 Fundamental aspects of conduction in charged ErMnO$_3$ domain walls


Published in: Advanced Electronic Materials

Document Version: Publisher's PDF, also known as Version of record

Queen's University Belfast - Research Portal: Link to publication record in Queen's University Belfast Research Portal

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Download date:30. Apr. 2024
It is now well-established that ferroelectric domain walls, at which there are discontinuities in polarization, are usually electrically conducting. Yet, there is a dearth of rather basic information on the physics underpinning conductivity. Here, Kelvin Probe Force Microscopy (KPFM)-based experiments are reported, which allow significant new insights regarding charge transport at domain walls in ErMnO$_3$. In one set of experiments, KPFM is used to spatially map the Hall potential, developed at the surface of polished single crystals. These maps provide direct experimental evidence that n-type head-to-head domain walls arise in otherwise p-type material. In another set of experiments, the geometry for current flow is restricted, by cutting sub-micron thick lamellar slices of ErMnO$_3$ (using a Focused Ion Beam microscope). Separate contacts are made to n and p-type walls and the potential profiles, when driving source-drain currents, are measured (again using KPFM). Current-electric field functions showed Ohmic behaviour for p-type walls, with an intrinsic room temperature conductivity value of $\approx 0.4$ Sm$^{-1}$. The n-type walls showed non-Ohmic behaviour and a significantly lower conductivity, supporting the prediction that electrons are in a polaronic state; an upper bound for the room wall conduction influencing datasets, or noted surprisingly high currents (beyond transient displacement currents) developing during switching. Despite these reports, it was not until 2009, when Seidel et al. published...
conducting Atomic Force Microscopy (cAFM) images on 
BiFeO$_3$,[5] that the notion of enhanced electrical transport in 
ferroelectric domain walls became widely accepted. Since then, wall 
conduction has been found across a variety of other systems, 
Cu$_4$B$_7$O$_{13}$Cl$_2$ [14,15] (Ca,Sr)$_3$Ti$_2$O$_7$ [16] and a number of different 
hexagonal manganite materials.[17–19]

Even though 2D conducting systems are now commonplace, 
domain walls are exceptional; they can be moved around, within 
the insulating ferroelectric matrix, as domains expand and con-
tract in response to externally applied fields.[20–24] They may be 
created and destroyed, during domain nucleation and co-
alescence. They are therefore strongly confined electrical con-
ductants that are both mobile and ephemeral in nature.[25] New 
demonstrator devices, that make use of these exciting prop-
erties of conducting domain walls, have been widely reported 
and this is still a rich area for ongoing research.[26–33] How-
ever, experimental elucidation of the fundamental nature and 
origin of domain wall conduction has been slow to emerge. Ex-
perimental insights into carrier properties (types, densities, 
mobilities and masses), local band structures and transport 
mechanisms have been undertaken[19,34–37] but the general pic-
ture is still rather poorly developed. Only a few magnetotrans-
port measurements have been performed[35–39] and, notwithstanding a few notable exceptions,[40,41] simple current-voltage 
measurements have usually involved two-point measure-
ments,[6,42–44] where interfacial barrier resistances can often obscure the 
intrinsinc behaviour of the domain wall itself. Recent work by 
Sharma et al.,[45] is, however, noteworthy: although measure-
ments were still two-probe, treating domain walls as compo-
nents in a transmission line, between source and drain ele-
ctrodes, allowed the confounding effects of contact resistance 
to be overcome and quantitative statements on BiFeO$_3$ domain wall 
conductivity to be made (found to be $\approx 3 \times 10^{-3}$ S m$^{-1}$ at room 
temperature).

Herein, we present and discuss results from two sets of exper-
iments, on conducting domain walls in single crystal ErMnO$_3$, 
that were designed to generate reliable information on funda-
mental aspects of transport. Previous work had established that, in 
ErMnO$_3$, tail-to-tail (T-T) wall types consistently conduct more 
strongly than bulk. In contrast, head-to-head (H-H) walls have 
a more complex response, transitioning from insulating to con-
ducting only when sufficiently large bias fields are applied.[18]

Our first set of experiments revealed that, under the same ori-
entations of electric and magnetic fields, walls supporting dif-
f erent senses of polar discontinuity generate opposite signs of 
Hall voltages at the crystal surface; since it has already been es-
blished that conduction, along T-T walls, is mediated by p-type 
carriers, the opposite sign in Hall voltage, found at H-H walls in 
our work, means that here carriers must be predominantly n-type.

Domain walls in hexagonal manganites meander in complex 
networks and so it is extremely difficult, in a bulk three di-
cisional crystal, to discern the current pathway between two 
points on the crystal surface. Recent work addressed this is-
sue, evaluating contributions from domain walls in near-surface 
regions.[2] Here, in our second set of experiments, we take a 
complementary approach; we spatially confine the current within 
a sub-micron-thick lamella, such that the geometry for cur-
rent propagation is well-defined. Thin film contact pads allowed 
separate source and drain electrodes to be deposited on both n-
type H-H and p-type T-T walls. Source-drain currents were 
driven along each wall type and the associated potential distri-
bution was mapped using KPFM. The specific potential drops 
along the two kinds of walls were monitored, as a function of 
keeping all aspects of the experiment the same, save for a reversal 
in the orientation of the magnetic field. By subtracting the mea-
sured potentials at each pixel, in the in-situ KPFM images taken 
at +B and -B, and dividing by 2, clear Hall voltage maps could be 
generated.

Figure 1 shows a number of meandering line features, in the 
background-corrected Hall potential map, which are mor-
phologically reminiscent of domain wall traces in the hexagonal 
manganides. Indeed, they can be easily correlated with the loci 
of domain walls, revealed by Piezoresponse Force Microscopy 
(PFM), in the same region (Figure 1). Inspection of the images 
and the superposition of the PFM and Hall voltage maps (Figure 1f) show that negative Hall voltages occur at T-T sections 
of domain walls, while positive Hall voltages are seen at H-H 
sections. In our previous work, it was established that conduc-
tion at T-T walls was mediated by p-type carriers.[35] Here, the 
positive sign of the measured Hall voltages in H-H walls, seen in 
Figure 1, clearly indicates that the conducting H-H domain walls 
must therefore be n-type. While it has long been theoretically pre-
dicted that carrier accumulation, or the formation of an inversion 
layer, in charged conducting ferroelectric domain walls, facilitate 
screening and are hence of opposite sign to that of the bound 
charge, explicit experimental evidence for this is rare.[19,35,44] 
The implications of our Hall mapping experiments therefore use-
fully add to the state-of-the-art. Parenthetically, p-n junctions are 
clearly evident, at the points at which domain walls intersect 
(referred to as “vortices” or “vertices” in the hexagonal man-
ganite literature); such p-n junctions are of significant current 
interest.[25,47,48]
Figure 1. Kelvin Probe Force Microscopy (KPFM) measurement of the electrostatic potential developed on a polished ErMnO$_3$ single crystal surface. In a), potentials result from the application of a perpendicular electric (E) and magnetic (B) field ($\approx 12$ kV m$^{-1}$ and $300$ mT respectively), with both oriented parallel to the imaged surface. Subtracting two such potential maps, at constant E field but with the B field orientation reversed, generated images of the spatial variation in the Hall potential b). The potential variations along specific scans (marked by blue and green dashed lines and arrows in a) and (b)) are presented in c,d). Note that, both in the Hall voltage image (b) and in the line trace (d), distinct features with both positive and negative Hall voltages can be seen (highlighted by yellow and cyan lollipop markers). Piezoresponse Force Microscopy (PFM) imaging of the same region e) shows that the positive and negative Hall voltages in (b) map well to domain walls that are distinctly head-to-head (H-H) and tail-to-tail (T-T) in nature. In panel f), PFM and Hall voltage images are superposed (making one image semi-transparent and placing it on top of the other) to emphasise that negative Hall voltages (dark contrast) correlate with T-T walls, while positive Hall voltages (light contrast) correlate with H-H walls. The change in the sign of the Hall voltage indicates that T-T walls are n-type and H-H walls are p-type. Scale bars marked in (b) and (d) represent $5 \mu$m.

3. Current-Field Measurements along Conducting Domain Walls

Lamellar slices of ErMnO$_3$ ($\approx 800$ nm in thickness and $\approx 5 \times 10^5$ m$^2$ in surface area) were machined from the single crystal, using a Focused Ion Beam (FIB) microscope. They were cut perpendicular to the polished surface of the bulk crystal (associated with the Hall maps discussed above) and parallel to the uniaxial polar axis of the ferroelectric. The final stages of lamellar processing used relatively low gallium ion energies and fluxes (typically at $5$ kV and $10$ pA), to minimise surface amorphaisation and ion implantation to a few unit cells in thickness.

Once cut free from the host crystal, lamellae were transferred onto electrically insulating single crystal alumina (“sapphire”) substrates and held in place with small electron-deposited thin film platinum tags (using local electron-assisted chemical vapour deposition, made possible in the dual-beam FIB microscope). Without further processing, clear PFM domain images could not be obtained (even a thin amorphous layer prevented strong PFM contrast). Gentle surface milling, removing the topmost several nanometres of material, using the scanning probe tip and modest pressure, was needed. Only then could microstructures be clearly imaged (Figure 2a,b). Thin film meso-scale gold contact pads were sputter-deposited onto the alumina, through

Figure 2. Integrating lamellae into circuits to allow source-drain contact to individual domain walls. Lamellae ($\approx 800$ nm in thickness) were cut, using a Focused Ion Beam (FIB), from the ErMnO$_3$ single crystals, such that lamellar surfaces were parallel to the polar axis. After placing on (001) single crystal Al$_2$O$_3$ substrates, lamellar top surfaces were gently milled by scanning the tip with modest force, in contact mode. Lateral Piezoresponse Force Microscopy (PFM) amplitude a) and phase b) mapping revealed, in one case, a microstructure with both head-to-head (H-H) and tail-to-tail (T-T) domain walls (the orientation of the cantilever is given by the tip schematic below the PFM maps and the scale bar represents $5\mu$m). A schematic of the lamella, placed onto the Al$_2$O$_3$ substrate, and the geometry used to make electrical contact with the domain walls is presented in c). PFM phase image d) after the successful addition of local electrodes, using electron beam-induced chemical vapour deposition (scale bar represents $5\mu$m).
Figure 3. Potential mapping along domain walls using Kelvin Probe Force Microscopy (KPFM). Source-drain currents were driven between the surface deposited electrodes and the associated potentials were mapped using KPFM on and around both tail-to-tail a,b) and head-to-head c,d) domain walls. The voltage on the electrodes was reversed in maps (a) and (b) and in maps (c) and (d). The fine dark lines in the panel images are equipotential contours with regions between contour lines block-coloured. The scale bar underneath panel (b) represents 2 μm. All maps presented are at the same scale.

In this geometry, bias could be applied along each domain wall separately. In general, a current control mode on the power source was used. Currents were varied, between −10 and +10pA, and the potential distributions along the walls were mapped, again using KPFM. Figure 3 illustrates the kinds of maps obtained, both for a T-T wall (a,b) and a H-H wall (c,d). All KPFM maps, at all applied biases, for these walls are given in Supporting Information. The potential along the wall loci was extracted and potential gradients were determined (Figure 4a); hence electric field-current functions were developed (Figure 4b). Because electric fields were evaluated using potential information safely away from the electrode-domain wall contact regions, the influences of

Figure 4. Potential, electric field and currents along ErMnO$_3$ domain walls. Measured potential as a function of position along the tail-to-tail (T-T) domain wall when driving 7pA of current (a), top panel), along with the derived local electric field (a), bottom panel). Field enhancements close to the electrode-domain wall contacts are clearly evident, but these regions were ignored, when determining the field needed to drive the current along the wall itself. With this information, source-drain current may be given as a function of the electric field developed along the domain walls and in surrounding domains b). Lines with gradients of 1 and 3 in the ln-ln plots indicate Ohmic and non-Ohmic transport behaviour associated with the T-T and H-H walls respectively. The units for current and field, from which the natural logarithms were determined, were standard SI units.
barrier resistance were obviated. The electric-field-current functions derived are hence equivalent to those that might be obtained through a conventional four-probe methodology, for example. A similar KPFM-based approach was employed in Ref.[40] for spatially resolved mapping of nanowire resistivity and characterisation of the contact resistivities. As can be seen in Figure 4b, field (E)-current (I) data for both H-H and T-T walls adhere reasonably to power law relations. In the T-T case, the exponent is unity, within error, indicating Ohmic behaviour (Eσ0I), as might be expected in a four-probe investigation of a metal or semiconductor in a relatively low-field regime. For the H-H case, the exponent is significantly larger (≈3), indicating a strongly non-Ohmic response. In terms of interpreting such non-Ohmic behaviour according to standard models, the elevated exponent could indicate a form of space-charge-limited conduction (SCLC), but a great deal of information would be needed to establish this categorically and extract carrier and defect-related information.[50] Conventional Poole-Frenkel field-induced de-trapping of carriers, as previously discussed based on 2-probe, measurements for T-T walls,[51] can probably be discounted for the H-H walls: analysis of ln(−I) as a function of √E generates a strongly scattered function (see Supporting Information) with the R² goodness of linear fit parameter of only ≈0.35. Equally, transport models involving overcoming (or tunnelling through) interfacial energy barriers are not pertinent, as the KPFM mapping used obviates the influence of contacts on the extracted transport current-voltage characteristics.

In analysing the data further, we need to acknowledge that room temperature conduction occurs through both domain walls and domains in the ErMnO₃ system, since the inherent band gap in ErMnO₃ is relatively modest (between 1.3 and 1.6 eV).[52,53] Moreover, conducting Atomic Force Microscopy (cAFM) measurements[54] shows less than an order of magnitude difference in currents developed by tip contact to domain walls and domains. However, due to the finite width of the tip, current spreading, and extrinsic effects (e.g., contact resistance), such measured values usually underestimate the domain wall conductance and it has been theoretically estimated to be ≈10² times larger than in the domains.[56] Nevertheless, an equivalent circuit, in which domains and domain walls act in parallel, is appropriate to confidently develop quantitative wall conductivity information.

The overall conductance (G_total) associated with the domains (D) and the domain wall (DW) has therefore been modelled as follows:

\[
G_{\text{total}} = \frac{I}{V} \approx \frac{I}{EL} = \frac{A_D \sigma_D}{L} + \frac{A_{DW} \sigma_{DW}}{L}
\]  

(1)

where I is the measured source-drain current, E is the measured electric field (potential gradient taken from KPFM data) that drives the source-drain current, and L is the distance between the electrodes. \(A_D\) is the cross-sectional area of the domains through which current propagates, and \(A_{DW}\) is the cross-sectional area of the conducting domain wall; \(\sigma_D\) and \(\sigma_{DW}\) are the conductivities of the domains and domain wall respectively. Note that \(A_D\) and \(A_{DW}\) are limited by the finite dimensions of the ErMnO₃ lamella.

4. Extracting Conductivity Information

Using this equivalent circuit, we can develop several fundamental insights. We can, for example, generate a useful upper bound value for the conductivity of the domains. Consider Equation (1), specifically for the current \(I_{H-H}\)-field (E) information associated with electrical contacts to the H-H wall (the more resistive of the datasets obtained):

\[
\frac{I_{H-H}}{E} = A_D \sigma_D + A_{DW} \sigma_{H-H}
\]  

(2)

where \(\sigma_{H-H}\) is the H-H wall conductivity. Note that \(I_{H-H}\) is the total current developed between the source and drain, not just the current component along the domain wall. The subscript H-H simply indicates that the electrode contacts are those made with the H-H wall, as opposed to the T-T one.

Neither of the right-hand terms in this expression can be negative and hence both must be \(\leq \frac{I_{H-H}}{E}\). The upper bound for the bulk conductivity of domains \(\sigma_D\)(max) can hence be obtained by assuming the domain walls to be perfectly insulating, such that:

\[
\sigma_D(\text{max}) = \frac{I_{H-H}}{EA_D}
\]  

(3)

We note that our in-operando KPFM imaging suggests that the field levels in domain walls and significant regions of adjacent domains are comparable (by inspection of the potential distributions in Figure 3). The effective cross-sectional area for current propagation between source and drain through domains should therefore be close to the order of the lamellar width x lamellar thickness (5µm × 800 nm = 4 × 10⁻¹² m²).

From our data, the interpolated current developed at a field of \(E = 1 \times 10^5\) V - m⁻¹ is \(\approx 2.5\) pA; substituting these values into Equation (3), as well as an effective domain cross-sectional area of \(4 \times 10⁻¹²\) m², gives:

\[
\sigma_D(\text{max}) \approx 6 \times 10⁻⁶\ \text{Sm}^{-¹}
\]  

(4)

which is the same order of magnitude as the bulk DC conductivity at 300K, measured by Ruff et al.[51] (≈3×10⁻⁶ Sm⁻¹). This is reassuring, as the ErMnO₃ crystals used for the Ruff et al. study are from the same batch as have been used in our work.

By also expressing the equivalent of Equation (2) for the T-T wall and subtracting one equation from the other, we can see that:

\[
\frac{1}{EA_{DW}} (I_{T-T} - I_{H-H}) = \sigma_{T-T} - \sigma_{H-H}
\]  

(5)

where \(I_{T-T}\) is the total current developed from the source to the drain with a field (E) dropped along the T-T wall (as well as adjacent domains). We note that Equation (5) is independent of any estimate for domain conductivity or current-carrying domain cross-sectional area. Assuming a domain wall thickness of 1 nm[46] and cross-sectional area 1×800 nm², our measurements (Figure 4a) give the following, at a field value of \(E = 3 \times 10^4\) V m⁻¹:

\[
\sigma_{T-T} - \sigma_{H-H} = 0.4\ \text{Sm}^{-¹}
\]  

(6)
Of course, this domain wall cross-sectional area is only appropriate if the wall runs perpendicular to the lamellar surface. It hence represents a lower bounding estimate, as the wall inclination angle and extent of meandering have not been explicitly established. Nevertheless, the lateral confinement of the lamella and the distances from the surface traces of the domain walls to the lamellar edges (the order of a few microns) suggest that the lower bound estimate should be well within an order of magnitude of the actual value (and more probably within a factor of 3).

By using Ruf et al.'s bulk conductivity as indicative of that associated with the domains, and assuming the same wall cross-sectional areas as above, we can also explicitly determine the conductivities of both types of domain walls (see Supporting Information). Using currents associated with fields of $E = 3 \times 10^3$ V m$^{-1}$ (for the T-T wall), we find:

$$\sigma_{T-T} = 0.4 \text{ Sm}^{-1}$$

(7)

Such a room temperature conductivity is reasonably comparable with the intrinsic conductivity of pure Ge ($\approx 1 \text{ Sm}^{-1}$) and is somewhat higher than that deduced for BiFeO$_3$ domain walls.$^{[45]}$ It is certainly significantly greater than the values discussed in previous studies (e.g., $\approx 1 \times 10^3$ in ref.[46]): here, we obtain the ratio $\sigma_{T-T}/\sigma_{H}$ to be $\approx 1 \times 10^{-3}$. Turner et al.$^{[18]}$ determined the p-type mobility in T-T walls to be $\approx 670$ cm$^2$V$^{-1}$s$^{-1}$ and made a crude estimate for the carrier density of $\approx 1 \times 10^{23}$ cm$^{-3}$. These parameters give $\sigma_{T-T} \approx 0.1 \text{ Sm}^{-1}$, although it should be noted that a domain wall width of $\approx 10$ nm had been assumed in determining the carrier density value in this work (an assumption of 1 nm would increase the implied conductivity by an order of magnitude). Nevertheless, consistency within an order of magnitude is apparent and this lends confidence to the result.

The equivalent treatment for determining the H-H wall conductivity yields a much smaller value, which we note is particularly sensitive to the assumed ErMnO$_3$ bulk conductivity (as this approaches $6 \times 10^{-6}$ Sm$^{-1}$, $\sigma_{H-H}$ progressively decreases toward zero). Such low conductivity is consistent with previous work, when the applied electric fields were relatively modest.$^{[18]}$ Importantly, our measurements indicate that any transition from strongly resistive to strongly conductive behaviour in these walls is not related to overcoming barrier effects at the electrode-wall contacts, as their influence is obviated by the methodology used. Thus, our observations are consistent with the theoretical prediction that electrons at H-H walls are in localized polaronic states, contributing to conduction only at higher electric fields.$^{[18]}

5. Summary and Conclusion

In summary, we have used in situ Kelvin Probe Force Microscopy (KPFM) to measure the Hall potential and the potential gradients (electric fields) needed to drive specific currents in both tail-to-tail (T-T) and head-to-head (H-H) ErMnO$_3$ domain walls. These measurements reveal that the carriers in H-H walls are of opposite charge to those in T-T walls and are hence n-type (as previous work had identified the carriers in T-T walls as p-type). They also allow: the determination of the difference in room temperature direct current conductivity between H-H and T-T walls, robust estimates for the conductivity of T-T walls, and less well-defined estimates for the conductivity of H-H walls in their low-field high-resistance state. Reassuringly, the upper-bound room-temperature value for the conductivity of ErMnO$_3$ domains, deduced from our data, is very much in line with previous impedance spectroscopy results on bulk crystals. The insights developed represent the most definitive carrier and room temperature conductivity information yet obtained for domain walls in this hexagonal manganite system.

6. Experimental Section

Kelvin Probe Force Microscopy (KPFM): Currents driven across the sample were supplied using an external Keithley 237 source-measure unit. Standard Pt/Ir-coated Si probes (Nanosensors, PPP-EFM with free resonance of $\approx 70$ kHz) were used to measure surface potential, using KPFM mode on the MFP-Infinity atomic force microscope (Asylum Research, Oxford Instruments). The interelectrode region was systematically mapped using KPFM, while driving different currents at a typical lift height of 20 nm. To ensure accuracy in the quantitative information obtained from the KPFM, the Pt-coated tip was calibrated, using a standard gold film (or a Au–Al Budget Sensors KPFM and EFMsample), to determine the work function of the tip, before making potential measurements. Upon calibration, stepped voltages were applied to a gold electrode, while measuring the surface potential via KPFM and values were found to be in agreement. Lateral potential gradients were applied to interelectrode gaps and also measured accurately using KPFM.

P FM Mapping: PFM measurements were carried out using the MFP-3D Infinity system with Pt/Ir-coated Si cantilevers showing contact resonance of $\approx 350$ kHz (stiffness $\approx 2.8$ N m$^{-1}$). A typical AC voltage of 3 V was applied, near the contact resonance, to obtain domain images.

Hall Voltage Mapping at Domain Walls: Hall voltage mapping was carried out on the MFP-3D Infinity system using the variable field module 2, capable of applying up to 0.8 T in the plane of the sample. KPFM mapping was undertaken with the application of opposite magnetic fields to extract Hall voltages developed at the walls (discussed in detail in Section 2 above).

Focused Ion Beam Lamellar Preparation: Cross-sectional lamellae were prepared using a Tescan-focused ion beam (FIB)-secondary electron microscope (SEM) Lyra 3. FIB milling was performed at accelerating voltages of 30 kV (rough milling), 5, 2, and 1 kV (fine polishing). Local Pt electrodes were deposited onto the lamellae using electron beam (operated at 5 kV)-induced chemical vapour deposition.

Supporting Information

Supporting information is available on the Wiley Online Library or from the author.

Acknowledgements

The authors are grateful for funding support from the Engineering and Physical Sciences Research Council (EPSRC) through grant number EP/P02453X/1, through studentship funding (partly associated with a Centre for Doctoral Training (EP/L015323/1)) and through the UKRI Future Leaders Fellowship programme (MR/T043172/1). D.M. acknowledges funding from the European Research Council (ERC) under the European Union’s Horizon 2020 research and innovation program (Grant Agreement No. 863691).

Conflict of Interest

The authors declare no conflict of interest.
Author Contributions
J. McCo. and P. W. T. performed most of the experimental work, with help from J. McCo., C. C. and K. H. C. C., R. G. P. McQ., A. K. and J. M. G. supervised the research. The experiments were conceived by J. M. G. The manuscript was primarily prepared and written by J. McCo. and J. M. G., who were also primarily responsible for the data analysis and interpretation. All authors contributed to project discussions and in making refinements to the data analysis, interpretation and presentation. J. McCo., C. C., K. H. C. C., R. G. P. McQ., A. K., D. M. and J. M. G. were all involved in manuscript editing.

Data Availability Statement
The data that support the findings of this study are available from the corresponding author upon reasonable request.

Received: February 8, 2024
Revised: March 28, 2024
Published online:

Keywords
charge transport, conductivity, ferroelectric domain wall, Kelvin Probe Force Microscopy


