**180° head-to-head flat domain walls in single crystal BiFeO$_3$**

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Abstract

We investigate flux-grown BiFeO$_3$ crystals using transmission electron microscopy (TEM). This material has an intriguing ferroelectric structure of domain walls with a period of approximately 100 nm, alternating between flat and sawtooth morphologies. We show that all domain walls are of 180° type and that the flat walls, lying on (112) planes, are reconstructed with an excess of Fe and a deficiency of Bi. This reconstruction is similar to that observed in several previous studies of deposited layers of BiFeO$_3$. The negative charge of the reconstructed layer induces head-to-head polarisation in the surrounding material and a rigid-body shift of one domain relative to the other. These characteristics pin the flat 180° domain walls and determine the domain structure of the material. Sawtooth 180° domain walls provide the necessary reversal of polarisation between flat walls. The high density of immobile domain walls suppresses the ferroelectric properties of the material, highlighting the need for careful control of growth conditions.

Keywords: Bismuth ferrite, ferroelectric domains, 180° domain walls, reconstructed domain walls

INTRODUCTION

BiFeO$_3$ is arguably the most investigated multiferroic material$^{[1-3]}$, simultaneously showing ferroelectric, antiferromagnetic, and ferroelastic order at room temperature$^{[4]}$. The coupling of ferroelectric and ferromagnetic properties makes it ideal for the promising application of mutual control of electrical and...
magnetic order in multiferroic devices\textsuperscript{[5,6]}.

BiFeO\textsubscript{3} has a rhombohedral structure (space group \textit{R3c}) below its Curie temperature \(T_c = 1,100\) K\textsuperscript{[4]}, with a pseudocubic lattice parameter of 3.965 Å and rhombohedral angle 89.4° (pseudocubic indexing is used throughout this paper). Its spontaneous ferroelectric polarisation (\(P_s\)) has a magnitude \(P_s\) approximately 100 μC cm\(^{-2}\) along [111] and mostly arises from the displacement of Bi ions relative to their surrounding FeO\textsubscript{6} cages\textsuperscript{[7]}. Oxygen octahedra are tilted antiphase around the three-fold [111] axis by 10°-14° (\textit{a}a\textit{a}a in Glazer notation\textsuperscript{[8]}). The formation of domains in BiFeO\textsubscript{3}, similar to other ferroelectric materials, occurs to minimise the total energy, comprised of electrostatic, depolarisation, and elastic components\textsuperscript{[9]}. Depending on the angle \(\phi\) between \(P_s\) of adjacent domains, the meeting of these domains at domain walls can result in either purely ferroelectric (\(\phi = 180°\)) or ferroelectric-ferroelastic (\(\phi = 71°\) or 109°). The orientation or habit plane of domain walls has been of great interest as it provides insight into the competing energy components\textsuperscript{[10-13]} and determines functional properties\textsuperscript{[14,15]}.

The batch of flux-grown single-crystals of BiFeO\textsubscript{3} investigated in this study have been subject to several previous investigations\textsuperscript{[16,17]}, all of which have revealed a dense array of parallel domain walls seen in piezoresponse force microscopy (PFM) and conventional transmission electron microscopy (TEM) as alternating sawtooth and flat bands of contrast [Figure 1]. The complex microdomain structure in these crystals is extremely stable, exhibiting no change upon observation, even in the thinnest specimens. Furthermore, no observable switching (or rapid back-switching into the pristine domain pattern) was found, even with 200 V applied to the surface with an AFM tip. The inability to access the functional properties of these high-quality single crystals is concerning and may have implications for the exploitation of BiFeO\textsubscript{3} in practical applications. Therefore, it is important to understand the nature and origin of this domain structure.

The first study of these crystals\textsuperscript{[16]} showed the domain walls to be either 180°- or 109°-type and due to the high predicted energy of 180°-type domain walls, it was proposed that they were probably 109°-type. A second study\textsuperscript{[17]} of the same batch of crystals using negative \(C_s\) high-resolution TEM imaging found a variety of domain wall types, including 71°-, 109°-, and 180°-type. Another recent study of similar material\textsuperscript{[18]} confirms the sawtooth walls to be 180°-type but proposes that flat domain walls are 109°-type. In this article, along with its companion publication\textsuperscript{[19]}, we revisit the domain structure in this same batch of crystals using a combination of PFM, conventional TEM, convergent beam electron diffraction (CBED), atomic resolution scanning TEM (STEM), and electron energy loss spectroscopy (EELS). The high stability of the domains ensured there was no influence of specimen preparation on the structure. We find that there are only 180° domains in the crystal, i.e., all domain walls are 180°-type. The difficulties experienced in previous work may potentially be attributed to projection effects when the three-dimensional domain structure is observed in an electron-transparent foil. To overcome this, we use a focused ion beam (FIB) technique to prepare multiple sections with different orientations from the same region of the crystal. We describe the sawtooth domains in a companion article\textsuperscript{[19]}; the focus of this article is on the flat 180° domain walls.

**MATERIALS AND METHODS**

BiFeO\textsubscript{3} single crystals were obtained from the same batch used in the studies of Berger \textit{et al.}\textsuperscript{[16]} and Jia \textit{et al.}\textsuperscript{[17]}, who described growth conditions in detail. In brief, crystals were precipitated from a Fe\textsubscript{2}O\textsubscript{3}/Bi\textsubscript{2}O\textsubscript{3}/B\textsubscript{2}O\textsubscript{3} flux cooled very slowly from 1,170 K to 875 K. Much of the growth took place below the paraelectric-ferroelectric phase transition at 1,098 K. The crystals were compact and had sizes of half to a few millimetres, with a top surface of four (\textit{hhl}) facets, only a few degrees away from (001). For this study, several well-formed single crystals that had no contact with the crucible sidewalls were selected.
Figure 1. (A) PFM out-of-plane amplitude image of the sectioned and polished (001) surface of the BiFeO$_3$ crystal. (B) Low magnification BF-STEM image of a (110) FIB-prepared lamella. (C) Low magnification BF-STEM image of a (010) FIB-prepared lamella. Arrows in (B and C) point towards increasing thickness. Examples of continuity of domain walls, where a sawtooth wall changes direction and becomes a flat wall, are marked by \( x \) in (A and B). Scale bars are 100 nm.

For PFM measurements, a crystal was ground and polished to (001) using diamond lapping film of decreasing sizes to 0.1 \( \mu \)m, finishing with a dilute 0.04 \( \mu \)m colloidal silica solution. PFM measurements were conducted on a Bruker Dimension Icon AFM at a scan rate of 0.5 Hz using a Bruker OSCM-PT-R3 tip with Pt coating, along with a drive frequency of 254 kHz and a drive voltage of 2 V.

Specimens for TEM were prepared by lift-out on a Tescan Amber Ga$^+$ FIB-SEM from a second crystal in its as-grown state, with (110), (010), and (1\( \overline{1} \)0) orientations. Cutting and thinning were performed using an ion beam energy of 30 kV, with a final low-energy polish at 2 kV. The specimens were taken from a compact region on the crystal, and their orientations were verified by their relative position, selected area electron diffraction (SAED) patterns, and atomic resolution images. To obtain the thinnest possible specimens for very high-resolution imaging, wedge-shaped lamellae were produced.

STEM images were taken with a double-corrected JEOL ARM 200F TEM/STEM operating at 200 kV, with a beam convergence semi-angle of 22 mrad. The annular bright field (ABF) and annular dark field (ADF) detectors covered 11.5-24 and approximately 45-180 mrad, respectively [11]. In atomic resolution STEM images, atom positions are determined by fitting two-dimensional Gaussian peaks [11,20].

EELS data and ADF images were acquired at the UK SuperSTEM Laboratory using a Nion UltraSTEM 100 MC operated at 60 kV, with a beam convergence semi-angle of 32 mrad. The acceptance semi-angles of EELS and ADF were 44 mrad and 79-185 mrad, respectively.

RESULTS AND DISCUSSION

Domains on the polished (001) surface imaged by PFM follow a regular stripe pattern running along [\( \overline{1} \)10] with reversal of both in-plane and out-of-plane phase and alternating sawtooth and straight domain walls, as shown in Figure 1A and Supplementary Figure 1A. No relation between domain structure and surface topography was present (see Supplementary Figure 1B). The spacing between stripes varies between 60 and 120 nm. Occasionally, domains can be found to terminate where sawtooth and straight domain walls meet (e.g., at x in Figure 1), indicating that these two types of walls separate the sample into only two different domains. Bright-field (BF) STEM images of (110) and (010) FIB lift-out lamellae are shown in Figure 1B and C (see Supplementary Figure 2A). In both (110) and (010) orientations, the domain walls appear as alternating sawtooth and planar bands of contrast. In these BF-STEM images, the crystal is
aligned to the zone axis, and local conditions, such as strain, composition, or a small change in crystal orientation across a boundary, can strongly affect the transmission of the electron probe. As a result, domain boundaries appear slightly darker. Their orientation varies depending on the direction of view. In the (110) section [Figure 1B], straight bands are parallel to the (001) surface, while in the (010) section [Figure 1C], they are inclined at an angle of approximately 25°, running along [¥01]. Sawtooth walls present different V-shapes when observed from different directions, and their structure is considered in detail elsewhere[19]. The domain appearance and PFM measurements agree with the observations of Berger et al.[16]. However, they interpreted the flat bands of contrast seen in Figure 1B and C as domains with a width of approximately 10 nm. The varying thickness of the lamellae in Figure 1 shows this to be incorrect. In Figure 1C, the lamella decreases to a thickness of just a few nm at the edge of the specimen on the top-right of the image. The width of the bands decreases as the specimen thickness decreases, indicating that they correspond to planes in the material that are inclined to the direction of view. The same effect can also be seen in Figure 1B, where the specimen thickness increases towards the bottom of the image. Therefore, the darker bands are not slabs of material that form domains, different to the adjacent lighter material. They are the projection of a plane that is inclined to the point of view.

The proposal that the flat bands of contrast in Figure 1B and C are planes rather than slabs of finite thickness is confirmed by the (¥10) section shown in Figure 2A. In this projection, their width does not depend on specimen thickness. Here, they are seen edge-on and appear as sharp, straight lines at an angle of 35° to (001) surface, i.e., lying on (112) planes. The sawtooth domain pattern can be recognised in the thinnest part of the specimen but becomes indistinct where the lamella is thicker due to overlapping projections. Higher magnification images [Supplementary Figure 2B] show that the average habit plane is slightly away from (112); the flat domain walls have a terraced structure, with (112) facets seen edge-on, separated by steps 1-2 nm in height.

High-resolution (¥10) STEM images of one of these domain walls, seen edge-on in the thinnest part of the specimen, are shown in Figure 2C and D. In any {110} section of BiFeO₃, the [111] polar axis can either lie within the plane of the section or at 35° to it, (i.e. with an out-of-plane component). The a a a antiphase tilting of oxygen octahedra in the R3c structure gives different projections for the two cases. When the polar axis is in the image plane, the antiphase tilting of octahedra is not visible, and oxygen columns appear as a dumbbell at the same location in every unit cell[21]. Conversely, when it has an out-of-plane component, O-Fe-O-Fe-O chains form an arc with a curvature indicating the direction of P[17] [Supplementary Figure 3]. In Figure 2C, the polar axis is in the image plane for both domains, consistent with a [¥10] BiFeO₃ projection [Figure 2B]. On a unit-cell level, polarisation Pₚ is often taken to be proportional to the negative displacement of Fe relative to the centre of the rectangle defined by its four Bi neighbours, namely -δFB[22,23]. Since the polar axis lies in the image plane, we observe a full projection of the displacement (in comparison, one can only observe approximately 57% or 81% of the displacement in [110] or [100] projections, respectively[23]). The quiver map of -δFB vectors overlaid on the ADF-STEM image of Figure 2D shows that polarisation changes by 180°, pointing towards the wall on both sides. (N.B. at the domain wall itself, δFB is not calculated because it is unclear how to define the unit cell.) Away from the domain wall, the magnitude of δFB is generally around 40 pm, in good agreement with the theoretical value of 41 pm for Pₚ of approximately 100 μC cm⁻²[21,23,24]. As a final observation in Figure 2D, Bi atom columns in the domain wall are not as bright as equivalent atom columns in the surrounding material, indicating a lower occupancy of Bi at the domain wall.
Figure 2. (A) Low magnification BF-STEM image of a (10) FIB-prepared lamella. The dark arrow at the thin edge of the lamella points towards increasing thickness. The width of the straight dark lines is independent of specimen thickness, showing that they correspond to planes seen edge-on. The crystal orientation applies to all images in this figure. (B) Ideal (10) projection of BiFeO$_3$, where Bi atoms are presented in red, Fe blue, and O grey. The direction of the $\delta_{FB}$ vector is also shown. (C) A high-resolution ABF-STEM image of a flat wall. White lines are aligned horizontally and vertically, allowing the rigid-body displacement of the domains along [110] and [001] to be seen. (D) An ADF-STEM image taken simultaneously with (C). Arrows in (D) show local $\delta_{FB}$ vectors, indicating the magnitude and direction of polarisation in each unit cell.

It is apparent from Figure 2C and D that these 180° head-to-head domain walls are reconstructed; the local structure is different from the bulk crystal, and the associated distortions are quantified in Figure 3. Examining the horizontal (001) planes of bright Bi atom columns in Figure 2D, downwards displacements (i.e., along [001]) are seen on the left, starting approximately 0.5 nm away from the domain wall, while upwards displacements along [001] are seen on the right. These displacements are shown as blue points and lines in Figure 3A and reach a value of approximately 0.1 nm at the domain wall itself. Far from the domain wall, these displacements tend to zero, showing that the (001) planes are aligned, as can be directly seen from the horizontal white lines in Figure 2C. The orange line in Figure 3A shows a similar measurement for the vertical (110) planes. Here, there is greater scatter as the displacements vary from place to place along the domain wall. Interestingly, there is a slight increase in displacements immediately adjacent to the domain wall. Additionally, a long-range misalignment of about 0.1 nm is observed, corresponding to a lateral shift of $\frac{1}{2}[110]$. This rigid-body displacement can be directly observed in Figure 2C by comparison of the vertical white lines with the position of atom columns. The average value of $P$, changes its sign abruptly
Figure 3. (A) Distortion of unit cells in the vicinity of the domain wall shown in Figure 2D, measured by the displacement of Bi atom columns away from (110) planes (orange) or (001) planes (blue). Points correspond to individual measurements, and the line is an average value. (B) Average polarisation $P_s$ obtained from $-\mathbf{\delta} FB$ vectors, measured in bands parallel to the domain wall.

(over approximately 1 nm) at the wall [Figure 3B], which is significantly different from the previously reported thicker (> 2 nm) widths for charged domain walls in BiFeO$_3$ without any local reconstruction$^{[23,25-27]}$. The narrow domain wall width indicates the presence of a strong electrical field related to the local change in structure at the domain wall.

More information on the reconstruction at these domain walls can be obtained from a second point of view, obtained from a (010) section shown in Figure 4. Although the domain wall is probably inclined to the electron beam direction, this image is taken at the thinnest part of the lamella, and the high electron beam convergence angle gives a reduced depth of field$^{[28]}$. Consequently, the domain wall is sharply delineated in ADF-STEM images. (The effect of reduced depth of field is shown in Supplementary Figure 4). At the domain wall [Figure 4A], the reconstruction appears as alternating bright and dark clusters of atoms, forming either $2 \times 1$ or $2 \times 2$ atom blocks, while $-\mathbf{\delta} FB$ vectors demonstrate the $180^\circ$ head-to-head nature [Figure 4B]. There is no obvious change in the magnitude of $P_s$ immediately adjacent to the reconstructed domain wall, which is approximately 1.5 unit cells in width and runs along $[201]$ in this projection. Just as in the $[\overline{1}10]$ images of Figure 2, (001) planes are fully aligned (horizontal white lines), while a $\frac{1}{2}[100]$ displacement is visible (vertical white lines). Since the component along the beam direction is not visible, this is in full agreement with the $\frac{1}{2}[110]$ rigid body shift observed in Figure 2. Bending of the (001) Bi planes can also be found at the domain wall (see Supplementary Figure 5). Figures 4E-G show core-loss EELS elemental maps of the domain wall, together with a simultaneously recorded ADF image in Figure 4D. The brightest atom columns in the ADF image are shown to be Bi in a separate EELS acquisition [Supplementary Figure 6]. Dark regions in the ADF image at the domain wall lack Bi and consist of Fe+O. These findings, along with the results presented in Figures 2 and 4, show that the flat domain wall has a structure and stoichiometry different from bulk BiFeO$_3$. They are consistent with each other both in the observed rigid-body shift, lower Bi content, bending of (001) Bi planes, and $P_s$ distribution around them.

The nonstoichiometry of the reconstruction in the flat $180^\circ$ head-to-head walls is an indication that they form during crystal growth, while the periodic domain structure indicates a degree of self-organisation. Synthesis of crystalline BiFeO$_3$ is only possible within a narrow range of conditions, both in the deposition of thin epitaxial layers$^{[21,29-32]}$ and as ceramics or single crystals$^{[33,33-35]}$. In bulk crystal growth, the secondary Bi-rich sillenite Bi$_{25}$FeO$_{39}$ and/or Fe-rich mullite Bi$_2$Fe$_4$O$_9$ phases readily form to accommodate deviations.
Figure 4. (A) An ADF-STEM image of a head-to-head 180° domain wall in a (010) FIB-prepared lamella. Large and bright atoms columns are Bi, and smaller ones are Fe. O atoms are not visible. (B) Quiver plot of local $\mathbf{\delta}_{FB}$ vectors, indicating the magnitude and direction of polarisation in each unit cell. (C) Ideal (010) projection of BiFeO$_3$: Bi atoms are red, Fe blue, and O grey. The direction of the $-\mathbf{\delta}_{FB}$ vector is also shown. (D-G) core-loss EELS data: image size is 1.32 $\times$ 3.13 nm. (D) HAADF-STEM; (E) an element map of Fe; (F) an element map of O; (G) a composite of ADF (red) and Fe (green).

from perfect stoichiometry, e.g., due to the relative volatility of Bi$_2$O$_3$[33,35]. In epitaxial thin film growth, locally nonstoichiometric planar defects that strongly resemble those shown in Figure 4 are often seen[22,29,36,32,37]. Maclaren et al. found iron-rich regions consisting of edge-sharing FeO$_6$ octahedra, resembling the structure of $\gamma$-Fe$_2$O$_3$ (and, indeed, mullite)[36,34,37]. Li et al. showed that they can be induced by a slight increase in substrate temperature during MBE deposition, which makes the surface Fe-rich[22,29,32]. Similar to the observed reconstruction in this study, these previously observed planar defects also have a half-unit-cell rigid body shift across them caused by the switch from corner-sharing to edge-sharing oxygen octahedra. The deviation from stoichiometry gives a local excess of oxygen anions, giving a negative charge density estimated to be between $-68 \ \mu$C cm$^{-2}$ and $-110 \ \mu$C cm$^{-2}$[22,34,37]. The effect of these negatively charged planar defects in the surrounding BiFeO$_3$ matrix is to induce an increased local polarisation towards them, producing charged head-to-head domain walls[32]. The intrinsic negative charge density accommodates the majority of the $-190 \ \mu$C cm$^{-2}$ polar discontinuity expected at the flat domain walls in our crystal, explaining the relatively narrow region of adjacent material that has a different polarisation to the bulk material.
Therefore, in our material, we may explain the origin of the domain microstructure as follows. Since the temperature during crystal growth is below $T_c$, ferroelectric domains are to be expected, and ideally, they would assume a form to minimise the competing ferroelectric/ferroelastic/electrostatic energy components of the system. In thin film growth, it is well established that domain microstructures may evolve during film deposition\cite{38} and, in turn, can influence growth morphology\cite{39}. Therefore, in our case of single-crystal BiFeO$_3$ growth, we may expect regions with positive and negative surface charges that have an effect on domain structure and subsequent growth. Importantly, Li\cite{29} showed that nonstoichiometric monolayers may form on polarisation-up, negatively charged BiFeO$_3$ growth surfaces. They found that as growth progresses, a region with polarisation pointing towards the growth surface may, therefore, reach a critical negative charge, causing the incorporation of FeO$_6$ octahedra at the crystal surface and producing nonstoichiometric monolayers of the defective material. They also showed that the negative charge induces and pins head-to-head polarisation\cite{32}. It seems plausible that a similar mechanism is responsible for the head-to-head domain walls that we observe. On a (hhl) growth surface, the FeO$_6$ octahedra would form edge-sharing chains along [100], partly defining the orientation of the wall. These negatively-charged defects stabilise the 180° domain walls and perpetuate incorporation of FeO$_6$ octahedra in the next monolayer of crystal growth. It is not clear if the (112) plane makes the best match of their structure to the adjacent R3c BFO or whether this habit plane is a result of the asymmetrical surface charge where the domain wall intersects the surface, which may vary with, for example, the growth rate. An array of such structures can only exist with a further reversal of polarisation between them, and tail-to-tail 180° domain walls are the most efficient way of achieving this. The presence of the sawtooth walls is thus topologically necessary. It has been shown that unpinned 180° domain walls are expected to develop a crenellated structure to balance polarisation and electrostatic energy\cite{40,41}. The crenellated structure of these walls indicates that they are not pinned in the same way as the flat walls, and a very recent study of these tail-to-tail walls indicates that they are, to some extent, mobile when an electric field is applied, while head-to-head flat walls are immobile\cite{18}. This is in good agreement with our measurements of $P_s$ hysteresis, which gave essentially no signal, indicating that the flat walls are completely pinned. It is commonly observed that measurements of $P_s$ in bulk BiFeO$_3$ crystals are usually an order of magnitude smaller than in thin films, even though our measured $\delta_{FB}$ vectors at the atomic scale are similar, equivalent to $P_s$ approximately 100 $\mu$C cm$^{-2}$. While this batch of material now dominates high-resolution structural studies of bulk BiFeO$_3$\cite{16,17}, there is nothing in the proposed origin of the domain structure that would indicate it to be specific to these particular crystals. On a macroscopic scale, the deviation from perfect stoichiometry is miniscule; approximating the defects as sheets of Bi with 50% occupancy, a spacing of 100 nm gives a Fe excess of only approximately 0.2 at.%, meaning that they are unlikely to be avoided by changes in starting composition.

**CONCLUSIONS**

Periodic domain structures in single crystal BiFeO$_3$ have been re-investigated. Our results show that all domain walls are of 180°-type, alternating between flat head-to-head and sawtooth tail-to-tail walls. We focus on the flat walls here, finding that they have an orientation close to (112) and a polarisation reversal that occurs over only approximately 1 nm, significantly less than seen in other charged domain walls in BiFeO$_3$. They are locally nonstoichiometric, with an atomic reconstruction of roughly a unit cell in thickness, similar to that seen in planar defects in thin film BiFeO$_3$ and related materials. The reconstructed region contains edge-sharing FeO$_6$ octahedra that produce a rigid-body shift of half a unit cell and a negative charge density that induces head-to-head polarisation. We propose that the periodic domain structure forms during crystal growth in regions where negative surface charges exceed a critical value, causing the incorporation of FeO$_6$ octahedra that is perpetuated and becomes self-organised as growth proceeds. The reconstruction and local charge strongly pin head-to-head 180° domain walls, explaining the poor response of these BiFeO$_3$ single crystals in measurements of polarisation. Avoiding their formation is,
therefore, a necessary prerequisite for high-performance BiFeO₃ crystals and ceramics.

**DECLARATIONS**

**Authors’ contributions**

Performed data acquisition, data analysis, and interpretation and wrote the first draft of the article: Ge W, Sanchez AM

Performed data acquisition (STEM+EELS maps) at SuperSTEM: Ramasse Q

Provided material, conception and design of the study and interpretation: Alexe M

Performed data analysis and interpretation and contributed to the development of the article: Beanland R, Sanchez AM

**AVAILABILITY OF DATA AND MATERIALS**

The data that support the findings of this study are openly available in WRAP at http://wrap.warwick.ac.uk/173964.

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**Conflicts of interest**

All authors declare that there are no conflicts of interest.

**Ethical approval and consent to participate**

Not applicable.

**Consent for publication**

Not applicable.

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**REFERENCES**


