomena of the reemerged second superconducting phase often occurred in many iron-based superconductors under high pressure, such as FeS [29], K$_x$Fe$_{2-y}$Se$_2$ [30], and (Li$_{1-x}$Fe$_x$)OHFeSe [31], although Te doping provides the negative chemical pressure.

In order to obtain more information about the properties of carrier transport by Te doping, the Hall resistivity $\rho_{xy}$ up to 9 T were measured at various temperatures. Hall resistivity $\rho_{xy}$ is defined as $\rho_{xy}(H) = \frac{\rho_{xy}(+H) - \rho_{xy}(-H)}{2}$. The field-dependent Hall resistivity for four typical selected samples FeS, FeS$_{0.98}$Te$_{0.02}$, FeS$_{0.95}$Te$_{0.05}$, and FeS$_{0.89}$Te$_{0.11}$ single crystals are shown in Fig. 4 a–d, respectively. For FeS, the $\rho_{xy}$ curves exhibit clear non-linear field-dependent behavior at low-temperature region, which is a signature of the multiband effect. With Te doping, this non-linear behavior is gradually suppressed and linear field-dependent behavior is recovered, which means single band dominates the electric conduction. For a single-band system, the Hall coefficient $R_H$ is only dependent on the charge density, and exhibits weak temperature dependence. However, mobility should be considered in the multiband system. According to the two-band model, the Hall coefficient $R_H$ at low fields can be expressed by $R_H = \frac{n_e u_e^2 - n_h u_h^2}{e(n_e u_e + n_h u_h)^2}$ [32], where $u_h$($u_e$) and $n_h$($n_e$) are the mobility and the charge density of holes (electrons), respectively. Te doping introduces more impurity scattering centers, which reduce the mobility of carriers, and especially the Hall conduction contribution from small band with high mobility. In consequence, multiband effect is suppressed and the single band effect is recovered gradually. The temperature dependence of $R_H$ is shown in Fig. 5.

![Fig. 5 The temperature dependence of Hall coefficient $R_H$ for FeS$_{1-x}$Te$_x$ samples. $R_H$ is determined by $R_H = \rho_{xy}/H$ at low fields](image)

![Fig. 6 Temperature-dependent magnetoresistance at various temperatures for a $x = 0$, b $x = 0.02$, c $x = 0.05$, and d $x = 0.11$ single crystals](image)
where $R_H = \rho_{xy}/H$ is determined by the linear part below 1 T. The negative $R_H$ of FeS$_{1-x}$Te$_x$ below 200 K indicates $\mu_e > \mu_m$, meaning that the conduction is dominated by electron-like charge carriers. The large change of $R_H$ for FeS in the whole temperature range is more obvious than that of doping samples due to the multiband effect.

As we all know, the information of the Fermi surface and the carrier scattering can be obtained by measuring the MR, which is defined as $\text{MR} = (\Delta \rho / \rho(0)) = (\rho(H) - \rho(0))/\rho(0)$. We have measured the field dependence of the MR up to 9 T at various temperatures, and the temperature dependence of MR for FeS$_{1-x}$Te$_x$ samples are plotted in Fig. 6 a–d. It is evident that the large value of MR for FeS is suppressed with gradually increasing Te concentration. This result is quite similar to that in S-oped FeSe single crystal [17], where the value of MR for FeSe is obviously larger than that of FeSe$_{0.86}$S$_{0.14}$. The similar work has also been reported in α-WP$_2$ single crystals, where the highest RRR exhibits the largest MR [33]. Therefore, the decrease of MR could be attributed to the disorder. Unlike the single-band system, the mobility should be considered in the multiband system, and the mobility is known to be inversely proportional to $\rho_0$ [34]. From Fig. 2 c, we can observe that $\rho_0$ increases with Te doping, which indicates that the mobility decreases by doping. Our results demonstrate that Te doping introduces more impurities scattering, which is consistent with the suppression of the MR value and the non-linear field dependence of $\rho_{xy}$ at low temperatures.

For many metallic materials with symmetrical Fermi surface, Kohler’s law is always obeyed. At different temperatures, the MR can be scaled by $\Delta \rho / \rho_0 = f(H/\rho_0) = f(\omega_c \tau)$, known as Kohler’s rule [35]. Figures 7 a–d show the MR as functions of $\mu_0 H / \rho(0)$ at $x = 0, 0.02, 0.05$, and 0.11, respectively. Clearly, all the curves superimpose into one curve, indicating that Kohler’s rule is obeyed and the scattering rate is isotropic in the Fermi surface [17]. However, Kohler’s law was obviously violated below $T_s$ in FeSe$_{1-x}$S$_x$ ($x = 0, 0.14$) [17]. The violation of Kohler’s rule may be related to the multiband effect or the anisotropic scattering on the Fermi surface [36, 37].

### 4 Conclusion

In summary, we have investigated the transport properties of FeS$_{1-x}$Te$_x$ ($0 \leq x \leq 0.15$) single crystals grown by
hydrothermal method. Due to the larger ionic size of Te relative to that of S, the Te substitution leads to the increase of lattice constant $c$. Magnetic and electric transport properties indicate that $T_c$ is obviously suppressed after Te doping, which also leads to the evolution of Hall coefficient $R_H$ from non-linear to linear behavior at low-temperature region, and accompanied by an obvious suppression of the MR. These results can be explained by doping-induced the impurity scattering. The negative $R_H$ in the whole temperature range reveals that the conduction is dominated by electron carriers. More advanced experimental synthesis methods need to be explored to further study the physical properties of samples in the unknown restricted region.

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