Simultaneous Optimization of Spin Fluctuations and Superconductivity under Pressure in an Iron-Based Superconductor

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(Received 14 May 2013; revised manuscript received 30 July 2013; published 4 September 2013)

We present a high-pressure NMR study of the overdoped iron pnictide superconductor $NaFe_{0.94}Co_{0.06}As$. The low-energy antiferromagnetic spin fluctuations in the normal state, manifest as the Curie-Weiss upturn in the spin-lattice relaxation rate $1/^{75}T_1T$, first increase strongly with pressure but fall again at $P > P_{opt} = 2.2$ GPa. Neither long-ranged magnetic order nor a structural phase transition is encountered up to 2.5 GPa. The superconducting transition temperature T_c shows a pressure dependence identical to the spin fluctuations. Our observations demonstrate that magnetic correlations and superconductivity are optimized simultaneously as a function of the electronic structure, thereby supporting very strongly a magnetic origin of superconductivity.

DOI: 10.1103/PhysRevLett.111.107004 PACS numbers: 74.70.−b, 76.60.−k

In the iron-based superconductors [1–4], charged dopants usually act to suppress an orthorhombic ground state with antiferromagnetic long-range order (AFMLRO) in favor of a tetragonal, paramagnetic, and superconducting phase. Multiple electron bands are observed [5,6], which may include all five Fe *d* orbitals. These results indicate that different electronic degrees of freedom, both orbital and magnetic, are involved in the fluctuations and possible broken-symmetry phases, and to date these complex correlation effects have obscured the pairing mechanism [7]. While spin fluctuations are a leading candidate for mediating superconductivity, orbital fluctuations have also been proposed for this role [8]. Direct evidence for the pairing mechanism continues to be the primary goal of the many studies investigating how the lattice structure, band structure, and magnetism determine the superconducting properties.

An applied pressure is a particularly clean method for controlling the physical properties of iron-based superconductors. The superconducting transition temperature T_c has been found to change strongly with pressure in $LaFeAsO_{1-x}F_x$ (1111 structure) [9], $BaFe_2(As_{1-x}P_x)_2$ (122) [10], $NaFe_{1-x}Co_xAs$ (111) [11], $Fe_{1+x}Se$ (11) [12], and many other systems [13]. To date, $NaFe_{1-x}Co_xAs$ has shown the most marked effects, even of rather moderate pressures, in its structural, magnetic, and superconducting properties. NMR studies of the parent compound NaFeAs show that the Néel temperature T_N increases with pressure up to 2.4 GPa [14], and x-ray measurements find a collapsed tetragonal phase above 3 GPa [15]. These observations leave open the question of how changes in T_c may be associated with competing spin fluctuations, AFMLRO, and/or changes in crystal structure and suggest a systematic study of correlation and pairing effects by changing the lattice parameters under pressure.

In this Letter, we present a high-pressure ^{75}As NMR study on the overdoped iron-based superconductor $NaFe_{1-x}Co_xAs$ with $x = 0.06$. In the normal state, the spin-lattice relaxation rate divided by temperature, $1/^{75}T_1T$, first grows significantly with pressure, showing a low-temperature Curie-Weiss upturn indicative of strongly enhanced low-energy spin fluctuations. However, $1/^{75}T_1T$ reaches a maximum at $P_{opt} \simeq 2.17$ GPa before decreasing again, a nonmonotonic pressure dependence not previously observed in iron-based superconductors. The superconducting transition temperature has an identical "dome" feature under pressure, with a maximal T_c at the same P_{opt}. These observations indicates clearly that the strong correlations between magnetism, superconductivity, and the details of the underlying lattice are quite different from the effects of doping and give strong support for a magnetic origin of superconductivity.

$NaFe_{1-x}Co_xAs$ is optimally doped at $x = 0.03$, where the maximal T_c is approximately 20 K [16]. We perform a systematic study of pressure effects on the structure and the magnetic fluctuations and of their correlation with superconductivity by avoiding both the structural and magnetic phase transitions; for this, we focus on a sample with significant overdoping, $x = 0.06$, where $T_c \approx 18$ K. $NaFe_{0.94}Co_{0.06}As$ single crystals were synthesized by the flux-grown method with NaAs as the flux. The doping was determined accurately from inductively coupled plasma atomic emission spectroscopy measurements.

For our high-pressure NMR measurements, we used a clamp-type pressure cell with Daphne oil as a pressure

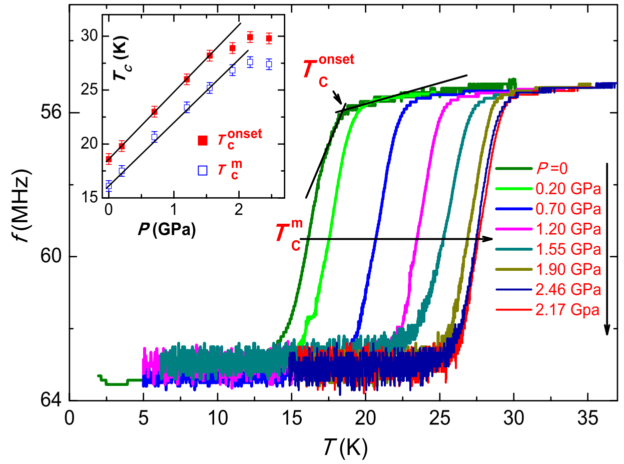

FIG. 1 (color online). Main panel: RF resonance frequency of the detuned NMR circuit measured as a function of temperature and pressure at zero field. The onset and midpoint superconducting transition temperatures, respectively T_c^{onset} and T_c^m, are indicated by the arrows. Inset: values of T_c^{onset} and T_c^m as functions of pressure.

medium achieving high homogeneity. The clamp cell is limited to 2.5 GPa at low temperatures, and although P cannot be changed externally below room temperature, it does change with T. For a complete calibration of the pressure at different temperatures we used a manometer of $\mathrm{Cu_2O}$, whose nuclear quadrupole resonance frequency is known very accurately [17]. We deduced $P(T)$ from $^{63}\nu_q(P,T)$, finding a pressure drop $\Delta P(T) \leq 0.15$ GPa from 300 to 150 K and negligible changes below 150 K. The pressures reported here are those we measured at 2 K. We stress that all our measurements under pressure were fully and reproducibly reversible. The superconducting transition under pressure was determined consistently by NMR and from the ac susceptibility. The ^{75}As NMR spectra were obtained by the spin-echo technique under a field of 7.63 T applied in the ab plane. The spin-lattice relaxation rate $1/^{75}T_1$ was measured by the spin-inversion method.

T_c can be determined accurately *in situ* at all pressures by the ac inductance change of the sample coil during cooling and warming at zero field. The superconducting transition is indicated (Fig. 1) by an increase in the resonance frequency of the NMR circuit, which measures the ac susceptibility, upon cooling. We define the onset (T_c^{onset}) and midpoint (T_c^m) temperatures from the frequency curve, as illustrated in Fig. 1. Both T_c^{onset} and T_c^m have a strong initial increase (6 K/GPa) with pressure (inset, Fig. 1). However, after reaching maximal values of 29.8 K (T_c^{onset}) and 27.4 K (T_c^m) at a pressure $P_{\text{opt}} \simeq 2.17$ GPa, both quantities then decrease slowly ($-$ 0.6 K/GPa) at higher pressures. This dome-shaped superconducting transition is consistent with the results of high-pressure transport studies [11].

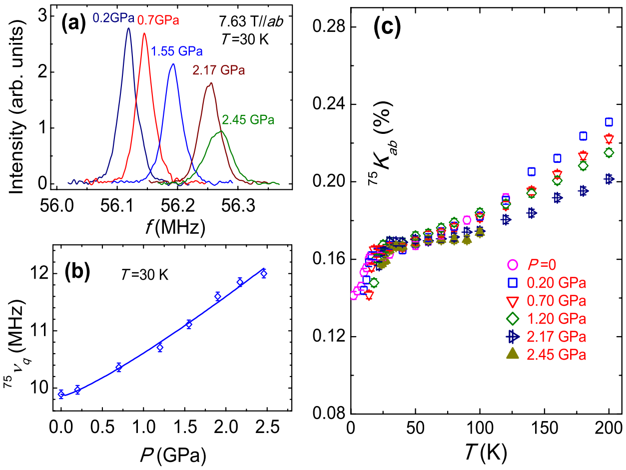

FIG. 2 (color online). (a) Center line of the ^{75}As NMR spectra at different pressures, with field applied in the crystalline ab plane. (b) Pressure dependence of the ^{75}As quadrupole frequency $^{75}\nu_q$ at $T = 30$ K. (c) Temperature dependence of the Knight shift $^{75}K_{ab}$ at different pressures.

We have measured ^{75}As ($S = 3/2$) NMR spectra over the full temperature range to 200 K, at a number of different pressures and with the field applied in the ab plane. Figure 2(a) shows the temperature-corrected center line of the spectrum at $T = 30$ K for several pressure values. The spectra shift monotonically to higher frequencies, primarily as a result of second-order corrections from the ^{75}As quadrupole frequency $^{75}\nu_q$, which we discuss below. The NMR line width increases from 25 kHz at $P = 0$ to 50 kHz at $P = 2.46$ GPa, showing a weak pressure inhomogeneity at higher pressures.

The quadrupole frequency is measured from the ^{75}As satellite spectra (data not shown). The low-temperature values of $^{75}\nu_q$ display an appreciable rise with pressure up to 2.46 GPa [Fig. 2(b)]. ν_q measures the local electric field gradient (EFG), which is very sensitive to the lattice parameters. This continuous increase of $^{75}\nu_q$ indicates a continuous lattice compression under pressure; neither the line shape nor the satellite frequency shows any abrupt changes with pressure or temperature. Thus the structure remains tetragonal and a transition to orthorhombic or collapsed-tetragonal symmetry is excluded up to 2.46 GPa, in contrast to the behavior observed in NaFeAs [14,15].

The in-plane Knight shift $^{75}K_{ab}$ deduced from the center line of the NMR spectrum is shown in Fig. 2(c). At a fixed pressure, $^{75}K_{ab}$ increases monotonically with temperature; the functional form $^{75}K_{ab} = A_0 + B_0T + C_0T^2$ is characteristic of additive contributions from itinerant electrons (A_0) and from predominantly two-dimensional (2D) local spin fluctuations (B_0) [18], with only weak three-dimensional (3D) contributions from interplane coupling (C_0). There are no abrupt changes in $^{75}K_{ab}$; taken together with constant Boltzmann-corrected spectral intensities down to 1.5 K at each pressure and the absence of

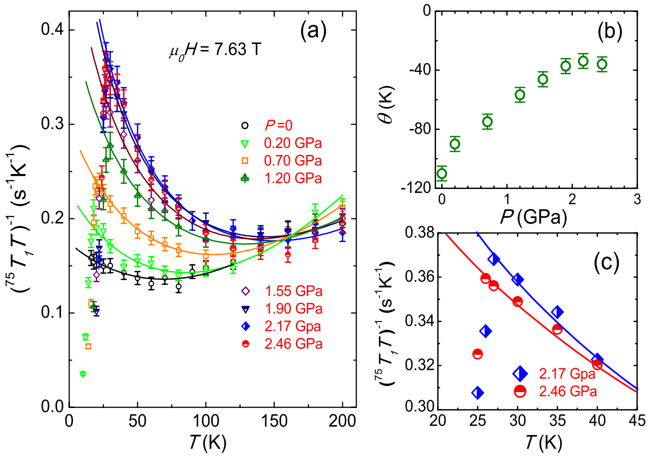

FIG. 3 (color online). (a) Temperature dependence of $1/^{75}T_1T$ at different pressures. The solid lines are fits to the form $1/^{75}T_1T = A_1/(T-\theta) + B_1T + C_1T^2$. (b) Pressure dependence of the Curie-Weiss temperature θ extracted from panel (a). (c) Comparison of $1/^{75}T_1T$ data near T_c at the two highest pressures [data and fitting lines as in panel (a)].

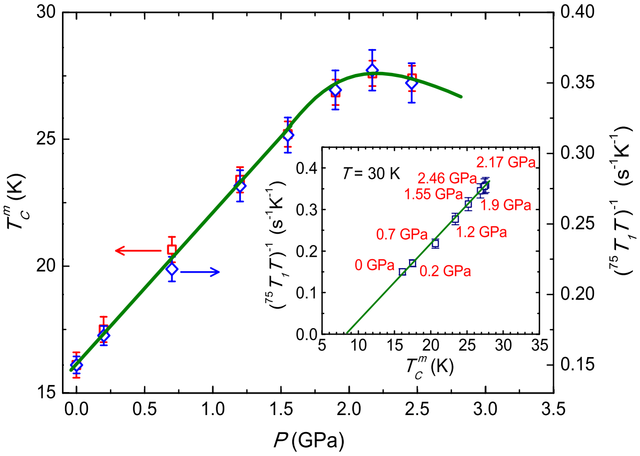

FIG. 4 (color online). Main panel: midpoint superconducting transition temperature T_c^m (squares) and normal-state spin-lattice relaxation rate $1/^{75}T_1T$ at $T = 30$ K (diamonds) as a function of pressure. Inset: scaling between T_c and normal-state $1/^{75}T_1T$.

diverging behavior in $1/^{75}T_1$ above T_c (shown below), this excludes a magnetic ordering transition below 2.46 GPa. At a fixed temperature $T > T_c$, $^{75}K_{ab}$ decreases with pressure. At $T < T_c$, $^{75}K_{ab}$ drops sharply, indicating a singlet superconducting order parameter. The values of T_c determined from the Knight shift are fully consistent with those from the ac susceptibility data (Fig. 1).

The ^{75}As spin-lattice relaxation rates ($1/^{75}T_1$) measured at each pressure are shown in Fig. 3(a) for temperatures up to 200 K. On cooling, $1/^{75}T_1T$ first decreases but then shows a broad, low-temperature upturn before falling abruptly below T_c. The upturn, which becomes increasingly prominent at high pressures, can be fitted rather well by the expression $1/^{75}T_1T = A_1/(T-\theta) + B_1T + C_1T^2$. The Curie-Weiss contribution (A_1) is consistent with 2D low-energy spin fluctuations [19] and demonstrates their increasing importance as pressure drives the system closer to a magnetic ordering transition. However, unlike underdoped NaFe$_{1-x}$Co$_x$As, where $1/T_1T$ diverges at the onset of AFMLRO [14], our overdoped sample shows no divergence. Instead, the values of $|\theta|$ extracted from the fit at each pressure, shown in Fig. 3(b), approach the divergent regime but then increase again. We stress that $1/^{75}T_1T$ at low temperatures shows the same nonmonotonic pressure-dependence as T_c [Fig. 3(c)]; the low-energy spin fluctuations are optimized at the same pressure P_{opt}. This behavior is also reflected in the maximum of θ [Fig. 3(b)], which maximizes the Curie-Weiss term.

We conclude our data analysis by performing a detailed comparison between T_c and the low-energy spin-fluctuation contribution to $1/^{75}T_1T$. Figure 4 shows $1/^{75}T_1T$ at $T = 30$ K, directly above T_c, and T_c^{m} taken from Fig. 1, for all measured pressure values. The two quantities have an initial linear increase, begin to flatten above 1.7 GPa, are maximal at 2.17 GPa, and fall beyond this. To our knowledge, such a simultaneous optimization of T_c and the low-energy spin fluctuations in an unconventional superconductor has not been demonstrated before. We have achieved this optimization through the pressure dependence of both quantities while avoiding the structural and magnetic phase transitions. To make the relationship between magnetic fluctuations and superconductivity yet more explicit, in the inset of Fig. 4 we plot $1/^{75}T_1T|_{T=30\ \mathrm{K}}$ against T_c with pressure as the implicit parameter. The pressure-induced changes $\Delta(T_c)$ and $\Delta(1/^{75}T_1T|_{T=30\ \mathrm{K}})$ show a simple linear scaling behavior, valid both below and above the optimal pressure.

We begin our discussion by considering the low-energy spin fluctuations. Irrespective of the connection to superconductivity, such an optimization of spin fluctuations by changing the lattice parameters has also not been observed previously. This nonmonotonic change clearly cannot be described by any sort of effective (negative) doping, because doping always leads to AFMLRO in Fe-based superconductors, with the dome of optimal doping arising due to the competition between magnetic order and superconductivity. The behavior we observe also contrasts strongly with the effects of pressure in FeSe, where spin fluctuations increase monotonically until AFMLRO sets in [12].

Because the spin fluctuations can be optimized by pressure without a change of structural symmetry, our results demonstrate that the magnetic interactions are extremely sensitive to the exact lattice parameters, and therefore supply information important for a microscopic model. Although the iron-based superconductors have a complex, multiorbital electronic structure, the Fermi surfaces of NaFe$_{1-x}$Co$_x$As and their orbital composition have been well characterized by angle-resolved photoemission spectroscopy (ARPES) [5,6,20]. $\mathrm{NaFe}_{1-x}\mathrm{Co}_x\mathrm{As}$ is a quasi-2D system whose band structure is only weakly dispersive along the c axis. Under these circumstances, one expects that the primary effect of an applied pressure will be to compress the c-axis lattice parameter; this interpretation is consistent with the large but continuous increase of $^{75}\nu_q$ [Fig. 2(b)], which is determined by V_{zz}, the principal EFG in the tetragonal phase. Because the As sites lie above and below the Fe layers, c-axis compression increases the overlap between the Fe d_{xz} and d_{yz} orbitals and the As p orbitals. The pressure-enhanced low-energy spin fluctuations should thus be associated with improved Fermi-surface nesting of the d_{xz} and d_{yz} orbitals, a result confirmed by a recent study combining ARPES and NMR measurements on $\mathrm{NaFe}_{1-x}\mathrm{Co}_x\mathrm{As}$ [6].

However, the decrease in spin fluctuations beyond P_{opt} raises further questions. High-pressure synchrotron x-ray powder diffraction studies of NaFeAs found that the FeAs planes achieve a structure where the FeAs_4 tetrahedra are completely regular (all internal angles equal to 109.4°) at approximately 3 GPa [15]. This regular structure appears to optimize the superconducting transition temperature in many iron pnictides [21,22]. Although we cannot probe the lattice structure by NMR, our results for Co-doped NaFeAs certainly display a similar optimization as a function of lattice distortion, presumably as the "horizontal" and "vertical" As–Fe–As bond angles approach the regular value from opposite directions under pressure. Our data therefore imply that the empirical observation of a maximal T_c and the achievement of completely regular FeAs_4 tetrahedra [21,22] may be connected by the optimization of magnetic correlations. A possible origin for this effect could lie in the optimization of Fermi-surface nesting.

Considering the spin fluctuations in more detail, our data show that they have two different types in $\mathrm{NaFe}_{1-x}\mathrm{Co}_x\mathrm{As}$. One is the low-energy spin fluctuations, responsible for the Curie-Weiss upturn at low temperatures in $1/^{75}T_1T$. These usually arise due to itinerant electrons and are observed both by ARPES [23] and by inelastic neutron scattering [24] in compounds with good Fermi-surface nesting; they are peaked at the wave vector of the incipient AFMLRO, and hence dominate $1/^{75}T_1T$ [25,26] but are scarcely evident in the Knight shift. However, this upturn is weak in overdoped 1111 materials [9], completely absent in the intercalated iron selenide $\mathrm{K}_y\mathrm{Fe}_{2-x}\mathrm{Se}_2$ [27], and weak in $\mathrm{NaFe}_{0.94}\mathrm{Co}_{0.06}\mathrm{As}$ at ambient pressure (Fig. 3), and yet these systems all have a high T_c value. To identify the origin of strong pairing interactions in these compounds, we note that their Knight shifts increase significantly with temperature, as observed respectively in Ref. [9], Ref. [18], and Fig. 2(c). In fact, this strong thermal enhancement appears in both $^{75}K_{ab}$ and $1/^{75}T_1T$, meaning at all wave vectors, and its functional form [the relative linear (B_0, B_1) and quadratic (C_0, C_1) coefficients] is consistent with other indicators of predominantly 2D or 3D nature. This behavior is characteristic of fluctuating local moments [18], rather than itinerant electrons and a band-structure description [28]. Our data show that the low-energy spin fluctuations are strongly enhanced by the pressure (Fig. 3), whereas the local spin fluctuations are strongest at low pressures but weaken as P increases [Fig. 2(c)].

Turning now to the connection with superconductivity, the paradigm of a spin-fluctuation-mediated pairing interaction whose strength diverges at the magnetic instability in the random phase approximation was the foundation for several theories of high-temperature superconductors. However, in cuprates the separation in doping between the AFMLRO phase and the dome-shaped maximum in T_c is impossible to reproduce in this scenario. Here, we obtain a direct proof for the correlation between low-energy spin fluctuations and superconductivity by their simultaneous optimization, using pressure as the control parameter. This is a very strong statement in favor of a magnetic origin for superconductivity. We reiterate that the pressure-enhanced T_c we observe is correlated more directly with the low-energy spin fluctuations, caused by itinerant electrons, than with the local ones. This behavior is also manifest in the doping dependence of the two spin-fluctuation types, where the high-energy ones were found [29] to change little with electron doping in $\mathrm{BaFe}_2\mathrm{As}_2$ whereas significant changes were found in the low-energy ones.

Our observations also shed light on the question of whether superconductivity in iron-based materials requires low-energy spin fluctuations at all, given that these seem to be weak or absent in some systems. By monitoring the evolution of NMR response with pressure, we have shown how superconductivity is correlated with two types of spin fluctuation. To distinguish between their contributions, we note in the perfectly linear relation between $1/^{75}T_1T$ and T_c (inset, Fig. 4) that T_c extrapolates to a finite value (around 8 K) as $1/^{75}T_1T \rightarrow 0$. This indicates that low-energy spin fluctuations are not the only contribution to pairing and that superconductivity may arise in their absence. Given the presence of local spin fluctuations, which are strong at low pressures [Fig. 2(c)], we suggest that these are the short-range magnetic correlation effects providing the additional pairing interaction, which is dominant in some materials. In $\mathrm{NaFe}_{0.94}\mathrm{Co}_{0.06}\mathrm{As}$, our data show both local and low-energy spin fluctuations contributing to superconductivity at ambient pressure, whereas the latter dominate at high pressures; this balance of contributions will change with sample doping.

Finally, spin fluctuations are not the only candidate pairing mechanism in Fe superconductors. Pairing mediated by orbital fluctuations has been proposed in a five-band model with electron-phonon coupling [8]. Our data resolve this question. The direct correlation of T_c and $1/^{75}T_1T$ favors unequivocally a magnetic origin. Furthermore, phonon-mediated interactions are expected to increase monotonically with pressure, and so a nonmonotonic change in T_c does not appear to be consistent with the orbital-fluctuation scenario. A further consequence of this mechanism would be a conventional s^{++} pairing symmetry, which should result in an NMR coherence peak robust against disorder. We are uniquely positioned to comment on the pairing symmetry, and we find that $1/^{75}T_1T$ drops sharply below T_c; the coherence peak is absent at all pressures. This result indicates an unconventional pairing symmetry such as s^{+-}, which is sensitive to impurity scattering [30], again contradicting the orbital-fluctuation prediction. We found no evidence for a change of pairing symmetry under pressure.

In summary, we have demonstrated a direct connection between superconductivity and low-energy spin fluctuations in a high-temperature superconductor. We chose to analyze $NaFe_{0.94}Co_{0.06}As$, an overdoped system where both the structural phase transition and antiferromagnetic long-range order are avoided. We performed NMR measurements under an applied pressure, which allows clean and detailed control of both the lattice and electronic structures. We show that the spin fluctuations and the superconducting transition temperature change in lock step and are optimized at exactly the same pressure. This result strongly supports a magnetic origin for superconductivity. Our measurements also demonstrate the presence of two types of spin fluctuation, namely low-energy ones arising from itinerant electrons and finite-energy ones with a local nature, and that both contribute to pairing in the superconducting state.

The authors acknowledge helpful discussions with W. Bao, D.L. Feng, S.L. Li, and S.C. Wang. Work at Renmin University of China is supported by the National Science Foundation of China (Grants No. 11174365, No. 11222433, and No. 11374364) and the National Basic Research Program of China (Grants No. 2010CB923004, No. 2011CBA00112, and No. 2012CB921704). Work at North China Electric Power University is supported by the National Science Foundation of China (Grant No. 11104070). Work at the University of Tennessee and Rice University is supported by the U.S. DOE Office of Basic Energy Sciences under Grant No. DE-FG02-05ER46202.

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