Antiferromagnetic Order and Superconductivity in Sr$_4$(Mg$_{0.5-x}$Ti$_{0.5+x}$)$_2$O$_6$Fe$_2$As$_2$
with Electron Doping: $^{75}$As-NMR Study

Keisuke YAMAMOTO$^1$, Hidekazu MUKUDA$^{1,4*}$, Hiroaki KINOUCHI$^1$, Mitsuharu YASHIMA$^{1,4}$, Yoshiro KITAOKA$^1$, Mamoru Yogi$^3$, Shinya SATO$^2$, Hiraoku OGINO$^{2,4}$, and Jun-ichi SHIMOYAMA$^{2,4}$

$^1$Graduate School of Engineering Science, Osaka University, Toyonaka, Osaka 560-8531
$^2$Department of Applied Chemistry, The University of Tokyo, Hongo, Bunkyo, Tokyo 113-8656
$^3$Department of Physics and Earth Sciences, Faculty of Science, University of the Ryukyus, Okinawa 903-0213
$^4$JST, TRIP (Transformative Research-Project on Iron-Pnictides), Chiyoda, Tokyo 102-0075

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We report an $^{75}$As-NMR study on iron (Fe)-based superconductors with thick perovskite-type block-layered Sr$_4$(Mg$_{0.5-x}$Ti$_{0.5+x}$)$_2$O$_6$Fe$_2$As$_2$ with $x=0$ and 0.2. We have found that antiferromagnetic (AFM) order sets in at $T_c=36$ K when $x=0.2$. These results reveal that the Fe-pnictides with thick perovskite-type block layers also undergo an evolution from the AFM order to the SC phase by doping electron carriers into FeAs layers through the chemical substitution of Ti$^{4+}$ ions for Mg$^{2+}$ ions, analogous to the $P_n$-substitution in LaFeAsO compound. The reason why the $T_c=36$ K at $x=0.2$ being higher than the optimally electron-doped LaFeAsO with $T_c=27$ K relates to the fact that the local tetrahedron structure of FeAs$_4$ is optimized for the onset of SC.

KEYWORDS: Sr$_4$(Mg$_{0.5-x}$Ti)$_2$O$_6$Fe$_2$As$_2$, perovskite blocks, superconductivity, antiferromagnetism, NMR

Iron-based high-$T_c$ superconductors comprise a two-dimensional layered structure of iron (Fe)-pnictogen (Pn) planes, which are separated by blocking layers, such as LnO (Ln=rare earth), alkaline earth metals and alkaline metals, and so on. Relatively high superconducting transition has been reported in Fe-pnictides with thick perovskite-type block layers, for example, $T_c$ is $\sim 47$ K for Ca$_4$(Mg$_{0.25}$Ti$_{0.75}$)$_2$O$_6$Fe$_2$As$_2$ and $\sim 37$ K for Sr$_2$V$_2$O$_6$Fe$_2$As$_2$ ($V$-$4202$), in which an interlayer distance between FePn layers ($L$) is longer than 13 Å. However, a ground state of undoped (FeAs)$^-$ layer and a relation with the onset of SC in the Fe-pnictides with thick perovskite blocks has not been identified sufficiently, although it is well known that the superconductivity (SC) in Fe-pnictide compounds emerges in close proximity to antiferromagnetism (AFM). In Sr$_4$(Mg$_{0.5-x}$Ti$_{0.5+x}$)$_2$O$_6$Fe$_2$As$_2$ (denoted as Mg$_{0.5-x}$Ti$_{0.5+x}$42622 hereafter), it has been reported that $T_c$ increases from 0 K to 36 K by the substitution of Ti for Mg, and up to 43 K by the application of high pressure. In this compound with $x=0$, the Fe$^{2+}$ state is formally expected as well as other parent Fe-pnictide compounds. Therefore, systematic investigations of these compounds will provide us with further insight into the intimate relationship between SC and AFM order inherent in FeAs layers with a highly two-dimensional electronic structure.

In this Letter, we report $^{75}$As-nuclear magnetic resonance (NMR) study of Mg$_{0.5-x}$Ti$_{0.5+x}$42622 with $x=0$ and 0.2, which unravels that an AFM order sets in at $x=0$ and an SC state emerges at $x=0.2$ by doping electron carriers into the FeAs layers through the chemical substitution of Ti for Mg. We remark that the ground state of (FeAs)$^-$ layer of undoped Mg$_{0.5-x}$Ti$_{0.5+x}$42622 resembles those of 1111 and 122 systems.

Polycrystalline samples of Mg$_{0.5-x}$Ti$_{0.5+x}$42622 were synthesized in quartz ampules at ambient pressure as described elsewhere. Powder x-ray diffraction measurement indicates that the samples are dominated by an intrinsic phase, whereas the $x=0.2$ sample contains small amounts of impurities such as SrFe$_2$As$_2$ and Sr$_2$TiO$_4$. However, the $^{75}$As-NMR signal inherent in Mg$_{0.5-x}$Ti$_{0.5+x}$42622 at $x=0.2$ is discriminated from that of SrFe$_2$As$_2$. Both the samples with $x=0.0$ and 0.2 are the same lattice parameters: $a$- and $c$-axis length of $a=3.94$ Å and $c=15.95$ Å, a height of pnictogen from Fe-plane $h_{\text{Fe-As}}$ $\sim 1.4$ Å, and a $Pn$-Fe-$Pn$ bond angle, $\alpha \sim 109.5^\circ$. These parameters are comparable to the optimum lattice parameters to reach a highest $T_c$ in various series of Fe based compounds, as suggested in the literature. The $T_c$s of Mg$_{0.5-x}$Ti$_{0.5+x}$42622 determined by the resistivity and susceptibility measurements are shown in Fig. 1(a). Here, $T_{\text{onset}}$ and $T_{\text{zero}}$ are the respective temperatures for an onset and zero resistance of SC in resistivity measurement, and $T_{\text{onset}}$ is a temperature for an onset of SC diamagnetism in susceptibility measurement. Note that $T_{\text{zero}}$ $\sim 5$ K when $x=0$ is not associated with a bulk SC, but a filamentary-induced one, since the SC diamagnetism does not point to a bulk nature. The substitution of Ti for Mg brings about a bulk SC transition at $T_{\text{onset}}$ $\sim 36$ K ($T_{\text{onset}}$ $\sim 22$ K), causing the distinct appearance of SC diamagnetism. $^{75}$As-NMR measurements have been performed for coarse powder samples of Mg$_{0.5-x}$Ti$_{0.5+x}$42622 ($x=0$ and 0.2).

Figure 1(c) shows the temperature($T$)-dependence of $^{75}$As-NMR ($I=3/2$) spectra for the powder sample of Mg$_{0.5}$Ti$_{0.5}$42622 with $x=0$. The spectrum at 100 K is a typical powder pattern affected by the nuclear quadrupole interaction in a paramagnetic state. Here, $^{75}I_{Q}$ was estimated to be $\sim 11.7$ MHz, which is slightly larger than in LaFeAsO. As $T$ lowers, the spectrum
order below $LaFeAsO$, which exhibits the commensurate stripe AFM spectral shape resembles that of the parent compound $density being large below 100 K$. Note that the broad NMR overlaps with a broad spectrum with its spectral intensity being comparable to $LaFeAsO$ and $BaFe$ $57$ $As$ $site$ are assumed in these values at the As site are assumed in these.

| $M_{AFM}$ | $T_N$ $[K]$ |
|----------|-------------|
| $Mg_{0.5}Ti_{0.5}$ $-42622$ $x=0$ | $50 \sim 100$ |
| $LaFeAsO$ | $5.3^{15}$ $\pm 1.6^{12}$ $0.8 \mu_B^{16}$ |
| $BaFe_2As_2$ | $5.4^{17}$ $\pm 1.4^{20}$ $0.87 \mu_B$ |
| $SrFe_2As_2$ | $8.9^{28}$ $\pm 2.2^{28}$ $1.03 \mu_B$ |
| $CaFe_2As_2$ | $10^{29}$ $\pm 2.6^{21}$ $0.8 \mu_B$ |
| $Sc_{-42622}$ | $1.65^{6}$ |

Table 1. Internal field at respective $^{57}$Fe and $^{75}$As sites from $^{57}$Fe Mössbauer and $^{75}$As-NMR studies for the undoped Fe-pnictides at low temperatures, together with the ordered moment $(M_{AFM})$ and $T_N$. $^{14}$

Fig. 1. (Color online) (a) Phase diagram of $Mg_{0.5-x}Ti_{0.5+x}$ $-42622$. Respective $T_{c}^{\nu_{Q}}$ and $T_{c}^{\nu_{Q}}$ are the onset of resistivity drop and SC diamagnetism in susceptibility, and $T_{c}^{\nu_{Q}}$ presents zero resistivity[Sato et al.]. The Néel temperature $(T_N)$ for $x=0$ is $50 \sim 100$ K, as revealed in this study(see text). (b) Phase diagram of $LaFeAsO_{1-y}$. $^{12}$ (c) and (d) are $T$-dependence of $^{75}$As-NMR spectra for $x=0$ and $LaFeAsO$ $(T_N=140$ K), respectively. The solid lines are the simulated spectra when the internal field $H_{int}^{[c]}$ and their $\nu_{Q}$ values at the As site are assumed in these compounds. (e) $T$ dependence of volume fraction of AFM domain evaluated from the fractional intensity of the broad spectra. Here, we assume that the difference of spin-spin relaxation time $T_2$ in two phases is neglected.

the broad spectra of AFM domains are tentatively reproduced by assuming $^{75}H_{int}^{[c]} \sim \pm 1.3$ T and $^{75}H_{Q}^{[c]}=11.7$ MHz. On the other hand, it should be noted that the $^{75}$As NMR spectrum of $LaFeAsO$ is well reproduced by assuming an internal field $^{75}H_{int}^{[c]}=\pm 1.6$ T and $^{75}H_{Q}^{[c]}=8.8$ MHz, revealing that no phase separation takes place in $LaFeAsO$, as indicated by the solid line in Fig. 1(d). $^{12}$

Table I presents a list of the internal fields at $^{57}$Fe and $^{75}$As sites, $T_N$ and ordered moments $M_{AFM}$ derived from the experiments of $^{57}$Fe-Mössbauer and $^{75}$As-NMR on mother compounds of FeAs based superconductors. The $^{57}H_{int}$ and $^{75}H_{int}$ are induced by $M_{AFM}$ through the respective hyperfine-coupling constants $^{57}A_{hf}$ and $^{75}B_{hf}$. In particular, the origin of $^{75}B_{hf}$ is attributed to an off-diagonal pseudodipole field induced by stripe-type AFM ordered moments lying on the ab-plane at the Fe site. $^{13}$

Since $^{75}H_{int}^{[c]} \sim \pm 1.3$ T in $Mg_{0.5}Ti_{0.5}$ $-42622$ is comparable with those values in $LaFeAsO$ and $BaFe_2As_2$, it is likely that its AFM ordered state is similar to those in $LaFeAsO$ and $BaFe_2As_2$. It differs from the static magnetic order of tiny moment from the FeAs layer, which was reported in $Sr_3Sc_2O_6Fe_2As_2$ (denoted as $Sc_{-42622}$). $^{6}$ Hence, we remark that a commensurate stripe AFM order being comparable to $LaFeAsO$ and $BaFe_2As_2$ is realized in the ground state of $Mg_{0.5}Ti_{0.5}$ $-42622$, even though Mössbauer and neutron scattering experiments in this compound are not yet reported.

In the powder sample of $Mg_{0.3}Ti_{0.7}$ $-42622$ with $x=0.2$, the broad spectrum arising from the AFM domains was not observed, suggesting that doping electron carriers expel the AFM domains. This fact suggests that Ti ions are in a tetravalent state of Ti$^{4+}$ with $3d^0$ in blocking layers, which contrasts with the trivalent state of V$^{3+}$ ions in $Sr_4V_2O_6Fe_2As_2$ which are magnetic. $^{7,22,23}$ Thus, the substitution of nonmagnetic Ti$^{4+}$ ions for Mg$^{2+}$ ions results in an increase in electron density and leads to the collapse of the AFM order. This is also corroborated by the fact that $^{75}\nu_{Q}$ $\sim 12.6$ MHz at $x=0.2$ is slightly larger than that at $x=0$, which also resembles the doping dependence of $^{75}\nu_{Q}$ in $LaFeAsO$ system. $^{11,24}$

The nuclear spin-lattice relaxation rate $1/T_1$ was measured at the central peak in the $^{75}$As-NMR spectra (see Fig. 1(c)). Here, $1/T_1$ was determined from the recovery curve of $^{75}$As nuclear magnetization following the theo-
Accordingly, it would be expected that short component $T_1$ can be determined by a single theoretical curve at high temperatures ($T > 120$ K), but not below 100 K. The fraction of the long $T_1$ component becomes large at low temperatures in accordance with the emergence of broad spectra below 100 K (see Fig. 1(c)).

Theoretical function for $I = 3/2$: $m(t) = \frac{M_0 - M(t)}{M_0} = 0.1 \exp(-t/T_1) + 0.9 \exp(-6t/T_1)$, where $M_0$ and $M(t)$ are the respective nuclear magnetizations for the thermal equilibrium condition and at a time $t$ after the saturation pulse. As shown in Fig. 2, the $m(t)$ at $x=0$ is fitted by a theoretical curve with a single component of $T_1$ at temperatures higher than 120 K, but lower than ~100 K. Therefore, a long component $T_{1L}$ and a short component $T_{1S}$ are tentatively deduced by assuming an expression given by $m(t) = A_{MS}S(t) + A_{ML}l(t)$. Here, $A_S$ and $A_L$ with $A_S + A_L = 1$ represent the respective volume fractions of domains with $T_{1S}$ and $T_{1L}$. Note that $A_L$ becomes larger upon cooling below ~100 K in association with the emergence of AFM domains. Accordingly, it would be expected that $T_{1L}$ and $T_{1S}$ are associated with the AFM domains and the paramagnetic domains, respectively, reflecting the phase separation in Mg$_{0.5}$Ti$_{0.5}$-42622 with $x=0$.

The $T$ dependences of $1/T_{1S}T$ and $1/T_{1L}T$ components are plotted in Fig. 3(a). The $1/T_{1S}T$ increases upon cooling below 100 K, but it decreases rapidly with a peak at 50 K, accompanied by a reduction in the volume fraction of the paramagnetic domains. On the other hand, $1/T_{1L}T$ decreases gradually upon cooling below ~100 K, accompanied by an increase in the volume fraction of the AFM domains. These results are consistently interpreted by the fact that AFM ordered domains develop progressively below ~100 K and their fraction exceeds the fraction of paramagnetic domains below 50 K, as presented in Fig. 1(c). The peak in $1/T_{1S}T$ at $T_N = 50$ K may suggest that some paramagnetic domains undergo an AFM order with a possible distribution of Néel temperature ($T_N$) in between 50 K and 100 K, depending on a possible spatial inhomogeneity of the local concentration of Mg/Ti atoms. It contrasts with the case of stoichiometric parent compounds LaFeAsO and BaFe$_2$As$_2$, as compared in Fig. 1(e), where the Fe$^{2+}$ states of LaFeAsO and BaFe$_2$As$_2$ are homogeneously realized on the (FeAs)$^-$ layer without any phase separation after the structural transition to the orthorhombic phase. We also note that $T_{NS}$ of $x=0$ and Sc-42622 in the previous report$^6$ are significantly lower than $T_N ≈ 140$ K for other parent compounds LaFeAsO and BaFe$_2$As$_2$, which may relate with the large interlayer distance between the FeAs layers.

![Fig. 2. (Color online) Recovery curves of $^{75}$As nuclear magnetization in $x=0$. The $1/T_1$ can be determined by a single theoretical curve at high temperatures ($T > 120$ K), but not below 100 K. The fraction of the long $T_1$ component becomes large at low temperatures in accordance with the emergence of broad spectra below 100 K (see Fig. 1(c)).](image)

![Fig. 3. (Color online) $T$ dependence of $^{75}$As-NMR-$(1/T_1T)$ for (a) Mg$_{0.5-x}$Ti$_{0.5+x}$-42622 ($x=0$ and 0.2) and Al-42622 ($T_c = 27$ K)$^{13}$, and (b) LaFeAsO (non-doped(ND) and optimally-doped(OPT) with $T_c = 28$ K$^{11}$ and heavily overdoped(OVD) with $T_c = 5$ K$^{26}$).](image)

Next, we deal with the $T_1$ results for Mg$_{0.3}$Ti$_{0.7}$-42622 with $x=0.2$, which are shown in Fig. 3(a). The onset of SC at $T_c = 36$ K is also corroborated by a distinct reduction in $1/T_1$. However, since its broad SC transition prevents us from deducing SC characteristics precisely, we focus only on normal-state properties of this SC compound. We remark that the $T$ dependences of $1/T_1T$ for $x=0$ and 0.2 resemble those in non-doped (ND) and optimally electron-doped LaFeAsO-based compounds$^{11,12,25,26}$, as compared in Figs. 3(a) and 3(b). The decrease in $1/T_1T$ upon cooling for $x=0.2$ is mostly attributed to the band structure effect$^{27}$ suggesting the suppression of AFM spin fluctuations in low energies. Since the lattice parameters do not change so much in the series of Mg$_{0.5-x}$Ti$_{0.5+x}$-42622, $^4$ probably due to the strong covalent bonding in the perovskite blocks, the SC in this compound takes place by increasing Ti$^{4+}$ content through the substitution of Ti for Mg, namely, by doping electron carriers. This contrasts with the optimally electron-doped LaFeAsO with $T_c = 28$ K in which either the F-substitution or the O-deficiency changes both the electron-doping level and the lattice parameters. In another context, note that the normal-state property for $x=0$ differs from the case of the related 42622 compound Ca$_4$Al$_2$O$_6$Fe$_2$As$_2$ (denoted as Al-42622)$^{5}$ as shown in Fig. 3(a). The latter compound was characterized by the development of AFM spin fluctuations at low energies$^{28}$ in association with the nesting between the hole and electron Fermi surfaces (FSs) being quite better$^{29}$ owing to the lattice parameters characterized by a very small $a = 3.71$ Å, a narrow $\alpha \sim 102.1°$, etc.
and a high $h_p \approx 1.50 \, \text{Å}$ \cite{5}. By contrast, MgTi-42622 with a nearly ideal FeAs$_4$-tetrahedron possesses slightly worse FS nesting properties than that of Al-42622 \cite{30}, but $T_c$ is higher than in Al-42622. This result suggests that AFM spin fluctuations are not only a unique parameter for enhancing $T_c$. We also note that a highest $T_c=36 \, \text{K}$ in the series of Mg$_{0.5-x}$Ti$_{0.5+x}$-42622 with the optimum electron doping level at $x=0.2$ is higher than $T_c=28 \, \text{K}$ in optimally doped LaFeAsO, which should be ascribed to the fact that the local tetrahedron structure of FeAs$_4$ is optimized. Within a spin-fluctuation mediated pairing theory on a five-orbital model, Usui et al. have theoretically claimed that not only the nesting of the hole and electron FSs but also the multiplicity of FSs are important to realize high-$T_c$ SC in Fe based compounds \cite{30}. According to this scenario, the higher $T_c$ in Mg$_{0.5-x}$Ti$_{0.5+x}$-42622 can be attributed to the larger multiplicity of FSs in Mg$_{0.5-x}$Ti$_{0.5+x}$-42622 than in Al-42622, whereas the nesting property of FSs are not perfect. Further systematic studies on the relationship between the local structure and electronic state in the related 42622 compounds are desired.

In summary, the $^{75}$As-NMR studies on Sr$_2$(Mg$_{0.5-x}$Ti$_{0.5+x}$)$_2$Fe$_2$As$_2$O$_6$ have unraveled that the AFM stripe order takes place for $x=0$ and the SC sets in at $T_c=36 \, \text{K}$ for $x=0.2$. The increase of Ti substitution from $x=0$ to 0.2 brings about the onset of SC with $T_c=36 \, \text{K}$ as a result of doping electron carriers into FeAs layers, which resembles the variation of the electronic states in the electron-doped LaFeAsO compounds through either F-substitution or O-deficiency. The phase diagram of the Fe-pnictides with thick perovskite-type blocking layers resembles those in other Fe-based superconductors which emerge in close proximity to the AFM phase by doping either electron or hole carriers. As for the SC state, the comparison with the related 42622 compound Ca$_4$Al$_2$O$_6$Fe$_2$As$_2$ with $T_c=27 \, \text{K}$ suggests that antiferromagnetic spin fluctuations are not a unique factor for enhancing $T_c$. The reason why the $T_c=36 \, \text{K}$ at $x=0.2$ is higher than the optimally electron-doped LaFeAsO with $T_c=28 \, \text{K}$ may relate to the fact that the local tetrahedron structure of FeAs$_4$ is optimized for the onset of SC.

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