Superconducting and normal-state properties of single-crystalline $Tl_{0.47}Rb_{0.34}Fe_{1.63}Se_2$ as seen via ^{77}Se and ^{87}Rb NMR

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We report both ^{77}Se and ^{87}Rb nuclear magnetic resonance (NMR) studies on $Tl_{0.47}Rb_{0.34}Fe_{1.63}Se_2$ single-crystalline superconductors ($T_c \approx 32$ K). Singlet superconductivity is suggested by a sharp drop of the Knight shift $K(T)$ below T_c, after subtracting the superconducting diamagnetic effect. However, the Hebel-Slichter coherence peak below T_c is not observed in the spin-lattice relaxation rate $1/T_1$, even with a low in-plane NMR field of 2.6 T. Just above T_c, no evidence of low-energy spin fluctuation is found in the spin-lattice relaxation rate on both the ^{77}Se and the ^{87}Rb sites. Upon warming, however, the Knight shifts and the spin-lattice relaxation rates of both nuclei increase substantially with temperature. In particular, the Knight shift is nearly isotropic and follow a function fit of $K = a + bT^2$ from T_c up to 300 K. Our observations should put a strong constraint to the theory of magnetism and superconductivity in the recently discovered iron selenide superconductors.

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I. INTRODUCTION

As the second type of high-temperature superconductors after cuprates, the iron-based superconductors[1–4] have attracted a lot of research interests in recent years. The iron pnictide superconductors are remarkable in that they originate from antiferromagnetic (AFM) semimetals[4] and have multiple electron and hole bands on the Fermi surface which are gapped in the superconducting state.[5,6] Lately, a family of iron selenide superconductors, with nominal chemical formulas $A_yFe_{2-x}Se_2$ or (Tl, $A)_yFe_{2-x}Se_2$ (A = K, Rb, Cs),[7–11] have been discovered with a T_c as high as 33 K. Comparing with iron pnictides, this iron selenide family shows qualitatively different properties in lattice structure, band structure, and magnetic properties, as described below, and thus enrich and challenge our understanding of the iron-based superconductivity.

First, the composition of this type of superconductors is suggested to be close to $A_{0.8}Fe_{1.6}Se_2$ by the x-ray and the neutron diffraction structure refinement studies.[12,13] This chemical stoichiometry leads to an almost perfect Fe-vacancy order in superconductors with a $\sqrt{5} \times \sqrt{5} \times 1$ supercell at low temperatures.[12–16] Second, the angle-resolved photoemission spectroscopy (ARPES) studies found that the hole bands centered at the Γ point sink below the Fermi level,[17,18] which is different from previous iron pnictide and chalcogenide superconductors.[19–21] Third, nodeless superconducting gaps are observed on the electron pockets on the Fermi surface.[17,19–21] NMR studies on nominal $K_{0.8}Fe_{2-x}Se_2$ show a singlet pairing symmetry from the Knight shift, whereas the Hebel-Slichter coherence peak is not observed in the spin-lattice relaxation rate.[22] The $s^{\pm}$ gap symmetry, proposed in iron pnictides with interband transitions,[23,24] may not be applicable here because of the absence of the hole band on the Fermi surface. Finally, an AFM order with large magnetic moments (2.3–3.3 μ_B/Fe) has been determined by neutron diffraction[13,25] for all of the these iron selenide superconductors and possibly coexists with bulk superconductivity, as indicated by the μSR and other bulk measurements.[13,26,27] From NMR, however, the Curie-Weiss upturn in $1/T_1T$, as an indication of low-energy spin fluctuations observed in most iron pnictides,[28–35] is not seen in $K_{0.8}Fe_{2-x}Se_2$ (Ref. 22).

In the previous NMR studies, the superconducting diamagnetic effect is not counted, which may preclude a strong evidence for a singlet pairing.[22,36,37] It should also be noted that the large NMR magnetic field used in those studies may suppress the Hebel-Slichter coherence peak. Furthermore, a large increase of the Knight shifts and the spin-lattice relaxation rates with temperature are also observed in the normal state with an unknown origin. So far NMR data are only available in $K_{0.8}Fe_{2-x}Se_2$. NMR studies in the same structure family are seeking to verify and understand these observations.

In this paper, we report both ^{77}Se and ^{87}Rb NMR studies on a single-crystalline $Tl_{0.47}Rb_{0.34}Fe_{1.63}Se_2$ superconductor. The paper is organized as following. First, we compare the Knight shifts of ^{77}Se and ^{87}Rb in the superconducting state. The superconducting diamagnetic effect are evaluated, owing to the differences in the hyperfine coupling strength of the two nuclei. Second, we investigate the spin-lattice relaxation rate under different NMR fields below T_c. The coherence peak is not observed even with a 2.6-T in-plane field. Finally, we study the Knight shift and the spin-lattice relaxation rate in the normal state and discuss the possible spin fluctuations. Detailed comparisons with $K_{0.8}Fe_{2-x}Se_2$ are also given in the paper.

II. EXPERIMENTAL TECHNIQUES AND RESULTS

The $Tl_{0.47}Rb_{0.34}Fe_{1.63}Se_2$ single crystals were synthesized by the Bridgeman method.[11] The chemical composition, determined by the inductively coupled plasma (ICP) analysis, is consistent with the Fe vacancy order in a general stoichiometric structure of $A_{0.8}Fe_{1.6}Se_2$ (or $A_2Fe_4Se_5$).[14] In this paper, we primarily report our results on one single crystal with dimensions of $\sim 5 \times 3 \times 1$ mm^3 ($T_c \approx 32$ K at zero field). The superconducting transition was monitored *in situ* by the ac susceptibility measurements with the NMR coil. In Fig. 1(a), T_c is shown at about 31 K with an 11.62-T field oriented along the crystalline c axis.

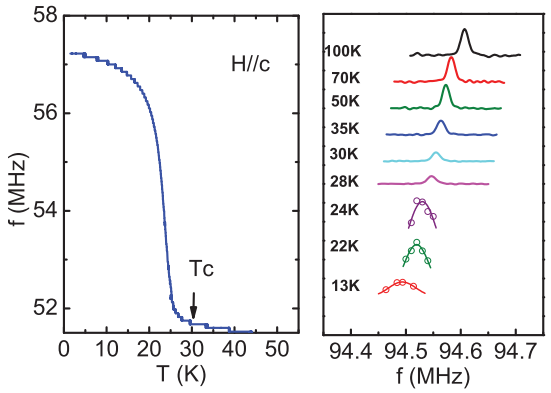

FIG. 1. (Color online) (a) The temperature shift of the tuning frequency f of a fixed NMR circuit, with the field (11.62 T) oriented along the crystalline c axis. The large frequency shift below T_c is related to the ac susceptibility χ_{ac} of the sample, $\Delta f \propto -\chi_{ac}$. T_c of the sample is determined as 31 ± 1 K with the applied field. (b) The ^{77}Se NMR spectra at different temperatures. For data below 28 K, the circles represent the integrated spectral intensity, and the solid lines are Gaussian fits to the data.

The ^{77}Se ($S = 1/2$) NMR is performed under an 11.62-T NMR field, and ^{87}Rb ($S = 3/2$) NMR is performed under 11.62- and 2.6-T fields. We also studied the field anisotropy effect with field along both the crystalline c axis and the ab plane. Typical ^{77}Se spectra with $H//c$ are shown in Fig. 1(b). A linewidth about 20 kHz is observed at $T = 35$ K, and narrows down as temperature increases. Below T_c, the spectra broaden and shift to lower frequencies. The ^{87}Rb has one center transition and two satellites ($\nu_q \approx 1.4$ MHz, data not shown).

The Knight shifts $^{77}K(T)$ and $^{87}K(T)$ are obtained from $K(T) = (f - \gamma_n B)/\gamma_n B$, where f is the measured resonance frequency at magnetic field B, and $^{77}\gamma_n = 8.131$ MHz/T and $^{87}\gamma_n = 13.931$ MHz/T are the gyromagnetic ratios of two respective nuclei. The Knight shift data of ^{77}Se and ^{87}Rb are shown in Figs. 2 and 3, respectively.

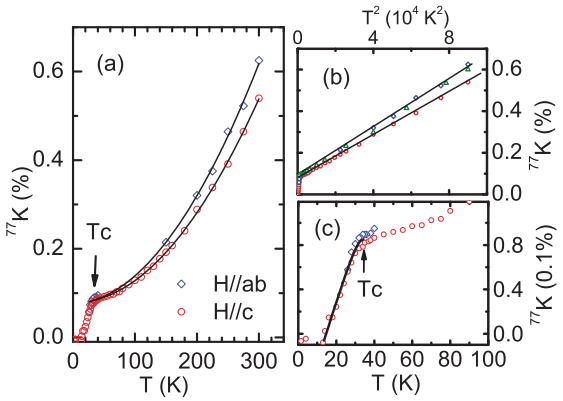

FIG. 2. (Color online) (a) The temperature dependence of the Knight shift ^{77}K under an 11.62-T NMR field with $H//ab$ (open diamonds) and $H//c$ (open circles). The solid lines are guides for the eye. (b) Replot of ^{77}K as a function of T^2 for both field orientations, and the solid lines are function fits to $K(T) = a + bT^2$. The triangles are data for $K_{0.8}Fe_{1-x}Se_2$ ($T_c \approx 32$ K) with $H//ab$, adapted from Ref. 22. (c) The enlarged view of the low-temperature behavior of ^{77}K with two field orientations.

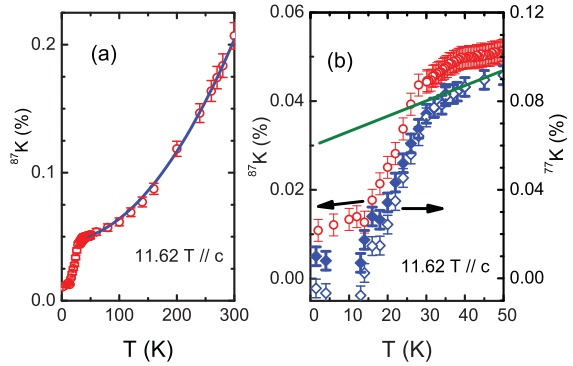

FIG. 3. (Color online) (a) The temperature dependence of the Knight shift ^{87}K under an 11.62-T NMR field with $H//c$. The solid line is a function fit to $K(T) = a + bT^2$. (b) Replot of the low-temperature part of ^{87}K (open circles) and ^{77}K (open diamonds) under the same field condition. The solid line is a guide to the normal state ^{77}K. The solid diamonds with error bars represent the values of ^{77}K after subtracting the superconducting diamagnetic effect (see text).

The spin-lattice relaxation rate $1/T_1$ is measured by the inversion-recovery method and is obtained from the fitting $I(t)/I(0) = 1 - ae^{-t/T1}$ for ^{77}Se and $I(t)/I(0) = 1 - a(0.1e^{-t/T_1} + 0.9e^{-6t/T1})$ for ^{87}Rb ($a \geqslant 1$). The fitting works nicely at all measured temperatures, indicating a single phase. The $1/^{77}T_1$ and $1/^{87}T_1$ data are shown in Figs. 4 and 5, respectively.

III. THE NMR KNIGHT SHIFT AND THE SINGLET SUPERCONDUCTIVITY

The ^{77}Se Knight shift data of $Tl_{0.47}Rb_{0.34}Fe_{1.63}Se_2$ are shown in Fig. 2(a). The measurements were performed with field along either the ab plane (open diamonds) or the c axis (open circles). Above T_c, the ^{77}K for both field orientations increases monotonically with temperature. In fact, both sets of data can be nicely fit by a simple function $K(T) = a + bT^2$ with temperature from T_c to 300 K, as shown in Fig. 2(b). The fitting parameters are given as

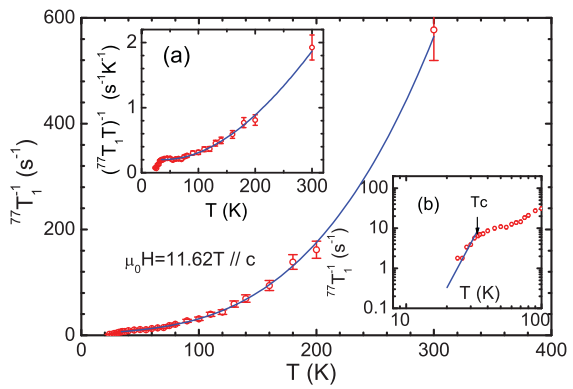

FIG. 4. (Color online) The temperature dependence of the spin-lattice relaxation rate $1/^{77}T_1$ with an 11.62-T field oriented along the c axis. The solid line is a guide for the eye. Insets: (a) $1/^{77}T_1T$ vs temperature; (b) the low-temperature $1/^{77}T_1$ data highlighted with a log-log scale. The solid line is a guide for the eye.

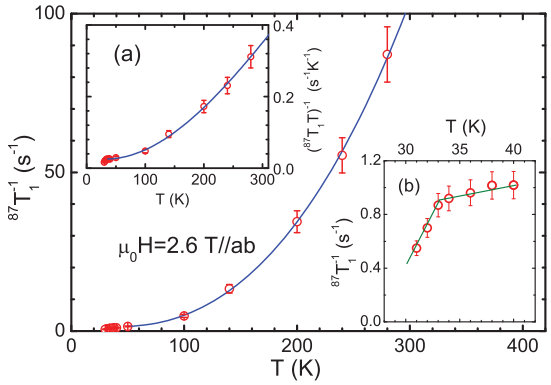

FIG. 5. (Color online) The temperature dependence of the spin-lattice rate $1/^{87}T_1$ with a 2.6-T field oriented along the ab plane. The solid line is a guide for the eye. Insets: (a) $1/^{87}T_1T$ vs temperature; (b) the highlighted low-temperature $1/^{87}T_1$ data.

$a \approx 0.1\%$ and $b \approx 6 \times 10^{-6}\%/\mathrm{K}^2$ for $H//ab$ and $a \approx 0.1\%$ and $b \approx 5.5 \times 10^{-6}\%/\mathrm{K}^2$ for $H//c$. The low-temperature part of $^{77}K(T)$ is highlighted in Fig. 2(c) to show a fast drop of ^{77}K from T_c to $T_c/2$ (about 15 K) with both field orientations. Below 15 K, the spectral intensity is very small due to RF screening, and we believe our Knight shift is not well determined because that vortex core also contributes to the spectrum.

The ^{87}Rb Knight shift data of $Tl_{0.47}Rb_{0.34}Fe_{1.63}Se_2$ are shown in Fig. 3(a). The measurements were performed with field along the crystalline c axis. Above T_c, the ^{87}K also increases with temperature, following the same $a + bT^2$ behavior (see the solid line fitting).

We first discuss the Knight shift data in the superconducting state and its implication to the pairing symmetry. Below T_c, the total shift K includes three parts $K = K_d + K_s + K_c$, where K_d, K_s, and K_c represent the superconducting diamagnetic shielding, the spin, and the chemical (or orbital) contribution to the frequency shift. In principle, K_c does not change with temperature, while K_d decreases from zero to negative below T_c, and K_s measures local electron susceptibility. Therefore, below T_c, the change of the shift follows $\Delta K(T) = \Delta K_s(T) + \Delta K_d(T)$ with ΔK defined as $\Delta K = K(T) - K(T_c)$. Usually, the K_s and the K_d are difficult to separate. In the current compound, due to a weaker hyperfine coupling on the interlayer ^{87}Rb site, as shown by the smaller Knight shift, K_s and K_d can be estimated by comparing the ^{77}K and the ^{87}K under the same field condition [see Fig. 3(b)], as described below.

Let us first look at the Knight shifts at a specific temperature $T = 16$ K. The ^{87}K decreases by about $0.028 \pm 0.004\%$ from T_c to 16 K, which gives the diamagnetic shift of $-0.028\% < \Delta K_d < 0$. It is reasonable to assume that K_d is the same for both nuclei from the same diamagnetic shielding; therefore, the value of $^{77}K_s + {}^{77}K_c$ for ^{77}Se should be moved up by the same amount $0.028 \pm 0.004\%$ (or less) from the $^{77}K(T)$ ($0.015 \pm 0.004\%$). This gives $^{77}K_s + {}^{77}K_c \leqslant 0.043 \pm 0.008\%$ at 16 K, which is far below the guide line of the normal state ^{77}K.

The K_d and K_s can also be calculated with a higher precision as follows. We first estimate $^{87}K_s/{}^{77}K_s \approx 0.4$ from the change of the Knight shift in the temperature range between T_c and 300 K, because the ^{77}K and the ^{87}K increase with the same T^2 behavior. Then the values of K_d and K_s are separated from the ^{77}K and ^{87}K data at each temperature below T_c, using $^{87}K_s/{}^{77}K_s = 0.4$. We plot the resulting value of $^{77}K_s + {}^{77}K_c$ below T_c in Fig. 3(b) (the solid diamonds). The data show that the diamagnetic shift is less than 0.02% even at the lowest temperature.

A sharp drop in ^{77}K just below T_c, after the subtraction of the diamagnetic effect, is clearly seen, indicating a singlet superconductivity. This confirms the previous results in $K_{0.8}Fe_{2-x}Se_2$, although the diamagnetic effect was not considered there.[22] The singlet pairing puts a strong constraint to the superconducting mechanism and narrows down the gap to s-wave, d-wave, or other even orbital symmetry.

IV. THE SPIN-LATTICE RELAXATION RATE AND THE COHERENCE PEAK

We further study the spin-lattice relaxation rate below T_c to understand the pairing symmetry. Usually, the $1/T_1$ far below T_c follows an activation behavior for a conventional s-wave superconductor. Unfortunately, the strong RF screening of our single-crystal limits our studies of T_1 to a narrow temperature range below T_c.

The $1/^{77}T_1$ data with field oriented along the c axis is shown in Fig. 4. Above T_c, the $1/^{77}T_1$ increases with temperatures. Below T_c, a sharp drop of $1/^{77}T_1$ is clearly shown by the low-temperature highlight in Fig. 4(b). In Fig. 5, we show the $1/^{87}T_1$ data of ^{87}Rb with a 2.6-T field oriented along the ab plane. We are able to measure $1/^{87}T_1$ below T_c due to the higher natural abundance of ^{87}Rb. A sharp drop of $1/^{87}T_1$ is also prominent, as shown in Fig. 5(b).

The above data show that the Hebel-Slichter coherence peak is absent in the $1/T_1$ just below T_c for both nuclei. Since the $H_{c2}^{ab}(0)$ is over 60 T for this class of iron selenide superconductors,[38,39] the coherence peak is unlikely to be suppressed with the 2.6-T in-plane field. The absence of the coherence peak in the current compound is consistent with the result reported in $K_{0.8}Fe_{2-x}Se_2$ (Refs. 22,36 and 37).

Usually, the absence of a coherence peak is interpreted as evidence for d-wave or other non-s-wave pairing symmetry. In most iron pnictides, the coherence peak is not observed by far,[40–46] which is proposed as a consequence of the $s^{\pm}$ pairing symmetry with interband impurity scattering.[47–51] In the current iron selenide superconductors, however, the $s^{\pm}$ scenario with interband transitions may not be applicable, because the hole band is absent on the Fermi surface close to the Γ point. The absence of the coherence peak suggests that the system is different from a conventional s-wave superconductor and needs to be further addressed. It may be worthwhile to note that an $s^{\pm}$ symmetry is proposed in $K_{0.8}Fe_{2-x}Se_2$ considering the coupling between the bonding and the antibonding states on the Fermi surface.[52]

V. THE LOW-TEMPERATURE SPIN DYNAMICS

Next we discuss the magnetic properties from the Knight shift and the spin-lattice relaxation rate data in the normal state. In Fig. 4(a), the $1/^{77}T_1T$ is plotted versus temperature to compare with the Fermi liquid behavior ($1/T_1T = \text{const.}$).

Our data show that $1/^{77}T_1T$ reaches a constant value when the temperature is close to T_c. $1/^{87}T_1T$ also shows a similar temperature dependence as seen in Fig. 5(a). The Knight shift data, ^{77}K and ^{87}K, also level off toward T_c [see Figs. 2(a) and 3(a)]. The levels off of both the Knight shift and the $1/T_1T$ suggest that the system approaches a normal Fermi liquid behavior at low temperatures, with a constant density of states at the Fermi level.

In contrast, the binary iron selenide (FeSe) superconductors show strong AFM spin fluctuations by a Curie-Weiss upturn in $1/T_1T \sim 1/(T+\Theta)$[28] above T_c. Similarly, the Curie-Weiss upturn in $1/T_1T$ has also been reported in many iron arsenide superconductors.[29–35,53] The existence of AFM spin fluctuations just above T_c in the previous iron-based superconductors draw a correlation between spin fluctuations and superconductivity.[28–35]

The absence of the Curie-Weiss upturn in $1/^{77}T_1T$ in the current material and also in $K_{0.8}Fe_{2-x}Se_2$[22] suggests that the spin fluctuations are weak in both compounds, although their T_c's are also high. These distinctive behaviors of the spin-lattice relaxation rate in the current iron selenides does not seem to support that the superconductivity has a magnetic origin. We note that in several iron pnictides with low T_c, the spin fluctuations appear to be weak.[54–56]

However, we should be aware that spin fluctuations may exist in other forms. For example, some spin fluctuations at the wave vector $(\pm\pi, \pm\pi)$ cannot be detected in the current spin-lattice relaxation rate, due to the cancellation effect on the Se and Rb sites which are located at the center of the Fe square. Furthermore, as we show below, the spin fluctuations may exist in the high-energy modes according to our high-temperature data.

VI. THE HIGH-TEMPERATURE SPIN DYNAMICS

To further compare with a Fermi liquid behavior, we plot $(T_1T)^{-0.5}$ against $K(T)$ in Figs. 6(a) and 6(b) for both nuclei. Usually, a Fermi liquid follows the Korringa relation $(T_1T)^{-0.5} \propto K_s$. As shown in Fig. 6(a), a linear relation between $(T_1T)^{-0.5}$ and $K(T)$, as demanded by the Korringa relation, is approximately satisfied for ^{77}Se in the low-temperature limit. However, the Korringa relation is not obeyed in the high-temperature range, indicating a deviation

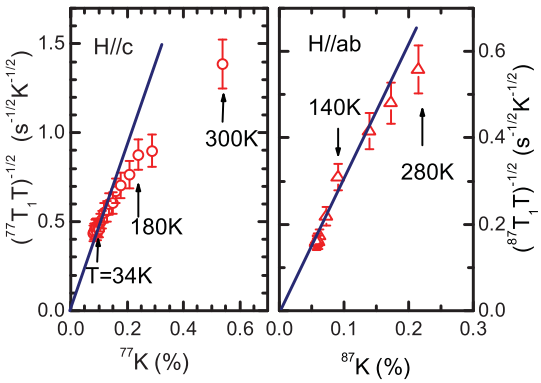

FIG. 6. (Color online) The plot of $(T_1T)^{-0.5}$ vs $K(T)$ with temperature as an implicit parameter. The solid line represents the Korringa relation.

from the Fermi liquid behavior. Here we estimated $K_c \approx 0$ based on the value of K at $T \ll T_c$, where $K_s(T) = 0$ is expected [see Fig. 3(b)]. For ^{87}Rb, the Korringa relation does not hold either at high temperatures.

Now we look closely at the high-temperature behavior of the Knight shift and the spin-lattice relaxation rate. The $1/T_1T$ of two nuclei increases substantially with temperature. The Knight shift data ^{77}K and the ^{87}K increase by over a factor of three, when the temperature increases from T_c to the room temperature, as shown in Figs. 2 and 3. The Knight shift ^{77}K of $K_{0.8}Fe_{2-x}Se_2$ with $H//ab$ is also plotted in Fig. 2(b), and it falls on the same line of the current compound. This suggests a universal electronic structure in these iron selenide superconductors. In contrast, in most iron pnictides, the Knight shift shows a much smaller increase with a linear T behavior.[32]

The anisotropy of the Knight shift is small with $K_s^{ab}/K_s^c \approx 1.1$ in the studied temperature range, as indicated by the slightly different slope in the T^2 plot of ^{77}K (see Fig. 2). This anisotropy may come from an anisotropic susceptibility or an anisotropic hyperfine coupling. As a local probe, the Knight shift data should indicate an intrinsic behavior of the magnetic susceptibility. In contrast, the magnetic susceptibility reported on $Tl_{0.58}Rb_{0.42}Fe_{1.72}Se_2$ shows a T^2 behavior with $H \parallel c$, but not with $H \parallel ab$ [Refs. 38], which may be affected by some unknown contributions. Furthermore, the ratio $^{87}K/^{77}K \approx 0.4$ indicates that the hyperfine field on the interlayer Rb site is comparable to that on the Se site, implying a three-dimensional-like system.

Such a large increase of the Knight shift is unlikely caused by charge excitations. In this compound, the charge excitation scenario will require an increase of the electron density over a factor of three up to the room temperature, which has not been indicated by any ARPES or transport measurements. Spin fluctuations constitute another possible scenario. The temperature behavior of the Knight shift suggest that a finite spin gap (or pseudogap) may exist, so that the spin fluctuations are suppressed at low temperatures and thermally activated at high temperatures. However, the experimental evidence for the physical origin of such a spin gap is lacking. Our evidence of nearly isotropic, three-dimensional Knight shift should constitute important constraints to possible theories.

We note that a magnetic transition at about 500 K has been reported by recent neutron scattering[25] and susceptibility measurements[27] on $(Tl, A)_yFe_{2-x}Se_2$ superconductors. However, the long-range antiferromagnetism has not been seen by NMR so far. It is possible that the sample is phase separated with magnetic regions and nonmagnetic regions at low temperatures. Further NMR measurements, in particular at high temperatures, are needed to address this issue.

VII. SUMMARY

To summarize, our ^{77}Se and ^{87}Rb Knight shifts of $Tl_{0.47}Rb_{0.34}Fe_{1.63}Se_2$ show bulk evidence for singlet superconductivity. However, the Hebel-Slichter coherence peak is not seen in the spin-lattice relaxation rate at a very low in-plane field, which suggests that the system is probably not a conventional s-wave superconductor. In the normal state, evidence for low-energy spin fluctuations is not observed close

to T_c, which raises questions about the correlation between spin fluctuations and the superconductivity. With increasing temperature, however, the Knight shift increases substantially with a T^2 behavior up to room temperature, which is in contrast to most iron pnictides. The spin-lattice relaxation rate also shows a large increase with temperature. The Knight shift and the spin-lattice relaxation may suggest spin excitations with a finite gap (or pseudogap) which are thermally excited at high temperatures.

Our resolved chemical composition of $Tl_{0.47}Rb_{0.34}Fe_{1.63}Se_2$ suggests that the $A_2Fe_4Se_5$ stoichiometric structure is formed in our superconducting compound. Furthermore, the similar NMR data of $Tl_{0.47}Rb_{0.34}Fe_{1.63}Se_2$ and $K_{0.8}Fe_{2-x}Se_2$ indicates the same electronic structure and pairing symmetry in both compounds, which points to a common origin of superconductivity in this structure family of the iron-based superconductors.

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