Locking of Electric Field Induced Non-180^o Domain Switching and Phase Transition in Ferroelectric Materials upon Cyclic Electric Fatigue

M. Liu and K.J. Hsia*

Department of Theoretical and Applied Mechanics, University of Illinois at Urbana-Champaign, Urbana, IL 61801

* Corresponding author. fax: +1-217-244-5707, E-mail: kj-hsia@uiuc.edu

Abstract

In-situ x-ray diffraction measurements are conducted on a polycrystalline ferroelectric material PZT-5H at different levels of static electric field. The locking of the electric field induced non-180^o domain switching and phase transition in PZT-5H after experiencing cyclic electric field is investigated by examining the changes in pseudo-cubic diffraction profiles. The results show that cyclic electric field with an amplitude lower than the coercive field has little effect on ferroelectric fatigue of the material, whereas cyclic field with an amplitude above the coercive field results in nearly complete locking of non-180^o domain switching and phase transition. The results also demonstrate that the locking occurs rather suddenly after $10^3 – 10^4$ cycles. This locking phenomenon can explain the dramatic change in piezoelectric coefficients in these materials upon cyclic electric field.

Perovskite ferroelectric ceramics are promising materials for applications in micro-actuators and nonvolatile memories[1-3]. However, under cyclic bipolar electric fields, ferroelectric materials often undergo electric fatigue, resulting in significant decrease in the number of switchable domains. The locking of switchable domains causes the loss of memory function, reduces the piezoelectric effect, and causes the actuators to function improperly. Such behavior is therefore the topic of extensive study in the past few years[4-17]. There are mainly two approaches to the analysis of ferroelectric fatigue. One approach is through investigation of the change of the material properties, such as the reduction of remnant polarization and piezoelectric constants[4-12]. Another approach is by observing the locking of switchable domains directly[13,14]. To date, the observation has only been conducted to detect the locking of 180^o domain switching, although some efforts have been made to distinguish the 180^o domain switching from the non-180^o domain switching[18,19].

A literature survey often shows conflicting conclusions as regard to the cause of ferroelectric fatigue. The change of materials constants usually indicates that the repeated 180^o domain switching causes locking of switchable domains[8,11]. Recent research, by applying an electric field inclined to the material's spontaneous polarization direction, showed that domain locking was likely caused by field-induced phase transitions[7]. On the other hand, the extraordinarily strong and nonlinear piezoelectric constants found in the ferroelectric materials with compositions near the Morphotropic Phase Boundary (MPB)[20-21] are believed to result from the combined electric field-induced non-180^o domain switching and phase transition, as well as from the residual stresses generated by the poling process[22-24]. A thorough understanding of the ferroelectric fatigue behavior, therefore, depends critically on direct, *in-situ* observations of non-180^o domain switching and phase transition under applied electric field.

In this work, we conduct *in-situ* x-ray diffraction study on freshly poled and electrically fatigued samples under different levels of static electric field. PZT-5H, a material with compositions near the MPB, is considered. Thin plates with dimensions of 2 cm × 2 cm × 375 μm, coated with nickel electrodes on top and bottom surfaces, and poled in the thickness direction, are used. The material has a coercive field of E_C = 8 kV/cm. The main objective of the measurements is to provide definitive information on the non-180^o domain switching and phase transition induced by static electric fields in freshly poled and electrically fatigued specimens.

The experiment is conducted in two steps. The first step involves subjecting three groups of specimens with different cyclic electric fields: the first group is electrically fatigued with cyclic electric field of ±1.1 E_C in amplitude at 50 Hz; the second group is electrically fatigued with cyclic electric field of ±0.8 E_C in amplitude at 50 Hz; and the third group, as a comparison group, is left in the freshly poled state. The number of cycles applied to the fatigued specimens is 10^3, 10^4, and 10^5. Since the terminating field level cannot be controlled during cyclic loading, to ensure consistency of the results, a static electric field of the same magnitude as the amplitude of cyclic electric field is applied in the initial poling direction for one minute. The second step involves recording *in-situ* x-ray diffraction profiles of each specimen at static electric field levels of 0.8 E_C, 0, -0.8 E_C, and -1.0 E_C, respectively. The positive static field is in the poling direction.

2θ-θ scans were performed using a Rigaku D-Max powder x-ray diffractometer. The instrument uses copper radiation with two lines $K\alpha_1$ and $K\alpha_2$ (wavelength λ_{α_1} =

1.5406 Å and λ_{α_2} = 1.5444 Å). Reflections at pseudo-cubic (200), (111), and (220) regions were recorded over a 3^{o}-4^{o} 2θ range at a speed of 0.3^{o}/min. Due to the material's morphotropic and polycrystalline microstructures, the diffraction profiles are rather complicated. Each pseudo-cubic peak consists of multiple peaks from different phases.

A peak fitting algorithm was used to identify the peaks of individual phases co-existing in the material. Figure 1 shows the reflections at pseudo-cubic (200) region and the fitted peaks of individual phases in a fresh sample at static electric field 0.0 E_C and -0.8 E_C, in which "T" and "R" stand for tetragonal and rhombohedral phases. Comparing the fitted peaks at static field levels of 0.0 E_C and –0.8 E_C in Figure 1, we can see that there exist 90° domain switching from T(002) to T(200) and phase transition from T(002) to R(200). Such non-180^{o} domain switching and phase transition dramatically change the profile of the pseudo-cubic (200) peak. The detailed description of the peak fitting method is given elsewhere[25].

Figure 2 shows the reflections at pseudo-cubic (200) region in a freshly poled sample at static field levels of 0.8 E_C, 0, -0.8 E_C, and -1.0 E_C, respectively. It is seen that, at 0.8 E_C, the left peak at pseudo-cubic (200) region increases slightly while the right peak decreases slightly, indicating some amount of non-180^{o} domain switching and phase transition that re-align some domains to the poling direction[25]. Upon a negative electric field closed to the negative coercive field (-0.8 E_C), profound phase and domain changes occur, which rotate the domain polarization to directions perpendicular or inclined to the poling direction. The changes can induce a large contraction (i.e., negative strain) in the poling direction, therefore enhances the piezoelectric effect and is responsible for the large minima in the strain-electric field hysteresis loop[26]. However, at -1.0 E_C, the polarization of domains switches to the negative poling direction, as demonstrated by the similar pseudo-cubic (200) reflections at 0.8 E_C and -1.0 E_C.

Figure 3 shows the effects of cyclic electric field (10^5 cycles) on the material's ability to undergo further non-180^o domain switching and phase transition upon static field. Figures 3a and 3b show the pseudo-cubic (200) reflections under different static field levels for samples fatigued at ±0.8 E_C and fatigued at ±1.1 E_C, respectively. It clearly shows that cyclic electric field has little effect on further domain and phase changes if the amplitude of the cyclic electric field is lower than the coercive field. However, if the amplitude of the cyclic field is beyond the coercive field (1.1 E_C as in Fig. 3b), the ability for the material to undergo further non-180^o domain switching and phase transition upon static electric field is nearly completely suppressed.

Figure 4 shows the change of the pseudo-cubic (200) peak upon a static electric field of -0.8 E_C after experiencing different number of cycles of electric field of ±1.1 E_C. It is clear that the locking of the non-180^o domain switching and phase change takes place between 10^3 and 10^4 cycles, which is a very short lifetime. Furthermore, such locking appears to occur relatively suddenly.

The present work employs *in-situ* x-ray diffraction to investigate the phenomenon of the locking of non-180° domain switching and phase transition in ferroelectric materials upon cyclic electric field. Major findings of this study are: 1) in a freshly poled PZT-5H, a static negative electric field of magnitude below the coercive field induces significant non-180° domain switching and phase transition, while a negative coercive field induces predominantly 180° domain switching; 2) the material's ability to undergo non-180° domain switching and phase transition is not affected when the material is electrically fatigued at amplitudes lower than the coercive field, but such ability is completely suppressed if the cyclic field amplitude is above the coercive field; 3) the locking of non-180° domain switching and phase transition occurs rather suddenly upon cyclic electric field between 10^3 and 10^4 cycles. Similar observations were found for other pseudo-cubic peaks such as (111) and (220). One effect of such locking phenomenon is to decrease the piezoelectric coefficients greatly, which may explain why there is only a small minima in the strain-electric field hysteresis loop measured in similar PZT materials[27].

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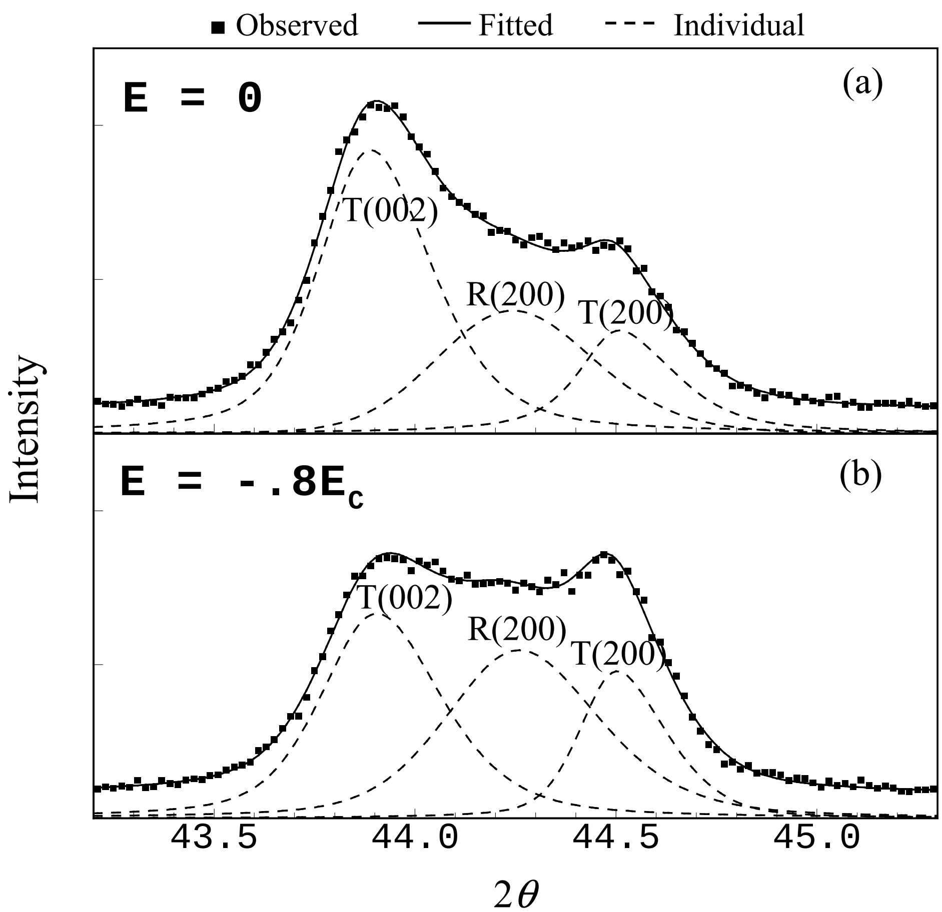

Fig.1 Profile of the pseudo-cubic (200) peak and the fitted peaks of individual phases in a freshly poled sample upon static electric field, (a) at $E = 0$, (b) at $E = -.8E_C$

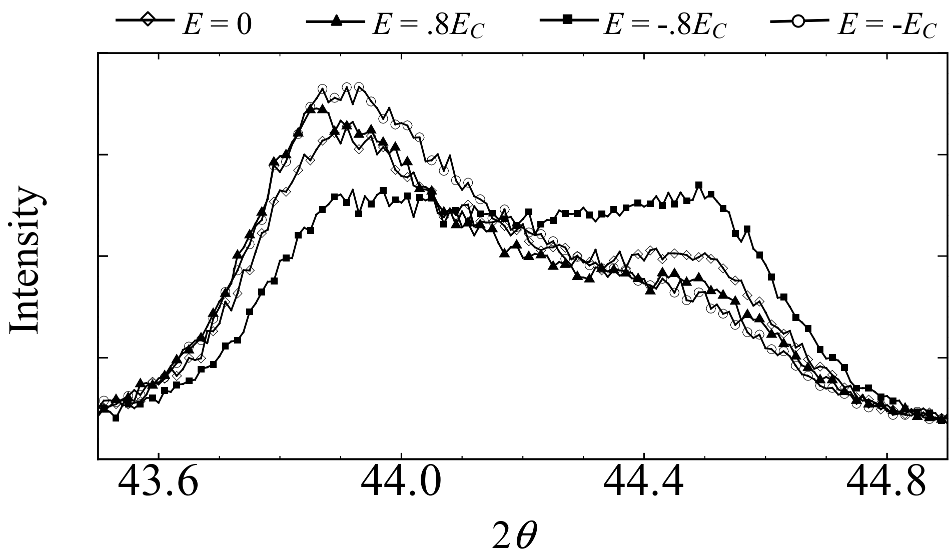

Fig.2 The pseudo-cubic (200) peaks at different levels of static electric field in a freshly poled sample

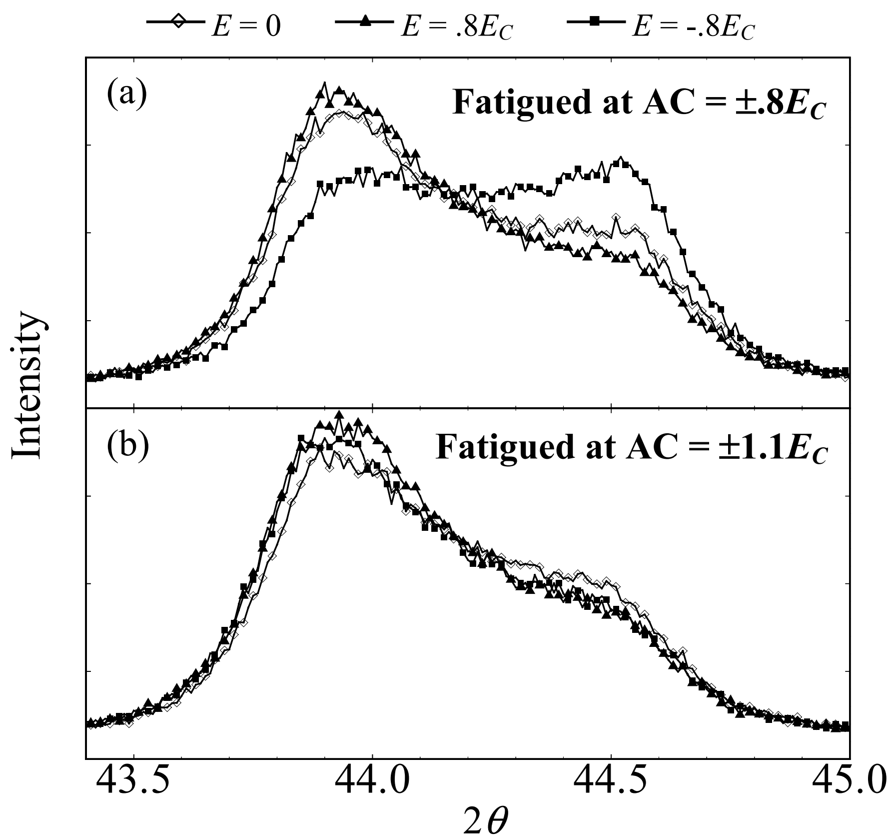

Fig.3 Effects of bipolar electric field (AC, 50Hz, 10^5 cycles) on the pseudo-cubic (200) peaks at different static electric field: (a) fatigued at AC amplitude of ±0.8 E_C; (b) fatigued at AC amplitude of ±1.1 E_C

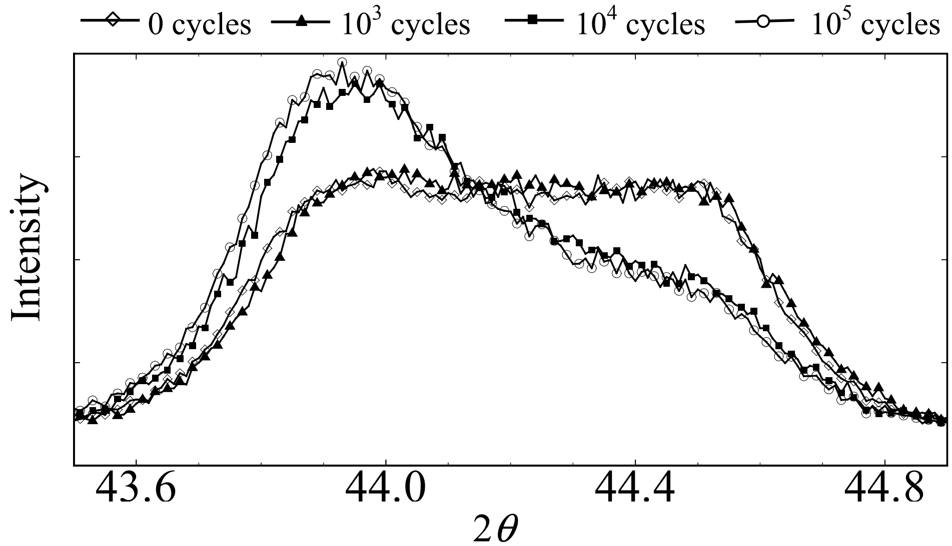

Fig.4 Effects of the number of cycles of bipolar electric fields (AC, 50Hz, ±1.1E_C) on the pseudo-cubic (200) peak at a static electric field of -0.8 E_C

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1005	Fried, E., and B. C. Roy	Gravity-induced segregation of cohesionless granular mixtures—*Lecture Notes in Mechanics*, in press (2002)	July 2002
1006	Tomkins, C. D., and R. J. Adrian	Spanwise structure and scale growth in turbulent boundary layers—*Journal of Fluid Mechanics* (submitted)	Aug. 2002

List of Recent TAM Reports (cont'd)

No.	*Authors*	*Title*	*Date*
1007	Riahi, D. N.	On nonlinear convection in mushy layers: Part 2. Mixed oscillatory and stationary modes of convection—*Journal of Fluid Mechanics* (submitted)	Sept. 2002
1008	Aref, H., P. K. Newton, M. A. Stremler, T. Tokieda, and D. L. Vainchtein	Vortex crystals—*Advances in Applied Mathematics* **39**, in press (2002)	Oct. 2002
1009	Bagchi, P., and S. Balachandar	Effect of turbulence on the drag and lift of a particle—*Physics of Fluids* (submitted)	Oct. 2002
1010	Zhang, S., R. Panat, and K. J. Hsia	Influence of surface morphology on the adhesive strength of aluminum/epoxy interfaces—*Journal of Adhesion Science and Technology* (submitted)	Oct. 2002
1011	Carlson, D. E., E. Fried, and D. A. Tortorelli	On internal constraints in continuum mechanics—*Journal of Elasticity* (submitted)	Oct. 2002
1012	Boyland, P. L., M. A. Stremler, and H. Aref	Topological fluid mechanics of point vortex motions—*Physica D* **175**, 69–95 (2002)	Oct. 2002
1013	Bhattacharjee, P., and D. N. Riahi	Computational studies of the effect of rotation on convection during protein crystallization—*Journal of Crystal Growth* (submitted)	Feb. 2003
1014	Brown, E. N., M. R. Kessler, N. R. Sottos, and S. R. White	*In situ* poly(urea-formaldehyde) microencapsulation of dicyclopentadiene—*Journal of Microencapsulation* (submitted)	Feb. 2003
1015	Brown, E. N., S. R. White, and N. R. Sottos	Microcapsule induced toughening in a self-healing polymer composite—*Journal of Materials Science* (submitted)	Feb. 2003
1016	Kuznetsov, I. R., and D. S. Stewart	Burning rate of energetic materials with thermal expansion—*Combustion and Flame* (submitted)	Mar. 2003
1017	Dolbow, J., E. Fried, and H. Ji	Chemically induced swelling of hydrogels—*Journal of the Mechanics and Physics of Solids* (submitted)	Mar. 2003
1018	Costello, G. A.	Mechanics of wire rope—Mordica Lecture, Interwire 2003, Wire Association International, Atlanta, Georgia, May 12, 2003	Mar. 2003
1019	Wang, J., N. R. Sottos, and R. L. Weaver	Thin film adhesion measurement by laser induced stress waves—*Journal of the Mechanics and Physics of Solids* (submitted)	Apr. 2003
1020	Bhattacharjee, P., and D. N. Riahi	Effect of rotation on surface tension driven flow during protein crystallization—*Microgravity Science and Technology* (submitted)	Apr. 2003
1021	Fried, E.	The configurational and standard force balances are not always statements of a single law—*Proceedings of the Royal Society* (submitted)	Apr. 2003
1022	Panat, R. P., and K. J. Hsia	Experimental investigation of the bond coat rumpling instability under isothermal and cyclic thermal histories in thermal barrier systems—*Proceedings of the Royal Society of London A* (submitted)	May 2003
1023	Fried, E., and M. E. Gurtin	A unified treatment of evolving interfaces accounting for small deformations and atomic transport: grain-boundaries, phase transitions, epitaxy—*Advances in Applied Mechanics* (submitted)	May 2003
1024	Dong, F., D. N. Riahi, and A. T. Hsui	On similarity waves in compacting media—*Advances in Mathematics Research* (submitted)	May 2003
1025	Liu, M., and K. J. Hsia	Locking of electric field induced non-180° domain switching and phase transition in ferroelectric materials upon cyclic electric fatigue—*Applied Physics Letters* (submitted)	May 2003
1026	Liu, M., K. J. Hsia, and M. Sardela Jr.	In situ X-ray diffraction study of electric field induced domain switching and phase transition in PZT-5H—*Journal of the American Ceramics Society* (submitted)	May 2003