Coupled magnetic and electric hysteresis in the multiferroic double perovskite
\(\text{Lu}_2\text{MnCoO}_6\)

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We investigate a magnetic hysteresis loop with a remanent moment that couples to electric polarization to create coupled hysteretic multiferroic behavior in \(\text{Lu}_2\text{MnCoO}_6\). Measurements of elastic neutron diffraction, muon spin relaxation, and micro-Hall magnetometry demonstrate an unusual mechanism for the magnetic hysteresis – namely the hysteretic evolution of a microscopic magnetic order, and not classic ferromagnetic domain effects. We show how the frustrated spin system evolves from antiferromagnetism with an incommensurate long-wavelength modulation and strong fluctuations towards a net magnetism. We also clarify the different temperature scales for the onset of ordering, dynamics, and hysteresis.

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Magnetic order that induces electric polarization is a focus area of multiferroic research. This cross coupling of magnetism and ferroelectricity creates a multifunctionality that has numerous possible applications in electronics, sensing and electronic memory.\[1\] For applications like data storage, it is desirable to have a net, hysteretic magnetization \(M\) with a switchable remanent magnetic moment that couples to a similarly hysteretic polarization \(P\).\[2\] However, this scenario is rare. Multiferroics generally fall into two categories.\[3\] In type I, the magnetic and electric order parameters are distinct from each other, and only a small fraction of \(M\) and \(P\) couple to each other. In type II multiferroics, \(P\) is entirely induced by magnetic order, so \(M\) and \(P\) are strongly coupled. This requires that the magnetic order breaks spatial-inversion symmetry (SIS), and such magnetic orders tend to be complex structures such as spirals with little net magnetization.\[9\] Thus it is a challenge to create a ferromagnetic-like \(M\) that couples to a ferroelectric \(P\). The observation of this effect in \(\text{Lu}_2\text{MnCoO}_6\) is thus significant.\[12\] In \(\text{Lu}_2\text{MnCoO}_6\), \(M(H)\) is hysteretic with a remanent magnetization after cycling the applied magnetic field. Above the coercive magnetic field, \(P\) is suppressed and remains suppressed until an electric field is applied. While the magnitude and temperatures of the effect are not ready for applications, the unusual phenomenology and underlying physics are of great interest.

\(\text{Lu}_2\text{MnCoO}_6\) forms in the \(\text{A}_2\text{BB’O}_6\) double perovskite structure with a slight monoclinic distortion.\[12\] Elastic neutron scattering measurements identify magnetic order with a net microscopic magnetization \(M = 0\) at \(H = 0\), despite the observed magnetic hysteresis loop that develops in applied \(H\).\[13\] The neutron scattering results indicate that the \(S = 3/2\) Co\(^{2+}\) and Mn\(^{4+}\) spins point along the c-axis, and form a pattern of \(\uparrow\text{Co} \downarrow\text{Mn} \downarrow\text{Co} \uparrow\text{Mn}\), or alternately \(\uparrow\text{Mn} \uparrow\text{Co} \downarrow\text{Mn} \downarrow\text{Co}\) propagating along the c-axis. This type of ordering can result for example from frustration between nearest and next-nearest neighbor spins.\[14\] Although this magnetic order breaks SIS along the c-axis, the electric polarization points along the b-axis.\[12\] The neutron diffraction data also show a long-wavelength modulation consistent with a propagation vector \(\bm{\tau} = (0.0223(8), 0.0098(7), 0.5)\) at \(H = 0\), which could be a mechanism for inducing \(P\).\[12\] How the magnetic order evolves in magnetic field is not clear. Another unresolved issue is why the magnetic hysteresis and electric polarization occur below 30 K while long-range magnetic order has been reported to develop below 45 K.\[13\] It is an open question as to whether the onset of \(P\) corresponds to a change in magnetic order, or reflects a change in the dynamics of the magnetization. Furthermore, a kink in \(M(T)\) curves at a third temperature, 12 K, also remains unexplained.

We explore these questions with elastic neutron scattering and muon spin relaxation (\(\mu\)SR) measurements on polycrystals, and sensitive Hall magnetometry of sintered monocrystalline grains taken from the polycrystals.

We begin with the magnetic field evolution of the magnetic ordering. Elastic neutron scattering results are shown in Fig.\[1\] on the same polycrystals as in Yañez-Vilar \textit{et al.}\[15\] These data were collected on the RITA-II spectrometer at the Paul Scherrer Institute using neutrons with a wavelength \(\lambda_n = 4.217\) Å, for \(H\) up to 12 T and temperatures down to 1.6 K. Fig.\[4\] focuses on...
A netic phase. Both scenarios account for the suppression of electric polarization with $H$: the low-field antiferromagnetic phase breaks SIS and so can induce electric polarization and the suppression or evolution of this phase can reduce $P$. In contrast to the (1 1 0) and IC AFM peaks, the (0 0 2) peak shows no resolvable magnetic field-dependence. This makes sense since (0 0 2) is sensitive to the $c$-axis components of the spins, which are very anisotropic. Finally, sharp jumps can be observed in the IC AFM and (1 1 0) intensities near the 2 T "coercive magnetic field" where $M$ switches sign. Note that the magnetic field range in which magnetic and electric hysteresis occur coincides with the presence of the IC AFM peak.

The neutron diffraction data support an unconventional mechanism for the magnetic hysteresis. The hysteresis in the magnetization matches the hysteresis observed in intensity of the (1 1 0) peak (Fig. 2(b)), with the small difference in the virgin curve due to the residual intensity of the structural Bragg peak. Thus, the hysteresis in the microscopic order accounts for the observed magnetic hysteresis. This is in contrast to conventional ferromagnets where the microscopic order remains essentially unchanged around the magnetic hysteresis loop since the coercive magnetic field only changes the size and configuration of the domains. A conventional ferromagnet thus would not show a significant change in the powder

three indicative peaks. There is a broad scattering peak centered at $2\theta = -70.9^\circ$, which we refer to as incommensurate antiferromagnetic (IC AFM) since it is due purely to the magnetic Bragg peaks $(1 - 1 0) + \tau$, $(-1 1 1) - \tau$, $(1 1 0) + \tau$, and $(-1 - 1 1) - \tau$ arising from the $H = 0$ antiferromagnetic order. There is also a peak centered at $-69.4^\circ$ containing contributions from the (0 0 2) structural Bragg peak as well as the (1 1 1) order. The integrated intensity of the structural Bragg peak is suppressed while the intensity of the (1 1 0) peak grows (Fig. 2). The magnetic and electric hysteresis are also suppressed over the intensity of the (1 1 0) peak (Fig. 2(b)), with the small difference in the virgin curve due to the residual intensity of the structural Bragg peak.

Fig. 2(c-e) shows the magnetic field evolution of the integrated intensity of these peaks, extracted from fits by Gaussian line-shapes. For comparison we also show $M(H)$ and $P(H)$ from Yañez-Vilar et al. in Fig. 2(a), and we compare $|M|$ to the intensity of the (1 1 0) peak in (b). In all three of the neutron diffraction peaks, the center positions and full widths at half maxima do not change with $H$ to within our resolution. However, $H$-dependence can be resolved in the intensity of the IC AFM and the (1 1 0) peak. These peaks have neutron momentum transfers containing components in the $ab$-plane, so they are sensitive to magnetic moments aligned along the $c$-direction. As the magnetic field increases, the intensity of the IC AFM peak is suppressed while the intensity of the (1 1 0) peak grows (Fig. 2). The magnetic and electric hysteresis are also suppressed over the same magnetic field range as the IC AFM. One interpretation of the data is that a single microscopic order evolves from antiferromagnetism towards another phase with a net magnetism, such as "↑↑↑↓↓". Another scenario is that the sample becomes spatially phase-segregated, and the phase with net magnetism occupies more volume with $H$ at the expense of the antiferromagnetic phase. Both scenarios account for the suppression of electric polarization with $H$: the low-field antiferromagnetic phase breaks SIS and so can induce electric polarization and the suppression or evolution of this phase can reduce $P$. In contrast to the (1 1 0) and IC AFM peaks, the (0 0 2) peak shows no resolvable magnetic field-dependence. This makes sense since (0 0 2) is sensitive to the $c$-axis components of the spins, which are very anisotropic. Finally, sharp jumps can be observed in the IC AFM and (1 1 0) intensities near the 2 T "coercive magnetic field" where $M$ switches sign. Note that the magnetic field range in which magnetic and electric hysteresis occur coincides with the presence of the IC AFM peak.

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change in dynamics. Regarding the question of where the electric polarization onsets, we note \( \Delta P \) in Fig. 3(a) indicates electric hysteresis, not electric order. On the other hand, the upturn in the dielectric constant below \( T_N \) in Fig. 3(c) strongly suggests that ferroelectric order is induced at \( T_N \), not \( T_H \).

We explore these temperature scales further with \( \mu \)SR measurements on polycrystals at the Swiss Muon Source using the GPS spectrometer. The samples were packed inside 25 \( \mu \)m Ag foil and mounted on a \( ^4 \)He flow cryostat. In the S.I., we present example \( \mu \)SR asymmetry spectra. These show monotonic relaxation, rather than oscillations, down to the lowest measured temperatures. The exponential time-dependence of the relaxation is consistent with dynamic fluctuations of the electronic spins, and not static disorder. These fluctuations are sufficiently strong that the muons decay before they can precess. Correlation times for the spin fluctuations are likely 10-100 ps. For example, a local magnetic field of 500 mT fluctuating with correlation times on the order of 10 ps would yield a relaxation rate on the order of 2 \( \mu \)s\(^{-1} \), close to what we observe. The data can be described as a single exponential relaxation below \( T_N \), and two relaxations with well-separated time constants above \( T_N \). The data can be fitted (described in detail in the S.I.), to a function of the form \( A(t) = A_1 e^{-\lambda t} + A_2 e^{-\lambda' t} \) and \( A_{bg} \) from \( \mu \)SR measurements, as described in the text and S.I.(f) T-dependence of the relaxation times \( \lambda \) and \( \lambda' \) from \( \mu \)SR data.

neutron diffraction pattern with magnetic field.

We now describe the \( T \)-dependence of the magnetic ordering. For reference we show in Fig. 3, b, and c, the \( T \)-dependent magnetic and electric data previously reported in Yáñez-Vilar et al.\(^{12} \) In Fig. 3(d)-(f) we show new neutron diffraction and \( \mu \)SR data as a function of \( T \). Three temperatures are labeled: the ordering temperature \( T_N \), the onset of magnetic and electric hysteresis at \( T_H \), and a kink in the magnetization at 12 K at \( T_f \). The neutron diffraction data in Fig. 3(d) reveals that ordering onsets below \( T_N = 50 \) K, which is slightly higher than 45 K previously estimated from magnetization data. 50 K corresponds to a kink in \( M(T) \) in (a); to the midpoint of the jump in \( \chi_{ac}(T) \) in (b); and finally to an upturn in the dielectric constant in Fig. 3(c). An important conclusion that we can draw from the neutron diffraction data is that there is only one magnetic ordering temperature and no changes in the microscopic magnetic order can be resolved at \( T_H \) or \( T_f \). Other magnetic Bragg peaks exhibit a similar \( T \)-dependence as the IC AFM peak, and are shown in the Supplementary Information (S.I.).\(^{12} \)

Below a lower temperature \( T_H \sim 30 \) K, we observe the onset of magnetic and electric hysteresis. This temperature corresponds to the peak in the dielectric constant, and a small frequency dependence in the ac susceptibility, which is indicative the magnetic dynamics. As mentioned previously, the neutron diffraction data does not resolve a change in magnetic order. So, \( T_H \) is purely a

FIG. 3. \( T \)-dependence of different properties, showing three temperature scales: \( T_N, T_H, \) and \( T_f \). a) shows \( M(T) \) after zero-field cooling (ZFC) and field-cooling (FC) in \( H = 1 \) kOe, as well as \( \Delta \chi = \chi(0 \text{~T}) - \chi(15 \text{~T}) \). b) ac magnetic susceptibility at 10 Hz, 100 Hz and 1 kHz, c) dielectric constant \( \varepsilon_r \) measured for \( E \parallel H \) at 0 and 14 T at 10 kHz d) \( T \)-dependence of the IC AFM peak for \( H = 0 \). e) \( T \)-dependence of \( A_\lambda \) and \( A_{bg} \) from \( \mu \)SR measurements, as described in the text and S.I.(f) T-dependence of the relaxation times \( \lambda \) and \( \lambda' \) from \( \mu \)SR data.
only for $H_{||}$—a sharp jump in $M$ at a coercive field of 2 T, consistent with the studies of Yáñez-Vilar et al., where sudden switching has been observed at 2 T for $T \leq 2$ K. We note that the single crystals show a larger $H_c$ of 5 T at 5 K compared to 2 T in the monoclinic and polycrystalline samples. Finally we show that our coercive field for $H_{\perp}$ peaks at $T_f \approx 12$ K (see Fig. 4 b)). This is the same temperature for which a freezing out of magnetic fluctuations is deduced from $\mu$SR.

Micro-Hall measurements have previously been used to track Barkhausen jumps in micron-sized magnetic particles due to pinning/depinning of magnetic domain walls. No indications for Barkhausen jumps in the magnetization for Lu$_2$MnCoO$_6$ corroborate the notion of a magnetization reversal mechanism that is not due to magnetic domain pinning effects on the nm scale.

Except for the just-mentioned differences in $H_c$, our polycrystalline data is similar to the $M$, $P$, and $c$ reported for single crystals, showing similar $T$ and $H$ evolution and hysteresis. A notable difference however is that in the single crystals, $P$ and $c$ are only sensitive to $H \perp E$ ($E \parallel b$ and $H \parallel c$). The polycrystals on the other hand, show $P$ and $c$ that can be suppressed by both $H \perp E$ and $H \parallel E$. One explanation for this discrepancy is that the differently-oriented grains of the polycrystal can couple to each other via their magnetic dipole moments, as can occur in ferromagnetic polycrystals. Thus the magnetic properties of the polycrystal will not simply be an average of the different directions of a single crystal.

We can now create a picture of the magnetic and electric behavior of Lu$_2$MnCoO$_6$. We resolve the nature of the three temperature scales: long-range magnetic and electric order occurs below a single temperature $T_N = 50$ K; kHz dynamics and hysteresis in $M$ and $P$ onset below $T_H \sim 30$ K and finally strong GHZ spin fluctuations begin to freeze below $T_f = 12$ K. We see no additional magnetic phase transitions as a function of $T$ below $T_N = 50$ K. The magnetic order was previously shown to be consistent with an “↑↑↓↓” spin configuration along the $c$-axis chains, subject to an incommensurate modulation propagating in the a-b-plane. As a function of $H$, we find that the magnetic order does evolve, and does so in a hysteretic fashion as evidenced from neutron diffraction. The evolving magnetic order creates and destroys $P$. This explains the origin of the coupling between an apparent ferromagnetic hysteresis loop and electric polarization. It is quite unusual for a magnetic field-induced phase transition to be so hysteretic as to create a remanent magnetization. In most cases when field-induced magnetic transitions are hysteretic, there is only a relatively small difference between the transition field on up and down sweeps of the magnetic field, and no remanent magnetization. The origin of hysteresis in typical ferromagnets on the other hand, is domain evolution and not changes in the microscopic magnetic order. Two classes of compounds that do show magnetic hysteresis with evolving magnetic order are 1) certain frustrated chain compounds with Ising spins, similar to Lu$_2$MnCoO$_6$, and 2) martensitic magnetic materials. Ca$_3$Co$_2$O$_6$ is a frustrated spin chain material where magnetic hysteresis is accompanied by hysteretic microscopic magnetic order. Like Lu$_2$MnCoO$_6$ it shows long wave-length incommensurability and slow dynamics and its frustrated magnetism has been analyzed in terms of the Axial Next-Nearest Neighbor Ising (ANNNI) model. Its isostructural family members including Ca$_3$CoMnO$_6$, Ca$_3$CoRhO$_6$, Sr$_3$CoIrO$_6$, and Sr$_3$NiIrO$_6$ also show magnetic hysteresis in Ising spin chains, though the origin of the hysteresis has not been explored. Of these materials, Ca$_3$CoMnO$_6$ is a multiferroic with qualitatively similar hysteretic behavior to Lu$_2$MnCoO$_6$ including $↑↑↓↓$ magnetic order along c-axis chains and $H$-suppressed $P$. A very important difference however is that in Ca$_3$CoMnO$_6$, $P$ points along the propagation of $↑↑↓↓$ and not perpendicular to it as in Lu$_2$MnCoO$_6$, suggesting a different origin for $P$. Large magnetic hysteresis loops associated with the evolution of magnetic order also occur in martensitic magnetic materials like the colossal magnetoresistant manganites (see e.g., Sr$_3$CoIrO$_6$). Here spontaneous phase segregation into domains of different magnetic order occurs due to an interplay between spin, charge and lattice strain. Our data could certainly be consistent with magnetic phase segregation. At $H = 0$ phase segregation has also been suggested in the double-perovskite Sr$_2$FeOsO$_6$. Thus, we suggest two possibilities for further exploration: 1) the magnetic hysteresis could be an unusual and intriguing consequence of very slow dynamics and pinning in strong frustrated chain systems or 2) the frustration could go one step further and phase-segregate the magnetic or...
order into different magnetic domains that occupy different fractions of the sample volume as a function of magnetic field.

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