NMR investigations toward understanding the variety of ground states in iron-based superconductors

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Abstract. We report NMR experiments on heavily electron-doped Fe-based superconductor in comparison with the results on the parent Fe-based compounds. The typical parent Fe-based compound LaFe(As₁₋ₓPₓ)O exhibits the re-emergent antiferromagnetic (AFM) order at x′ ~ 0.6 (AFM2) separated from the parent AFM order at x′ = 0 (AFM1). Systematic ³¹P-NMR study on [Sr₄Sc₂O₆]Fe₂(As₁₋ₓPₓ)₂(SrSc₄2622), which has local lattice parameters of iron-pnictogen (FePₙ) layer similar to the series of LaFe(As₁₋ₓPₓ)O, also revealed that the presence of AFM1 order is universal for most of parent Fe-based compounds. In contrast, the static AFM2 order was absent in this series, however, the dynamical low-energy AFM spin fluctuations are enhanced at around x ~ 0.8, indicating that the onset of the static AFM2 is quite sensitive to the local lattice parameters of FePₙ layer. In order to elucidate the further universality and diversity, we have carried out ⁷⁷Se-NMR measurement on Liₓ(NH₃)₁₋ₓFe₂₋ₓSe₂ (Tc = 44 K) in heavily electron-doped regime. Although the spin fluctuations at low energies does not significantly develops upon cooling, the moderate spin fluctuations were extracted at high temperatures from comparison of the temperature (T) dependences of Knight shift and nuclear relaxation rate (1/T₁T). We discuss the universality and diversity of the relationship between the Tc and the characteristics of the spin fluctuations in the Fe-based compounds from a microscopic point of the NMR measurements.

1. Introduction
A number of researches on iron (Fe)-based superconductors have unraveled a rich variety of antiferromagnetic (AFM), structural, and superconducting phases of various Fe-based compounds[1]. The typical parent compound LaFeAsO shows a stripe antiferromagnetic (AFM) order in association with the structural transition, which is denoted as AFM1 hereafter. The superconductivity (SC) usually appears by doping the electrons/holes, isovalent substitutions, and applying the pressure, however, the phase diagram strongly depends on the material families,
which prevent us to understand the mechanism for increasing $T_c$ in Fe-based compounds. For example, the series of LaFe(A$_{1-x}$P$_x$)$_2$O$_6$ exhibits the complicated phase diagrams including the AFM1 order at $x' = 0.0 - 0.2$, the SC1 at $x' = 0.2 - 0.4$, the re-emergent AFM order (AFM2) at $x' = 0.4 - 0.7$, and the SC2 at $x' = 0.7 - 1.0$[2, 3, 4, 5]. In the series [Sr$_4$Sc$_2$O$_6$]Fe$_2$(A$_{1-x}$P$_x$)$_2$, denoted as SrSc$_{2}$(A$_{1-x}$P$_x$)$_2$ hereafter, it was previously reported to exhibit the AFM order below $T_N = 35$ K at $x = 0$ [6], and the SC at $x = 1$, that is relatively high $T_c \sim 17$ K among the FeP-based compounds[7, 8]. Since this series SrSc$_{2}$(A$_{1-x}$P$_x$)$_2$ possess the local lattice parameters of the FeP$_n$ layer similar to those of the series LaFe(A$_{1-x}$P$_x$)$_2$O, this is very good material to investigate the universality of the SC. AFM1 and AFM2 phases for typical parent Fe-based compounds composed of hole and electron Fermi surfaces (FSs) in similar sizes. In contrast, the higher SC states have been also reported in Fe-based compounds in the heavily-electron doped regimes, such as in monolayer FeSe[9, 10], and intercalated FeSe systems[11, 12]. These electronic configurations are dominated by large electron FSs, which differs from that of typical parent compounds. Toward the universal understanding of the SC mechanism, it is important to challenge to unveil the essential features among the various Fe-based compounds.

In this report, we first review the systematic NMR studies on [Sr$_4$Sc$_2$O$_6$]Fe$_2$(A$_{1-x}$P$_x$)$_2$ and LaFe(A$_{1-x}$P$_x$)$_2$O, which provide an opportunity to discuss the universality of their ground states, the origin of the high $T_c$ state in FeP-based compounds, and the relationship between local lattice parameters and some segregated AFM and SC phases. To unravel the universality/diversity in the Fe-based superconductors in heavily-electron doped states, we show the recent NMR results on iron-selenide superconductor Li$_x$(NH$_3$)$_y$Fe$_{2-x}$Se$_2$. Although the spin fluctuations at low energies is not significant, the moderate spin fluctuations were extracted at high temperatures from the comparison of the temperature ($T$) dependences of Knight shift and nuclear relaxation rate ($1/T_1T$). We discuss the diversity and universality of the relationship between the $T_c$ and the characteristics of the spin fluctuations in Fe-based compounds from the NMR measurements.

2. Experimental

$^{31}$P-NMR measurements were performed on polycrystalline samples of [Sr$_4$Sc$_2$O$_6$]Fe$_2$(A$_{1-x}$P$_x$)$_2$ and LaFe(A$_{1-x}$P$_x$)$_2$O over a wide range of $x$($x'$). The Knight shift ($K$) and the nuclear-spin lattice-relaxation rate $^{31}(1/T_1)$ in the normal state was measured by $^{31}$P-NMR mainly at an external field of $B_0 \sim 11.93$ T. Polycrystalline sample of Li$_x$(NH$_3$)$_y$Fe$_{2-x}$Se$_2$ was prepared through the process that high-quality FeSe crystals and Li metal in the appropriate ratio were filled with liquid NH$_3$, as described in detail elsewhere[13]. The magnetic susceptibility $\chi$ measured by a SQUID magnetometer shows a single sharp SC transition at $T_c = 44$ K. $^{77}$Se-NMR measurement was carried out at $B_0 \sim 11.96$ T. The nuclear spin-lattice relaxation rate ($1/T_1$) was obtained by fitting the recovery curve of the nuclear magnetization.

3. Results and Discussion

First we review the NMR result on the series of [Sr$_4$Sc$_2$O$_6$]Fe$_2$(A$_{1-x}$P$_x$)$_2$ (SrSc$_{2}$(A$_{1-x}$P$_x$)$_2$) [14], as typical parent compounds dominated by the electronic configuration of hole and electron FSs in similar sizes. Figure 1(a) shows the $T$ dependence of Full-width-at-half-maximum (FWHM) at $^{31}$P site as functions of $T$ and $x$, revealing the evolution of the internal field. The FWHM increases significantly below the broken line extrapolated to $T_N = 35$ K at $x = 0$ (See Fig. 1(a)), suggesting that the AFM1 order phase disappears at $x = 0.3 - 0.4$. We found that the AFM1 phase disappears when the pnictogen height from Fe-plane ($h_p$) is $h_p \leq 1.3 - 1.32$ Å, which is insensitive to Fe-Fe bond length ($d_{Fe-Fe}$) for many parent Fe-based compounds, as shown in Fig. 1(c). This plot represents the ground states for some isoivalent-substituted parent compounds with a formal valence of Fe$^{2+}$ as functions of $h_p$ and $d_{Fe-Fe}$[14, 15, 16], which were derived from previous results on [Sr$_4$Sc$_2$O$_6$]Fe$_2$(A$_{1-x}$P)$_2$[6, 7, 14], LaFe(A$_{1-x}$P/Sb)O[2, 4, 5, 17, 18],
Figure 1. (a) FWHM at $^{31}$P site in $[\text{Sr}_4\text{Sc}_2\text{O}_6]\text{Fe}_2(\text{As}_{1-x}\text{P}_x)_2$, revealing the evolution of the internal field as functions of $T$ and $x$. (b) Contour plot of low-energy AFMSFs $1/(T_1T)_{\text{AFM}}$ for $[\text{Sr}_4\text{Sc}_2\text{O}_6]\text{Fe}_2(\text{As}_{1-x}\text{P}_x)_2$. (c) Plot of the ground states of parent and isovalent substituted Fe-pnictides with a formal valence of Fe$^{2+}$ as functions of $h_{pm}$ and $d_{\text{Fe}-\text{Fe}}$, which are derived from previous results on $[\text{Sr}_4\text{Sc}_2\text{O}_6]\text{Fe}_2(\text{As},\text{P})_2[6, 7, 14]$, LaFe(As,P/Sb)O$[2, 4, 5, 17, 18]$, and $[\text{Ca}_4\text{Al}_2\text{O}_6]\text{Fe}_2(\text{As},\text{P})_2[15, 19]$. The solid, open, and cross symbols represent the AFM order, the SC, and non-SC states, respectively. The high $T_c$ region in the literatures$[20, 21]$ appears by doping electrons/holes at around $h_{pm} = 1.36 - 1.38$ Å and $\alpha \sim 109.5^\circ$ as shown by the dashed circle.

and $[\text{Ca}_4\text{Al}_2\text{O}_6]\text{Fe}_2(\text{As},\text{P})_2[15, 19]$. Here, the solid, open, and cross symbols represent the AFM order, the SC, and non-SC states, respectively. On the other hand, the static AFM2 phase reported in the range $0.4 \leq x' \leq 0.7$ for LaFe(As$_{1-x'}$P$_{x'}$)O was not observed in $0.6 \leq x \leq 1$ for SrSc42622(As$_{1-x}$P$_x$) even at a comparable value of $h_{pm}$. It indicates that the onset of the static AFM2 is sensitive to the local lattice parameters such as $d_{\text{Fe}-\text{Fe}}$. Instead, we observed the re-enhancement of dynamical low-energy AFM spin fluctuations in SrSc42622(As$_{1-x}$P$_x$) as follows. To deduce the development of AFM spin fluctuations (AFMSFs) $[4, 16, 22, 23, 24]$, we assume that the observed $1/(T_1T)$ is decomposed as $(1/T_1T) = (1/T_1T)_{\text{AFM}} + (1/T_1T)_0$. Here the first term $(1/T_1T)_{\text{AFM}}$ represents the contribution of AFMSFs with the finite wave vectors $Q$, which is generally described as

$$\left(\frac{1}{T_1T}\right)_{\text{AFM}} \propto \lim_{\omega \rightarrow \omega_0 \sim 0} |A_{\text{hf}}(Q)|^2 \frac{\chi''(Q, \omega)}{\omega},$$

where $A_{\text{hf}}(Q)$ is the hyperfine-coupling constant at $q \sim Q$, $\chi''(Q, \omega)$ is the dynamical spin susceptibility at $q \sim Q$ and energy $\omega$, and $\omega_0$ is the NMR frequency approximated as $\omega_0 \sim 0$. The second term $(1/T_1T)_0$ represents the $q$-independent one in proportion to $N(E_F)\omega^2$, which can be evaluated by $K_0^2(T)(=(K - K_{\text{chem}})^2)$. Here, $N(E_F)$ is the density of states at Fermi level, and $K_{\text{chem}}$ is the chemical shift$[14]$. As a result, the contour plot of $(1/T_1T)_{\text{AFM}}$ is shown as functions
of $x$ and $T$ in Fig. 1(b)[14]. At $x < 0.4$, the AFMSFs evolve upon cooling toward the AFM1 order phase. Although these are suppressed once at $x = 0.3$ - 0.4, the AFMSFs are enhanced again significantly in the region of $x = 0.6$ - 1.0, which are not continuous from the AFM1 phase. The AFMSFs at $x = 0.6$ - 1.0 can be naturally attributed to the instability of the AFM2 order phase, because the local lattice parameters of their FePn layers are similar to that of the AFM2 phase for $x' = 0.4$ - 0.7 of LaFe(As$_{1-x}$P$_x$)O[2, 3, 4, 5]. The band calculation suggested that the AFM2 phase is derived from the accidentally good nesting of the hole and electron FSs in the local lattice parameters of LaFe(As$_{0.4}$P$_{0.6}$)O[25], and thus it is considered that these well-nested FSs in the parameter regions of LaFe(As$_{0.4}$P$_{0.6}$)O may be missed by slightly longer $d_{\text{Fe-Fe}}$ in SrSc$_4$2622(As$_{1-x}$P$_x$)[14]. Despite the absence of the static AFM2 order, the possible instability of the AFM2 phase induces the dynamical low-energy AFM spin fluctuations, which plays a significant role in enhancing the $T_c$ to 17 K at $x = 0.8$ - 1.0[7, 8, 14], resulting in the highest $T_c$ among FeP-based superconductors.

![Figure 2.](image)

Figure 2. (a) $T$ dependence of $1/T_1 T$ and $K^2_s$, and (b) a plot of $(1/T_1 T)^{0.5}$ vs. $K$ in the normal state of Li$_x$(NH$_3$)$_y$Fe$_{2-\delta}$Se$_2$. The slight deviation from the linear dependence known as Korringa relation is shown by the hatched region in (b), corresponding to the component of spin fluctuations in $1/T_1 T (= (1/T_1 T)_{\text{AFM}})$.

Next, we address the $^{77}$Se-NMR result on the heavily electron-doped superconductor Li$_x$(NH$_3$)$_y$Fe$_{2-\delta}$Se$_2$, denoted as [Li(NH$_3$)]FeSe hereafter. This compound is expected to possess the electronic configuration dominated by larger electron FSs in contrast to that of typical parent compounds. X-ray diffraction pattern indicates that the sample is composed by almost single phase of [Li(NH$_3$)]FeSe. A single sharp SC transition at $T_c = 44$ K is confirmed in the magnetic susceptibility measurement. We observed very sharp $^{77}$Se-NMR spectra with FWHM is $\Delta K \sim 300$ ppm[26]. These experimental facts guarantee the high-quality crystal dominated by homogeneous electronic states of high $T_c$ phase over the sample. As shown in Fig. 2(a), the $1/T_1 T$ in the normal state exhibits a monotonous decrease upon cooling, which resembles that of the Knight shift $(K^2_s(T) = (K - K_{\text{chem}})^2)[26]$. The results are similar to the previous reports for many intercalated FeSe-based compounds in the electron-doped regimes[27, 28, 29, 30]. We note that this feature differs from the typical cases of the parent Fe-based superconductors, as seen in $x = 0.8$ of SrSc$_4$2622(As$_{1-x}$P$_x$), for example, where the spin fluctuation at low energies are strongly
enhanced toward low temperatures. Here we applied the analyses to extract the component of the AFM spin fluctuations as discussed previously. If the compound is the normal metal without any spin fluctuations, the linear dependence (Korringa relation) should be observed in the plot of $(1/T_1T)^{0.5}$ vs. $K(=K_s+K_{\text{chem}})$, as indicated by the solid line of Fig. 2(b). The slight deviation from this solid line suggests the presence of the non-negligible component of spin fluctuations at finite wave-vector $Q$ in $1/T_1T$, i.e. $(1/T_1T)_{\text{AFM}}$, as displayed by the hatched area in the figure. The subtracted $(1/T_1T)_{\text{AFM}}$ from this analysis is shown in Fig. 2(a), which represents that the spin fluctuations develop from high temperatures and decrease toward lower temperatures through the maximum around 200 K. The result suggests that there is a spin-gap at low energies in spin excitation spectra of dynamical spin susceptibility $\chi''(Q, \omega)$. We point out that this behavior can be seen in the many other related intercalated compounds in electron-doped regimes, such as Li$_x$(C$_2$H$_5$N)$_2$Fe$_2$Se$_2$, K$_x$Fe$_2$Se$_2$, [Tl$_{0.47}$Rb$_{0.53}$]Fe$_{1.63}$Se$_2$[27, 28, 29, 30], if we apply the same analysis. Such behavior is in contrast with the case of bulk FeSe($T_c \sim 8$ K), in which the strong development of spin fluctuations is observed toward low temperatures[31]. The band calculation for the related compounds suggested that the FS topology is composed by the tiny hole FS (or incipient hole-like band) and large electron FSs, dominated mainly by the $d_{xy}$ orbitals[32, 33]. This configuration is far from those of the parent compounds with well-nested or moderately-nested FSs composed by the hole and electron FSs in similar size. Recently, it has been reported that in FeSe$_{1-x}$S$_x$ more moderate spin fluctuation at low energies in the $C_4$ symmetry with higher $T_c$ values than in the $C_2$ symmetry with lower $T_c$ values, although the development of low energy spin fluctuations are important for enhancing the $T_c$ values in both cases[34]. We suggest that the low energy part of the spin fluctuations is generally important for spin-mediated Cooper pairing mechanism, but the finite energy part of the spin fluctuations also contribute to the Cooper pairing in the Fe-based compounds. Further spectroscopic experiments are necessary for general understanding of the universality and diversity for the high-$T_c$ SC states of various Fe-based compounds.

4. Summary

We have investigated the universality and diversity for understanding the complicated ground states and the high-$T_c$ SC states in various Fe-based compounds from a microscopic point. The systematic NMR studies on [Sr$_4$Sc$_2$O$_6$]Fe$_2$(As$_{1-x}$P$_x$)$_2$ revealed that the parent AFM1 phase at $x = 0$ disappears in the range $x = 0.3 - 0.4$. By contrast, the static AFM2 order reported in LaFe(As$_{0.4}$P$_{0.6}$)O does not appear at approximately $x \sim 0.8$ of [Sr$_4$Sc$_2$O$_6$]Fe$_2$(As$_{1-x}$P$_x$)$_2$ although the local lattice parameters of the FePn layer are close to each other. Despite the absence of the static AFM2 phase, the dynamical low-energy AFMSFs develop significantly at approximately $x \sim 0.8$. We remark that such re-enhancement of AFMSFs derived from the instability of the AFM2 order phase play a significant role in enhancing the $T_c \sim 17$ K at $x = 0.8 \sim 1$ of these compounds. In contrast, as for Li$_x$(NH$_3$)$_y$Fe$_2$Se$_2$ in the heavily-electron doped regime, we found the moderate spin fluctuations with gap at low energies, which may be common feature for the heavily-electron doped FeSe-based SC compounds. To reveal the general understanding the high-$T_c$ SC states, further spectroscopic experiments are necessary for various Fe-based compounds over wide doping regions.

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