# Using EBSD to Map Domain Structures in Ferroelectrics

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Electron backscatter diffraction (EBSD) has undergone a rapid transformation in the past 15 years from a curiosity and a minority interest into an important part of our arsenal of microstructure characterisation techniques. In this time, it has been very widely applied in metallurgy, especially in understanding the influence of processing on local crystallographic textures and on boundary populations. More recently, it has been used in a diverse range of different fields including semiconductors, ceramics, geological materials and even palaeontology. In this article we will show how EBSD can yield invaluable information about domain structures in ferroelectrics.

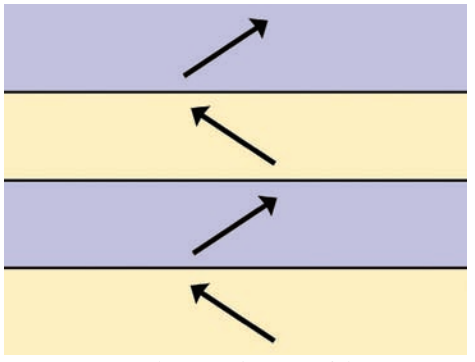

*Fig. 1: Schematic diagram of domains in a ferroelectric.*

Ferroelectrics are a key class of functional materials that display an electrostatic polarisation below a certain temperature, the Curie temperature. The properties that come from this including a high dielectric constant, the piezoelectric effect (where the shape changes in response to an electric field or vice versa), and the ability to retain a strong remanent polarisation after exposure to field are of great practical benefit. Ferroelectrics are therefore used in capacitors, transducers, micro-positioning systems, actuators, and ferroelectric memories for computers.

A key feature of ferroelectrics is that the whole crystal does not have a single polarisation orientation, but breaks up into domains where the polarisation is oriented differently as shown in Figure 1, separated by domain boundaries. These domain boundaries usually form on specific planes to minimise stress, for example in tetragonal ferroelectrics such as $BaTiO_3$ or Ti-rich $Pb(Zr,Ti)O_3$ (PZT) the angle of rotation is approximately 90° and the domains sit on {101} planes resulting in highly regular lamellar structures (see Figure 2).

The complication with doing EBSD on these materials is that they are insulators and charge easily under an electron beam. At the same time, the features of interest are under 100 nm, so a fine spot size is needed, which can be a problem with using a raised pressure in an environmental scanning electron microscope since the gas atom scattering can cause significant beam spreading even as it

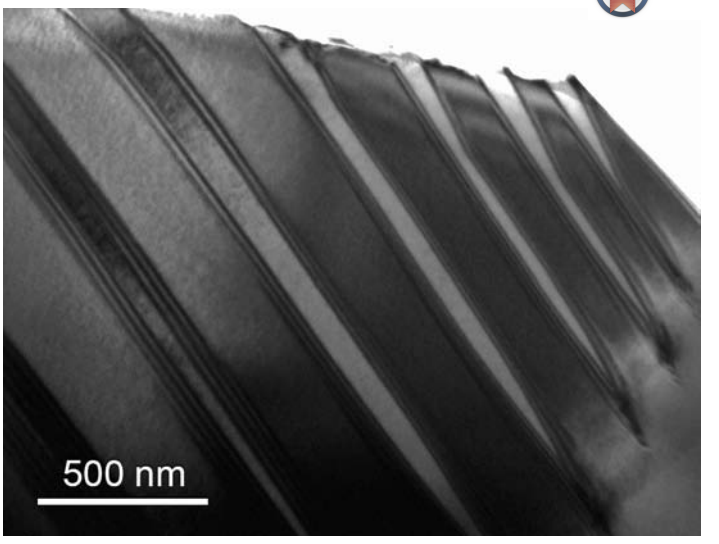


*Fig. 2: Bright-field transmission electron micrograph of lamellar "90°" domains in $PbZr_{0.5}Ti_{0.5}O_3$.*

ameliorates the charging problem. We overcame this problem, at least partially, by coating the sample with a thin layer of carbon; too thin and the conductivity is too poor to prevent charging, too thick and the EBSD pattern is degraded by scattering from the amorphous coating. Even at the optimum coating thickness, we found that it was necessary to work quickly, use 4×4 binning of the patterns from the camera, and only scan relatively small areas. Even then, not all maps were successfully acquired and some suffered from serious drift and charging before they could be completed.

Figure 3 shows one of our maps collected from PZT with a Zr:Ti ratio of 37.5:62.5 and doped with 1% La and 2% Sr (provided by Prof. Michael Hoffmann and Dr. Hans Kungl at the University of Karlsruhe, Germany). This shows clear mapping of the lamellar domains with only a few points producing a questionable indexing (*e.g.* the pink points in the centre of the lilac domain). One great point about the EBSD technique for ferroelectrics is that we can extract the orientation of the polar *c*-axis at any point and thus view how polarisation behaves across a sample (although please note that only the orientation, and not the sense of this axis can be determined). Another advantage for the domain analysis is that we can calculate misorientations between domains; in this case the misorientations are about the [010] axis with

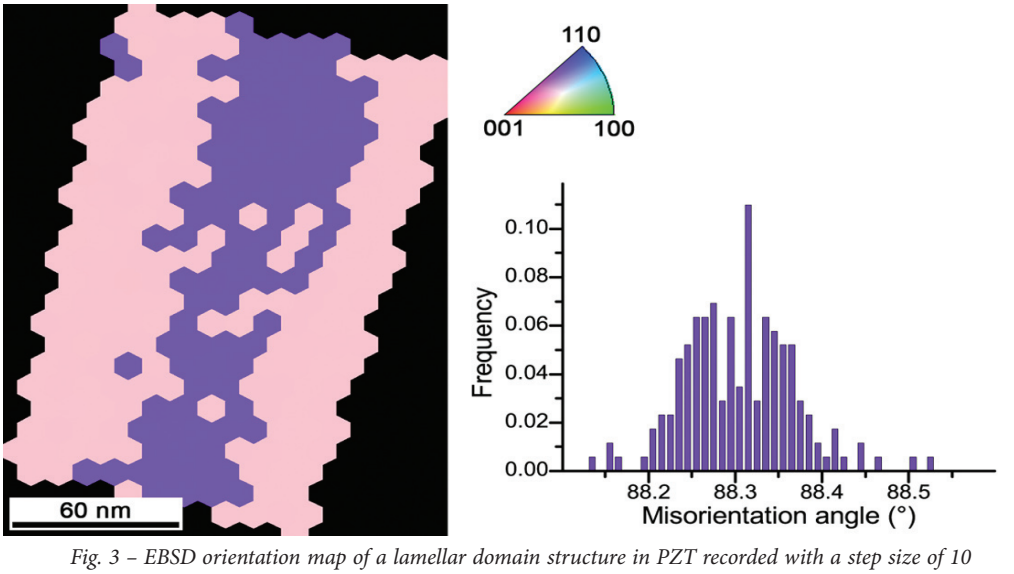


*Fig. 3 – EBSD orientation map of a lamellar domain structure in PZT recorded with a step size of 10 nm coloured according to the inverse pole figure colours in the top right. A histogram of the misorientations between points in the pink and lilac domains.*

angles given in the histogram in Figure 3. The average angle of this distribution is related to the *c/a* ratio of the material via 2 arctan(*a/c*), so in this case we have a local *c/a* ratio of 1.030 ± 0.001 in reasonable agreement with the bulk value 1.034 determined using X-ray diffraction[1]. This gives us a method to determine local lattice parameters in such domain structured materials, which would be particularly useful in inhomogeneous materials or devices where bulk measurements by X-ray diffraction would not be possible.

It is also possible to examine more complex domain structures such as the herringbone domain junction shown in Figure 4. We have studied such structures in PZT and $BiFeO_3$-$PbTiO_3$ and an example from the latter material (provided by Mr. Tim Burnett, Dr. Timothy Comyn and Prof. Andy Bell at the University of Leeds, UK) is shown in Figure 4. We have been able to show an excellent agreement between geometric/crystallographic models of such structures and the EBSD measurements of the misorientations across the domain boundaries. Moreover, such data gives us ways of estimating stress concentrations at the junction – the junction regions shows a lot of missing or random coloured points for a reason – the stress there is so high (~GPa!!) that the EBSD patterns are of very poor quality and cannot be reliably indexed. A full analysis of the crystallography of these junctions is beyond the scope of this article but the interested reader is referred to our upcoming publication for details[2].

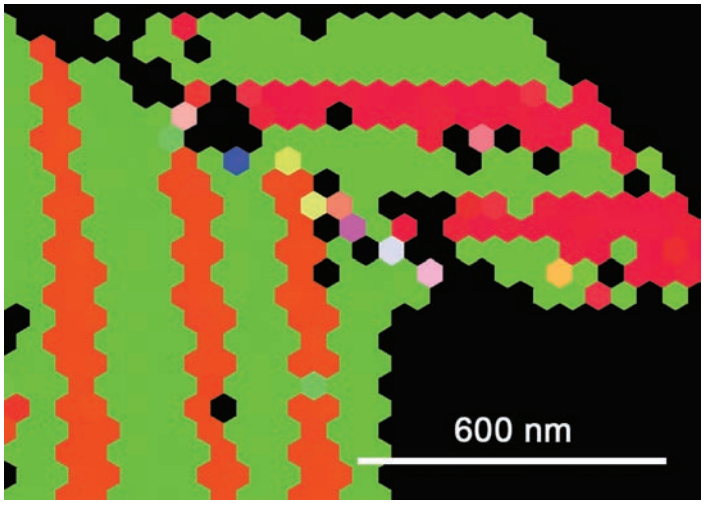


*Fig. 4 – Mapping of a herringbone domain junction in $BiFeO_3$-$PbTiO_3$, the arrows and dotted circle symbols show the orientation of the c-axis in each domain.*

In conclusion, domain structures in ferroelectrics can be mapped using EBSD, the maps can be processed to reveal information about the local crystallography, the local polarisation orientations are available from the maps, and complex junction structures can be examined using this technique. Whilst the approach described here is already clearly useful in providing an improved micro-scale and sub-micro-scale understanding of domain structures in ferroelectrics, there are many other classes of materials that also form regular domain structures, and where this technique would be equally applicable. For instance, it could be a useful way of studying structures in lath martensites, magnetostrictive materials, ferroelastics, and multiferroic materials and heterostructures. Hopefully, this article inspires somebody to have a go at using EBSD to study domain structures in a material we haven't even considered yet! ■

## References


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2. M.U. Farooq, R. Villaurrutia, I. MacLaren, T.L. Burnett, T.P. Comyn, A.J. Bell, H. Kungl, and M.J. Hoffmann, J. Appl. Phys., 104, 024111 (2008).




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