Critical current density and vortex pinning mechanism of Li$_x$(NH$_3$)$_y$Fe$_2$Te$_{1.2}$Se$_{0.8}$ single crystals

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Abstract
We grew Li$_x$(NH$_3$)$_y$Fe$_2$Te$_{1.2}$Se$_{0.8}$ single crystals successfully using the low-temperature ammonothermal method, and the onset superconducting transition temperature $T_c$ is increased to 21 K compared to 14 K in the parent compound FeTe$_{0.6}$Se$_{0.4}$. The derived critical current density $J_c$ increases remarkably to $2.6 \times 10^5$ A/cm$^2$ at 2 K. Further analysis indicates that the dominant pinning mechanism in Li$_x$(NH$_3$)$_y$Fe$_2$Te$_{1.2}$Se$_{0.8}$ single crystal is the interaction between vortex and surface-like defects with normal core, by variations in the charge-carrier mean free path $l$ near the defects ($\delta l$ pinning). Moreover, flux creep is important to the vortex dynamics of this material.

Keywords: Li$_x$(NH$_3$)$_y$Fe$_2$Te$_{1.2}$Se$_{0.8}$ single crystals, critical current density, vortex pinning mechanism

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

1. Introduction
Iron-based superconductors (IBSCs) have induced great interest since their discovery almost a decade ago. The family of IBSCs exhibits rather high superconducting transition temperature $T_c$, large upper critical field $H_{c2}$ and critical current density $J_c$. These unique properties are important not only for basic sciences but also for practical applications. Among iron-chalcogenide SCs, FeCh (Ch = S, Se, and Te) has nearly isotropic $\mu_0H_{c2}$ and rather large $J_c$ [1–5], but the relatively low $T_c$ limits its applications to some extent. When monovalent metals A (A = K, Rb, Cs, and Tl) are intercalated into FeCh, the $T_c$ is raised up to about 32 K with rather high $\mu_0H_{c2}$ ($\sim$56 T for $H||c$ at 1.6 K) [6, 7]. However, for A$_x$Fe$_{2-y}$Se$_2$, there are Fe vacancies in the FeCh layer [8]. More severely, the superconducting phase always intergrows with the insulating phase A$_{0.8}$Fe$_{1.6}$Se$_2$, leading to a mesoscopic phase separation [9]. Correspondingly, the superconducting phase takes over only small parts of the total phase. On the one hand, this impedes the investigation of intrinsic superconducting properties of these materials. On the other hand, it also results in the rather small $J_c$ of A$_x$Fe$_{2-y}$Se$_2$ even compared to FeCh [10, 11].

Recently, superconductivity with $T_c$ up to about 45 K has been reported in AM$_x$(NH$_3$)$_y$Fe$_2$Ch$_2$ (AM = alkali, alkaline-earth, and rare-earth metals) [12–18]. Previous studies indicate that Fe vacancies are almost absent in these materials [15, 18]. Thus, it is promising that AM$_x$(NH$_3$)$_y$Fe$_2$Ch$_2$ will have a relatively high $J_c$. Moreover, the enhanced $T_c$ and mass anisotropy due to large interlayer distance along the c axis in AM$_x$(NH$_3$)$_y$Fe$_2$Ch$_2$ could result in the significant increase of the Ginzburg number $G_0$, i.e. the vortex motion and fluctuations would become quite strong. It causes some very interesting phenomena in vortex dynamics, such as giant-flux creep and thermally-activated flux flow, etc. Because of the difficulty of single crystal growth for AM$_x$(NH$_3$)$_y$Fe$_2$Se$_2$, related study is still absent.

In this work, we report the study of the $J_c$ of Li$_x$(NH$_3$)$_y$Fe$_2$Te$_{1.2}$Se$_{0.8}$ (LiFeTeSe-122) single crystals synthesized by the low-temperature ammonothermal method. The $J_c$ reaches $2.6 \times 10^5$ A/cm$^2$ at 2 K. Detailed analysis suggests that the main pinning sources are surface-like defects...
with normal core and flux creep cannot be ignored when analysing vortex dynamics of this material.

2. Experimental

FeTe_{0.6}Se_{0.4} single crystals were grown by self-flux method with nominal ratio of Fe : Te : Se = 1 : 0.6 : 0.4, Fe pieces (99.98%), selenium shots (99.999%), and Te grains (99.99%) were mixed and loaded into an alumina crucible, which was sealed in the quartz tube under partial argon atmosphere. The sealed ampoule was heated to 1273 K and kept at this temperature for 24 h. Then it was cooled to room temperature slowly. The LiFeTeSe-122 single crystals were synthesized by the low-temperature ammonothermal technique [12–14, 19]. The pieces of Li metal and FeTe_{0.6}Se_{0.4} single crystals in the molar ratio of 1 : 2 were loaded into the high-pressure vessel (25 ml) with a magnetic stirrer. All of these processes were carried out in an argon-filled glovebox with O_2 and H_2O content below 0.1 ppm. Then, the vessel was taken out from the glovebox and connected to a vacuum line equipped with a molecular pump and a NH_3 gas line. The vessel was evacuated by using a molecular pump (~1 × 10^-3 Pa) before introducing NH_3 and placed in an ethanol bath cooled to ~238 K, then the NH_3 gas was condensed into the vessel for 20 min. After that, the vessel was taken out from the cooling bath and stirred for 2 d at room temperature in order to facilitate the reaction and to improve the homogeneity of intercalation. Finally, the NH_3 gas was evacuated using a molecular pump. X-ray diffraction (XRD) patterns were collected using a Bruker D8 x-ray Diffractometer with Cu Kα radiation (λ = 0.15418 nm) at room temperature. Rietveld refinement of the XRD patterns was performed using the code TOPAS4 [20]. The elemental analysis was performed using inductively coupled plasma atomic emission spectroscopy (ICP-AES). Magnetization measurements were performed in a Quantum Design Magnetic Property Measurement System up to 5 T.

3. Results and discussion

Figure 1(a) shows the XRD patterns of FeTe_{0.6}Se_{0.4} and LiFeTeSe-122 single crystals. Only (00l) reflections can be indexed, indicating that the surfaces of crystals are parallel to the (00l)-plane. The diffraction peaks of LiFeTeSe-122 shift to a lower angle when compared to FeTe_{0.6}Se_{0.4}, suggesting a larger interlayer distance in the former. The typical size of LiFeTeSe-122 single crystals is about 1 × 2 mm^2 (inset of figure 1(b)), similar to the size of parent crystals, i.e. the shape of the crystal is roughly unchanged during the intercalation process. It shows that the intensity of XRD diffraction peaks of LiFeTeSe-122 is weaker than that of FeTe_{0.6}Se_{0.4}, leading to more obvious background signal in the former. The weakened diffraction intensity could be due to the increased roughness of the surface of the crystals after intercalation. Figure 1(b) shows the powder XRD pattern of LiFeTeSe-122 and the fitted a- and c-axial lattice parameters are 3.8270(8) and 18.17(1) Å, consistent with previous results [18]. There are weak diffraction peaks originating from Fe(Re, Se). This is not due to the incomplete intercalation of Li-NH_3, but the decomposition of LiFeTeSe-122 during grinding for powder XRD measurement. If the intercalation is incomplete, the superconducting transition of Fe(Re, Se) with T_c ~ 15 K would be clearly observed in the curves of magnetic susceptibility, which is not the case in our experiment (shown below). The atomic ratio of Li : Fe : Te : Se determined from the ICP-AES analysis is 0.16 : 1 : 0.60 : 0.38. The molar ratio of Fe to Se is perfectly consistent with the nominal ratio of the parent compound. More importantly, there is no Fe vacancy in the LiFeTeSe-122 crystals. The temperature dependence of magnetic susceptibility 4πχ(T) at low-temperature region for H∥c (figures 1(c) and (d)) clearly shows superconducting transition in both samples. The onset superconducting transition temperature is T_{c onset} ~ 14 K for FeTe_{0.6}Se_{0.4}, consistent with previous results in the literature [18]. After intercalation, the T_{c onset} is enhanced to about 21 K, which is slightly higher than that of the powder sample [18]. After considering the demagnetization effect of the sample by using the formula

\[ 4πχ_{eff} = 4πχ/(1 - 4πNdH) \]

where \( N_d \) is demagnetization factor [21] (0.70 and 0.82 for FeTe_{0.6}Se_{0.4} and LiFeTeSe-122, respectively), the estimated superconducting volume fractions (SVFs) from zero-field-cooling 4πχ_{eff} curves at 2 K for both samples are ~100%, clearly indicating bulk superconductivity in these crystals. On the other hand, the smaller SVFs determined from the field-cooling curves imply that both of them are type-II superconductors with rather strong vortex pinning effects.

The magnetization hysteresis loops (MHLs) of LiFeTeSe-122 and FeTe_{0.6}Se_{0.4} single crystals at T = 2 K for H∥c are shown in figure 2(a). The symmetrical shapes of MHLs for two samples are typical of type-II superconductors, indicating that bulk pinning is dominant without ferromagnetic impurity in the samples. Importantly, the MHL of the intercalated crystal is much larger than that of the parent one, implying that the pinning force is greatly enhanced in the intercalated sample. According to the Bean model [22, 23], the critical current density can be determined from the MHLs. For a rectangularly-shaped crystal with dimension \( c \times a \times b \), when \( H∥c \), the in-plane critical current density \( J^{ab}_c(\mu_0H) \) is given by

\[ J^{ab}_c(\mu_0H) = \frac{20ΔM(\mu_0H)}{a(1 - a/3b)} \]

where \( a \) and \( b \) (\( a < b \)) are the in-plane sample size in cm, \( ΔM(\mu_0H) \) is the difference between the magnetization values for increasing and decreasing fields at a particular applied field value (measured in emu/cm^3), and \( J^{ab}_c(\mu_0H) \) is the critical current density in A/cm^2. As shown in figure 2(b), at \( T = 2 \) K, the \( J^{ab}_c(\mu_0H) \) of FeTe_{0.6}Se_{0.4} at self-field is about 6.7 × 10^5 A/cm^2. In contrast, the \( J^{ab}_c(\mu_0H) \) of LiFeTeSe-122 at self-field is about four times larger than that of the parent sample and reaches 2.6 × 10^5 A/cm^2. Moreover, the decrease of \( J^{ab}_c(\mu_0H) \) is rather slow with increasing magnetic field, suggesting strong vortex pinning effect in the sample. It has to be mentioned that because of the bulk
superconductivity with large SVF in LiFeTeSe-122, its $J_{c}^{ab}$ is much larger even than that of K$_{x}$Fe$_{2-y}$Se$_{2}$ which has much higher $T_{c}$ ($\sim$32 K) [24, 25]. However, the $J_{c}^{ab}$ of LiFeTeSe-122 is still low when compared to the iron pnictide superconductors where the typical self-field $J_{c0}^{ab}$ is above 10$^6$ A/cm$^2$ at 2 K [26].

Figures 3(a) and (b) show the MHLs of LiFeTeSe-122 single crystal for $H || c$ at the temperature ranges of 2 K to 8 K and 10 K to 18 K, respectively. The hysteresis area decreases with increasing temperature, indicating the $J_{c}^{ab}$ decreases as temperature increases. The derived $J_{c}^{ab}(\mu_{0}H)$ of LiFeTeSe-122 single crystal at different temperatures from the MHLs using equation (1) is shown in figure 3(b). The $J_{c}^{ab}(\mu_{0}H)$ is robust against the applied field at low temperatures, but the slopes of $J_{c}^{ab}(\mu_{0}H)$ versus $\mu_{0}H$ become larger at high temperatures, indicating a significant thermally-activated depinning process.

The vortex pinning force $F_{p}(=\mu_{0}H \times J_{c}^{ab})$ can provide more information about the vortex pinning mechanism in LiFeTeSe-122 single crystals. According to the Dew-Huges model [27], if one pinning mechanism is dominant in a certain temperature range, the normalized vortex pinning force $f_{p}(h)$ at different temperatures should be proportional to $h^{p}(1 - h)^{q}$, where $F_{p}^{\text{max}}$ is the maximum pinning force, the indices $p$ and $q$ are determined by the pinning mechanism, $h = H/H_{irr}$ is the normalized field, and the irreversibility field $H_{irr}$ is estimated by extrapolating $J_{c}^{ab}(T, \mu_{0}H)$ to zero. Figure 4(a) shows the relationship between $f_{p}(h)$ and $h$ at different temperatures ($T > 6$ K) for $H || c$. It can be clearly seen that the $f_{p}(h)$ as a function of $h$ exhibits a temperature independence scaling law, suggesting the dominance of a single pinning mechanism. The fitting using $f_{p}(h) \propto h^{p}(1 - h)^{q}$ gives $p = 0.63(3)$ and $q = 2.52(8)$. The value of $h_{\text{max}}^{\text{in}}$ calculated by $p/(p + q)$ equals 0.202, consistent with the peak positions obtained from the experimental curves at all temperatures $h_{\text{exp}}^{\text{in}} \approx 0.207$. Moreover, for $T = 6$ and 8 K, the $H_{irr}$ could be estimated by locating the field of $F_{p}^{\text{max}}$ at $h_{\text{exp}}^{\text{in}}$. Partial $f_{p}(h, T)$ curves at $T = 6$ and 8 K also exhibit the same
scaling law, suggesting that the same pinning mechanism is dominant above 6 K. On the other hand, when \( T < 6 \) K, the scaling behavior cannot be analyzed because of the absence of \( F_{p}^{\text{max}} \). The values of \( p \), \( q \) and \( h_{\text{fit}}^{\text{max}} \) are close to the expected values \( (p = 0.5, q = 2, \text{ and } h_{\text{fit}}^{\text{max}} = 0.2) \) for the pinning of surface-like defects with normal core [27]. The slightly larger values of \( p \) and \( q \) than theoretical predictions suggest that flux creep might have some influence on pinning force [28]. Interestingly, similar values of \( p \) and \( q \) have also been observed in FeS and FeS\(_{0.94}\)Se\(_{0.06}\) single crystals prepared by deintercalating potassium from \( K_{x}Fe_{2-y}(S, Se)_{2} \) using the hydrothermal method [29]. It suggests that there is a common type of pinning center in these materials in contrast to FeS-\( e_{0.5}Te_{0.5} \) thin films, \( K_{x}Fe_{2-y}(S, Se)_{2} \), and \( Ba_{0.6}K_{0.4}Fe_{2}As_{2} \), where point-like defects with normal core are the dominant pinning sources [24, 26, 30]. Moreover, \( F_{p}^{\text{max}} \) can be fitted using \( F_{p}^{\text{max}} = A(h_{0}H_{\text{fit}})^{\alpha} \) (inset of figure 4(a)) and the obtained \( \alpha \) is 1.67(4), also close to the theoretical value \( (\alpha = 2) \) [27].

On the other hand, the self-field \( J_{c}^{\text{ab}}(\mu_{0}H) \) reduces quickly at a low-temperature region and this trend then becomes milder at higher temperatures (figure 4(b)). It implies that flux creep needs to be considered in vortex dynamics of LiFeTeSe-122 single crystals. In the framework of the thermally-activated
According to the collective theory, there are two pinning mechanisms, we have $J_{\text{c}}(T)$ = $xJ_{\text{c}}^{\text{ab}}(T) + (1-x)J_{\text{c}}^{\text{bl}}(T)$, where $x$ represents the contribution of $J_{\text{c}}^{\text{ab}}(T)$. The $J_{\text{c}}^{\text{bl}}(T)$ can be well fitted using equation (2) (figure 4(b)). The fitted $x$ is 0.15(5) with $\mu = 1.1$ (1). The value of $\mu$ is between the prediction of small-bundle and large-bundle regimes and similar to the value observed in FeTe$_{0.99}$Se$_{0.4}$ single crystals [35]. The small but non-zero $x$ indicates that both $\delta T_c$ and $\delta l$ pinning mechanisms play roles in LiFeTeSe-122 single crystals, but the latter one is dominant.

As shown in figure 4(c), the temperature dependence of $\mu_0H_{\text{c1}}(T)$ agrees well with the theoretical fitting based on the flux creep model and the obtained parameters are $K = 40(1)$, $m = 1.9(1)$, and $\gamma = 0.20(3)$. The values of $m$ and $\gamma$ are similar to those in Hg-based cuprate superconductors and SmFeAsO$_{0.85}$ [37, 38].

Finally, it has to be mentioned that the $J_c$ of FeTe$_{0.99}$Se$_{0.4}$ single crystals studied in the present work is not the highest one when compared to those reported in the literature [35]. This could be due to different conditions of crystal growth and a number of defects in the crystals. If the quality of parent compounds Fe(Fe, Se) can be improved further, the $J_c$ of LiFeTeSe-122 could be even higher. On the other hand, the Fe(Fe, Se) films can have larger $J_c$ than single crystals because of various kinds of external factors such as interface effects, nonmagnetic/magnetic point/nanorod defects/inclusions introduced during the preparation process of the films, pinning of grain boundaries, etc [39–42]. Moreover, the enhancement of $T_c$ has been observed in Fe(Fe, Se) films and it is inextricably linked to the strain induced during the epitaxial growth [40, 43]. It would be very interesting to examine whether the $J_c$ and $T_c$ of films could increase further when intercalating Li-NH$_3$ with the proper doping level of electron carriers.

4. Conclusion

In summary, we investigate the critical current density $J_c$ of LiFeTeSe-122 single crystals grown using the low-temperature ammonothermal method. The cointercalation of Li and NH$_3$ not only increases the $T_c$ from 14 K to 21 K, but also significantly increases the $J_c$ to $2.6 \times 10^5$ A/cm$^2$ at 2 K. Detailed analysis of the vortex dynamics indicates that the dominant pinning sources are surface-like defects with normal core. Moreover, lux creep is important to the vortex dynamics of LiFeTeSe-122 single crystals and the analysis of self-field $J_{\text{c}}^{\text{ab}}$ suggests that the $\delta l$ pinning mechanism due to spatial fluctuations of the charge-carrier mean free path is dominant at the measured temperature range.

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**Figure 4.** (a) Normalized vortex pinning force $f_p = F_p/F_{\text{pin}}^{\text{max}}$ as a function of normalized field $h = H/H_m$ for LiFeTeSe-122 single crystal at various temperatures. Solid line represents the fitting curve using $f_p \propto h^\gamma(1 - h)$ is set. Inset shows $F_{\text{pin}}^{\text{max}}$ as a function of $\mu_0H_{\text{c1}}$. The fitting result using $F_{\text{pin}}^{\text{max}} = A(\mu_0H_{\text{c1}})^\beta$ is shown as a solid line. The measured and estimated $\mu_0H_{\text{c1}}$ are shown as closed and open circles. (b) Reduced temperature dependence of self-field $J_{\text{c}}^{\text{bl}}(T)$. The solid line is the theoretical curve obtained based on equation (2) with the coexistence of $\delta l$ and $\delta T_c$ pinning mechanisms. (c) Temperature dependence of $\mu_0H_{\text{c1}}(T)$. Solid line is the fitting using the flux creep model.
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