Domain phenomena in single crystalline and ceramic ferroics: unresolved and attractive problems

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Abstract

Domain-related properties of ferroic materials offer a number of application aspects. However, some of the involved domain characteristics have not been fully explained and may initiate an interesting field of further research. Here we specify and discuss several such still unresolved problems related to single crystalline and ceramic samples. Those concerning static domain pattern include the topic of domain wall thickness and of possible macroscopic wall properties, macroscopic properties of ceramic samples *depoled* in different ways and application of multidomain systems in surface acoustic wave devices. Discussed problems related to dynamic domain phenomena include the issue of visibility and thickness of moving domain walls, the effect of nucleation and the topic of extrinsic contributions to macroscopic properties of multidomain samples.
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1. Introduction

Samples of ferroic materials can be produced in a number of assemblage types; as single crystals, ceramics, polymers, glass ceramic, composites and thin (thick) films. All of them may contain domains since the existence of the latter is dictated by the symmetry reduction specifying the phase transition. Here we address only single crystals and ceramics. Understandably, most unsolved problems in single crystals indicate open questions even in ferroics of other systems. In contrast, many essential domain-related problems in non-single-crystalline types of samples are connected with boundary conditions or composition-nonhomogeneity aspects that are irrelevant in single crystals.

Some interesting domain-related features remain unsolved; some are very difficult to be solved,

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some have not been readdresses because there are no application-related aspects; and some were not accessed because no proper experimental techniques were available, etc.

Out of a number of such problems we have selected just a few which we believe would be worth addressing whether experimentally and/or theoretically. Roughly speaking, all open issues can be also classified into those connected with static aspects of domain patterns and those related to dynamic aspects of wall motion and switching. Here we have made a narrow selection of problems related to both these viewpoints.

2. Domain walls and domains at rest

2.1. Domain wall thickness

The problem of wall thickness d_w was repeatedly addressed starting from the early stages of ferroelectric domain investigations. From the recent points of view of application aspects it may play a substantial role: when using specific properties of walls their thickness d_w would be essential, in addition to the domain density.

Here we shall concentrate on nonferroelastic walls in ferroelectrics; the problem was probably first discussed for the classical ferroelectric $BaTiO_3$. The early models [1] and calculations [2] indicated that d_w of its 180° walls is just a few lattice parameters; in most discussions of properties of multidomain samples this extremely small wall thickness is generally assumed. And indeed a very large number of TEM investigations (see, e.g., Ref. [3,4]) of this material indicated the d_w values between 10 and 100 Å. Also in some other perovskites this small wall thickness was confirmed. For TGS, a ferroelectric of a significantly different composition, a similar value $d_w \cong 80$ Å was reported [5], based on atomic force microscopy data.

The mentioned information on d_w has been primarily based on observations of either extremely thin samples or of thin surface regions of crystals. Repeatedly, much thicker domain walls have been detected using X-ray topography which can reveal walls inside a sample; e.g., d_w of the order of 1 μm was reported for $NaNO_2$ [6], $SC(NH_2)_2$ [7] or TGS [8,9]. While data based on this method have been disputed because of its possible limited spatial resolution, we have to admit that reliable reports on d_w values inside samples based on other methods are missing.

We wish to mention two examples indicating the possibility of existence of thick nonferroelastic walls. Kawata et al. [10] investigated 180° domains in thick *a*-oriented plates of defect-free $BaTiO_3$ by surface reflection topographs and optical micrographs. Their data refer to static situations. Antiparallel domains terminate by *oblique* optically visible 180° walls. The analysis of X-ray and optical data showed that d_w values were of the order of 1 μm. The ε_{23} strain component was found to be responsible for the optical contrast of oblique walls in which **P** makes an angle 1–2° with $\mathbf{P}_S$. The origin of the significantly increased thickness cannot be of a simple mechanical-compatibility origin. It could be related either to the structural aspects of walls of $(h,k,l \neq 0)$ orientation or to the fact that they may be charged. The latter possibility seems to be less probable since the mentioned investigations did not proceed fast and charge compensation could be assumed. Thus these results seem to indicate that an inclined mechanically fully compatible wall can be in fact significantly thicker than generally assumed.

It has been assumed from the early stages of investigations that wall thickness could be strongly

influenced by lattice defects; either walls can be assumed to prefer locations with high density of defects or defects could diffuse into the existing walls. The problem was repeatedly addressed for ferroelastic walls. Darinskii and Sidorkin [11] were probably the first to address theoretically the influence of defects on the observed d_w of a nonferroelastic 180° wall. They defined the effective wall thickness $d_{w,eff}$ as the width of a layer containing the domain wall which is locally deformed by point defects. For defect concentration 10^{18} cm^{-3} $d_{w,eff}$ increased from a few unit cells to 300 Å, and for linear defects parallel to the polar direction $d_{w,eff}$ might reach the value of 3000 Å. When the charge of local defects is specifically taken into account [12], the broadening of walls has distinctive features; in particular, the wall can contain extended areas of curved sections where it makes an angle with the $\mathbf{P}_S$ direction.

Much more recently the problem was finally addressed also experimentally, by Gupta and co-workers [13,14]. Indeed it has been shown that the thickness of 180° nonferroelastic wall can be considerably influenced by the presence of lattice defects. In congruent crystals which are Li-deficient, the 180° walls formed at room temperature have been reported to reveal very specific properties: large birefringence within regions of thickness of the order of 1 μm [13,14]. Based on these and some macroscopic properties, strain components connected with walls were evaluated [15] and the existence of strong local electric fields was suggested. In stoichiometric crystals strain and birefringence tend to disappear; thus these effects and the large wall width are clearly connected with lattice defects. Kim et al. [16] used X-ray synchrotron diffraction imaging to study strain components when crossing the wall and found that they represent wall thickness of several μm in both congruent $LiNbO_3$ and $LiTaO_3$.

To summarize briefly, we can state that two features have been proved to influence considerably the thickness of a static 180° nonferroelastic wall: the wall orientation and lattice defects. Although representative types and properties of defects resulting in wide domain walls do not seem to have been specified, the latter feature in particular may open a new field of applications of ferroelectric crystals with a large wall density; a new area of domain wall engineering [17,18]. $LiNbO_3$ samples with appropriate non-stoichiometry and with engineered dense domain pattern might offer macroscopic properties new in the sense of symmetry and/or in the sense of values of some material coefficients.

2.2. Domain wall symmetry

Let us now pay attention to the 'ideal' wall structures, uncharged and uninfluenced by defects. Can they offer some macroscopically detectable properties differing from those of a single domain material? It would be an interesting question from the practical point of view if the wall width and/or their density would provide a non-negligible sample volume. The lowest-order tensor which could represent the macroscopic properties of walls is a vector, polarization. Thus one asks the question whether a domain wall in a nonferroelectric sample can carry a dipole moment. A positive answer to this question was suggested by Walker and Gooding [19] who showed theoretically that domain walls in the room temperature phase α of quartz can carry a dipole moment. Between the parent phase β and phase α there exists an incommensurate phase containing triangular domain systems of two variants; in one variant domain walls carry dipole moments along the 3-fold axis, in the other the dipoles are of opposite direction. Snoeck et al. [20] showed that applied field results in changes of this domain pattern and could be interpreted as a result of sidewise motion of the polar walls. Another indirect proof of polar walls was presented by Jorio et al. [21] for crystals of Cs_2HgBr_4. Raman

scattering and infrared reflectivity dates led to the conclusion that domain walls carry a dipole moment lying in the wall plane. We are not aware of other data representing polar walls; symmetry-based analyses [22,23] indicate that they may be present in both nonferroelastic and ferroelastic nonferroelectric species and recent theoretical study [24] predicted that even antiphase domain walls may be polar, in $SrTiO_3$ crystals in particular.

Polarity of domain walls in defect-free samples of classical ferroics does not seem to have been unambiguously proved. However, when an attribute is allowed by symmetry, it may be just a matter of time and of finding the proper material to demonstrate its existence.

2.3. Applications of static domain systems: SAW

Let us now mention two specific domain-related application aspects. Surface acoustic waves play an important role in some applications of piezoelectric crystals and $LiNbO_3$ plays an essential role. Since two domain states of this material differ in elastic coefficients and in signs of piezoelectric constants, different domain geometries can be considered influencing the propagation of SAW. Here we wish to point out that even a simple system of two domains D1 and D2 (Fig. 1) can offer properties which may be useful for SAW applications. The SAW propagates from the interdigital transducer IDT1 to IDT2. Each domain is provided with electrodes (A1, A2) so that electric fields perpendicular the the propagation direction of SAW can be applied. As shown before [25], by static or slowly changing electric field it is possible to influence the group velocity of SAW in either of the domains and this can be achieved in each domain in a different way. The effect can be described by the change of elastic and piezoelectric coefficients: $c* = c + aE$, $e* = e + bE$. As a result, two time delay lines are produced on one substrate. On the domain wall considered here to be perpendicular to the signal propagation, significant reflection of the surface acoustic wave can occur [26]. Interference of the original and reflected SAW can be influenced by the electric field in domain D1. The whole system can be used for signal processing.

2.4. Static domain systems in depoled ceramic samples

Another interesting domain-related aspect concerns ferroelectric ceramics. Consider we wish to depole a previously poled ceramic sample. This can be achieved in several ways; the resulting
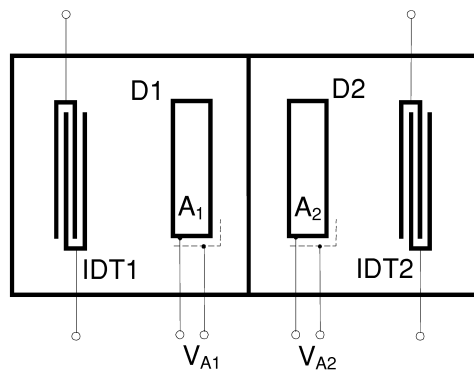

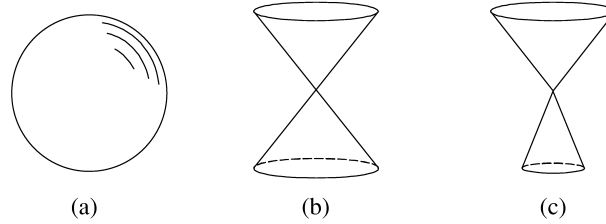Fig. 1. A two-domain system for SAW applications; see text.

Fig. 2. Representation of symmetries of depoled ceramic samples: (a) thermally depoled sample; (b) sample depoled by ac field; (c) sample depoled when traversing the hysteresis loop.

symmetries are represented in Fig. 2. Thermal depoling (a) would result in the $\infty\infty m$ symmetry with zero piezoelectricity. When a low frequency ac field is applied whose amplitude is reduced with time, it can be expected that the arrangement of $\mathbf{P}_S$ directions results in zero value of $\bar{P}$ but does not return to a homogeneous distribution of spherical symmetry; we would rather expect the vector distribution corresponding to a double-cone of symmetry ∞/mm. Tensorial properties of a sample depoled in this way (b) do not seem to have been investigated. Let us note that tensorial properties in the samples depolarized by the two mentioned ways would differ, by symmetry; e.g., in tensors of symmetries V^2, $[V^2]$, $\varepsilon V[V^2]$, $V[V^3]$ and $[[V^2]^2]$ [27].

Yet a ceramic sample can be depolarized in still another manner: a $P(E)$ hysteresis loop is slowly traversed. When the loop crosses the horizontal axis ($\bar{P}=0$), it can be expected that the domain geometry in this state will differ significantly from those depoled by the above mentioned methods. Can a ceramic sample depoled in this way be piezoelectric? This question was discussed by Luchaninov and co-workers [28,29]; and the answer is positive. Here we present the simplest approach to the problem. Obviously, in this state the $\mathbf{P}_S$ distribution could be represented by an asymmetric double cone: the distribution of $\mathbf{P}_S$ vectors around the previous poling direction can be narrower than the distribution around the opposite direction. Thus the ∞mm symmetry would still be retained although in the arrived state $\bar{P}=0$. The sample could still exhibit piezoelectric properties. Investigations of ceramic samples depoled in the three mentioned ways might offer interesting information.

3. Dynamic aspects: moving domain walls and nucleation

3.1. Visibility and thickness of moving 180° walls

Above we have addressed the problem of the thickness of 180° walls at rest. However, the issue of the thickness and structure of moving 180° walls may be found even more interesting. It was discussed a long time ago and seems to be an almost forgotten issue although it can play a substantial role in ferroelectric memories.

If nonferroelastic 180° walls in a high quality $BaTiO_3$ crystal were as thin as discussed above, they could not be observed optically. However, Miller and Savage [30] reported that during polarization reversal 180° domains are visible when viewed along the c-axis between crossed polaroids. Nakamura et al. [31] showed that when a field is applied to an a-oriented $BaTiO_3$ crystalline plate, slightly oblique static walls can be observed for a short time in a polarizing microscope as bright lines.

Theoretical discussion [32] showed that the effective thickness of a moving wall with average ($hk0$) orientation may be very large because of the nucleation process. However, it did not include any aspects which would lead to an optical contrast; either a modified symmetry of domain walls or an inclined wall orientation. The visibility of sidewise moving walls viewed along the *c*-axis was discussed in detail by Kobayashi et al. [33]. They showed that the optical contrast in the zones of moving walls indicated birefringence which increased when the wall moved faster; after the wall had stopped moving, it took some time for the contrast to disappear. Using X-ray diffraction they confirmed that the symmetry of a moving wall is influenced by a monoclinic deformation which results also in optical visibility of the wall. We may note that the shear strain specified by Kawata et al. [34] in a static wall is about one order of magnitude smaller than that observed by Kobayashi for the moving wall. Independently, Kobayashi [35] also demonstrated that moving 180° walls in $BaTiO_3$ are visible even when viewing along the *a*-axis.

In the observations referred to above, $BaTiO_3$ crystals were used grown by the original Remeika's method. It might be interesting to readdress the problem using crystals grown by Linz's top-seeded flux method.

It appears that the presently applied theories of switching do not take into account the specific structure and thickness of moving domain walls. This applies in particular to switching in thin films where the moving wall thickness may in fact be comparable to the sample thickness. In fact we miss even some basic experimental data on antiparallel switching processes in thin film samples of materials presently used in memory devices.

3.2. Nucleation

It was accepted in the early of investigations that polarization reversal in classical ferroelectrics proceeds in three steps: nucleation of domains with antiparallel polarization and their forward growth followed by sidewise expansion. Experimental data on which the switching models are based are of two kinds: macroscopic properties—hysteresis $P(E)$ or switching current $i(t)$ dependencies—and observations of domains during the switching process. We now address the question whether the nucleation process, meaning random formation of small domains with antiparallel $\mathbf{P}_S$, really plays a role.

It is well known that Landauer [36] pointed out in the early stages of investigations of $BaTiO_3$ that the critical nucleation energy ΔU^*, in fact a potential barrier to be overcome to form a stable nucleus with antiparallel polarization, is too high ($10^8 kT$) to allow for any realistic nucleation rate. It is a result of combined contributions of depolarizing field energy and surface energy of the nucleus. Even if the depolarizing field were compensated by free charges from the electrodes, the surface energy term alone is too large ($10^5 kT$) for nucleation aided by thermal fluctuations to be possible. Only in applied field orders of magnitude higher [37] than the real field required for switching the activation energy could be reduced to $10kT$. For PZT, in the applied field 100 kV/cm, Tagantsev's estimation (A.K. Tagantsev, personal communication) of the barrier was $10^3 kT$. To solve this problem, a number of experimental investigations were performed and some theoretical approaches were proposed.

Understandably, many attempts have been devoted to analyzing the nucleation stages experimentally. Stadler and Zachmanidis [38] used etching to reveal domains formed in $BaTiO_3$ after the application of several pulses of field; to each cylindrical domain a nucleus was attributed. Depending on the sign of the 'internal bias', 10–30% of observed domains were 'repeaters', indicating nucleation

occurring at predetermined sites. Thus the remaining percentage of nuclei might be formed chaotically, based on statistical probability. Total nucleation rate dn/dt was found to be proportional to $E^{1.4}$. It has to be pointed out these results were obtained by etching the samples a long time after the application of field. Therefore the obtained data, while interesting, need not represent the nucleation process.

Much more recently, Gruverman et al. [39] investigated nucleation in lead germanate; the specific advantage of this material is that its species $\bar{6}-Pd-3$ allows for optical observation of nonferroelastic domains in real time, due to optical activity. The authors specified three time intervals: t_d in which the first 'through' domains appear; it was found to depend on the quality of sample surface; t_{tr} during which the nucleation rate increases till a steady state regime (constant value of dn/dt) is reached; this regime lasted for more than 2 ms. The essential point is that the authors used stroboscopy to observe the nucleated and grown domains. The possibility to use this method indicates that the nuclei occurred repeatedly at the same spots which must have been predetermined by defects. It would be interesting to study crystals of the same material grown with the aim to control their defect structure and to reach maximum purity.

The fact that nucleation has been repeatedly observed at surfaces of samples need not be connected with the ideal nucleation energy considerations. Preparation of samples usually includes mechanical modification of their surfaces which may lead to facilitating the nucleation or even to built-in nuclei. It is interesting that this may play a substantial role even in nonferroelastic samples. Venables [40] showed that surface polishing of $LiTaO_3$ samples leads to formation of dislocations with [001] oriented Burgers vectors, which served as nuclei of antiparallel domains; their densities reached a value of 10^7 per cm^2.

Other data are available concerning the number of nuclei and its time dependence. However, because of the limited spatial and time resolution to obtain information on the *real* nucleation process is difficult. To refer to small observed domains as to nuclei may be questionable.

The mentioned estimations and data point to the question whether the nucleation process plays an essential role in the observed macroscopic properties, like hysteresis loops or switching currents. It was shown already by Janta [41] that to interpret the $P(E)$ loop does not really require the nucleation stage. In his model, the presence of lattice defects results in the existence of residual domains. Using the $\exp(-\alpha/E)$ law for wall velocities together with the domain coalescence described by the Avrami theorem, hysteresis loops can be calculated whose amplitude and frequency dependencies agree well with experimental data. Ishibashi and Takagi [42] developed a phenomenological theory of the switching current in pulse field. They solved the problem under two different assumptions: (a) the nucleation rate is constant throughout the switching period; (b) latent nuclei of a given number are assumed to exist and no further nucleations occur. The available experimental data did not allow to reach ultimate conclusions regarding the role of the nucleation process; however, the assumption of latent nuclei did not contradict any observed properties.

The problem of nucleation is still repeatedly addressed. A new theoretical approach was offered by Molotskii et al. [43]. They consider the following mechanism for the formation of nuclei. When E is applied, electrons tunnel from an electrode to states of neutral defects aggregates. Those captured by defects form single-electron fluctuons; a fluctuon is a bound state of an electron (or a hole) with a fluctuation of local polarization. The coalescence of such fluctuons leads to the appearance of stable multielectron fluctuons which may be regarded as repolarization nuclei. While the energy of an equilibrium fluctuon is found to be negative, there is still a barrier of the order of several eV which is

expected to be overcome by thermal fluctuations. Let us stress that even the fluctuon model assumes that lattice defects play a role in the whole process.

Recently, Urenski et al. [44] referred to the offered models [36,43] of how the activation energy could be reduced by assuming domain nucleation on the polar crystal surface where the required charge carriers may be injected from the switching electrodes. They pointed out that when studying $KTiOPO_4$ crystals we have the possibility to compare two situations. At room temperature the material possesses superionic conductivity (mobile K ions) allowing for compensation of depolarizing field. This may lead to easy nucleation in the bulk of the crystal. In contrast, at low temperatures the material is a true dielectric for which nucleation at the electrodes may be more probable. Indeed an obvious difference was observed in these two temperature regions: In the dielectric state the domains are nucleated at the electrodes and grow straightforwardly from the top to the bottom surface. In the superionic state the observed domains appear in the crystal bulk and on growing are strongly widened. These interesting observations do not rule out the possibility of built-in nuclei.

To conclude, all results support or at least do not exclude the assumption that in the observed switching processes the ideal defect-independent nucleation is irrelevant. However, the possibility cannot be excluded that in different materials the nucleation processes are of different origins. It would be of interest to concentrate further research on one crystalline material grown with well defined concentrations of defects and working with samples whose surfaces would be prepared in a well controlled way.

At the end of this section, we wish to point out that the problem of nucleation is closely related to the aspect of 'writing' domains which is essential for memory applications of thin films. Eng et al. [45] addressed the issue also for thick samples, using scanning force microscopy. They showed that local polarization reversal can be induced by applied field in any required spot (specified with the accuracy of about 10 nm). Till now such a nucleation process in a highly nonhomogeneous field has not been addressed theoretically. It has been suggested [46] that even higher order ferroic effects might be involved.

3.3. Modes of extrinsic contributions

The last problem we wish to mention is the classical issue of domain wall (extrinsic) contributions to small-signal macroscopic properties of ferroics. In a nonferroelastic ferroelectric, wall shifts by Δx increase the measured permittivity. Assuming the applied field amplitude $E_0 = 1$ V/cm and a realistic density of walls at room temperature, in typical crystals of TGS Δx at 1 kHz is of the order of 0.1 Å. In a ferroelastic ferroelectric, wall shifts by Δx lead to extrinsic parts of permittivity, elastic constant and piezoelectric coefficient. Estimations for RbH_2PO_4 based on the data of all three properties lead to Δx of the order of 0.1–1 Å [47], again for $E_0 = 1$ V/cm, at a below the resonance frequency.

Let us consider the 'ideal' wall thickness just a few unit cells, referring to TEM observations or to wall theories, both macroscopic or microscopic. Then such small values of shifts Δx represent rather a change of the structure of walls rather than their uniform displacements. The problem can be solved in two different ways. First, the walls inside thick samples may be actually much thicker; this would be in agreement with some observations mentioned above. Alternatively, the walls can be pinned by defects and the mentioned Δx represents just a spatially averaged value. Yang et al. [48] used near field scanning optical microscope to investigate domain walls in thick samples of $LiTaO_3$. When electric field was applied, curved sections of 180° walls were clearly observed, demonstrating that

even nonferroelastic walls can be pinned by defects. The curvature radius corresponded to the applied field.

This observation showed clearly that—at least in nonferroelastic ferroelectrics—the Δx calculated from the measured macroscopic quantities is an averaged value. In addition, because the internal structure of a bent wall may be different from that of a planar wall, the extrinsic contributions may combine two aspects—volume changes of positively and negatively oriented domains as well as changes of the internal wall structures, due to bending.

The problem of extrinsic contributions has been repeatedly addressed for several decades; yet it still offers an area of interesting research which might also open new and essential application aspects.

4. Concluding remark

At the end we wish to stress that in many issues mentioned above the role of crystal lattice defects is expected to be essential; yet we still know very little about the background of defect-induced phenomena. Growing defect-free and defect-defined samples will be expensive but surely very interesting.

We have mentioned only a small selection of interesting and open problems related to domain phenomena in ferroics. There are many other issues worth addressing. They offer interesting physical aspects and material properties and we expect that the domain research will belong to very active and strongly supported research areas in the coming years.

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