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Possible Multiple Gap Superconductivity with Line Nodes in Heavily Hole-Doped Superconductor KFe_2As_2 Studied by ^{75}As Nuclear Quadrupole Resonance and Specific Heat

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We report the ^{75}As nuclear quadrupole resonance (NQR) and specific heat measurements of the heavily hole-doped superconductor KFe_2As_2 (superconducting transition temperature $T_c \simeq 3.5$ K). The spin-lattice relaxation rate $1/T_1$ in the superconducting state exhibits a gradual temperature dependence with no coherence peak below T_c. The quasiparticle specific heat C_{QP}/T shows a small jump, which is about 30% of the electronic specific heat coefficient just below T_c. The C_{QP}/T suggests the existence of low-energy quasiparticle excitation at the lowest measurement temperature $T = 0.4\,\mathrm{K} \simeq T_c/10$. The T dependences of $1/T_1$ and C_{QP}/T can be explained by a multiple nodal superconducting gap scenario rather than by a multiple fully gapped $s_{\pm}$-wave scenario determined using simple gap analysis.

KEYWORDS: KFe_2As_2, multiple superconducting gap, nuclear quadrupole resonance, specific heat
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After the discovery of superconductivity in F-doped LaFeAsO with a superconducting transition temperature $T_c = 26$ K,[1] K-doped (hole-doped) $BaFe_2As_2$ was reported as the first oxygen-free iron-pnictide superconductor with $T_c = 38$ K.[2] The crystal structure of $Ba_{1-x}K_xFe_2As_2$ is of the $ThCr_2Si_2$ type. We performed ^{75}As nuclear magnetic resonance (NMR) measurements of $Ba_{1-x}K_xFe_2As_2$ ($T_c \simeq 38$ K),[3] which also exhibits the first-order antiferromagnetic (AF) ordering associated with a structural phase transition. Our results clearly revealed that the coexistence of AF and superconducting (SC) states[4,5] is not a microscopic coexistence but a phase separation.

The important feature of $Ba_{1-x}K_xFe_2As_2$ is that superconductivity occurs even for $x = 1$,[4–6] although T_c itself is much lower ($T_c \sim 3.5$ K) than the optimum T_c. This implies the lower essentialness of carrier doping for the occurrence of superconductivity in 122 systems than in high-T_c cuprates. However, looking at the phase diagram of $Ba_{1-x}K_xFe_2As_2$ in ref. 4, T_c once decreases to zero from a maximum T_c side toward $x = 0.75$ with increasing x, but remains low at a value ($T_c \sim 10$ K) above about $x = 0.75$. Nearly the same tendency of the phase diagram is also seen in $Sr_{1-x}K_xFe_2As_2$.[6] This is probably related to the disappearance or shrinkage of the electron-like Fermi surface around the M point in the Brillouin zone induced by hole doping, which is indeed observed by angle-resolved photoemission spectroscopy (ARPES)[7] or confirmed by band calculation in KFe_2As_2.[8] Furthermore, this might bring a change in SC symmetry. In iron arsenide superconductors, it has been proposed that the multiple fully gapped $s_{\pm}$-wave Cooper pairing is preferable in many experimental and theoretical approaches.[9–16] On the other hand, the nodal-line SC symmetry scenario is proposed to explain the superconductivity in LaFePO due to the modification of the Fermi surface.[11,17,18] In this sense, KFe_2As_2 is a suitable candidate for verifying the general tendency of SC symmetry in Fe-based superconductors, since it is potentially cleaner than other substituted SC compounds and SC even with an extremely modified Fermi surface. In this Letter, we report the ^{75}As nuclear quadrupole resonance (NQR) and specific heat measurements of KFe_2As_2 ($T_c \simeq 3.5$ K). The characteristic temperature T dependences of the spin-lattice relaxation rate $1/T_1$ and the quasiparticle specific heat C_{QP}/T can be understood by the multiple nodal SC gap scenario.

Polycrystalline KFe_2As_2 was synthesized by a high-temperature and high-pressure method.[3] X-ray diffraction analysis revealed that the sample was of nearly single phase. The obtained lattice parameters namely, $a = 3.846$ Å and $c = 13.87$ Å, were consistent with previous reports.[4,5]

In order to check sample quality, we measured the resistivity $\rho(T)$ by a standard four-probe method (Fig. 1). The residual resistivity ratio (RRR) is $\rho(300\,\mathrm{K})/\rho(4.2\,\mathrm{K}) = 67$, which is comparable to the reported value (= 87) for single crystals of KFe_2As_2 ($T_c = 2.8$ K).[19] The onset of T_c is about 4.0 K and ρ becomes nearly zero at 3.5 K. We also determined T_c and SC volume fraction with a commercial superconducting-quantum-interference-device magnetometer (inset of Fig. 1). To reduce the extent of demagnetization effect, we used a plate-shaped sample with dimensions of $3.0 \times 3.0 \times 1.0\,\mathrm{mm}^3$ and an applied magnetic field parallel to the largest plane. The T_c of the sample is about 3.5 K, and the SC volume fraction is 80%. These results indicate that the quality of our sample is sufficient and that the superconductivity in KFe_2As_2 is a bulk property of the sample.

An NQR experiment on the ^{75}As nucleus ($I = 3/2$, $\gamma/2\pi = 7.292$ MHz/T) was carried out using phase-coherent pulsed NQR spectrometers. The samples were powdered for use in the experiments. The measurement above 1.4 K was performed using a ^4He cryostat, and between 0.3 and 1.2 K with a ^3He refrigerator. We measured specific heat by a

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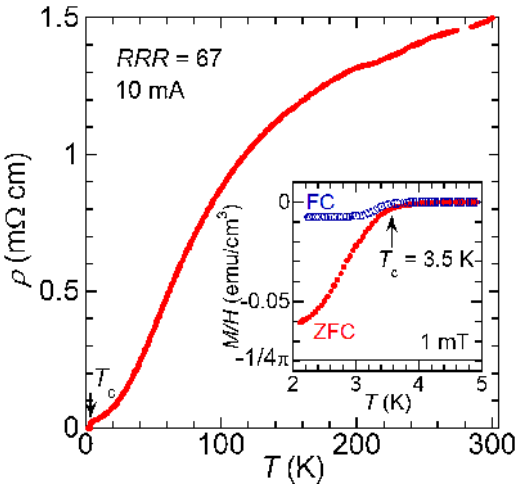

Fig. 1. (Color online) Resistivity of KFe_2As_2. The inset shows the magnetic susceptibility of KFe_2As_2 in the paramagnetic state. Solid and open symbols denote the data obtained after zero-field cooling (ZFC) and field cooling (FC), respectively.

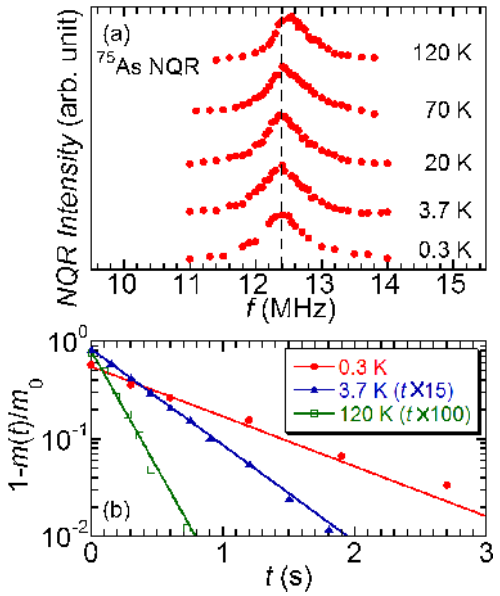

Fig. 2. (Color online) (a) ^{75}As NQR spectra of KFe_2As_2 at various temperatures. (b) Nuclear magnetization recovery curves of KFe_2As_2 at 0.3, 3.7, and 120 K. Solid lines denote the fitting curve obtained using the formula in the text.

thermal relaxation method between 0.4 and 10 K with a commercial calorimeter.

In Fig. 2(a), we show the ^{75}As-NQR spectra of KFe_2As_2 at various T's. A clear single peak signal was observed. With decreasing T, the spectral center decreases and remains nearly constant below about 70 K. Below this temperature, the NQR frequency ν_Q was 12.4 MHz. The principal axis of the electric field gradient is along the crystal c-axis since the As site has a local fourfold symmetry around the c-axis. With increasing x, the ν_Q in $Ba_{1-x}K_xFe_2As_2$ increases from $\nu_Q =$ 2.2 MHz ($x = 0$)[20,21] to 12.4 MHz ($x = 1$) through an intermediate value of $\sim$5 MHz ($x = 0.4$).[3] The full width at half maximum (*FWHM*) of the spectra for $x = 1$ remains nearly constant above T_c and is about 740 ± 20 kHz. This is about half of the magnitude of the linewidth of oxygen-deficient $LaFeAsO_{1-\delta}$[22] and F-doped $LaFeAsO_{1-x}F_x$.[23] The *FWHM* slightly increases below T_c and becomes about 850 kHz at 0.3 K. However, we cannot conclude that this is due to magnetic order since the increase in *FWHM* is too small.

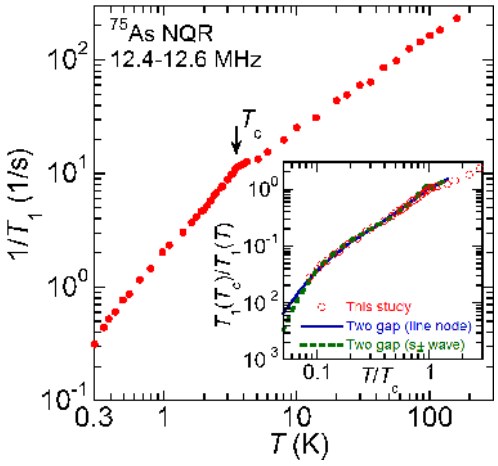

Fig. 3. (Color online) $1/T_1$ of KFe_2As_2. The inset shows the normalized results of the experiment and analysis. We assumed a two-independent-SC-gap model for a nodal gap or a full gap.

In Fig. 2(b), we show the nuclear magnetization recovery curves of KFe_2As_2 at 0.3, 3.7, and 120 K. All the obtained recovery curves followed the single exponential curve expected for ^{75}As NQR ($I = 3/2$);[24]

$$1 - \frac{m(t)}{m_0} = \exp\left(-\frac{3t}{T_1}\right),$$

where $m(t)$ and m_0 are the nuclear magnetization after a time t from the NQR saturation pulse and the thermal equilibrium magnetization, respectively. The small deviation in data points from the fitting curve at 0.3 K may be due to the distribution of T_c. However, its fitting error is within the marker size in Fig. 3. We checked the absence of sample heat up by changing NQR pulse width and power.

In Fig. 3, we show the T dependence of $1/T_1$ of KFe_2As_2. In the normal state, $1/T_1$ follows $T^{0.8}$ between 5 and 160 K. This is slightly weak compared with the T linear dependence of the conventional Korringa law, which suggests that antiferromagnetic fluctuation is much suppressed and that the system is nearly Pauli paramagnetic. The most striking feature of $1/T_1$ is its T dependence below $T_c = 3.5$ K. No coherence peak was observed just below T_c, and $1/T_1$ follows only $T^{1.4}$ between 0.6 K and T_c. This is different from the $1/T_1$ below T_c in $Ba_{1-x}K_xFe_2As_2$ with $T_c \simeq 38$ K in which $1/T_1$ follows $T^3 - T^5$ with no coherence peak.[3,16,25] The $1/T_1$ of KFe_2As_2 more steeply decreases below about 0.6 K. Such a weak T dependence of $1/T_1$ in the SC state was observed in $La_{0.87}Ca_{0.13}FePO$.[26]

In order to understand the nature of the above-mentioned anomalous T dependence, we analyzed the data by assuming a simple two-independent-SC-gap model with a gap with a line node or a full gap ($s_\pm$ wave type). Similar analysis procedure was adopted in refs. 16 and 25. Large two-hole Fermi surfaces (α and β in ref. 7) are observed around the Γ point by ARPES in KFe_2As_2.[7] These Fermi surfaces have different gaps in $Ba_{1-x}K_xFe_2As_2$ ($T_c \simeq 38$ K),[14–16,26] and are considered to mainly contribute to the total density of states (DOS) at the Fermi level in KFe_2As_2.[7,8] Therefore, it is natural to analyze the two-independent-SC-gap model.

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Furthermore, we assumed two SC gap symmetries because these symmetries are experimentally and theoretically favorable for iron-based superconductors.[9–17,25] The $1/T_1$ in the SC state is proportional to

$$\frac{1}{T_1} \propto \sum_{i=1,2} n_i^2 \int_0^\infty [N_S^i(E)^2 + M_S^i(E)^2] f(E)[1 - f(E)]\, dE,$$

where $N_S^i(E)$, $M_S^i(E)$, and $f(E)$ are the DOS, anomalous DOS arising from the coherence effect of Cooper pairs, and Fermi distribution function, respectively. n_i represents the fraction of DOS of the i-th gap and $n_1 + n_2 = 1$. In both cases of SC gaps, we assumed that the integral of $M_S^i(E)$ becomes zero because of the sign-changing SC gaps. In the full-gap case, we averaged $N_S^i(E)$ at approximately $E \sim \Delta_i(T)$ with the width $2\delta_i$ [$< \Delta_i(T)$] in order to reduce the size of the coherence peak.[27,28] Here, $\Delta_i(T)$ is the SC gap; for simplicity, the (θ, φ) dependence is given by $\Delta_i(T, \theta, \varphi) = \Delta_i(T) \cos\theta$ for the nodal gap and by $\Delta_i(T, \theta, \varphi) = \Delta_i(T)$ for the full gap. A concrete function for the average $N_S^i(E)$ is described in ref. 27. This procedure corresponds to the anisotropy of the SC gaps or finite lifetime of Cooper pairs arising from pair breaking.[29] In order to distinguish the nodal- and the full-gap models, we utilized the above procedure, which is different from that described in refs. 16 and 30, in which a finite DOS appears at the Fermi level in the full-gap model. The clear difference in total DOS is depicted in Fig. 4 using the parameters described below.

In the inset of Fig. 3, we show the normalized results of the experiment and analysis. In Table I, we summarized the obtained values of $2\Delta_i(0)/T_c$, n_1, and δ_1/Δ_1 (for the full-gap model). The gap parameters are nearly the same for all models, and the fitting to the experimental results is sufficiently good. This suggests that the ^{75}As-NQR down to the lowest measurement temperature of $\sim T_c/10$ cannot solely determine the SC symmetry of KFe_2As_2. The larger gap is of moderately strong coupling, while the smaller gap is of considerably weak coupling. This indicates that the smaller gap is induced by the emergence of a larger gap. Note that single gap analysis ($n_1 = 1$) does not entirely work for both SC symmetries.

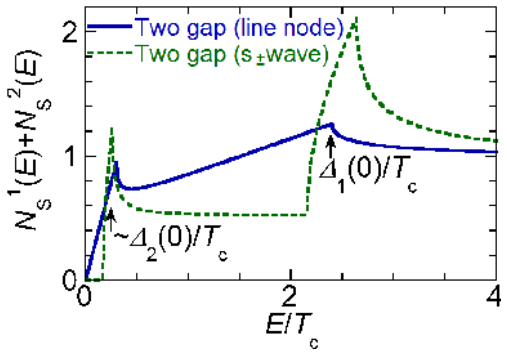

Fig. 4. (Color online) Total density of states used for the analysis.

Table I. Analyzed SC gaps, fractions of DOS, and widths of broadening of gap for the experimental $1/T_1$.

Type of gap	$2\Delta_1(0)/T_c$	$2\Delta_2(0)/T_c$	n_1	δ_1/Δ_1
Line node	4.8	0.60	0.45	—
Fully gapped $s_\pm$	4.8	0.44	0.48	0.1

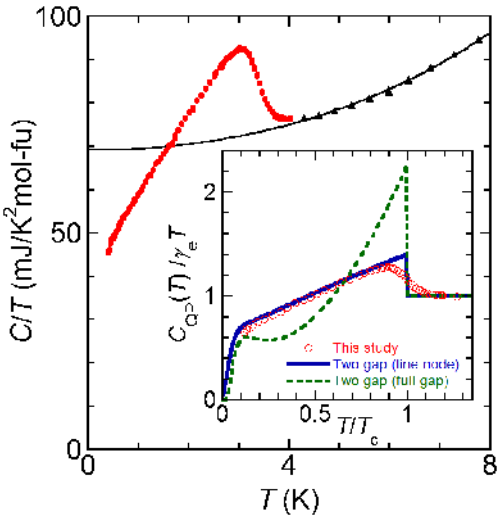

Fig. 5. (Color online) Specific heat divided by temperature $C(T)/T$ of KFe_2As_2. The solid curve represents the fitting (C_{fit}/T) for the data between 4 and 10 K (data below 8 K is shown). The fitting function is described in the text. The inset shows the normalized results of the experiment and numerical calculations. We assumed a two-independent-SC-gap model for a nodal gap or a full gap and used the parameters summarized in Table I.

In Fig. 5, we show specific heat divided by the temperature $C(T)/T$ of KFe_2As_2. A clear specific heat jump was observed, which again indicates the bulk nature of superconductivity. The midpoint of the jump is 3.4 K. In order to estimate the electronic specific heat coefficient γ_e and lattice contribution, we performed the fitting ($C_{fit}/T = \gamma_e + \beta T^2 + \varepsilon T^4$) for the data between 4 and 10 K (solid curve in Fig. 5). The obtained γ_e is 69.1(2) mJ/(K^2·mol), which is comparable to 63.3 mJ/(K^2·mol) for a single crystal of $Ba_{0.6}K_{0.4}Fe_2As_2$.[31] Note that the Schottky specific heat of ^{75}As is negligible since the Schottky specific heat calculated from the NQR frequency $\nu_Q = 12.4$ MHz is significant below about 0.1 K.

By subtracting the lattice contribution $\beta T^2 + \varepsilon T^4$, we obtained the quasiparticle specific heat C_{QP}/T. The most evident feature of C_{QP}/T is that the specific heat jump is only about 30% of γ_e. One of the reasons for this is the broadening of the SC transition arising from the small distribution of T_c. However, by taking into account this broadening, the specific heat jump is roughly expected to be at most 60% of γ_e. Another important feature is the finite $C_{QP}/T \simeq 45$ mJ/(K^2·mol-fu) even at the lowest measurement temperature $\sim T_c/10$. This suggests the existence of low-energy quasiparticle excitation.

In the inset of Fig. 5, we compared the normalized results of experiment and numerical calculations. Here, we again assumed a simple two-independent-SC-gap model with a gap with a line node or a full gap. Quasiparticle specific heat is given as

$$\frac{C_{QP}}{T} \propto \sum_{i=1,2} \frac{n_i}{T^3} \int_0^\infty N_S^i(E) \left[E^2 - \frac{T}{2} \frac{d\Delta_i(T)^2}{dT} \right] \times f(E)[1 - f(E)]\, dE.$$

To calculate specific heat, we used the parameters summarized in Table I. Clearly, the calculated result of the nodal SC gap model is in good agreement with the experimental result, except for the shape around the SC transition. In

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particular, this calculated result has a small specific heat jump and exhibits low-energy quasiparticle excitation. Therefore, we can conclude that the multiple gap superconductivity with a gap with a line node is realized in KFe_2As_2 by a simple two-gap analysis. The E linear part between 0.5 and 2 in the total DOS for the nodal gap model in Fig. 4 gives rise to a wide T linear part in C_{QP}/T between $T_c/5$ and $4T_c/5$. Note that such a correspondence between C_{QP}/T and total DOS can be seen in the results of the specific heat (Fig. 7 in ref. 31) and $1/T_1$ [Fig. 4(d) in ref. 16] of $Ba_{0.6}K_{0.4}Fe_2As_2$. It is also suggestive that the specific heat jumps divided by γ_e, $\Delta C_{QP}/\gamma_e T_c$, in other typical multiple gap superconductors are small ($\Delta C_{QP}/\gamma_e T_c \simeq 0.75$) for nodal-gap Sr_2RuO_4[32] and reasonably large ($\Delta C_{QP}/\gamma_e T_c \simeq 1.2$) for full-gap MgB_2.[33] In our present analysis, the rapid decreases in $1/T_1$ and C_{QP}/T are expected below $T_c/10$, below which the effect of residual DOS becomes significant. Further studies below this temperature using single crystals are needed.

In the present study, we clarified that iron-pnictide superconductors with an intrinsically low $T_c \sim 10$ K including FeP-based compounds[17,18] have a nodal SC gap. This is supported by a theory from the viewpoint of modified Fermi surfaces and the change of favorable wave vectors for superconductivity,[11] although its band calculation is based on rather lightly doped LaFeAsO and LaFePO. Moreover, it is suggested that an SC symmetry change occurs at $x \sim 0.75$ ($= 3/4$) in the phase diagram of $A_{1-x}K_xFe_2As_2$ (A = Ba, Sr). Hence, it is required from both experimental and theoretical aspects to determine which Fermi surface or wave vector is responsible for superconductivity in KFe_2As_2 and to clarify the properties of the possible phase boundary at $x \sim 3/4$.

In summary, we performed ^{75}As NQR and specific heat measurements of the heavily hole-doped superconductor KFe_2As_2 ($T_c \simeq 3.5$ K). The $1/T_1$ in the normal state reflects a nearly Korringa-like T dependence. The $1/T_1$ in the SC state exhibits a weak T dependence with no coherence peak. The C_{QP}/T shows a small jump just below T_c. The C_{QP}/T suggests the existence of low-energy quasiparticle excitation at $T = 0.4\,\mathrm{K} \simeq T_c/10$. The T dependences of $1/T_1$ and C_{QP}/T can be explained by a multiple nodal superconducting gap scenario rather than by a multiple fully gapped $s_\pm$-wave scenario which is the most plausible scenario to describe the SC state in $Ba_{1-x}K_xFe_2As_2$ with $T_c \simeq 38$ K. Further studies using theoretical and experimental methods (e.g., ARPES, de Haas–van Alphen effect, and penetration depth), especially using single crystals at lower temperatures, are required to more precisely determine the SC gap symmetry. Moreover, Knight shift measurement below T_c is also required to determine the spin part symmetry of KFe_2As_2.

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