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Effect of Zn doping on magnetic order and superconductivity in LaFeAsO

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Abstract. We report Zn-doping effect in the parent and F-doped LaFeAsO oxy-arsenides. Slight Zn doping in $LaFe_{1-x}Zn_xAsO$ drastically suppresses the resistivity anomaly around 150 K associated with the antiferromagnetic (AFM) spin density wave (SDW) in the parent compound. The measurements of magnetic susceptibility and thermopower confirm further the effect of Zn doping on AFM order. Meanwhile Zn doping does not affect or even enhances the T_c of $LaFe_{1-x}Zn_xAsO_{0.9}F_{0.1}$, in contrast to the effect of Zn doping in high-T_c cuprates. We found that the solubility of Zn content (x) is limited to less than 0.1 in both systems and further Zn doping (i.e. $x \geqslant 0.1$) causes phase separation. Our study clearly indicates that the non-magnetic impurity of Zn^{2+} ions doped in the Fe_2As_2 layers affects selectively the AFM order and superconductivity remains robust against the Zn doping in the F-doped superconductors.

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Contents

1. Introduction

Since the discovery of superconductivity at 26 K in 1111 phase $LaFeAsO_{1-x}F_x$ [1], T_c has been raised up to 56 K [2]–[5] quickly in a series of doped oxy-arsenides. Although a BCS-like superconducting gap has been observed by the studies of Andreev spectroscopy [6] and angle-resolved photoemission spectroscopy (ARPES) [7, 8], theoretical calculations have excluded a conventional pairing mechanism based on electron–phonon interaction [9]. Thus various theoretical models have been proposed [10]–[17]. The evolvement of electronic state with charge carrier doping indicates that superconductivity occurs on the verge of antiferromagnetic (AFM) ordering state (for example see [18]), suggesting similarities with high-T_c cuprates.

Elemental substitution is a very useful approach to explore new superconducting materials and to elucidate the intrinsic factors which determine T_c in unconventional superconductors. In the case of high-T_c cuprates, the partial substitution of Cu with other 3d elements such as Ni and Zn in CuO_2 planes severely destroys superconductivity [19, 20]. However, the band structure calculations [21]–[23] and theoretical analysis [24] reveal the itinerant character of Fe 3d electrons in the iron-based oxy-arsenides, in comparison with the localized nature of Cu 3d electrons in cuprates. Therefore different behavior could be expected when Fe is partially substituted by other 3d transition metal elements. Indeed superconductivity has been observed in both 1111 phase and 122 phase by doping magnetic elements Co [25, 26] or Ni [27]. Although the previous report has discovered that LaCoAsO is a ferromagnetic metal with a Curie temperature $T_c = 66$ K [28], which suggests that Co^{2+} ions should be magnetic in this structure, we found that the Co 3d electrons are basically itinerant in $LnFe_{1-x}Co_xAsO$ system (Ln: lanthanides, $x \leqslant 0.3$) [25]. These studies have revealed that there are essentially different doping mechanisms between iron-based arsenides and cuprates and suggested a fundamental difference between the two classes of high-temperature superconductivity. The effect of non-magnetic Zn doping in the iron-based arsenides has not been reported yet. In high-T_c cuprates, Zn doping not only suppresses superconductivity severely but also induces local magnetic moments and other anomalous properties [29]–[31]. Therefore it is a very interesting issue to address.

In this paper, we report the investigation on the doping effect of the non-magnetic element Zn in the parent and F-doped LaFeAsO systems. We found that even slight Zn doping in $LaFe_{1-x}Zn_xAsO$ drastically suppresses the resistivity anomaly around 150 K, which is associated with formation of AFM spin density wave (SDW) in the undoped parent compound [32]. Measurements of magnetic susceptibility and thermopower provide further

evidence for the suppression of SDW order. Meanwhile slight Zn doping has little effect on superconductivity in $LaFe_{1-x}Zn_xAsO_{0.9}F_{0.1}$, in contrast to the effect of Zn doping in high-T_c cuprates. Our result implies that the Zn doping selectively destroys the SDW order rather than Cooper pairs.

2. Experimental

The polycrystalline $LaFe_{1-x}Zn_xAsO$ and $LaFe_{1-x}Zn_xAsO_{0.9}F_{0.1}$ samples were synthesized by solid state reaction in vacuum using powders of LaAs, La_2O_3, FeAs, Fe_2As, LaF_3 and ZnO. LaAs was presynthesized by reacting stoichiometric La pieces and As powders in evacuated quartz tubes at 1223 K for 24 h. FeAs and Fe_2As were prepared by reacting stoichiometric Fe powders and As powders at 873 K for 10 h, respectively. The powders of these intermediate materials were weighed according to the stoichiometric ratio of $LaFe_{1-x}Zn_xAsO$ ($x = 0$, 0.02, 0.05, 0.10 and 0.15) and $LaFe_{1-x}Zn_xAsO_{0.9}F_{0.1}$ ($x = 0$, 0.02, 0.05, 0.1 and 0.15), respectively and then thoroughly mixed in an agate mortar. The mixtures were pressed into pellets under a pressure of 2000 kg cm^{-2}. All the processes were operated in a glove box filled with high-purity argon. The pellets were sealed in evacuated quartz tubes and heated uniformly at 1433 K for 40 h.

Powder x-ray diffraction (XRD) was performed at room temperature using a D/Max-rA diffractometer with Cu K_α radiation and a graphite monochromator. The XRD diffractometer system was calibrated using standard Si powders. Lattice parameters were refined by a least-squares fit using at least 20 XRD peaks.

The electrical resistivity was measured by a standard four-terminal method. The samples for transport property measurements were cut into a thin bar with typical size of 4 mm × 2 mm × 0.5 mm. The size of the contact pads leads to a total uncertainty in the absolute values of resistivity of 10%. Thermopower was measured by using a steady-state technique. The temperature dependence of dc magnetic susceptibility was measured on a Quantum Design magnetic property measurement system (MPMS-5). The applied magnetic field was 1000 Oe for the non-superconducting $LaFe_{1-x}Zn_xAsO$, and 10 Oe for the superconducting $LaFe_{1-x}Zn_xAsO_{0.9}F_{0.1}$. The superconducting samples used for magnetic measurements were made in roughly cubic shape so that a shape demagnetizing factor (N) of about 1/3 was used to calculate the demagnetizing effect in the superconducting state.

3. Results and discussion

3.1. Effect of Zn doping in $LaFe_{1-x}Zn_xAsO$

Figure 1 shows the XRD patterns of $LaFe_{1-x}Zn_xAsO$ samples and the variations of the lattice constants with Zn content. For the parent compound and slightly Zn-doped compounds ($x \leqslant 0.05$), the XRD peaks can be well indexed based on a tetragonal cell with the space group of P4/nmm, which indicates that the samples are essentially single phase. With further increasing Zn content, peak splitting can be observed for the peaks (102), (112) and (200), which indicates that there is a possible phase separation for $x \geqslant 0.10$. Since the lattice constants of LaZnAsO are quite a bit larger than those of LaFeAsO [33], a phase separation of LaZnAsO from the uniform $LaFe_{1-x}Zn_xAsO$ phase could occur. From the figure 1(b), it can be seen that the a-axis increases slightly with x, but the c-axis shrinks slightly. The cell volume decreases

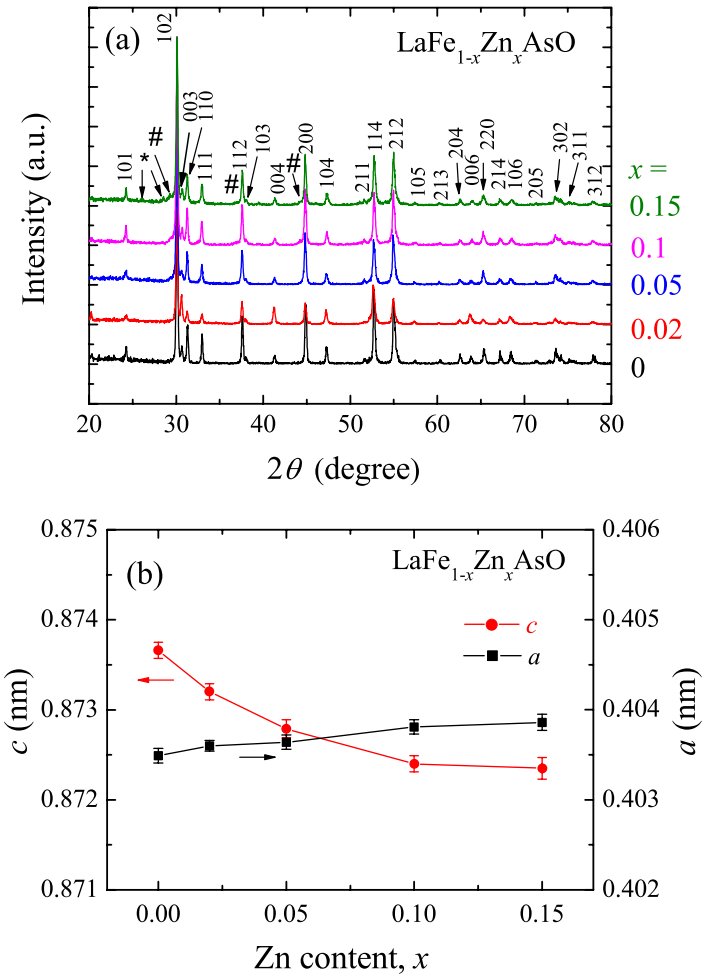

Figure 1. (a) Powder XRD patterns of $LaFe_{1-x}Zn_xAsO$ ($x = 0$, 0.02, 0.05, 0.1 and 0.15) samples. The symbols ♯ and ⋆ denote the impurity phases LaZnAsO and La_2O_3, respectively. (b) Variations of lattice parameters as a function of Zn content.

first and then increases slightly for $x > 0.05$. For larger Zn content, the change in the lattice constants a and c becomes saturated, which is in agreement with the phase separation as $x \geqslant 0.1$.

Figure 2 shows the temperature dependence of resistivity for the $LaFe_{1-x}Zn_xAsO$ samples. The resistivity of the undoped parent compound shows a drop around T^* of 150 K, where T^* is defined as the onset point of this anomaly. As shown in the inset, the anomaly becomes clearer in the plot of $d\rho/dT$ versus T. Such an anomalous drop in the resistivity is associated with the structural transition and/or formation of AFM SDW [32]. Even with addition of only 2% Zn content ($x = 0.02$), the anomaly becomes less significant and moves to a lower temperature ($T^* \sim 140$ K) and the resistivity itself becomes semiconductor-like. As x increases to 0.05, this anomaly almost disappears. Only a very tiny kink around 120 K can be observed in the curve of $d\rho/dT$ versus T for $x = 0.05$, which might result from the residual SDW order. On the other hand, in contrast to the case of Co doping on the Fe site [25, 26], no superconductivity in the Zn-doped $LaFe_{1-x}Zn_xAsO$ samples is observed for temperature down to 2 K. Instead, the

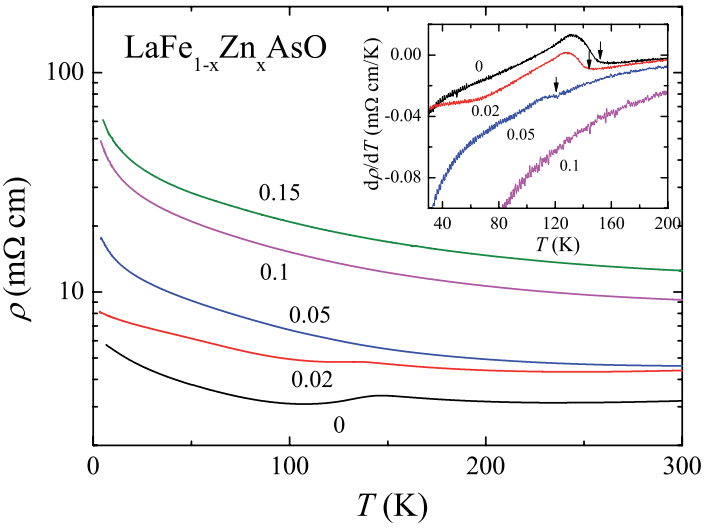

Figure 2. The temperature dependence of resistivity (ρ) for $LaFe_{1-x}Zn_xAsO$ samples. The inset shows the derivative of resistivity versus temperature. The arrows indicate the anomaly in resistivity, which could be associated with structural and/or magnetic transitions.

resistivity is semiconductor-like for all the Zn-doped samples. Actually, the resistivity increases roughly logarithmically with decreasing temperature at low temperatures instead of usual thermally activated conductivity in typical semiconductors. It should also be noted that the resistivity value increases gradually with increasing Zn content, suggesting the effect of disorder caused by Zn doping. For $x \geqslant 0.10$, there is a large increase in the resistivity, consistent with the fact that the $LaFe_{1-x}Zn_xAsO$ samples become phase separated for $x \geqslant 0.10$ as shown by the XRD patterns.

Figure 3 shows the temperature dependence of magnetic susceptibility for the $LaFe_{1-x}Zn_xAsO$ samples. A clear drop in susceptibility related to SDW order can be observed around 150 K for the parent compound, which is consistent with previous reports [1, 32]. With increasing Zn content, the susceptibility increases gradually, and the linear temperature dependence of susceptibility for the temperature above the SDW transition, which could result from the fluctuations of 'SDW moments' of Fe^{2+} ions [34], becomes less and less significant. The Curie-like upturn observed at low temperatures could be mainly due to an extrinsic origin (such as defects and trace impurities). For $x = 0.02$, the drop in susceptibility can still be observed around 137 K, consistent with the resistivity result. However, no anomalous change in susceptibility can be observed for $x \geqslant 0.05$. The magnetic data show that the SDW order is very sensitive to Zn doping.

In order to confirm further the suppression of SDW order by Zn doping, the measurement of thermopower (S) was also performed. As demonstrated by several previous reports on the novel superconductors such as $NbSe_2$ and Sr_2RuO_4, the thermopower is very sensitive to subtle changes in the electronic structure [35, 36]. Figure 4 shows the temperature dependence of thermopower for the $LaFe_{1-x}Zn_xAsO$ samples. A remarkable increase in S occurs around the structural and magnetic transitions. Similar behavior has been observed in the previous reports [37, 38]. This anomaly in S could result from opening of a pseudo-gap in Fermi surface.

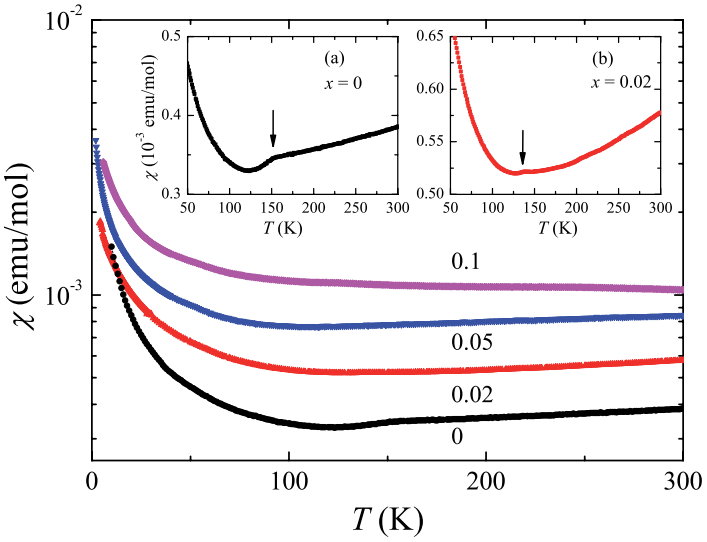

Figure 3. The temperature dependence of magnetic susceptibility (χ) of $LaFe_{1-x}Zn_xAsO$ samples. The insets show the enlarged plots for $x = 0$ and 0.02. The arrows indicate the anomaly in susceptibility related to structural and/or magnetic transitions.

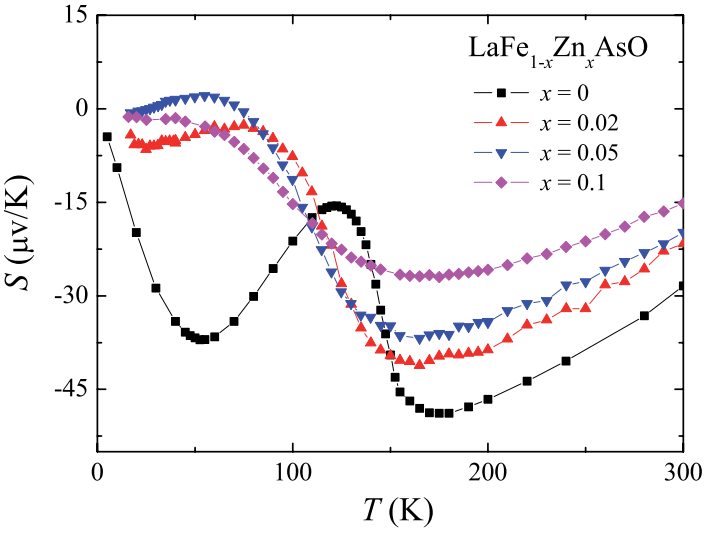

Figure 4. The temperature dependence of thermopower (S) for $LaFe_{1-x}Zn_xAsO$ samples.

Optical spectra indeed revealed the evidence for pseudo-gap opening below structrual/SDW transitions [23]. Because of the multi-band nature, a quantitative analysis of the thermopower data is quite difficult. However, the anomalous change in S can be regarded as experimental evidence of the SDW transition. It can be seen from figure 4 that the anomaly in S becomes less significant and moves to a lower temperature for $x = 0.02$, still observable for $x = 0.05$, but almost disappears for $x \geqslant 0.10$. The thermopower data confirm further that the SDW order is severely suppressed by Zn doping, essentially consistent with the resistivity and susceptibility

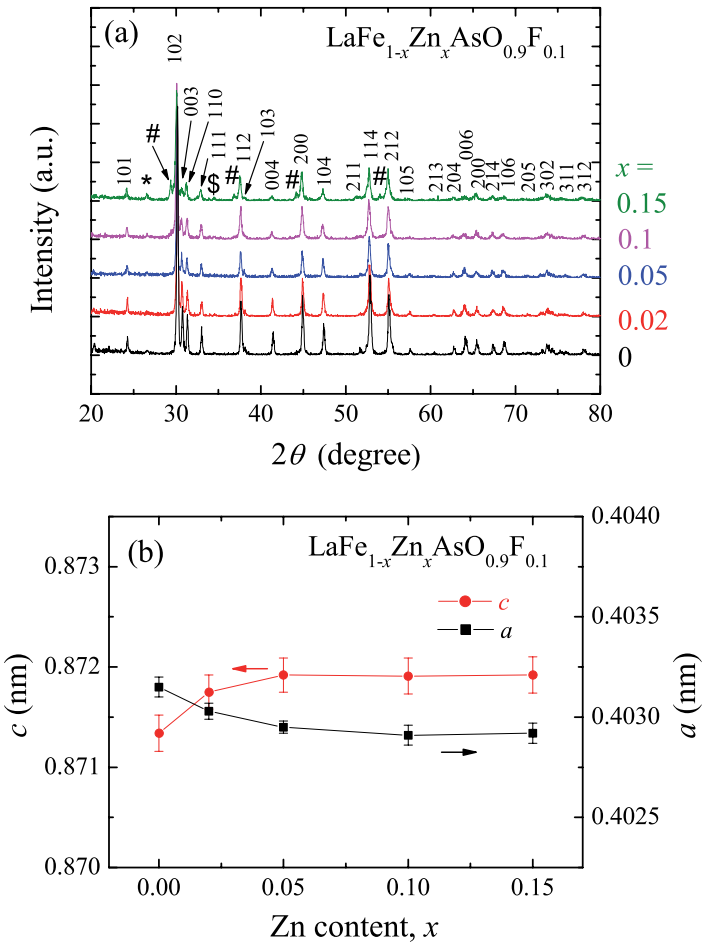

Figure 5. (a) Powder XRD patterns of $LaFe_{1-x}Zn_xAsO_{0.9}F_{0.1}$ ($x = 0$, 0.02, 0.05, 0.1 and 0.15) samples. The symbols ♯, ⋆ and \$ denote the impurity phases LaZnAsO, La_2O_3 and FeAs, respectively. (b) Lattice parameters as a function of Zn content.

data. The effect of Zn doping on the SDW order is striking, which implies that the magnetism in the Fe_2As_2 layers can be affected severely by addition of non-magnetic Zn^{2+} ions. However, as shown below, there is almost no influence of Zn doping on superconductivity in the F-doped $LaFe_{1-x}Zn_xAsO_{0.9}F_{0.1}$.

3.2. Superconductivity in Zn doped $LaFe_{1-x}Zn_xAsO_{0.9}F_{0.1}$

Figure 5(a) shows the XRD patterns of $LaFe_{1-x}Zn_xAsO_{0.9}F_{0.1}$ samples and figure 5(b) shows the variations of the lattice constants with Zn content. For small Zn doping level, all the peaks can be well indexed based on a tetragonal cell with the space group of $P4/nmm$. Obviously F doping causes remarkably shrinkage of both a- and c-axes, similar to the previous report [1]. With the addition of Zn in the F-doped samples, the a-axis decreases slightly, whereas the c-axis increases. The change in the c-axis becomes saturated for $x \geqslant 0.05$, indicating that the samples could become phase separated. A few tiny foreign XRD peaks corresponding to

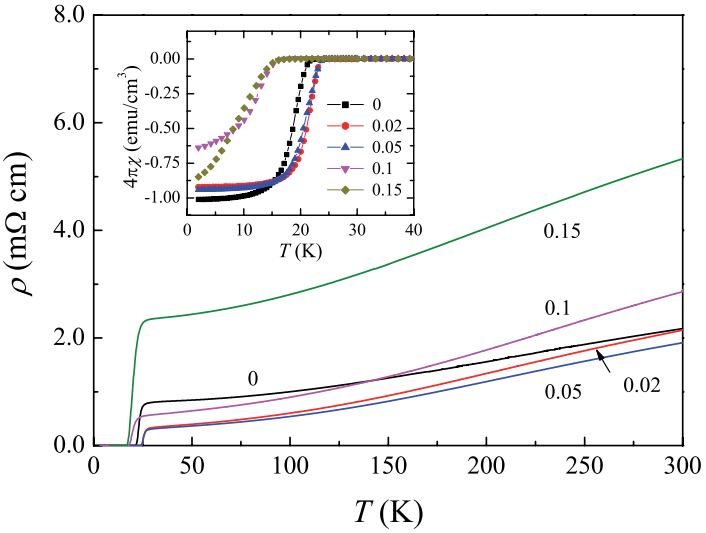

Figure 6. The temperature dependence of resistivity (ρ) of $LaFe_{1-x}Zn_xAsO_{0.9}F_{0.1}$ samples. Inset: the dc magnetic susceptibility (χ) versus temperature measured under magnetic field of 10 Oe.

the impurity phases LaZnAsO, La_2O_3 and FeAs appear for large x (indicated by symbols ♯, ⋆ and \$, respectively). As in the case of $LaFe_{1-x}Zn_xAsO$, there is a limit of Zn solubility on the Fe-site and superabundant Zn doping leads to phase separation. The solubility of Zn doping in the F-doped $LaFeAsO_{0.9}F_{0.1}$ is even smaller compared to the parent compound.

Figure 6 shows the temperature dependence of resistivity for the $LaFe_{1-x}Zn_xAsO_{0.9}F_{0.1}$ ($x = 0$, 0.02, 0.05, 0.1 and 0.15) samples and the inset shows the temperature dependence of dc magnetic susceptibility measured under H of 10 Oe. Without Zn doping, the 10% F doping (almost at the optimal doping level) induces superconductivity at T_c^{onset} of 26.8 K and T_c^{mid} of 23.0 K, where T_c^{onset} and T_c^{mid} are defined as the onset point and midpoint temperature in the resistive transition, respectively, consistent with the previous report [1]. To our surprise, the temperature dependence of resistivity becomes even more metallic for the Zn-doped samples. Meanwhile T_c^{mid} slightly increases to 25.5 and 25.4 K for $x = 0.02$ and 0.05, respectively. The volume fraction of superconducting magnetic shielding is nearly 100% for the samples with $x = 0$, 0.02 and 0.05 estimated from the magnetic susceptibility. The demagnetizing factor N is taken into account in our estimation, but the volume fraction could still be a little over-estimated due to other factors. The sharp transitions in the magnetic susceptibility suggest bulk superconductivity and high homogeneity of the Zn-doped samples for $x \leqslant 0.05$. For $x \geqslant 0.10$, there is a large increase in the normal state resistivity and the superconducting transition becomes broad due to the phase separation. Meanwhile, the volume fraction of superconducting magnetic shielding also decreases quickly for $x \geqslant 0.05$, indicating the existence of non-superconducting impurity phases. To our surprise, Zn doping does not suppress superconductivity in the single-phase F-doped samples and it even enhances T_c instead.

The temperature dependence of thermopower for the $LaFe_{1-x}Zn_xAsO_{0.9}F_{0.1}$ samples is shown in figure 7. The thermopower drops to zero quickly below T_c. Similar to the resistive and magnetic superconducting transitions, the transition in S for $x \leqslant 0.05$ is very sharp, but it becomes broad for $x = 0.1$ due to the phase separation. The absolute value of thermopower,

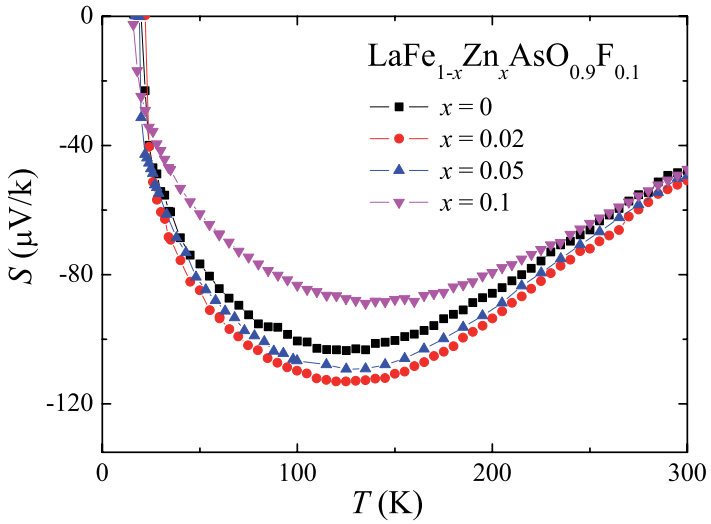

Figure 7. The temperature dependence of thermopower (S) for $LaFe_{1-x}Zn_xAsO_{0.9}F_{0.1}$ samples.

$|S|$, is much larger than that of the parent compound LaFeAsO. There seems to exist a close correlation between the absolute value of thermopower and T_c, namely, the higher T_c corresponds to larger normal state $|S|$, as proposed in the previous reports [25]. There should be a relationship between the origin of the enhanced thermopower and the superconducting mechanism.

As is well known, Zn doping on the CuO_2 planes causes radical suppression of superconductivity in the high-T_c cuprates [19, 20], and interpretations in the frame of pair-breaking or decrease in the superfluid density were proposed (for example see [39] and references therein). In contrast to the case of cuprates, Zn doping almost has no influence on superconductivity in the Fe-based oxy-arsenides. This result reveals a significant difference between high-T_c ferro-arsenides and high-T_c cuprates. In the latter case, any substitution of Cu by other elements in the CuO_2 layers can lead to sharp suppression of superconductivity. Meanwhile in the ferro-arsenides superconductivity can survive when the Fe element in the conducting layer is partially substituted by other 3d transition elements such as Co, Ni and Zn no mater whether the substitution is magnetic or non-magnetic. However, there is still an essential difference between Co doping and Zn doping. That is, the Zn doping itself cannot induce superconductivity in LaFeAsO system. The calculation of density of states (DOS) versus energy for LaOMAs (M = Mn, Fe, Co and Ni) by Xu *et al* [40] shows that the main feature of total DOS remains unchanged in these systems, except that Fermi level shifts toward the top of valence band with band filling (adding electrons) one by one from M = Mn, Fe, Co to Ni. This study indicates that the 3d electrons of M = Mn, Fe, Co and Ni are essentially itinerant. The electron configuration of Zn^{2+} ion is $3d^{10}$, very different from that of Mn, Fe, Co or Ni. A recent band structure calculation of LaZnAsO shows that the fully occupied Zn 3d states are located about -7 eV to the Fermi level [41], namely, the 3d electrons of Zn in this structure are localized. Thus the partial substitution of Fe by Zn is not expected to add more itinerant electrons into FeAs layers and it causes mainly disorder instead, which should account for the difference between the effect of Zn doping and that of Co or Ni doping in this structure.

Whereas the anomaly in the resistivity is completely suppressed in the parent compound, the superconductivity in the F-doped system remains almost unperturbed by Zn doping, which reveals that doped Zn^{2+} ions affect selectively AFM order and meanwhile the disorder caused by Zn doping has little effect on the superconducting electron pairing. Neutron studies have revealed that the parent compound LaFeAsO is a long-range ordered antiferromagnet with a simple stripe-type AFM structure within the plane at low temperatures [32]. Such a stripe-type AFM long-range order can be easily destroyed by disorder. The substitution of magnetic Fe^{2+} ions with non-magnetic Zn^{2+} ions destroys the long-range AFM order and short-range AFM fluctuations could still remain. In the F-doped $LaFe_{1-x}Zn_xAsO_{0.9}F_{0.1}$ superconductor, the stripe-type long-range AFM order no longer exists and thus the main effect of Zn doping is to induce disorder in the FeAs layers. All the results of Co-, Ni- or Zn-doped superconducting systems show that high-T_c superconductivity is quite robust against the disorder in the conducting FeAs layer, which may be due to the quite three-dimensional superconductivity of the iron-based arsenide superconductors [42].

4. Conclusion

In summary, we have investigated the effect of non-magnetic Zn doping on AFM order and superconductivity in the parent and F-doped LaFeAsO. A striking discovery is that Zn doping suppresses severely the AFM order without occurrence of superconductivity in the parent compound. Meanwhile superconductivity remains almost unperturbed by Zn doping in the F-doped LaFeAsO systems. We also found that the solubility of Zn doping on Fe site is limited to $x = 0.1$. Our results may shed light on the mechanism of superconductivity and the relationship between AFM (SDW) order and superconductivity.

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