Synthesis and physical properties of LaO$_{1-x}$F$_x$FeAs

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Abstract. We have prepared the newly discovered Fe-based superconducting material LaO$_{1-x}$F$_x$FeAs (0 $\leq$ $x$ $\leq$ 0.2) in polycrystalline form and have investigated the samples by means of structural, thermodynamic and transport measurements. Our investigations reveal a non superconducting phase at 0 $\leq$ $x$ $\lesssim$ 0.04 which for $x$ = 0 is characterized by a structural transition towards an orthorhombic distortion at $T_N$ $\approx$ 160 K and antiferromagnetic spin order at $T_N$ $\approx$ 138 K. Both transitions lead to strong anomalies in various transport properties as well as in magnetization and in specific heat. Remarkably, the transition temperatures are only weakly doping dependent up $x$ $\lesssim$ 0.04. However, the transitions are abruptly suppressed at $x$ $\geq$ 0.05 in favour of a superconducting phase with a critical temperature $T_c$ $\gtrsim$ 20 K. Upon further increasing the F-doping $T_c$ increases up to a maximum of $T_c$ = 26.8 K at $x$ = 0.1 which is followed by a decrease down to $T_c$ $\approx$ 10 K at $x$ $\geq$ 0.15.

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1 Introduction

The ongoing search for new superconductors has recently yielded a new family of Fe-based compounds composed of alternating LaO$_1$-$x$F$_x$ and FeAs layers with transition temperatures $T_c$ up to 28 K [1]. Rapidly after the discovery, $T_c$ has been raised to above 50 K [2, 3, 4, 5, 6] by replacing La with other Rare Earths, and thus the first non-copper-oxide superconductor with $T_c$ exceeding 50 K has emerged. Both, theoretical treatments and experimental findings indicate unconventional multiband superconductivity in these materials. Ab-initio calculations of the electron-phonon coupling are incompatible with a conventional electron-phonon pairing mechanism, which is consistent with experimental findings [10]. The unusual nature of superconductivity is experimentally also evident from the absence of a Hebel-Slichter peak in NMR experiments [11] and high magnetic field experiments [12,13].

Various mechanisms of superconductivity have been discussed and different pairing symmetries of the superconducting ground state including spin-triplet p-wave pairing have been proposed [9,14,15,16,17]. Intriguingly, there is evidence for a close interplay between superconductivity and magnetism as it is well established for other unconventional superconductors. A commensurate spin-density wave (SDW) and an orthorhombic distortion have been observed below $\approx$ 150 K in the undoped parent compound [18,19] which appear to be fully suppressed once superconductivity emerges upon doping [20,21]. Note, that the SDW ground state has also been established for isostructural Rare Earth (R) based RO$_1$-$x$Fe$_x$As [22], and even in the parent material of other iron-pnictide superconductors such as Ba$_{1-x}$K$_x$Fe$_2$As$_2$ which exhibit a different crystal structure but similar Fe$_2$As$_2$ layers [23,24,25]. While the superconducting state of LaO$_{1-x}$F$_x$FeAs has been shown to exhibit no traces of static magnetic order [20,21], the coexistence of static magnetic order and superconductivity has been reported [22,26,27,28].

In this paper, we investigate the impact of the structural and magnetic transitions of undoped LaOFeAs on bulk physical properties and study the evolution as a function of doping. In particular we present investigations of LaO$_{1-x}$F$_x$FeAs by means of structural, thermodynamic and transport measurements. Our investigations reveal a non superconducting phase at 0 $\leq$ $x$ $\lesssim$ 0.04 where the physical properties are dominated by a two successive transitions. At higher temperatures ($T_s$ $\approx$ 160 K at $x$ = 0), there is a transition from tetragonal to orthorhombic structure which is accompanied by distinct drops in the magnetic susceptibility $\chi$ and the electrical resistivity $\rho$. At slightly lower temperature ($T_N$ $\approx$ 138 K at $x$ = 0), a magnetic transition occurs towards long range spin density wave (SDW) antiferromagnetic order, which leads to an inflection point in the resistivity [19]. Remarkably these transition temperatures are only weakly doping dependent up to $x$ $\lesssim$ 0.04. However, the transitions are abruptly sup-

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pressed at $x \geq 0.05$ in favour of a superconducting phase with a critical temperature $T_c \geq 20$ K, i.e. close to the boundary in the phase diagram where superconductivity emerges. Upon increasing the F-doping, $T_c$ increases up to a maximum of $T_c = 26.8$ K at $x = 0.1$ which is followed by a decrease with $T_c \approx 10$ K at $x > 0.15$. Intriguingly, for lower doping levels $0.05 \leq x \leq 0.075$ of these superconducting samples the temperature dependence of the resistivity at lower temperatures is reminiscent of that of the non-superconducting doping levels, i.e. $\rho(T)$ shows a weakly insulating behavior at $T \lesssim 80$ K. At higher doping levels $x \geq 0.1$, a quadratic temperature dependence evolves in the normal state just above $T_c$ with increasing doping.

2 Experimental

2.1 Sample preparation and characterization

Polycrystalline samples of LaO$_{1-x}$F$_x$FeAs ($0 \leq x \leq 0.2$) were prepared from pure components using a two-step solid state reaction method, similar to that described by Zhu et al. [29]. In the first step, Fe powder (Alfa Aesar, Puratronic, 99.999%) and powdered As lump (Alfa Aesar, 99.999%) were milled, mixed and pressed into pellets and annealed at 500°C for 2 h and at 700°C for 10 h in an evacuated silica tube. In the second step, the FeAs pellets were milled and mixed with lanthanum powder (Goodfellow, 99.99%, 60 mesh), annealed La$_2$O$_3$ powder (Chempur, 99.99%), and anhydrous LaF$_3$ powder (Alfa Aesar, 99.99%) and pressed into pellets under a pressure of 1 GPa. All preparation steps were carried out under argon atmosphere. The samples were then heated in an evacuated silica tube at 940°C for 8 h and at 1150°C for 40 h. Due to the formation of a liquid phase at 1007°C, determined by DT A/TG analysis, the 940°C annealing step was extended compared to Zhu et al. [29] in order to improve the homogeneity.

Since the oxidation state of the system depends strongly on the oxygen stoichiometry, the oxygen content of the starting metals (La and Fe) was measured [30] and taken into account in the weighting procedure of the components. The amount of oxygen in the powders was found to be 2.8 at% for Fe and 3.3 at% for La, respectively. Note that these values differ from lot to lot of the same supplier. A wrapping of the samples in Ta-foil during the annealing process as is described in the literature was not used because this induces an extended incorporation of As into the Ta-foil up to 4 wt% which leads to As deficiency in the samples and, hence, changed physical properties of LaO$_{1-x}$F$_x$FeAs [12]. However, an elevated As vapour-pressure was observed during annealing which mainly depends on the three factors temperature, ampoule volume and sample mass, which allows to optimize the process.

The appearance of the quartz ampoules after annealing (see Figure 1 for a representative example) indicates no significant reaction of the pellets with the quartz wall, which confirms that annealing without Ta foil is possible. Only in the case of the high fluorine doped samples the quartz was slightly attacked.

The annealed pellets were ground and polished and the local composition of the resulting samples was investigated by wavelength-dispersive X-ray spectroscopy (WDX) in a scanning electron microscope. Moreover, room temperature Mo $K_{\alpha}$ powder x-ray diffraction (XRD) in transmission geometry was performed on the samples in order to check the phase purity and to obtain structural information via Rietveld refinement. The data was collected over a $2\theta$ range of 4° to 55° with a step size of 0.01°. The refined parameters include zero point offset, lattice parameters, scale factors, overall isotropic displacement parameters, Lorentzian isotropic crystallite size, Lorentzian isotropic strain, and preferred orientation. In order to determine the lattice parameters as a function of doping with high absolute precision, another set of diffraction patterns was recorded in Bragg-Brentano reflection geometry after mixing the samples with Si-powder for calibration (Cu $K_{\alpha1}$ radiation). Temperature dependent measurements of the lattice parameters were undertaken in Bragg-Brentano geometry using a He evaporation cryostate.

2.2 Physical properties

The electrical resistivity of the samples was measured using a standard four-point method in the temperature range 5-300 K. The electrical contacts to the cuboid-shaped specimens were made with silver paint (DuPont 4929). Magnetization measurements have been performed in the temperature range 2-350 K in a static magnetic field of 1 T using a SQUID magnetometer MPMS-XL5 from Quantum Design. Specific heat measurements in zero magnetic field have been done at 2-250 K by means of a relaxation technique in a Quantum Design PPMS system. Thermal conductivity and Seebeck coefficient were measured in a home-made device using a steady-state method with an SMD resistor as a heater and a Au-Chromel differential thermocouple for determining temperature gradient [31].

3 Results and discussion

3.1 Sample preparation and analysis

The prepared pellets are to a large extent porous. They exhibit grains of LaO$_{1-x}$F$_x$FeAs with typical dimensions
of some tens of micrometers as is revealed by electron microscopy in backscattered electron imaging (BSE mode), cf. Figure 2 for a representative example. We find typical mass densities which amount to about 65% of the theoretical value. The chemical composition of the main phase was confirmed by wavelength dispersive X-ray analysis mode. The chemical composition of the main phase was measured in WDX mode are FeAs and LaO.

Representative results of the Rietveld refinement are shown in Figure 3. The majority of the diffraction peaks are explained by the phase LaFeAsO which has a tetragonal structure with space group $P4/nmm$, in agreement with previous reported data [32]. The remaining peaks correspond to impurity phases FeAs and LaFO. The quality of the overall refinement, i.e. $\chi^2$, of all samples was found between 0.947 and 2.68 (corresponding to the samples with $x = 0.125$ and $x = 0.2$, respectively) which indicates a satisfactory fit. As can be seen in Figure 3, the diffraction peaks of both impurity phases are well resolvable at the doping level $x = 0.2$. These secondary phases appear with fractional concentrations lower than about 3% except for the samples with $x = 0.04$ and $x = 0.2$, where the total amount of secondary phases is found to be around 5% and 11%, respectively.

Figure 4 shows the variation of the lattice parameters as function of the F content. It shows that both $a$ and $c$ decrease systematically by substituting O$^{-2}$ by F$^{-}$ up to the highest doping level. It appears noteworthy that the reduction of the lattice volume seems to set in above 4% doping, i.e. above the critical doping level where the two low-temperature transitions are suppressed (see the inset of Figure 4).

3.2 Physical Properties

Our data of the temperature dependence of $\rho$, $\chi$, $\alpha$- and $\epsilon$-lattice constants, $c_p$, $S$ and $\kappa$ at zero doping level $x = 0$ are summarized in Figure 5. The data presented in this figure show a strong impact of the structural (cf. Figure 5) and magnetic transitions at $T_N \approx 160$ K and $T_N = 138$ K on all quantities shown in the figure. As has already been pointed out in References [19,33,34] an inti-
mate coupling exists between the two transitions and the electronic and magnetic properties. The structural transition is well resolved through the splitting of the (220) reflection (tetragonal notation) into the (400)$_O$ and (040)$_O$ (orthorhombic notation) at $T_S \approx 160$ K. The resistivity (see Figure 5h) exhibits a maximum at $T_S$ where it starts to decrease upon lowering the temperature. Interestingly, at the onset of long range antiferromagnetic SDW order at $T_N = 138$ K [19,18] the drop of the resistivity becomes weaker (visible through an inflection point in $\rho$ at the onset of long range antiferromagnetic SDW order (Figure 5d), which show a sign change near $T_N$). The data confirm a jump in $c_p$ at $T_S$ which is indicative of a second order phase transition and an additional anomaly at $T_N$. The similarity between $d(\chi T)/dT$ and the measured specific heat anomaly indicates that the total entropy change connected with both transitions is proportional to that of magnetic origin. Note, however, that the former comprises contributions from structural and charge degrees of freedom in addition to the magnetic ones.

The strong impact of the transitions on the lattice properties is reflected in the thermal conductivity $\kappa$, which is presented in Figure 5. According to the Wiedemann-Franz law applied on the resistivity data, the electronic contribution to $\kappa$ is negligible and hence $\kappa$ of LaOFeAs is of phononic origin. The most prominent feature of the measured data is a significant deviation from a $\sim T^{-1}$ increase which is usually expected for phononic heat conductivity at higher temperature. Instead, a strong suppression connected with a kink-like anomaly near $T_N$ and $T_S$ is observed which signals the sudden freezing of fluctuations connected with the transitions [30,31].

Figure 6 summarizes our data for $\rho$, $\chi$, the lattice constants as well as $d\rho/dT$ and the magnetic specific heat $d(\chi T)/dT$ for the highest non-superconducting doping level $x = 0.04$. A first glance on the figure suggests that doping LaO$_{1-x}$F$_x$FeAs up to a level of $x \approx 0.04$ leaves the qualitative properties of the material essentially unchanged. The structural transition is still clearly present as can be inferred from the orthorhombic splitting with a similar magnitude as in the undoped case. As in the undoped case, the structural transition leads to clear anomalies in $\rho$ and $\chi$. Note, however, that the transition occurs at a somewhat lower temperature $T_S \approx 150$ K and that the connected anomalies in $\rho$ and $\chi$ are less pronounced and broadened. The resistivity maximum even occurs a few Kelvin below $T_S$. A similar conclusion holds for the onset of long range SDW order, which sets in at $T_N \approx 122$ K [21]. While $T_N$ is still well reflected through the inflection point of $\rho$, a corresponding anomaly is barely resolveable in the magnetic susceptibility.

Superconductivity occurs abruptly with a rather high $T_c = 20.6$ K at a slightly higher doping level $x = 0.05$, and the superconducting phase persists up to the highest studied doping level $x = 0.2$. Thereby $T_c$ increases with doping up to 26.8 K at $x = 0.1$ and then quickly diminishes with further increasing $x$ down to $T_c = 19.4$ K at $x = 0.125$ and $T_c \approx 10$ K for $x = 0.15$ and 0.2. All these superconducting samples show a strong diamagnetic response in zero field cooled (ZFC) as well as in field cooled (FC) measurements (cf. Figure 7). Note, that for $x = 0.15$ and $x = 0.2$, the diamagnetic signal becomes significantly weaker.
In the following we describe the magnetic, structural and electrical transport properties for two selected and representative concentrations. Figure 8 summarizes our results for the lattice parameters, $\rho$ and $\chi$ at $x = 0.05$, i.e. at the lowest doping level where superconductivity occurs. The structural data clearly show that the transition to the low-temperature orthorhombic phase is absent in the investigated temperature range. Note, that also static or slowly fluctuating magnetism is clearly absent as revealed by $\mu$SR experiments [21]. Interestingly, the suppression of the structural/magnetic transitions and the occurrence of superconductivity is accompanied by strong changes of the normal state resistivity. A low-temperature upturn ($T \lesssim 60$ K) is still present just above $T_c = 20.6$ K, which is rem-
The highest critical temperature of our LaO$_{1-x}$F$_x$FeAs samples occurs at the doping level $x = 0.1$. Also for this compound no indication of a structural transition towards the low-temperature orthorhombic phase is observed (cf. the structural data in Figure 9). The magnetic susceptibility is qualitatively and quantitatively very similar to that of the lower superconducting doping levels. The only change is a slightly more pronounced positive curvature. A drastic systematic change is present, however, in the electrical resistivity $\rho(T)$ at doping levels $0.1 \leq x \leq 0.2$. Here, instead of the low-$T$ upturn, we observe $\rho(T) = \rho_0 + AT^2$ ($\rho_0 = \text{const}$) from just above $T_c$ up to $\sim 200$ K [34], consistent with previous data [32] for $x = 0.11$.

3.3 Discussion

The physical properties exhibit a systematic change as a function of doping. Apparently, there exist three major regions in the electronic phase diagram of LaO$_{1-x}$F$_x$FeAs as a function of doping level $x$. At low doping levels $0 \leq x \leq 0.04$ the material is a non-superconducting metal with long range SDW order as the ground state. As is revealed by our data, the transition towards the SDW state and the accompanying structural transition has a strong impact on the physical properties of the system. More precisely, our data show that the transitions have a combined effect on the charge degrees of freedom (resistivity, thermopower), magnetism (magnetic susceptibility) and structure (diffraction, thermal transport). At slightly higher doping levels ($0.05 \leq x \leq 0.075$) the transitions are sup-

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**Fig. 6.** Temperature dependence of $\rho$, $\chi = M/B$, $d\rho/dT$, the magnetic specific heat $d(\chi T)/dT$ and the lattice constants for the highest non-superconducting doping level $x = 0.04$.

**Fig. 7.** Temperature dependence of $\rho$, $\chi$ in the vicinity of $T_c$ for representative doping levels in the superconducting regime of the phase diagram. Magnetic measurements have been performed in a field of 2 mT.
pressed in favour of superconductivity at rather high temperature $T_c > 20$ K. This clearly suggests that SDW order and superconductivity compete in the system. In this second region of the phase diagram, no traces of static magnetism is observed in $\mu$SR experiments [21], in contrast to the Sm-based pendant of the system, SmO$_{1-x}$F$_x$FeAs, where SDW order and superconductivity apparently may coexist [22, 26]. Despite this interesting finding, the resistivity shows a low temperature upturn just before the onset of superconductivity, which is reminiscent of the low-temperature upturn of the non superconducting species.

The third region of the phase diagram comprises even higher doping levels $0.1 \leq x \leq 0.2$, which includes the finding of maximum $T_c = 26.8$ K at $x = 0.1$ and also the lowest $T_c \approx 10$ K at $x = 0.15$ and $x = 0.2$. Here, instead of a the low-temperature upturn we observe a monotonically increasing resistivity curve with $\rho(T) = \rho_0 + AT^2$ ($\rho_0 = \text{const}$) at low temperature.

4 Summary

We have prepared polycrystals of the new superconducting material LaO$_{1-x}$F$_x$FeAs. The characterization of the samples clearly shows that the materials are of high quality in the sense of phase purity and systematic variation of the lattice constants as a function of the fluorine doping level. Our investigation of the structural, thermodynamic and transport properties of the samples reveal a non superconducting phase at $0 \leq x \lesssim 0.04$ which for $x = 0$ is characterized by a structural transition towards an orthorhombic distortion at $T_s \approx 160$ K and antiferromagnetic spin order at $T_N \approx 138$ K. Both transitions have a strong impact on electronic, magnetic and structural degrees of freedom. Remarkably, these transition temperatures are only weakly doping dependent up $x \lesssim 0.04$. At slightly higher doping level ($x \geq 0.05$) superconductivity emerges with a concurrent suppression of the magnetic
and structural transitions. Upon increasing the F-doping a maximum $T_c = 26.8$ K is observed at $x = 0.1$ followed by a decrease with $T_c \approx 10$ K at $x \geq 0.15$. While the electrical resistivity shows pronounced doping dependence in the normal state, the magnetic susceptibility is only weakly doping dependent.

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