Thickness dependence of domain size in 2D ferroelectric CuInP$_2$S$_6$ nanoflakes

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ABSTRACT
Two-dimensional (2D) ferroelectrics refer to those ferroelectrics with layered structure and weak interlayer interactions (e.g., van de Waals interlayer coupling). A number of basic physical issues in the framework of ferroelectricity deserve clarifications, and one of them is the size effect regarding the dependence of ferroelectricity on material thickness. In this work, we investigate the ferroelectric domain structures of 2D ferroelectric CuInP$_2$S$_6$ nanoflakes attached on heavily doped Si wafers and polarization switching using the piezoresponse force microscopy. While the domain structure shows highly irregular morphology and 180° domain walls, the statistics on domain size (diameter) $W$ and nanoflake thickness $d$ demonstrate the remarkable thickness dependence of domain size, illustrated by the shrinking domain size from 630 nm to 75 nm with decreasing thickness $d$ from $\sim$130 nm to $\sim$11 nm. This dependence fits the Landau-Lifshitz-Kittel (LLK) scaling law with the scaling exponent of $\sim$0.65, slightly larger than 0.5 for 3D ferroelectrics. It is suggested that the size effect in terms of the LLK scaling law does not show an essential difference between the 2D and 3D ferroelectric systems.

I. INTRODUCTION
The finite size effect or dimensionality in condensed matters represents a long-standing topic with which a series of emergent phenomena have been revealed and understood. This effect is also highly concerned in ferroics such as ferroelectrics and magnetics, which was once the hot topic for advanced magnetism and ferroelectricity. Along with the increasing attention toward low-dimensional systems, the issue of size effect is now extended to two-dimensional (2D) magnetics and ferroelectrics. Continuous symmetries at finite temperature ($T$) in dimensions no more than two for those lattices of only short-range exchanges. The so-called continuous symmetry here implies that the magnetocrystalline anisotropy would be negligible. Nevertheless, recent experiments did evidence the long-range magnetic ordering in several 2D materials, suggesting that those concepts well established in 3D systems may not be applicable to 2D materials and thus those highly concerned issues should be rechecked in 2D systems.

In parallel, the finite size effect of ferroelectricity has for a long time been a topic of interest, and similar questions can be raised for 2D ferroelectrics, in response to requirements of high density integrated ferroelectric devices. Earlier works focused on the dimension-shrinking of 3D ferroelectric perovskite oxides such as BaTiO$_3$ and PbTiO$_3$. While a remarkable size effect is well believed, no size-downlimit of 3D ferroelectricity down to 1–2 nm in the characteristic size has been identified. The size effect for 3D...
ferroelectrics can be described by the quadratic scaling law proposed by Landau, Lifshitz, and Kittel [the well-known Landau-Lifshitz-Kittel (LLK) scaling),

\[ W \sim d^m, \]

where \( W \) is the characteristic size of ferroelectric domains (e.g., domain diameter), \( d \) is the sample dimension (thickness for thin film or diameter for nanodots), and \( m = 1/2 \) is the scaling exponent. The underlying physics can be understood phenomenologically by the competition between the depolarization energy and the domain wall energy.

It is certainly imperative to check this scaling law in 2D ferroelectrics, noting that there is one dimension with which the interaction is much weaker than those along the other two dimensions. Several issues can be discussed and questioned. First, one expects that the long-range ferroelastic effect should be weak at least along the out-of-plane direction due to the strong anisotropy in the chemical bonding. If it is true, the well-ordered domain structures often observed in 3D systems such as twinlike domain stripes would be hard to develop in these 2D systems. Second, 2D ferroelectrics would exhibit different degeneracy in the preferred polarization orientations from 3D counterparts, making the domain structure quite different. It is seen that most 2D ferroelectrics in ultrathin geometry still have nonzero out-of-plane polarization that is usually believed to be suppressed due to the strong depolarization field when the thickness goes down to the zero limit. Third, ferroelectric ordering can be phenomenologically viewed as the spatially ordered alignment of electric dipoles, and such an ordering can be simply described by electrostatic energy. In such a sense, one sees no substantial difference of a 3D lattice structure from that for a 2D layered structure with weak interlayer interaction. It seems that the size effect such as the LLK scaling, in some slightly modified form, should be applicable to 2D ferroelectrics too.

Certainly, these issues deserve checking in 2D ferroelectrics. So far, there have been reported a few 2D ferroelectrics, including CuInP\(_2\)S\(_6\) (CIPS), In\(_2\)Se\(_3\), and MoS\(_2\). We take CIPS as the object for the present study of size effect. CIPS is a room-temperature 2D ferroelectric with a Curie point of \( T_C \sim 315 \) K. This ordering occurs close to room temperature, and thus, the coercive field should be low and the self-poling effect should be weak, benefiting access to the equilibrium domain structure at room temperature. Structurally, CIPS consists of sulfur octahedral cages stacked layer by layer. The cages in the layers form different triangular sublattices which are, respectively, filled with Cu, In, and P-P pairs, as shown in Figs. 1(a) and 1(b), and the layers are coupled with the vdW interaction. It is argued that a unit cell must include two adjacent layers, considering the site exchange between Cu and P-P pair from one layer to another. Therefore, the whole structure is a collinear two-sublattice ferrielectric system. The spontaneous polarization is generated upon the ferroelectric transition from the high-\( T \) \( \text{C}_{2/c}\) phase to the low-\( T \) \( \text{C}_{c}\) phase. The off-center shifting in the Cu sublattice and cation displacement in the In-sublattice are responsible for the out-of-plane spontaneous polarization, allowing only the 180° domain structure and thus providing an advantage for checking the size effect of ferroelectricity using the piezoresponse force microscopy (PFM) technique in the out-of-plane mode.

II. EXPERIMENTAL DETAILS

In this work, we focus on the size effect of CIPS nanoflakes. We start from a series of CIPS nanoflakes of different thicknesses \( d \) and probe the ferroelectric domain sizes of these nanoflakes at room temperature \( T = 293 \) K. Based on the domain size data, we check the thickness dependence of domain size, noting that a recent work on the domain structure of CIPS did reveal such a dependence. In our experiments, the CIPS bulk crystals were deposited...
in evacuated silica ampules (quartz glass substrates) using the chemical vapor transport method, and details of the deposition procedure were described in Refs. 32 and 33. Subsequently, the CIPS thin nanoflakes were obtained by the standard mechanical exfoliation technique and transferred to the n⁺-doped Si wafers that are flat at the atomic level and act as bottom electrodes in the subsequent measurements. Fortunately, this exfoliation method allowed us to obtain nanoflakes of different thickness on one Si wafer. We did collect a sufficient number of CIPS nanoflakes with variation in the range from 11 nm to 290 nm. Unfortunately, data for even thinner nanoflakes are not available due to the spatial resolution of the PFM domain imaging (~20 nm, in-plane resolution), and this deficiency limits our probing from the downlimit of a few of unit layers in thickness.

Here, it should be mentioned that the wafer used for supporting the CIPS nanoflakes may impose the interface field effect due to the difference in electronic property between the nanoflakes and wafer. Here, the heavy-doped Si wafers were used. Since CIPS nanoflakes are micrometers in the in-plane dimension, we had no approach to experimentally characterize the effect of the interface field. Nevertheless, when the Pt thin plates were used to transfer the nanoflakes, the observations showed that domain structures of the nanoflakes on the Pt and Si plates are similar in terms of the domain size. However, the Pt plates have coarse surface, making the domain structure imaging difficult. Since Pt and Si have quite different surface structure and electronic properties, one may assume that the introduced interface field from the Si wafer would not be an issue.

Subsequently, we probed these samples one by one using the PFM technique. The probing was carried out using the commercial atomic force microscope (AFM, Bruker multimode 8) with the advanced PFM mode. The Co/Cr-coated conductive tip (MESP-RC-V2, Bruker) was used, and we obtained the PFM images and PFM hysteresis loops with the scan frequency of 1.0 Hz and 0.1 Hz, respectively. It was shown that the nanoflakes show quite clear domain contrast with this operation state and the imaging can be well reproduced.

III. RESULTS AND DISCUSSIONS

As a representative example, we first show in Figs. 1(c) and 1(d) the topology and out-of-plane (OP) phase images of a CIPS nanoflake, which was 5–10 μm in the in-plane dimension. It is seen that the nanoflake’s surface consists of several steplike regions, corresponding to different film thicknesses, as marked in Fig. 1(c). On each step, one can observe fresh domains with clear black and white contrast. The black regions are domains with downward polarization, while those white domains have the upward polarization.

There are several morphology features to be highlighted. First, the domains are different in shape and show broad distribution in size. No remarkable domain coarsening with time was observed in our experiments, suggesting that these domain structures are in the fresh and well-developed state. Second, our in-plane PFM probing reveals no well-defined contrast, suggesting that these domains are the 180° domains with the out-of-plane polarization. Third, these domains show irregular shapes, and this feature is different from most 3D ferroelectrics in the bulk form where usually a well-shaped domain pattern is developed and the domain walls have well-defined characteristic scale and smooth shapes. Indeed, irregular wall shapes were also observed in some 3D systems in the ultrathin film form where the domain morphology may be described using the fractal geometry. Here, a similar description can be applicable. Such an irregular shape feature implies that no long-range elastic energy is involved in this system due to the dominant vdW interlayer bonding.

Subsequently, we checked a huge number of CIPS nanoflakes with different thicknesses and performed sufficient statistics on the domain size $W$ and flake thickness $d$. Due to the irregular domain shapes, the size $W$ for every domain was obtained by taking the average of the long and short cross-edge lengths. As typical examples, we present in Figs. 2(a)–2(f) the phase images of observed domain structures for several selected samples with thickness $d$ marked on the lower right corner of each image. It is seen that the larger domains usually have stronger black/white contrast in the PFM amplitude and phase images, suggesting the larger out-of-plane polarization. While the large domains have sharp wall contrast, the walls of those fine dendrite or treelike fine domains are far from sharp. For the sample as thin as 14 nm, as shown in Fig. 2(f), no clear contrast separating the domains and their walls can be identified by eyes.

![FIG. 2. The measured out-of-plane PFM phase images of a set of nanoflakes with different thickness $d$. (a) $d = 125$ nm, (b) $d = 80$ nm, (c) $d = 56$ nm, (d) $d = 29$ nm, (e) $d = 23$ nm, (f) $d = 14$ nm.](image-url)
FIG. 3. The measured piezoelectric amplitude (amp) and phase (phase) in response to the cycling of the tip voltage, generating the amplitude and phase hysteresis loops on a series of CIPS nanoflakes of thickness $d = 125$ nm (a), 50 nm (b), 29 nm (c), 23 nm (d), 14 nm (e). The evaluated coercive field $E_c$, i.e., the electric field corresponding to the amplitude valleys and phase reversals, as a function of $d$, is plotted in (f).

For a quantitative characterization of these CIPS nanoflakes, we measured the PFM hysteresis response of various samples, as shown in Fig. 3, where several sets of data were collected at a fixed voltage sweeping rate and the value of $d$ is inserted in each plot. It is seen clearly that the phase hysteresis shape is seriously thickness-dependent, evident with the double-butterfly patterns of the amplitude hysteresis. One may highlight the measured results. First, the phase hysteresis is square shaped for thick samples and becomes slim and inclined with decreasing thickness. Second, the hysteresis data show the large amplitudes and sharp valleys corresponding to the domain switching for the thick samples, while the amplitude decreases rapidly with reduced sample thickness from $\sim 4.0$ mV at $d \sim 125$ nm to $0.3$ mV at $d \sim 14$ nm. Third, the reduced coercive field $E_c$ in the thicker samples as shown in Fig. 3(f), a well-known fact for 3D ferroelectrics, is revealed in our samples too. In short, the remarkable reduced amplitude and seriously distorted phase-loop shape reflect the serious suppression of ferroelectricity in thin samples.

In consistence with these facts, we performed the polarization reversal experiments by writing a fixed squared region on these samples. Two representative examples are shown in Figs. 4(a) and 4(b), where the writing voltages applied to the bottom electrode are labeled and the black contrast shows the downward polarization. It is roughly shown that the written patterns in the thick samples [Fig. 4(a), $d = 79$ nm] are stable without serious back-recovery. However, for the thin sample ($d = 23$ nm), the written domains are highly unstable and the squared region recovers gradually backward to the original domain structure, as shown in Fig. 4(b). This fact suggests the essential role of the domain wall energy in competition with the large electrostatic energy due to the depolarization. The final part of the measured data includes the measured average domain size $W$ as a function of sample thickness $d$, in order to check the LLK scaling law. For the measurements, it should be mentioned that the domains may be even larger than the step regions on sample surface if the sample is very thick. For examples, some domains will exceed 2 $\mu$m in size when the sample is thicker.

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than 130 nm. Also, the irregular shape and scattered domain size make the data errors relatively remarkable. The results are plotted in Fig. 5(a) where the blue coarse line is the fitting by the LLK law. Although the data show some scattering, the fitted scaling exponent \( n \approx 0.65 \), slightly larger than 1/2 predicted by the LLK law. It seems that the LLK scaling law is still applicable to CIPS in the sample thickness range covered in this work although the scaling exponent does differ from 1/2 slightly.

To this stage, we have presented the measured dependence of the domain structure on the thickness of CIPS samples. It is revealed that the size effect in 2D ferroelectrics like CIPS here does not show large difference from that observed in 3D ferroelectrics with negligible long-range elastic energy. This result seems to be surprising since it is generally expected that the 2D systems would have very different behaviors from the 3D counterparts in terms of the size effect. For a simplified discussion, we start from the electrostatic interaction scenario. We consider as an extreme case a single layer 2D structure, as shown by one layer in Fig. 5(b). Since the spontaneous polarization is out-of-plane oriented, one expects a huge depolarization energy which is believed to eventually suppress the ionic displacement by screening the electric dipole alignment. This is the basis in the conventional understanding of the size effect. Nevertheless, even though the interlayer coupling is of the van der Waals type and very weak, a stacking of such single layers along the \( z \)-axis would enable the compensation of the depolarization field which decreases rapidly with increasing \( d \), as shown in Fig. 5(b), and thus, the electrostatic interaction becomes similar to the 3D ferroelectrics when the nanoflakes become a bit thick. We actually find no qualitative difference in the size effect between the 2D and 3D ferroelectrics, and the LLK scaling law is roughly followed in the present CIPS samples where the domain size is determined by the competition between the depolarization energy and the domain wall energy.

Unfortunately, due to the spatial resolution of the PFM facility, we cannot access those samples or regions with \( d < 10 \) nm, where the domain size should be smaller than 20 nm. What we can claim is that the size effect as \( d > 10 \) nm does satisfy the LLK scaling law. When the thickness tends to the limit of one or two unit cells, experimental observation did find nonzero polarization, implying that the LLK law no longer works in the ultrathin limit. In fact, the invalidity of the LLK law for 3D ferroelectrics in such a limit has also been suggested.14 On the other hand, the scaling exponent \( m = 0.65 > 0.50 \) implies that the domain size shrinking with decreasing \( d \) in those thin regions becomes weaker, consistent with the fact that the single layer 2D system still exhibits ferroelectricity, although no details of the physics are accessible to us at this moment. In the other words, whether the LLK law applies to the small-\( d \) limit remains to be an open issue.

IV. CONCLUSION

In conclusion, we have investigated carefully the domain structures of mechanically exfoliated but Si wafer-attached CIPS nanoflakes with different thicknesses and checked the size effect, using the advanced PFM technique. It has been revealed that the CIPS nanoflakes exhibit the typical 180° and irregular domain structure with out-of-plane ferroelectric polarization. The remarkable thickness-dependence of the domain structure has been found, characterized by the transition from the square and sharp piezoelectric...
response loops observed in the thick samples to the slim and weak piezoelectric response loops in the thin samples. In the range of sample thickness covered in this study, the LLK scaling law for the 3D ferroelectrics is also satisfied by the measured average domain size as a function of CIPS nanoflake thickness although the evaluated scaling exponent \( m \approx 0.65 \) is slightly larger than that predicted by the LLK law \( (m = 0.50) \). While the 2D piezoelectricity remains to be an appreciated issue, this study demonstrates the remarkable size effect of 2D ferroelectrics.

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