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Characterizations of fatigue and crack growth of ferroelectrics under cyclic electric field

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Abstract The behavior of fatigue and crack growth of PZT based ferroelectrics under electric field was characterized. It is found that the crack growth shows an anisotropic effect. Under cyclic electric field, crack in the direction perpendicular to the polarization direction grows very fast, whereas in the direction parallel to the polarization direction no clear crack propagation is observed. Low cyclic electric field can also result in crack growth. The results also show that high electric field leads to degradation of the ferroelectric property more quickly than low electric field. The mechanism of the phenomenon is discussed.

Keywords Fatigue · Crack · Ferroelectrics · Cyclic electric field

1 Introduction

$Pb(Zr_x,Ti_{1-x})O_3$ (PZT) ferroelectric ceramics are extensively used in the production of actuators, sensors and memories due to their superior ferroelectric, piezoelectric and pyroelectric properties [1–3]. Above the Curie temperature, they show simple cubic structures, while at lower temperatures they become tetragonal or rhombohedral structures. At a certain temperature, whether a tetragonal or a rhombohedral structure is formed is determined by the composition x. The phase boundary in the phase diagram, which separates the tetragonal from rhombohedral region, is called the morphotropic phase boundary (MPB). Recently, it was suggested that there exists a morphotropic phase region between tetragonal and rhombohedral regions in the phase diagram [4]. Large piezoelectric constants are found in the ferroelectric materials with compositions near the MPB, which are believed to be because of the coexistence of morphotropic phases and the associated phase changes.

It is well known that ferroelectric materials are often applied in conditions of cyclic bipolar electric field, resulting in significant decrease of switchable polarization. This causes the loss of memory function, reduction of the piezoelectric effect and leads to improper working of electric devices. The polarization fatigue is one of the restrictive factors in the full commercialization of PZT ceramics in ferroelectric memories [5, 6]. On the other hand, high mechanical stresses and intense electric fields in the ceramics may cause microcracks to develop which have been considered to significantly affect the performance of the devices [7]. However, only a few experiments have been performed to characterize the behavior of crack growth under cyclic electric field till now. Therefore, further characterization and understanding of cyclic electric field induced crack growth of polarized piezoelectric ceramics are very important to improve the reliability and durability of the electric devices.

In the present article, crack growth in PZT based ferroelectric ceramics was investigated under applied cyclic electric field higher or lower than the coercive field strength by using the Vickers indentation technique. The change in crack length was discussed in connection with the influence of domain switching on the residual stress generating from crack. Ferroelectric fatigue properties of the materials were also characterized.

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2 Experimental procedure

PZT based ceramic powders with compositions near the MPB were synthesized by conventional oxide mixing method. The precursor oxides with high purities were mixed, ball milled, then dried and calcined to form perovskite powders. After remilling, drying and sieving, the powders were pressed into columns with the size of 80×8×4 mm under the pressure of 100 Mpa. The samples were buried in the same composition powders and sintered in a sealed alumina crucible at 920°C for 4 h then cooled down to room temperature in the furnace. Silver electrodes were coated on the two 80×8 mm surfaces and polarization was done alone the 4 mm directions.

The specimens to be used for indentation were polished and heat-treated at 600°C for 30 min to release the residual stress during the polishing. Vickers indentations were made with a standard load of 1.0 kg on the 80×4 mm plane. The ferroelectric properties and electric fatigue process were carried out on RT6000HVS high voltage test system, which measured the polarization using a virtual ground mode. A sine-wave electric field was subjected to the samples with amplitudes 1.1 or 0.11 times the coercive electric fields of the ceramics, and the frequency was 50 Hz, which was slow enough for the full switching of ferroelectric domains. Hysteresis loops were measured before and after the fatigue process for comparison. Fatigue crack propagations were observed through an optical microscope (Hirox KH-1000 Hi-scope).

3 Results and discussions

Figure 1 shows the evolution of the Vickers indentation under cyclic electric fields with the amplitude E=1.1 Ec, here Ec represents the coercive electric field of the materials. It can be seen clearly that after 50,000 electric cycles, the propagations of the cracks in different directions is unlike. There exists a strong anisotropy of crack growth. In the orientation perpendicular to the polarization direction, the crack grows markedly and fast, whereas no obvious propagation is found in the orientation parallel to the polarization direction. This phenomenon can be explained by the domain switching that occurs at the crack tip region. It is well known that most domains are likely to be arranged in the same direction as the polarization direction in the polarized samples. When a crack grows in polarized piezoelectric ceramics there exists an irregular tensile stress field at the crack tip region. If the crack growth direction is parallel to the polarization direction, i.e., the direction of most domains, 90° domain switching occurs at the tip of the crack due to the existence of both the cyclic electric fields and mechanical stress resulting from the piezoelectric effect of the polarized piezoelectric ceramics. And this consequently leads to local stress relaxation and blocks the crack growth. However when the crack growth direction is perpendicular to the polarization direction, 90° domain switching induces an increase of the tensile stress and consequently lead to a continuous growth of the crack.

In order to investigate the influence of cyclic electric field intensity on the crack growth, an electric field with amplitude of 0.11 Ec was subjected to the samples. The result is demonstrated in Fig. 2. It shows that low electric field intensity far from Ec can also result in electric induced fatigue crack growth. The characteristics of the crack growth is the same as that under high electric field, that is to say, the cracks transverse to the polarization direction grow lager and longer than those parallel to the polarization direction after fatigue process. The results differ from some published researches in which the authors preferred a threshold of 0.85 Ec to cause the fatigue crack growth for PIC 151 piezoelectric ceramic [8].

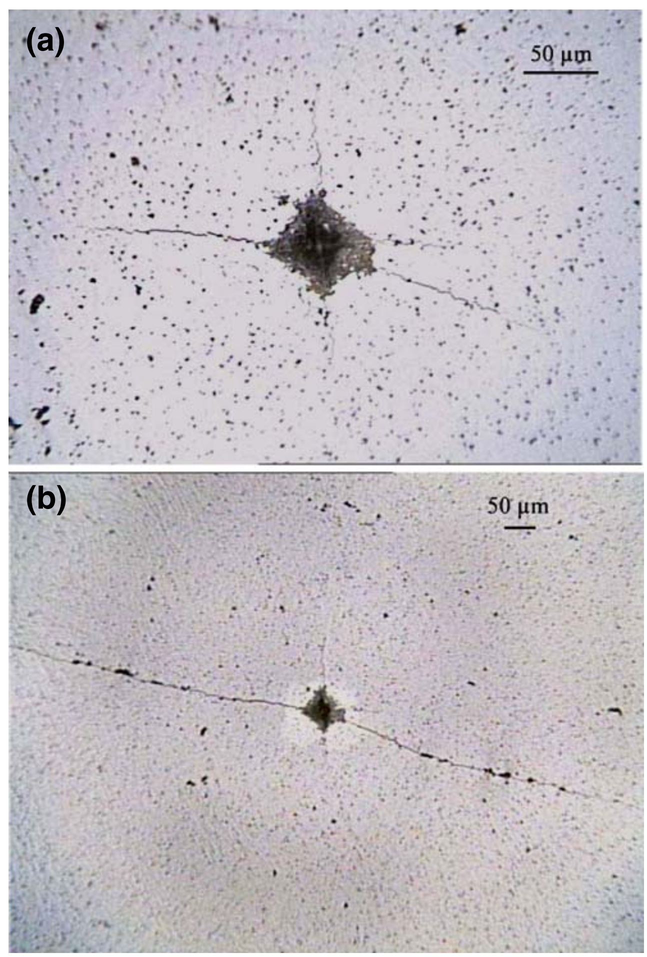

Fig. 1 Micrographs of the Vickers indentation: (**a**) initial, (**b**) after 50,000 electric cycles fatigue (E=1.1 Ec and the polarization direction is vertical)

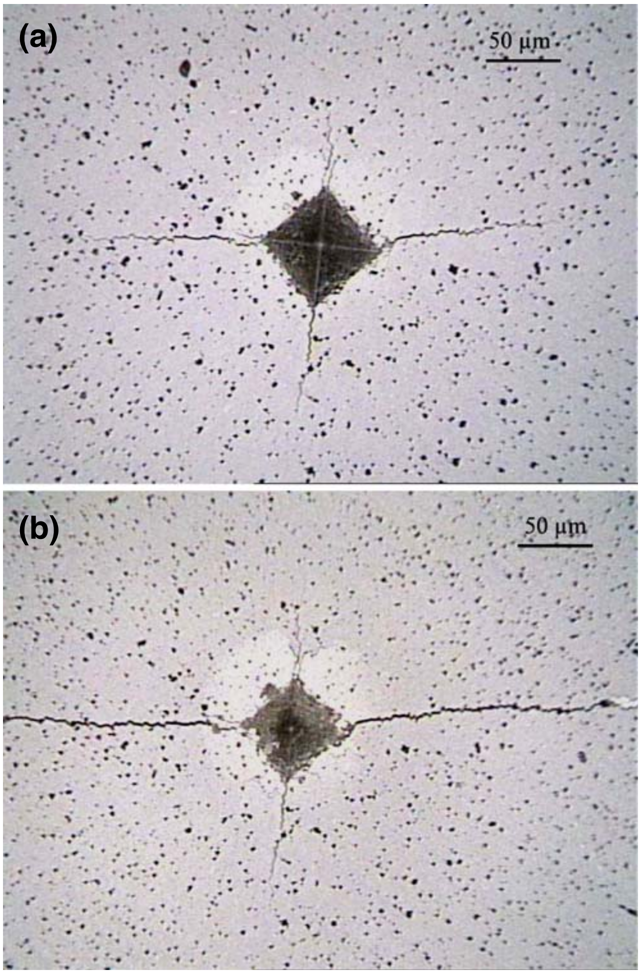

Fig. 2 Micrographs of the Vickers indentation: (**a**) initial, (**b**) after 30,000 electric cycles fatigue (E=0.11 Ec and the polarization direction is vertical)

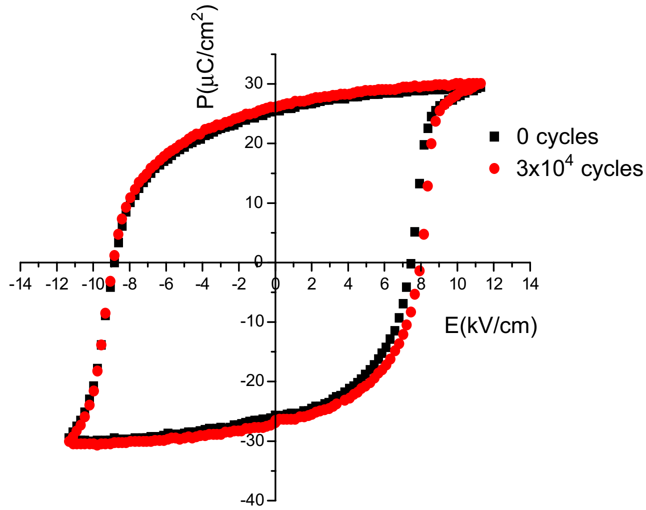

Fig. 4 Evolution of the hysteresis loops under a cyclic electric field with the amplitude E=0.11 Ec

Figure 3 gives the evolution of the hysteresis loops under a cyclic electric field with the amplitude E=1.1 Ec. After 50,000 cycles fatigue, the saturated polarization and remanent polarization decreased whereas the coercive electric field value almost had no change. Figure 4 demonstrates the hysteresis loops before and after the electric fatigue, and the intensity of the cyclic electric field subjected to the sample was 0.11 Ec. It is clear that the two curves almost superposed each other and no apparent decreases of the saturated polarization and remanent polarization were observed after 30,000 cycles fatigue, and the coercive electric field had no clear change. The normalized switchable polarizations vs. cumulative cycles under different electric field intensities are illustrated in Fig. 5. For the electric field intensity higher than Ec (E= 1.1 Ec), the switchable polarization decreased continuously, but for the electric field intensity much lower than Ec (E=

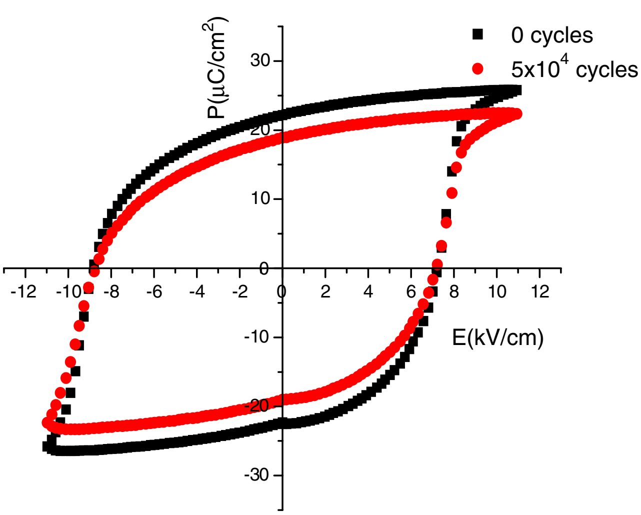

Fig. 3 Evolution of the hysteresis loops under a cyclic electric field with the amplitude E=1.1 Ec

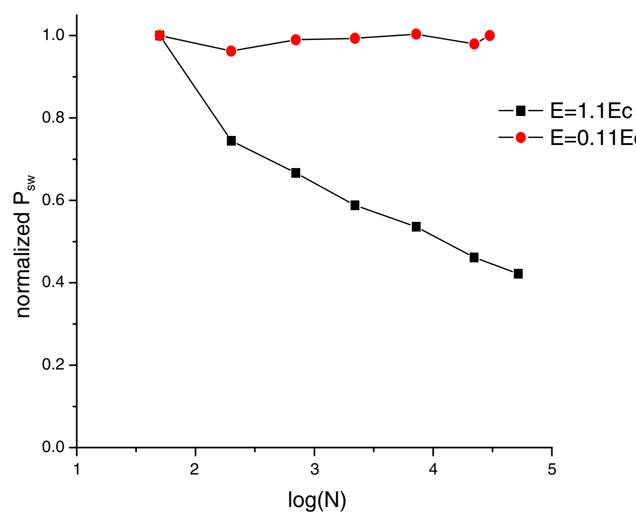

Fig. 5 Fatigue properties of the samples under different electric fields

0.11 Ec), the switchable polarization changed slightly during the whole fatigue process. It is suggested that high electric fields (>Ec) can induce relatively full switching of the ferroelectric domains thus leads to the declines of the ferroelectric properties, and low electric fields far from Ec can not provide full switching of the ferroelectric domains therefore have little effect on the ferroelectric properties of the ceramics though they may lead to the fatigue crack growth. It indicates that the crack growth has no apparent influence on the ferroelectric properties.

4 Conclusions

There exists a strong anisotropy of crack growth on the plane parallel to the polarization direction for polarized ferroelectric samples with a composition near MPB. In the orientation perpendicular to the polarization direction, the crack grows markedly and fast, whereas no obvious propagation is found in the orientation parallel to the polarization direction. High electric field intensity leads to both the degradation of the ferroelectric properties and the fatigue crack growth. Low electric field intensity far from Ec can also result in electric induced fatigue crack growth although it has little effect on the ferroelectric properties of the ceramics. Domain switching is considered to be the main mechanism for the fatigue crack growth. The crack growth has no apparent influence on the ferroelectric properties.

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