Temperature-dependent local structure of NdFeAsO$_{1-x}$F$_x$ system using arsenic K-edge extended x-ray absorption fine structure

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Abstract

Local structure of NdFeAsO$_{1-x}$F$_x$ ($x = 0.0, 0.05, 0.15$ and $0.18$) high temperature iron-pnictide superconductor system is studied using arsenic K-edge extended x-ray absorption fine structure measurements as a function of temperature. Fe–As bond length shows only a weak temperature and F-substitution dependence, consistent with the strong covalent nature of this bond. The temperature dependence of the mean square relative displacements of the Fe–As bond length are well described by the correlated Einstein model for all the samples, but with different Einstein temperatures for the superconducting and non-superconducting samples. The results indicate distinct local Fe–As lattice dynamics in the superconducting and non-superconducting iron-pnictide systems.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

The role of structural topology in the magnetic and superconducting properties of the newly discovered iron-based superconductors (FeSCs) is one of the active themes of investigation [1–3] focusing on finding the mechanisms of the unconventional superconductivity. Several studies have shown unprecedented sensitivity of Fermi surface topology to the anion (pnictogen or chalcogen) height above the Fe plane in these materials. The anion height is seen to influence the density of states near the Fermi energy and to influence the electron-pairing properties of the FeSC [4–10]. In addition, local structural studies have pointed out the importance of the local order–disorder in the spacer layer in the FeSC [11, 12]. The structurally simplest systems among the FeSCs, FeTe$_{1-x}$Se$_x$, are found to have lower local structural symmetry than the average crystallographic symmetry, with the Se and Te occupying distinct sites [13, 14]. This local inhomogeneity is found to have a direct consequence on the electronic properties of the system [15]. All of this points to the importance of the diverging local structure from the average one, putting the FeSC on the same platform as cuprates, revealing lattice anomalies and local inhomogeneities [16], closely related to the superconducting properties. Some recent extended x-ray absorption fine structure (EXAFS) studies on F-doped LaFeAsO and SmFeAsO have shown a weak anomaly in the Fe–As bond length fluctuations close to the superconducting transition [17, 18]. However, such anomalies are found to be much weaker compared to those observed in the cuprates [16].

Here, we have used EXAFS, a fast and site-specific experimental tool [19, 20], to probe systematically the local structure of NdFeAsO$_{1-x}$F$_x$ oxypnictides as a function of temperature and charge density. Arsenic K-edge EXAFS has been used to retrieve direct information on the Fe–As bond lengths in superconducting ($x = 0.15$ and $0.18$) and non-superconducting ($x = 0.0$ and $0.05$) samples. The bond lengths show weak temperature and F-doping dependence, consistent with the strong covalent nature of the Fe–As bonds. The corresponding mean square relative displacements (MSRDs), well described by the correlated Einstein model, reveal different Einstein temperatures ($\Theta_E$)
for the superconducting ($T_c = 348 \pm 12$ K) and non-superconducting ($T_c = 326 \pm 12$ K) samples. The superconducting sample with maximum $T_c$ ($x = 0.18$) appears to show a weak temperature-dependent anomaly in the Fe–As MSRDs. However, the anomaly is well within the experimental uncertainties, similar to the case of SmFeAsO$_{0.931}$F$_{0.069}$. These results indicate that there exist distinct lattice dynamics in the superconducting and non-superconducting systems, albeit the differences are smaller.

2. Experimental methods

Temperature-dependent x-ray absorption measurements were performed in transmission mode on powder samples of NdFeAsO$_{1-x}$F$_x$ ($x = 0.0, 0.05, 0.15$ and $0.18$; the $x$ values given correspond to the nominal composition, the actual values are lower than the nominal values for higher $x$ [21]) at the BM26A beamline [22] of the European Synchrotron Radiation Facility, Grenoble (France). The synchrotron light emitted by a bending magnetic source was monochromatized by a double-crystal Si(111) monochromator. The samples, synthesized by the conventional solid state reaction method [21], were characterized for their structural, magnetic and superconducting properties prior to the experimental run. While the samples $x = 0.0$ and $0.05$ are non-superconducting, showing a tetragonal-to-orthorhombic structural phase transition, the samples with $x = 0.15$ and $0.18$ are superconducting with $T_c = \sim 20$ K and $\sim 40$ K, respectively, without any evidence of the structural phase transition [21]. For the temperature-dependent measurements (15–300 K), a continuous-flow He cryostat with a temperature control within an accuracy of $\pm 1$ K was used. A minimum of two scans (with high signal-to-noise ratio) were measured at each temperature to make sure of the reproducibility. Standard procedure was used to extract the EXAFS oscillations from the absorption spectrum [19, 20].

3. Results and discussions

Figure 1 shows Fourier transform (FT) magnitudes of the arsenic K-edge EXAFS oscillations for the NdFeAsO$_{1-x}$F$_x$ ($x = 0.0, 0.05, 0.15$ and $0.18$). Corresponding EXAFS oscillations are shown in the insets. The FTs are not corrected for the phase shifts, thus representing raw experimental data. A systematic temperature dependence is evident from the gradual change in the FT intensities.

In the NdFeAsO$_{1-x}$F$_x$ system, the arsenic atoms have the nearest neighbors as Fe atoms, and their contributions to the arsenic K-edge EXAFS are well separated from all other shell contributions. This makes the arsenic K-edge EXAFS data highly suitable for extracting quantitative information on the Fe–As bond lengths and the related MSRDs. Here we have exploited this possibility and performed single-shell model fits to the EXAFS oscillations due to the Fe–As bond lengths. The EXAFS amplitude depends on several factors and is given by the following general equation [19, 20]:

$$
\chi(k) = \sum_i N_i \frac{S_i^2}{kR_i^2} f_i(k, R_i)e^{-2k\sigma_i^2} \sin[2kR_i + \delta_i(k)],
$$

where $N_i$ is the number of neighboring atoms at a distance
$R_i$, $S_0^2$ is the passive electron reduction factor, $f_i(k, R_i)$ is the backscattering amplitude, $\lambda$ is the photoelectron mean free path, $\delta_i$ is the phase shift and $\sigma_i^2$ is the correlated Debye–Waller (DW) factor, measuring the MSRDs of the photoabsorber–backscatterer pairs. Apart from these variables, the photoelectron energy origin, $E_0$, is another input needed for the modeling of the EXAFS. In the present case, we have used a single-shell EXAFS modeling [19] to extract the Fe–As bond correlations. For such a single-shell analysis, we have used the WINXAS package [23], with backscattering amplitudes and phase shifts calculated using the FEFF code [24] with the crystal structure data from diffraction [21] as input. In the single-shell least-squares fit, the number of independent data points, $N_{\text{ind}} \sim (2\Delta k \Delta R)/\pi$ [19] were about 13 (\(\Delta k = 14$ Å\(^{-1}\) \((k = 3 \sim 17$ Å\(^{-1}\)) and $\Delta R = 1.5$ Å), but we used only two parameters, the radial distance $R_i$ and the corresponding MSRD $\sigma_i^2$, as the refinable variables, fixing all other parameters ($S_0^2 = 1$) to obtain reliable information on the Fe–As bond correlations. The uncertainties in the derived two parameters, $R$ and $\sigma^2$, were estimated by the standard EXAFS method [20]. The canonical approach to error estimation is to determine the region around the best fit that contains the true value with a certain probability $\beta$. The projection of that volume onto an axis corresponding to a parameter gives the parameter errors, for a chosen value of $\beta$. In general these uncertainties are dependent on the experimental as well as EXAFS data extraction procedures, in addition to the uncertainties coming from the statistical $\chi^2$-procedure. In the present case, due to the adoption of identical experimental conditions and EXAFS data extraction procedures, the first kind of uncertainties are minimal. However, we have set the error bars on derived parameters, $R$ and $\sigma^2$, to two times the highest uncertainty estimated. This is to underline the fact that we are interested in discussing the relative changes rather than the absolute values of these parameters. Figure 2 shows the single-shell model fit in the real and $k$-space at 12 K for samples with different $x$. Here it should be mentioned that the samples used in the present study are phase pure with the amount of impurities below the sensitivity of the x-ray diffraction. The main impurity phase present in the doped samples is the NdOF phase, which showed an F-doping dependence [21]. However, in the present case, this impurity phase has no influence on the arsenic K-edge EXAFS data. The impurities which can possibly interfere with the present EXAFS results, like the FeAs phases and NdAs phases, were less than 1% [21], thus ruling out any contribution from such impurity phases in the data presented here.

Figure 3 shows temperature dependence of the Fe–As bond distances determined from the single-shell analysis of the arsenic K-edge EXAFS data. The Fe–As distances in all the samples are very similar, around 2.39 Å. The overall temperature dependence of the distance also looks very similar. However, one can notice a small change in the superconducting samples somewhat close to the transition temperature, with
no such changes evident in the parent compound. An earlier temperature-dependent x-ray diffraction study on the NdFeAsO_{1−x} superconductor has shown an abrupt change in the Fe–As distance around T_c [27]. Present results also show that the Fe–As distance tend to change near T_c for the superconducting (x = 0.15, 0.18) samples (figure 3 upper panels). But we do not see significant changes in the Fe–As bond lengths or corresponding MSRD across the structural phase transition for the parent compound, which is consistent with earlier EXAFS studies on LaFeAsO [17, 28], SmFeAsO [18] and BaFe_2As_2 [29], showing no change in the Fe–As bond length and corresponding MSRD across the structural phase transition. As shown in a recent diffraction study of the REFeAsO (RE stands for rare-earth) system, data from single crystals are important for a better understanding of structural phase transition properties [25]. For example, a recent study on the NdFeAsO single crystal revealed two low temperature phase transitions in addition to the tetragonal-to-orthorhombic transition at T_o ∼ 142 K [26], indicating the importance of single crystals in revealing the intricate properties of FeSCs. Naturally EXAFS studies using single crystals are also going to be important, in addition to the angular-dependent local information provided by the polarization-dependent EXAFS measurements [16].

Figure 4 shows mean square relative displacements (σ^2) of the Fe–As pair, describing the distance–distance correlation function (correlated DW factors). The MSRD is a sum of temperature-independent (σ_0^2) and temperature-dependent terms [19], i.e.

\[
\sigma_{Fe-As}^2 = \sigma_0^2 + \sigma_{Fe-As}(T).
\]

In several cases, the temperature-dependent term can be described by the correlated Einstein model [19, 20]:

\[
\sigma_{Fe-As}^2(T) = \frac{h}{2\mu_{Fe-As}\omega_E} \coth \left( \frac{\hbar \omega_E}{2k_B T} \right),
\]

where μ_{Fe-As} is the reduced mass of the Fe–As bond and ω_E is the Einstein frequency. The related Einstein temperature can be obtained from the expression θ_E = hω_E/κ_B. In the present case, temperature dependence of σ_{Fe-As}^2 is found to follow the correlated Einstein model. The description of σ_{Fe-As}^2 using this model yields θ_E values to be 348 ± 12 K and 326 ± 12 K, respectively, for the x = 0.18 and 0.0 samples. Notice that the uncertainty in θ_E given above is obtained from the least-squares fit considering the error bars on the data points. The obtained Einstein frequencies are in the range of phonon frequencies observed in Raman studies for the modes involving arsenic and iron atoms [31, 32]. In addition, different Einstein frequencies (ω_E) indicate different local force constants (k = μ_{Fe-As}ω_E^2) for the Fe–As bonds in the superconducting and non-superconducting samples. The calculated local force constants for the Fe–As bonds are ~6.65 eV Å^−2 and ~5.85 eV Å^−2, respectively, for the superconducting and non-superconducting samples. Thus the Fe–As bond length seems to get harder in the superconducting regime. This observation is an indication of the non-negligible role of the lattice modes in the superconductivity of these materials.

Comparing the already available local structural data on different oxypnictides [17, 18, 28, 29, 33, 34] one can see that the Fe–As bond in these systems shows only small changes with doping and temperature. An EXAFS study on a series of oxypnictides [33] has shown that the Fe–As bond length and the related MSRDs hardly show any change with the varying rare-earth size, consistent with the strong covalent nature of the Fe–As bonds. On the other hand, earlier studies on F-doped La-1111 [17] and Sm-1111 [18] compounds have shown the presence of an anomaly in the temperature dependence of the Fe–As MSRDs. But these anomalies were seen to be very weak compared to those observed for the cuprates [16]. Indeed another study on the superconducting La-1111 system [28] did not indicate any such anomaly. Such anomalies were also not seen in the K-doped Ba-122 system [29] and F-doped Ce-1111 system [30]. From the results presented in figure 4, for the F-doped Nd-1111 system, there is no evidence for a significant anomaly in the MSRD of Fe–As bonds associated with the superconducting transition. Here, we make an explicit comparison of the Fe–As MSRDs of the superconducting Sm-1111 and Nd-1111 samples to underline that the Fe–As MSRDs tend to change around the superconducting transition
temperature, albeit the anomalies are indeed weak compared to what has been seen in the copper oxide superconductors [16].

Figure 5 shows the temperature dependence of the MSRDs of the Fe–As bond for the NdFeAsO, compared with that of the LaFeAsO [28], SmFeAsO [18] and BaFe$_2$As$_2$ [29]. In all cases, the MSRD data are extracted from the arsenic K-edge EXAFS. The correlated Einstein model fit to the data is also indicated in the figure (as dotted lines). From figure 5, it can be seen that the Fe–As MSRDs show almost identical temperature dependence for these different oxypnictide systems. Although the temperature dependence looks very similar, there seems to be a systematic change in the Einstein temperature. The $\Theta_E$ values are, respectively, 316 ± 5 K, 318 ± 10 K, 326 ± 12 K and 328 ± 12 K for LaFeAsO, BaFe$_2$As$_2$, NdFeAsO and SmFeAsO. There is a clear increase in the force constant of the Fe–As bonds, with increasing rare-earth size in the ‘1111’ series. Optical studies using single crystals of the ‘1111’ series reveal that the Fe–As stretching mode in these oxypnictides shows substantial hardening with the rare-earth size, being harder for the NdFeAsO and SmFeAsO showing higher $T_c$ in comparison to the LaFeAsO [35]. The $E_u$ mode frequency for the BaFe$_2$As$_2$ [36] is also found to be similar to that of the ‘1111’ series [35]. In the ‘1111’ family, the changing rare-earth ionic size leads to a change in the pnictogen height above the Fe plane [33] and thus a different interlayer atomic correlation [11, 12]. Local structural studies using atomic pair distribution function analysis on Ba$_{1-x}$K$_x$Fe$_2$As$_2$ show that, although the changes in the Fe–As bonds are minimum, the FeAs$_4$ tetrahedra show a systematic evolution with K doping [34]. Indeed a small change in the pnictogen height is known to have a significant effect on the electronic properties of the system through changing degeneracy between different Fe 3d bands (in particular between the $d_{x^2}$ and $d_{yz}$, $d_{zx}$), with a direct implication on the magnetic structure and superconductivity [8, 10]. Very recent photoemission studies have shown this pnictogen-height-dependent change in the electronic structure [37], confirming the importance of even very small local structural changes in determining the properties of the FeSCs.

To further enlighten the possible anomaly around the superconducting transition temperature, we have made an explicit comparison between the temperature-dependent Fe–As MSRDs for the F-doped highest $T_c$ Nd-1111 and Sm-1111 [18] superconductors (see, e.g., figure 5(d)). The inset in figure 5(d) is a zoomed view around $T_c$. The error bars given here are identical to those given in reference [18]. For the NdFeAsO$_{0.93}$F$_{0.07}$ sample, there is a weak anomaly in the temperature dependence of the Fe–As MSRDs similar to that observed in the superconducting SmFeAsO$_{0.93}$F$_{0.07}$ [18]. However, the anomaly is well within the experimental uncertainties. This situation is different from that of cuprates [16] where several measurements showed clear lattice anomalies associated with the superconducting transition. Although more experiments are needed, the weakness of the anomalies does not imply that lattice fluctuations have a minor role in the Fe-based superconductors. Indeed, the empirical relationship between the superconducting transition temperature and the FeAs$_4$ tetrahedra in FeSCs [4–6] clearly establishes the role of the local lattice in the superconducting properties. Local structural studies also underline the same point [33, 34]. The increased force constant of the Fe–As bonds in the superconducting sample compared to the parent compound in the Nd-1111 system indicate the importance of lattice dynamics in determining the properties. Systematic changes in the Fe–As force constant within the different iron-pnictide compounds, as revealed by the present study, further support this.

4. Conclusions

In conclusion, we have carried out a systematic temperature-dependent local structural studies of the NdFeAsO$_{1-x}$F$_x$
(x = 0.0, 0.05, 0.15 and 0.18) high temperature pnictide superconductor using arsenic K-edge EXAFS. In all the samples, the temperature dependence of the MSDRs of the Fe–As bonds are found to follow the correlated Einstein model. However, the Einstein frequency for the superconducting sample is higher than that of the parent compound, indicating a hardening of the Fe–As bond in the former compared to the sample is higher than that of the parent compound, indicating a hardening of the Fe–As bond in the former compared to the latter. The overall temperature dependence of the MSDRs of the Fe–As bond seems to be similar in NdFeAsO, SmFeAsO, LaFeAsO and BaFe$_2$As$_2$, but with a systematic variation of the Fe–As bond seems to be similar in NdFeAsO, SmFeAsO, LaFeAsO and BaFe$_2$As$_2$, but with a systematic variation of the corresponding force constants. The changes occurring in the FeAs$_4$ tetrahedra, together with the coupling between the active layer and the spacer layer, may account for the changes in properties of different iron oxypnictide superconductors.

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