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Effect of wall thickness on the ferroelastic domain size of BaTiO<sub>3</sub>

**Authors: G. Catalan · I. Lukyanchuk · A. Schilling · J.M. Gregg · J.F. Scott**

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Abstract Extremely regular self-organized patterns of  $90^\circ$  ferroelastic domains have been reported in free-standing single crystal thin films of ferroelectric  $\text{BaTiO}_3$ . Lukyanchuk et al. [Phys Rev B 79, 144111 (2009)] have recently shown that the domain size as a function of thickness for such free standing films can be well described assuming that the domains are due to stress caused by a surface tension layer that does not undergo the paraelectric–ferroelectric transition. From the starting point of Lukyanchuk’s model, it is shown here that the “universal” relationship between domain size and domain wall thickness previously observed in ferroelectrics, ferromagnets and multiferroics is also valid for ferroelastic domains. Further analysis of experimental data also shows that the domain wall thickness can vary considerably (an order of magnitude) from sample to sample even for the same material ( $\text{BaTiO}_3$ ), in spite of which the domain size scaling model is still valid, provided that the correct, sample dependent, domain wall thickness is used.

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2 **Effect of wall thickness on the ferroelastic domain size of BaTiO<sub>3</sub>**

3 **G. Catalan · I. Lukyanchuk · A. Schilling ·**  
4 **J. M. Gregg · J. F. Scott**

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7 **Abstract** Extremely regular self-organized patterns of  
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16 Lukyanchuk’s model, it is shown here that the “universal”  
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18 ness previously observed in ferroelectrics, ferromagnets  
19 and multiferroics is also valid for ferroelastic domains.  
20 Further analysis of experimental data also shows that the  
21 domain wall thickness can vary considerably (an order of  
22 magnitude) from sample to sample even for the same  
23 material (BaTiO<sub>3</sub>), in spite of which the domain size  
24 scaling model is still valid, provided that the correct,  
25 sample dependent, domain wall thickness is used.

**Introduction**

An important feature of all ferroic materials—including ferroelectrics—is that they can display regions of different polarity (domains) separated by domain walls. The domains often arrange themselves in surprisingly regular patterns in the form of periodic stripes. The period of the domain scales non-linearly with the size of the device: specifically, for thin films, the domain size typically grows proportional to the square root of the film thickness [1–4]. This square root dependence has the consequence that, on approaching the nanoscale, the domain size decreases very rapidly and thus the number density of domains—and domain walls—can rapidly become very large. The practical consequences of these are twofold: first, as emphasized by Scott [5], the switching characteristics of a ferroelectric device are ultimately limited by the ability of domains to nucleate and grow, so the scaling of domain size is closely correlated with the scaling of switching properties such as coercive field [5]. Second, domain walls have functional properties distinct from those of the domains themselves: superconductivity in the ferroelastic walls of non-superconducting WO<sub>3-x</sub> [6], polarization in the twin walls of paraelectric SrTiO<sub>3</sub> [7] and conductivity in the ferroelectric walls of insulating BiFeO<sub>3</sub> [8] are some examples. Given a sufficiently large volume density of domain walls, then, the functional behavior of the sample may be dominated not so much by the properties of the domains as by those of the domain walls.

In this context, recent studies by groups in Belfast, Cambridge, and Amiens have been aimed at studying and rationalizing the formation of regular domain patterns in “ideal” samples of nanoferroelectrics. The experimental study, pioneered by Gregg et al. [9, 10], is based on the idea that ferroelectric thin films need not be grown on a

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62 substrate, but can instead be “carved out” of a bulk single  
 63 crystal of, e.g., BaTiO<sub>3</sub> (BTO) using a focussed ion beam.  
 64 When placed under a transmission electron microscope  
 65 (TEM), such single crystal lamella display astonishingly  
 66 regular stripe domain patterns, with the polarization in-  
 67 plane alternating at 90° between the *x* and *y* axes [11]. The  
 68 fact that the polarization is entirely in-plane means that  
 69 depolarization effects do not play a significant role and that  
 70 the domain configuration and size must respond to elastic  
 71 constraints instead [4]. Extension of the thin film work to  
 72 non-planar structures such as nano-wires [12–14] and  
 73 nano-dots [15] invariably shows the same feature: the  
 74 appearance of ferroelectric–ferroelastic domains in regular  
 75 patterns and in a configuration such that the macroscopic  
 76 shape of the sample tends to be preserved.

77 Until very recently, however, the shape-preserving  
 78 aspect of the domains had remained rather intriguing. After  
 79 all, if there is no external constraint on the shape of the  
 80 sample (such as that which may be expected, for example,  
 81 from clamping to a rigid substrate [4, 16, 17]), why should  
 82 the samples want to keep any specific shape? In theory, the  
 83 minimum energy configuration for a free-standing film  
 84 should be one with a single domain (so as to minimize  
 85 energy from domain walls) and with the polarization  
 86 pointing in-plane (so as to minimize depolarization). A  
 87 possible explanation proposed recently by Lukyanchuk  
 88 et al. [18] is that the domains appear in order to relieve the  
 89 stress generated by an encapsulating surface layer that does  
 90 not itself experience the structural phase transition. Thus,  
 91 when the interior of the films becomes ferroelectric, it  
 92 automatically experiences the stress imposed by the  
 93 untransformed surface layer. This model reproduces well  
 94 the observed dependence of domain size on lamellar  
 95 thickness. However, it also poses new questions: (i) is this  
 96 surface layer extrinsic and sample-dependent or intrinsic  
 97 and therefore unavoidable? (ii) Does the proportionality  
 98 between domain size and domain wall thickness, previ-  
 99 ously reported for ferroelectrics and ferromagnets, hold  
 100 also true for this ferroelastic twinning? And (iii) if the  
 101 domain walls are extrinsically broadened, does this affect  
 102 the domain periodicity? The present article looks at these  
 103 issues.

## 104 Theory

105 According to the model of Lukyanchuk et al. [18], the  
 106 domain width (*w*) is given by:

$$w = \frac{w_0}{1 - \frac{w_0}{kt_{SL}}} \quad (1)$$

108 where *k* = 7.4 is a numerical constant, *t*<sub>SL</sub> is the thick-  
 109 ness of the surface layer and *w*<sub>0</sub> is the Roytburd-type

equilibrium domain size of the ferroelastic domains, which  
 is given by

$$w_0 = \sqrt{kDt_{FL}} = 2.72\sqrt{D(t - 2t_{SL})} \quad (2)$$

where *t*<sub>FL</sub> is the thickness of the ferroelectric layer of the  
 film, here expressed as the difference between the total film  
 thickness (*t*) and the thickness the two surface layers. *D* is  
 the elastic length-scale parameter of the system, which is  
 given by [18]:

$$D = \frac{\sigma}{Ge_{ac}^2} \quad (3)$$

where  $\sigma$  is the surface energy density of the domain wall,  
 which is ~2–4 mJ/m<sup>2</sup> for BTO [19], *G* is the shear modulus  
 (~50GPa for BTO [20]) and  $e_{ac}^2$  is the tetragonal strain:  
 $e_{ac} = \frac{c-a}{b}$ , with *a* and *c* the tetragonal lattice parameters of  
 BTO, and *b* the pseudocubic lattice parameter of the  
 paraelectric phase. Physically, this length-scale parameter  
 coincides with the half-thickness of the ferroelastic domain  
 wall. This can be seen noticing that  $\frac{1}{2}Ge_{ac}^2$  is the elastic  
 energy density per unit volume of domain wall (as in  
 Hooke’s law, where *G* is the elastic constant and *e* is the  
 deformation). Multiplying this volume energy density by  
 the thickness of the domain wall ( $\equiv \delta$ ), one obtains the  
 energy density per unit surface of the domain wall:

$$\sigma = \delta \times \frac{1}{2}Ge_{ac}^2 \quad (4)$$

Comparing Eqs. 3 and 4 it is immediately obvious that  
 the domain wall thickness is  $\delta = 2D$ . The calculated value  
 for the intrinsic thickness of the ferroelastic domain wall is  
 thus estimated to be  $\delta \sim 1$  nm for BTO. It is important to  
 note, however, that this wall thickness is based only on the  
 elastic interactions. If the ferroelectric polarization is  
 incorporated, one obtains instead the classic results for  
 ferroelectric–ferroelastic domain wall thickness of Zhirnov  
 [19] and Cao et al. [21, 22]. The actual experimental values  
 of the domain wall thickness differ for different studies, but  
 our calculated intrinsic value of  $\delta \sim 1$  nm coincides with  
 the lower limit of the range of thicknesses directly  
 measured by Zhang et al. [23] using electron holography.

Equation 2 can be re-written as:

$$\frac{w_0^2}{\delta} = 3.7(t - 2t_{SL}) \quad (5)$$

where  $\delta = 2D$  is the domain wall thickness. Here we note  
 that Eq. 5 is essentially identical to that previously  
 obtained for domain size scaling in 180° stripe domains in  
 ferromagnets and ferroelectrics [24–26], provided that the  
 surface layer is sufficiently thin in comparison with the  
 total thickness of the film (i.e.,  $2t_{SL} \ll t$ ). This suggests  
 that, theoretically at least, the proportionality between the  
 square of the spontaneous domain size and the domain wall

156 thickness is indeed a universal feature of all ferroics,  
157 irrespective of the specific forces involved.

158 Finally, we would like to comment on the possibility  
159 that the surface layers of BTO be harder than the bulk  
160 structure. Surface-tension induced hardening is of course  
161 not unusual in nature, and Lukyanchuk et al. showed that,  
162 if the surface layer of the ferroelastic material is harder  
163 than the interior, Eq. 1 should be modified to incorporate  
164 the different shear moduli of bulk ( $G_b$ ) and surface ( $G_s$ ),  
165 with the resulting equation being

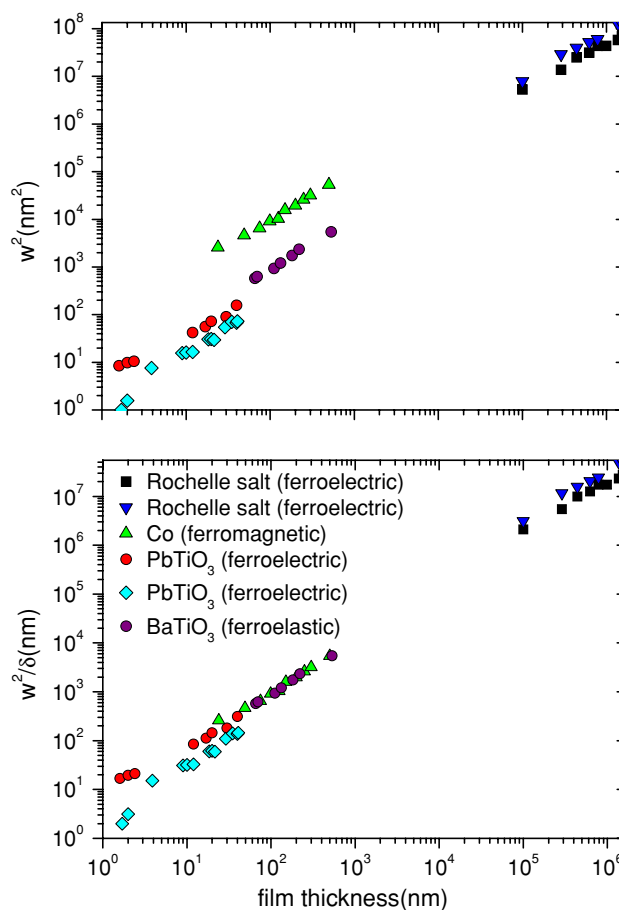
$$w = \frac{w_0}{1 - \frac{w_0 G_b}{k t_{SL} G_s}} \quad (6)$$

167 Thus, while surface hardening does not affect  $w_0$ , which  
168 is the Kittel–Roitburd square root limit for domain size  
169 dependence on thickness, it does affect the range of surface  
170 layer thicknesses ( $t_{SL}$ ) for which one can assume that  
171  $w \approx w_0$ . Specifically, the harder the surface layer is, the  
172 smaller the second term in the denominator and thus the  
173 better the square root approximation is. In this context,  
174 there have been recent calculations by Gharbi et al. [27]  
175 which show that indeed the hardness of BTO increases  
176 with decreasing thickness, with the surface being nearly  
177 twice as hard as the interior. This result extends range of  
178 validity of Eqs. 2 and 5 as reasonable approximations.

## 179 Comparison with experiment

180 In Fig. 1 we have compared the domain size of the ferro-  
181 elastic  $90^\circ$  domains of Schilling et al. with the domain size  
182 of  $180^\circ$  stripes in ferroelectrics and ferromagnets. The  
183 samples in which the ferroelastic domains were studied  
184 were thin lamellae that were carved out of bulk BTO single  
185 crystals using a focussed ion beam. The lamellae were  
186 heated above the Curie temperature and subsequently  
187 cooled down so that the domains appeared spontaneously.  
188 The comparison between the different types of ferroic  
189 domains shows that, while the domain size as a function of  
190 thickness is quite different for the different ferroics, once  
191 normalized by the domain wall thickness all data fall in the  
192 same “universal” curve.

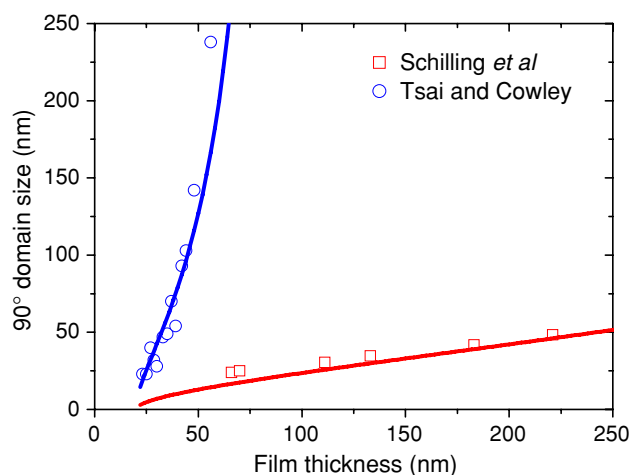
193 A second aspect worth considering is the fact that the  
194 wall half-thickness given by (3) is a lower limit and is not  
195 necessarily the same as the experimentally measured value  
196 in real ferroelectrics. For example, defects are known to be  
197 attracted to domain walls, and this can considerably  
198 broaden them. The domain wall thickness is thus sample-  
199 specific and should either be directly measured for the  
200 samples being analyzed or else treated as a fitting param-  
201 eter. This has been verified here by comparing the size of  
202 ferroelastic  $90^\circ$  domains in single crystal BTO films made  
203 in two different laboratories, as shown in Fig. 2.



**Fig. 1** (Above) Square of the domain size as a function of thickness for different types of ferroic domains found in the literature:  $180^\circ$  ferroelectric domains in  $\text{PbTiO}_3$  [30] (the red and blue sets correspond to different configurations of the stripe domains), and Rochelle salt [3] (rapidly cooled (black) and slowly cooled (blue)),  $180^\circ$  ferromagnetic domains in ferromagnetic Co [31] and  $90^\circ$  ferroelastic domains in  $\text{BaTiO}_3$  [11]. (Below) When the square of the domain size is normalized by the domain wall thickness, all ferroic domains, including the ferroelastic ones, fall into approximately the same “universal” curve

The domain size as a function of thickness is strikingly different between the two sets of samples, even though both the ferroelectric material (BTO) and the type of domain ( $90^\circ$  in-plane) are purportedly the same. And yet, in spite of their very different quantitative trends, both sets of data can be well fitted using Eq. 1. In the modeling of Tsai’s data, we have used their experimentally measured surface layer thickness,  $t_{SL} = 10$  nm [28], and a domain wall half-thickness of  $D = 10$  nm instead of the intrinsic  $D = 0.5$  nm of Schilling’s data. The domain wall half-thickness used for modeling Tsai and Cowley’s data, though apparently thick, is not an arbitrary fitting parameter: it is the value measured in their work for the half-thickness of the boundaries between their domains; though they associated such boundaries to needle domains, we





**Fig. 2** Experimental results (symbols) and theoretical fit (solid lines) of the domain size as a function of thickness for the samples of Schilling et al. [11] and Tsai and Cowley [28]. The difference between the two fits was the domain wall thickness, 1 nm for the data of Schilling et al. and 20 nm in the data of Tsai and Cowley

219 believe that they were in fact the domain walls, and our  
220 results here are consistent with that view. This wall  
221 thickness is also the same as directly measured by Franck  
222 et al. [29] using piezo-response atomic force microscopy.

223 Finally, in the experiments of Schilling et al., the  
224 encapsulating layer could be associated with extrinsic  
225 surface damage caused by Ga ion implantation during  
226 focussed ion milling process. However, samples annealed  
227 to remove the extrinsic surface damage still showed regular  
228 ferroelastic domain patterns [18]. Likewise, the samples of  
229 Tsai and Cowley [28] were made by conventional TEM  
230 preparation techniques and so they were, in principle, free  
231 from ion implantation damage, in spite of which they also  
232 showed a surface relaxation layer. All these observations  
233 suggest that the existence of non-ferroelectric layers  
234 around ferroelectrics may be an intrinsic feature, and that  
235 the influence of surface tension on domain formation  
236 should therefore be generally taken into account when  
237 modeling nanoscale ferroelectrics.

## 238 Conclusions

239 In summary, comparison between the surface tension  
240 model for ferroelastic domain size in free-standing ferro-  
241 electrics and experimental data from different sources  
242 allows establishing three principal conclusions:

- 243 (i) The square of the domain size is linearly proportional to  
244 domain wall thickness in all ferroic materials, irrespec-  
245 tive of the forces at play and the type of domain;

- (ii) The proportionality holds true even for extrinsically  
246 broadened domain walls. The equations are based on  
247 thermodynamic stability; hence it is the experimen-  
248 tal value of sample-dependent wall thickness,  $\delta$ , that  
249 is involved, whatever its origin, and not the ideal  
250 defect-free, strain-free value of one or two unit cells,  
251 and  
252  
(iii) Ferroelastic domains appear even in the absence of  
253 extrinsic surface layers, suggesting an intrinsic ori-  
254 gin, such as surface tension, for the strain relaxation  
255 layer. There are indications that the intrinsic surface  
256 layer is harder than the interior [27], and this further  
257 reinforces effect of the surface layer on twinning.  
258

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