Fluctuations and correlations in a frustrated $S = 1/2$ square lattice with competing ferromagnetic and antiferromagnetic interactions: a $\mu$SR study

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Zero and longitudinal field $\mu$SR measurements in Pb$_2$VO(PO$_4$)$_2$ and BaCdVO(PO$_4$)$_2$, two prototypes of the frustrated $S = 1/2$ square lattice model with competing ferromagnetic and antiferromagnetic interactions, are presented. Both systems are observed to undergo a phase transition to a long-range magnetic order at $T_N \simeq 3.46$ K, for Pb$_2$VO(PO$_4$)$_2$, and at $T_N \simeq 0.99$ K, for BaCdVO(PO$_4$)$_2$. In Pb$_2$VO(PO$_4$)$_2$ both the temperature dependence of the order parameter and the longitudinal relaxation rate above $T_N$ are consistent with a two-dimensional XY model. On the other hand, for BaCdVO(PO$_4$)$_2$, which lies very close to the magnetically disordered region of the phase diagram where a bond-nematic order was predicted, a peculiar logarithmic increase of the relaxation is observed above $T_N$. In both systems a rather broad distribution of internal fields at the muon sites is noticed below $T_N$. The origin of this distribution is discussed in the light of the $\mu$SR experiments already performed on $S = 1/2$ frustrated antiferromagnets on a square lattice.

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I. INTRODUCTION

Strongly correlated electron systems with competing interactions are known to show rather rich phase diagrams, with crossovers or phase transitions which depend on the relative magnitude of the competing energy scales. In certain cases rather complex scenarios are observed together with the insurgence of novel phases of matter, a situation typically found in frustrated magnets where, for instance, spin ice or spin liquid ground-states arise. In these insulating systems either the geometry of the underlying spin lattice or the geometry of the interactions causes the suppression of the long-range magnetic order and the onset of exotic phases. For instance, for a frustrated $S = 1/2$ square lattice with competing ferromagnetic and antiferromagnetic interactions (hereafter QFFMSL for short) it has been proposed that for exchange couplings yielding to the disappearance of long-range magnetic order, a nematic order could be present. QFFMSL are characterized by a ferromagnetic nearest neighbour (n.n.) exchange coupling $J_1$, along the sides of the square spin lattice, competing with the antiferromagnetic next nearest neighbour (n.n.n.) coupling $J_2$, along the diagonals. For a frustration ratio $-0.4 \geq J_2/J_1 \geq -0.7$ long-range magnetic order should disappear and a nematic order of spin bonds should arise. In this phase, the individual spins do not carry any orientational order but the traceless tensor

$$O^{\alpha \gamma} = \frac{1}{2}(S_i^\alpha S_j^\gamma + S_j^\gamma S_i^\alpha) - \frac{1}{3}S^\alpha S^\gamma \langle \vec{S}_i \cdot \vec{S}_j \rangle,$$

with $\alpha, \gamma = x, y, z$ and $i, j$ running over spin lattice sites, does order and gives rise to stripy like correlations in the plane. This interesting theoretical result lacks of any experimental confirmation, basically because there is no material characterized by a frustration ratio $J_2/J_1 \simeq -0.5$. Nevertheless, in the last years there has been a considerable effort to synthesize novel vanadates, containing $V^{4+}$, $S = 1/2$ ions, which have exchange couplings in this region of the $J_1 - J_2$ phase diagram. Several prototypes QFFMSL have been synthesized, with ratios $J_2/J_1$ ranging from $-1.8$ for Pb$_2$VO(PO$_4$)$_2$ to -0.9 for BaCdVO(PO$_4$)$_2$, very close to the boundary between the long-range magnetic order and the nematic order. On the other hand, it has been recently pointed out by Tsirlin and Rosner that in these compounds the n.n. and n.n.n. exchange couplings along different sides and diagonals of the square lattice, respectively, might be inequivalent due to the buckling and stretching of the magnetic layers. The difference between the exchange couplings appears to be less pronounced for BaCdVO(PO$_4$)$_2$ than for Pb$_2$VO(PO$_4$)$_2$.

![FIG. 1: Time evolution of the ZF$\mu$SR asymmetry in Pb$_2$VO(PO$_4$)$_2$ at two representative temperatures, above and below $T_N$. The solid lines are best fits according to Eq. 2 (data at $T = 3.7 K$) and 3 (data at $T = 3.37 K$) in the text.](image)

In order to study the evolution of the local microscopic...
properties of QFFMSL upon decreasing the ratio $J_2/J_1$ towards the critical value $J_2/J_1 \approx -0.7$ and how they compare to the ones of frustrated $S = 1/2$ square lattice systems with competing antiferromagnetic interactions, \cite{8,9} (QFAFLS, i.e. for $J_2/J_1 > 0$), we have performed zero field (ZF) and longitudinal field (LF) $\mu$SR experiments on BaCdVO(PO$_4$)$_2$ and Pb$_2$VO(PO$_4$)$_2$ powders. It was found that although both systems are characterized by a magnetically ordered ground-state below $T_N$, significant differences with respect to the behaviour observed for QFAFLS are present. The correlated spin dynamics in Pb$_2$VO(PO$_4$)$_2$ for $T > T_N$ and the temperature dependence of the magnetic order parameter are both consistent with a two-dimensional (2D) XY model. On the other hand, in BaCdVO(PO$_4$)$_2$ a rather peculiar behaviour is observed, with a logarithmic increase of the longitudinal muon relaxation rate ($\lambda$) on cooling for $T > T_N$. This behaviour is reminiscent of the one observed in one-dimensional systems and might suggest the onset of bond-nematic correlations above $T_N$.

II. TECHNICAL ASPECTS AND EXPERIMENTAL RESULTS

A polycrystalline sample of Pb$_2$VO(PO$_4$)$_2$ was synthesized by solid state reaction technique using PbO (99.99%), (NH$_4$)$_2$H$_2$PO$_4$ (99.9%), and VO$_2$ (99.99%) as starting materials. In the first step, the intermediate compound Pb$_2$P$_2$O$_7$ was prepared firing the stoichiometric mixtures of PbO and (NH$_4$)$_2$H$_2$PO$_4$ in air at 750 °C for 24 hours. In the second step, the intermediate product was mixed with VO$_2$ in appropriate molar ratio and heated for 48 hours at 680 °C in dynamic vacuum with one intermediate grinding and pelletization. Synthesis of BaCdVO(PO$_4$)$_2$ was done following the same procedure reported in Ref. \cite{6}. Single phase materials were confirmed by x-ray diffraction performed with a STOE powder diffractometer (CuKα radiation). Both materials, Pb$_2$VO(PO$_4$)$_2$ and BaCdVO(PO$_4$)$_2$, contained a minor (2-3%) fraction of unreacted diamagnetic phosphates Pb$_2$P$_2$O$_7$ and BaCdP$_2$O$_7$, respectively. These impurities are non-magnetic and, therefore, are irrelevant for the discussion to be presented in the following.

ZF and LF$\mu$SR measurements were performed on EMU and MUSR beam lines at ISIS pulsed muon facility, using 29 MeV/c spin-polarized muons. The powders of Pb$_2$VO(PO$_4$)$_2$ and BaCdVO(PO$_4$)$_2$ were pressed and attached to a silver sample-holder, whose background contribution ($B$) to the muon asymmetry was determined from the slowly decaying part of the longitudinal polarization. We checked that there were no history dependent effects associated with a poor sample thermalization onto the dilution fridge cold plate.

In Pb$_2$VO(PO$_4$)$_2$ at temperatures above $T_N = 3.46 \pm 0.01$ K the decay of the muon polarization $P_\mu(t)$, either in zero (Fig. 1) or in a longitudinal field, could be fitted by the expression

$$P_\mu(t) = A_1 e^{-\sigma t} J_0(\gamma_\mu B_\mu t) + A_2 e^{-(\lambda t)^\beta} + B ,$$

In ZF, for $T \gg T_N$, $\beta$ was found to increase slightly above unity which indicates that at high temperature a non-negligible contribution to the relaxation arises from the field distribution generated by the dipolar interaction with the nuclei, which typically gives a Gaussian decay. On the other hand, when a LF of 1 kGauss was applied in order to quench the nuclear dipole contribution, $\beta$ was found to decrease from unity to about 0.7 on approaching $T_N$.

Below $T_N$ ZF experiments showed the insurgence of oscillations in the muon asymmetry (Fig. 1) which clearly indicate the presence of a long-range magnetic order, which generates an internal field at the muon site $B_\mu$. Accordingly one observes a precessional signal at a frequency $\omega_\mu = \gamma_\mu B_\mu$, with $\gamma_\mu$ the muon gyromagnetic ratio. Nevertheless, the initial decay of the muon polarization can hardly be fitted by assuming a well defined field at the muon. On the other hand, one can fit $P_\mu(t)$ over a broad temperature range below $T_N$ with the expression

$$P_\mu(t) = A_1 e^{-(\sigma t)} J_0(\gamma_\mu B_\mu t) + A_2 e^{-(\lambda t)^\beta} + B ,$$

where $J_0(x)$ is the zero-th order Bessel function of the first kind, which characterizes the decay of the muon polarization in the presence of a distribution of internal fields which is further broadened by the decay constant $\sigma$. In fact, if one performs the Fourier transform of the oscillating component of $P_\mu(t)$ one finds quite a broad spectrum (Fig. 2). Upon decreasing the temperature below $T_N$ one observes a loss of the initial asymmetry, possibly due to a further broadening of the field distribution. The temperature dependence of $B_\mu$, derived by

![FIG. 2: Power of the Fourier transform of the oscillating component of the ZF$\mu$SR asymmetry for Pb$_2$VO(PO$_4$)$_2$, BaCdVO(PO$_4$)$_2$ and Li$_2$VOSiO$_4$, for $T < T_N$. It is evident that while in Li$_2$VOSiO$_4$, a well defined internal field is present, in Pb$_2$VO(PO$_4$)$_2$ and BaCdVO(PO$_4$)$_2$ a broad distribution of internal fields is probed by the muons.](image-url)
fitting the ZF decay of the muon asymmetry with Eq. 3 is reported in Fig. 3. In Fig. 4 the temperature dependence of the longitudinal relaxation rate $\lambda$ is reported. A clear divergence of the relaxation rate is observed at $T_N$.

Also for BaCdVO(PO$_4$)$_2$ at temperatures above $T_N = 0.99 \pm 0.01$ K the decay of the muon polarization can be nicely fitted with Eq. 2, with an exponent decreasing from $\beta = 1$ for $T \approx 10$ K to $\beta = 0.6$ on approaching $T_N$ (Fig. 5). On the other hand, although in ZF below $T_N$ oscillations in $P_\mu(t)$ are observed, confirming the onset of a long-range magnetic order, the decay of the muon polarization can hardly be fitted by Eq. 3 and a much broader distribution of internal fields is present. In fact, by looking at the Fourier transform of the oscillating component of the muon asymmetry (Fig. 2) one clearly notices that a significant distribution of internal fields is present, much broader than in QFAFSL$_{12}$. Still, it is possible to fit the slowly decaying non-oscillating component of $P_\mu(t)$ with a stretched exponential in order to derive the temperature dependence of $\lambda$ over all the explored temperature range. Again a peak is present at $T_N$ (Fig. 6). However it is noticed that for $T > T_N$ the increase of $\lambda$ on cooling is rather different from the one observed in Pb$_2$VO(PO$_4$)$_2$.

III. DISCUSSION

First we shall address the temperature dependence of the muon longitudinal relaxation rate and the temperature dependence of $B_\mu$ in Pb$_2$VO(PO$_4$)$_2$. The temperature dependence of $B_\mu$ corresponds to the one of the order parameter. In fact, in zero-field the local field at the muon can be written as $\vec{B}_\mu = A < \vec{S} >^{10}$, where $A$ is the hyperfine coupling tensor describing the coupling of the muon with the surrounding V$^{4+}$ $S = 1/2$ spins and $< \vec{S} >$ the corresponding spontaneous spin polar-
ization. In spite of the distribution of local fields evidenced in Fig. 2, which yields some uncertainty in the estimate of $B_\mu$, the basic trend of $B_\mu(T)$ is reminiscent of the one observed for Li$_2$VOSiO$_4$ QFAFSL. In this latter compound it has been observed that for $T \to T_N$ the order parameter increases according to the critical power law $B_\mu \propto (1 - T/T_N)^{\beta_\mu}$, with a critical exponent $\beta_\mu \simeq 0.235$, the one predicted for a 2D XY system. If one reports $B_\mu$ data for Pb$_2$VO(PO$_4$)$_2$ in the proximity of $T_N$ as a function of the reduced temperature $(1 - T/T_N)$ (see the inset of Fig. 3), one realizes that also for Pb$_2$VO(PO$_4$)$_2$ $\beta_\mu \simeq 0.235$.

The temperature dependence of the muon longitudinal relaxation rate $\lambda$ in BaCdVO(PO$_4$)$_2$. In the inset the same data are reported vs. $1/T$ in a linear-log scale in order to evidence the logarithmic increase of $\lambda$ above $T_N$.

The temperature dependence of the muon longitudinal relaxation rate above $T_N$ originates from the one of the in-plane correlation length. In fact, if we neglect the nuclear contribution to the relaxation which is temperature independent and in any way, quenched by the application of a longitudinal field, the critical increase of $\lambda$ on approaching $T_N$ is of dynamical nature and associated with spin-lattice relaxation processes. Then one can write

$$\lambda = \frac{x^2}{2N} \sum_{q,\alpha} |A_q|^2 S^{\alpha\alpha}(q, \omega)$$

where $S^{\alpha\alpha}(q, \omega)$ are the components of the dynamical structure factor at the muon Larmor frequency and $A_q$ is the form factor describing the hyperfine coupling of the spin excitations at wave-vector $q$ with the nuclei. By resorting to scaling arguments, which are expected to be valid for $T \to T_N$, one finds that for a 2D spin system $\lambda \propto \xi^z$ with $\xi$ the in-plane correlation length and $z$ the dynamical scaling exponent. Considering that Pb$_2$VO(PO$_4$)$_2$ lies in the sector of the $J_1-J_2$ phase diagram where the antiferromagnetic $J_2$ coupling is larger, one could at first take for $\xi$ the temperature dependence expected for a $S = 1/2$ 2D Heisenberg antiferromagnet on a square lattice. Namely, $\xi \propto \exp(2\pi \rho_s/T)^{12}$, with $\rho_s$ the spin-stiffness constant, which is possibly reduced with respect to the value $\rho_s = 1.15 \sqrt{J_1/2\pi}$ expected for a non-frustrated systems. Accordingly one should observe that $\lambda \propto \exp(2\pi \rho_s/T)$. However, we have found that this expression can fit the data only over a limited temperature range, no matter which value one takes for $\rho_s$. On the other hand, the temperature dependence of the order parameter below $T_N$ suggests considering for $\xi$ the form predicted for a 2D XY model, namely $\xi \propto \exp[(B_{XY}/(T - T_N)]^{1/2}$, where $B_{XY}$ is of the order of magnitude of the exchange coupling constant. Then, one should find

$$\lambda = c \propto \exp(z^2 B_{XY}/(T - T_N))^{1/2}$$

Indeed, Pb$_2$VO(PO$_4$)$_2$ data can be nicely fitted with Eq. 5 over almost all the explored temperature range (Fig. 4). It is worth comparing the behaviour observed for Pb$_2$VO(PO$_4$)$_2$ with the one found for a prototype of the $S = 1/2$ 2D XY model, Sr$_2$CuO$_2$Cl$_2$. In Fig. 4 we compare the temperature dependence of $\lambda$ for Pb$_2$VO(PO$_4$)$_2$ and for Sr$_2$CuO$_2$Cl$_2$, reported as a function of $\sqrt{J_1/(T - T_N)}$, with $J_1 = \sqrt{J_1^z + J_1^\perp}$ an effective exchange coupling. One notices that for both systems Eq. 5 is followed very well.

Now we shall address the temperature dependence of the order parameter describing the BaCdVO(PO$_4$)$_2$ ground-state. A broad distribution of local fields at the muon site, even larger than the one found for Pb$_2$VO(PO$_4$)$_2$, is noticed (Fig. 4). This field distribution should not be associated with sample inhomogeneities since the phase transitions detected by $\lambda$ or specific heat measurements appear rather sharp. On the other hand, it should be mentioned that in QFAFSL as Li$_2$VOSiO$_4$, where the buckling along the $a$-axis is not present and the unit cell is tetragonal, such a broad distribution is absent (Fig. 4). Thus, at first sight one could think that the different structure could yield a distribution of muon sites, leading to a distribution of hyperfine coupling constants which could justify the broadening of the Fourier transform spectra. However, it is rather difficult to associate such a broad distribution with the presence of a discrete number of $\mu^+$ sites and moreover the distribution is larger in BaCdVO(PO$_4$)$_2$ where the $ab$ plane is less distorted. Furthermore, even if a priori the presence of several inequivalent muon sites cannot be excluded, we mention that in oxides with superstructures and much larger unit cells, as Bi$_2$Sr$_2$YCu$_2$O$_{8+}$ and Sr$_{14}$Cu$_2$O$_{19}$ for instance, just up to two muon sites are observed.

If the significant broadening in the Fourier transform is not due to a distribution of inequivalent probes it means that $< \hat{S} >$ varies from lattice cell to lattice cell, namely
either the underlying magnetic lattice is not commensurate with the crystal lattice structure or domains are present at the microscopic level. In this respect it is instructive to compare our results to neutron scattering experiments in Pb$_2$VO(PO$_4$)$_2$ indicate the presence of a columnar order, a broad background distribution of the energy integrated dynamical structure factor is noticed in q-space, even well below $T_N$. The origin of this broad distribution could suggest the presence of small size domain like structures. Indeed Pb$_2$VO(PO$_4$)$_2$ and BaCdVO(PO$_4$)$_2$ are in a sector of the $J_1-J_2$ phase diagram where a two-fold degenerate magnetic ground-state is present and one could speculate that under certain conditions the coexistence of domains of both phases, characterized by different magnetic wave vectors, could be observed. Although, we cannot draw a clear conclusion on the origin of the broadening of the spectra, it is clear that in BaCdVO(PO$_4$)$_2$ and Pb$_2$VO(PO$_4$)$_2$ a larger degree of structural and possibly of magnetic disorder is present with respect to QFAFSL as Li$_2$VOSiO$_4$.

In BaCdVO(PO$_4$)$_2$ the application of a moderate magnetic field is observed to affect significantly the spectrum of the excitations and the specific heat. Therefore, in order to probe the intrinsic low-energy excitations of this system we have performed zero-field relaxation experiments. Under these conditions an additional contribution to the decay of the muon polarization, associated with the dipolar interaction with the nuclei, might be present. Nevertheless, the functional form of $P_\mu(t)$ suggests that in BaCdVO(PO$_4$)$_2$ this contribution is less significant than in Pb$_2$VO(PO$_4$)$_2$, where the nuclear contribution to the relaxation is already minor for $T \rightarrow T_N$. In fact, in this latter compound $\lambda$ is observed to diverge for $T \rightarrow T_N$ according to Eq.5 also inZF.

On the other hand, the temperature dependence of the zero-field relaxation rate in BaCdVO(PO$_4$)$_2$ shows a rather peculiar behaviour, not observed in Pb$_2$VO(PO$_4$)$_2$. At high temperature, for $T > 4$ K, $\lambda$ is temperature independent, as expected for a non-correlated spin system. On decreasing the temperature towards $T_N$, one would expect for a 2D system, on the basis of Eq. 4, an exponential growth of $\lambda$ which is found to increase logarithmically, i.e. $\lambda \propto \ln(1/T)$ (Fig. 6), a trend which is rather characteristic of one-dimensional systems. This behaviour should not be related to the filtering of the critical fluctuations by the form factor in Eq. 4. In fact, if this was the case one should expect the cancellation of the magnetic field at the muon site below $T_N$, at variance with the experimental findings (Fig. 2). On the other hand, since BaCdVO(PO$_4$)$_2$ should lie very close to the boundary with the bond-nematic phase, one could expect that nematic correlations of stripy character arise above $T_N$ and yield a logarithmic increase of $\lambda$ with decreasing temperature. This occurs until the XY anisotropy and/or the interlayer coupling cause the crossover to a three-dimensional long-range order at a finite temperature resulting in an abrupt increase of $\lambda$ very close to $T_N$ (see Fig. 6).

IV. CONCLUSIONS

In this manuscript we have presented new $\mu$SR measurements in two prototypes of QFFMSL: Pb$_2$VO(PO$_4$)$_2$ and BaCdVO(PO$_4$)$_2$. In both compounds a broad distribution of local fields at the muon site is evidenced and is tentatively associated either with an incommensurate magnetic order or with the formation of mesoscopic domains. In Pb$_2$VO(PO$_4$)$_2$ the overall temperature dependence of the longitudinal relaxation rate and of the order parameter below $T_N$ are both consistent with a 2D XY model. On the other hand, in BaCdVO(PO$_4$)$_2$ a rather peculiar behaviour of the longitudinal relaxation rate was evidenced, which can be hardly associated with conventional 2D spin correlations. The logarithmic increase of $\lambda$ on cooling suggests the onset of one-dimensional correlations which might be related to the insurgence of novel type of correlations in this QFFMSL.

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