The effect of impurity and the suppression of superconductivity in Na(Fe$_{0.97-x}$Co$_{0.03}$T$_x$)As (T = Cu, Mn)

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Abstract
We report the successful growth of and the effect of impurity scattering in single crystals of Na(Fe$_{0.97-x}$Co$_{0.03}$T$_x$)As (T = Cu, Mn). The temperature dependence of the DC magnetization at high magnetic fields is measured for different concentrations of Cu and Mn. Detailed analysis based on the Curie–Weiss law indicates that Cu doping weakens the average magnetic moments, while doping with Mn enhances the local magnetic moments greatly, suggesting that the former may produce nonmagnetic or very weak magnetic impurities, and that the latter may give rise to magnetic impurities. However, it is found that both doping with Cu and doping with Mn will enhance the residual resistivity and suppress the superconductivity, at similar rates, in the low doping region, which is consistent with the prediction of the S$^\pm$ model. For the Cu-doped system, the superconductivity is suppressed completely at a residual resistivity of $\rho_0 = 0.87$ m\$\Omega$ cm, for which a strong localization effect is observed. However, in the case of Mn doping, the suppression of $T_c$ becomes much weaker beyond $x = 0.03$ and superconductivity is maintained even up to a residual resistivity of 2.86 m\$\Omega$ cm. Clearly the magnetic Mn impurities may even be not as detrimental as the nonmagnetic or very weak magnetic Cu impurities to the superconductivity in the high doping regime.
1. Introduction

The discovery of iron-based superconductors in 2008 has brought new vigor and vitality to the study of unconventional superconductivity [1]. The pairing symmetry is very much crucial for understanding the superconducting mechanism. The pairing symmetry in the cuprates was proved to be d-wave type [2], while its form in the iron-based superconductors is still under debate. Theoretically, a pairing model with the gap structure of $S^{\pm}$ was proposed. This model is based on the assumption that the pairing is established by exchanging the paramagnons given by the antiferromagnetic spin fluctuations between two electrons with opposite momenta and spins. This kind of pairing will naturally lead to a sign reversal of the order parameter between the electron and the hole Fermi pockets [3–7]. This model has gained support from many experiments, including scanning tunneling spectroscopy (STS) measurements [8, 9] and inelastic neutron scattering experiments [10]. On the other hand, a pairing state without sign reversal of the gaps, namely $S^{++}$, was proposed for the scenario where the pairing is mediated by orbital fluctuations [11–13]. In some special cases, even a d-wave gap may be expected in iron-based superconductors [4, 14, 15].

For the superconducting state, the impurity scattering effect is closely related to the gap structure, the characteristics of the impurities and the underlying electronic structure. The impurity scattering effect may give some important clues as regards how to unravel the pairing gap structure as well as the pairing mechanism. According to Anderson’s theorem [16], in a conventional s-wave superconductor a magnetic impurity can break the Cooper pairs easily, while superconductivity is retained robustly with the presence of nonmagnetic impurities in the unitary limit. In sharp contrast, Anderson’s theorem is seriously violated for unconventional superconductors, where both magnetic and nonmagnetic impurities are detrimental to superconductivity. For cuprate superconductors with a d-wave gap structure, significant $T_c$ suppression was observed with doping with Zn [17–19]. For the case of an $S^{\pm}$ pairing state, it was pointed out that both magnetic and nonmagnetic impurities can suppress $T_c$ rapidly [11, 20]. In efforts to resolve the mystery of the superconducting mechanism, plenty of experiments have been carried out on the impurity effects in iron-based superconductors. Ways to introduce scattering centers include chemical substitutions [21–33] and particle irradiations [34–38]. Unfortunately, the conclusions remain highly controversial. Previous studies on Mn impurities showed that $T_c$ was strongly suppressed in Ba$_{0.5}$K$_{0.5}$Fe$_2$As$_2$ [21, 23] and Ba(Fe$_{1-y}$Co)$_2$As$_2$ systems [24], while $T_c$ is not suppressed or is even enhanced in Mn-doped FeSe$_{0.5}$Te$_{0.5}$ superconductors [26]. Zn impurity suppresses $T_c$ rapidly in BaFe$_{1.89}$Zn$_{0.1}$Co$_{0.11}$As$_2$ [25], whereas the superconducting state is retained robustly in Fe$_{1-y}$Zn$_y$Se$_{0.3}$Te$_{0.7}$ [27]. Theoretically it is proposed that the pairing symmetry in iron-based superconductors can differ from material to material [7]. A study of the LaFe$_{1-y}$Zn$_y$AsO$_{1-y}$F$_y$ system showed that the superconducting transition temperature increases in the underdoped regime with doping with Zn impurities, remains unchanged in the optimally doped regime and is severely suppressed in the overdoped regime, suggesting that the pairing symmetry could change from s-wave to $S^{\pm}$ or even d-wave states with increasing doping concentration [28]. In order to have a better understanding of the
pairing symmetry and superconducting mechanism in Fe-based superconductors, further experimental studies, especially with known properties of the impurities, are highly desired.

In this study, we investigate the impurity effect of doping Cu and Mn into the 111-type iron-based superconductors Na(Fe$_{0.97}$Co$_{0.03}$)As. Our study reveals that the Cu doping weakens the average magnetic moments (below 0.4 $\mu_B$/Fe site), while doping with Mn enhances the local magnetic moments greatly, suggesting that Cu dopants behave as nonmagnetic or very weakly magnetic impurities, and Mn dopants are magnetic ones. It is found that both doping with Cu and doping with Mn can enhance the residual resistivity and suppress the superconductivity rapidly, which is consistent with the prediction of the S$^\pm$ pairing model in the low doping region.

2. Experimental methods

The single crystals of Na(Fe$_{0.97}$Co$_{0.03}$)As (named as the pristine sample) and Na(Fe$_{0.97}$−$x$Co$_{0.03}$T$_x$)As (T = Cu and Mn) were synthesized by the self-flux method using NaAs as the flux. Firstly, NaAs was prepared as the precursor. The Na (purity 99%, Alfa Aesar) was cut into pieces and mixed with As powders (purity 99.99%, Alfa Aesar), and the mixture was put into an alumina crucible and sealed in a quartz tube in vacuum. Then it was slowly heated up to 200°C and held there for 10 h; this was followed by cooling down to room temperature. Then the resultant NaAs and Fe (purity 99.9%, Alfa Aesar), Co (purity 99.9%, Alfa Aesar), and Cu or Mn (purity 99.9%, Alfa Aesar) powders were weighed and mixed with an atomic ratio of NaAs:Fe:Co:T = 4:0.97−$x$:0.03:$x$ and ground thoroughly. The mixture was loaded into an alumina crucible, then sealed in an iron tube under Ar atmosphere. The iron tube was then sealed in an evacuated quartz tube to prevent oxidization of the iron tube. Then it was placed into the furnace and heated up to 950°C and held there for 10 h; this was followed by cooling down to 600°C at a rate of 3°C h$^{-1}$, to grow single crystals. Single crystals with shiny surfaces and typical dimensions of 2−5 mm with thicknesses of about 0.2−0.6 mm were obtained. In the preparation process, the weighing, mixing, and grinding were conducted in a glove box under an argon atmosphere with the O$_2$ and H$_2$O below 0.5 ppm. X-ray diffraction (XRD) measurements were performed using a Bruker D8 Advanced diffractometer with Cu K$_\alpha$ radiation. DC magnetization measurements were carried out with a SQUID-VSM-7 T (Quantum Design). The in-plane resistivity measurements were done using a PPMS-16 T (Quantum Design) with the standard four-probe method.

3. Results and discussion

Figure 1 shows the XRD patterns of the Na(Fe$_{0.97}$−$x$Co$_{0.03}$T$_x$)As (T = Cu, Mn) single crystals. Only (00$l$) peaks can be observed with a relatively small full width at half-maximum (FWHM), indicating that the cleavage plane is the $ab$ plane and showing the high quality of the samples. As shown in figures 2(a) and (b), the peaks of the (004) reflection shift monotonically in 2\theta with increase of the doping concentration, indicating that the impurities were doped into the crystal lattice successfully. This conclusion is also supported by the monotonic increase of the residual resistivity versus the doping level in both systems. The $c$-axis lattice parameter is obtained and plotted as a function of the doping concentration $x$, as shown in figure 2(c). For the pristine
sample, the $c$-axis lattice parameter is 7.048 Å, which is consistent with the previously reported results within experimental error [39]. We can see that the $c$-axis lattice parameter slightly increases with increase of the Mn doping concentration, while it decreases in the Cu-doped samples. This behavior is similar to the results presented in other reports [23, 32].

3.1. Magnetization and resistivity measurements

In figure 3, we present the temperature dependence of the in-plane resistivity from 2 K to 300 K for Na(Fe$_{0.97-x}$Co$_{0.03}$Cu$_x$)As single crystals. Obviously, $T_c$ drops down and the residual resistivity goes up with increase of the Cu concentration. In the low doping region, the resistivity decreases upon cooling, and this is followed by a superconducting transition. For the highly doped sample, an upturn is observed at low temperatures. It is interesting to note that, for the sample $x = 0.03$, the transition seems to be broad; an initial drop of resistivity starts at about 16 K, which is followed by a sharper drop at about 8–9 K. This may be induced by a chemical segregation. But the strange point is that this broadened transition occurs in most of the measured curves for this doping level, even from the samples of different batches. We notice that actually the normal state starts to show a low $T$ upturn at this doping point. The low $T$ upturn gets stronger with increase of the Cu content, which may be induced by a stronger localization effect. A strong semiconducting-like temperature dependence of the resistivity is observed for the sample with $x = 0.05$–0.06. Interestingly, it is found that $T_c$ is suppressed to
Figure 2. (a), (b) Peaks of the (004) reflections of Na(Fe$_{0.97-x}$Co$_{0.03}$T$_x$)As (T = Cu and Mn) single crystals. (c) The $c$-axis lattice parameter plotted as a function of the doping concentration $x$ for Cu- and Mn-doped samples.

Figure 3. Temperature dependence of the in-plane resistivity of Na(Fe$_{0.97-x}$Co$_{0.03}$Cu$_x$)As.
zero at the threshold of strong semiconducting behavior. For the sample with $x = 0.05$, a downward trend of resistivity is observed at about 2.5 K. So the doping level $x = 0.05$ is regarded as the critical doping concentration which suppresses $T_c$ to zero. We define the residual resistivity $\rho_0$ by extrapolating normal state data in a linear way in the low temperature region to zero temperature, as shown in figure 4(a). One can see that $T_c$ decreases monotonically with increase of $\rho_0$, and it is suppressed to zero at a residual resistivity of 0.87 m$\Omega$ cm. Figure 4(b) shows the temperature dependence of the DC magnetization taken at 20 Oe after the zero-field-cooling (ZFC) and field-cooling (FC) procedures for the superconducting Na(Fe$_{0.97-x}$Co$_{0.03}$Cu$_x$)As single crystals. In this study, $T_c$ is defined on the magnetization curve using the crossing point of the normal state background line and the extrapolated linear line of the steep transition, which is nearly consistent with the point where the resistivity reaches zero. For the pristine sample, $T_c$ reaches about 20.5 K.

The temperature dependence of the in-plane resistivity and the DC magnetization of Na(Fe$_{0.97-x}$Co$_{0.03}$Mn$_x$)As are shown in figure 5 and figure 6(b), respectively. As for the Cu-doped samples, the residual resistivity is determined by a linear extrapolation of normal state data to zero temperature, as shown in figure 6(a). It is clear that the residual resistivity increases with increasing doping level, and consequently $T_c$ is also suppressed monotonically. Interestingly, a similar broadening effect of the resistive transition occurs at about $x = 0.035$; this is similar to

**Figure 4.** (a) Temperature dependence of the resistivity in the low temperature region for Na(Fe$_{0.97-x}$Co$_{0.03}$Cu$_x$)As. The dashed lines represent the linear extrapolations of the normal state data to zero temperature. (b) DC magnetization of Na(Fe$_{0.97-x}$Co$_{0.03}$Cu$_x$)As.
Figure 5. Temperature dependence of the in-plane resistivity of Na(Fe_{0.97-x}Co_{0.03}Mn_{x})As.

Figure 6. (a) The resistivity curve in the low temperature region for Na(Fe_{0.97-x}Co_{0.03}Mn_{x})As. The dashed lines represent the linear extrapolation of the normal state data to zero temperature. (b) DC magnetization of a Na(Fe_{0.97-x}Co_{0.03}Mn_{x})As single crystal.
the case for doping with Cu, and seems to be some kind of intrinsic feature. Compared with Cu-doped samples, Mn-doped ones show much weaker localization even to a very high doping. The enhancements of the residual resistivity versus the doping are similar in the two systems below about $x = 0.03$, but it gets much faster in the Mn-doped system beyond this doping. Interestingly, superconductivity still exists in the sample with a residual resistivity, even up 2.86 mΩ cm. One may argue that the Mn dopants may not be successfully doped into the system when the concentration is higher than a certain value. However, this argument cannot be supported by the doping dependence of the $c$-axis lattice constant, as shown in figure 2(c). Moreover, the residual resistivity of the Mn-doped samples also increases continuously, which again indicates a successful doping of Mn into the material. Clearly, the Mn impurity shows a weaker $T_c$ suppression effect than Cu in the high doping regime. This remains an interesting and unresolved observation.

3.2. DC magnetization and analysis

In order to study the impurity scattering mechanism, it is crucial to determine whether the impurity is magnetic or nonmagnetic. To evaluate the magnetic moments induced by Cu and Mn dopants, we have done the magnetization measurements under high magnetic fields. The raw data from the magnetization measurements at 1 T are shown in figures 7(a) and (b). The clear divergence of the magnetic susceptibility at low temperatures can be understood as indicating the existence of some local magnetic moments. One can see that the low temperature upturn gets enhanced clearly by the Mn doping, but not enhanced by the Cu doping, indicating that Mn is a magnetic impurity, while Cu behaves as a nonmagnetic or very weak magnetic impurity. To further investigate the magnetic moments induced by Cu and Mn dopants, we assume that the low temperature magnetization can be written as the Curie–Weiss law

$$\chi = M/H = \chi_0 + C_0/(T + T_0),$$

(1)

where $C_0 = \mu_0 \mu_{\text{eff}}^2 / 3k_B$, $\chi_0$ and $T_0$ are the fitting parameters, and $\mu_{\text{eff}}$ is the local magnetic moment per Fe site. The first term, $\chi_0$, comes from the Pauli paramagnetism of the conduction electrons, which is related to the density of states (DOS) at the Fermi energy. The second term, $C_0/(T + T_0)$, is given by the local magnetic moments of the ions at the Fe sites (including dopants like Co, Cu, and Mn). The fitting process is not straightforward; for a precise evaluation of the local magnetic moments, we adjust the $\chi_0$ value to get a linear function of $1/(\chi - \chi_0)$ versus $T$ in the low temperature limit. Then we fit the data with a linear function, as shown in figure 8. The slope of the linear line gives $1/C_0$, and the intercept delivers the value of $T_0/C_0$. Once $C_0$ is obtained, we can get the average magnetic moment of a single Fe site (including the contribution of Fe and the dopants). The results are shown in figure 7(c). Clearly, doping with Mn induces strong local magnetic moments, especially when $x$ exceeds 0.35, while doping with Cu seems to even weaken the average local moments. These facts suggest that Mn ions here play the role of magnetic impurities, while Cu dopants act as nonmagnetic or weak magnetic impurities. One possible picture in which to interpret this is that the Cu dopant may have a full shell of the $d^{10}$ configuration with the ionic state of Cu$^{1+}$ as predicted by the theoretical calculations [40, 41]. Similar results are observed for Cu-doped Fe$_{1+y}$Te$_{0.6}$Se$_{0.4}$ [31].
3.3. Discussion

The residual resistivity of Na(Fe$_{0.97-x}$Co$_{0.03}$)$_2$As ($T = $ Cu, Mn) plotted as a function of doping concentration $x$ is shown in figure 9. One can see that both doping with Cu and doping with Mn can enhance the residual resistivity. In the low doping region ($x = 1\%, 2\%, 3\%)$, the residual resistivity increases with the same ratio, 0.18 m\(\Omega\) cm/%, for the Cu-doped and the Mn-doped samples. However, the residual resistivity increases rapidly for highly Mn-doped samples. Surprisingly, superconductivity still exists even up to a residual resistivity of 2.86 m\(\Omega\) cm for the case of Mn doping. A similar phenomenon is observed for Co-doped Fe$_{1+y}$Te$_{0.6}$Se$_{0.4}$ superconductor [31], where superconductivity is maintained even up to a residual resistivity of 6 m\(\Omega\) cm. We must emphasize that the Mn elements have been doped into the Fe sites without doubt, because the lattice constant changes monotonically, and the high residual resistivity
Figure 8. Temperature dependence of $1/(\chi - \chi_0)$ for Na(Fe$_{0.97}$Co$_{0.03}$)As and Na(Fe$_{0.97-x}$Co$_{0.03}$T$_x$)As ($T = \text{Cu and Mn}$) single crystals under 1 T with $\chi$ the DC magnetic susceptibility. By adjusting $\chi_0$ we obtain a linear relation of $1/(\chi - \chi_0)$ with the temperature in the low temperature limit. The red lines represent the linear fits of the data. The slope gives $1/C_0$ and the intercept provides the value of $T_0/C_0$.

Figure 9. Residual resistivity of Na(Fe$_{0.97-x}$Co$_{0.03}$T$_x$)As ($T = \text{Cu, Mn}$) plotted as a function of the doping concentration $x$. 
increases monotonically with the doping level of Mn up to 4%. For a simple $S^\pm$ pairing model, this is very difficult to understand. A further in-depth understanding is highly desired.

It seems that both Cu and Mn act as charge donors. In this case, one may argue that the suppression of $T_c$ may result from the change of the charge carrier density. However, we have reason to believe that the $T_c$ suppression of the Na(Fe$_{0.97-x}$Co$_{0.03}$T$_x$)As (T = Cu, Mn) system is due to impurity scattering. If Cu behaves as an electron dopant, the carrier density will be increased, and result in a decrease of the residual resistivity. Our results showed that the residual resistivity increases rapidly with increase of the doping content for both Cu- and Mn-doped samples. So it is reasonable to say that Cu behaves more like random scatterers than an electron dopant. And we believe that such a rapid increase of the residual resistivity in Mn-doped samples is mainly attributable to impurity scattering. Plenty of studies on some other iron-based superconductors have revealed that Cu 3d states do not provide additional free carriers to the Fermi level [31, 41, 42]. Similarly, it is proved that in Mn-doped BaFe$_2$As$_2$, Mn dopants act as magnetic impurities rather than hole donors [43].

In the above, we have discussed the influence of doping with Cu and with Mn on the lattice parameter, magnetization and resistivity. We have also discovered that Mn doping gives rise to a continuous increase of the local magnetic moments, and that the higher dopings with Mn can especially enhance the local magnetic moments greatly, whereas Cu behaves as a nonmagnetic or very weakly magnetic impurity. On the basis of these results, in the following, we focus on the discussion of the pair breaking mechanism for Na(Fe$_{0.97-x}$Co$_{0.03}$T$_x$)As (T = Cu, Mn) superconductors. According to the $S^\pm$ scenario with equal gaps of opposite signs on different Fermi surfaces, $T_c$ is expected to be markedly suppressed due to the potential scattering by substituted nonmagnetic impurities, and it obeys a universal Abrikosov–Gor'kov formula [5], $-\ln t = \psi \left(1/2 + \alpha/2t\right) - \psi \left(1/2\right)$, where $t = T_c/T_{c0}$ with $T_{c0}$ and $T_c$ the transition temperatures of the pristine and the doped samples respectively, $\psi (x)$ the digamma function, and $\alpha$ the pair breaking parameter [44]. The Abrikosov–Gor’kov formula would lead to $T_c$ vanishing at $\alpha_{\text{theory}} = 0.28$. However, plenty of previous studies of the impurity effect on the iron-based superconductor have revealed that the critical value of $\alpha$ is much larger than 0.28, which means that the rate of $T_c$ suppression found previously is too small to be explained by the $S^\pm$ scenario [22–25, 27, 33]. According to the theoretical calculations based on the five-orbital model [11], $\alpha = z\hbar\Gamma /2\pi k_B T_{c0}$, where $z = m/m^*$ is the renormalization factor and $\Gamma$ is the scattering rate. We use the relation $\Gamma = ne^2 \Delta \rho_{0}/m^* = e \Delta \rho_{0}/m^* R_H$, where $n$ is the carrier density, $R_H$ is the Hall coefficient, and $m^*$ is the effective mass. In this study, we use $z = 1/4.2$ obtained from optical spectroscopy experiments for Na-111 superconductor [45, 46], and $R_H = -7 \times 10^{-9}$ m$^3$/C is obtained from the transport measurements by extrapolating $R_H$ data to zero temperature, which is consistent with the result reported before [39]. The critical value of $\alpha$ obtained, for $T_c$ to vanish, based on our experimental data for Cu-doped samples, is about 0.64, which is still much larger than the theoretically expected value $\alpha_{\text{theory}} = 0.28$, but the gap between the experimental and the theoretical values becomes much smaller compared with the case for previous studies on the Ba-122 system [23–25].

To further study the pair breaking mechanism in the Na(Fe$_{0.97-x}$Co$_{0.03}$T$_x$)As (T = Cu, Mn) system, we calculated the critical residual resistivity for $T_c$ to vanish on the basis of the $S^\pm$
scenario; it is proportional to the pair breaking parameter $\alpha$. The relationship between $T_c$ and $\alpha$ can be transformed into a relationship between $T_c$ and the residual resistivity by using the relation 

$$ \rho_c = 2\pi k_B T_c / \rho_0 + \rho_0^\text{pri} $$

where $\rho_0^\text{pri}$ represents the residual resistivity of the pristine sample. As shown in figure 10, $T_c$ was plotted as a function of $\rho_0$. The dashed line represents the relationship between $T_c$ and the residual resistivity based on the $S^\pm$ scenario, which follows a universal Abrikosov–Gor’kov formula. One can see that the critical residual resistivity for $T_c$ to vanish in the $S^\pm$ model is 0.44 m$\Omega$ cm. As we can see, in the low doping region, our data can be roughly fitted to this model, which means that the impurity effect in Na(Fe$_{0.97-x}$Co$_{0.03}$)As induced either by Mn or by Cu can be explained by the $S^\pm$ scenario in the low doping regime. In the case of Mn doping, the suppression of $T_c$ gets much weaker beyond $x = 0.03$ and the superconductivity is retained even up to a residual resistivity of 2.86 m$\Omega$ cm, indicating that the magnetic Mn impurities are even not as detrimental as the nonmagnetic Cu impurities to the superconductivity in the high doping region. This is an interesting observation; further theoretical and experimental efforts are expected to examine why the superconductivity can be so robust under Mn doping.

4. Conclusion

In summary, we studied the impurity effect in single crystals of Na(Fe$_{0.97-x}$Co$_{0.03}$)As ($T = \text{Cu, Mn}$). Analysis of the DC magnetization based on the Curie–Weiss law indicates that Mn doping gives magnetic impurities, whereas Cu dopants behave as nonmagnetic or very weak magnetic impurities. However, it is found that both doping with Cu and doping with Mn can enhance the residual resistivity and suppress the superconductivity, at the same rate, in the low doping region, which is consistent with the prediction of the $S^\pm$ model. For the Cu-doped system, the superconductivity is suppressed completely at a residual resistivity of 0.87 m$\Omega$ cm, when a
strong localization effect is observed. However, in the case of Mn doping, the suppression of $T_c$ gets much weaker beyond $x = 0.03$ and superconductivity survives even up to a residual resistivity of $2.86 \text{ m} \Omega \text{ cm}$. Clearly the magnetic Mn impurities are even not as detrimental as the nonmagnetic Cu impurities to the superconductivity in the Na(Fe$_{0.97}$Co$_{0.03}$)As system in the high doping regime.

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