Coexistence of Superconductivity and Magnetism in LaFeAs(O$_{0.94}$F$_{0.06}$) Probed by Muon Spin Relaxation

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The presence of macroscopic phase separation into superconducting and spin-glass-like magnetic phases in LaFeAs(O$_{1-x}$F$_x$) is demonstrated by muon spin rotation measurement in a sample near the phase boundary ($x = 0.06$). Both magnetism and superconductivity develop simultaneously below a common critical temperature, $T_m \simeq T_c \simeq 18$ K. This remarkable accordance strongly suggests that the electronic correlations leading to these two competing ground states share a common origin.

KEYWORDS: oxypnictide superconductor, muon spin rotation, electronic correlation, magnetism

The recent discovery of the oxypnictide superconductor LaFeAsO$_{1-x}$F$_x$ (LFAO-F) with a critical temperature ($T_c$) of 26 K$^1$ and the successful revelation of much increased $T_c$ upon the substitution of La for other rare-earth elements (such as Sm, leading to $\sim$ 43 K$^2$) and the application of pressure for LFAO-F ($\sim$ 43 K$^3$) have triggered broad interest in the mechanism yielding a relatively high $T_c$ in this new class of compounds. They have a layered structure like high-$T_c$ cuprates, where the dopant and conducting layers are so separated that the doped carriers (electrons introduced by the substitution of O$^{2-}$ with F$^-$ in the La$_2$O$_2$ layers) move within the layers consisting of strongly bonded Fe and As atoms. They exhibit another qualitative similarity to cuprates in that superconductivity occurs upon carrier doping of pristine compounds that exhibit magnetism.$^4$ Some preliminary results of the muon spin rotation/relaxation (µSR) experiment on a variety of oxypnictide superconductors showed that the superfluid density $n_s$ falls on the empirical line on the $n_s$ vs $T_c$ diagram observed for

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the *underdoped* cuprates,\textsuperscript{5,6} from which possibility of the common mechanism of superconductivity is argued between oxypnictides and cuprates.

However, in terms of the doping phase diagram, there are certain differences between these two systems, e.g., (i) \( T_c (> 0 \) for \( 0.4 < x < 0.12 \)) does not vary much with \( x \) as in cuprates known as “bell-shaped” and (ii) the magnetic (spin density wave, SDW) phase shares a boundary with the superconducting phase near \( x \simeq 0.04 \).\textsuperscript{1,7} The insensitivity of \( T_c \) to \( x \) is reasonably understood from the conventional BCS theory where condensation energy is predicted to be independent of carrier concentration. The close relationship of magnetism and superconductivity suggests that a detailed investigation of how these two phases coexist (and compete) near the phase boundary will provide important clues to elucidating the paring mechanism. Among various techniques, \( \mu \text{SR} \) has a great advantage in that it can be applied in systems consisting of spatially inhomogeneous multiple phases, providing information on respective phases according to their fractional yield. Our \( \mu \text{SR} \) measurement in the LFAO-F sample with \( x = 0.06 \) (\( T_c \simeq 18 \text{ K} \)) reveals that these two phases indeed coexist in the form of macroscopic phase separation, and more interestingly, that a spin glass-like magnetic phase develops in conjunction with superconductivity in the paramagnetic phase. This accordance strongly suggests a common origin of the electronic correlation leading to these two competing phases.

Although the oxypnictide with rare-earth (\( R \)) substitution \( R\text{FeAsO}_{1-x}\text{F}_x \) exhibits higher \( T_c \) than that of LFAO-F, strong random fields from rare-earth ions preclude a detailed study of the ground state using sensitive magnetic probes like \( \mu \text{SR} \). Therefore, we chose the original LFAO-F system for our \( \mu \text{SR} \) study. The target concentration of LaFeAsO\(_{1-x}\)F\(_x\) is set near the phase boundary, \( x = 0.06 \), for which a polycrystalline sample was synthesized by solid state reaction. The detailed procedure for sample preparation is described in an earlier report.\textsuperscript{1} The sample was confirmed to be mostly of single phase using X-ray diffraction analysis. Of two possible impurity phases, namely, LaOF and FeAs, only the latter exhibits a magnetic (helical) order with \( T_N \simeq 77 \text{ K} \).\textsuperscript{8} As shown in Fig. 1, magnetic susceptibility exhibits no trace of FeAs phase or local magnetic impurities except below \( \sim 50 \text{ K} \) where a small upturn is observed. The susceptibility at a lower field [shown in Fig. 3(a)] provides evidence of bulk superconductivity with \( T_c \simeq 18 \text{ K} \) from the onset of diamagnetism. Conventional \( \mu \text{SR} \) measurement was performed using the LAMPF spectrometer installed on the M15 beamline of TRIUMF, Canada. During the measurement under a zero field (ZF), residual magnetic field at the sample position was reduced below \( 10^{-6} \text{ T} \) with the initial muon spin direction parallel to the muon beam direction \([\vec{P}_\mu(0) \parallel \hat{z}]\). For longitudinal field (LF) measurement, a magnetic field was applied parallel to \( \vec{P}_\mu(0) \). Time-dependent muon polarization \([G_z(t) = \hat{z} \cdot \vec{P}_\mu(t)]\) was monitored by measuring decay-positron asymmetry along the \( \hat{z} \)-axis. Transverse field (TF) condition was realized by rotating the initial muon polarization so that \( \vec{P}_\mu(0) \parallel \hat{x} \), where the
Fig. 1. (Color online) Magnetic susceptibility of LaFeAsO$_{1-x}$F$_x$ with $x = 0.06$ for the sample used for $\mu$SR measurement. Inset shows a reduced view of the region below 35 K.

asymmetry was monitored along the $\hat{x}$-axis to obtain $G_x(t) = \hat{x} \cdot \vec{P}_{\mu}(t)$. All the measurements under a magnetic field were made by cooling the sample to the target temperature after the field equilibrated.

ZF-$\mu$SR is the most sensitive technique for examining magnetism in any form, where the development of local magnetic moments leads to either the spontaneous oscillation (for long-range order) or exponential damping (inhomogeneous local magnetism) of $G_z(t)$. Figure 2 shows examples of ZF-$\mu$SR time spectra collected at 2 and 30 K. The spectrum at 30 K ($> T_c$) exhibits a Gaussian-like relaxation due to weak random local fields from nuclear magnetic moments, indicating that the entire sample is in the paramagnetic state. Meanwhile, the spectrum at 2 K is split into two components, one that exhibits a steep relaxation and the other that remains to show Gaussian-like relaxation. This indicates that there is a finite fraction of implanted muons that sense hyperfine fields from local electronic moments. The absence of oscillatory signal implies that the hyperfine field is highly inhomogeneous, so that the local magnetism is characterized by strong randomness or spin fluctuation. The fractional yield of the component showing steep relaxation is as large as 25% (see below), which is hardly attributed to impurity and therefore implies that the sample exhibits a macroscopic phase separation into two phases.

The magnitude of the hyperfine field and that of spin fluctuation are evaluated by observing the response of the $\mu$SR spectrum to a longitudinal field (LF). It is shown in Fig. 2 that the relaxation in the paramagnetic component is quenched by applying a weak magnetic field (LF=5 mT), which is perfectly explained by the suppression of static nuclear dipolar fields ($< 10^9$ mT). Meanwhile, the faster relaxation (seen for $0 < t < 1 \mu$s) due to the magnetic
phase is recovered only gradually over a field range of $10^{1-2}$ mT, and there still remains a slow relaxation even at the highest field of 60 mT. This residual depolarization under LF is a clear sign that local spins are slowly fluctuating, leading to the spin-lattice relaxation of $\vec{B}_\mu(t)$. Such quasi-two-step relaxation is also observed in dilute spin-glass systems, which is understood as a distribution of spin correlation time. A detailed analysis is made considering that these two components coming from the magnetic phase (see below).

Under a transverse field, implanted muons experience an inhomogeneity of the field $[B_z(r)]$ due to flux line lattice formation below $T_c$ that leads to relaxation, in addition to those observed under a zero field. The TF-$\mu$SR time spectrum in Fig. 2 (envelop part of the oscillation) obtained under a field of 50 mT exhibits complete depolarization at 2 K, indicating that the entire volume of the paramagnetic phase falls into the superconducting state. The rapidly relaxing component observed under ZF is also visible (although the coarse binning of the spectra for extracting the envelop part makes it slightly obscure), indicating that the corresponding part of the sample remains magnetic.

Considering the presence of the magnetic phase besides the paramagnetic (=superconducting below $T_c$) phase, we take special precaution to analyze both TF and ZF/LF $\mu$SR spectra in a consistent manner. For the determination of physical parameters describing the behavior of signals from the magnetic phase, we first analyze ZF/LF spectra at 2 K using the $\chi$-square minimization fit with the relaxation function

$$G_z(t) = [w_1 + \sum_{i=2}^3 w_i \exp(-\Lambda_i t)] \cdot G_{KT}(\delta_N : t), \quad (1)$$

Fig. 2. (Color online) $\mu$SR time spectra observed in LaFeAsO$_{1-x}$F$_x$ with $x = 0.06$ at 2 K under a longitudinal field (LF), a zero field (ZF), and a transverse field (TF), and that under ZF at 30 K. The spectrum under TF is plotted on a rotating reference frame of 6.78 MHz to extract the envelop function.
where \( G_{\text{KT}}(\delta_N : t) \) is the Kubo-Toyabe relaxation function for describing the Gaussian damping due to random local fields from nuclear moments (with \( \delta_N \) being the linewidth). \( w_1 \) is the fractional yield for the paramagnetic phase, \( w_2 \) and \( w_3 \) are those for the magnetic phase \((\sum w_i = 1)\) with \( \Lambda_2 \) and \( \Lambda_3 \) being the corresponding relaxation rate described by the Redfield relation

\[
\Lambda_i = \frac{2\delta_i^2\nu_i}{\nu_i^2 + \omega_{\mu}^2} \quad (i = 2, 3),
\]

where \( \omega_{\mu} = \gamma_{\mu}H_{\text{LF}} \), \( \gamma_{\mu} \) is the muon gyromagnetic ratio \( (= 2\pi \times 135.53 \text{ MHz/T}) \), \( H_{\text{LF}} \) is the longitudinal field, \( \delta_2 \) and \( \delta_3 \) are the means of the hyperfine fields exerting on muons from local electronic moments, and \( \nu_2 \) and \( \nu_3 \) are the fluctuation rates of the hyperfine field. The solid curves in Fig. 2 show the result of analysis where all the spectra at different fields (only ZF and LF=5mT are shown here) are fitted simultaneously using eqs. (1) and (2) with common parameter values (except \( \omega_{\mu} \) that is fixed to the respective value for each spectrum), which show excellent agreement with all the spectra. The deduced parameters are as follows: \( w_1 = 0.754(9), w_2 = 0.165(9), w_3 = 0.081(4), \delta_2 = 0.71(5) \mu s^{-1}, \delta_3 = 3.9(3) \mu s^{-1}, \nu_2 = 1.7(2) \mu s^{-1}, \) and \( \nu_3 = 4(1) \mu s^{-1}. \) Although the depolarization in the magnetic phase is approximately represented by two components with different hyperfine couplings \( (\delta_i) \), the fluctuation rates \( (\nu_i) \) are close to each other \( (10^7 \text{ s}^{-1} \text{ at } 2 \text{ K}) \), suggesting that the randomness is primarily due to the distribution in the size of local moments (or in their distances to muons). Since no impurity phase with a fraction as large as 25% is detected by X-ray diffraction analysis, it is concluded that this magnetic phase is intrinsic.

In the analysis of temperature-dependent TF spectra, we used the relaxation function

\[
G_x(t) = \exp(-\frac{1}{2}\delta_N^2t^2)[w_1 \exp(-\delta_2^2t^2)\cos(2\pi f_1t + \phi) + (w_2 + w_3) \exp(-\Lambda_m t)\cos(2\pi f_{\text{m}}t + \phi)],
\]

where \( w_i \) and \( \delta_N \) are fixed to the values obtained by analyzing ZF/LF-\( \mu \)SR spectra. The first component in the above equation represents the contribution of flux line lattice formation in the superconducting phase, where \( \delta_s \) corresponds to the linewidth \( \sigma_s = \sqrt{2}\delta_s = \gamma_{\mu}\langle(B(r) - B_0)\rangle^{1/2} \) [with \( B_0 \) being the mean \( B(r) \)], while the second term represents the relaxation in the magnetic phase. Here, the relaxation rate for the latter is represented by a single value \( \Lambda_m \) (instead of \( \Lambda_{2,3} \), as it turns out that the two components observed under LF are hardly discernible in TF-\( \mu \)SR spectra. [This does not affect the result of the analysis, because the amplitude is fixed to \( w_2 + w_3 \) so that \( \Lambda_m \) may represent a mean \( \simeq (w_2\Lambda_2 + w_3\Lambda_3)/(w_2 + w_3) \).] The fit analysis using the above form indicates that all the spectra are perfectly reproduced while the partial asymmetry is fixed to the value determined from ZF-\( \mu \)SR spectra. This strengthens the presumption that the paramagnetic phase becomes superconducting below \( T_c \). The result of analysis is summarized in Fig. 3, together with the result of dc magnetization measured in the sample from the same batch as that used for \( \mu \)SR.
It is interesting to note in Fig. 3(b) that, although the central frequency in the superconducting phase ($f_s$) does not show much change below $T_c \simeq 18$ K probably owing to a large magnetic penetration depth (it is indeed large, see below), that in the magnetic phase ($f_m$) exhibits a clear shift in the negative direction below $T_m \simeq T_c$. The magnitude of the shift is as large as $\sim 1\%$ and thus is readily identified despite a relatively low external field of 50 mT. As shown in Fig. 3(c), the relaxation rate in the magnetic phase ($\Lambda_m$) also develops below $T_c$ in accordance with the frequency shift, demonstrating that a spin-glass-like magnetism sets in below $T_c$. Here, we note that the development of magnetic phase is already evident in the ZF/LF-$\mu$SR spectra, and results are fully consistent with each other. The onset of superconductivity below $T_c$ is also confirmed by an increase in $\delta_s$, as observed in Fig. 3(c). This remarkable accordance of onset temperature between magnetism and superconductivity strongly suggests that there is an intrinsic relationship between the superconducting and magnetic phases that leads to a common characteristic temperature.

The temperature dependence of $\sigma_s$ in Fig. 3(d) is compared with theoretical predictions for a variety of models with different order parameters. The weak-coupling BCS model ($s$-wave, single gap) apparently fails to reproduce the present data, as they exhibit a tendency to vary with temperature over the region $T/T_c < 0.4$. Although a fit using a two-gap model shown by a dotted line seems to exhibit reasonable agreement with data, the deduced gap parameters ($2\Delta_i/k_BT_c$) are largely inconsistent with the prediction of the weak-coupling BCS model (see Table I). These observations suggest that the superconducting order parameter is not described by a $s$-wave symmetry with a single gap. When a power law, $\sigma_s = \sigma_0[1-(T/T_c)^\beta]$, is used in fitting the data, we obtain a curve shown by the broken line in Fig. 3(d) with an exponent $\beta \simeq 2$. This is in good agreement with the case of $d$-wave symmetry at the dirty limit.

In the limit of extreme type II superconductors [i.e., $\lambda/\xi \gg 1$, where $\lambda$ is the effective London penetration depth and $\xi = \sqrt{\Phi_0/(2\pi H_{c2})}$ is the Ginzburg-Landau coherence length, $\Phi_0$ is the flux quantum, and $H_{c2}$ is the upper critical field], $\sigma_s$ is determined by $\lambda$ using the relation $\sigma_s/\gamma_\mu = 2.74 \times 10^{-2}(1-h)\left[1+3.9(1-h)^2\right]^{1/2}\Phi_0\lambda^{-2}$, where $h = H_{TF}/H_{c2}$.

### Table I. Parameters for defining the lines in Fig. 3(d).

| Two-gap | Power law |
|---------|-----------|
| $T_c$ (K) | $T_c$ (K) |
| $\sigma(0)$ ($\mu$s$^{-1}$) | $\sigma(0)$ ($\mu$s$^{-1}$) |
| $w$ | $\beta$ |
| $2\Delta_1/k_BT_c$ | $2\Delta_2/k_BT_c$ |
Fig. 3. (Color online) Temperature dependence of dc magnetic susceptibility measured at 1 mT (a), and that of physical parameters deduced by analyzing TF-μSR spectra in superconducting ($f_s$, $\delta_s$) and magnetic ($f_m$, $\Lambda_m$) phases (b-c), and of $\sigma_s (=\sqrt{2}\delta_s)$ proportional to superfluid density (d). Curves in (d) are fits by models described in the text.

and $H_{TF}$ is the magnitude of external field. From $\sigma_s$ extrapolated to $T = 0$ and taking $H_{c2} \simeq 50$ T (ref.13), we obtain $\lambda=595(3)$ nm. Because of the large anisotropy expected from the layered structure of this compound, $\lambda$ in the polycrystalline sample would be predominantly determined by in-plane penetration depth ($\lambda_{ab}$). Using the formula $\lambda = 1.31\lambda_{ab}$ for such a situation,14 we obtain $\lambda_{ab}=454(2)$ nm. This value coincides with that expected from the aforementioned empirical relation between $\lambda_{ab}^{-2}$ superconductors.6,15 However, this may not be uniquely attributed to the superfluid density because $\lambda$ depends not only $n_s$ but also on the effective mass, $\sigma_s \propto \lambda^{-2} = n_s e^2/m^*c^2$. 

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Finally, we discuss the feature of the spin glass-like phase. Assuming that the local moments are those of off-stoichiometric iron atoms with a moment size close to that in the SDW phase ($\sim 0.36 \mu_B$), the mean distance between muon and iron moments in the relevant phase is estimated to be $\sim 0.5$ nm from an average of $\delta_i$. Given the unit cell size ($a = 0.403$ nm, $c = 0.874$ nm$^1$), this would mean that more than a quarter of iron atoms in the magnetic phase (i.e., $\simeq 7\%$ of the entire sample) should serve as a source of local moments. It is unlikely that such a significant fraction of iron atoms remains as impurities in the present sample.

It might also be noteworthy that there is an anomaly near $T_{m2} \simeq 12$ K in the susceptibility [the onset of ZFC/FC hysteresis in Fig. 1 and a steplike kink in Fig. 3(a)]. This seems to be in accordance with the onset of a steeper increase in $\Lambda_m$ below $T_{m2}$, suggesting a change in magnetic correlation.

$\mu$SR studies of LFAO-F have been made by a number of groups. According to those preliminary studies, no clear sign of magnetism is observed in the sample over relevant doping concentrations, except for a weak relaxation observed far below $T_c$ for $x = 0.05$ and 0.075 in ZF-$\mu$SR spectra and an unidentified additional relaxation observed in TF-$\mu$SR spectra for $x = 0.075$.\textsuperscript{5-7} This led us to recall the sensitivity to chemical stoichiometry in the emergence of the spin glass-like $A$-phase observed near the boundary between the antiferromagnetic and superconducting phases in CeCu$_2$Si$_2$.\textsuperscript{16} In addition to the $A$-phase, the present LFAO-F system exhibits a closer similarity to this classical heavy-fermion superconductor such as the phase diagram against pressure/doping.\textsuperscript{17} Further study of the dependence of fractional yield for the magnetic phase with varying $x$ (in small steps near the phase boundary) would provide further insight into the true nature of these phases and the mechanism of superconductivity itself that is working behind the coexistence/competition.

In summary, it has been revealed by our $\mu$SR experiment that superconducting and magnetic phases coexist in LaFeAs(O$_{0.94}$F$_{0.06}$) with $x = 0.06$. These two phases simultaneously develop just below $T_c$, strongly suggesting an intimate and intrinsic relationship between these two phases. The result of TF-$\mu$SR measurement suggests that the superconductivity of LaFeAs(O$_{0.94}$F$_{0.06}$) cannot be explained by the conventional weak-BCS model (single gap, $s$-wave).

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