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Abstract
^{57}Fe Mössbauer spectroscopy has been used to investigate the magnetic order of non-superconducting NdFeAsO ($T_N = 140$ K) and superconducting NdFeAsO$_{0.88}$F$_{0.12}$ ($T_c = 45$ K). A magnetic hyperfine field B_{hf} was observed at the ^{57}Fe nucleus below $T_N \sim 140$ K for NdFeAsO. Below $\sim$2 K an increase of B_{hf} relative to the saturation value was attributed to the transferred B_{hf} at the Fe site resulting from the collinear antiferromagnetic (AF) spin structure of the Nd moments. The analysis of the spectra is consistent with a commensurate AF order of Fe spins. No B_{hf} is observed in superconducting NdFeAsO$_{0.88}$F$_{0.12}$ down to 1.5 K.

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

1. Introduction

Since the discovery of superconductivity at 26 K in LaFeAsO$_{1-x}$F$_x$ [1] a large effort has been made to understand their crystallographic, magnetic and superconducting properties [2–13]. This is mainly due to the subsequent rising of T_c up to $\sim$55 K with substitution of La by other rare earths RFeAsO$_{1-x}$F$_x$ (R = Sm, Ce, Nd, Pr or Gd). Although the parent compounds are not superconductors they show interesting low temperature structural and magnetic orderings [2–18]. The LaFeAsO undergoes an abrupt structural distortion below $\sim$155 K and at $\sim$137 K the compound develops a long range SDW-type antiferromagnetic (AF) order with a small Fe moment and simple magnetic structure [2, 5, 6]. The parent compounds of the new superconductors with magnetic rare earths have an electronic structure similar to that of LaFeAsO, with all five d orbitals contributing to a low density of states at the Fermi level [20–24]. They have the same structural distortions, going from a tetragonal to an orthorhombic phase with critical temperature depending on the rare earth. Moreover at lower temperatures they exhibit a stripe-like commensurate AF ordering of the iron spins [2, 10, 12, 14–17]. The rare earth moments order magnetically even at lower temperatures (below 14 K) [10, 12, 14–18].

The NdFeAsO compound studied in this work displays an orthorhombic distortion below $\sim$150 K [15]. The iron spins order at 141 K with the same in-plane spin configuration as was found for all the parent compounds [16] and the Nd spins order below 2 K [15]. Electron doping the system with fluorine suppresses the structural distortion and static long range AF order, favoring superconductivity [2–13]. Competing orders is the one of the main features of these layered compounds [19]. Several experimental techniques are being used to study these unusual systems. Mössbauer effect (ME) spectroscopy is one of the methods which gives relevant information about the presence of impurities in the samples, the determination of the Fe moments and their ordering in parent compounds, and the absence of magnetism in fluorine doped compounds [5, 25, 26]. However until now there have been no ME systematic studies as a function of temperature to
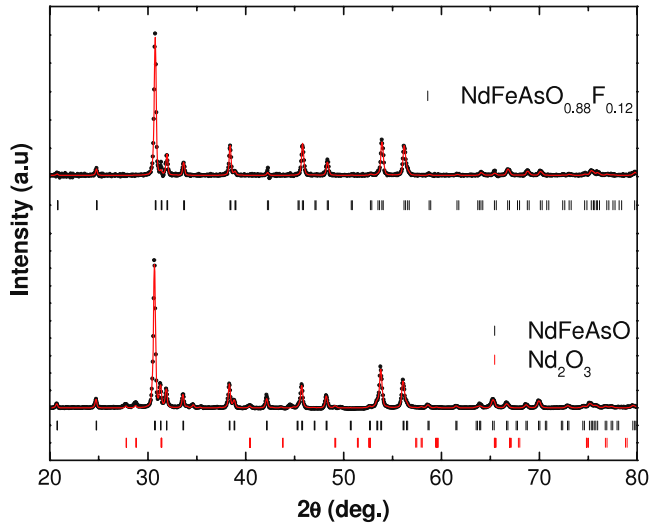
Figure 1. Rietveld plots of NdFeAsO$_{0.88}$F$_{0.12}$ and NdFeAsO at $T = 295$ K. The observed data points are indicated by closed circles, while the calculated patterns are shown by solid lines. The bars at the bottom show the calculated Bragg diffraction positions of the phases. No impurity is detected for the NdFeAsO$_{0.88}$F$_{0.12}$ sample. For the NdFeAsO sample three small impurity phases (Nd$_2$O$_3$, FeAs and FeAs$_2$) were included in the analysis of the x-ray data. The Bragg peaks of Nd$_2$O$_3$ ($\sim$5 wt%) are indicated while those for FeAs and FeAs$_2$ and phases below 5% were omitted.

help to elucidate their magnetic structure. We have shown that ^{57}Fe ME spectroscopy can be a powerful tool in the study of the spin structure and the interplay between superconductivity and magnetism in the RNi$_2$B$_2$C magnetic superconductors wherein the Fe substitutes Ni with a negligibly small magnetic moment [27, 28]. This method can be applied to study the new kind of superconductor with the advantage that, in this case, the Fe is a constituent element of the system. We use ME to study the structural and magnetic phase transition in polycrystalline non-superconducting NdFeAsO and superconducting NdFeAsO$_{0.85}$F$_{0.12}$ compounds. For NdFeAsO we find that Fe spins order magnetically below $\sim$140 K and the analysis of the low temperature spectra is consistent with a commensurate AF structure for Fe spins. At 1.5 K a significant increase of the hyperfine field was observed and related to a commensurate AF structure for Nd spins below $\sim$2 K. No hyperfine magnetic field (B_{hf}) or change in the electrical quadrupole splitting (ΔE_Q) was observed at the ^{57}Fe nucleus in superconducting NdFeAsO$_{0.88}$F$_{0.12}$ at any temperature. This result is consistent with the absence of magnetic order and suppression of structural distortion.

2. Experimental details

Polycrystalline samples of NdFeAsO and NdFeAsO$_{0.88}$F$_{0.12}$ were synthesized at Beijing National Laboratory for Condensed Matter Physics following the methods published elsewhere [8–11]. In addition, the samples were characterized by magnetization and powder x-ray diffraction (XRD) methods with Cu Kα radiation at room temperature. The magnetization measurements show a clear magnetic order below $\sim$140 K for
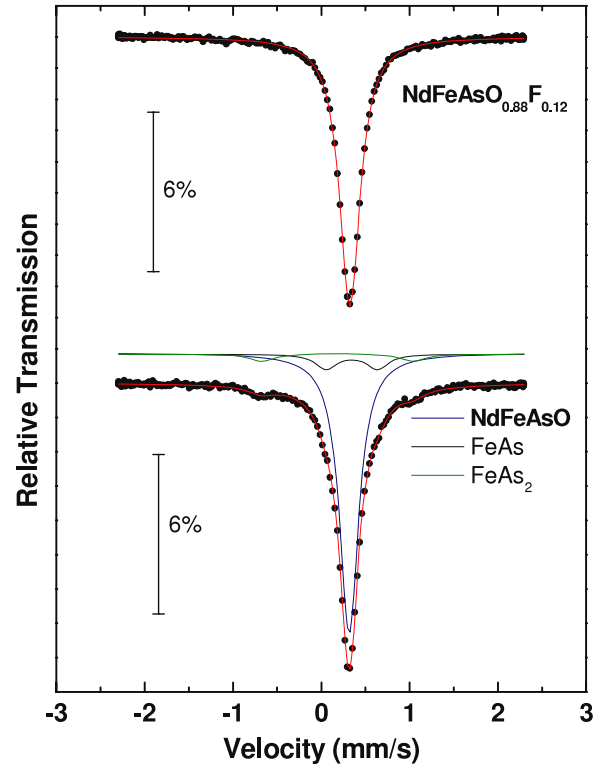

Figure 2. ^{57}Fe Mössbauer spectra for NdFeAsO$_{0.88}$F$_{0.12}$ and NdFeAsO at 295 K. For NdFeAsO the three subspectra are shown explicitly.

pure NdFeAsO and a superconducting transition temperature $T_c = 40$ K for fluoride doped NdFeAsO$_{0.88}$F$_{0.12}$.

^{57}Fe ME spectroscopy experiments were performed in the temperature range 1.5–300 K with the samples in a variable temperature Oxford helium cryostat and the ^{57}Co:Rh source, kept at the absorber temperature, moving in a sinusoidal mode inside the cryostat.

3. Results and discussion

The powder x-ray patterns for NdFeAsO$_{0.88}$F$_{0.12}$ and NdFeAsO compounds are shown in figure 1. The spectra are well indexed on the basis of the tetragonal ZrCuSiAs-type structure with the space group $P4/nmm$ having lattice parameters $a = b = 3.961$ Å, $c = 8.555$ Å for NdFeAsO$_{0.88}$F$_{0.12}$ and $a = b = 3.969$ Å, $c = 8.580$ Å for NdFeAsO, in agreement with the values found in the literature [11]. This Rietveld analysis showed that NdFeAsO$_{0.88}$F$_{0.12}$ and NdFeAsO are nearly single phase and only impurities of Nd$_2$O$_3$, FeAs and FeAs$_2$ below 5% are inferred for the parent compound while for the F doped compound no signal of impurity phases was found. Such impurities were also found in similar compounds and are inherent to the preparation method [1, 25, 26, 29].

The room temperature (RT) ^{57}Fe ME spectra for NdFeAsO and NdFeAsO$_{0.88}$F$_{0.12}$ compounds are compared in figure 2. The RT NdFeAsO$_{0.88}$F$_{0.12}$ spectrum can be fitted with only one doublet, with the hyperfine parameters quadrupole splitting $\Delta E_Q = 0.02(2)$ mm s^{-1}, isomer shift
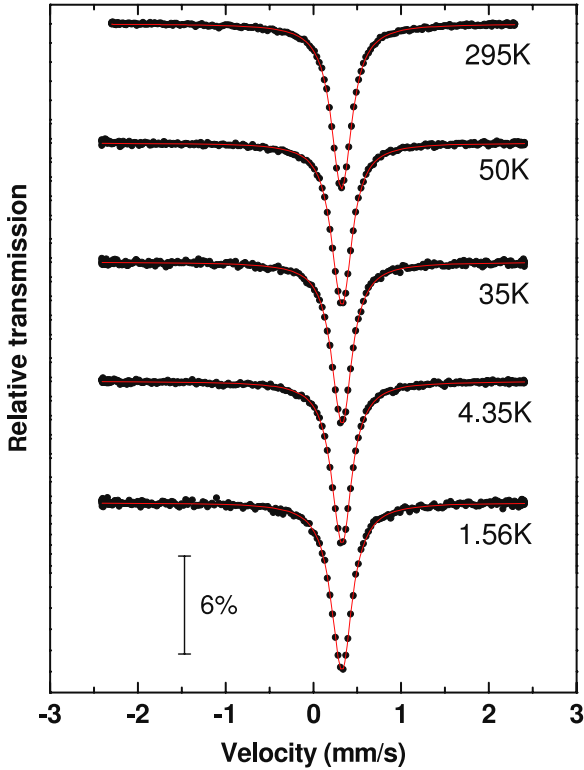
Figure 3. ^{57}Fe Mössbauer spectra of NdFeAsO$_{0.88}$F$_{0.12}$ in temperature range of 295 K $\leqslant T \leqslant$ 1.56 K.

Figure 4. ^{57}Fe Mössbauer spectra of NdFeAsO in the temperature range of 150 K $\leqslant T \leqslant$ 1.5 K. The three components used to fit the spectra are explicitly shown. The smaller components corresponding to impurities were omitted.

$\delta_{\text{IS}} = 0.437(1)$ mm s^{-1} and linewidth $\Gamma = 0.275(3)$ mm s^{-1}, indicating a unique phase for Fe. The NdFeAsO spectrum needs three quadrupole doublets to be analyzed. The main doublet ($\sim$87% of total absorption area (A)) has hyperfine parameter $\Delta E_Q = 0.00(1)$, $\delta_{\text{IS}} = 0.429(2)$ mm s^{-1} and $\Gamma = 0.265(2)$ mm s^{-1}, very similar to the ones for NdO$_{0.88}$F$_{0.12}$FeAs, and attributed to Fe in the NdFeAsO phase. The δ_{IS} ($\sim$0.44 mm s^{-1}) values found for Fe in these two compounds are typical for low or intermediate spin Fe(II). The other two additional weak doublets were identified as FeAs ($\Delta E_Q = 0.582(1)$, $\delta_{\text{IS}} = 0.46(2)$, $A \sim$ 8%) and FeAs$_2$ ($\Delta E_Q = 1.71(3)$, $\delta_{\text{IS}} = 0.29(2)$, $A \sim$ 5%) impurities also observed by means of x-ray diffraction. It is important to mention that both doublets due to the impurity phases were taken into account in all the low temperature ^{57}Fe ME spectra analysis. A magnetic transition below 77 K occurs in FeAs [30] while FeAs$_2$ remains paramagnetic down to 1.5 K [31].

Cooling the NdFeAsO and NdFeAsO$_{0.88}$F$_{0.12}$ samples down to 1.5 K results in quite different ^{57}Fe ME spectra for both compounds and for this reason in the following we will discuss each of them separately.

The ^{57}Fe ME spectra of NdFeAsO$_{0.88}$F$_{0.12}$ taken at different temperatures are compared in figure 3. Down to 1.5 K there is no obvious difference: the isomer shift, linewidth and quadrupole splitting values are almost the same within the experimental errors. The hyperfine parameters agree within the experimental errors: $\Delta E_Q = 0.03 \pm 0.01$, $\delta_{\text{IS}} = 0.437 \pm 0.002$ and $\Gamma = 0.279 \pm 0.006$. Therefore, no hyperfine magnetic field (B_{hf}(1.5 K) $\leqslant$ 0.1 T) and no structural
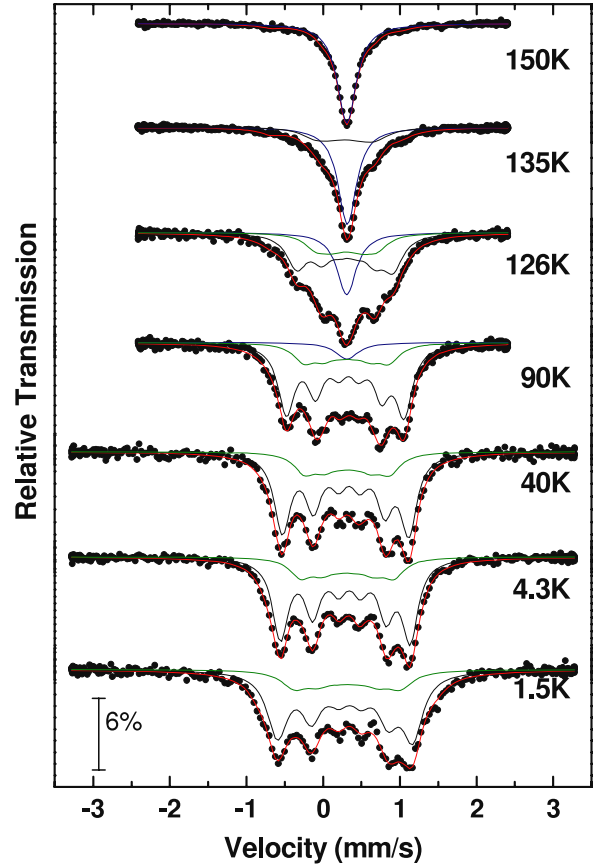
change were observed at the ^{57}Fe nucleus in superconducting NdFeAsO$_{0.88}$F$_{0.12}$ from 295 to 1.5 K. Similar behavior has been observed for the superconductors LaFeAsO$_{0.89}$F$_{0.11}$ [32] and NdFeAsO$_{0.82}$F$_{0.18}$ [33].

NdFeAsO is known to display magnetic order below 140 K [16] and a long range SDW-type AF order was suggested for these parent compounds [2, 9, 11]. As is evident from the ^{57}Fe ME spectra shown in figure 4, a fraction of the paramagnetic phase (singlet) begins to broaden below $\sim$140 K indicating the onset of the magnetic transition. Since the B_{hf} field is small it was necessary to analyze the spectra with the full hyperfine Hamiltonian, including electrical quadrupole and magnetic interactions. Furthermore, the following assumptions have been made: (1) the quadrupole asymmetry parameter $\eta = 0$, as is expected for ^{57}Fe with a regular symmetry; and (2) the absence of spin texture in the polycrystalline powder sample.

Several models can be used to fit the spectra below $T_{\text{N}} \sim$ 140 K and the simplest one involves a magnetic hyperfine field distribution. This analysis works very well for all the spectra and is compatible, in principle, with an incommensurate SDW structure for the NdFeAsO below T_{N}. Such an incommensurate SDW should lead to a very broad hyperfine field distribution ranging from zero to a maximum field value; however for temperatures lower than 100 K this does not occur and the

smaller magnetic field components disappear. So, this model is not appropriate for explaining the magnetic structure of Fe ions in this compound.

It was recently suggested that such a spectrum could be analyzed as a superposition of commensurate SDW with a minor component of an incommensurate field distribution due to oxygen deficient phases [29]. This model seems to work well for temperatures just below the magnetic transition temperature T_N and precludes the inclusion of any paramagnetic component. However μSR experiments on NdFeAsO have revealed a remaining paramagnetic component below T_N which gradually vanishes for lower temperatures [34]. So, this model does not agree with μSR results. Furthermore, for lower temperatures where the paramagnetic fraction vanishes, the smaller magnetic field components also disappear. As discussed above, an attribution of an incommensurate SDW, even for a minor oxygen deficient component, is not possible through this model. As we see later, an incommensurate SDW for a minor oxygen deficient component could be analyzed in terms of the angle between B_{hf} and V_{zz} rather than a hyperfine field distribution.

An analysis considering two magnetic components is also possible. Although this model works well for lower temperatures, it leads to large values of the quadrupole splitting (0.7 mm s^{-1}) between 140 K $\leqslant T \leqslant$ 100 K. Recent Mössbauer studies of LaFeAsO [5] have shown an increase of $\sim$0.3 mm s^{-1} for the quadrupole splitting due to a small deformation of the FeAs$_4$ tetrahedron below the structural phase transition. Therefore this model was excluded because such a large value for the quadrupole splitting is not consistent with the orthorhombic distortion observed in such compounds.

In this work we analyzed the spectra below 140 K using three components: two magnetic (sextets) and one paramagnetic (singlet). Between 140 K $\leqslant T \leqslant$ 120 K the spectra were fitted with one sextet and one singlet; below 120 K a second sextet was added. The paramagnetic fraction is observed to decrease gradually below T_N with a consequent increase of the magnetic fraction area (see figure 5(b)). This model is consistent with recent μSR measurement for a NdFeAsO sample prepared by the same group [34].

Below 120 K the ΔE_Q, δ_{IS}, sign of V_{ZZ} (the main component of the electrical field gradient tensor), B_{hf} field, and angle θ between the directions of B_{hf} and V_{ZZ} were the free fitting parameters. This analysis yields to the following results: $\Gamma = 0.285(2)$ mm s^{-1}, $\Delta E_Q = 0.07(2)$ mm s^{-1}, $\delta_{IS} = 0.44(1)$ mm s^{-1}, $V_{ZZ} > 0$, $\theta \approx 90° \pm 5°$ for the main component and a temperature dependent B_{hf} (from $B_{hf} \sim 0$ T at 140 K to $B_{hf} \sim 5.2$ T at 4.2 K). The minor broadened magnetic component spectra ($\sim$18% of absorption area) were analyzed, keeping ΔE_Q and δ_{IS} fixed at the same values of the main component leading to $\theta \approx 55° \pm 7°$. The temperature dependence of B_{hf} is shown in figure 4(a) and the onset of magnetic ordering at $T_N \sim 140$ K is clearly seen. The magnetization curve seems to reach saturation at 4.2 K; however a jump in the B_{hf} value for both components is observed at 1.5 K and can be attributed to B_{hf} being transferred due to the order of the Nd moments. The variation of the ΔE_Q values obtained from the analysis shows a clear increase below $\sim$138 K, reflecting the structural phase transition.
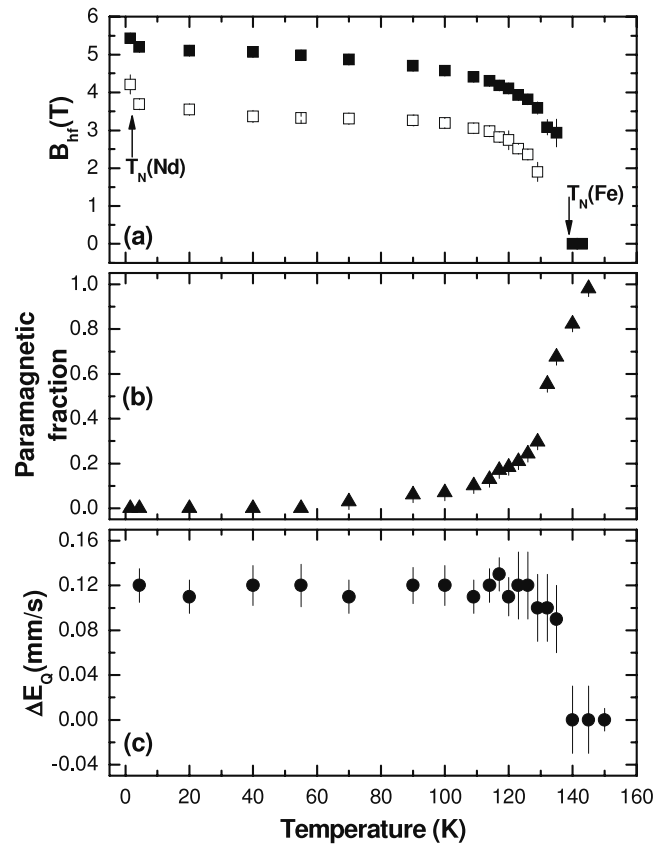

Figure 5. (a) Temperature dependence of the magnetic hyperfine field B_{hf} of ^{57}Fe in NdFeAsO. Full squares correspond to Fe in the NdFeAsO phase and open squares to a minor oxygen deficiency phase. The magnetic orderings of Fe and Nd spins are indicated. (b) Fraction of Fe in a paramagnetic state estimated from the resonant absorption area. (c) Temperature dependence of the quadrupole splitting $|\Delta E_Q|$ for NdFeAsO reflecting the structural phase transition at $\sim$138 K.

Now we will discuss why the ^{57}Fe ME spectrum at low temperatures varies strongly with the F doping for the different NdFeAsO$_{1-x}$F$_x$ samples. Our experiments show that there exists *no* measurable magnetic hf field ($B_{hf} < 0.1$ T) at the ^{57}Fe nucleus in NdFeAsO$_{0.88}$F$_{0.12}$ down to 1.5 K. This can be explained as a result of fluorine doping, i.e., doping with carriers the two-dimensional Fe–As conducting layer and compressing the lattice, a strong covalence of Fe and As atoms seems to occur. This strong covalence could lead to a delocalization of the spin density from the Fe 3d to adjacent As atoms, reducing the moment, and consequently could vanish the B_{hf} at the iron nucleus. In addition, Nd moment ordering can contribute to the magnetic hyperfine field at the ^{57}Fe nucleus (through a B_{hf} transferred from the Nd–O layer to the Fe layer) as seen for NdFeAsO in figure 5(a). The absence of B_{hf} indicates that either the Nd moments do not order even at 1.5 K or they order in a collinear AF structure such that the transferred magnetic field cancels at the Fe site. Neutron study of the superconductor NdFeAsO$_{0.80}$F$_{0.20}$ did not reveal any magnetic phase transition down to 1.5 K [15]. Thus, our Mössbauer results support the absence of magnetic ordering of Nd moments in NdFeAsO$_{0.88}$F$_{0.12}$ down to 1.5 K.

Furthermore, the quadrupole splitting remains constant down to 1.5 K showing no signal for structural transitions. This compound becomes superconducting below $T_c \sim 45$ K and no indications of magnetic order or structural transitions have been observed by Mössbauer spectroscopy. So, doping the system with fluorine suppresses the Fe moments, magnetic order and structural distortion, favoring superconductivity.

For NdFeAsO, however, the situation is quite different: we observe a magnetic B_{hf} field below $T_N \sim 140$ K (see figure 5(a)) which is related to the Fe spin ordering. Additional conclusions about the spin structure in NdFeAsO can be drawn from the measured angle θ between the directions of V_{ZZ} and B_{hf}. Another system structurally closely related to RFeAsO is the RNi$_2$B$_2$C borocarbides, whose tetragonal structure can be viewed as a stacking of Ni$_2$B$_2$ layers spatially separated by R–C planes. In borocarbides the B atoms are placed around the Fe atoms in a tetrahedral arrangement, like the As atoms around the Fe in NdFeAsO. Mössbauer studies of borocarbides [27, 28] established that the direction of V_{ZZ} has to be a c direction. From Mössbauer measurements on CaFe$_2$As$_2$ single crystal [35], which has the same local structure around Fe, we found that the V_{ZZ} direction is along the c axis. For NdFeAsO (main component) we find $\theta \approx 90° \pm 5°$. Having established that V_{ZZ} is in the c direction we can conclude that for NdFeAsO, B_{hf} lies in the (a, b) plane. Such a field direction is expected for a commensurate AF spin structure, for example with the Fe moments in the (a, b) plane. This result is in complete agreement with the neutron studies for NdFeAsO where an AF structure was found below T_N with the Fe moments pointing in the a direction [16].

The isomer shift δ_{IS} does not change with the temperature; however below 140 K a slight but clear increase in the quadrupole splitting ΔE_Q is observed (figure 5(c)). The ΔE_Q value changes from 0.01 mm s^{-1} at 145 K to 0.12 mm s^{-1} at 126 K, keeping this value for lower temperatures. The change in ΔE_Q, close to the temperature of the magnetic transition, indicates a structural transition concomitant with the magnetic ordering, as observed from neutron measurements.

A secondary magnetic component also appears below 140 K having a magnetic hyperfine field $B'_{hf}(T) \sim \frac{1}{\sqrt{2}}B_{hf}(T)$, where $B_{hf}(T)$ is the value corresponding to the main component. The angle θ is found to be $55° \pm 7°$; this is either just the so-called 'magic angle' ($\theta \sim 55°$) or is not well defined, i.e. all angles in the range $0 \leqslant \theta \leqslant 90$ occur with equal probability [28]. The last statement means a random angle between the magnetic moment direction and the c axis (the V_{ZZ} direction) and compatible with a spiral or a not well defined spin magnetic structure for the Fe in this phase. Considering that the reduced B'_{hf} is just the root mean square of a hypothetical sinusoidal function of B_{hf}, we can propose a kind of spiral commensurate SDW structure for Fe moments in this minor phase. On the other hand, this secondary component has a small fraction ($\sim$18%) and is rather broadened ($\Gamma = 0.35$ mm s^{-1}), indicating a slightly inhomogeneous magnetic state, which could be caused by small variations of the oxygen content. Furthermore, it was found that oxygen deficiency in this kind of compound leads to a disappearance of magnetism and gives rise to the establishing

of a superconducting state [36–39]. So, a reduction (and disappearance) of B_{hf} at the Fe nucleus is expected for oxygen deficient samples [40]. We want to point out that this minor component is not observed at room temperature because the paramagnetic Mössbauer spectra of pure NdFeAsO and of their corresponding oxygen deficiency phase are indistinguishable, i.e. the two phases have almost the same electrical hyperfine parameters (δ_{IS} and ΔE_Q) at room temperature. Therefore, considering that no impurities other than FeAs and FeAs$_2$ were detected by means of x-ray diffraction, we attribute this minor component, with a reduced hyperfine magnetic field, to the Fe in an oxygen deficiency phase.

For the Fe in the NdFeAsO phase, B_{hf} increases as the temperature decreases below T_N, reaching a value of about 5.2 T at 4.2 K (see figure 5(a)). Below 4.2 K a significant increase ($\sim$0.2 T) in B_{hf} is observed. This increment is of the same order of magnitude of the transferred hyperfine field, via RKKY or dipolar interaction, from ordered rare earth moments to the Ni site in borocarbides [27, 28]. Therefore the increase of B_{hf} at the temperature where the Nd moments order, as seen from neutron diffraction [15], is attributed to a transferred magnetic hyperfine field, from Nd moments. At the same time it is known that superconductivity does not appear at all in this compound [11]. From the value of B_{hf} (4.2 K) = 5.2 T we can estimate a magnetic moment for the Fe in NdFeAsO as 0.35 μ_B. These hyperfine parameters are in very good agreement with those reported in [5, 25, 26, 41] for LaFeAsO.

4. Conclusion

For non-superconducting NdFeAsO a magnetic hyperfine field B_{hf} was observed at the ^{57}Fe nucleus below $T_N \sim 140$ K indicating the onset of the magnetic transition. The analyses of the low temperature Mössbauer spectra are consistent with a commensurate AF structure with the Fe spins lying in the (a, b) plane. A slight change in the ΔE_Q values around 140 K indicates a structural transition concomitant with the magnetic transition for this compound. Below $\sim$2 K an increase of B_{hf}, relative to the saturation value at $\sim$4.2 K, was observed and this is attributed to the transferred B_{hf} at the Fe site resulting from the collinear AF spin structure of the Nd moments. On the other hand, no changes in ΔE_Q or B_{hf} were observed in superconducting NdFeAsO$_{0.88}$F$_{0.12}$ down to 1.5 K. These results indicate that both magnetic and structural transitions are suppressed by fluorine doping, favoring the establishing of a superconducting state.

Acknowledgments

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