Efficient charge carriers induced by extra outer-shell electrons in iron-pnictides: a comparison between Ni- and Co-doped CaFeAsF

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Abstract. A comprehensive study of the difference between CaFe1−xNi,xAsF and CaFe1−xCo,xAsF systems has been carried out by measuring the efficient charge carrier concentration, the valence states and the superconducting phase diagram. It is found that at the same doping level, Ni doping introduces nearly twice the number of charge carriers as Co doping. However, x-ray absorption near-edge spectroscopy measurements reveal that the valence state of Fe in both systems is close to 2, indicating that there is no valence mismatch. We suggest that the charge carriers in CaFe1−xM,xAsF (M = transition metal elements) are not induced by valence mismatch but come from the difference in the number of outer-shell electrons. We also suggest that with Ni and Co doping, the systems change from a multi-band material in the underdoped regions to a single-band state in the overdoped regions.

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1. Introduction

The high-temperature superconducting mechanism has become one of the most mysterious subjects in condensed matter physics. After 24 years since the discovery of high-temperature cuprate superconductors [1], the fundamental understanding of the physical properties of cuprates is still controversial. The discovery of high-temperature superconductivity in iron-pnictides has provided a new chapter in superconductivity research [2]–[4]. The superconductivity community is excited to have this new class of materials, because the comparison between the two classes of superconducting materials may help in solving the question of the high-temperature superconducting mechanism.

Unlike the Mott-insulating state of the undoped cuprate superconductors, the parent iron-pnictide compounds exhibit metallic or semiconducting behavior [5, 6]. It is argued that the electronic correlations are weak, leading to a small Coulomb repulsion $U$ in iron-pnictides compared to the large Coulomb repulsion in cuprates [7]. Despite these differences, there are many similarities between the two systems. Like the cuprates, the iron-pnictides are layered, containing planes of iron and arsenic interleaved with planes of oxygen (fluorine) and rare-earth elements (or alkaline-earth metals and alkali metals). Similar to the so-called ‘conducting layer’ of the CuO$_2$ plane in cuprates, the FeAs plane takes the role of a ‘conducting layer’ in iron-pnictides, with the other part remaining in the ‘charge reservoir layer’. The superconductivity emerges when one ‘dopes’ certain non-isovalent impurities in the charge reservoir layer, e.g. either electron-type doping (LaFeAsO$_{1−x}$F$_x$) or hole-type doping (La$_{1−x}$Sr$_x$FeAsO) in the LaFeAsO system [2, 8], and hole-type doping (Ba$_{1−x}$K$_x$Fe$_2$As$_2$) in the BaFe$_2$As$_2$ system [3].

With increasing impurity content, the superconducting phase diagram undergoes evolution from the underdoped region to the optimally doped region to the overdoped region. Eventually, the system becomes ordinary metal with an even higher doping content. The similarity between the cuprates and iron-pnictides seems to suggest that a valence mismatch is important in producing the efficient charge carriers that are necessary for both the conductivity and the superconductivity.

However, it is a surprise to find the superconductivity when a certain amount of iron in iron-pnictides is replaced by cobalt or nickel, atoms of almost the same valence state but with one or two extra outer-shell electrons [4], [9]–[12]. In this case, the efficient charge carriers are apparently not coming from the valence mismatch as in cuprates. Up to now, how the efficient charge carriers can be induced into the FeAs conducting layer has remained an open question. In this paper, we perform a systematic study of the transport and other properties of the CaFe$_{1−x}$Ni$_x$AsF and CaFe$_{1−x}$Co$_x$AsF systems. We confirm that the substitution of Ni and Co at the Fe site does not lead to any valence evolution of Fe. Meanwhile, efficient charge carriers...
are substantially introduced into the FeAs conducting layers of the systems. We suggest that the charge carriers in CaFe$_{1-x}$M$_x$AsF ($M =$ transition metal elements) are not induced by any valence mismatch. The extra outer-shell electrons of Ni or Co compared to Fe play an important role in providing the charge carriers in the CaFe$_{1-x}$Ni$_x$AsF and CaFe$_{1-x}$Co$_x$AsF systems.

2. Experiment

Polycrystalline samples with nominal compositions CaFe$_{1-x}$Ni$_x$AsF and CaFe$_{1-x}$Co$_x$AsF were prepared by the conventional solid-state reaction method using high-purity Ca$_3$As$_2$, Fe$_3$As$_2$, Ni$_3$As$_2$, Co$_3$As$_2$ and CaF$_2$ (powder, 99.999%, Alfa-Aesar) as starting materials. Ca$_3$As$_2$ was synthesized by heating Ca (granules, redistilled, 99.5%, Alfa-Aesar) with As (powder, 99.99%, metals basis, Alfa-Aesar) at 600°C for 10 h in an evacuated silica tube. Fe$_3$As$_2$, Ni$_3$As$_2$ and Co$_3$As$_2$ were prepared from powders of mixed elements at 750°C for 6 h. The raw materials were thoroughly ground and pressed into pellets. The pellets were wrapped up in Ta foil and sealed in an evacuated quartz tube. They were pre-heated at 1000°C for 10 h and cooled slowly to room temperature. The pellets were ground again and pressed into new pellets and heated at 1000°C for 12 h.

X-ray diffraction (XRD) was carried out by a Rigaku-D/max-gA diffractometer using high-intensity Cu-Kα radiation to screen for the presence of an impurity phase and changes in structure. The lattice parameters were determined from the $d$ value of XRD peaks by a standard least-squares refinement method. The resistivity was measured using a standard four-probe method in a closed-cycle helium cryostat. The magnetic susceptibility of the samples was determined by a superconductivity quantum interference device (SQUID) magnetometer (Quantum Design, MPMS). The applied magnetic field was 10 Oe with both zero-field cooling and field cooling. The Hall coefficient was measured by the four-lead method in a magnetic field of up to 7 T, using a permanent magnet. The Hall coefficient was measured at selected temperatures from 2 to 300 K. The Fe K-edge x-ray absorption near-edge structure (XANES) spectra of the powder samples were measured at the U7C beamline of the National Synchrotron Radiation Laboratory (NSRL), China. The XANES signals were collected in fluorescence mode with a seven-element high-purity Ge solid detector.

3. Results and discussion

Figure 1 shows the powder XRD patterns of the CaFe$_{1-x}$Ni$_x$AsF and CaFe$_{1-x}$Co$_x$AsF samples. The XRD results reveal the formation of nearly single-phase products for samples with 0 $\leq$ $x$ $\leq$ 0.25. The minor impurity peaks, which reflect very small amounts of unreacted impurities, such as CaF$_2$ and Fe$_2$As, are marked in figure 1. It is clear that all the main peaks can be indexed to the FeAs-1111 phase with the tetragonal ZrCuSiAs-type structure$^4$. There is no macroscopic structure phase transition with Fe-site doping. By having a closer look, one can find that the diffraction peaks shift slightly to higher angles with increasing Ni or Co doping content, suggesting a shrinkage of the lattice constants. By using the software of the XRD program, we take a general fit to the XRD data of each sample and determine the lattice constants. The doping dependence of the lattice constants calculated from the XRD data for several representative samples is listed in table 1. The monotonous shrinkage of both the $a$-axis and the $c$-axis with

$^4$ JCPDS-International Centre for Diffraction Data 1990, 40-1100.
Figure 1. Powder XRD patterns of polycrystalline samples CaFe$_{1-x}$Ni$_x$AsF and CaFe$_{1-x}$Co$_x$AsF.

Table 1. Unit cell parameters of CaFe$_{1-x}$Ni$_x$AsF and CaFe$_{1-x}$Co$_x$AsF samples.

| Sample composition    | $a$ (Å) | $c$ (Å) |
|-----------------------|---------|---------|
| CaFeAsF               | 3.8950(2) | 8.5006(2) |
| CaFe$_{1-x}$Ni$_x$AsF, $x = 0.05$ | 3.8913(2) | 8.4933(5) |
| CaFe$_{1-x}$Ni$_x$AsF, $x = 0.10$ | 3.8907(1) | 8.4878(3) |
| CaFe$_{1-x}$Ni$_x$AsF, $x = 0.15$ | 3.8891(2) | 8.4818(4) |
| CaFe$_{1-x}$Ni$_x$AsF, $x = 0.20$ | 3.8867(8) | 8.4725(3) |
| CaFe$_{1-x}$Ni$_x$AsF, $x = 0.25$ | 3.8863(3) | 8.4685(6) |
| CaFe$_{1-x}$Co$_x$AsF, $x = 0.10$ | 3.8911(6) | 8.4843(1) |
| CaFe$_{1-x}$Co$_x$AsF, $x = 0.20$ | 3.8883(4) | 8.4715(5) |

increasing Ni- or Co-doping content suggests that the Ni and Co atoms have substantially gone into the crystal lattice of the CaFeAsF system.

The temperature dependence of the electrical resistivity of the CaFe$_{1-x}$Ni$_x$AsF and CaFe$_{1-x}$Co$_x$AsF samples is plotted in figures 2(a) and (b), respectively. From figure 2 it can be seen that the resistivity of CaFe$_{1-x}$Ni$_x$AsF (and CaFe$_{1-x}$Co$_x$AsF) decreases monotonously with increasing Ni (and Co)-doping content, indicating that the effective charge carriers are introduced into the Fe(Ni, Co)As conducting plane. Similar to previous reports [4], there is an anomalous decrease in resistivity in the parent compound CaFeAsF below $T^* \sim 120$ K, which is associated with the spin wave density (SDW) transition. The resistivity anomaly is shifted to low temperature with increasing Ni-doping content. For example, $T^*$ is about 100 K for $x = 0.01$ and about 85 K for $x = 0.02$. Besides the low-temperature shifting of $T^*$, the magnitude of the decrease in resistivity below $T^*$ is significantly weakened with Ni doping, consistent with the suppression of the SDW transition. On further increasing Ni-doping.
content, the anomaly in resistivity disappears. For \( x \geq 0.03 \) samples, the samples exhibit typical semiconducting conduction behavior at low temperature. For \( x = 0.045 \), a superconducting transition occurs below \( T_c^{\text{onset}} \) (defined as the onset temperature point in resistive transition) at 6.9 K, but zero resistivity cannot be reached even for temperatures as low as 4 K. At the optimal doping level \( x = 0.09 \), \( T_c^{\text{onset}} \) reaches a maximum of 23.1 K, and \( T_c^{\text{zero}} \) is about 13.3 K. The relatively large transition width \( \Delta T_c \) of 9.8 K is due to the grain boundary effects in the polycrystalline samples. For the CaFe\(_{1-x}\)Co\(_x\)AsF system, the resistivity anomaly also shifts to low temperature with increasing Co-doping content, indicating the destruction of the SDW transition by Co doping. However, it is apparent that the suppression of SDW in the Co-doped system is much slower than that in the Ni-doped system. Superconductivity appears when \( x \geq 0.06 \). The optimal doping level in the CaFe\(_{1-x}\)Co\(_x\)AsF system is \( x = 0.12 \), where \( T_c^{\text{onset}} \) is 27.3 K and \( T_c^{\text{zero}} \) is about 19.1 K. Figure 3 shows the temperature dependence of the \( dc \) magnetic susceptibility of CaFe\(_{1-x}\)Ni\(_x\)AsF near the transition temperature. For each sample, the superconducting transition temperature determined from the magnetic susceptibility is consistent with that determined from the resistivity measurement. In the inset of figure 3, we give the magnetic susceptibility below 300 K. For the parent compound CaFeAsF, the sharp decrease in magnetic susceptibility is due to the SDW transition. It can be seen that a very small amount of Ni doping leads to dramatic suppression of the SDW transition, which is consistent with the resistivity measurements.

In order to see the difference between the Ni-doped case and the Co-doped case, we plot the superconducting phase diagram of the CaFe\(_{1-x}\)Ni\(_x\)AsF and CaFe\(_{1-x}\)Co\(_x\)AsF systems in figure 4. In detail, superconductivity emerges in the CaFe\(_{1-x}\)Ni\(_x\)AsF system at \( x_c = 0.045 \). The maximum \( T_c^{\text{mid}} \) value of about 13.9 K is achieved at \( x_m = 0.09 \) (here \( T_c^{\text{mid}} \) is defined as the middle point in the superconducting transition). The superconductivity disappears at \( x_d \geq 0.16 \). For the CaFe\(_{1-x}\)Co\(_x\)AsF system, the superconductivity emerges at \( x_c = 0.06 \).
Figure 3. Temperature dependence of the magnetic susceptibility of CaFe$_{1-x}$Ni$_x$AsF samples under field-cooled conditions with 10 G.

Figure 4. Superconducting phase diagram of CaFe$_{1-x}$Ni$_x$AsF and CaFe$_{1-x}$Co$_x$AsF systems.

The maximum $T_{c}^\text{mid}$ value of about 22.6 K is achieved at $x_m = 0.12$. The superconductivity disappears at $x_d \geq 0.28$. From the phase diagram, two distinct differences are given between the CaFe$_{1-x}$Ni$_x$AsF and CaFe$_{1-x}$Co$_x$AsF systems. One is that the superconductivity appears faster in the CaFe$_{1-x}$Ni$_x$AsF system compared to the CaFe$_{1-x}$Co$_x$AsF system. The other is that the optimal doping level is lower in the CaFe$_{1-x}$Ni$_x$AsF system. The superconducting region in the phase diagram is much broader in the CaFe$_{1-x}$Co$_x$AsF system. These facts seem to suggest that Ni doping is more effective in introducing charge carriers than Co doping.
In order to obtain more information about the conducting carriers, we measured the Hall effect of the CaFe\(_{1-x}\)Ni\(_x\)AsF and CaFe\(_{1-x}\)Co\(_x\)AsF samples. Figure 5(a) shows the magnetic field dependence of Hall resistivity for the CaFeAsF parent compound at different temperatures. In the experiment, \(\rho_{xy}\) was taken as \(\rho_{xy} = (\rho(+H) - \rho(-H))/2\) at each point to eliminate the effect of the misaligned Hall electrodes. It is clear that all curves in figure 5(a) have good linearity versus the magnetic field. Moreover, \(\rho_{xy}\) data are linear with a negative slope in \(H \leq 7\) T, giving a negative Hall coefficient \(R_H = \rho_{xy}/H\), which indicates the existence of dominant electron-type charge carriers. Figure 5(b) gives the temperature dependence of the Hall coefficient for the CaFe\(_{1-x}\)Ni\(_x\)AsF samples. For the parent compound CaFeAsF, with decreasing temperature, \(|R_H|\) jumps to higher values at \(T^*\), indicating the development of an...
SDW gap in the Fermi surface, which leads to a reduction in carriers. However, we do not observe the change in the carrier type below \( T^* \), as previously reported [13]. With increasing Ni content, \(|R_{\text{H}}|\) exhibits a monotonous decrease while keeping its sign negative, indicating that electron-type effective charge carriers are introduced in the CaFe\(_{1-x}\)Ni\(_x\)AsF samples. For \( x \leq 0.08 \), \( R_{\text{H}} \) is strongly temperature dependent, especially at low temperature. It is known that for a conventional metal with Fermi-liquid features, the Hall coefficient is almost independent of temperature. The strongly temperature-dependent \( R_{\text{H}} \) in \( x \leq 0.08 \) samples suggests that these samples have multi-band behavior [14], similar to cuprate superconductors [15]. For samples with \( x > 0.08 \), \( R_{\text{H}} \) is almost temperature independent, suggesting that these samples exhibit a more simple single-band feature. This fact actually indicates that with Ni doping the system changes from a multi-band material in the underdoped regions to a single-band state in the overdoped regions.

In figure 5(c), we compare the temperature dependence of the Hall coefficient between the CaFe\(_{1-x}\)Ni\(_x\)AsF and CaFe\(_{1-x}\)Co\(_x\)AsF systems at several representative doping levels. It is obvious that there is an increase in \(|R_{\text{H}}|\) in the CaFe\(_{0.90}\)Co\(_{0.10}\)AsF sample with decreasing temperature, especially when \( T \) is below 50 K. In the CaFe\(_{0.88}\)Co\(_{0.20}\)AsF sample, the Hall resistivity is nearly temperature independent. Similar to the Ni-doped system, the CaFe\(_{1-x}\)Co\(_x\)AsF system also involves an evolution from a multi-band material in the underdoped regions to a single-band state in the overdoped regions. From figure 5(c), we notice a very interesting fact. That is, at a certain Ni doping content \( x \), the efficient charge carrier concentration in the CaFe\(_{1-x}\)Ni\(_x\)AsF sample is almost the same as that in the CaFe\(_{1-2x}\)Co\(_{2x}\)AsF sample. In other words, one Ni ion can provide twice the amount of effective charge carrier into the FeAs conducting layer as two Co ions provide.

The Hall resistivity results suggest that at the same doping content, Ni doping can introduce nearly twice the number of charge carriers as Co doping introduces. To learn whether the Ni and Co doping induce any valence mismatch in the FeAs-conducting layer, we determine the valence state of Fe in the two systems by performing XANES measurements. The Fe K-edge XANES spectra for the CaFe\(_{1-x}\)Ni\(_x\)AsF and CaFe\(_{1-x}\)Co\(_x\)AsF samples, together with the Fe K-edge XANES spectra for reference samples FeO and Fe\(_2\)O\(_3\), are shown in figure 6. The main absorption peak located at about 7120 eV corresponds to the excitation of 1s core level electrons to the 4p unoccupied orbital of the absorption atoms. In a tetrahedral crystal symmetry, the pre-edge peak at about 7110 eV is due to the excitation of 1s electrons to the unoccupied 3d bands. In the FeO and Fe\(_2\)O\(_3\) samples, the 1s to 3d excitation is much weakened compared to the tetrahedral CaFeAsF system. In determining the valence state, the absorption threshold is defined as the first inflection point of the pre-edge peak (1s to 3d excitation). In order to see the threshold distinctly, we plot in the inset of figure 6 the pre-edge structure of the samples. One can see that the absorption threshold (marked by the arrows) for the parent compound CaFeAsF is located at 7109.76 eV, which is almost the same as that of the FeO reference sample, indicating that the valence state of Fe in CaFeAsF is close to +2. It can be seen that the Fe K-edge XANES features of the CaFe\(_{1-x}\)Ni\(_x\)AsF and CaFe\(_{1-x}\)Co\(_x\)AsF samples are all similar to that of the CaFeAsF parent compound, which means that the doping of Ni does not lead to any structural changes or valence variation in Fe. Combining the results taken from the Hall coefficient and XANES measurements, we suggest that charge carriers in the CaFe\(_{1-x}\)Ni\(_x\)AsF and CaFe\(_{1-x}\)Co\(_x\)AsF systems are introduced, but not from any valence mismatch.

In cuprate superconductors, a valence mismatch between the so-called ‘charge reservoir layer’ and the ‘conducting layer’ is necessary in transferring effective charge carriers from the...
Figure 6. XANES spectra at the Fe K-edge of CaFe$_{1-x}$Ni$_x$AsF and CaFe$_{1-x}$Co$_x$AsF samples, measured at 300 K. The absorption edge, defined as the first inflection point, is indicated by arrows.

‘charge reservoir layer’ to the CuO$_2$ conductive plane. In iron-pnictide superconductors, initial research seems to support the fact that the iron-pnictides show similar behavior to that of the cuprates. For example, electron-type charge carriers or hole-type charge carriers can be introduced into the FeAs-conducting layers by F doping or Sr doping in the RE-O ‘charge reservoir layer’ in REFeAsO systems. In either case, a valence mismatch between the ‘charge reservoir layer’ and the ‘conducting layer’ is established due to anisovalent doping in the ‘charge reservoir layer’. With the increase in charge carrier concentration, both the cuprates and the iron-pnictides evolve from the low-temperature antiferromagnetic state into the underdoped superconducting state. With further increasing charge carriers, the systems change into the overdoped superconducting region. Eventually, Fermi-liquid normal metal behavior appears. However, the superconducting behavior in the Co- and Ni-doped iron-pnictides is very different from the cuprates, because neither Co doping nor Ni doping induce any valence mismatch in the
systems. It is also well known that in cuprate superconductors a very small amount of Co- or Ni doping in the CuO$_2$ conducting plane leads to dramatic depression of the superconductivity. In cuprates, the Co and Ni ions are generally considered as random impurities, magnetic scattering centers and Cooper-pair breakers. In contrast, the Co and Ni ions take an absolutely different role in iron-pnictides from that in cuprates. In the CaFe$_{1-x}$Ni$_x$AsF and CaFe$_{1-x}$Co$_x$AsF systems, partial substitution of Fe with Co or Ni results in the production of efficient charge carriers into the Fe(Co,Ni)As conducting layers, which are favorable to superconductivity. Since the Co and Ni dopants do not lead to any valence mismatch, it is most likely that the efficient charge carriers induced by Co and Ni doping are due to the difference in the number of outer-shell 3d electrons between Fe ions and Co(Ni) ions. That is, an Fe$^{2+}$ ion has a 3d$^6$ configuration in its outer shell, while it is 3d$^7$ and 3d$^8$ in Co and Ni, respectively. In the iron-pnictide parent compounds, the ground state is theoretically predicted and experimentally proved to be antiferromagnetic at low temperature \cite{5}, \cite{16}–\cite{18}. At a macroscopic length scale, the sum of the magnetic moment contributed by all the Fe$^{2+}$ ions is close to zero. The 3d$^6$ electrons lose their itinerancy. When the Fe sites are partially replaced by Co or Ni ions, these dopants introduce one or two ‘extra’ outer-shell electrons. These ‘extra’ outer-shell electrons, which have 3d$^7$ and 3d$^8$ configurations, destroy the antiferromagnetic correlations and make the system more or less paramagnetic-like. At the same time, it is actually these ‘extra’ electrons that act as the itinerant charge carriers. In this way, the doping of Co and Ni increases the density of mobile electrons in the conducting plane. That the efficient charge carriers come from the ‘extra’ electrons in the 3d$^7$ and 3d$^8$ configurations compared to the 3d$^6$ configuration is supported by the fact that, at the same doping level, the doping of Ni can introduce nearly twice the number of charge carriers as the doping of Co introduces. That is, the Ni ion, which has a 3d$^8$ configuration, carries two ‘extra’ electrons compared with the Fe ion, while the Co ion carries only one ‘extra’ electron compared with Fe.

4. Conclusion

In summary, we have established and compared the superconducting phase diagram of the CaFe$_{1-x}$Ni$_x$AsF and CaFe$_{1-x}$Co$_x$AsF systems. It is found that Ni doping is more effective in introducing mobile charge carriers compared to Co doping. We suggest that the introduction of charge carriers is not due to any valence mismatch like that in cuprate superconductors. In iron-pnictide systems, the loosely bound electrons in the outer shell of the transition metals play an important role in providing efficient charge carriers into the FeAs-conducting layer.

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