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# In-situ Observation of Crack Growth and Domain Switching Around Vickers Indentation on $BaTiO_3$ Single Crystal Under Sustained Electric Field

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The crack propagation and domain switching process around the indentation on the surface of barium titanate single crystal under the external electric field was investigated by atomic force microscope and polarized light microscope. The evolutions of domain switching and crack propagation were in-situ observed when a 90° $a-c$ domain wall moved across the indentation which was driven by external electric field. The results show that the incompatible strain induced by domain switching in the residual stress zone around the indentation is the driving force of the anisotropic crack propagation. The crack propagation results in the changes of the fine domain stripes around the crack tip.

**KEY WORDS: Domain switching; Indentation; Crack propagation; $BaTiO_3$; AFM**

## 1. Introduction

As a class of the most important function materials, ferroelectrics have wide applications in electromechanical devices, such as micro-positioner, sensors, and detectors. However, they have a major disadvantage of brittleness. The ferroelectric devices tend to degrade after longtime working under electrical and mechanical loads due to the nucleation and growth of cracks. It is important to study the degradation mechanism in ferroelectrics to improve their reliability. The mechanical and fracture behaviors of ferroelectrics were considered to related to the domain switching. In recent years, much effort has been dedicated to a clear understanding of the crack growth and the domain switching around crack tips in ferroelectrics under mechanical or electric field[1−13]. For example, using Vickers indentation method[14−19], it has been revealed that the crack growth is distinctly anisotropic in the direction either parallel or perpendicular to the poling direction, which is much shorter in the former case. Although, the correlation between the crack and the domain switching is important to reveal the fracture mechanism, it is far from clear up to now, where a high-resolution technique can be very useful to in-situ observe the physical processes in microscopic scale.

Scanning force microscopy (SFM) is a powerful, non-destructive technique with great potentials to image and control the domain structures in ferroelectric materials in the nanometer scale. Over the past few years significant progress has been made in high-resolution visualization of ferroelectric domains in bulk crystals and polycrystalline films by means of SFM[20−27]. But little result focused on the relation of dynamic domain behavior with crack. In this article, the crack growth and the domain switching around Vickers indentation on the surface of barium titanate single crystal under an external electric field were in-situ studied by atomic force microscope (AFM) and polarized light microscope (PLM).

## 2. Experimental

Commercial tetragonal $BaTiO_3$ single crysta

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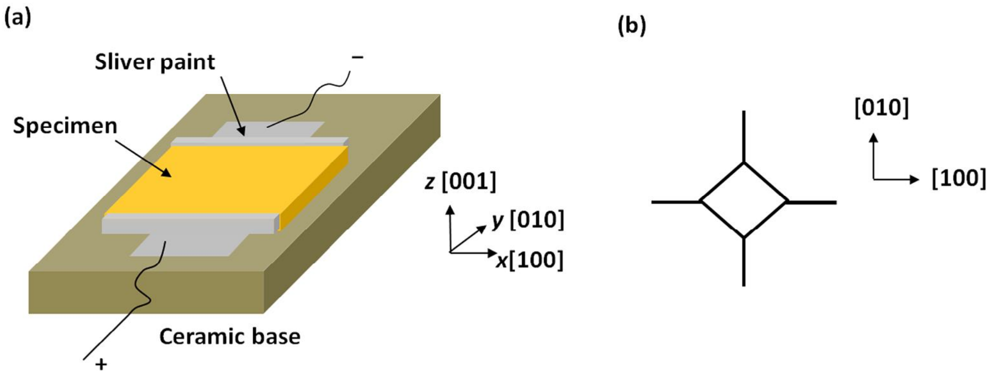

**Fig. 1** Schematic illustration of electrical load (a) and indentation cracks (b) in (001) surface

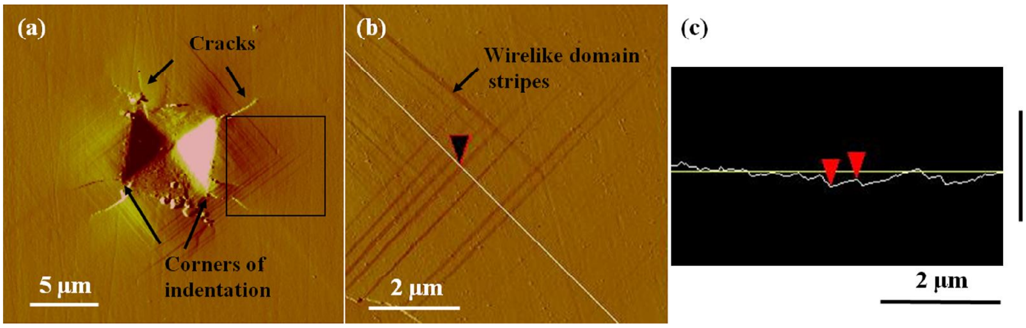

**Fig. 2** Indentation cracks and domain switching zones characterized by AFM: (a) morphology of the indentation with 25 g load; (b) wirelike ***a*** domain stripes around the indentation; (c) section analysis of the 90° ***a***-***c*** domain wall

the size of 5 mm×2 mm×1 mm was provided by the Institute of Physics, Chinese Academy of Sciences. The sample was polarized along [001] direction at room temperature, so that only "out-of plane" orientated *c* domains survive in (001) surface. For investigation, the (001) surface was mechanical polished by diamond abrasive. Electrodes were connected to two side faces and copper wires were attached using silver paint, as shown schematically in Fig. 1(a). The pre-cracks were initiated by Vickers indentation on the (001) polished surface. The load was 25 g with a dwell time of 20 s. The diagonal directions of the indentation are along the [100] and [010], as shown in Fig. 1(b). The commercial AFM (Veeco Dimension 3100, Nanoscope III) and PLM (Olympus BX60) were used to characterize the surface morphology and the domain configuration, before and after an external electric field was applied by a high voltage DC power source along the [010] direction. In the AFM measurement, the contact mode was used.

## 3. Results and Discussion

The whole observed surface is ***c*** domian, whose polarization vector directs out of plane, so the indentation on the surface is isotropic and the cracks with almost the same length emanated from each indent corner, as shown in Fig. 2(a). At the same time, the domain stripes induced by indentation cracks were also observed. It can be seen from Fig. 2(a) and Fig. 2(b) some ***c*** domains switched to ***a*** domains and formed wirelike domain stripes between two corners of the indentation. The direction of domain stripes was perpendicular to the crack tips, as shown in Fig. 2(a) and Fig. 2(b). Fig. 2(c) shows that the height of the wirelike domain wall was about 6 nm through the section analysis. Since the theoretical value of a 90° ***a***-***c*** domain wall in tetragonal BaTiO$_3$ is 0.6°, the measured topological inclination angle, $\theta$ of about 0.55° gave a proof of the orientation of these wirelike domain stripes. Therefore, the dark wirelike domain stripes were ***a*** domains induced by the stress of indentation.

The electric field was gradually increased along the [010] direction until ***c*** domain began to switching to ***a*** domain. Then, remained this sustained electric load unchanged. Under the sustained electric load, a long straight 90° ***a***-***c*** domain wall perpendicular to the electric field direction appeared and moved forward, that is, ***c*** domains switched to ***a*** domains. The ***a***-***c*** domain wall moved across the indentation. The velocity
domain wall movement was about $1.4\times10^{-4}$ n
Fig. 3 shows that the crack propagation aroun
indentation during the ***a***-***c*** domain wall travers
indentation. In Fig. 3, the dark zones are ***a*** do

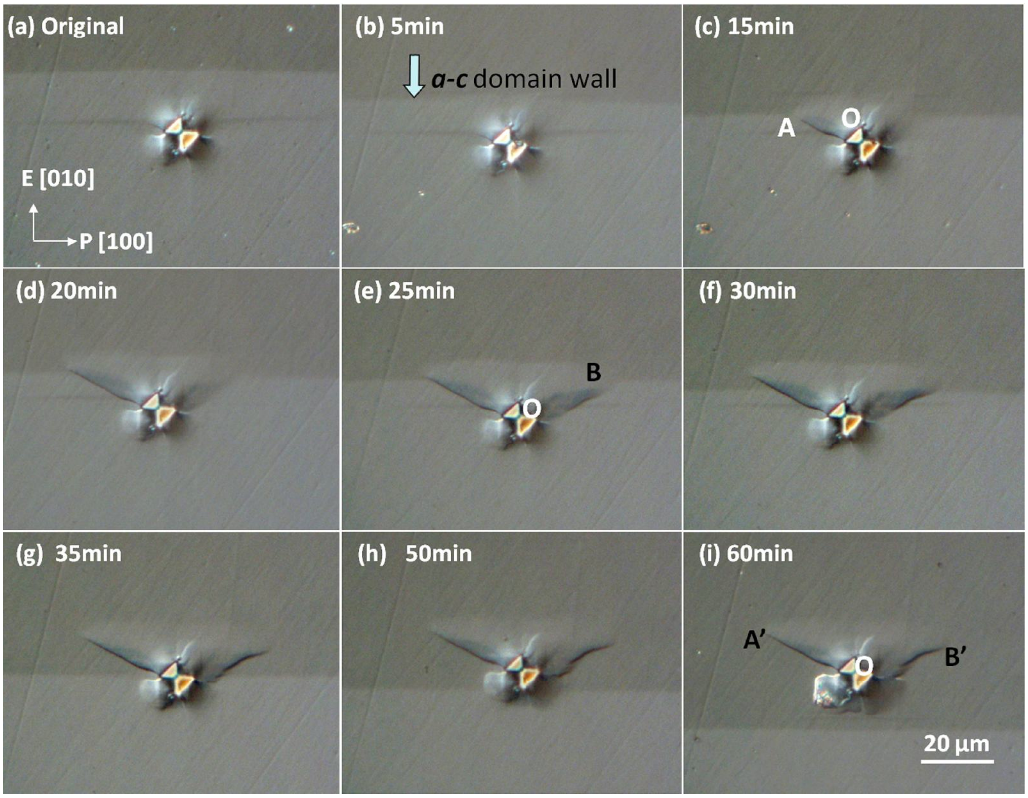

**Fig. 3** PLM observation of the changes of indentation cracks during the 90° *a*-*c* domain wall traversed the indentation: (a) original, (b) 5 min, (c) 15 min, (d) 20 min, (e) 25 min, (f) 30 min, (g) 35 min, (h) 50 min, (j) 60 min

and the bright zones are *c* domains. The blue arrow points to the movement of 90° *a*-*c* domain domain wall. Moved near to the indentation, the domain wall was hampered and a small bowl-shaped zone appeared, as shown in Fig. 3(b) and Fig. 3(c). Then, two cracks, OA and OB, appeared and propagated towards of *a* domain zone and the bowl-shaped zone became bigger, as shown in Fig. 3(d), Fig. 3(e) and Fig. 3(f). It can be seen from Fig. 3(h) and Fig. 3(i), the cracks stopped propagating and the bowl-shaped domain zone remained when the *a*-*c* domain wall traversed completely the indentation.

The details of domain switching and crack propagation in the bowl-shaped zone can not be examined by PLM, so we carried out the profound observation using AFM, as shown in Fig. 4 and Fig. 5. The dark zones are *a* domains and the bright zones are *c* domains in Fig. 4 and Fig. 5. In agreement with the PLM results, the 90° *a*-*c* domain wall was hampered when it moved near to the indentation, and a small bowl-shaped domain zone and a crack OA appeared at a coner of the indentation, as shown in Fig. 4(b). With the movement of the 90° *a*-*c* domain wall, the crack OA propagated to OA' and a new crack OB appeared, as shown in Fig. 4(c). It can be seen from Fig. 4(e), when the 90° *a*-*c* domain wall moved gradually, the crack OB propagated to OB' and two short cracks (OC and OD) grew a bit and connected each other, because the internal stress around the indentation was smaller, which was relaxed by the former crack propagation (OA and OB). Finally, the bowl-shaped zone remained on one side of the indentation, as shown in Fig. 4(f). For PLM observation, the bowl-shaped zone looks like *c* domain, but in fact some fine domain stripes are exist around the crack. Fig. 5 shows that the changes of the fine domain stripes with the crack propagation. When the 90° *a*-*c* domain wall traversed completely the indentation, the domain stripes in the bowl-shaped zone did not change.

In the past researches, the external electric field was usually applied at the same time or before the indentation[14–18]. The indentation cracks are isotropic or anisotropic on the surface with different polarization states. On the surface of *a* domains, the indentation cracks are anisotropic, ones are parallel to the poling direction which are much shorter than those perpendicular to the poling direction. On the surface of *c* domains, the isotropic cracks at four corners are also short with almost the same length. In our experiment, a dynamic evolution from the isotropic crack to anisotropic cracks was in-situ observed, as well the domain switching details a
the indentation. In literatures, there are re
stresses correlated to the crystal orientation a
the indentation[14,28,29]. The anisotropy of the i
tation crack can be explained by the residual

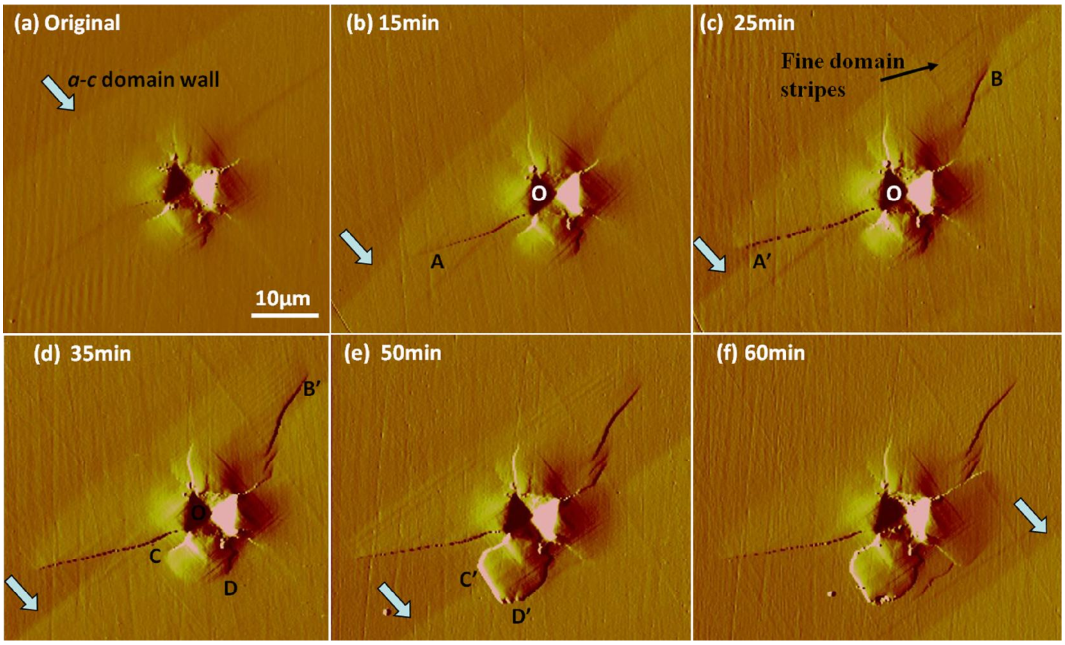

**Fig. 4** AFM images of the crack propagation and domain switching when the 90° ***a-c*** domain wall traversed the indentation: (a) original, (b) 15 min, (c) 25 min, (d) 35 min, (e) 50 min, (f) 60 min

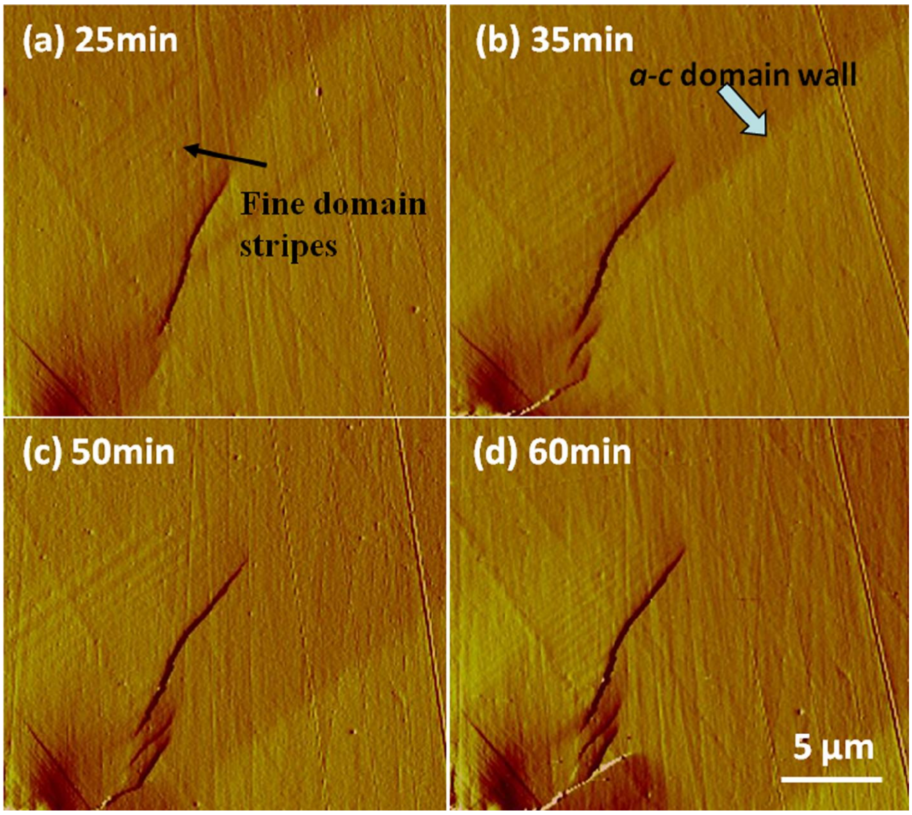

**Fig. 5** Details of the domain stripes around a crack when the 90° ***a-c*** domain wall traversed the indentation: (a) 25 min, (b) 35 min, (c) 50 min, (c) 60 min

around the indentation. In fact, the movement of the 90° ***a-c*** domain wall is a polarization process from ***c*** domains to ***a*** domains. The 90° ***a-c*** domain wall is hampered by residual stress zone and begin to bend along the edge of residual stress zone, when it ı near to the indentation. The incompatible stra duced by 90° ***a-c*** domain switching on the be position of domain wall results in the crack p gated to outside of the residual stress zone. At this moment, the residual stress is relaxed by the crack propagation (OA and OB) on one side of the indentation. Therefore, the cracks on the other side of indentation hardly propagate (OC and OD). Moreover, the crack propagation also lead to the incompatible strain, resulting in the changes of the fine domain stripes around the crack tip.

## 4. Conclusions

The crack propagation and domain switching around the indentation under an external electric field were in-situ investigated using PLM and AFM. The wirelike 90° $\boldsymbol{a}$–$\boldsymbol{c}$ domain stripes were observed around the four corners of the indentation. Under the external electric field, $\boldsymbol{c}$ domains switched to $\boldsymbol{a}$ domains through the movement of a long straight 90° $\boldsymbol{a}$-$\boldsymbol{c}$ domain wall perpendicular to the electric field direction. When the 90° $\boldsymbol{a}$-$\boldsymbol{c}$ domain wall traversed the indentation, the domain wall was hampered by the residual stress around the indentation. The driving force of the crack propagation was the incompatible strain induced by domain switching on the bending position of 90° $\boldsymbol{a}$-$\boldsymbol{c}$ domain wall at the edge of the residual stress zone. The changes of fine domain stripes were induced by the crack propagation.

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