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# Evidence for a full energy gap in the nickel pnictide superconductor $LaNiAsO_{1-x}F_x$ from $^{75}$As nuclear quadrupole resonance

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We report systematic $^{75}$As-NQR and $^{139}$La-NMR studies on nickel pnictide superconductors $LaNiAsO_{1-x}F_x$ ($x$=0, 0.06, 0.10, and 0.12). The spin-lattice relaxation rate $1/T_1$ decreases below $T_c$ with a well-defined coherence peak and follows an exponential decay at low temperatures. This result indicates that the superconducting gap is fully opened and is strikingly different from that observed in iron-pnictide analogs. In the normal state, $1/T_1T$ is constant in the temperature range $T_c \sim 4$ K $\leq T \leq 10$ K for all compounds and up to $T$=250 K for $x$=0 and 0.06, which indicates weak electron correlations and is also different from the iron analog. We argue that the differences between the iron and nickel pnictides arise from the different electronic band structure. Our results highlight the importance of the peculiar Fermi-surface topology in iron pnictides. We also provide a possible interpretation for the "pseudogap-like" behavior in the normal state which is also seen in iron pnictides.

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Superconductivity in $ReFeAsO_{1-x}F_x$ (*Re*: rare-earth element) at the transition temperature up to $T_c$=55 K (Refs. 1 and 2) has received considerable attention. These compounds have a ZrCuSiAs-type structure ($P4/nmm$) in which FeAs forms a two-dimensional network similar to the $CuO_2$ plane in the case of cuprate high-$T_c$ superconductors. By replacing O with F, electrons are doped and superconductivity emerges. The Fermi surface consists of two hole pockets centered at (0, 0) (Γ point) of the unfolded Brillouin zone, and two electron pockets around $(\pi,0)$.[3] Nuclear magnetic resonance (NMR) measurement in $PrFeAsO_{0.89}F_{0.11}$ suggested that there are multiple superconducting gaps.[4] Angle-resolved photoemission spectroscopy (ARPES) has directly observed the gaps on different Fermi surfaces.[5]

However, the symmetry of the superconducting gap and the mechanism of the superconductivity remains unclear. The spin-lattice relaxation rate $1/T_1$ shows neither a coherence peak just below $T_c$ nor an exponential decay at low temperature ($T$) (Refs. 4, 6, and 7) expected for conventional fully gapped superconductors. The result was interpreted as indicative of nodes in the gap function. On the other hand, tunneling[8] and ARPES (Ref. 5) measurements have suggested a full gap. The penetration depth measurements by different groups[9,10] have led to different conclusion on whether there are nodes in the gap function or not.

In the normal state, antiferromagnetic spin fluctuations with wave vector $Q=(\pi,0)$ is expected due to the nesting between the electronlike Fermi surface and the holelike Fermi surface.[11–14] Indeed, signatures of such spin fluctuations are seen in $LaFeAsO_{0.92}F_{0.08}$ (Ref. 15) and $Ba_{1-x}K_xFe_2As_2$ (Refs. 16–18) where $1/T_1T$ increases with decreasing temperature. It has been proposed that such spin fluctuations may promote superconductivity with $s^{\pm}$-wave gap that changes sign on different Fermi surfaces.[11,12,19] In such case, the scatterings between different Fermi surfaces may reduce the coherence peak in the $T$ dependence of $1/T_1$,[13,20–23] thereby reconcile the discrepancy between NMR and tunneling/ARPES measurements. However, experimental verification of such exotic superconducting state remains to be carried out.

Meanwhile, the nickel analog of $LaFeAsO_{1-x}F_x$, namely, $LaNiAsO_{1-x}F_x$, are superconducting but with a lower $T_c \sim 4$ K.[24] Local-density approximation calculation has revealed a striking difference in Fermi surface compared to the Fe analog. Namely, there are no hole pockets around the (0, 0) point for $LaNiAsO_{1-x}F_x$.[25] Since interband scattering between the electron and hole pockets with the wave vector $Q=(\pi,0)$ is proposed to be responsible for both the superconductivity and the normal-state properties for $LaFeAsO_{1-x}F_x$, the $LaNiAsO_{1-x}F_x$ without such Fermi-surface nesting is an ideal compound to compare with and to obtain insight into $LaFeAsO_{1-x}F_x$. In particular, identifying

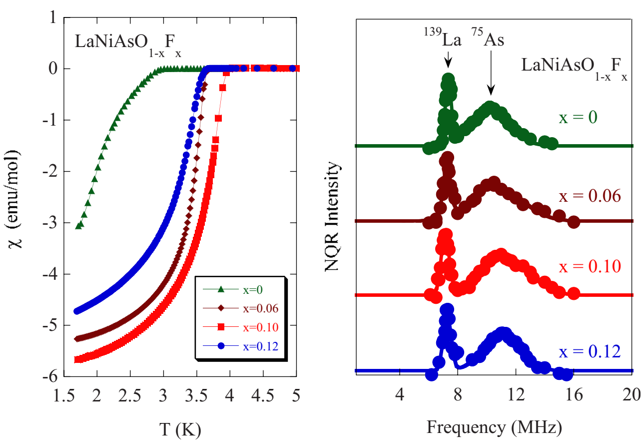


FIG. 1. (Color online) (Left) dc susceptibility below $T$=5 K measured at $H$=10 Oe (zero-field cooling). (Right) NQR spectra at $T$=4.2 K. The baseline (the horizontal line associated with each spectrum) is shifted for clarity. The curves are guide to the eyes. The left peak is from the $\pm 7/2 \leftrightarrow \pm 5/2$ transition of $^{139}$La and the right peak is from $^{75}$As.

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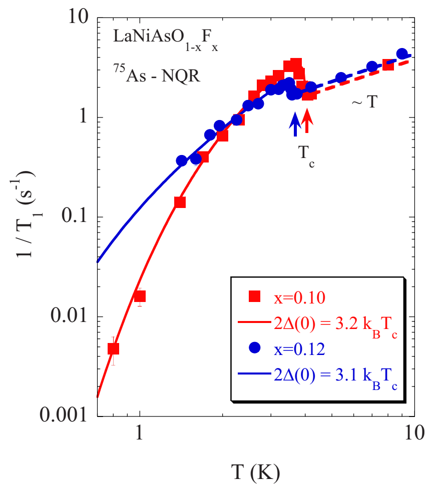


FIG. 2. (Color online) The $T$ dependence of the spin-lattice relaxation rate, $1/T_1$, for $LaNiAsO_{1-x}F_x$ ($x$=0.10 and 0.12). The arrows indicate $T_c$. The broken straight lines show the relation $1/T_1 \propto T$, and the curves below $T_c$ are fits to the BCS model with the gap size indicated in the figure.

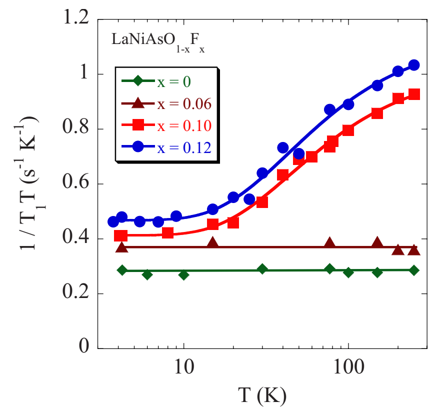


FIG. 3. (Color online) The quantity $^{75}(1/T_1T)$ in the normal state of $LaNiAsO_{1-x}F_x$ above $T_c$. The errors are within the size of the symbols. Below $T$=10 K, $1/T_1T$ is constant for all F contents, indicating weak electron correlations. The straight lines are guide to the eyes. The curves for $x$=0.10 and 0.12 are fits to $1/T_1T = (1/T_1T)_0 + b\exp(-2E_g/k_BT)$ with $(1/T_1T)_0$=0.41 $s^{-1}\,K^{-1}$, $b$=0.61 $s^{-1}\,K^{-1}$, and $E_g/k_B$=22.5 K for $x$=0.10 and $(1/T_1T)_0$=0.46 $s^{-1}\,K^{-1}$, $b$=0.67 $s^{-1}\,K^{-1}$, and $E_g/k_B$=22 K for $x$=0.12.

the superconducting gap symmetry and the nature of electron correlations, if any, in $LaNiAsO_{1-x}F_x$ will help test the theoretical proposals and understand the mechanisms of high-$T_c$ superconductivity in iron pnictides.

In this Rapid Communication, we report a microscopic measurement on $LaNiAsO_{1-x}F_x$ ($x$=0, 0.06, 0.10, and 0.12) using the $^{75}$As nuclear quadrupole resonance (NQR) and $^{139}$La-NMR techniques. The spin-lattice relaxation rate, $1/T_1$, shows a well-defined coherence peak just below $T_c$ and decays exponentially with further decreasing $T$, indicating a fully opened superconducting gap on the entire Fermi surface. This is clear NMR/NQR evidence for a full gap in the pnictide superconductors. In the normal state above $T_c$, no antiferromagnetic spin fluctuations were observed. These features are striking different from $LaFeAsO_{1-x}F_x$ where high-$T_c$ unusual superconductivity emerges with moderate strength of antiferromagnetic spin fluctuation as a background. The difference is understood by the different topology of the Fermi surface of the two classes of materials. Our results highlight the importance of the Fermi-surface topology in Fe pnictides and shed lights on the mechanism of superconductivity and electron correlations there.

The polycrystalline samples of $LaNiAsO_{1-x}F_x$ were synthesized by the solid-state reaction method using NiO, Ni, As, La, and $LaF_3$ as starting materials.[24] LaAs was prepared by reacting La chips and As pieces at 500 °C for 15 h and then at 850 °C for 2 h. The raw materials were thoroughly ground and pressed into pellets and then annealed at 1150 °C for 50 h. Powder x-ray diffraction indicates that the samples are of single phase. For NQR measurements, the pellets were ground into powder. $T_c$ was determined by dc susceptibility measurement on the powders using a superconducting quantum interference device and by ac susceptibility measurement using the *in situ* NQR coil. The results by the two methods agree well. The NQR spectra were taken by changing the frequency point by point while the NMR spectra were taken by sweeping the magnetic field at a fixed frequency. The $1/T_1$ was measured at zero magnetic field (NQR) by using a single saturation pulse. Measurements below $T$=1.3 K were conducted by using a $^3$He-$^4$He dilution refrigerator.

Figure 1 shows the dc susceptibility around $T_c$ and the NQR spectra of $^{75}$As ($I$=3/2). The nuclear quadrupole frequency $\nu_Q$ increases slightly with increasing F content.

Figure 2 shows the $T$ dependence of the spin-lattice relaxation rate, $1/T_1$, at zero magnetic field, for the two samples with higher $T_c$. The $T_1$ was measured at the peak of the spectrum and determined from an excellent fit of the nuclear magnetization to the single exponential function $1-M(t)/M_0=\exp(-3t/T_1)$, where $M_0$ and $M(t)$ are the nuclear magnetization in the thermal equilibrium and at a time $t$ after the saturating pulse, respectively. The single component of $T_1$ indicates that the sample is homogeneous.

As seen in the figure, $1/T_1$ shows a well-defined coherence (Hebel-Slichter) peak just below $T_c$, which is a characteristic of superconductors with an isotropic gap. This is in sharp contrast to various Fe pnictides reported so far.[4,6,7,15–17] At low temperatures, $1/T_1$ decreases as an exponential function of $T$. The solid curves in Fig. 2 are calculations using the BCS model. The $1/T_{1S}$ in the superconducting state is expressed as $\frac{T_{1N}}{T_{1S}}=\frac{2}{k_BT}\int\int(1+\frac{\Delta^2}{EE'})N_s(E)N_s(E')f(E)[1-f(E')]\delta(E-E')dEdE'$, where $1/T_{1N}$ is the relaxation rate in the normal state, $N_s(E)$ is the superconducting density of states (DOS), $f(E)$ is the Fermi distribution function, and $C=1+\frac{\Delta^2}{EE'}$ is the "coherence factor." Following Hebel,[26] we convolute $N_s(E)$ with a broadening function $B(E)$ which is approximated with a rectangular function centered at $E$ with a height of $1/2\delta$. The solid curves below $T_c$ for the two samples shown in Fig. 2 are calculations with $2\Delta(0)=3.2k_BT_c$ and $r\equiv\Delta(0)/\delta=5$ for $LaNiAsO_{0.90}F_{0.10}$ and $2\Delta(0)=3.1k_BT_c$ and $r$=1.5 for $LaNiAsO_{0.88}F_{0.12}$.

Such $T$ dependence of $1/T_1$ in the superconducting state is in striking contrast to that for Fe pnictides where no co-

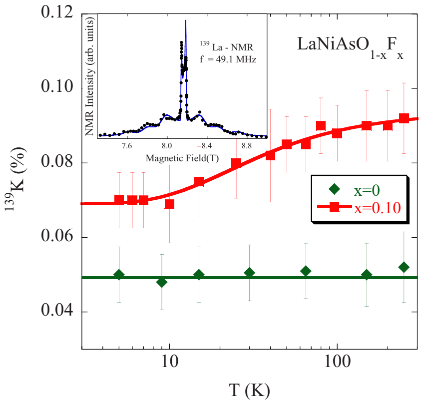


FIG. 4. (Color online) The $^{139}$La Knight shift in the $ab$ plane of $LaNiAsO_{1-x}F_x$ ($x$=0 and 0.10) above $T_c$. The curve for $x$=0.10 is a fit to $K=K_0+K_1 \exp(-E_g/k_BT)$ with $K_0$=0.07%, $K_1$=0.025%, and $E_g/k_B$=23 K. The straight line for $x$=0 is a guide to the eyes. The inset shows the NMR spectrum at 49.1 MHz and $T$=5 K for $x$=0.10. It consists of two central peaks corresponding to, respectively, $\theta$=90° (left) and 41.7° (right), accompanied by six satellite peaks since the nuclear spin is 7/2. Here $\theta$ is the angle between $H$ and the $c$ axis. The curve is a theoretical calculation with $\nu_Q$ =2.35 MHz for a randomly oriented powders. The sample appears to be partially oriented in the $ab$ plane, as in $LaFeAsO_{0.9}F_{0.1}$ (Ref. 7) and $PrFeAsO_{0.89}F_{0.11}$(Ref. 4).

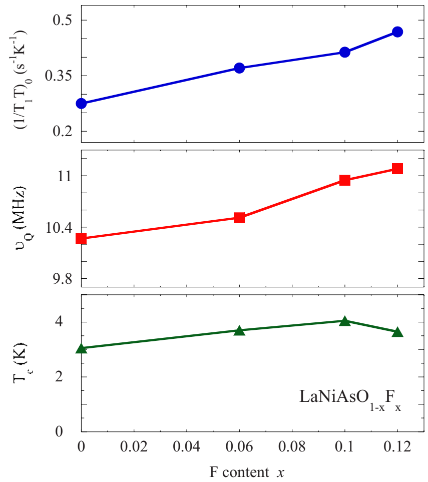


FIG. 5. (Color online) Phase diagram obtained from the present study. $\nu_Q$ is the NQR frequency at $T$=4.2 K and $(1/T_1T)_0$ is the averaged value for $T_c \leq T \leq 10$ K, which is proportional to the squared DOS at the Fermi level.

herence peak was observed and the $T$ dependence at low $T$ does not show an exponential behavior. The striking difference may be ascribed to the different topology of the Fermi surfaces. For Fe pnictides, it has been proposed that $d$-wave[27,28] or sign reversal $s$-wave gap[11,12] can be stabilized due to nesting by the connecting wave vector $Q=(\pi,0)$. In $LaNiAsO_{1-x}F_x$, however, there is no such Fermi-surface nesting,[25] and thus the mechanism for the proposed gap symmetry does not exist. Our result therefore highlights the important role of the Fermi-surface topology in the superconductivity of Fe pnictides.

Figure 3 shows the quantity $1/T_1T$ as a function of $T$. For $x$=0 and 0.06, $1/T_1T$ is a constant independent of $T$, which indicates that the electron correlations are weak as in conventional metals. This feature is also in striking contrast to the Fe analog. In $LaFeAsO_{1-x}F_x$, a magnetically ordered state is realized below $T_N$=140 K for $x$=0.[29] For small $x$, $1/T_1T$ increases with decreasing $T$,[15] indicating electron correlations as seen in high-$T_c$ cuprates. The explanation based on Fermi-surface nesting is a promising scenario to account for the magnetic order.[11,30] Fermi-surface nesting is also proposed to be responsible for the spin fluctuations with $Q$ $=(\pi,0)$.[11,12] In $LaNiAsO_{1-x}F_x$, however, such Fermi-surface nesting does not exist, therefore the spin fluctuations are not expected. Thus, the striking difference in the normal state between $LaNiAsO_{1-x}F_x$ and $LaFeAsO_{1-x}F_x$ can also be understood by the different topology of the Fermi surfaces.

On the other hand, for $LaNiAsO_{0.90}F_{0.10}$ and $LaNiAsO_{0.88}F_{0.12}$, $1/T_1T$ increases with increasing $T$. A similar feature was also seen in highly doped $LaFeAsO_{1-x}F_x$ with $x \geq 0.1$ (Refs. 6, 31, and 32) and $Ba(Fe_{0.9}Co_{0.1})_2As_2$.[33] Clearly, such behavior cannot be ascribed to electron correlations which are believed to be responsible for a similar phenomenon in high-$T_c$ cuprates (called pseudogap).[34] It was proposed that such pseudogap behavior in $LaFeAsO_{1-x}F_x$ is due to the band structure.[35] In $LaFeAsO_{1-x}F_x$ with larger electron doping of $x \geq 0.1$, the large DOS due to the hole pocket around the $\Gamma'$ point, namely $(\pi,\pi)$, sinks to below the Fermi level.[35,36] At low $T$, this part of DOS does not contribute to $1/T_1$. Upon increasing $T$, however, the thermal activation associated with such DOS will contribute to $1/T_1$, giving rise to the pseudogap behavior. At present, the origin for the pseudogap behavior in $LaNiAsO_{1-x}F_x$ is unclear. But there is a good possibility that the same mechanism[35] also applies. Upon doping, the bands around X and R points in $LaNiAsO_{1-x}F_x$ (Ref. 25) could sink to below the Fermi level,[37] resulting in a pseudogap behavior similar to $LaFeAsO_{1-x}F_x$ ($x \geq 0.1$). The $^{139}$La Knight shift ($K$) shown in Fig. 4 supports such speculation since the spin part of $K$ is proportional to the DOS at the Fermi level $[N(E_F)]$ while $1/T_1T \propto N(E_F)^2$. The curves for $x$=0.10 and 0.12 in Fig. 3 are fits to $1/T_1T=(1/T_1T)_0+b \exp(-2E_g/k_BT)$. The first term is proportional to $N(E_F)^2$ at low $T$ and the second term comes from the thermal activation effect of the DOS beneath the Fermi level. The obtained $E_g/k_B$ is 22.5 K and 22 K for $x$ =0.10 and 0.12, respectively. The curve in Fig. 4 is a fit to $K=K_0+K_1 \exp(-E_g/k_BT)$ with resulting $E_g/k_B$=23 K, in good agreement with the $1/T_1T$ result. To conclude, the pseudogaplike behavior commonly seen in nickel and iron pnictides, in spite of strikingly different $T_c$, suggests that this feature is unrelated to the mechanism of the superconductivity.

Figure 5 summarizes the results. Upon replacing O with F, the value of $(1/T_1T)_0$ at low temperatures increases, indicating electron doping into the system. This is also supported by the F-content dependence of $\nu_Q$ (middle panel) which is generally dominated by the on-site charge distribution. The $T_c$ initially increases upon doping but becomes less $x$ dependent beyond 0.06.[24] And most remarkably different, $T_c$ is smaller by nearly an order of magnitude compared to $LaFeAsO_{1-x}F_x$. If the superconductivity in $LaFeAsO_{1-x}F_x$ is promoted by the spin fluctuations with $Q=(\pi,0)$ as proposed,[11,12] then the much lower $T_c$ in $LaNiAsO_{1-x}F_x$ can be naturally understood since such spin fluctuation is not expected due to the different Fermi surface topology and indeed is not observed experimentally.

In summary, we have presented the NQR and NMR results on the electron-doped, low-$T_c$ nickel pnictides $LaNiAsO_{1-x}F_x$ ($x=0$, 0.06, 0.10, and 0.12). We find that the superconducting gap is fully opened on the entire Fermi surface. Namely, the spin-lattice relaxation rate shows a well-defined coherence peak just below $T_c$ and decays exponentially with further decreasing $T$. In the normal state, no antiferromagnetic spin fluctuations were observed. These features are in striking contrast with $LaFeAsO_{1-x}F_x$ where high-$T_c$ unusual superconductivity emerges in a background of moderate antiferromagnetic spin fluctuation. All these differences are understood by the different topology of the Fermi surface for the two classes of materials. Our results highlight the peculiarity and importance of the Fermi-surface topology in $LaFeAsO_{1-x}F_x$ and shed light on the mechanism of superconductivity and electron correlations there.

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