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Ferroelectricity at the nanoscale

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<u>Abstract.</u> The properties of ferroelectrics at the nanoscale are reviewed. The term nanoscale is here related to the ferroelectric film thickness (which is by an order of magnitude the size of the critical domain nucleus). The three aspects considered are ferroelectric switching, the scaling of the coercive field, and the bulk photovoltaic effect. While ferroelectricity at the nanoscale has a twenty-year history of study, it is only in the last few years that perovskite ferroelectric films have become a focus of interest.

1. Introduction: some historical remarks

In 1945–1946, Vul [1] in Russia and von Hippel [2] in the US discovered ferroelectric properties in BaTiO₃, a discovery that stimulated Ginzburg to develop the ferroelectric mean-field theory (also known as the Landau–Ginzburg–Devon-shire, or LGD, theory) [3, 4].

The LGD theory predicts switching to occur for $E > E_c$, where E_c is the coercive field. We use the term 'intrinsic coercive field' to refer to the value of E_c determined from the LGD theory; hence, the term intrinsic (or uniform) for LGD switching. Figure 1, taken from Ginzburg's work [3], shows the LGD-calculated dependence of the free energy Φ on the polarization *P* for different values of the external field *E* and illustrates intrinsic (uniform) switching. It can be seen that LGD switching has a characteristic limit field, $E = E_{c,in}$, below which no switching occurs. Until 1998, no one ever observed intrinsic switching, either in bulk crystals or in films. The measured coercive field $E_{c,ex}$ was found to be 2–3 orders

V M Fridkin Shubnikov Institute of Crystallography, Russian Academy of Sciences, Leninskii prosp. 59, 119333 Moscow, Russian Federation E-mail: fridkin@ns.crys.ras.ru S Ducharme Department of Physics and Astronomy, Nebraska Center for Materials and Nanoscience, University of Nebraska-Lincoln; NE 68588-0299, USA E-mail: sducharme1@unl.edu

Received 14 August 2013, revised 2 October 2013 Uspekhi Fizicheskikh Nauk **184** (6) 645–651 (2014) DOI: 10.3367/UFNr.0184.201406d.0645 Translated by E G Strel'chenko; edited by A M Semikhatov of magnitude less than the intrinsic values $E_{c, ex} \ll E_{c, in}$ predicted by the LGD theory.

This discrepancy remained unresolved for some years until the discovery of domains in Seignette's salt by Chernyshova and coworkers [5]. Later, Merz discovered that $BaTiO_3$ has a domain structure and that polarization switching is due to the motion of domains in an external field [6]. Indenbom reached the same conclusion from his study of Seignette's salt [7]. Then Ishibashi and Takagi [8] used Kolmogorov's statistics to develop a theory of domain (extrinsic) switching, known as the Kolmogorov–Avrami– Ishibashi (KAI) theory. A comprehensive review of the intrinsic (domain) mechanism is given in Ref. [9].

Thus, while the LGD mean-field theory is successful in explaining the totality of ferroelectric phenomena, including first-order close to second-order ferroelectric transitions, it so



far has failed to account for the switching phenomenon and its details. The discovery and study of Langmuir–Blodgett (LB) polymer ferroelectric nanofilms [10, 11] changed this situation. The LB method allows controlling the film thickness to a single monolayer (0.5 m) and uses ellipsometry and atomic force microscopy to control the species and orientation of molecules.

The properties and growth of LB nanofilms of polyvinylidene fluoride-trifluoroethylene copolymer have been studied by a number of authors [12–15]. While the study and application of thin ferroelectric films has a long history [16], it is the LB method that first enabled the fabrication and study of films with a thickness of the order of the critical domain nucleation size. Because, as is known, critical domain nuclei have their size l^* in the range $\approx 1-10$ nm [16–18], nanosize ferroelectric films allow only nucleation and initial domain growth. In spite of this, ferroelectric LB films exhibit the phenomenon of switching [11], something that the KAI theory does not explain.

2. Switching at the nanoscale

It was conjectured in [19, 20] that switching in ferroelectric films in the nanorange (1–10 nm) occurs by an intrinsic (uniform) LGD mechanism. The kinetics of intrinsic switching are described within the uniform mean-field framework by the Landau–Khalatnikov equation

$$\xi \, \frac{\mathrm{d}P}{\mathrm{d}t} = -\alpha P - \beta P^3 - \gamma P^5 + E\,,\tag{1}$$

where *P* is the spontaneous polarization, α , β , and γ are the well-known Landau–Ginzburg coefficients for first-order ferroelectrics, *E* is the external field, and ξ is the damping coefficient. Equation (1) admits the critical solution [19]

$$\tau^{-1} = \frac{1}{\tau_0} \left(\frac{E}{E_{\rm c,in}} - 1 \right)^{1/2},\tag{2}$$

where τ is the switching time, $\tau_0 \approx 6.3\gamma\xi/\beta^2$, $E_{c,in}$ is the intrinsic coercive field, and $E_{c,in} \approx P/\varepsilon\varepsilon_0$. As can be seen from Eqn (2), switching occurs only for $E > E_{c,in}$ and does not occur for $E < E_{c,in}$.

Thus, as already noted, intrinsic LGD switching is characterized by a limit field $E = E_{c,in}$, in contrast to the KAI domain mechanism, where this notion is irrelevant. In the simplest case, the KAI theory yields the following expression for the switching time [8]:

$$\tau^{-1} = \tau_0^{-1} \exp\left(-\frac{E_0}{E}\right),\tag{3}$$

where τ_0 and E_0 are constants.

3. Kinetics of intrinsic switching

Intrinsic LGD switching was first discovered in a 1 nm thick LB copolymer film [11], and its kinetics were extensively studied using Merz's and Chynoweth's methods [19, 21, 22] and piezoelectric atomic force microscopy (PAM) [23, 24].

In Fig. 2, the inverse switching time (Fig. 2b) measured by the Merz method (Fig. 2a) is shown as a function of the external voltage V for an LB polymer. In Fig. 2b, the value $E_{c,in} \approx 10^9 \text{ V cm}^{-1}$, corresponding to the thickness l = 15 nm, correlates with the value of the intrinsic coercive field



Figure 2. Intrinsic and extrinsic switching in a ferroelectric copolymer. t_m indicates the maximum of the switching curve. ML stands for monolayer.

for polyvinylidene fluoride-trifluoroethylene [20]. The continuous curve in Fig. 2b corresponds to expression (2). Figure 2c shows the same plots for the thicknesses l = 5,



Figure 3. (a) Epitaxial BTO film. Electron diffraction images of (b) MgO/Pt and (c) Pt/BTO interfaces.

15 and 50 nm (10, 30, and 200 monolayers, respectively). The thicker films exhibit the behavior predicted by the KAI theory in Eqn (3). Two films with thicknesses of 5 nm and 15 nm (10 and 30 monolayers), despite experimental scatter, show a limit value V = 4 V. The continuous curves correspond to Eqn (2) for 5 nm and 15 nm and to Eqn (3) for 50 nm (100 monolayers). The data were obtained using the Chynoweth method. PAM measurements yielded the same results [23, 24].

The observation of uniform LGD switching in ferroelectric polymer films [19, 21, 24] in the nanoscale range led many to conclude that this switching behavior is a polymerspecific exception [9]. Perovskite ferroelectric films in the nanometer range were obtained fairly recently and have not yet been investigated for switching uniformity. A comprehensive review on the growth of perovskite superlattices in the nanoscale range is given in Ref. [25]. Ferroelectricity was found in perovskite films with a thickness of three unit cells (1.2 nm) [26–29], but no study has been made on whether their exhibited switching is intrinsic. The idea of uniform switching in PbTiO₃ nanofilms was suggested in only one work, study [30], which failed to study switching kinetics, however.

Uniform intrinsic switching in perovskites was first discovered in Ref. [31], which used a capacitor circuit and atomic force microscopy to study switching kinetics in ultrathin heteroepitaxial BaTiO₃ films and which demonstrated good agreement between the observed limit coercive field and its LGD value.

In Refs [32, 33], ultrathin barium titanate (BTO) films were laser epitaxially grown on a Pt/MgO (100) substrate in a single vacuum cycle, and the resulting composite systems were studied for their structural properties (including the film thickness) using Rutherford backward scattering spectroscopy and transmission electron microscopy. The heteroepitaxial growth of BR films on a Pt surface is illustrated by the data obtained on the MgO/Pt and Pt/BaTiO₃ interfaces using high-resolution Fourier-transform electron microscopy (Fig. 3). The thickness of the BaTiO₃ films, l = 2, 8, and 40 nm, was measured (to within a 10-percent or better accuracy) by Rutherford backward scattering from 2 MeV He⁺⁺ ions [32]. To perform capacitor measurements, a second (Au) electrode was deposited on a barium titanate



Figure 4. Measurements on a capacitor using PAM.

film in an ultra-high vacuum (Fig. 4). The measurements were made with a piezoelectric atomic force microscope with its tip in contact with the gold electrode. The top 20 nm gold electrode was deposited on the barium titanate surface in an ultra-high vacuum (see Fig. 4).

All films in the thickness range 4–40 nm exhibited a stable monodomain state.

The difference between intrinsic behavior and extrinsic behavior is shown in Fig. 5, which plots the switching rate versus the applied voltage for an ultrathin (8 nm) epitaxial barium titanate film (Figs 5a, b) and for a 1 mm thick singlecrystal [001] plate of barium titanate (Fig. 5c). Shown in Fig. 5a is a τ^{-2} versus V plot for barium titanate in a capacitor. Figure 5b is the same for the tip in contact with the free surface of the film (the so-called tip regime). The



Figure 5. Intrinsic and extrinsic switching in a nanometer BTO film.



Figure 6. (a) Schematic of domain switching and (b) schematic of uniform switching (from Ref. [36]).

experimental data are in good agreement with Eqn (1) and indicate the coercive field $E_c = E_{c,in}$. Despite the small difference in the switching time, both regimes exhibit the same intrinsic switching behavior.

Referring to Fig. 5c, we see that for a single crystal, on the contrary, the switching kinetics are in agreement with Eqn (3), with switching occurring at $V = V_{c,ex} \ll V_{c,in}$.

Switching kinetics measurements confirm the occurrence of homogeneous switching in nanoscale ferroelectric films with a thickness of the order of the critical domain nucleation size. If we change from crystals to ferroelectric films, the KAI mechanism is superseded by the uniform LGD mechanism. In this thickness range, there is competition between these two mechanisms, both of which depend on the film thickness and the external field. This point has recently been given a firstprinciple justification [34]. Transitions between the two mechanisms are due to the difference in the switching time versus field dependence between thick films (Eqn (3), an exponential dependence) and thin films (Eqn (2), a slower dependence). The occurrence of extrinsic switching at high fields was recently discovered in Ref. [35]. We believe that this competition between the two mechanisms is a common feature of all ferroelectric films comparable in thickness to the size of the critical domain nucleus.

Clearly, the phenomenological LGD theory cannot explain how switching works microscopically. Some preliminary notes on this point have already appeared [30]. In accordance with Ref. [30], we suggest the following microscopic mechanism of switching (Fig. 6).

In the nanoscale range, both the LGD and KAI switching mechanisms operate simultaneously. At thicknesses close to the critical domain nucleation size (which is in the range 1–10 nm according to Refs [16–18]; Miller and Weinreich speak of 5 nm), the dominant mechanism is the inherent uniform one, implying domainless switching in such nanoscale films.

4. Coercive field scaling

All ferroelectric films have their coercive field dependent on the thickness, $E_{c,ex} = E_{c,ex}(l)$. The intrinsic LGD coercive field $E_{c,in}$ is thickness independent and determined by the Landau–Ginzburg coefficients [20].

Figure 7a plots $E_c = E_c(l)$ for an LB film of ferroelectric copolymer P[VDF-TrFE] [20]. It is seen that for the film thickness less than 10 nm, $E_c = E_{c,in} \approx 10^9 \text{ V m}^{-1}$, and the coercive field is independent of the film thickness. The coercive field strength is in good agreement with the intrinsic LGD value. Thicker films, which were prepared by the



Figure 7. (a) Scaling of the coercive field in a ferroelectric copolymer. (b) Inverse capacitance of the capacitor as a function of the film thickness.

spinning technique, exhibit scaling behavior. The authors of Ref. [20] therefore conjectured that 1–10 nm copolymer films exhibit uniform switching, as in Eqns (1) and (2), as indeed was later found in [19, 21–24]. In contrast to this, in films thicker than 10 nm, the coercive field is found to be thickness dependent, implying the domain switching dynamics [8].

The results shown in Fig. 7a were obtained in an Al– P[VDF-TrFE]–Al capacitor using the Sawyer–Tower method.

It was hypothesized in [36] that the saturation in Fig. 7a is due to the Thomas–Fermi screening gap between the electrode and the copolymer film. However, no dependence of the inverse capacity of the capacitor on the film thickness was observed (Fig. 7b) [37].

Here, for the first time, a scaling behavior of the same type is reported for nanoscale barium titanate films grown by laser epitaxy [38]. Measurements were performed on a Pt-BaTiO₃-Au capacitor using PAM (see Fig. 4). Electrodes were formed on a barium titanate surface by lithography and were circular with a radius of a few micrometers. Figure 8 shows hysteretic loops obtained for films with thicknesses of 3, 8, and 40 nm (Figs 8a-c). Also shown is the variation of the coercive field with the film thickness in the range 3-40 nm (Fig. 8d). Scaling occurs at thicknesses above 10 nm. In the range 3-10 nm, the coercive field is only weakly thickness dependent, and its magnitude $E_{c,in} \approx 10^8 \text{ V m}^{-1}$ is in close agreement with that given by the LGD theory. Accordingly, films with thicknesses of 3 and 8 mm exhibit uniform LGD switching, as given by Eqns (1) and (2), whereas thicker films follow the domain dynamics, as in Eqn (3).



5. Bulk photovoltaic effect in nanoferroelectrics

In this section, we make some brief preliminary remarks on the bulk photovoltaic effect (BPE) in the nanometer range.

Until the 1970s, there were two known varieties of the photovoltaic effect. The reasons for the first of these, the Dember effect, are the nonuniform illumination of the crystal and the strong absorption of light; the effect is then due to the diffusion of nonequilibrium carriers and the mobility difference between holes and electrons. The second variety is due to the separation of nonequilibrium carriers at crystal nonhomogeneities, the well-known example being the photovoltaic effect on p–n junctions currently used in solar cells. Thus, the photovoltaic effect as known until the early 1970s was due to either innomogeneities in or nonuniform illumination of the crystal. In either case, the photovoltage generated in the crystal (or in an individual p–n junction) does not exceed the band gap width of the crystal (which is a few volts).

In the early 1970s, another effect, the so-called bulk photovoltaic effect, was discovered [39], which arises when a crystal without central symmetry is exposed to uniform illumination. Unlike its earlier counterparts, neither crystal not illumination inhomogeneities play any role in this effect. There are two major points about the BPE: the bulk photovoltaic current depends on the light polarization, and the photovoltage in an open circuit crystal can exceed the band gap by orders of magnitude. The BPE is observed only in noncentrosymmetric crystals, and hence for in only twenty pyroelectric point symmetry groups. In other words, we here have an analogy to the violation of parity in weak interactions, for example, in neutron beta decay in ⁶⁰Co experiments (which brought the Nobel Prize in physics to Lee and Yang in 1957 [40, 41]). Landau was the first to hypothesize (in his lecture at the Shubnikov Institute of Crystallography in 1957) that the nonconservation of parity has a relation to the asymmetry of the Universe.

The microscopic BPE theory introduces a violation of the detailed balance principle for photoexcited carriers in noncentrosymmetric crystals [39]. The detailed balance principle assumes that the probability $W_{kk'}$ of transition from the state with a momentum k' to the state with a momentum k equals that of the opposite transition, $W_{k'k}$. Earlier, it was believed that this principle holds in kinetic theory for all solids, but already Boltzmann questioned this. In centrosymmetric crystals (such as Ge or Si), this principle does apply, and the BPE, accordingly, does not exist. In noncentrosymmetric crystals, where $W_{kk'} \neq W_{k'k}$, the violation of detailed balance leads to a nonequilibrium momentum distribution of conduction-band electrons or holes, producing a photovoltaic current in a homogeneous crystal under uniform illumination. This is schematically illustrated in Fig. 9.

Irrespective of the microscopic mechanism, the BPE involves the excitation of nonthermalized electrons (holes) in the crystal. From Fig. 9a, which illustrates the intrinsic excitation of a centrosymmetric crystal, we see that the electron distribution in the band is symmetric and that the



Figure 9. (a) Symmetric distribution of nonequilibrium carriers in a band of a centrosymmetric crystal; (b) nonsymmetric distribution of non-equilibrium carriers in a crystal with no center of symmetry.

current is absent. The excitation of a noncentrosymmetric crystal (Fig. 9b) results in the nonthermalized carriers having their momenta distributed asymmetrically (as shown by the arrow). The photoexcited nonthermalized carriers lose their energy and fall to the bottom of the conduction band, which causes them to shift by l_0 and gives rise to the bulk photovoltaic current

$$J = \alpha G^L I,$$

where α is the absorption coefficient and *I* is the light intensity. The amplitude of the third-rank tensor G^L is

$$G^{L} = e l_0 \zeta \varphi(h v)^{-1} , \qquad (4)$$

where φ is the quantum yield, the parameter ζ characterizes the excitation asymmetry and depends on the detailed balance violation mechanism, *e* is the electron charge, and G^L is a component of the third-rank tensor, whose experimental value is $10^{-8}-10^{-9}$ cm V⁻¹. In accordance with Ref. [39], $\zeta \approx 10^{-1}-10^{-2}$, and l_0 is in the range 10–100 nm. If the shift of a nonthermalized electron is of the order of the ferroelectric film thickness, the energy conversion efficiency η can be large. The estimate in [39] is

$$\eta = \frac{1}{4} G^L l_0 \zeta(\mu \tau)^{-1} , \qquad (5)$$

where in the nanometer range (10–100 nm), the mobility μ and the lifetime τ are nonthermalized.

Substituting $l_0 = 10 - 100$ nm, $G \approx 10^{-8}$, and $\zeta \approx 10^{-1} - 10^{-2}$ in Eqn (5) for barium titanate films, we obtain

$$\eta \approx 10^{-15} (\mu \tau)^{-1}$$
. (6)

The thermalization time is $\tau \approx 10^{-12} - 10^{-14}$ s [39]. However, the uncertainty in the estimate of the effective mass of the polaron prevents estimating the mobility of a nonthermalized electron μ .

Finally, we mention a series of recent experiments [42–44] that measured the photovoltaic effect and the energy conversion efficiency in the nanometer range, but failed to uncover the physical mechanism of these phenomena.

6. Conclusion

To conclude, ferroelectric properties in the nanometer range occur not because of any dimensional effect but because of the coexistence of intrinsic and extrinsic switching mechanisms, their respective roles depending on the film thickness and the external field. For ferroelectric films with thickness $l > l^*$, the dominant factors are the extrinsic coercive field and switching. For $l < l^*$, intrinsic uniform switching and the coercive field take over, as they should according to the LGD phenomenology.

The LGD mean-field theory of 1945 was developed for a homogeneous medium and was unable to explain the details of switching. The 1970 KAI theory developed for a domainstructured medium both explained the switching kinetics and predicted the value of the coercive field in crystals and films. Ultrathin films with a thickness of the order of the critical domain nucleation size can be considered homogeneous. For such films, the LGD phenomenology accounts for all ferroelectric properties, including switching (see the literature from 1993 to 2013).

The bulk photovoltaic effect in the nanometer range seems to hold promise for high-efficiency energy conversion.

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