Electromechanical Response of Polycrystalline Barium Titanate Resolved at the Grain Scale

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Ferroic materials are critical components in many modern devices. Polycrystalline states of these materials dominate the market due to their cost effectiveness and ease of production. Studying the coupling of ferroic properties across grain boundaries and within clusters of grains is therefore critical for understanding bulk polycrystalline ferroic behavior. Here, three-dimensional X-ray diffraction is used to reconstruct a 3D grain map (grain orientations and neighborhoods) of a polycrystalline barium titanate sample and track the grain-scale non-180° ferroelectric domain switching strains of 139 individual grains in situ under an applied electric field. The map shows that each grain is located in a very unique local environment in terms of intergranular misorientations, leading to local strain heterogeneity in the as-processed state of the sample. While primarily dependent on the crystallographic orientation relative to the field directions, the response of individual grains is also heterogeneous. These unique experimental results are of critical importance both when building the starting conditions and considering the validity of grain-scale modeling efforts, and provide additional considerations in the design of novel ferroic materials.

Keywords: X-ray methods; polycrystalline materials; microstructure; domains

I. Introduction

Ferroic materials showing switchable order parameters under an external stimulus are key components in many modern devices. An example is electromechanical materials that directly couple mechanical stress with electrical charge via the piezoelectric effect and are used for actuation applications. Lead–zirconate–titanate (PZT)-based piezoelectric ceramics dominate industrial applications, primarily due to the combination of large piezoelectric coefficients and good temperature stability. Ceramics are also easier to manufacture and more cost-effective than single crystals. Recent regulations in North America, Europe, and elsewhere are pushing for lead-free alternatives, leading to the discovery of some high-performance lead-free piezoceramics and reinvigorating studies into the fundamental nature of electromechanical coupling.

Barium titanate (BT) is a prototypical perovskite piezoelectric material and was one of the first piezoceramics discovered. It is readily produced in ceramic form and is commonly used as an end-member component of many disordered perovskites that have been identified as potential high-performance lead-free piezoelectrics. Above the Curie temperature, \( T_C \), of roughly 130°C, BT is in the paraelectric cubic phase, transforming to a ferroelectric tetragonal phase upon cooling through \( T_C \). Since any of the primary cubic directions may become the anisotropic tetragonal \( c \)-axis, the transformation results in a domain structure within each grain, composed of both 180° and non-180° domains. The domains form such that the total energy of the system (e.g., elastic, electrical, etc.) is minimized, resulting in complex microstructures across many length scales. Additionally, due to the anisotropic thermal expansion coefficient, the interlocked, differently oriented grains develop thermal stresses during cooling. This results in a spatially distributed, built-in electric field in the as-processed ceramic, generated via the direct piezoelectric effect. The magnitude of this electric field is maximum at grain boundaries and corners, where the stresses are largest.

Non-180° domain wall motion represents a significant contribution to the overall strain response of piezoceramics. Powder diffraction studies have demonstrated that, on average, domain switching behavior depends on grain orientation, with the polarization tending to align as closely with the electric field direction as allowed by the crystallite orientation. This has made texture a very important topic with regard to engineering lead-free piezoceramics with properties approaching those of their PZT counterparts. It is clear, however, that it is not just the grain orientation that plays a role in domain switching. Simulations reveal that domain switching tends to initiate at grain boundaries and corners, where stress concentrations exist. Surface measurements by piezoresponse force microscopy reveal large deviations in the hysteresis loops of adjacent grains, with the effect again most pronounced at the grain boundaries. It has been suggested that there may exist characteristic grain boundaries that serve to either enhance or inhibit domain switching and strain response. Domains have also been observed crossing grain boundaries, suggesting the possibility of longer range coupling. Such correlated mesoscale phenomena have often proven difficult to study, and benefit greatly from a combination of modeling and imaging techniques. Powder diffraction studies give insight into the average mechanics, while grain-scale information revealed by imaging techniques is generally limited to two-dimensional surface measurements. Simulations and modeling offer an opportunity for in-depth investigation of length scales that are inaccessible experimentally. Phase field models have in particular contributed to the understanding of domain structures under an electrical field. However, models require experimental input for validation, typically macroscopic measurements such as texture, piezoelectric constants, and hysteresis loops. It is
important to consider that a model in macroscopic agreement with experiment may not always capture local effects, as demonstrated by comparisons between self-consistent models, where the strain interaction is smoothed, and finite element models, where the strain interaction is preserved.28

In this work, we present a three-dimensional microstructure measurement where the grain morphology, orientation, and neighborhood of a course-grained BT sample is reconstructed. Domain volume fractions of the three crystallographically unique tetragonal non-180° domain variants are extracted for individual grains and used to calculate the grain-scale non-180° domain switching strains in the as-processed state of the ceramic, as well as under the stimulus of an externally applied electric field. The data are measured using a combination of near-field and far-field three-dimensional X-ray diffraction (3DXRD). We present results regarding the effect of microstructural grain features on the grain-scale ferroelastic switching strain. Additionally, we discuss the possible use of this unique dataset for polycrystal model input and validation. Such integrated experimental and modeling approaches have previously been used to study, for example, deformation twinning in hexagonal close-packed metals.29-31 Three-dimensional experimental data have been identified as key for the direct validation of computational materials science approaches2,3,23 that are becoming increasingly important for both academia and industry.

II. Experimental Procedure

BT ceramic was prepared with a grain size of approximately 50–70 μm and cut and polished into a cuboid sample of dimensions 300 μm × 300 μm × 400 μm. The sample was mounted in a setup that allows application of a high electric field with minimal risk of dielectric breakdown (additional details can be found in Daniels et al.34).

The 3DXRD experiments were performed at beamline ID11 of the European Synchrotron Radiation Facility (ESRF). For the far-field experiment, which yields domain volume fraction and strain information, the beam was focused to a planar geometry of height 100 μm, illuminating the entire width of the specimen, with an energy of 78.395 keV (Pt edge). The sample was mounted 485 mm away from the Frelon4M detector (ESRF, Grenoble, France) with 2048 × 2048 pixels of 50 μm × 50 μm and diffraction images were collected while rotating the sample in the angular range of 345° about the vertical z-axis in steps of 0.1°. Three adjacent 100 μm layers of the sample were mapped in the as-processed state (F0), at an intermediate electric field strength close to the coercive field (F1) and at a field exceeding the coercive field (F2), with the electric field direction coincident with the rotational z-axis.

For the subsequent near-field experiment, which yields a grain map of the 3D microstructure, an X-ray beam energy of 37,010 keV (just below the Ba K-edge) was used. The beam dimensions were limited to 500 μm horizontally and 100 μm vertically with lenses and slits. Again three adjacent 100 μm layers of the sample were mapped through 360° in steps of 0.1° for the first layer and 0.2° for the remaining two. Near-field mapping employs two detectors simultaneously: the Frelon4M detector mentioned previously at a distance of 223 mm and the first screen of the Riso 3D-detector (DTU, Roskilde, Denmark) with 2048 × 2048 pixels of 1.4 μm × 1.4 μm at a distance of 8 mm.

The collected diffraction image stacks were analyzed using the Fable software suite (2014) (ESRF and DTU), described in detail in Sørensen et al.37 The individual grains were indexed from the far-field data using GrainSpotter,38 and the indexed grain orientations were then used as seeds for the reconstruction of a grain map from the near-field data by means of a 3D generalization of GrainSweeper.39 For each 2 μm × 2 μm ×2 μm voxel in the reconstruction, the seed orientation with the highest completeness ratio of expected to observed number of reflections was assigned. The three adjacent reconstructed layers were stacked along the z-axis—the common poling and rotation axis—in order to obtain the 3D orientation map, and voxels with completeness less than 60% were eliminated from the map. The orientation map was then registered to a grain map by assigning adjacent voxels with pseudocubic misorientations less than 1° to the same grain. Finally, microstructural information, such as grain neighbors, was extracted from the grain map using DREAM.3D (BlueQuartz Software, Springboro, OH).40

One hundred sixty-five unique grains were indexed from the far-field data measured at F0, F1, and F2 using Fable (2014) and GrainSpotter,38 resulting in a list of their orientations and 3D positions, as well as a list of diffraction spots assigned to each grain. The (200)/(002) peaks of these grains were then extracted from the data since these peaks contain the information about the volume fractions, v200, v020, and v002, of the three unique ferroelastic domain variants, d200, d020, and d002, distinguishable with the current method. Each reflection was integrated along the rotation, φ, and the azimuth, η, yielding a radial profile along 2θ, which was then fit with Gaussian peaks centered at the split peak positions 2θmin and 2θmax, as shown for a single grain at F0 and F2 in Fig. 1(a).

If a portion of the grain falls outside of the illuminated volume, the measured peak intensities may no longer be representative of the true domain volume fractions (e.g., if the unilluminated part of the grain contains many domains of the d002 variant), hence grains of this type were excluded from further analysis. After this procedure, domain volume fractions were fit based on the intensity ratios of the (200)/(002) reflections and error bars were estimated as outlined by Oddershede et al.41 for 139 embedded grains.

The non-180° domain switching strain resolved along the poling direction, εp, was then calculated from the extracted domain volume fractions along the unit vector l as:42

\[
ε_p = \left( \frac{c - a}{a^2} \right) \left( v_{200} l_1 + v_{020} l_2 + v_{002} l_3 \right) \frac{1}{3}
\]

where c and a are the tetragonal lattice parameters and, without an independent measure of the cubic lattice parameter, a0 = \sqrt{3}ca, which assumes that there is no volume change at TC. For l = <111> or v200 = v020 = v002 = 1/3 the strain εp = 0 because v200 + v020 + v002 = 1 and l1^2 + l2^2 + l3^2 = 1. The maximum poling strain of 2(c−a)/3a0 is obtained for a single domain with <100> aligned along the electric field direction, while the minimum is (a−c)/3a0. Propagation of error bars on v200, v020 and v002 to an error bar on εp was performed.41

The relationship between the domain volume fractions and εp is demonstrated in Fig. 1(b), where the bars are divided into three parts representing the volume fraction of each of the three non-180° ferroelastic domains, labeled with the domain c-axis misorientation with the poling direction, and the line is εp. The applied electric field results in the expected increase of εp from −0.126% ± 0.003% at F0 to 0.492% ± 0.014% at F2, a direct result of the growth of d002 at the expense of d200 and d020. This change is again evident in the shifting of intensity from relatively even ratios at F0 to a very strong peak at F2 [Fig. 1(a)]. The dominant growth of d002 is expected since it is by far the most favorably aligned with respect to the electric field vector at a misorientation of 10.5° [see inset in Fig. 1(b)].

III. Results and Discussion

(1) The Effect of Thermal Stresses in the As-Processed State

Figure 2 shows the distribution of non-180° domain switching strains for all grains at F0. While the strains
within the polycrystal balance and the mean volume weighted strain of the sample as a whole is \( \varepsilon_p \). \( \varepsilon_0 = -0.002\% \pm 0.001\% \), the as-processed strain state of the sample is heterogeneous at the grain scale and individual grains do not contain equal volume fractions of the three possible ferroelastic domain variants [which would yield \( \varepsilon_p = 0 \) from Eq. (1)]. These observations are consistent with recent micromechanical modeling of related ferroelectric/ferroelastic materials, which show a large spread in intergranular residual stresses even when no field is applied and the average internal stress is zero.43

Heterogeneity in the grain-scale domain volume fractions is expected to result from the anisotropic thermal expansion coefficients of tetragonal BT, and is consistent with simulation results that show a built-in, spatially distributed polarization in the as-processed state.42 The resultant anisotropic ferroelastic strain must inevitably induce large elastic compliance strains at the grain scale to compensate, as observed in powder diffraction studies.1 Additionally, the electrostatic energy associated with ferroelectric domain interactions at grain boundaries must outweigh the increased elastic energy caused by the strain heterogeneity.25

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**Fig. 1.** For the same [001]-oriented grain (a) the typical raw and integrated (200)/(002) diffraction spots at the initial (F0) and final (F2) electric fields, and (b) the change in volume fraction of domains (bars) with poling and the corresponding non-180° domain switching strain along the poling direction (line). Each segment of the bar represents a domain within the grain of interest and is labeled with the misorientation between the domain \( c \)-axis and the applied field direction as illustrated in the inset (field direction out of the plane).
The present experimental results show that while, at the bulk level, the ferroelastic strain response is as expected, i.e., zero at the initial state before increasing upon application of an electric field ($\varepsilon_{p,F1} = 0.088\% \pm 0.001\%$ and $\varepsilon_{p,F2} = 0.135\% \pm 0.001\%$), the ferroelastic strain response is heterogeneous at the grain scale. To study the effect of grain response averaging, the RMS distance to the trend line in Fig. 3(b) was calculated for an average over a group of grains of similar orientation at a variety of group sizes. It was found that the RMS distance was halved for groups of 10 similarly oriented grains as compared to individual grains, which agrees well with our previous measurements of grain-scale heterogeneity. The present work builds on this by combining the domain volume-fraction information with the 3D microstructure grain map, reconstructed from the near-field data and shown in Fig. 4, where individual grains are colored by the non-180° domain switching strains at (a) $\varepsilon_{p,F0}$ and (b) $\varepsilon_{p,F2}$, extracted from far-field data. The combination of these two results enables a correlative study of ferroelastic strain response with microstructural features of the grains themselves as well as their local neighborhoods. Figure 5(a) shows the effect of grain size on ferroelastic strain response relative to the trend line in Fig. 3(b). The grain diameter here is calculated from the volumes derived from the grain map by assuming that the grains are spheres. The calculated correlation coefficient of 0.1 indicates no significant correlation between grain diameter and ferroelastic strain response. While previous experiments and simulations have demonstrated the effect of average sample grain size on domain switching behavior and material response, this effect does not appear to be present at the individual grain level of the current coarse-grained sample, consistent with the fact that scaling effects become significant only at grain sizes below 10 µm.

It is well-known that the boundary conditions experienced by a grain at the surface of the sample are different from those of a grain in the bulk. To investigate how this is reflected in the current experimental results, the distribution

![Fig. 2. Distribution of non-180° domain switching strains in the initial as-processed state of the barium titanate sample. The distribution width is an indicator of the grain-scale strain heterogeneity in the as-processed state.](image)

In the present dataset, there is even evidence of such local intragranular lattice strains. For instance, all the raw data in Fig. 1(a) have more intensity between the two peaks corresponding to $2\theta_{\text{min}}$ and $2\theta_{\text{max}}$ than justified by the combined tails of the peaks, indicating a lattice strain distribution within the domains that tends toward the common pseudocubic orientation of the domains. This scattering has been speculated to result from strain compatibility at the domain boundaries.

Fig. 3. (a) The orientation of the 139 indexed grains color coded according to the ferroelastic strain response from step F0 to F2, and (b) $\varepsilon_{p,F2} - \varepsilon_{p,F0}$ as a function of $\cos^2\phi_{100}$ where $\phi_{100}$ is the misorientation between the electric field vector and the closest <100> direction in the given grain. The trend line in (b) corresponds to the average behavior expected for a given grain orientation.

(2) Strain Response Heterogeneity

The inverse pole figure in Fig. 3(a) shows that the grains are randomly oriented, as expected for the ceramic processing technique used, a result also confirmed by a Mackenzie type analysis. The color represents the difference in non-180° domain switching strain between the F0 and F2 states, from here on termed the ferroelastic strain response. From Fig. 3(a) it is clear that there is a general trend toward maximum and minimum ferroelastic strain response occurring at grain orientations with a <100> and <111> direction lying close to the electric field vector, respectively, as expected from the definition of $\varepsilon_p$ in Eq. (1) as well as from previous powder diffraction results. However, in addition to the observed first-order correlation between grain orientation and ferroelastic strain response there are significant variations, or second-order perturbations, within groups of grains with similar orientations. To highlight these variations, Fig. 3(b) shows the ferroelastic strain response as a function of $\cos^2\phi_{100}$, where $\phi_{100}$ is the misorientation between the electric field vector and the closest <100> direction in the given grain. Although it would be reasonable to think that grains with an initially negative strain would have more significant correlation between grain diameter and ferroelastic strain response there are significant variances of cos $2\phi_{100}$ with similar orientations. To highlight these variations, second-order perturbations, within groups of grains and ferroelastic strain response there are significant variances. In the present dataset, there is even evidence of such local intragranular lattice strains. For instance, all the raw data in Fig. 1(a) have more intensity between the two peaks corresponding to $2\theta_{\text{min}}$ and $2\theta_{\text{max}}$ than justified by the combined tails of the peaks, indicating a lattice strain distribution within the domains that tends toward the common pseudocubic orientation of the domains. This scattering has been speculated to result from strain compatibility at the domain boundaries.
of ferroelastic strain response deviation from the trend line is shown in Fig. 5(b) for the 50 bulk grains (dark) and 89 surface grains (light). The average of the bulk distribution is $0.01\%$ with an absolute spread of $0.11\%$, while the average and absolute spread for the surface grains are $0.01\%$ and $0.07\%$, respectively. This implies that there is no significant difference between bulk and surface grains in terms of their mean deviation from the linear trend, however, the spread of response magnitude in the surface grains is significantly lower than for bulk grains, with the probability of equal spread in the two distributions calculated as $0.06\%$ for all data points and $3\%$ for data points within the range of $\pm 0.2\%$ from the trend line. A more homogeneous response is expected for surface grains since they experience fewer constraints due to the relaxed elastic and electrostatic boundary conditions at the sample surface.

The grain size and location in the sample are also intimately linked with the number of contact neighbors that a grain has, i.e., small/surface grains generally have fewer neighbors than large/bulk grains. However, the correlation coefficient between the ferroelastic strain response away from the trend line and the number of neighbors is merely 0.02, hence it is concluded that neither the grain diameter nor number of neighbors nor the location of grains within the sample have any significant effect on the ferroelastic strain response of individual bulk grains within the polycrystalline BT sample.

### (3) Effects of the Grain Neighborhood

In order to experimentally assess the effects of grain interactions, two bulk grains, A and B, have been selected for further analysis. Some of their properties and those of their neighbors have been summarized in Table I. The grains were chosen specifically to be of similar orientation relative to the electric field vector ($5^\circ < \phi_{100} < 15^\circ$ for both) and similar size (both have grain diameters slightly smaller than the sample average of 64 $\mu$m), yet exhibiting significantly different ferroelastic strain responses. Grain A, which was used as an example in Fig. 1, experiences a relatively large ferroelastic strain response, while grain B experiences a ferroelastic strain response approximately equal to the average of all grains of similar orientation.

The local environments of grains A and B and their neighbors, shown in Fig. 6, have been analyzed in detail in order to quantify various aspects of the grain neighborhoods. Both grains have a similar number of neighbors, 10 for A and 14 for B. This is comparable to the average number of 11 neighbors per grain in the sample. The neighbors of grain A are on average larger than those of grain B, which is expected considering the similar grain diameters of A and B and the difference in number of neighbors. However, this average does not make clear that grain A has two very large neighbors (diameters 157 and 170 $\mu$m), while the largest neighbor of grain B has a diameter of only 125 $\mu$m.

The Mackenzie plot in Fig. 7(a), generated for all neighboring grain pairs in the sample, shows that for a random grain in the polycrystalline BT sample it is statistically likely that the grain is surrounded by neighbors that come very close to the average random texture of the entire sample.
However, Fig. 7(b) showing the distributions of neighbor misorientations for grains A (dark) and B (light), clearly demonstrate that in reality an individual grain sees a local neighborhood that is significantly different from the statistical average, as also indicated by the mean and spread of the two distributions given in Table I. So, while the bulk macroscopic properties and response are predictable, at the grain-scale the material behavior is far more localized in nature, as has been predicted from multigrain models and simulations.

Figure 6 also indicates that it is not only grain A that has a larger strain response than B, but so does its immediate neighborhood as a whole. A reason could be that grains in the neighborhood of grain A are generally more favorably aligned relative to the field vector. Another possible explanation could be the presence of the so-called Σ3 boundaries with neighbor misorientations close to 60° that have been suggested to play a special role in connection with continuity of ferroelectric domain structures across grain boundaries. However, neither A nor B exhibit any such neighbor misorientations.

The two grains are not representative of the sample texture as a whole, but rather selected to represent extremes of the response distribution caused by intergranular interactions. Consideration of such local environments is essential for predicting the local maximum stresses that are important for the reproducibility and reliability of the ferroelectric response in a ceramic. Depending on the actuation mechanisms within a specific ceramic material, the stress concentrations may vary significantly due to the degree of strain anisotropy. In cases where large intergranular stresses exist, these may accelerate the initiation of cracks that lead to fatigue and failure of the material.

We note that the spatial resolution of the current experimental results does not enable a distinction between individual domains within grains, only the domain volume fractions. Hence, a direct comparison between experiment and model is best performed for models not concerned with the specific domain structure. In particular, crystal plasticity-based finite elements models, where the incremental transformation by domain wall motion is analogous to incremental slip on a slip system for deformation studies, would greatly benefit from the present data both for input and validation purposes. There the state of the sample is described by the volume fractions of each domain type, making the modeling results directly comparable with their experimental counterparts.

Previous modeling attempts have considered the effect of grain orientation, assuming that all grains are of equal size with all domains of equal volume fraction in either 2D or 3D, while the present study has clearly demonstrated that this is not the case. The first 3D simulation of faceted grains of variable size (made by Voronoi tessellation from a random texture) represents an important step toward modeling real structures. In some cases, real 3D microstructures measured by serial sectioning and EBSD have been used as input, however, it is most often the case that microstructural input relies on simulation and extrapolation of selected grain morphology and orientation measurements.

Macroscopic measurements such as hysteresis loops and piezoelectric constants are often used for validation. While these are useful measures, it has been shown that such macroscopic measurements can agree without the model capturing the local interactions that are life limiting for the component. The dataset presented in this work is a reconstruction of a real, three-dimensional sample, where the domain volume fractions of individual bulk grains can be tracked from the initial state and with subsequent application of an electric field. Using the microstructure as direct input to finite element or other simulations, models can be validated on the grain scale, in addition to macroscopic measurements.

### IV. Conclusions

Using 3DXRD grain mapping, the grain-scale domain volume fractions in 139 grains of a BT ceramic were successfully extracted, and from this information the ferroelastic strain response of each grain was derived. In agreement with previous experiments (i.e., powder diffraction), it was found that tetragonal domains with c-axes more closely aligned with the poling direction ([001] aligned grains) tend to grow with the application of an electric field, thus resulting in larger ferroelastic strain responses. Although the average ferroelastic strain response of the sample was as expected, large
validity of grain-scale modeling efforts, and provide field is applied. These results are of critical importance both in the as-processed state of the sample and when an electrical cause of the observed grain-scale strain heterogeneities both and ensure stress equilibrium. All of these results suggest that strains within domains, probably mainly in the grain-bound-
tion, size and number of neighbors that were found to exhi-
bit very different ferroelastic strain responses. In addition, the diffraction data showed evidence of substantial lattice bit very different ferroelastic strain responses. In addition,
variations exist in the behavior of individual grains of similar orientations. An important observation is that these variations are not restricted to the ferroelastic strain response, they were in fact observed already in the initial as-processed state of the sample. It is suggested that these grain-scale variations arise from local strain and electrostatic neighborhoods being highly heterogeneous within the bulk polycrystal. The neighborhood heterogeneities were clearly demonstrated from the grain map for two selected bulk grains of similar orientation, size and number of neighbors that were found to exhib-
Fig. 7. Mackenzie type plot (a) for all misorientations of neighboring grains within the sample volume and the theoretical distribution of random orientations (line), and (b) neighbor misorientation distribution for grain A (dark) and grain B (light). All misorientations are calculated as the minimum rotation angle around an arbitrary axis needed to make the crystal lattice of a grain coincide with the crystal lattice of each of its neighbors.

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