Multiband behavior and non-metallic low-temperature state of K$_{0.50}$Na$_{0.24}$Fe$_{1.52}$Se$_2$

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We report evidence for multiband transport and an insulating low-temperature normal state in superconducting K$_{0.50}$Na$_{0.24}$Fe$_{1.52}$Se$_2$ with $T_c \approx 20$ K. The temperature-dependent upper critical field, $H_{c2}$, is well described by a two-band BCS model. The normal-state resistance, accessible at low temperatures only in pulsed magnetic fields, shows an insulating logarithmic temperature dependence as $T \to 0$ after superconductivity is suppressed. This is similar as for high-$T_c$ copper oxides and granular type-I superconductors, suggesting that the superconductor-insulator transition observed in high magnetic fields is related to intrinsic nanoscale phase separation.

The powder XRD data (Fig. I(a)) demonstrate the phase purity of our samples without any extrinsic peak present. The pattern is refined in the space groups $I4/mmm$ and $I4$ with fitted lattice parameters $a = 0.3870(2)$ nm, $c = 1.4160(2)$ nm and $a = 0.8833(2)$ nm, $c = 1.4075(2)$ nm, respectively, reflecting phase separation and small sample yield. With Na substitution, the lattice parameter $a$ decreases while $c$ increases when compared to K$_{0.8}$Fe$_2$Se$_2$, consistent with lattice parameters of NaFe$_2$Se$_2$. The average stoichiometry was determined by EDX, measuring multiple positions on the crystal. The obtained composition K$_{0.50(1)}$Na$_{0.24(4)}$Fe$_{1.52(3)}$Se$_{2.00(5)}$ suggests vacancies on both K and Fe sites. FeSe-122 superconductors feature an intrinsic phase separation into magnetic insulating and superconducting regions. As shown in the SEM image of Fig. II(b), K$_{0.50(1)}$Na$_{0.24(4)}$Fe$_{1.52(3)}$Se$_{2.00(5)}$ also exhibits a similar array of superconducting grains in an insulating matrix. The observed pattern is somewhat inhomogeneous with sizes ranging from about several microns to probably several tens
of nanometers,\textsuperscript{7} below our resolution limit. It would be of interest to investigate the local structure and electronic properties of $K_{0.50(1)}Na_{0.24(4)}Fe_{1.52(3)}Se_2$, since Na substitution provides chemically induced pressure which might suppress the phase separation similar as for $Rb_{1-x}Fe_{2-x}Se_2$\textsuperscript{20}.

The investigated single crystal becomes superconducting at 20 K after and at 28 K before the annealing and quenching procedure [Fig. 2(a) main part and inset, respectively]\textsuperscript{14,15} For the quenched crystal, the superconducting volume fraction at 1.8 K increases significantly up to 72%, albeit with a reduction of $T_c$. The post-annealing and quenching process results in a surface oxidation of some crystals which then dominates the magnetization signal. However, Fe$_3$O$_4$ is not visible in either of our laboratory or synchrotron X-ray studies\textsuperscript{14,15}. The magnetic hysteresis loops (MHL) of the quenched $K_{0.50(1)}Na_{0.24(4)}Fe_{1.52(3)}Se_2$ single crystal reflects the improvement in crystalline homogeneity since it is much larger and symmetric when compared to an as-grown sample [Fig. 2(b)] due to stronger pinning forces and bulk pinning\textsuperscript{24}. Also similar to $K_xFe_{2-x}Se_2$, there is an enhancement of the in-plane critical-current density calculated from the Bean model\textsuperscript{27,28} $J_c^0(\mu_0 H) = \frac{20\Delta M(\mu_0 H)}{a(1-a/\xi_0)}$, where $a$, $b$, and $c$ are the lengths of a rectangularly shaped crystal ($b > a > c$). In view of the improved volume fraction and homogeneity, further investigations of the electronic transport properties were performed on the quenched crystal.

The resistance of an inhomogeneous sample contains contributions from both metallic ($R_m$) and nonmetallic ($R_i$) regions. At $T < T_c$, due to superconductivity ($R_m = 0$) the insulating part of the sample is short-circuited. The isolating regions have a several orders of magnitude higher resistivity than the metallic part; hence, around $T_c$ and when $T \to 0$ in the high-field normal state $R(T) \approx R_m(T)$. This is similar to the resistance of a polycrystalline sample in the presence of grain boundaries and in agreement with the observation that isolating regions do not contribute to the spectral weight in angular resolved photoemission data in the energy range near $E_F$.\textsuperscript{20} In what follows below, we focus on the temperature-dependent sample resistance, $R(T)$.

The superconducting transition in $R_{ab}(T)$ is rather wide and shifts to lower temperatures in applied magnetic fields [Figs. 3(a-d)]. The shift is more pronounced for $H \parallel c$, which implies an anisotropic $\mu_0 H_{c2}$. The temperature-dependent upper critical fields shown in Fig. 3(c) were determined from the resistivity drops to 90%, 50%, and 10% of the normal-state value. It is clear

\begin{table}[h]
\centering
\begin{tabular}{|c|c|c|c|}
\hline
$T_c$ & $(d\mu_0 H_{c2}/dT)|_{T-T_c}$ & $\mu_0 H_{c2}(0)$ & $\xi(0)$
\hline
\hline
$H \perp c$ & 14.1(5) & -4.3(3) & 150〜160 & 2.62〜2.95
\hline
$H \parallel c$ & 14.1(5) & -1.1(2) & 38〜48 & 0.75〜0.79
\hline
\end{tabular}
\caption{Superconducting parameters of the quenched $K_{0.50(1)}Na_{0.24(4)}Fe_{1.52(3)}Se_2$ single crystal.}
\end{table}

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{fig1}
\caption{(Color online) (a) Powder XRD pattern of $K_{0.50(1)}Na_{0.24(4)}Fe_{1.52(3)}Se_2$. The plot shows the observed (+) and calculated (solid red line) powder pattern with the difference curve underneath. Vertical tick marks represent Bragg reflections in the I4/mmm (upper green marks) and I4/m (lower blue marks) space group. (b) SEM image of the crystal.}
\end{figure}

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{fig2}
\caption{(Color online) (a) Temperature dependence of the ac magnetic susceptibilities of as-grown (magnified in the inset) and quenched $K_{0.50(1)}Na_{0.24(4)}Fe_{1.52(3)}Se_2$. (b) Magnetic hysteresis loops of as-grown (triangles) and quenched (inverted triangles) samples at $T = 1.8$ K (closed symbols) and $T = 300$ K (open symbols) for $H \parallel c$. (c) Superconducting critical current densities, $J_c^0(\mu_0 H)$, at $T = 1.8$ K.}
\end{figure}
that all experimental data feature a similar temperature dependence irrespective of the criteria used. All data for $H||c$ are above the expected values for the single-band Werthamer-Helfand-Hohenberg (WHH) model (dotted lines). We proceed our further analysis using the 10% values, similar as done for LaFeAsO$_{0.85}$F$_{0.15}$.

From $H_{c2}(T)$ curves are linear for $H \perp c$ near $T_c$ and show an upturn at low $T$ for $H||c$ [Fig. 3(e)]. The initial slope near $T_c$ for $H \perp c$ is much larger than for $H||c$ [Fig. 3(f) and Table I]. These slopes are similar to values for as-grown and quenched K$_2$Fe$_2$As$_2$.

There are two basic mechanisms of Cooper-pair breaking by magnetic field in a superconductor. Orbital pair breaking imposes an orbital limit due to the induced screening currents, whereas the Zeeman effect contributes to the Pauli paramagnetic limit of $H_{c2}$. In the single-band WHH approach, the orbital critical field is given by

$$
\mu_0H_{c2}(0) = \frac{-0.693T_c(d\mu_0H_{c2}/dT)_{T=T_c}}{\pi T_T^{2D}}
$$

For K$_{0.50(1)}$Na$_{0.24(4)}$Fe$_{1.52(3)}$Se$_2$, this leads to $42(3)$ T for $H \perp c$ and $10(2)$ T for $H||c$ [Fig. 3(f)]. On the other hand, the Pauli-limiting field is given by

$$
\mu_0H_p(0) = \frac{1.86T_c(1+\lambda_{c-ph})^{1/2}}{\sqrt{2}}
$$

$\mu_0H_p(0)$ is $32(1)$ T. This is larger than the orbital pair-breaking field for $H||c$ estimated above, yet smaller than the value for $H \perp c$, which possibly implies that electron-phonon coupling is much stronger than for typical weak-coupling BCS superconductors.

The experimental data for $\mu_0H_{c2}(0)$ lie above the expected values from WHH theory [Fig. 3(f)], suggesting that multiband effects are not negligible. In the dirty limit, the upper critical field found for the two-band BCS model with orbital pair breaking and negligible interband scattering is

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

$$
U(x) = \psi(1/2 + x) - \psi(1/2),
$$

where $t = T/T_c$, $\psi(x)$ is the digamma function, $\eta = D_2/D_1$, $D_1$ and $D_2$ are diffusivities in band 1 and band 2, $h = H_{c2}/D_1/(2\phi_0T)$, and $\phi_0 = 2.07 \times 10^{-15}$ Wb is the magnetic flux quantum. $a_0 = 2w/\lambda_0$, $a_1 = 1 + \lambda_1/\lambda_0$, and $a_2 = 1 - \lambda_1/\lambda_0$, where $w = \lambda_{11} \lambda_{22} - \lambda_{12} \lambda_{21}$, $\lambda_0 = (\lambda_1^2 + 4\lambda_1\lambda_2)^{1/2}$, and $\lambda_1 = \lambda_{11} - \lambda_{22}$. $\lambda_{11}$ and $\lambda_{22}$ are pairing (intraband coupling) constants in band 1 and 2, and $\lambda_{12}$ and $\lambda_{21}$ quantify interband couplings between bands 1 and 2. For $D_2 = D_1$, Eq. (1) simplifies to the one-band model (WHH) in the dirty limit. When describing our data by use of the two-band BCS model fitting, we consider two different cases, $w > 0$ and $w < 0$, which imply either dominant intraband or dominant interband coupling, respectively. The solid lines in Figs. 3(f) and 3(i) are fits using Eq. (1) for $\lambda_{11} = \lambda_{22} = 0.5$ and $\lambda_{12} = \lambda_{21} = 0.25$ which indicates strong intraband coupling.

The extrapolated $\mu_0H_{c2}(0)$ is $38$ T for $H||c$ and $150$ T for $H \perp c$. Further, the dashed lines in Figs. 3(f) and 3(i) show fits with $\lambda_{11} = \lambda_{22} = 0.49$ and $\lambda_{12} = \lambda_{21} = 0.5$ for strong interband coupling that give $\mu_0H_{c2}(0) \approx 48$ T for $H||c$ and $\approx 160$ T for $H \perp c$.

From these fits we obtain $\eta$ values of 0.063 and 0.021 for dominant intraband ($w > 0$) and interband ($w < 0$) coupling, respectively, i.e., largely different $D_1$ and $D_2$ implying different electron mobilities in the two bands. The upward curvature of $\mu_0H_{c2}(T)$ is governed by $\eta$; it is more pronounced for $\eta \ll 1$. The large difference in the intraband diffusivities could be due to pronounced differences in effective masses, scattering, or strong magnetic excitations. The fit results are not very sensitive to the choice of the coupling constants, yet they mostly depend on $\eta$. This indicates either similar interband and intraband coupling strengths or that their difference is beyond our resolution limit. Our results are consistent
FIG. 4. (Color online) Temperature dependence of the resistance in several DC and pulsed magnetic fields for $H||c$.

with the data obtained on pure crystals, i.e., the large difference of electronic diffusivities for different Fermi surface sheets is maintained in the doped crystal. This is in agreement with the band-structure calculations that showed negligible contribution of K to the Fermi surface and density of states at the Fermi level. On the other hand, we find no enhancement of the superconducting $T_c$ with Na substitution in $K_xFe_{2−y}Se_2$ ($T_c \sim 30$ K). This is somewhat surprising because $Na_xFe_2Se_2$ and $NaFe_2Se_2$ that crystallize in $I4/mmm$ space group have $T_c$'s of 45 and 46 K. The Na substitution might affect the magnetic order in phase separated $K_xFe_{2−y}Se_2$ since the existence of a large magnetic moment in the antiferromagnetic phase was proposed to be important for the relatively high $T_c$.

Due to the limited data points, it is difficult to unambiguously estimate $\mu_0H_{c2}(0)$ for $H \perp c$. Based on results reported for similar Fe-based superconductors, $\mu_0H_{c2}(0)$ shows a pronounced upward curvature for $H||c$ while it tends to saturate for $H \perp c$. The real $\mu_0H_{c2}(0)$ for $H \perp c$ might be smaller than we estimated. The calculated coherence lengths, using $\mu_0H_{c2}(0) = \phi_0/2\pi\xi_{\perp}(0)\xi_{\parallel}(0)$ and $\mu_0H_{c2}''(0) = \phi_0/2\pi\xi_{\perp}(0)^2$ based on the two-band BCS fit results, are similar to values obtained for as-grown and quenched $K_xFe_{2−y}Se_2$ and are shown in Table I.

Superconductivity in $K_{0.50(1)}Na_{0.24(4)}Fe_{1.52(3)}Se_2$ is completely suppressed above about 60 T for $H||c$, allowing for a clear insight into the low-temperature electronic transport in the normal state (Fig. 3). Interestingly, we do not observe metallic transport below about 40 K implying that a superconductor-to-insulator transition (SIT) is induced in high magnetic fields. Kondo-type magnetic scattering is not very likely since a field of 62 T should suppress spin-flip scattering. A thermally activated semiconductor-like transport or variable range hopping (VRH) as occurring for Anderson localization is unlikely since the resistance in 62 T cannot be fit by $ln R \propto −1/T$, $ln R \propto T^{−\beta}$, with $\beta = 1/2, 1/3, 1/4$, and $ln R \propto T^{−\beta}$ instead. The resistance increases logarithmically with decreasing temperature in the normal state at 62 T as shown with the dashed line in Fig. 3. Hence, the SIT might originate from the granular nature of $K_{0.50(1)}Na_{0.24(4)}Fe_{1.52(3)}Se_2$. In a bosonic SIT scenario, Cooper pairs are localized in granules. When $H > H_{c2}$, virtual Cooper pairs form, yet they cannot hop to other granules when $T \rightarrow 0$ which induces the increase in resistivity as temperature decreases. The grain size can be estimated from $H_{c2}^0 \sim \phi_0/L_{\xi}$, where $L$ is the average grain radius and $\xi \approx 0.77$ nm is the average in-plane coherence length. The obtained $L = 62$ nm is in agreement with the phase-separation distance. The bosonic SIT mechanism in granular superconductors predicts $R = R_0\exp(T/T_0)$ (‘inverse Arrhenius law’) in the superconducting region near the SIT when $H < H_{c2}$ due to the destruction of quasi-localized Cooper pairs by superconducting fluctuations. Our data in 14, 20, and 30 T might be fitted with this formula (solid lines in Fig. 3).

In summary, we reported the multiband nature of superconductivity in $K_{0.50}Na_{0.24}Fe_{1.52}Se_2$ as evidenced in the temperature dependence of the upper critical field and a SIT in high magnetic fields. Granular type-I but also copper-oxide superconductors are also intrinsically phase separated on the nanoscale. Hence, a SIT in high magnetic fields seems to be connected with the intrinsic materials’ granularity in inhomogeneous superconductors. This suggests that the insulating states found in cuprates as a function of magnetic field or doping might involve Josephson coupling of nanoscale grains as opposed to quasi-one-dimensional metallic stripes bridged by Mott-insulating regions in the spin-charge separated picture.

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