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Muon spin rotation investigation of the $S = 1/2$ triangular lattice $LiNiO_2$

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Abstract

We have investigated $S = 1/2$ triangular lattice $Li_{0.98}Ni_{1.02}O_2$ by a zero-field (ZF) and longitudinal-field (LF) μSR technique. The muon signals from $Li_{0.98}Ni_{1.02}O_2$ could be fitted by a stretched exponential function at low temperatures. At about 30 K the power k of the stretched exponential function becomes about $k = 2$ for the ZF signal, i.e. the relaxation becomes practically a Gaussian. For the LF-μSR signals the exponent k tends to become 1 above $T = 30$ K, i.e. the signal becomes exponential. The relaxation rate λ becomes very high at temperature below $T = 10$ K for the ZF data whereas λ corresponding to LF data shows a maximum at $T \approx 10$ K. The LF relaxation rate remains finite ($0.4\ \mu s^{-1}$) at the lowest temperature ($T \approx 2$ K) investigated. This indicates that even at very low temperature muon spin becomes depolarized because muons pick up low energy excitations from the spin dynamics. We get a rough estimate of the magnetic exchange interaction of about 1 meV from the peak in the spin–spin correlation function.

A number of theoretical ideas have recently been proposed in order to explain the possible spin–orbital or flavour liquid state in $S = 1/2$ triangular layered lattice $LiNiO_2$ which has no long range order of orbital or spin degrees of freedom down to the lowest temperature investigated. In many transition metal oxides, the electron configuration on the metal ion has both spin and orbital degeneracy. In such systems the signs of spin–spin couplings depend on the orbital occupancy, leading to interesting magnetic properties. The possibility of quantum fluctuations being enhanced and magnetic long range order being destroyed by orbital fluctuations has been proposed by Feiner *et al* [1]. The search for physical realization of this novel physics has continued since then, but the best candidate for showing this still seems to be $LiNiO_2$. Since the $SU(2)$ Heisenberg model on a triangular lattice is believed to have long range order [2, 3], the absence of magnetic long range ordering in $LiNiO_2$ is logically attributed to the orbital degrees of freedom. The Ni^{3+} ions in $LiNiO_2$ have a low spin state with twofold orbital

degeneracy. However, no structural distortions have been observed in $LiNiO_2$ contrary to what is expected from the valence bond ordered spin liquid of [1]. The $SU(4)$ symmetric Kugel–Khomskii model [4, 5], in which orbital and spin degrees of freedom play symmetric roles, has been attracting considerable attention from theoretical condensed matter physicists. The additional orbital degrees of freedom provide possibilities of finding a spin liquid (or more generally a flavour liquid) state in two or three dimensions. Li *et al* [6] have discussed the model on the triangular lattice. The antiferromagnetic (AF) Heisenberg $S = \frac{1}{2}$ model on the triangular lattice is believed to order in a three-sublattice 120° structure [2, 3]. With an orbital degeneracy, such a spin ordering is no longer favoured. Li *et al* [6] have compared the estimated energies for various long range ordered states, including the classical Néel state, the orbital polarized spin ordered state and a valence bond state, with the $SU(4)$ singlet plaquette state. The $SU(4)$ singlet is a singlet of spin, orbital and the orbital–spin crossing operator and is a generalization of the $SU(2)$ singlet of the spin only system. Unlike for the spin only problem in which the classical Néel state and the valence bond state are degenerate, the plaquette state has much lower energy than others. Also the plaquette state can resonate to still lower energy and become a flavour liquid; Li *et al* [6] have speculated that the ground state may be a resonant plaquette state with neither spin nor orbital long range order. Mostovoy and Khomskii [7] have contested the idea of a spin–orbital or flavour liquid picture obtained from the $SU(4)$ symmetric Kugel–Khomskii Hamiltonian and have pointed out the importance of the superexchange interaction in the frustrated Jahn–Teller system with transition metal ions connected by the 90° metal–oxygen–metal bonds. Although their calculations can explain the situation in $NaNiO_2$, they have provided no plausible explanation of the non-conventional ground state of $LiNiO_2$. They have attributed the absence of orbital and magnetic ordering in $LiNiO_2$ to disorder due to non-stoichiometry. Recently Vernay *et al* [8] studied a general spin–orbital model on the triangular lattice. A mean-field approach reveals the presence of several singlet phases between the $SU(4)$ symmetric point and a ferromagnetic phase, a conclusion supported by the exact diagonalization of finite clusters. One of the phases, characterized by a large number of low lying singlets associated with dimer coverings of the triangular lattice, could explain the properties of $LiNiO_2$, while a ferro-orbital phase that lies nearby in parameter space can explain the magnetic properties of $NaNiO_2$, at least partially. The main result of this study is that, in contrast to the result of Mostovoy and Khomski [7], it is found that the absence of orbital and spin ordering in $LiNiO_2$ can be intrinsic. The above ideas seem to fit to the intriguing low temperature properties of $LiNiO_2$ measured so far, including those of the present μSR investigations.

The preparation and characterization of $LiNiO_2$ were done by Goodenough *et al* [9] a long time ago. Inspired by the earlier theoretical works [10–12], Hirakawa [13] first suggested that $LiNiO_2$ is a good candidate for showing spin liquid behaviour. Following this suggestion, $LiNiO_2$ has been synthesized and the physical properties have been measured by many workers [14–33]. The substance also received attention as a very important industrial positive electrode material. Lithium nickelate $Li_{1-x}Ni_{1+x}O_2$, which is always non-stoichimetric, forms an $S = \frac{1}{2}$ triangular lattice. The crystal structure of $Li_{1-x}Ni_{1+x}O_2$ is isotypic with that of α-$NaFeO_2$, having a rhombohedral $R\bar{3}m$ space group. This is a modified NaCl structure in which close-packed triangular lattices of each kind of atoms are stacked in the layer order Ni, O, Li, O. The true formula of lithium nickelate is $Li_{1-x}Ni_{1+x}O_2$, in which x varies in the 0.01–0.20 domain. The non-stoichiometry of lithium nickelate results from the instability of trivalent nickel ions at high temperatures and, irrespective of the experimental condition, lithium nickelate always contains divalent nickel ions. The existence of extra nickel ions in the lithium site coupled with the lithium deficiency requires the presence of $2x$ divalent and $1 - x$ trivalent nickel ions in the structure. Owing to the steric considerations, the

nickel ions situated in the lithium layers are divalent. The divalent Ni ions have $S = 1$ whereas trivalent Ni ions are in low spin $S = 1/2$ states. The older publications on the magnetic properties of $Li_{1-x}Ni_{1+x}O_2$ were full of confusion and contradictions. Quantum spin liquid [13, 15], spin glass [17, 20, 21, 28], frustrated antiferromagnetism [14, 17, 25, 29], a quantum disordered state without a spin gap [22] and other scenarios [18, 19, 23, 24, 26, 29–32] have been proposed for $LiNiO_2$. The reason for this confusion is perhaps the problem of the non-stoichiometry x of the samples studied by these authors. The magnetic properties of $Li_{1-x}Ni_{1+x}O_2$ are known to depend crucially on the stoichiometry. This has been shown by more recent publications. A recent publication by Reynaud *et al* [33] has helped enormously in removing much of the confusion created in the literature. These authors have interpreted the results of their ESR, magnetization and magnetic susceptibility measurements by recognizing the importance of the orbital degrees of freedom in $LiNiO_2$. Near-stoichiometric samples of $LiNiO_2$ have been prepared and there is a general consensus that, although the isostructural compound $NaNiO_2$ shows both orbital and spin ordering [34], the ground state of $LiNiO_2$ seems to be both orbital and spin liquid like. We believe that the resolution of the ground state problem of $LiNiO_2$ is not likely to be achieved by theoretical speculations alone. More experimental results on well characterized near-stoichiometric $LiNiO_2$ samples will be valuable meanwhile.

It is now well recognized that muon spin rotation or μSR is a very sensitive and useful magnetic probe. If the spin polarized μ^+ are stopped or implanted in a sample in which they are subjected to magnetic interactions, their polarization $\vec{P}$ may become time dependent, given by $\vec{P}(t)$. Its time evolution can be monitored by measuring the positron counting rate in a particular direction, say along the direction of the initial muon polarization $\vec{P}(0)$. The positron counting rate $\mathrm{d}N_{e^+}(t)/\mathrm{d}t$ as a function of the elapsed time is given by [35]

$$\frac{\mathrm{d}N_{e^+}(t)}{\mathrm{d}t} = N_0 \frac{1}{\tau_\mu} \exp(-t/\tau_\mu) \left(1 + \frac{A\vec{P(t)} \cdot \vec{P(0)}}{P(0)}\right) \tag{1}$$

where A is the asymmetry parameter, $\tau_\mu = 2.2$ μs is the μ^+ lifetime, the exponential factor accounts for the decay of the μ^+. $\vec{P(t)} \cdot \vec{P(0)}/P(0)^2$ can be identified with the normalized μ^+ spin autocorrelation function

$$G(t) = \frac{\langle \vec{S(t)} \cdot \vec{S(0)} \rangle}{\langle S(0)^2 \rangle}. \tag{2}$$

The function $G(t)$ contains all the information that can be extracted from the time evolution of the muon polarization $\vec{P(t)}$. $P(t)$ is defined as the projection of $\vec{P(t)}$ onto the initial polarization $\vec{P(0)}$, chosen to be the direction of positron observation. Thus

$$P(t) = \frac{\vec{P(t)} \cdot \vec{P(0)}}{P(0)} = G(t)P(0). \tag{3}$$

$P(t)$ is called the μSR signal. It is also called the asymmetry since it determines the effective decay asymmetry in the distribution given by equation (1).

We have performed zero-field (ZF) and longitudinal-field (LF) μSR investigations on the nearly stoichiometric $Li_{0.98}Ni_{0.02}O_2$ on the GPS beam line of the Paul-Scherrer-Institut. The sample used for the present investigation is from the same batch as was used by Reynaud *et al* [33] and was prepared following the procedure described by Rougier *et al* [25]. The polycrystalline sample was pressed into cylindrical plates of radius of about 8 mm and thickness of about 2 mm by applying high pressure. One such plate shaped sample was fixed on top of the sample holder made of silver plate of very high purity. The sample holder was mechanically fixed to the cold tip of the helium flow cryostat of the GPS beam line. The incident muon

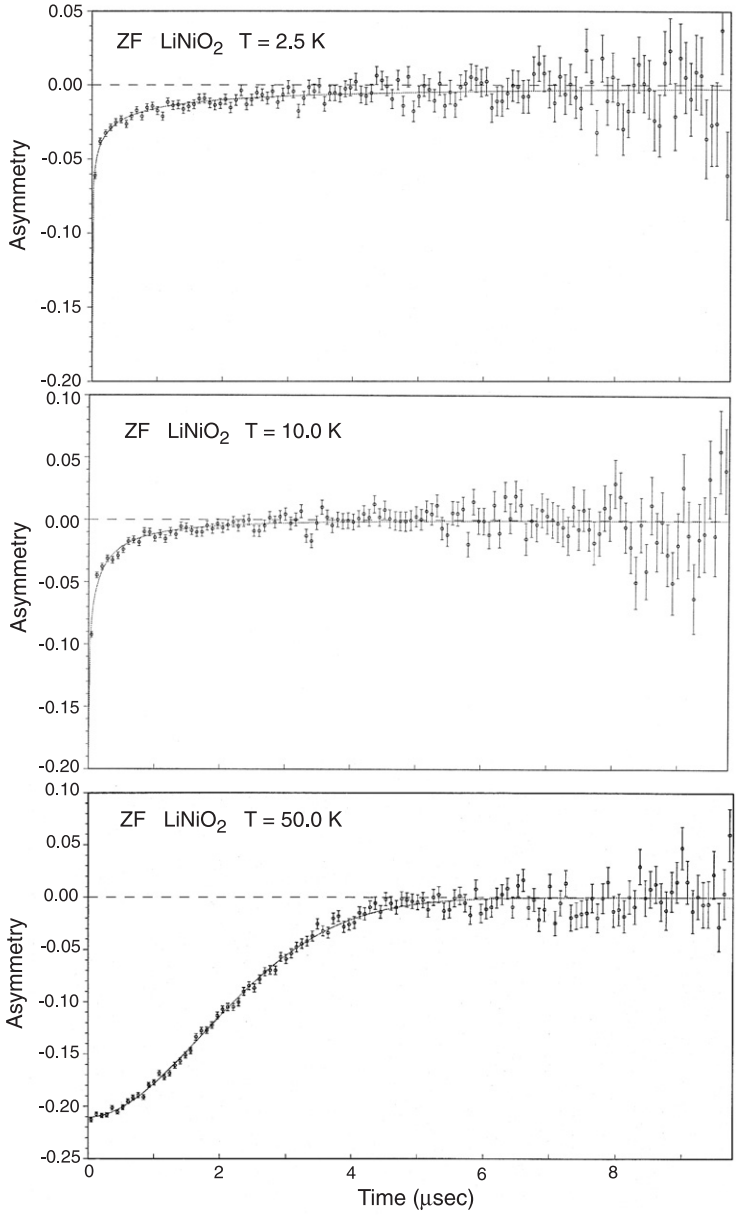

Figure 1. The zero-field muon depolarization from $Li_{0.98}Ni_{1.02}O_2$ at a few selected temperatures as a function of time. The continuous curve is the result of a fit with the stretched exponential function.

beam was perpendicular to the sample plate. The initial muon polarization was parallel to the beam. The muon signals were recorded at several temperatures in the range from 2.5 to 100 K at zero field and also in a longitudinal field of 6000 G. Figure 1 shows the zero-field (ZF) muon depolarization (asymmetry) from $Li_{0.98}Ni_{1.02}O_2$ at a few selected temperatures as a function of time. Figure 2 shows the same in a LF of 6000 G. Both ZF and LF signals showed no

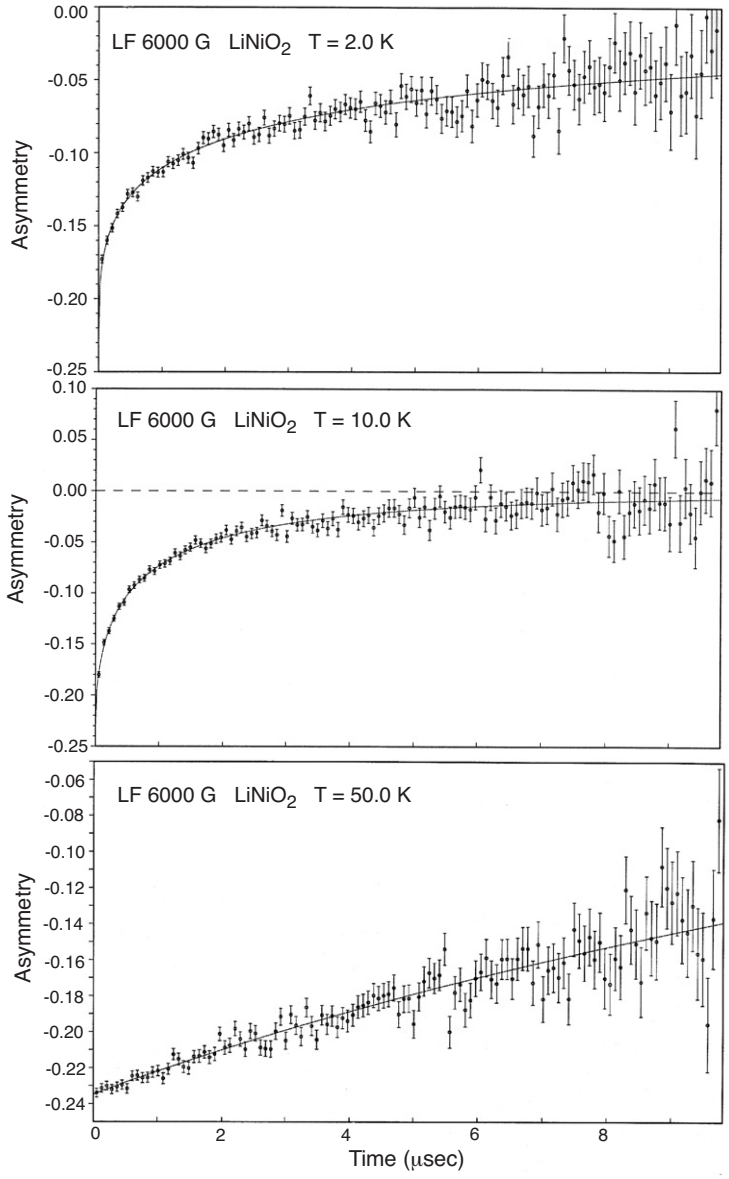

Figure 2. The longitudinal-field muon depolarization from $Li_{0.98}Ni_{1.02}O_2$ at a few selected temperatures as a function of time. The continuous curve is the result of a fit with the stretched exponential function.

oscillations but showed relaxations which could be fitted by a stretched exponential function

$$P(t) \propto \exp[-(\lambda t)^k] \tag{4}$$

where λ is the depolarization rate and k is an exponent. Figure 3 shows the temperature variation of the ZF muon relaxation rate λ and the exponent k of the stretched exponential function fitted to the muon signals. The relaxation rate at $T = 2.5$ K is $60 \pm 7\ \mu s^{-1}$. The relaxation rate decreases rapidly with increasing temperature and becomes of the order of

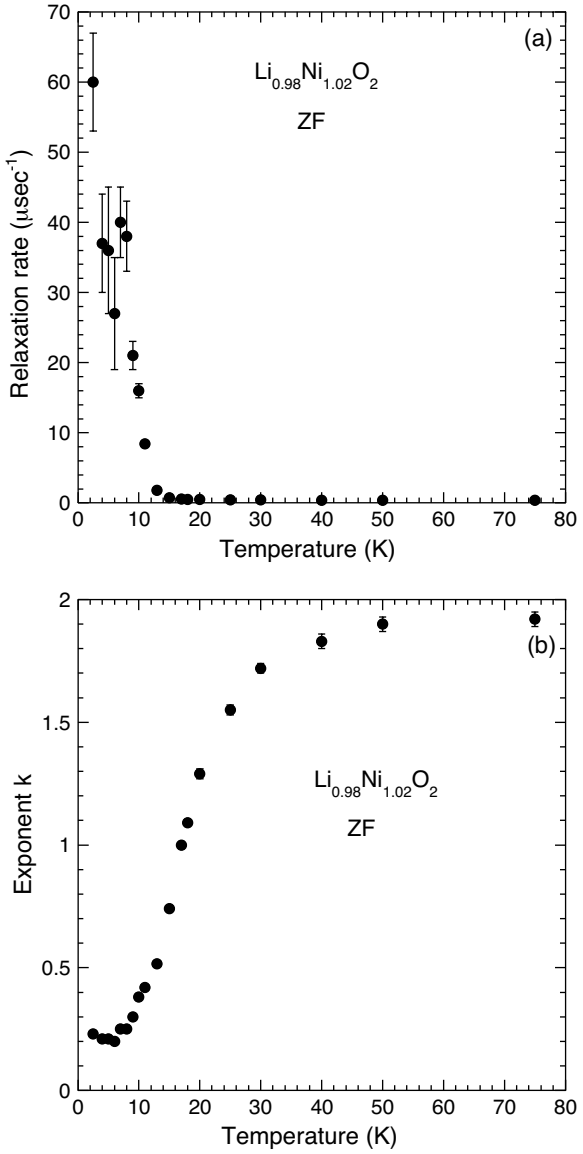

Figure 3. (a) The zero-field muon relaxation rate λ of $Li_{0.98}Ni_{1.02}O_2$ as a function of temperature. (b) The exponent k of the stretched exponential function fitted to the muon signal from $Li_{0.98}Ni_{1.02}O_2$ as a function of temperature.

0.4 μs^{-1} at about $T = 10$ K, and it retains approximately this value at higher temperatures up to 100 K. The exponent k of the stretched exponential is 0.23 ± 0.01 at $T = 2.5$ K. The value is retained up to about 10 K but increases rapidly at temperatures above about 10 K. The value of k becomes saturated at about $T = 50$ K: 1.90 ± 0.03. The value $k = 2$ corresponds to Gaussian relaxation. The temperature variations of both the relaxation rate and the exponent of the stretched exponential suggest that there is some sort of spin freezing below about $T = 10$ K. However, one expects dynamic effects below $T = 10$ K rather than static spin freezing.

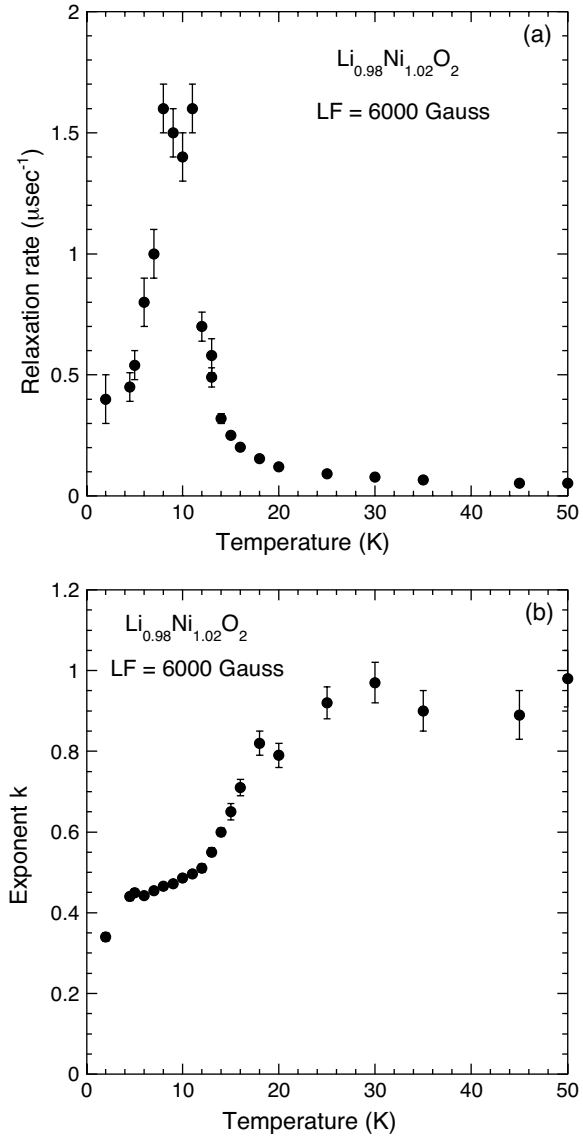

Figure 4. (a) The longitudinal-field muon depolarization rate λ of $Li_{0.98}Ni_{1.02}O_2$ as a function of temperature. (b) The exponent k of the stretched exponential function fitted to the LF muon signal from $Li_{0.98}Ni_{1.02}O_2$ as a function of temperature.

To suppress the static effects, we investigated the temperature dependence of the muon signal in an applied longitudinal field of 6000 G or 0.6 T. We know from magnetization measurements [33] on $LiNiO_2$ up to 15 T that a magnetic field of 0.6 T does not change the magnetic properties of $LiNiO_2$. On the other hand, a LF of 0.6 T is strong enough to decouple the static effects from the spin dynamics probed by μSR. We fitted a stretched exponential function $Ae^{(-\lambda t)^k}$ to the muon signals. Figure 4 shows the muon relaxation rate λ and the exponent k as a function of temperature. We note that the relaxation rate λ is much reduced by the application of a longitudinal field. For example, at $T = 2.5$ K the fitted relaxation rate

is only about 0.4 μs^{-1} compared to the zero-field value of about 60 μs^{-1}. More importantly, the temperature variation of the relaxation rate λ now shows a maximum at about $T = 10$ K. At higher temperature the muon relaxation rate decreases again and becomes 0.05 μs^{-1} at $T = 50$ K. The exponent is $k = 0.33$ at $T = 2.5$ K. It increases with increasing temperature and becomes about 1 (exponential) at about $T = 30$ K.

We summarize the results of the present μSR investigations on $Li_{0.98}Ni_{1.02}O_2$:

(1) Both ZF and LF depolarization from $Li_{0.98}Ni_{1.02}O_2$ can be fitted satisfactorily by a stretched exponential function below about $T = 30$ K. At higher temperatures the ZF muon depolarization becomes Gaussian ($k = 2$) whereas the LF polarization becomes exponential ($k = 1$).
(2) ZF signals below $T = 10$ K have huge depolarization rates and also have loss of asymmetry. The LF signals have the full expected asymmetry.
(3) The temperature dependence of the relaxation rate determined from the LF signal shows a maximum at about $T = 10$ K.

In the following paragraphs we will attempt to understand the implications of the above results.

The stretched exponential depolarization has been observed in a wide variety of spin systems. Although first detected in spin glasses, it is by no means restricted to such spin systems [36]. Experiments on moderately concentrated spin glasses (5–10% magnetic sites) show depolarization functions above the spin glass temperature T_g which can be fitted by a stretched exponential function. The relaxation rate λ increases as the temperature is lowered towards T_g from the high temperature side while the exponent k drops from a value near 1 to a limiting value of about $1/3$ as T_g is approached. This behaviour seems to be very general and indicates a wide spectrum of local spin relaxation rates, combined with non-exponential relaxation at individual sites. A limiting value of k of approximately $1/3$ when T_g is approached has been observed in an impressive number of systems. The crossover to exponential depolarization always occurs well above T_g, generally at temperatures $5T_g$–$10T_g$. Such behaviour has been observed not just in canonical metallic spin glasses, but has been observed also in CMR manganites [37] and frustrated pyrochlore lattices [38].

Let us now discuss the muon depolarization in a longitudinal field. Ignoring the direct effect of the field on the spin dynamics, the form of depolarization in a field is directly related to the power spectrum of the dipole-field fluctuations at the muon site. Thus the μSR method yields spin–lattice relaxation rates of the local probes (implanted positive muons) which are sensitive to fluctuations of the dipolar local field $h(t)$ at muon sites. This yields a broad average over all q, as in nuclear magnetic resonance (NMR). The time dependence of the local-field correlation function $S_h(\vec{q}, t)$, averaged over all $\vec{q}$, can be measured indirectly using μSR in a longitudinal field and the result can be compared with $S_\sigma(\vec{q}, t)$, obtained by using the neutron spin echo (NSE) in zero field. The two techniques are complementary in the time domain, since the NSE is most sensitive to correlation times in $S_\sigma(\vec{q}, t)$ between 10^{-8} and 10^{-12} s, whereas the μSR is most sensitive to times between 10^{-4} and 10^{-11} s. So the present LF muon depolarization rate contains very useful information about the spin dynamics on a timescale between 10^{-4} and 10^{-11} s. For comparison of the present μSR results with theoretical models of the ground states of $LiNiO_2$, it would be very helpful to calculate the temperature dependence of spin–spin time autocorrelation function of the spin dynamics of a frustrated spin–orbital system like $LiNiO_2$. Although such calculations are not available at the moment, we can still draw some qualitative conclusions from the temperature dependence of the LF muon spin relaxation rate. The peak in the temperature dependence of the spin–spin time correlation function is not unexpected in a frustrated spin–orbital system such as $LiNiO_2$. The $S = 1/2$ and 1 linear Heisenberg antiferromagnetic chains also show such a peak in

the temperature dependence of the susceptibility. The temperature at which the peak appears can be identified as the strength of the corresponding exchange interaction. At $T = 0$, the spin–spin time autocorrelation function should become zero for a system with a gap (Haldane gap [39]) such as the $S = 1$ linear Heisenberg chain and should remain finite for a gapless system such as the $S = 1/2$ linear Heisenberg chain [40]. The peak observed at $T \approx 10$ in the temperature dependence of the LF muon spin relaxation rate gives approximately the strength of the exchange interaction in $LiNiO_2$. The exchange interaction is about 1 meV. The exchange interaction is at least predominantly antiferromagnetic. Similar conclusions were also drawn by Reynaud *et al* [31] from their ESR, magnetization and magnetic susceptibility measurements on $LiNiO_2$. It is not quite clear whether a realistic spin–orbital $S = 1/2$ model on a triangular lattice has an energy gap or not, but intuitively and also from the analogy with the results from other $S = 1/2$ models one may perhaps think that the energy spectrum should be gapless. The LF muon spin relaxation rate given in figure 4(a) shows a finite relaxation rate ($0.4\ \mu s^{-1}$) at the lowest temperature investigated. This indicates that even at very low temperature the muon spin becomes depolarized due to muons picking up low energy excitations from the spin dynamics. So the system ought to be gapless. This may be a crucial test for theoretical models which have been put forward.

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