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13. ABSTRACT (Maximum 200 words)  Effects of electrical fields on cracking behavior of ceramics were examined in four different ceramics, namely, glass, magnesium oxide (MgO), lead zirconate titanate (PZT) and lead lanthanum zirconate titanate (PLZT). When the electrical field was non-orthogonal to the crack plane, deflection of microcracks occurred. The deflection angle was as high as 90 degrees from the original crack-plane, depending on the relative orientation of the electrical field to the crack plane. In piezoelectric ceramics, crack growth was observed under both DC and AC electric fields. The crack growth under AC electric fields was a strong function of the mean electrical field. Micromechanisms of cracking were examined by a new in-situ transmission electron microscopy technique, developed for observations of electric-field induced cracking in ferroelectric ceramics. With this technique, cracking of the domain boundary under both static and alternating electric field was directly observed for the first time in PMN-PT crystals and in a commercial EC65 PZT ceramic. Domain alignments and switching were also directly imaged near crack-like flaws.				
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## **FINAL PROGRESS REPORT**

### **Statement of the problem studied**

The extreme hardness and thermal resistance of ceramics have made interesting materials for a wide range of Army structures. However, the high susceptibility of ceramics to cracking has limited their widespread use. The purpose of our research was to examine effects of electric fields on crack propagation in ceramics and to seek mechanistic understanding of the cracking process under the influence of the electric field, so that crack propagation in ceramic materials and structures may be ultimately controlled or prevented.

### **Summary of the most important results**

#### **A. Effects of electric fields on crack propagation**

Effects of electrical fields on cracking behavior of ceramics were examined in four different oxides, namely, glass, magnesium oxide (MgO), lead zirconate titanate (PZT) and lead lanthanum zirconate titanate (PLZT). Our efforts were focussed on two significant aspects of the field-influenced crack growth, namely field-induced crack deflection and field-induced cracking of PZT and PLZT. Deflection of microcracks occurred when the electrical field was non-orthogonal to the crack plane. The deflection angle was as high as 90 degrees from the original crack-plane, depending on the relative orientation of the electrical field to the crack plane. Under both DC and AC electric fields, crack growth was observed in piezoelectric ceramics. The crack growth under AC electric fields was a strong function of the mean electrical field.

#### **1. Deflection of microcracks by electrical field**

In ceramics, microcracks oriented perpendicular to tensile stress axis are most dangerous in triggering catastrophic failures. In this study, the potential use of electric field in diverting crack away from the original crack plane (assumed plane of the maximum tensile stress) was explored in PZT and PLZT ceramics. The role of electric field in deflecting microcracks was examined by applying electric fields parallel, perpendicular, and inclined to the crack plane.

##### **a). Perpendicular Field**

When the electric field was perpendicular to the crack plane, the crack extends in a self-similar manner by propagating ahead on the original crack-plane, as shown in Fig. 1. The crack growth was stable up to very long length and crack speed was apparently independent of the crack length.

If the specimen is poled along the direction perpendicular to the applied AC electric field, the original crack forked to two branches, with each branch lying 45

degrees from the original plane (Fig. 2). Crack growth was much slower after the fork developed.

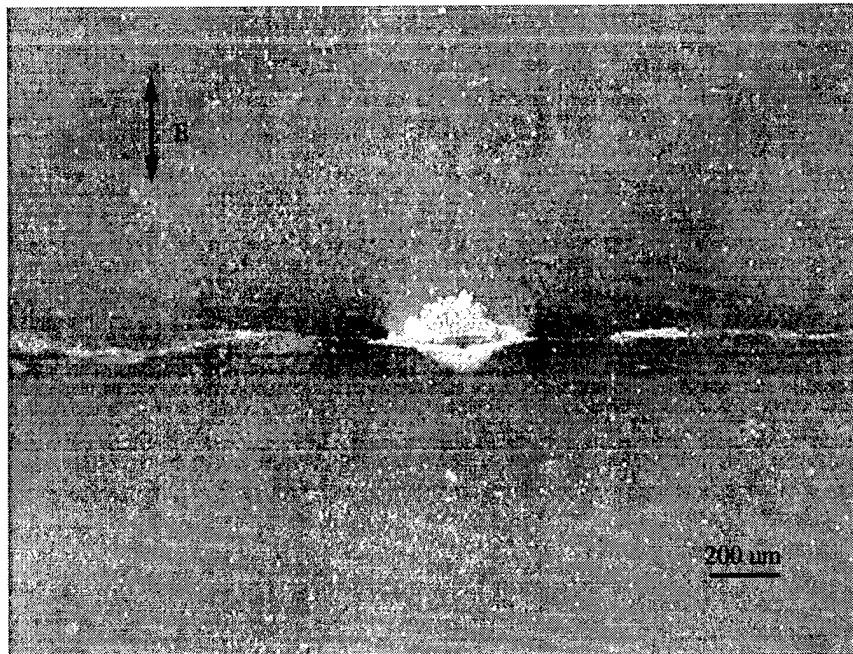


Fig. 1 Crack growth in PZT under a perpendicular electric field of 12 kV/cm.

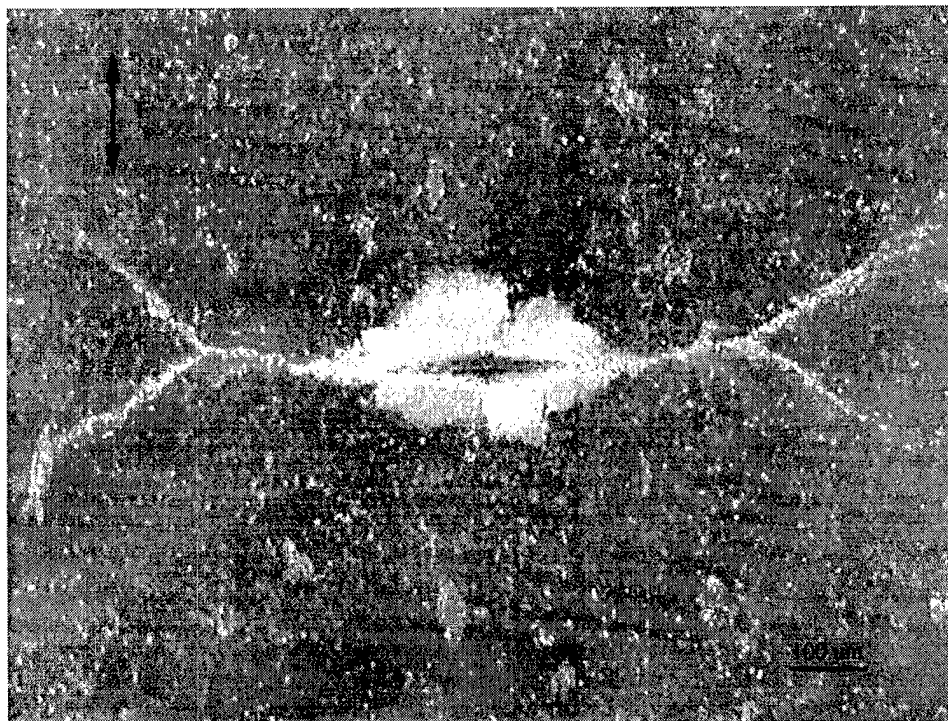


Fig. 2 Crack deflection in poled PZT under a perpendicular field of 14 kV/cm.

### b). Parallel Field

In the virgin state, a parallel field had no effect on the original indentation crack. After the specimen was poled along the crack orientation, a parallel field extended the original crack for a small distance and then turned the crack into a fork. The deflected crack branches propagated along a plane 70-80 degrees away from the original crack plane (Fig. 3). Both branches came to arrest after some distances.

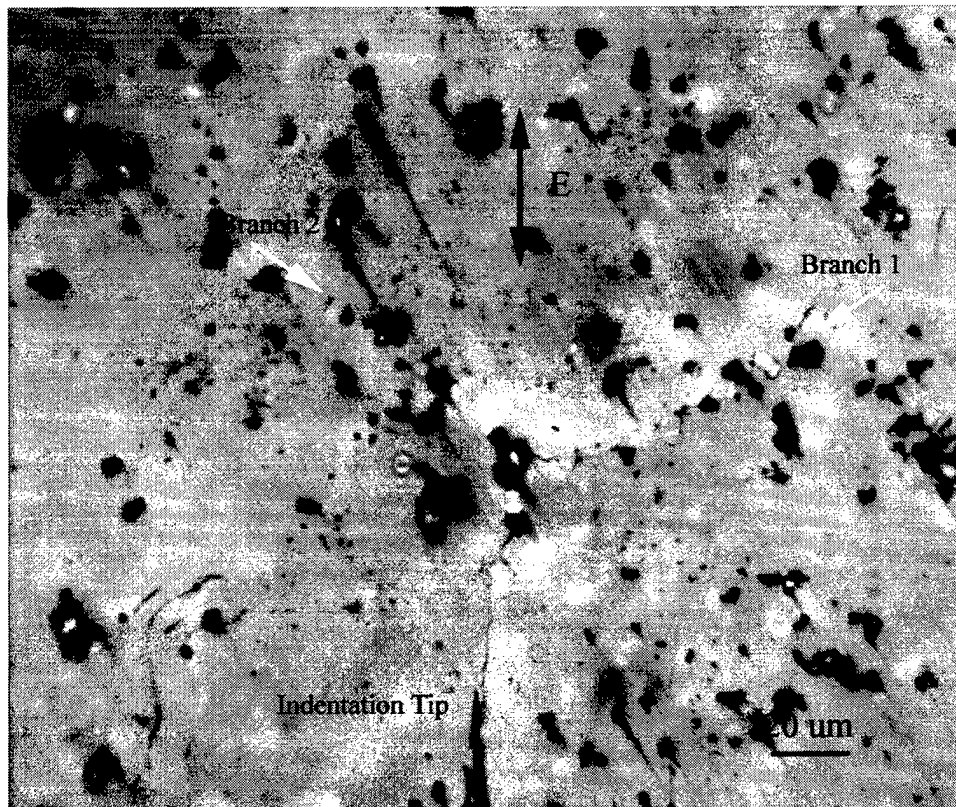


Fig. 3 Crack deflection in poled PZT under a parallel field of 14 kV/cm.

### c) Inclined Field

When the crack plane was inclined to the electric field, crack was deflected away from the original crack plane. As shown in Fig. 4, crack in PLZT preferred to extend on the plane perpendicular to the applied electric field. After deflection, the crack grew at nearly the same rate as that for the crack extending under a perpendicular field.

In the poled PZT specimen, when the applied field was perpendicular to the poling direction, but inclined 45 degrees to the crack plane, crack grew a small distance along the original crack plane, and then turned nearly 90 degrees (Fig. 5).

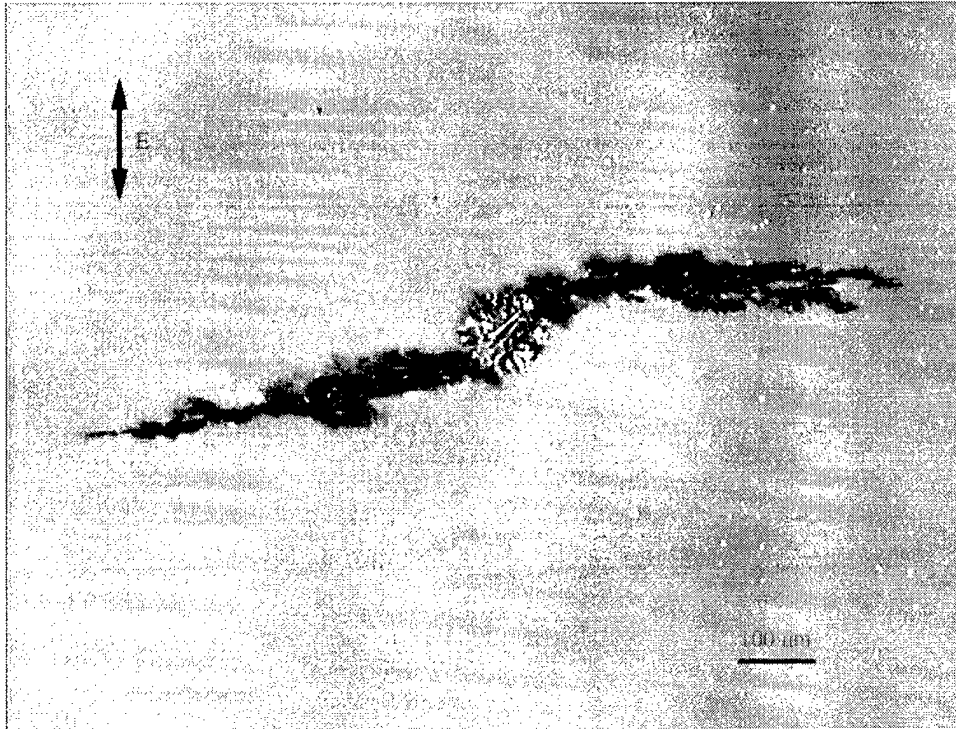


Fig. 4 Crack deflection in PLZT under an inclined field of 18 kV/cm.

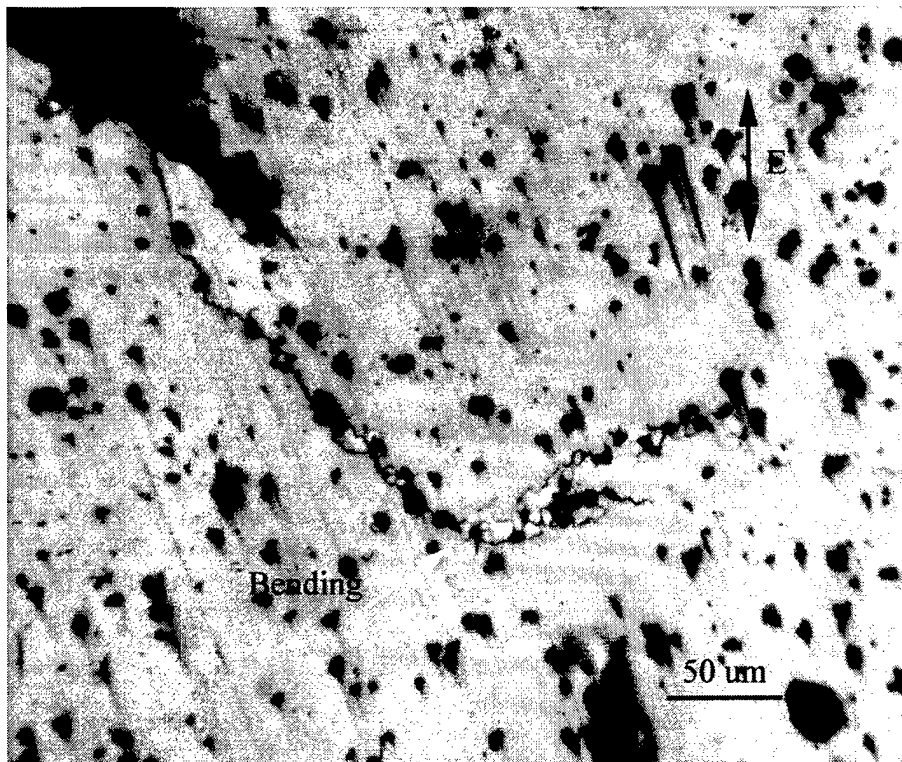


Fig. 5 Crack deflection in poled PZT under an inclined field of 14 kV/cm.

## 2. Crack growth in PLZT under DC and AC electric fields

DC electric field and sinusoidal AC fields of various amplitudes and mean levels were applied to mechanically precracked PLZT beams and crack growth rates were obtained from precracked specimens. As shown in Fig. 6, under AC electric field, the mechanical precrack began to grow when the peak field strength reached 8.5 kV/cm and continued its growth as long as comparable or higher field strength was maintained. The crack eventually grew very close to the end of the specimen and caused specimen fracture.

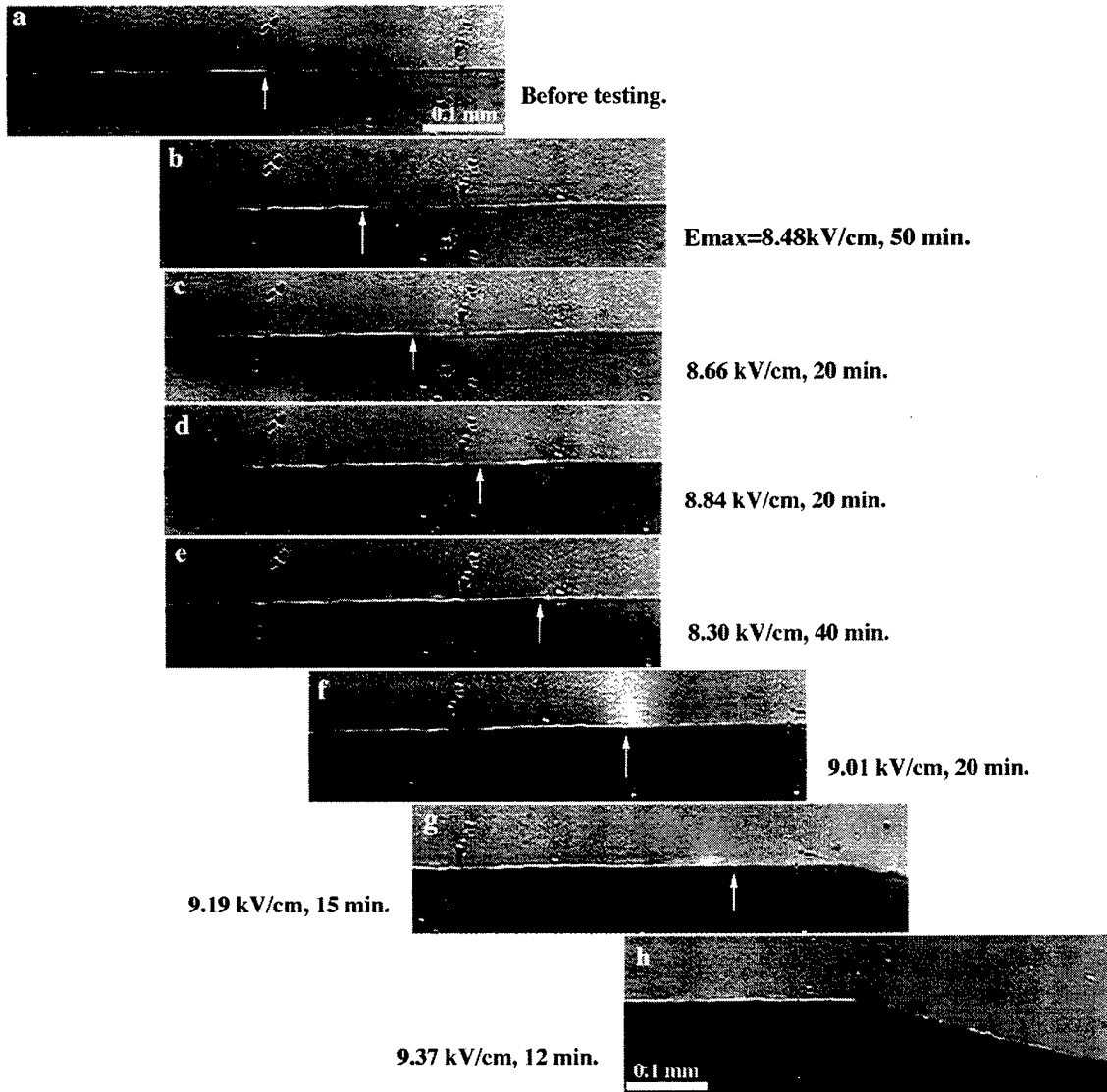


Fig. 6. Crack growth in PLZT under AC electric fields.

The relationship between crack growth rate and electric-field strength is shown in Fig. 7 at different minimum to maximum field ratios ( $E_{\min}/E_{\max}$ ). As the field ratio or the mean level of the AC field increases, the threshold electric field increases, but the range of the field strengths within which stable crack growth may be obtained becomes smaller. Distinct differences in fracture mechanisms were noted at different electric-field ratios. Fracture mechanism under completely reversed AC electric field ( $E_{\min}/E_{\max} = -1$ ) was transgranular, similar to the fracture mechanism under mechanical loading. However, crack growth turned mixed intergranular and transgranular at the field ratio of zero, and was predominantly intergranular at the field ratio of 0.2 and 1.0 (DC field).

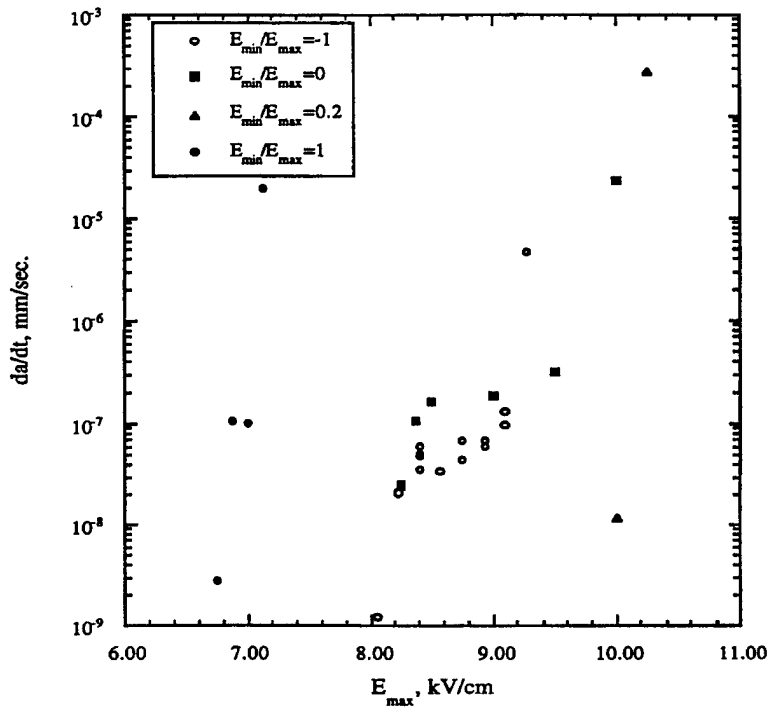


Fig. 7. Crack velocity as a function of the maximum field under DC and AC fields.

## B. Micromechanisms of cracking under electric fields

While the first half of our work had clearly shown that crack growth behavior in ceramics could be greatly modified by the application of an electric field, the micromechanism of the field-induced cracking remained elusive. In addressing this problem, we came up with a new experimental technique, which allowed direct in-situ observations of the micromechanisms of field-induced cracking to be made for the first time in a transmission electron microscope. We have used this new technique to obtain direct evidence of domain-boundary cracking, domain alignment and domain switching near crack-like flaws in piezoelectric crystals and ceramics.

## 1. Domain-boundary Cracking

Figure 8(a)-(d) are photomicrographs taken from an in-situ TEM study of the field-induced cracking in PMN-PT piezoelectric crystal. It shows the response of a small defect, a fine pore of  $\sim 0.22 \times 0.15 \mu\text{m}^2$  in size (Figure 8(a) and (c)), to an applied alternating field along the marked direction. When an alternating electric field of 25kV/cm was applied to the specimen for 20 cycles, microcracks were developed, as illustrated in Figure 8(b) and (d). According to the electron diffraction analysis, the microcrack followed the  $\{110\}$  domain boundary of the crystal.

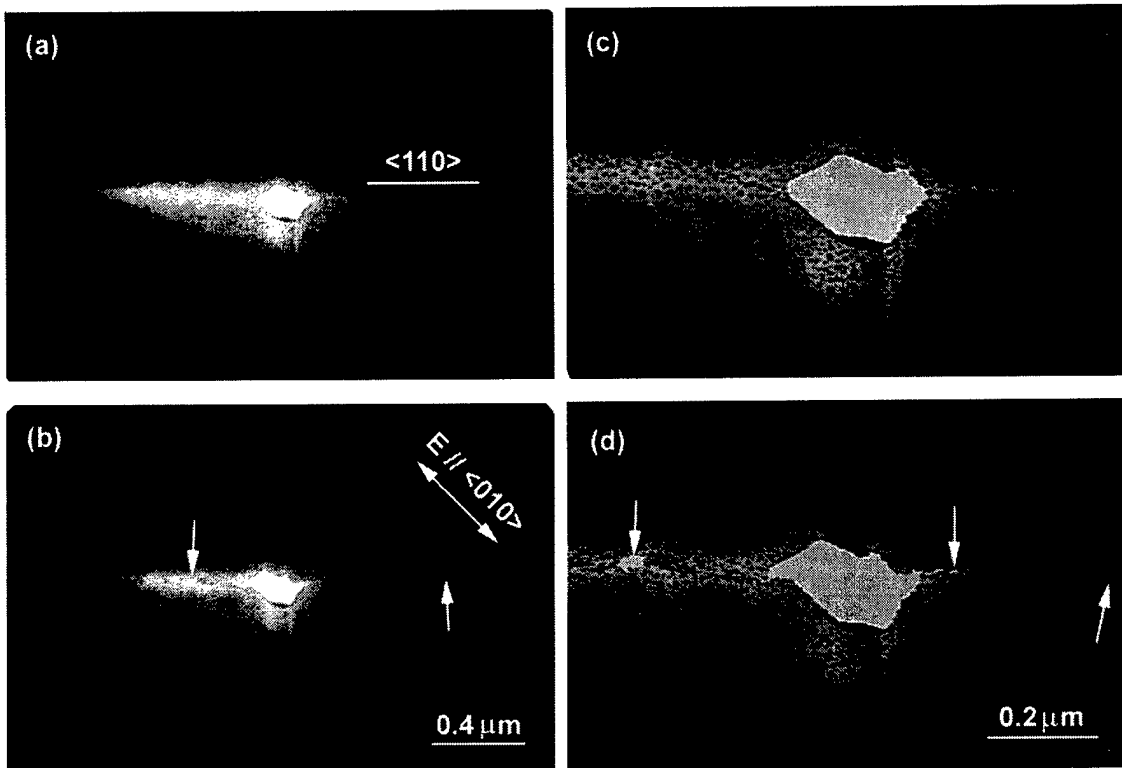


Figure 8. Effects of a cyclic electric field on microcrack initiation in single crystal 0.66PMN-0.34PT specimen. (a) and (c)  $E = 0$ ; and (b) and (d)  $E = \pm 25 \text{ kV/cm}$  for 20 cycles.

## 2. Domain Alignment around Crack-like Flaw

Figure 9 (a)-(c) were taken from an in-situ TEM study of the field-induced cracking in EC65 PZT ceramic, proving direct evidence of domain alignment near a crack-like flaw. Figure 9a shows the original domain structure of the annealed (depoled) specimen. Randomly oriented nano-domains with short range tweed features were observed. Diffraction analysis indicated these tweed features were along  $\{110\}$  plane, as noted by the plane trace line in the figure. Upon the application of a static field of

+4.0MV/m, nano-domains were forced to align along another {110} plane and the long range tweed structure emerged, as shown in Figure 9b. Release of the field could not resume the original domain structure. The reversion of the static field moved the tweed structure farther, as shown in Figure 9c.

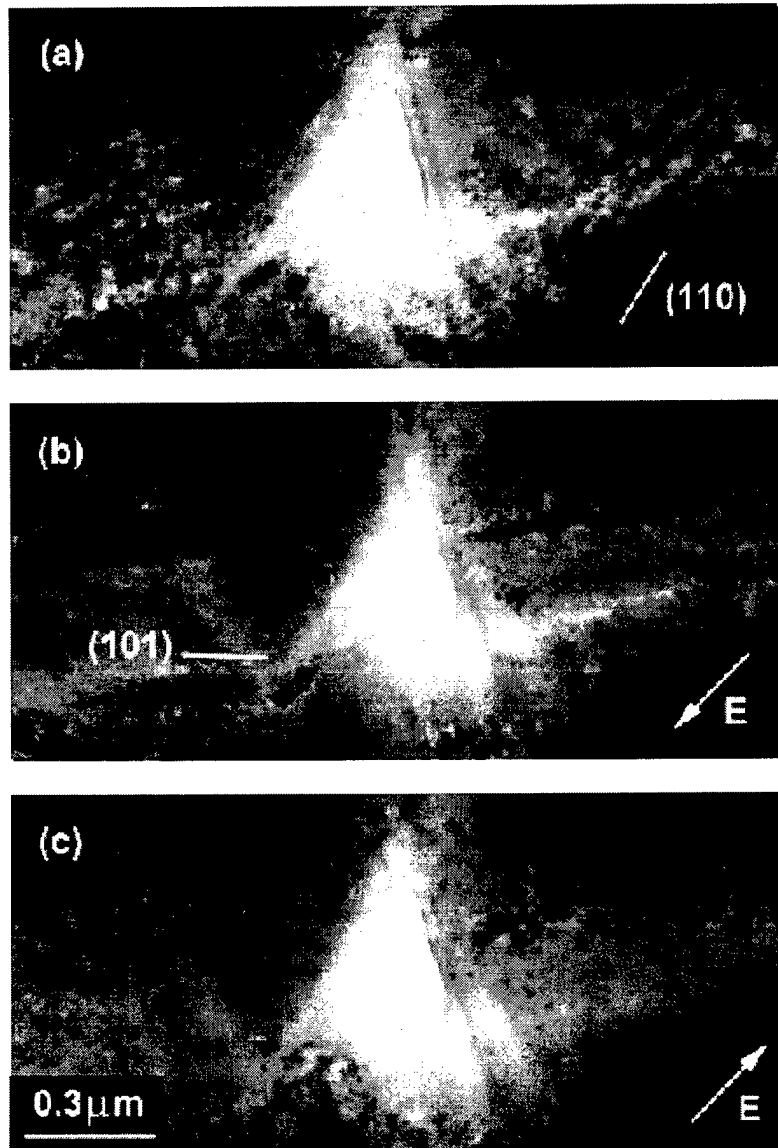


Figure 9. Electric-field-induced domain alignment and microcrack within a single grain: (a) domain structure at original annealed state; (b) electric field +4.0 MV/m; (c) electric field -4.0 MV/m.

### 3. Domain Switching at Crack Tip

Figure 10(a)-(d) were TEM images obtained from an in-situ TEM study of a fine-grained Mn-doped PZT ceramic, showing domain switching at the tip of a preexisting

crack in a complete electric cycle. Domain switching was seen as changes in the domain contrast or movement/disappearance of the domain walls. The microcrack was intergranular and just at the tip of it there was a grain with well-defined domains, indicated by a triangle in Figure 10a. Upon the application of the electric field, changes of the domain width in this grain were observed, as shown in Figure 10b. The contrast of these domains faded away (see Figure 10c) as the electric field reversed its polarity. Figure 10d shows that as the field was released, the domain configurations resumed to the state before the application of the electric field.

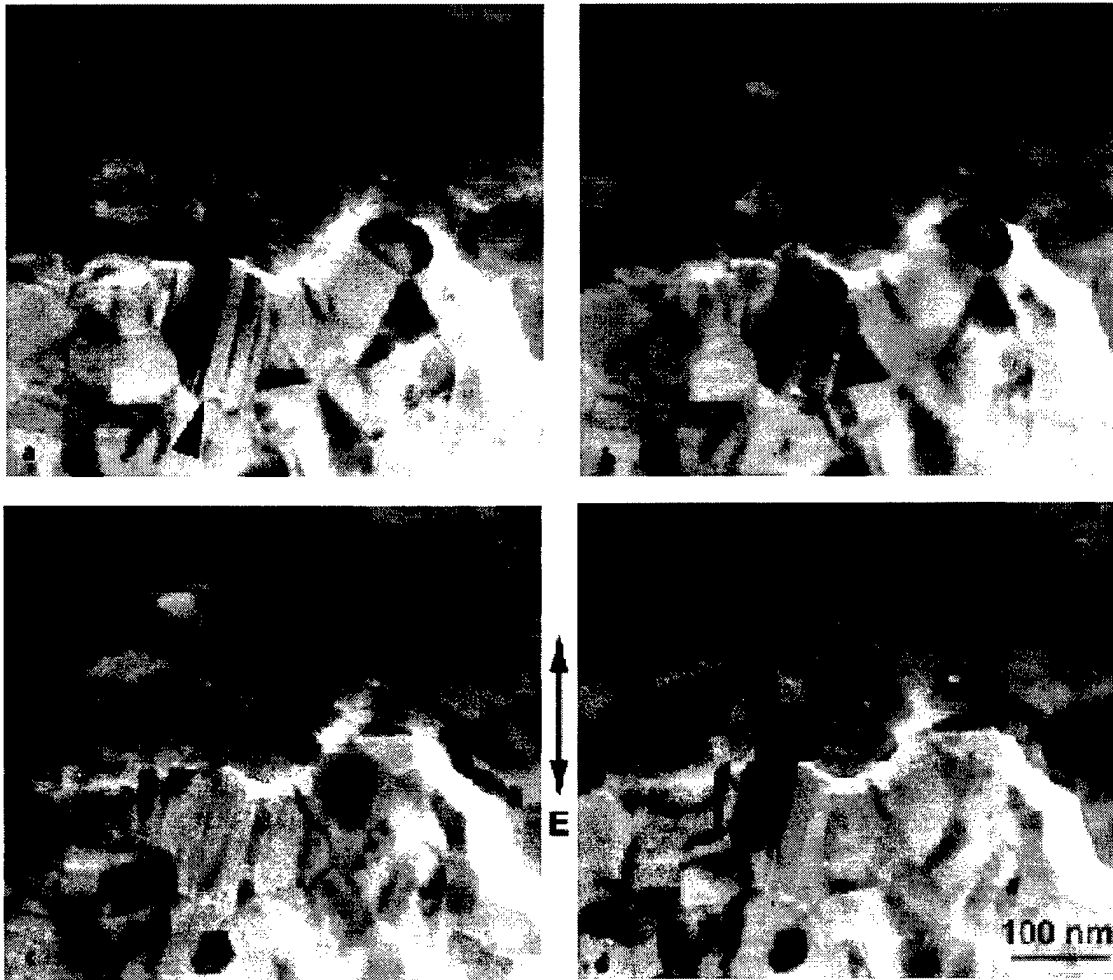


Figure 10. Domain switching at the crack tip in a complete electric cycle in PZT ceramic doped with 1% Mn: (a) electric field 0; (b) electric field +4.0 MV/m; (c) electric field -4.0 MV/m; (d) electric field released.

## **Publications:**

1. "Crack Deflection in Relaxor Ferroelectric PLZT under Inclined Cyclic Electric Field," X. Tan and J. K. Shang, to appear in Scripta Mater., 2000.
2. "In situ TEM study of electric-field-induced crack initiation and growth in single crystal  $0.66\text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3-0.34\text{PbTiO}_3$ ," Z. Xu, X. Tan, and P. Han and J.K. Shang, Appl. Phy. Lett., vol. 76, No. 25, 3732, 2000.
3. "Direct observations of electric field-induced domain boundary cracking in  $\langle 001 \rangle$  oriented piezoelectric  $\text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3-\text{PbTiO}_3$  single crystal," X. Tan, Z. Xu, J.K. Shang and P. Han, submitted to Appl. Phy. Lett., 2000
4. "DC electric-field driven crack propagation in mechanically-precracked La-modified lead zirconate titanate ceramics", Z. Xing, J. Li, E. Furman, Z. Xu, J. Shang and D. Viehland, submitted to J. Appl. Phys., 1999.

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