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ORIGINAL PAPER

Temperature-Dependent As K-Edge EXAFS Studies of $LaFe_{1-x}Co_xAsO$ ($x = 0.0$ and 0.11) Single Crystals

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Abstract We report the experimental results of temperature-dependent *polarized* As K-edge extended X-ray absorption fine structure (EXAFS) of $LaFe_{1-x}Co_xAsO$ ($x = 0.0$ and 0.11) single crystals. By aligning the Fe–As bond direction in the direction of the X-ray beam polarization, we have been able to identify an anomaly in the Fe–As bond correlations at the tetragonal to orthorhombic transition at 150 K, while previous investigations with standard unpolarized EXAFS of undoped LaFeAsO powder samples were not able to detect any such anomaly. Using our approach, we have been able to identify in the superconducting doped sample, $LaFe_{0.89}Co_{0.11}AsO$, a broad anomaly around 60 K. The low-temperature anomaly has good correlations with the temperature dependence of several properties like resistivity, magnetic susceptibility and linear thermal expansion, indicating the emergence of the dynamical oscillations of the Fe–As pairs.

Keywords EXAFS · Fe based superconductors · Fe-As bond correlations · Local structure

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1 Introduction

The excitement in the condensed matter community on the iron-based superconductors (FeSCs) continues to grow even after several years of intense worldwide research efforts, thanks to the several interesting experimental and theoretical investigations. The F-doped LaFeAsO systems [1] and the related lanthanide compounds [2] (so-called 1111 systems) have attracted a renewed interest once quality single crystals of these systems become available [3, 4], thus making way for experimental investigations [4–10]. These new experiments allow one to test the available theoretical predictions and also to give further experimental inputs needed for the possible formulation of an effective unified theory for the description of high-temperature superconductors. The most important and puzzling characteristics of these compounds are the occurrences of magnetism and superconductivity in close vicinity in the phase diagram and the interplay between the two [10]. Magnetism in these systems has attracted a large theoretical interest [11–13], especially the unusually small magnetic moment associated with the Fe lattice. To obtain an overall agreement between the different theoretical tools and experimental observations, it was proposed that the 1111 systems posses a strong magnetic ground state; however, with fluctuating domains which prelude the experimental detection. Recently, using single crystals, in the NdFeAsO system, two additional

phase transitions associated with the Nd and Fe magnetic moments were observed at low temperatures [14], indicating that indeed the rare-earth and Fe magnetic moments play a more active role than those revealed by the earlier experiments [15–17]. Magnetic and superconducting transitions have a direct correlation with the changes in the electronic density of states, which in turn is driven by the subtle structural changes, in many cases directly visible as anomalies in the mean-square relative displacements of the participating atomic bonds. Systematic local structural studies are necessary for the detection of such anomalies. In cuprates, such anomalies were detected in several systems using extended X-ray absorption fine structure (EXAFS) spectroscopy studies [18, 19] and neutron diffraction-based atomic pair distribution function (PDF) analysis [20, 21]. EXAFS [22–29] and X-ray PDF [30–34] studies also yielded several important information on the FeSC; however, a lack of high-quality single crystals limited those studies. Here, we report a systematic EXAFS study on the $LaFe_{1-x}Co_xAsO$ ($x = 0$ and 0.11) system using millimetre-sized single crystals, which demonstrates the important lattice effects in determining the properties of the system.

2 Materials and Methods

$LaFe_{1-x}Co_xAsO$ ($x = 0$ and 0.11) single crystals of size around ~2 mm × 2 mm × 0.2 mm were grown under ambient pressure in NaAs flux [4]. Temperature-dependent As K-edge EXAFS on these single crystals was carried out in fluorescence yield mode at the beamline, BM 29 of the European synchrotron radiation facility, Grenoble (France). Measurements were carried out between 15 and 300 K (with more than 25 temperature points for each crystal). The crystal was oriented so that the direction of the Fe–As bond is parallel to the electric field direction of the polarized X-ray photon beam. A continuous flow liquid He cryostat was used for the low-temperature measurements. Sample temperature during measurements was monitored and controlled within ±1 K. A minimum of three scans (many cases up to five) were taken at each temperature on both samples. From the absorption spectra, EXAFS data were extracted following the standard procedures [35]. At each temperature, the average of the different scans was used for the analysis.

EXAFS is a unique fast (with a measuring time scale of 10^{-15} s) local tool to get the instantaneous Fe–As bond length distribution without time averaging [18, 19, 35–39] which has first detected the short-range charge density wave [18], the polaron size [37] and the nanoscale stripe phase [38] in cuprates. The structure of LaFeAsO is made of an alternate stacking of the active [FeAs] active and spacer [LaO] layers [3, 39] as shown in Fig. 1a. The misfit strain [2, 40] and the proximity of the chemical potential to an electronic topological Lifshitz transition [41, 42] drive these layered systems to an arrested nanoscale phase separation [43, 44].

3 Results and Discussion

The raw experimental data (the Fourier transform magnitude) extracted from the As K-edge EXAFS in the complete temperature range are presented in Fig. 1b, d. Instead, a zoom over the three main peaks (representing the different

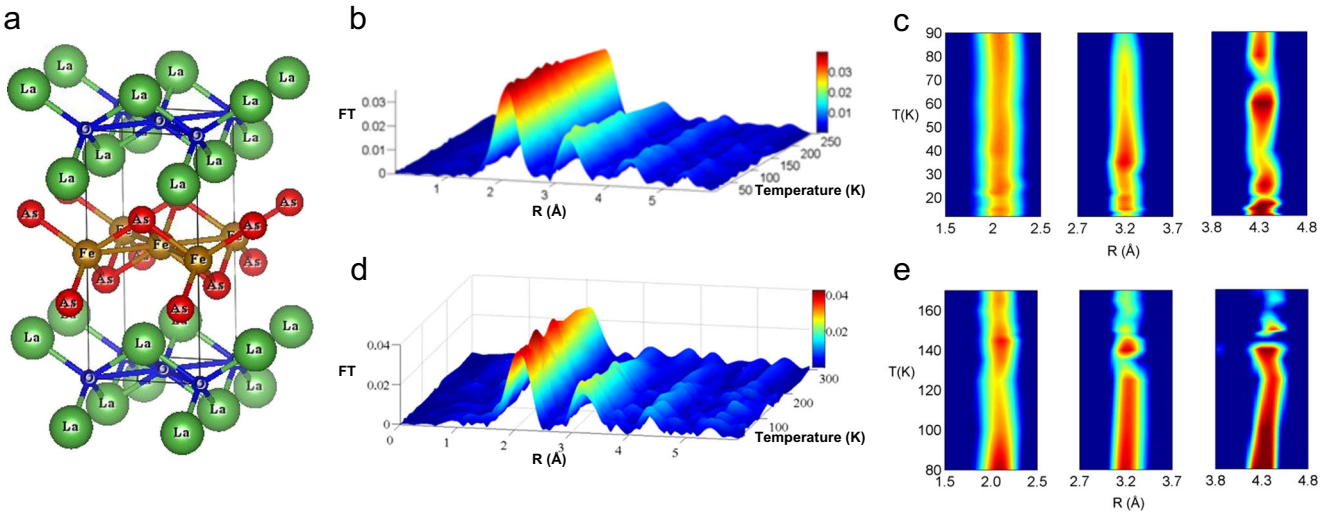

Fig. 1 Unit cell of LaFeAsO (**a**). Fourier transform (*FT*) magnitudes of the As K-edge EXAFS oscillations at different temperatures for the $LaFe_{1-x}Co_xAsO$ ($x = 0.11$) (**b**, **c**) and LaFeAsO (**d**, **e**) single crystals. FT magnitudes are not corrected for the phase shifts and represent raw experimental data

atomic shells around the As) at two temperature ranges for the Co-substituted and parent compounds is shown in Fig. 1c, e. Anomalies in the intensity are clear from this 2D colour plots in the Fourier transform peaks, close to the structural phase transition at around 150 K in the parent compound, whereas the Co-doped superconducting sample shows clear anomalies around 60 K.

In case of the As K-edge EXAFS in a La-1111 system, the first shell contribution involving As–Fe bonds is well separated from other contributions and, thus, a single shell modelling is very effective in extracting the quantitative Fe–As bond distributions [22, 24, 26]. The structure of $LaFe_{1-x}Co_xAsO$ has a tetragonal symmetry at room temperature. For $x = 0.0$, a structural transition to an orthorhombic phase appears below 150 K [2, 6, 8–10]. For the As site (probed by the As K-edge), there are four Fe near neighbours at a distance of ∼2.4 Å. The next nearest neighbours of As are La and O atoms. Contributions of these distant shells appear mixed, and it is very difficult to extract quantitative information from those distant shells. However, the contribution of the Fe–As bonds is well separated from other contributions and can be analysed using a single shell fit to extract the quantitative information on bond correlations.

As mentioned earlier, As K-edge EXAFS has been analysed using a single shell fit to extract quantitative information on the Fe–As bond correlations. For the single shell fit, except the radial Fe–As distance and related meansquare relative displacements (MSRDs), describing the correlated Debye-Waller factor (σ^2), all other parameters (like the photo-electron energy origin and the number of near neighbours) were kept fixed in the conventional least squares modelling, using the phase and amplitude factors calculated using the FEFF program [26]. For such a two parameter fit, the number of independent data points for the present modelling: $N_{\rm ind} \sim (2\Delta k \Delta R)/\pi$, where Δk and ΔR are respectively the ranges in k and R space over which the data are analysed [25], is 7 ($\Delta k = 10$ Å^{-1} and $\Delta R = 1.2$ Å). This makes the present analysis quite suitable for obtaining quantitative Fe–As bond correlations, minimising the correlation effects among the free parameters involved in the modelling.

The upper and lower left panels of Fig. 2 show respectively the temperature dependence of the Fe–As radial

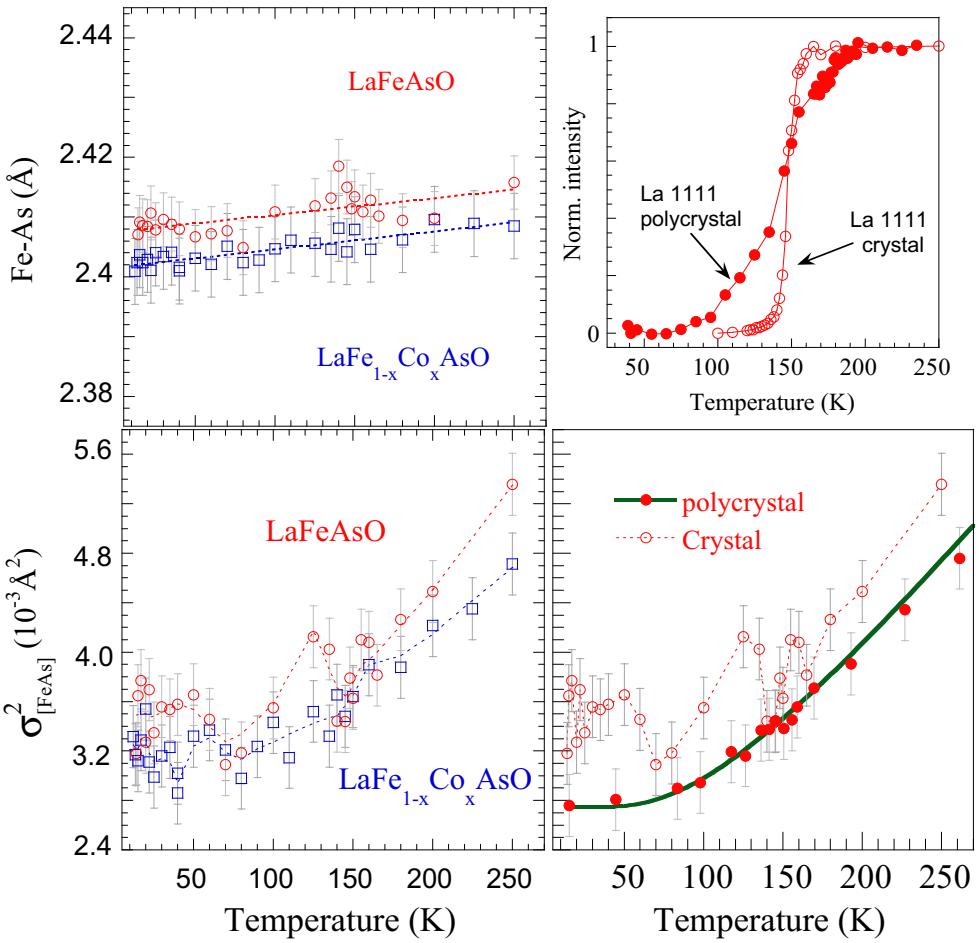

Fig. 2 Variation of the Fe–As bond length and the corresponding meansquare relative displacements (MSRDs) with temperature for the $LaFe_{1-x}Co_xAsO$ ($x = 0$ and 0.11) single crystals extracted from the single shell modelling of the As K-edge EXAFS (respectively *upper* and *lower left panels*). *Lower right panel* shows the temperature dependence of the Fe–As MSRD for the LaFeAsO single crystal and polycrystalline powder [26] samples. Variation of the intensity of the diffraction reflection corresponding to the tetragonal (220) peak with temperature for the single crystal and polycrystalline powder samples of LaFeAsO [9] is shown in the *right upper panel*

distance and the related MSRDs, describing the correlated Debye-Waller factor (σ^2), extracted from the above-mentioned single shell modelling of the As K-edge EXAFS data. The Fe–As distance for the Co-doped sample is slightly lower than that for the undoped sample in line with the reduced lattice parameters for the Co-doped [47] sample.

Overall, the Fe–As bonds show weak temperature dependence (negligible linear thermal expansion). A careful look reveals that the temperature dependence is not smooth, similar to the recent results from high-resolution diffraction measurements [48]. Temperature dependence of the MSRD is different between the doped and undoped samples (Fig. 3, lower left panel). For the parent compound, the Fe–As MSRDs show abrupt changes around 150 K. No such changes are seen in the Co-doped sample. The orthorhombic distortion for our sample is reported to be occurring below 154.5 K and the onset of magnetic order to be around 140 K. Both these transitions are absent in the Codoped sample, which shows a superconducting transition around 10 K.

Clearly, the anomalies seen in the Fe–As MSRD are related to the phase transitions in the system. Interestingly, such anomalies were not reported in the earlier EXAFS studies on these systems, where a polycrystalline powder sample was used for the measurements. To highlight the difference, in Fig. 2 (lower right panel), we present the temperature dependence of the Fe–As MSRD for the single crystal and the polycrystalline powder sample. The temperature dependence of the Fe–As MSRD is smooth for the polycrystalline powder samples across the structural phase transition temperature [22, 26], whereas for the single crystals, the Fe–As MSRDs show a clear anomaly. In a systematic diffraction study, Ricci et al. highlighted the difference in the structural phase transition (SPT) properties of the single crystals and the corresponding polycrystalline

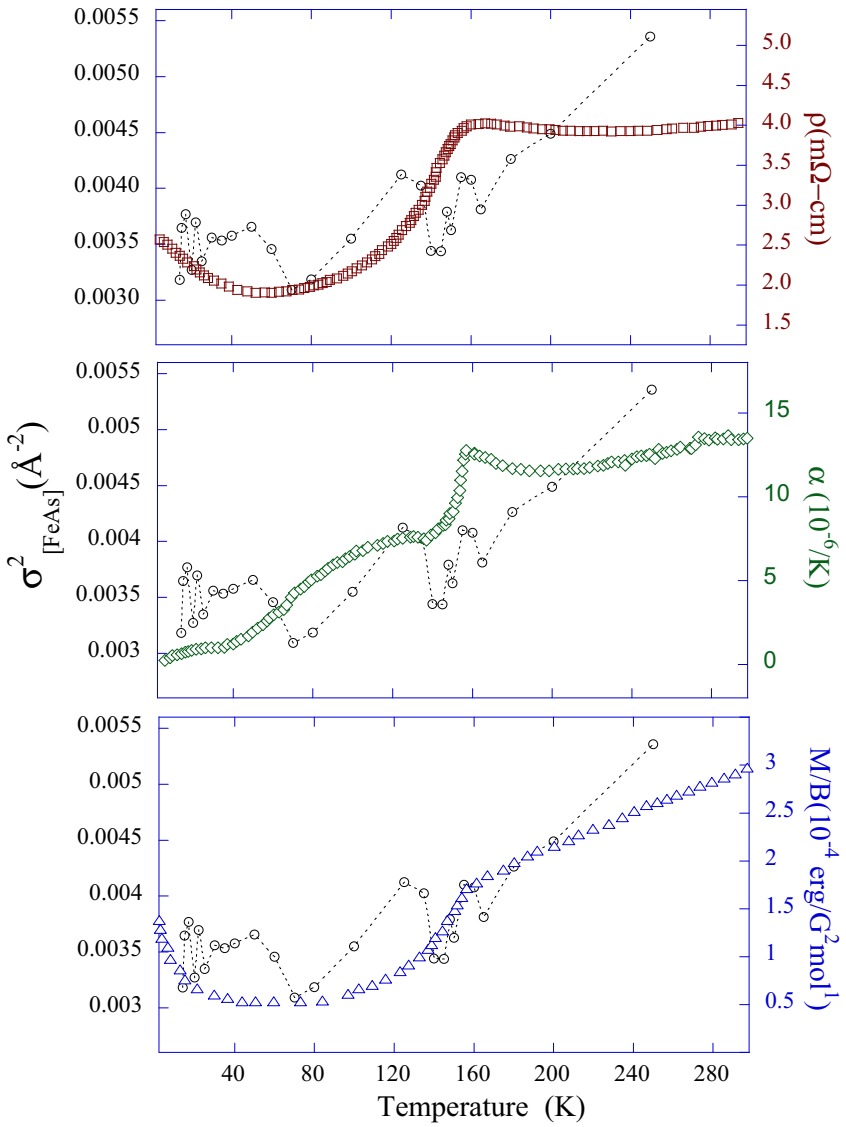

Fig. 3 Variation of the Fe–As meansquare relative displacements (MSRD) with temperature for the LaFeAsO single crystal extracted from the single shell modelling of the As K-edge EXAFS together with resistivity [49], thermal expansion [50] and magnetization [51] data reported for the same system

powders of the 1111 pnictides [9]. In Fig. 2, upper right panel, we show the variation of the intensity of the diffraction reflection corresponding to the tetragonal (220) peak with temperature for the single crystal and polycrystalline powder samples of LaFeAsO [9]. In case of the polycrystalline powder sample, the SPT is not sharp and extends over a temperature window of around 80 K [9]. Such a broad transition can mask the observation of the possible lattice anomalies of the participating bonds in the local structural measurements.

A further comparison of the temperature dependence of the Fe–As MSRD of the single crystal and polycrystalline powder (Fig. 2) shows that the MSRD of the single crystal also deviates from that of the polycrystalline powder below 60 K. In fact, several properties of the LaFeAsO show a discontinuous behaviour both at the structural phase transition and at a lower temperature below 60 K. In Fig. 3, we make a comparison of the temperature dependence of the Fe–As MSRD with the resistivity [49], thermal expansion [50] and magnetic susceptibility [51] of the LaFeAsO. Very recent angle-resolved photoemission spectroscopy (ARPES) measurements on the single crystal sample of LaFeAsO show a gap opening after the structural phase transition (SPT) [52]. The observed band shift (which happens around SPT and goes through spin-density wave (SDW) transition smoothly) is interpreted as spin-density wave fluctuation (short-range magnetic order) at the structural phase transition. Incidentally, from optical spectroscopy measurements, using single crystals [4], formation of a partial energy gap below the structural phase transition [52] and strong electron-phonon coupling were observed in the 1111 parent compounds [5]. The anomaly we observe around 150 K in the Fe–As bond correlations in LaFeAsO single crystal is mostly related to such gap-opening phenomena. It is worth recalling that the Cu–O bond length fluctuations observed in cuprate superconductors are identified as related to the pseudogap phenomena [18, 19, 21]. A recent ARPES study on CeFeAsO single crystal also showed the gap opening associated with the SPT/SDW transitions [8]. Temperature-dependent pseudogap phenomena are observed in the LaFeAsO system by laser photoemission studies [53]. The puzzling nature of magnetic and lattice phase transitions of FeSC is investigated via a first-principles Wannier function analysis of the representative parent compound LaFeAsO. A rare ferro-orbital ordering is found to result in a highly anisotropic magnetic coupling and drives both phase transitions—without resorting to a widely employed frustration or nesting picture [54].

As evident from Fig. 3, in the undoped sample, there is a clear anomaly in the MSRDs in the low-temperature regime, in addition to the SPT. This low-temperature anomaly clearly correlates with the observed low-temperature discontinuity of resistivity (Fig. 3, upper panel), thermal expansion (Fig. 3, middle panel) and magnetic susceptibility (Fig. 3, lower panel). It is possible that fluctuating domain boundaries in a nanoscale phase separation scenario in iron-based superconductors [55–58] like in cuprates [42–46] could explain the disagreement between theory and experiment in several crucial features of the FeSC.

Indeed, the magnetic ground state in the 1111 Fe systems has attracted a lot of theoretical interest [11–13, 55], and it is seen that the moment ordering of the Fe in a ferromagnetic state leads to a destruction of magnetism. In view of the above studies, it is very tempting to attribute the second anomaly observed in the Fe–As MSRD to a possible low-temperature charge density ordering in the parent compound. Interestingly, although weak in nature (considering the noise level), such an anomaly seems to be also present in the doped sample as well (Fig. 3, lower left panel). In a recent susceptibility study, the interplay of magnetism and superconductivity in $LaFeAsO_{1-x}F_x$ is clearly shown [51]. These authors show that while antiferromagnetic SDW formation is suppressed by superconductivity, the data provide strong evidence for robust local antiferromagnetic correlations persisting even in the superconducting regime of the phase diagram. The charge distribution in $RFeAsO_{1-x}F_x$ (R = La, Sm) iron pnictides probed by arsenic nuclear quadrupole resonance indicates that the undoped and optimally doped or overdoped compounds feature a single-charge environment, while a two-charge environment exists for the underdoped region [56]. From the temperature dependence of electron spin resonance in $LaFeAsO_{1-x}F_x$ ($x = 0$ and 0.13), Wu et al. suggested the existence of local moments in these materials [57]. The present results seem to support a nanoscale phase separation scenario in FeSC [25, 41, 42, 51, 56–60] which has been observed also in cuprates [42–44].

In conclusion, the temperature-dependent Fe–As bond correlations of $LaFe_{1-x}Co_xAsO$ ($x = 0$ and 0.11) single crystals were studied using the As K-edge extended X-ray absorption fine structure (EXAFS) spectroscopy which show the presence of anomalies in the Fe–As mean-square relative displacements (MSRDs) that were not seen in the corresponding polycrystalline powder sample. The anomalies of the Fe–As MSRD around 150 K in LaFeAsO single crystal are well correlated with the structural and spin-density wave-associated phase transitions. Such an anomaly is absent in the Co-doped sample where no such long-range structural or magnetic transitions exist. The absence of any anomaly in Fe–As MSRD in the polycrystalline powder samples of LaFeAsO can be understood, considering the difference in the sharpness of the phase transition in polycrystalline powder and single crystal [9]. Interestingly, both undoped and Co-doped $LaFe_{1-x}Co_xAsO$ samples show a low-temperature anomaly around 60 K, with a much weaker strength in the latter. The anomaly has a good correlation

with the temperature dependence of the resistivity magnetic susceptibility, linear thermal expansion experiments, indicating the importance of the structural effects in determining these properties. The results seem to support the phase separation scenario observed in several Fe-based superconductors [25, 41, 42, 51, 56–60].

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