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Abstract

We review our work on continuous Ni films coupled via strain to ferroelectric substrates of $BaTiO_3$ (BTO) and $0.68Pb(Mg_{1/3}Nb_{2/3})O_3–0.32PbTiO_3$ (PMN-PT). We show that magnetic force microscopy (MFM) and photoemission electron microscopy (PEEM) of the Ni films (during or after electrical treatment) permit to reveal nanoscale converse magnetoelectric effects (CMEs) that are novel and elude macroscopic measurements. As examples, we discuss magnetization reversal without applied field in multilayer capacitors (MLCs), shear-strain-mediated CMEs in thin Ni films on PMN-PT and reversible switching of perpendicular magnetization from out-of-plane to in-plane in Ni films on BTO. In this latter case, we show that PEEM can be used to measure both magnetic and ferroelectric domains, thus providing key mechanistic insight in the magnetoelectric coupling mechanism.

1. Introduction

Over the last two decades, the magnetoelectric effect [1] has evolved from being a fascinating but *niche* activity, to a mainstream hot topic of research [2-4]. This revival has been driven by the discovery of sizeable converse magnetoelectric effects (CMEs) in new magnetoelectric multiferroic materials that are promising for low-power magnetic memories.

In single-phase multiferroics that show intrinsic magnetoelectric coupling between electrical and magnetic long-range orders, CMEs permit voltage control of magnetism [5-8], but the outcomes are generally weak at room temperature. Large CMEs at room temperature can be found in composite multiferroics comprising ferromagnetic films that are coupled either *via* exchange-bias [9-12] or strain [13-17] with an underlying ferroelectric (exchange-bias requires the ferroelectric to be antiferromagnetic, e.g. $BiFeO_3$). Reports of CMEs in these systems include switching of in-plane magnetic domains [18], modification of a large in-plane magnetization [12, 19], reorientation of magnetic easy axes [20, 21], coercivity modulation [22], and control of magnetic domain wall motion [23]. More recent studies involved composite multiferroics with patterned ferromagnetic films, where voltage control of magnetic single domains [24], onion states [25] and vortex states [26-28] has been demonstrated.

The technological appeal of composite multiferroics for low-power magnetoelectric (electric-write/magnetic-read) memories is long-known [29-31], but still flourishing, as shown by the recent proposal by *Intel* for a magnetoelectric, spin–orbit coupled logic permitting a transformative energy performance of 1 attojoule per bit [4].

Here we review our work on composite multiferroics where ferromagnetic (and thus magnetostrictive) Ni films are coupled via strain to ferroelectric (and thus piezolectric) substrates of $BaTiO_3$ (BTO) and $0.68Pb(Mg_{1/3}Nb_{2/3})O_3$–$0.32PbTiO_3$ (PMN-PT). We will discuss electrically driven magnetization reversal in multilayer capacitors (MLCs) [32], sub-90° rotation of the easy-axis of Ni films due to shear strain from PMN-PT [33] and electrical control of perpendicular magnetisation in Ni films grown on BTO [13]. These nanoscale CMEs eluded macroscopic measurements and were revealed by using either Magnetic Force Microscopy or PEEM with contrast from x-ray magnetic circular dichroism (XMCD).

Experimental Methods

All the MFM work presented here was performed at room temperature using a Dimension 3100 either in varying applied electric fields or at electrical remanence. MFM scans were performed at lift heights of 40–60 nm using low-moment ASYMFMLM Asylum Research tips of stiffness 2 N m^{-1}, coated with 15 nm of CoCr. To unambiguously identify magnetic contrast, the MFM tip-field was reversed by placing the tip and holder in $\mu_0 H = 1$ T along the tip axis. The MFM tip and the metal-coated surface under study were grounded to minimize noise, and to avoid the possible influence of electric-field gradients when measuring in applied electric field.

All the PEEM work presented here was performed on the I06 beamline at Diamond Light Source. The beamline is equipped with an Elmitec SPELEEM III microscope to map secondary electron emission arising from the absorption of soft polarized x-rays that were incident on the sample surface at a grazing angle of 16°. The probe depth of ~7 nm is sufficient to sample the thin films of interest through either 3 nm thick Cu or 2 nm Pt protective caps or electrodes. The lateral resolution was typically ~50 nm. A 300 V power supply was connected to the top and bottom electrodes via feedthroughs in the sample holder.

Raw XMCD-PEEM images were acquired during 1 s exposure times with right (R) and left (L) circularly polarized light, both on the Ni L_3 resonance at 851 eV, and off this resonance at 842 eV. The pixels in a XMCD-PEEM image describe the XMCD asymmetry $\left(I^{\mathrm{R}}-I^{\mathrm{L}}\right)/\left(I^{\mathrm{R}}+I^{\mathrm{L}}\right)$, which represents the projection of the local surface magnetization on the incident-beam direction. Here, $I^{\mathrm{R/L}}=\left(I_{\mathrm{on}}^{\mathrm{R/L}}-I_{\mathrm{off}}^{\mathrm{R/L}}\right)/I_{\mathrm{off}}^{\mathrm{R/L}}$ is the relative intensity of secondary electron emission evaluated from the intensities measured on and off the L_3 resonance (the comparison between intensities on and off resonance serves to minimise the effect of any inhomogeneous illumination.)

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Raw PEEM images with X-ray linear dichroism (XLD) contrast were acquired during 1 s exposure times with vertically (V) and horizontally (H) polarized light, both on the Ti L_3 resonance at 457 eV, and off this resonance at 446 eV. The pixels in a raw XLD-PEEM image describe the XLD asymmetry $\left(I^{\mathrm{V}}-I^{\mathrm{H}}\right)/\left(I^{\mathrm{V}}+I^{\mathrm{H}}\right)$, which represents the projection of the local surface polarization on the incident-beam direction. Here, $I^{\mathrm{V/H}}=\left(I_{\mathrm{on}}^{\mathrm{V/H}}-I_{\mathrm{off}}^{\mathrm{V/H}}\right)/I_{\mathrm{off}}^{\mathrm{V/H}}$ denotes the relative intensity for secondary electron emission due to x-ray absorption on ($I_{\mathrm{on}}^{\mathrm{V/H}}$) and off ($I_{\mathrm{off}}^{\mathrm{V/H}}$) the Ti L_3 resonance.

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We averaged 100 XMCD-PEEM (XLD-PEEM) raw images to obtain one image of ferromagnetic (ferroelectric) domains. Magnetic vector maps were obtained by combining two such XMCD-PEEM images for orthogonal sample orientations, after correcting for drift and distortion via an affine transformation that was based on the averaged XAS-PEEM image for each sample orientation.

All PEEM data presented here were collected at room temperature, however on I06 sample temperature can be readily varied between 100 K and 400 K and we have exploited this feature in Ref. 13. The sample holders have up to 6 pins that can be used to control temperature, inject currents, apply electric or small magnetic fields during imaging. Samples can be rotated in a 200° range with respect the the surface normal, allowing to construct vector maps of the magnetization direction (see later).The I06 beamline will soon be upgraded with the installation of an aberration corrected PEEM with extended temperature range (< 20 K) and higher transmission.

MFM is a top bench technique that compares favourably with XMCD-PEEM only because it is simpler, cheaper and more available. Compared to surface sensitive PEEM, it has superior probing depth, which permits to image the magnetism of buried layers (even through layers with thickness of a few tens of nm). However, PEEM outperforms MFM in all other respects. MFM maps only vertical stray field gradients (and therefore the link with magnetization maps is indirect), but PEEM with XMCD contrast directly probes both in-plane (IP) and out-of-plane (OOP) magnetization components since the incident x-ray polarisation has components both in and out-of-plane (we exploited this feature in [13]). In samples with zero OOP magnetization 2D vector maps of the magnetization can be built.

It is easy to image both ferroelectricity and ferromagnetism with PEEM because it is straight forward to switch between the XMCD and XLD imaging modes. This offers unique mechanistic insights into magnetoelectric coupling thanks to the possibility of imaging voltage-driven switching in both magnetic and ferroelectric phases in the same field of view (MFM and piezoresponse force microscopy are usually performed with different types of tip, and therefore can be combined only in principle to achieve this).

PEEM does not suffer from limitations due to tip-sample dipolar interactions that can arise with MFM. These dipolar interactions are obviously of magnetic origin in standard measurements, but imaging in electric fields can present the additional challenge of spurious electrostatic forces that can arise between the metallic coatings of tips and samples.

3. Nanoscale magnetoelectric effects in continuous Ni films on ferroelectric substrates

In this section, we review our results on local magnetoelectric effects in three different composite multiferroics made of continuous Ni films on ferroelectric BTO and PMN-PT.

3.1 Voltage-driven magnetization reversal in MultiLayer Capacitors (MLCs)

We investigated commercial MLCs [32] that comprised 2 μm-thick polycrystalline magnetostrictive electrodes of Ni, separated by polycrystalline piezoelectric layers of doped BTO. Sizeable converse strain-mediated magnetoelectric effects had been previously reported [34] in these highly inhomogeneous systems, and we were therefore inspired to explore them locally. We used mechanical polishing to expose alternate Ni and BTO layers (Fig. 1) and we performed MFM studies of these Ni layers in varying applied voltages. These applied voltages resulted in both

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volatile and non-volatile ME effects in the local out-of-plane magnetization, even if the OOP global magnetization M_z showed no electrically driven change. The most remarkable result was the discovery of a magnetic feature that underwent repeatable non-volatile switching with no applied magnetic field (Fig. 2). The MFM data that we present are consistent with full magnetization reversal, because the large asymmetric phase shifts (Fig. 2e) are similar to those that we observed before and after MFM tip reversal while imaging a single domain magnetized OOP [32]. We were able to understand and model this result by recognizing that a fast temporary reduction in the local magnetic anisotropy can trigger magnetization precession. In the presence of a small transverse magnetic field, this precession can in turn lead to magnetization reversal. In our experiment, this transverse field was the internal field provided by the magnetic domains surrounding the feature. The fast reduction in anisotropy near the feature was provoked by the strain released by a ferroelectric domain switching (realistic switching times are 1-10 ns).

3.2 Shear strain-mediated magnetoelectric effects

We have recently reported giant magnetoelectric effects in Ni films grown on single crystals of PMN-PT [33]. This rhombohedral ferroelectric with 8 polar directions along a pseudocubic <111>$_{pc}$ (Fig. 3) presents giant piezoelectric effects [35] that can be repeatably driven. For its outstanding electromechanical properties PMN-PT has been widely exploited in magnetoelectrics and for commercial applications (e.g. actuators and transducers). Initial work in magnetoelectrics used PMN-PT crystals with the (001) orientation [36], but the (011) orientation is now preferred because it permits non-volatile magnetoelectric effects [37].

Ferromagnetic films grown on (011) PMN-PT crystals have been reported to undergo electrically driven 90° rotations of the easy axes of their macroscopic magnetization [20, 21, 38-41], which are ascribed to a dominant component of normal strain along y $[011]_c$ (ref. 42). These reports were based on macroscopic magnetoelectric measurements but were not contradicted by rare imaging studies (38, 43).

Our study of magnetoelectric effects in Ni films on PMN-PT was the first to compare global and local magnetoelectric measurements in detail. Global ME effects were measured with a vibrating sample magnetometer, and the local ME effects were recognized using high-resolution vector maps of magnetization derived from XMCD–PEEM images. Both our global and local data seemed initially consistent with previous reports of an electrically driven 90° degree rotation of the magnetisation. However we will show through a pixel-by-pixel analysis that this rotation is in fact significantly smaller than 90° due to the effect of insofar neglected shear strains associated with ferrolectric switching.

The Ni films were initially isotropic, but poling of the PMN-PT substrate introduced a permanent easy-axis as shown by hysteresis loops measured in zero electric-field ($E_1 = 0$) at varying angles with the applied magnetic field (Fig. 4a&c). Hysteresis loops measured along two orthogonal in-plane directions when electric fields $E_2 = +0.167$ MV m^{-1} (Fig. 4d) and $E_3 = +1$ MV m^{-1} (Fig. 4g) are applied, are consistent with the expected 90° rotation of a global easy axis [20, 21, 37-40] created by poling. In fact, both the first ($E_1 \rightarrow E_2$) and second ($E_2 \rightarrow E_3$) field steps appear to interconvert the hard and easy directions, as seen more clearly by comparing polar plots of magnetic-hysteresis-loop squareness at each electric field (blue data, Fig. 4c,f,i).

To investigate local ME effects we used XMCD-PEEM to build 50 μm-wide vector maps of magnetization direction ϕ for the same electric fields E_1, E_2 and E_3. At E_1 (Fig. 4b) and E_3 (Fig. 4h), the vector maps consist of a small number of large domains which appear to be separated by 180° domain wall (consistent with the uniaxial magnetic anisotropy of the Ni film).

The vector map for the magnetic state at E_2 (Fig. 4e) shows that the domain structure at E_1 broke down into a large number of small domains, whose magnetizations appear to have generally switched from ±y to approximately along ±x. Plotting the corresponding pixel magnetization directions in our vector maps on polar plots (red data, Fig. 4c,f,i) seems to suggest that each electric-field step typically rotated the local magnetization by roughly the hitherto expected value of 90° (refs. 37-42).

However, the polar plot at E_2 has a complex fine structure which is absent in plots at E_1 and E_3, which consists of two clearly defined lobes. To understand this difference, we undertook a pixel-by-pixel comparison of our vector maps.

For both $E_1 \rightarrow E_2$ (Fig. 5a) and $E_2 \rightarrow E_3$ (Fig. 5d), we mapped changes of magnetization direction $\Delta\phi$, after excluding the small (white) areas between magnetic domain walls in the vector maps at E_1 (Fig. 4b) and E_3 (Fig. 4h) for which the magnetization did not return to its original direction after the $E_2 \rightarrow E_3$ step. (The fact that the walls between domains along ±y have slightly different positions at E_1 and E_3 implies that magnetization in the white areas underwent a ~180° rotation).

Fig. 5b shows the number N' of green pixels in our vector map at E_1 that underwent magnetization direction change $\Delta\phi$ during $E_1 \rightarrow E_2$, while the number N' of purple pixels in our vector map at E_1

that underwent magnetization direction change $\Delta\phi$ during $E_1 \rightarrow E_2$ is plotted in Fig. 5c. Similarly, the number N' of green pixels in our vector map at E_3 that resulted from magnetization direction change $\Delta\phi$ during $E_2 \rightarrow E_3$ is plotted in Fig. 5e, while the number N' of purple pixels in our vector map at E_3 that resulted from magnetization direction change $\Delta\phi$ during $E_2 \rightarrow E_3$ is plotted in Fig. 5f. By considering separately each type of magnetic domain in our vector maps at E_1 and E_3 (green and purple pixels, after excluding white areas), we see that the magnetization of many pixels switched by large angles $\Delta\phi$ that are significantly smaller than the hitherto expected [37-42] value of 90° (Fig. 5b,c,e,f). Moreover, the two full-width at half-maximum (FWHM) peaks in Fig. 5b (in Fig. 5c) are essentially interchanged in Fig. 5e (in Fig. 5f), which suggests that the net effect of the two field steps was to switch and switch back the magnetization of many pixels by large angles of typically less than 90°.

This pixel-by-pixel comparison permits to understand the complex structure of the PEEM polar plot at E_2 because it shows that the step $E_1 \rightarrow E_2$ does not drive a simple rotation of the easy axis, but rather a change in symmetry of the magnetic anisotropy, from unixial with EA along y, to biaxial with two non-orthogonal EA lying at less than 90° with respect to ±y. Only a change in simmetry explains the existence of 4 distinct peaks in Fig. 5b,c,e,f (2 for the green pixels, and 2 for the purple ones), revealing 4 equilibrium directions for the magnetization at E_2.

We have shown that the formation of a pair of misaligned EA at E_2 is due to shear strains generated from ferroelectric domains switching in rhombohedral PMN–PT [33]. These shear strains have been so far surprisingly neglected even though they can be quantified by investigating the distortions of the unit cell that arise when the polarization switches in and out of the x–y plane lying parallel to the $(011)_{pc}$ surface of the substrate. Our model of polarization switching predicts that the misaligned axes will form angles of ±62.6° to y. The two experimental modal angles of 62° and −64° approximately match our predicted values of ±62.6°, while the six remaining modal angles adopt larger values. Our prediction is based on a single domain model, but PMN-PT crystals contain many ferroelectric domains. Strain-mediated interactions between ferroelectric domains could inhibit shear strains and thus favor the EA to lay close to 90° with respect to y (zero shear strain corresponds to a 90° rotation).

3.3 Voltage control of perpendicular magnetization in Ni films on BTO substrates

In polycrystalline films of negative-magnetostriction ferromagnets (e.g. Ni, Co) isotropic tensile growth strain gives rise to a uniaxial perpendicular magnetoelastic anisotropy which competes with the IP shape anisotropy yielding a canted and inhomogeneous local magnetization. This inhomogeneity arises because the out-of-plane component of the local magnetization alternates in sign to create flux-closure domain structures that minimise stray-field energy. As a result, above a critical thickness, the surface magnetic domain structure appears in the form of a stripe pattern whose width approximatively matches the film thickness.

The growth strain responsible for stripe domains is generated at grain boundaries and thus the properties of the resulting magnetic structures are independent of the substrate.This can be seen in our 100 nm-thick polycrystalline Ni films grown by e-beam evaporation where substrate choice (quartz, silicon, PMN-PT or BTO) does not influence the presence of stripe domains or stripe width [13].

In Ni films on BTO we demonstrated both thermal and electrical control of the magnetic stripe domain structure, but here we briefly discuss electric control only. The electrically driven magnetic changes are shown in the composite images of Fig.6, where we used XMCD-PEEM to image the Ni film near a zig-zag edge and XLD-PEEM to image nearby ferroelectric domains in the exposed BTO substrate.

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This result is intriguing because it reveals strong-coupling between ferroelectric and ferromagnetic domains in relatively thick films. Moreover, given that here PEEM probes only ~3 nm below the Ni surface (penetration depth is ~7 nm, but these films feature a 4 nm thick Cu cap) these data show that strain developed at the interface propagates across the whole 100 nm thickness of the Ni film, thus confirming that magnetoelectric coupling mediated by strain is exceedingly more long-range than those mediated by exchange-bias [10].

Conclusions

Composite multiferroics are promising for low-power memory applications because they present large CMEs at room temperature. In Ni films grown on ferroelectric BTO and PMN-PT, we have used MFM and PEEM to reveal nanoscale magnetoelectric effects that eluded macroscopic measurements, thus demonstrating that imaging is a powerful tool in magnetoelectrics. For magnetic imaging, XMCD-PEEM outperforms MFM because it directly maps the IP and OOP components of the magnetization, and when the OOP magnetization is zero, vector maps can be built. Moreover, PEEM permits to switch between imaging ferromagnetism and imaging ferroelectricity allowing to visualize coupling between ferroelectric and ferromagnetic domains.

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Figure captions

Figure 1. Schematic of an MLC prepared for MFM imaging by mechanical polishing. Ni electrodes that are sufficiently near the surface for MFM (yellow) may be exposed (E) or lightly buried (B) by BTO. We studied both exposed and lightly buried Ni and the results presented in Figs 2 were obtained on exposed electrodes near the thin end of the wedge (arrowed).

Figure 2. Non-volatile electrically driven repeatable magnetization reversal with no applied magnetic field. Flattened MFM images (9.8 μm × 9.8 μm) for a region of nominally exposed Ni at (a) magnetic remanence after poling, and subsequently in zero electric field after having applied (b) +200 V, c) -200 V and (d) +200 V across MLC terminals. The feature undergoing reversal is saturated in these images, as seen from (e) profiles along the corresponding coloured lines in (a-d). Image and caption after Ref. 32.

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Figure 3. Cubic representation of the pseudocubic PMN-PT (011) unit cell. Black arrows denote Cartesian directions x || $[100]_c$, y || $[0\text{-}11]_c$ and z || $[011]_c$ that lie parallel to the orthogonal substrate edges (c denotes cubic). The x-y plane represents the PMN-PT surface on which the Ni film was deposited. The eight $<111>_c$ directions (red arrows) correspond closely to permitted directions of local polarization in the pseudocubic unit cell, but the correspondence is not exact due to the pseudocubic distortion. Numbered directions of polarization P lying antiparallel to each other are identified via + and - subscripts.

Figure 4. Global and local magnetization for magnetoelectric switching in Ni//PMN PT (011)-pc. For electric field (a-c) $E_1 = 0$, (d-f) $E_2 = 0.167$ MV m^{-1} and (g-i) $E_3 = 1$ MV m^{-1}, we show: (a,d,g) reduced magnetization components M_x/M_s (blue) and M_y/M_s (red) versus collinear applied magnetic field H; (b,e,h) 50 μm diameter magnetic vector maps of IP magnetization direction ϕ; and (c,f,i) polar plots of loop squareness M_r/M_s (blue) derived from plots that include those shown in (a,d,g), and polar plots of $N^{1/2}$ (red), where N is the number of pixels in (b,e,h) with magnetization direction ϕ. We use $N^{1/2}$ rather than N, so that the area under the curve in an infinitesimal angular sector is proportional to N rather than N^2. Green and red arrows in (b,e,h) denote the IP projections of the grazing incidence x-ray beam, M_r denotes remanent magnetization, and M_s denotes saturation magnetization. Prior to data acquisition, we poled the sample by applying and removing an electric field of $E = 1$ MV m^{-1}. Twenty bipolar sweeps did not reveal any evidence of fatigue. Image and caption after Ref. 33.

Figure 5. Changes in the local magnetization for magnetoelectric switching in Ni//PMN PT (011)pc. Comparison of the magnetic vector maps in Fig. 4b,e,h for (a-c) $E_1 \rightarrow E_2$ and (d-f) $E_2 \rightarrow$

E_3. Data between the magnetic domain walls at E_1 and E_3 correspond to the white regions in all five images, and are likewise excluded from the four histograms. (a,d) 50 μm diameter maps showing changes of pixel magnetization direction 180° ≤ $\Delta\phi$ ≤ 180°. (b,c,e,f) The number of pixels N' that undergo a change of magnetization direction $\Delta\phi$, for pixels that are (b,e) green and (c,f) purple at (b,c) E_1 and (e,f) E_3, with modal angles specified. Data colour represents either a modal angle, 0° or ±180° on the colour wheel in (a,d). Image and caption after Ref. 33.

Figure 6. Concomitant electrical control of stripe domains and ferroelectric domains. Composite images obtained at room temperature for (a) 0 V, (b) 300 V, and (c) 0 V following an initial electrical cycle. As shown in the schematic, these images were spliced together on either side of a zig zag edge in the film, thus combining a XMCD-PEEM image of the film with an XLD-PEEM image of the exposed substrate. Inferred and observed a and c domains of BTO are labelled. Arrow shows IP projection of incident-beam direction. Data for Sample 5. Image and caption after Ref. 13.

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