Comparative $\mu^+\text{SR}$ study of the zigzag chain compounds NaMn$_2$O$_4$ & LiMn$_2$O$_4$

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Abstract. The magnetic nature of the CaFe$_2$O$_4$-type NaMn$_2$O$_4$ and Li$_{0.92}$Mn$_2$O$_4$, in which Mn ions form a zigzag chain along the b-axis, have been investigated by muon-spin rotation and relaxation ($\mu^+\text{SR}$) and susceptibility ($\chi$) measurements in the temperature range between 1.9 and 250 K using polycrystalline samples. Weak transverse-field $\mu^+\text{SR}$ measurements revealed the existence of a bulk antiferromagnetic (AF) transition for NMO at $T_N = 39$ K and for Li$_{0.92}$Mn$_2$O$_4$ at $T_N = 44$ K, although the $\chi(T)$ curve did not exhibit a clear anomaly around $T_N$ for either compound. Below $T_N$, however, the zero-field $\mu^+\text{SR}$ spectra in both samples did not show the oscillatory signal characteristic of long-range magnetic order. Instead, a slowly relaxing tail is observed. Comparisons with the calculated internal magnetic field for the AF ordered phase suggests that the AF spin structure is disordered and/or rapidly fluctuating. In the paramagnetic region above 100 K, a nuclear magnetic field in Li$_{0.92}$Mn$_2$O$_4$ was found to be dynamic, whereas that in NaMn$_2$O$_4$ was almost static. This implies the diffusive motion of Li$^+$ ions in Li$_{0.92}$Mn$_2$O$_4$ even below 250 K.

1. Introduction
The nature of the ground state of the quasi-1D (Q1D) spin system, having characteristic 1D zigzag chains in the structure, is attracting considerable theoretical and experimental interests [1, 2]. In these systems, there exists a competition between the first nearest neighbor (FNN) and the second nearest neighbor (SNN) interactions. Furthermore, the interaction between the zigzag chains, i.e., the inter-chain interaction, plays an essential role in the formation of magnetic order/disorder in the real systems, since a zigzag chain is never isolated. The spin density of the zigzag chain is the yet other significant factor that determines the ground state of the compounds.

We have initiated experimental studies of zigzag chain compounds to understand how the competition among FNN, SNN, and inter-chain interactions affects the magnetic ground state by means of positive muon-spin rotation/relaxation ($\mu^+\text{SR}$). Recently, two new zigzag chain
Figure 1. Crystal structure of NMO. The Mn$_2$O$_4$ double-chains, i.e., zigzag chains are formed by a network of edge-sharing MnO$_6$ octahedra align along the b-axis so as to make an irregular hexagonal 1D channel. The Na (or Li) ions are located at the center of the 1D channel.

Compounds $A$Mn$_2$O$_4$ ($A =$ Na and Li) have been synthesized using a high-pressure technique. The valence state of the Mn ions is naturally expected to be +3.5, although both compounds are insulating even at ambient $T$. Their crystal structure is described as a CaFe$_2$O$_4$(CFO)-type orthorhombic system with space group $Pnma$ (see Fig. 1). In NaMn$_2$O$_4$ (NMO) a cooperative Jahn- Teller distortion suggests charge ordering of the Mn$^{3+}$ and Mn$^{4+}$ ions, while for Li$_{0.92}$Mn$_2$O$_4$ (LMO) there is no indication of charge-order.

2. Experimental

A polycrystalline sample of NMO was prepared by a solid-state reaction technique under a pressure of 6 GPa using Na$_2$O$_2$ and Mn$_2$O$_3$ powders as starting materials [3]. A mixture of the two powders was packed in an Au capsule in an Ar-filled glove-box, then heated at 1300°C for 1 hour under $P$=6 GPa, and finally quenched to ambient $T$. For the synthesis of the CFO-type LMO, the spinel-type LMO powder was, at first, prepared by a conventional solid-state reaction technique at ambient pressure using Li$_2$CO$_3$ and Mn$_2$O$_3$ powders as starting materials [4]. A mixture of the two powders was heated at 850°C. Then, the obtained spinel-type LMO powder was packed in an Au capsule, heated at 1300°C for 1 hour under $P$=6 GPa, and finally quenched to ambient $T$. A powder x-ray diffraction (XRD) analysis showed that the NMO sample was almost single phase with the CFO-type orthorhombic system of space group $Pnma$ at ambient $T$, while the LMO sample consisted of the CaFe$_2$O$_4$-type orthorhombic phase and Li$_2$MnO$_3$, as in the previous report. The volume fraction of the Li$_2$MnO$_3$ impurity phase was estimated to range around 10%. DC susceptibility ($\chi$) measurements show the presence of an AF transition at $T_N = 15$ K for NMO, but the absence of a clear AF transition for LMO.

Seven polycrystalline discs with 6 mm diameter and 5 mm thickness were set on the muon-veto sample holder. Conventional time-differential $\mu^+\mathrm{SR}$ spectra were collected at the M20 surface muon channel at TRIUMF, Vancouver, Canada, using an experimental setup and techniques described elsewhere [5].
3. Results

As we reported previously [6], the weak transverse field (wTF-) measurements were carried out with applying field of 50 Oe below 80 K. As decreasing $T$, the wTF asymmetries, which correspond to the volume fraction $V_F$ of paramagnetic (PM) phases in a sample, showed a clear drop at 39 K, indicating occurrence of a magnetic transition at $T_N = 39$ K. The discrepancy of $T_{N_S}$ between that obtained by susceptibility measurement and wTF-$\mu$SR spectra is probably due to the difference of time windows between these methods. In order to investigate the magnetic properties below $T_N$, we measured the $\mu^+$SR spectrum with zero external field (ZF) down to 1.9 K. However, the ZF-spectrum exhibits no oscillation but a fast relaxation in an early time domain even at 1.9 K. Moreover, since the relaxation rate is too fast for $\mu^+$SR, we do not observe the full asymmetry ($\sim 0.24$) at $T \leq 30$ K. This implies the existence of a large fluctuating internal magnetic field below $T_N$. The ZF-spectrum is phenomenologically fitted by a combination of three exponential relaxation functions:

$$A_0 P_{ZF}(t) = A_1 \exp(-\lambda_1 t) + A_2 \exp(-\lambda_2 t) + A_3 \exp(-\lambda_3 t),$$

where $A_i$ ($i=1, 2,$ and 3) is the the asymmetries of the three components of the ZF-$\mu^+$SR signal, and $\lambda_i$ is their relaxation rate. In addition, in order to obtain reasonable values for the three signals even under the absence of the full asymmetry at $t = 0$, the total asymmetry is fixed as 0.24, i.e., $\Sigma A_i = A_0 = 0.24$.

Figure 2 shows the $T$ dependences of (a) the normalized asymmetries ($N_{A_i} = A_i/A_0$, with $i=1, 2,$ and 3), (b) $\lambda_1$, and (c) $\lambda_2$ and $\lambda_3$ for NMO. As $T$ decreases from $T_N$, $N_{A_i}$ increases rapidly down to $\sim 30$ K, and then levels off to a constant value ($\sim 0.7$) with further lowering $T$. In contrast to $N_{A_1}$, both $N_{A_2}$ and $N_{A_3}$ decrease rapidly with decreasing $T$ below $T_N$, and then they are almost $T$-independent below $\sim 30$ K. The $\lambda_1(T)$ curve exhibits roughly a similar $T$ dependence to the $N_{A_1}(T)$ curve, while both the $\lambda_2(T)$ and $\lambda_3(T)$ curves are less $T$-dependent even below the vicinity of $T_N$. Considering the magnitude of $N_{A_1}(\sim 2/3)$ at low $T$ and a very small magnitude of $\lambda_2$ and $\lambda_3$ compared with $\lambda_1$, both $A_2$ and $A_3$ signals are most likely to be the “$1/3$ tail” signal.

In order to further elucidate the magnetic behavior around $T_N$, we also carried out ZF-$\mu^+$SR measurements in the $T$ range between 35 and 100 K. As a result, the relaxation rate was found to increase rapidly with decreasing $T$ from 100 K. In fact, it is difficult to fit the spectrum using a simple exponential relaxation function $[\exp(-\lambda t)]$ in the whole $T$ range between 35 and 100 K, but the ZF-$\mu^+$SR spectrum is well fitted by a power exponentially relaxing signal:

$$A_0 P_{ZF}(t) = A_{ZF} \exp(-\lambda_{ZF} t)^\beta),$$

Note that “a power exponentially relaxing signal” has been observed for many dense-moment disordered magnetic systems [7, 8] in a paramagnetic state. The fit results are shown in Fig. 3. As $T$ decreases from 100 K, $\beta \sim 1.4$ [between Gaussian relaxation ($\beta = 2$) and simple exponential ($\beta = 1$)] down to 60 K. Then, $\beta$ decreases rapidly as $T$ is lowered further, dropping below $\beta=0.5$ (root exponential for a dilute disordered magnet) [9]. Ultimately, $\beta$ approaches a value of 1/3, indicating the existence of a dense disordered phase [10]. This means a gradual increase in the number density of localized magnetic moments, which are detectable with the muon-time scale, with decreasing $T$ below $\sim 60$ K. Accompanying the change in $\beta$, $\lambda_{ZF}$ also increases very rapidly below 70 K with decreasing $T$, suggesting a broadening of the distribution of $H_{int}$. This result is, therefore, consistent with the scenario that disordered moments appear below $\sim 60$ K and develops with decreasing $T$, and finally magnetic order or disorder completes below $T_N$.

As we described in previous paper [6], the wTF-$\mu^+$SR measurements on LMO also demonstrate the existence of a bulk magnetic transition located at $T_N=44$ K, although there
Figure 2. $T$ dependences of (a) $N_{Ai} (= A_i/A_0$, with $i = 1, 2, \text{ and } 3$), (b) $\lambda_1$, and (c) $\lambda_2$ and $\lambda_3$ for NMO. The data was obtained by the fitting of the ZF-spectrum using Eq. (1).

are no clear anomalies in the $\chi(T)$ curve around $T_N$, as in the case for NMO, the ZF-spectrum for LMO is very similar to that for NMO. However, the absence of the fast relaxing signal in the early time domain of the ZF-spectrum for LMO suggests the existence of a larger internal magnetic field in LMO at low $T$ than that in NMO.

4. Discussion
Unfortunately, there are no oscillations in the ZF-spectrum for both NMO and LMO even at $1.9 \text{ K}$. It is therefore impossible to determine the muon sites from the ZF-spectrum at $T$ below $T_N$. However, since the ZF-spectrum exhibits a Kubo-Toyabe behavior above $100 \text{ K}$ for both compounds, the implanted muons are most likely to see the internal magnetic field due to nuclear magnetic moments of $^{23}\text{Na}$, $^7\text{Li}$, $^6\text{Li}$, and $^{55}\text{Mn}$ at high $T$. More correctly, the ZF-spectrum is well fitted by the following function;

$$A_0 P_{ZF}(t) = A_{KT} \exp(-\lambda_{KT} t) G_{DGKT}(\Delta, \nu, t),$$

where $G_{DGKT}(\Delta, \nu, t)$ is a dynamic Gaussian Kubo-Toyabe function, $\lambda_{KT}$ is its relaxation rate, $\Delta$ is the static width of the local frequency at the disordered sites, and $\nu$ is the field fluctuation rate. The exponential relaxation is likely to come from the spin fluctuation of the $d$ electrons.
Figure 3. Temperature dependences of (a) $\beta$, and (b) $\lambda_{ZF}$ for the NMO powder, particularly above $T_N$. The ZF data were obtained by fits using Eq. (2).

Table 1. $\Delta$, $\lambda_{KT}$, and $A_{KT}$ for NMO and LMO. The data were obtained by a global fitting of the ZF- and LF-spectra at $T$ between 100 and 250 K using Eq. (3).

| Compound | $\Delta$ (10$^6$ s$^{-1}$) | $\lambda_{KT}$ (10$^6$ s$^{-1}$) | $A_{KT}$ |
|----------|-----------------------------|---------------------------------|----------|
| NMO      | 0.116(2)                    | 0.052(1)                        | 0.232(4) |
| LMO      | 0.204(4)                    | 0.033(2)                        | 0.220(3) |

probably due to a local Jahn-Teller instability of the Mn$^{3+}$ ions with $t_{2g}^{3}e_{g}^{1}$ configuration, as in the case of the spinel LMO, while $G^{DGK}T(\Delta, \nu, t)$ corresponds to the nuclear magnetic field. A preliminary fit using Eq. (3) for the ZF-spectrum at each $T$ between 100 and 250 K indicated that the three parameters, i.e., $\Delta$, $\lambda_{KT}$, and $A_{KT}$ are approximately independent of $T$. All of the ZF and LF-spectra were, therefore, fitted by Eq. (3) using common $\Delta$, $\lambda_{KT}$, and $A_{KT}$—i.e., a global fitting technique (see Table 1).

On the other hand, electrostatic potential calculations suggest that there are four stable muon sites in the crystallographic lattice, as in the case of NaV$_2$O$_4$ [11]. That is, muons locate at the vicinity of the O$^{2-}$ ions but ~1Å away [see Fig. 4(a)]. Then, dipole field calculations at the four sites provide that $\Delta = 0.32(1) \times 10^6$ s$^{-1}$ for NMO and $\Delta = 0.41(4) \times 10^6$ s$^{-1}$ for LMO. Although the calculations reproduce the relationship $\Delta_{NMO} < \Delta_{LMO}$, the calculated values are larger by 150-200% than the measured ones. Making comparison with the sites predicted by the potential calculations, the real muon sites are likely to deviate far away from the O$^{2-}$ ions, probably due to a variation of the atomic positions with $T$. Furthermore, if we fix $\lambda_{KT} = 0$ in Eq. (3), the global fit yields that $\Delta = 0.324(1) \times 10^6$ s$^{-1}$ for NMO and $\Delta = 0.307(3) \times 10^6$ s$^{-1}$ for LMO, although the fitting accuracy is rather poorer than that using a non-zero $\lambda_{KT}$. This would imply a possible interaction between the spin fluctuation of Mn ions and nuclear magnetism, for the details are unknown.

Figures 4(b) and 4(c) show the $T$ dependences of $\Delta$ and $\nu$ for NMO and LMO. It is found...
Figure 4. (a) possible four muon sites (I - IV) predicted by electrostatic potential calculations and $T$ dependence of the field distribution width ($\Delta$) and the field fluctuation rate ($\nu$) above 100 K for (b) NMO and (c) LMO. $\Delta_{\text{calc}}$ means the calculated $\Delta$ for the muon sites in (a).

that $\nu_{\text{NMO}} \leq 0.06 \times 10^6 \text{s}^{-1}$ in the whole $T$ range measured, while $\nu_{\text{LMO}}$ increases with increasing $T$. Furthermore, $\nu_{\text{LMO}}$ is about 4 times larger than $\nu_{\text{NMO}}$ even at 100 K. This indicates the diffusive motion of the Li ions even at 100 K in the LMO lattice. Indeed, the electrochemical property of LMO was reported in Ref. [4], in order to investigate its performance as a cathode material for reversible Li-ion batteries. Assuming that $\nu_{\text{LMO}}$ corresponds to the jump rate of the Li ions between the neighboring sites, the diffusion coefficient of the Li ions ($D_{\text{Li}}$) would be given by [12]:

$$D_{\text{Li}} = \frac{1}{2} Z_v s^2 \nu,$$

where $Z_v$ is the fraction of the vacancy of the Li site and $s$ is the jump distance. Here, we also assume the diffusion along the $b$-axis, i.e., along the 1D channel, because it is most unlikely that the Li$^+$ ions jump across the Mn$_2$O$_4$ zigzag chain. Therefore, $s$ is equivalent to the $b$-axis length and $Z_v$ is 0.08 from the neutron study [4]; that is, the $D_{\text{Li}}$ is thus estimated as $0.136(1) \times 10^{-10} \text{cm}^2\text{s}^{-1}$ at 250 K, although $D_{\text{Li}}$ evaluated by the other techniques for LMO is currently unavailable. We should here note that the diffusive motion in LMO is most likely enhanced by the Li vacancies ($\sim 8\%$), and would not be an intrinsic feature of the stoichiometric...
NMO and LMO in which the Mn ions form a zigzag by means of positive muon-spin rotation and relaxation (μ+SR).

| Compound | AF Structure | Site I | Site II | Site III | Site IV | Average |
|----------|--------------|-------|---------|----------|---------|---------|
| NMO      | CaV$_2$O$_4$-type | $H_{\text{int}}/\mu_{\text{ord}}$ (Oe/μB) | 1397 | 1276 | 2317 | 1367 | 1600 ± 200 |
| LMO      | CaV$_2$O$_4$-type | $H_{\text{int}}/\mu_{\text{ord}}$ (Oe/μB) | 1567 | 1296 | 1465 | 1775 | 1520 ± 90 |
| NMO      | random       | $H_{\text{int}}/\mu_{\text{ord}}$ (Oe/μB) | 1066 | 972 | 1365 | 948 | 1090 ± 90 |
| LMO      | random       | $H_{\text{int}}/\mu_{\text{ord}}$ (Oe/μB) | 1173 | 1004 | 1386 | 1103 | 1170 ± 70 |

LMO. In order to further investigate the diffusive motion of the Li ions, we need to measure the ZF- and LF-spectra at high $T$ using a pulse-muon facility, such as, ISIS in the UK or J-PARC in Japan.

Here, we discuss the magnitude of the internal magnetic field ($H_{\text{int}}$) in the AF phase below $T_N$. The AF spin structure of the related zigzag chain compound CaV$_2$O$_4$ was determined by neutron experiments [13, 14]; that is, AF order along the two 1D legs in the zigzag chain, and AF order between the zigzag chains. This AF structure is also supported by NMR and μ+SR experiments [15, 11]. Hence, in order to roughly estimate $H_{\text{int}}$ below $T_N$, it would be reasonable to assume the same AF structure for NMO and LMO. We also estimated $H_{\text{int}}$ for the fully disordered phase, i.e., a spin-glass-like phase, although the $\chi(T)$ curve for NMO is likely to suggest the AF order. The result of dipole field calculations for the AF phase is summarized in Table 2.

Assuming that $\mu_{\text{ord}}$ is comparable to $\mu_{\text{eff}}$ for both compounds, $H_{\text{int}} = 5360$ Oe for NMO and 6080 Oe for LMO at low $T$. Note that this estimation provides the upper limit of $H_{\text{int}}$. Then, $H_{\text{int}}$ is directly converted into the muon-spin precession frequency, using the muonic gyromagnetic ratio $[\gamma_\mu/(2\pi) = 13.553$ kHz/Oe]. We should, therefore, expect an oscillatory signal with 73 MHz for NMO and 82 MHz for LMO. Since both frequencies range in the μ+SR timescale, the absence of well-defined oscillatory signal in the ZF-spectrum indicates either a broad distribution of $H_{\text{int}}$ or a rapidly fluctuating $H_{\text{int}}$. Both are consistent with the result of the heat capacity measurements on LMO [4] and neutron and μ+SR results on the spinel LMO [16, 17]. In order to determine whether the magnetic nature is static or dynamic, particularly for a disordered state, an appropriate longitudinal field (LF) is usually applied to decouple the effect of the disordered moments. However, because of the lack of the meaningful signal in an early time-domain of the ZF-spectrum for the both samples, it is very difficult to deduce the nature of the magnetic phase by LF measurements.

As described in previous paper, the $\chi$ measurements imply that the magnitudes of the effective magnetic moment of Mn ions and the paramagnetic Curie temperature for LMO are larger than those for NMO [6]. This would suggest that $H_{\text{int}}$ for LMO is larger than that for NMO. Indeed, if we ignore the oscillatory signal from the Li$_2$MnO$_3$ phase in the ZF-spectrum for LMO, the spectrum eventually consists of only the tail component even at 1.9 K. This suggests that in LMO, a rapidly relaxing signal is hidden below 0.01 μs, in contrast to the case for NMO. Thus, it is reasonable that $H_{\text{int}}$(LMO) > $H_{\text{int}}$(NMO). This could also indicate a different spin structure between NMO and LMO, but the details are unknown based only on the μ+SR experiment. In order to further clarify the AF nature for both compounds, we unambiguously need to perform a neutron experiment.

5. Summary
By means of positive muon-spin rotation and relaxation (μ+SR), we clarified the magnetic nature of NMO and LMO in which the Mn ions form a zigzag Mn$_2$O$_4$ chain. For NMO (LMO), weak transverse field (wTF-) and zero field (ZF-) μ+SR measurements suggest the appearance of a random internal magnetic field at $T$ below 60 K (80 K) and the existence of a bulk magnetic
transition at $T_N = 39$ K (44 K). Although the spin structure below $T_N$ is still unknown, the AF order is thought to be highly disordered, as in the case of a spinel LMO.

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