Magnetic Field Induced Transition in Vanadium Spinels

E. D. Mun\textsuperscript{1}, Gia-Wei Chern\textsuperscript{2}, V. Pardo\textsuperscript{3}, F. Rivadulla\textsuperscript{4}, R. Sinclair\textsuperscript{5}, H. D. Zhou\textsuperscript{5}, V. S. Zapf\textsuperscript{3} and C. D. Batista\textsuperscript{2}

\textsuperscript{1} NHMFL Materials Physics and Applications, \textsuperscript{2}Theoretical Division, T-4 and CNLS, Los Alamos National Lab (LANL), Los Alamos NM 87545 (USA)
\textsuperscript{3}Departamento de Física Aplicada and \textsuperscript{4}CQUS, Universidad de Santiago de Compostela, 15782 Santiago de Compostela, Spain and \textsuperscript{5}Department of Physics and Astronomy, University of Tennessee, Knoxville, Tennessee 37996-1200 (USA)

(Dated: May 11, 2014)

We study vanadium spinels $AV_2O_4$ ($A = \text{Cd, Zn, and Mg}$) in pulsed magnetic fields up to 65 T. A jump in magnetization at $\mu_0H \approx 40$ T is observed in the single-crystal MgV$_2$O$_4$, indicating a field induced quantum phase transition between two distinct magnetic orders. In the multiferroic CdV$_2$O$_4$, the field-induced transition is accompanied by a suppression of the electric polarization. By modeling the magnetic properties in the presence of strong spin-orbit coupling characteristic of vanadium spinels, we show that both features of the field-induced transition can be successfully explained by including the effects of the local trigonal crystal field.

PACS numbers: 75.10.Jm, 71.70.Gm, 75.50.Ee

The interplay between spin and orbital degrees of freedom of highly frustrated magnets becomes particularly relevant when the ground state manifold of the dominant interaction is massively degenerate\textsuperscript{[1]}. This is the ideal scenario for finding rich phase diagrams because small interactions become the primary selection mechanism of spin-orbital ordering. A high susceptibility to small interactions opens the possibility of inducing phase transitions with moderate external pressure or magnetic fields. The vanadium spinels $AV_2O_4$ ($A = \text{Cd, Zn, and Mg}$) are archetypical realizations of highly frustrated spin-orbital systems\textsuperscript{[2–12]}. The magnetic V$^{3+}$ ions reside on a frustrated pyrochlore lattice and contain two $d$ electrons in the three $t_{2g}$ orbitals. These materials exhibit a cubic to tetragonal transition at a temperature $T = T_S$ and the onset of a Q $= 2\pi(0, 0, 1)$ antiferromagnetic (AFM) ordering below $T_N < T_S$ [see Fig. 1(a)]\textsuperscript{[3–6]}. The pyrochlore lattice can be viewed as a collection of cross-linking chains running along the (110) directions. Below $T_N$, the $xy$ ($z \parallel c$-axis) chains exhibit the usual Néel ordering, while chains oriented along $xz$ and $yz$ directions exhibit a $\uparrow\uparrow\uparrow\downarrow\downarrow$ superstructure\textsuperscript{[2–4]}. This ordering induces an electric polarization $P \approx 5\mu C/m^2$ $\hat{z}$ in CdV$_2$O$_4$, that arises from different oxygen displacements along $xz$ and $yz$ bonds\textsuperscript{[11]}, giving opposite contributions to $P$ depending on whether the bond is ferromagnetic (FM) or AFM. Because the magnitude of the displacements is different for FM and AFM bonds, the $\uparrow\uparrow\downarrow\downarrow$ structure induces a net $P \parallel \hat{z}$.

The ground state manifold of these spinels is extensively degenerate if only nearest-neighbor (NN) exchange is included. The $Q = 2\pi(0, 0, 1)$ ordering must then be selected by residual interactions which arise as a sequence of two selection mechanisms. The tetragonal distortion reduces the frustration by increasing the exchange along the $xy$ chains and inducing AFM spin correlations\textsuperscript{[4]}. This distortion is accompanied by long-range ferro-orbital ordering (occupied $xy$ orbitals) and the onset of a magnetic easy $z$-axis. However, the exchange between crossing $xy$ chains remains frustrated. In the Mott limit, relevant for CdV$_2$O$_4$, the $\uparrow\uparrow\downarrow\downarrow$ ordering is stabilized by a weak third NN AFM exchange.\textsuperscript{[7]} For spinels near the Mott transition, like ZnV$_2$O$_4$\textsuperscript{[13–14]}, the $\uparrow\uparrow\downarrow\downarrow$ superstructure could originate from Fermi point nesting of the quasi-1D $yz$ and $xz$ chains\textsuperscript{[13]}.

The weak nature of the interactions that select the magnetic ordering in highly frustrated magnets suggests that moderate fields could induce a different spin ordering. Indeed, multiple magnetic field-induced transitions have been reported in pyrochlore antiferromagnets and spin ice $R_2Ti_2O_7$\textsuperscript{[17–21]}. While most transitions result from the competition between various spin-spin interactions and the Zeeman coupling, the presence of orbital degrees of freedom makes vanadium spinel ideal candidates for studying similar phenomena in frustrated spin-orbital systems. By including the subtle interplay between spin-orbit coupling and lattice distortion, our model predicts a new high-field magnetic order induced by a local trigonal distortion, which was not regarded as an important factor in previous studies of vanadium spinels.

Here we show experimental evidence of a quantum phase transition induced by relatively small fields in MgV$_2$O$_4$ and CdV$_2$O$_4$ spinels, in spite of their large Curie-Weiss temperature $|\Theta_{CW}| \approx 300$ K\textsuperscript{[6]}. For a single-crystal of MgV$_2$O$_4$, we observe a clear jump in the magnetization at $\mu_0H \simeq 40$T,

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{FIG1.png}
\caption{3D orderings for $AV_2O_4$ viewed from [001] at $H = 0$ (a), and predicted to occur at high fields (b). $\bigcirc$ and $\bigotimes$ denote the $+z$ and $-z$ component of the moments. The arrows indicate the transverse spin components induced by the trigonal distortion. $J$ (solid blue) $J^x$ (dashed red) and $J_3$ are NN, next NN and 3rd NN exchange constants. A and B denote tetrahedra with opposite orientations.}
\end{figure}
while the multiferroic ordering of powder CdV$_2$O$_4$ is suppressed for $\mu_0 H \gtrsim 30$ T. We explain this transition with a new selection mechanism based on a trigonal distorsion, which is intrisic to spinel structures and rotates the local easy-axis toward the (111) direction of each V$^{3+}$. This rotation induces a weak FM component perpendicular to the Néel order parameter of each $xy$ chain [see Fig. 1(a)]. While the FM components of different chains cancel out for the low-field $Q = (001)$ structure of Fig. 1(a), the $Q = 0$ spin ordering shown in Fig. 1(b) acquires a net transverse FM component, which makes it energetically favorable for large enough $H$. This new structure is not ferroelectric, which is in agreement with our experiments.

Ref. [16] shows how the electronic structure of $A$M$_2$X$_4$ spinels containing $t_{2g}$ electrons is affected by a varying trigonal field. One component arises from the trigonal distorsion of the oxygen octahedron, as shown in Fig. 2. An opposing effect is caused by the surrounding trigonal anti-prism of $t_{2g}$-based metals (V ions). Both contributions eventually cancel when moving from ZnCr$_2$O$_4$ to HgCr$_2$O$_4$ because the oxygen trigonal field increases while the Cr trigonal field decreases. For CdV$_2$O$_4$ and the structure proposed in [22], a very small tetragonal field is expected below $T_T$ that is accompanied by a larger trigonal distorsion. The structure proposed in [11] (and also in [13] for the related compound ZnV$_2$O$_4$) explains the origin of the additional tetragonal term that further stabilizes the $d_{xy}$ orbital. From ab initio calculations we get $\Delta = 350$ meV and $\delta = 250$ meV for the $t_{2g}$ splittings induced by the tetragonal and trigonal distorsions, respectively [13].

We first present $P(H)$ and $M(H)$ data for CdV$_2$O$_4$ and show that the multiferroic $\uparrow \uparrow \downarrow \downarrow$ ordering of CdV$_2$O$_4$ is suppressed for $\mu_0 H > 30$ T. Similar features in $M(H)$ are observed in single-crystalline MgV$_2$O$_4$, which is too conductive to measure $P(H)$. Polycrystalline samples of CdV$_2$O$_4$ were prepared by solid-state reaction, and the single crystals of MgV$_2$O$_4$ by a travelling floating zone method, as described in the Supplement. Low-field $M(H)$ and $P(H)$ measurements are consistent with previous publications [11] [23].

The high-field data are plotted in Fig. 3 and more details are described in the Supplement. For CdV$_2$O$_4$, $M(H)$ increases linearly until a field between 30 and 40 T and then shows an upturn for $T < T_N$ [see Fig. 3(a)]. The $M(H)$ curve of CdV$_2$O$_4$ reaches 0.4 $\mu_B$/V$^{3+}$ at $\mu_0 H = 65$ T, which is far below the saturated value for V$^{3+}$ $S = 1$ ($2\mu_B$/V$^{3+}$). By linear extrapolation, the saturation value would be reached for $\mu_0 H > 200$ T, consistent with $\Theta_{CW} \approx 300$ K.  

Fig. 3(b) shows $\Delta P(H)$ of CdV$_2$O$_4$ for $H \parallel P$ (similar data for $H \perp P$ are shown in the Supplement). $P(H)$ remains constant up to a field between 20 and 40 T, depending on the temperature, and then begins to decrease. No change in $P$ with $H$ is observed for $T > T_N = 32.5$ K (see 33 K curve). Just below $T_N$ ($T = 29$K), $P(65$ T) - $P(0)$ is close to the value of $\Delta P(T)$ across temperature-induced phase transition at $T_N$ indicating that the 65 T field mostly suppresses ferroelectricity near $T_N$. However, $P(65$ T) - $P(0)$ shrinks for $T \leq 29$ K, indicating that a finite $P$ remains for $H > 65$ T.

Finally, Figs. 3(c) and (d) show $M(H)$ curves of MgV$_2$O$_4$ for $H \parallel [011]$ and $H \parallel [111]$, respectively. MgV$_2$O$_4$ is too electrically conductive to measure electric polarization. Like for CdV$_2$O$_4$, $M(H)$ is linear up to a field between 30 and 40 T and then shows an upturn or a jump. In the Supplement, we describe a magnetic hysteresis observed for $H \parallel [111]$.  

Fig. 4 shows the $H - T$ phase diagram of CdV$_2$O$_4$ and MgV$_2$O$_4$ that results from the measured $M(H)$ and $\Delta P(H)$ curves. Because CdV$_2$O$_4$ is polycrystalline, we find it likely that the observed behaviors of $P(H)$ and $M(H)$ are caused by a single field-induced transition that is broadened by anisotropy, i.e., the beginning of the upturn in $M(H)$ and downturn in $P(H)$ correspond to the transition field for $H$.
parallel to the easy-axis, while the transition field along the hard axis is not reached by 65 T. The phase diagram includes peaks in $d\Delta P/d\mu_0 H$ and the onset of the deviation from linear behavior in $M(H)$ (determined by intersecting the linear behaviors below and above the upturn).

We now introduce a model Hamiltonian that includes the effect of the tetragonal and trigonal distortions:

$$\mathcal{H} = J \sum_{(ij)} S_i \cdot S_j + J' \sum_{(ij)} S_i \cdot S_j - D \sum_i (S_i \cdot \hat{n}_i)^2 + J_3 \sum_{\langle(ij)\rangle} S_i \cdot S_j - g\mu_B \sum_i \mathbf{H} \cdot \mathbf{S}_i. \quad (1)$$

$J$ is the NN AFM exchange that couples the $V^{3+}$ spins along the $xy$ chains and $J'$ is the NN interaction along $xz$ and $yz$. $D$ is the single-ion anisotropy, $\hat{n}_i$ is a unit vector along the easy-axis, $J_3$ is the third NN exchange, $g$ is the gyromagnetic factor, and $\mu_B$ is the Bohr magneton.

As in other vanadium spinels [7,9], $J$ and $J'$ depend on the occupancy of the relevant $d$ orbitals. The occupancy of the $d_{xy}$-orbital, and thus the strength of $J$, becomes dominant after the tetragonal distortion below $T_S$. $J'$ is determined by the orbital ordering of the remaining $d_{xz}$ and $d_{yz}$ orbitals. Two types of orbital states have been proposed for vanadium spinels: staggered real [2] and a uniform complex orbital ordering [8]. Both cases result in $|J'| < J$. Because $J'$ is also geometrically frustrated, the final 3D ordering depends on residual perturbations. Here we consider two competing perturbations: the Zeeman coupling to the external field and a 3rd NN exchange $J_3$. The 3rd NN pairs are separated by twice the NN distance on the same $(110)$ chains [Fig. 1(a)].

The $D$ term originates from the relativistic spin-orbit interaction. We estimate the orientation of the easy axes $\hat{n}_i$ and the anisotropy strength $D$ by diagonalizing the single-ion Hamiltonian $H_{t_{2g}} = \Delta \lambda_b + \delta(\lambda_1 + \lambda_4 + \lambda_6) - \mathbf{AL}' \cdot \mathbf{S}$ in the two $d$-electron basis. $\lambda_b$ are Gell-mann matrices, $L'$ is the effective angular momentum of length $L' = 1$ for the $t_{2g}$ electronic configuration, and $\lambda \approx 20$ meV is the effective spin-orbit coupling constant. The crystal-field splitting estimated from our ab initio calculations ($\Delta \approx 350$ meV and $\delta \approx 250$ meV) leads to $D \approx 15.5$ meV and an easy axis $\hat{n}_i$, which is tilted about $\theta = 35^\circ$ from the $z$ axis toward the local (111) direction.

Specifically, the easy axes at the four sublattices of the pyrochlore are $\hat{n}_m = \cos \theta \hat{z} + \sin \theta \hat{e}_m$, where the in-plane unit vectors $\hat{e}_m$ point along the [110], [111], [110], and [110] directions for $m = 0, 1, 2, 3$, respectively (see Fig. 1). We note that the essential physics discussed below is largely independent of the specific values of these parameters.

We first consider the zero field magnetic order. The frustration of the $J'$ couplings between the crossing $xy$-chains is relieved by $J_3$. For zero trigonal distortion, $\delta = 0$, $J_3$ favors a collinear $\uparrow\uparrow\downarrow\downarrow$ ordering of spins along the $yz$ and $xz$ chains (the 3rd neighbor pairs on the $xy$ chains are parallel to each other due to a dominant $J$), giving rise to the Q = 2$\pi$(0,0,1) 3D ordering shown in Fig. 1(a). To characterize the AFM order in the pyrochlore lattice we introduce two Néel order parameters $L_x = S_0 + S_1 - S_2 - S_3$ and $L_y = S_0 + S_2 - S_1 - S_3$ for a tetrahedron [24], where $S_m$ is the magnetization of sublattice $m$. The corresponding values for the structure shown in Fig. 1(a) are $L_x(\mathbf{r}_A) = 4S \hat{z} e^{iQ \cdot \mathbf{r}_A}$ and $L_y(\mathbf{r}_B) = 4S \hat{z} e^{iQ \cdot \mathbf{r}_B}$, where $\mathbf{r}_A$ and $\mathbf{r}_B$ are coordinates of tetrahedra of type-A and B, respectively. Because $\exp(iQ \cdot \mathbf{r}_{A,B}) = \pm 1$, the sign of the Néel order parameters alternate between successive layers.

The trigonal crystal field $\delta$ rotates the easy axis and gives rise to a net magnetization $M = S_0 + S_1 + S_2 + S_3$ in each tetrahedron. This small FM component is modulated: $M(\mathbf{r}_A) \approx 4S \sin \theta \hat{y} e^{iQ \cdot \mathbf{r}_A}$, $M(\mathbf{r}_B) \approx 4S \sin \theta \hat{x} e^{iQ \cdot \mathbf{r}_B}$, and the net magnetization vanishes: $\langle M(\mathbf{r}) \rangle = 0$. The tetragonal symmetry is preserved by this magnetic order as the system is invariant under a $\pi/2$ rotation in the $xy$ plane ($A \leftrightarrow B$) accompanied by the exchange $L_x \leftrightarrow L_y$. Because the Q = (001) order is selected by $J_3$ out of many degenerate states consisting of decoupled AFM $xy$ chains, the Zeeman coupling to a large enough magnetic field should overwhelm $J_3$ and select the state with finite $M$ that is shown in Fig. 1(b). We suggest that the transition between these two ordered phases explains our measurements. The magnetic order in Fig. 1(b) has a wave vector $Q = 0, i.e.,$ all tetrahedra are in the same magnetic structure. This state is described by magnetic order parameters: $L_x(\mathbf{r}) \approx 4S \cos \theta \hat{z}$, $L_y(\mathbf{r}) = 0$, and $M(\mathbf{r}) = 4S \sin \theta \hat{y}$, for both types of tetrahedra. The tetragonal symmetry is broken in this case and the Zeeman energy gain is $E_Z = 4S g\mu_B \sin \theta \mathbf{H}$ per tetrahedron for a transverse magnetic field $\mathbf{H} = (0, H, 0)$. A first-order quantum phase transition thus takes place at $g\mu_B H_c \approx \text{const} \times J_3 / \sin \theta$ that
removes the polarization by suppressing the $\uparrow\uparrow\downarrow\downarrow$ structure.

We verified the above picture by performing classical Monte Carlo (MC) simulations of $H$ for $J' = 0.16J$, $J_3 = 0.01J$, and $D = 0.7J$. ($S = 1$ spins are approximated by classical unit vectors). We use the standard Metropolis algorithm and periodic boundary conditions for lattices of up to $N_s = 16 \times 8^3$ spins. Fig. 5(a) shows the $M(H)$ curves for $H \parallel \hat{y}$ obtained at different temperatures. A sharp discontinuity at $gH/\mu_B \approx 0.4J$ and $T = 0$ indicates a first-order transition. The discontinuity decreases with increasing temperature and disappears at $T \approx 0.15J$. The resulting $H-T$ phase diagram [Fig. 5(b)] includes the two ordered phases shown in Figs. 1(a) and (b). The transition to the high-$T$ paramagnetic phase is always continuous (the phase boundaries were estimated by the crossing of the Binder’s cumulant). The first-order line between the two ordered phases was determined with the method of mixed initial state (each of the two coexisting orders occupies half of the lattice) [25]. Relating $T_c(H = 0)$ with the experimental value of $T_c \approx 33$ K (Fig. 4), we estimate a transition field $\mu_0 H_c \approx 47$ T, in good agreement with our measurements. The first-order transition is consistent with the magnetic hysteresis observed in MgV$_2$O$_4$ (Supplement).

In summary, we observe a field-induced phase transition marked by a magnetization jump at $H \approx 40$ T in a single-crystal of MgV$_2$O$_4$, and an upturn in the $M(H)$ slope of polycrystalline CdV$_2$O$_4$. We also present a model, which includes the effect of orbital degrees of freedom, lattice distortion, and spin-orbit interactions, and predicts a field-induced $Q = 0$ magnetic order that is stabilized by relatively small magnetic fields in comparison to the dominant exchange. The measured field-induced transition is attributed to the onset of the $Q = 0$ order. Contrary to the $Q = 2\pi(0, 0, 1)$ zero field magnetic ground state, the new field-induced state does not support ferroelectricity, in agreement with the suppression of $P$ that we observe in the multiferroic spinel CdV$_2$O$_4$. This $Q = 0$ state is only possible in the presence of a small trigonal distortion, which has been observed in many vanadium spinels. For example, the trigonal distortion is essential for stabilizing the staggered orbital order of the ferrimagnetic vanadate MnV$_2$O$_4$ [26, 27]. Our theory thus underscores the importance of including the trigonal distortion, that exists in both zero and applied fields, for describing the magnetism of vanadium spinel compounds. In particular, the presence of the trigonal distortion in vanadium spinels indicates that the orbital ordering in the vanadium spinels family is most likely of the staggered type [7], as indeed the case for CdV$_2$O$_4$. Finally, because the $Q = 0$ state also breaks the tetragonal symmetry, a similar phase transition might also be driven by epitaxial strain.

We thank N. Perkins and D. Khomskii for useful discussions and Brian Scott for single crystal orientations. Work at the LANL was performed under the auspices of the U.S. DOE contract No. DE-AC52-06NA25396 through the LDRD program. The NHMFL facility is funded by the NSF through cooperative grant DMR-1157490, by the DOE and by the State of Florida. This material is based upon work supported in part by the NSF under Grant No. PHY-1066293 and the hospitality of the ACP. V.P. acknowledges financial support from the Ramon y Cajal Program.

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