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Domain texture dependent fracture behavior in mechanically poled/depoled ferroelectric ceramics

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Abstract

The influence of domain texture on the fracture behavior was investigated in both the mechanically poled (MP) and the mechanically depoled (MD) PZT ceramics. Firstly, we fabricated axisymmetric domain textures in the MP and MD PZT samples subjected to a series of compressive stresses of up to 400 MPa. Then the Vickers indentation was employed to measure the fracture toughness parallel to ($K_{IC}^{\parallel}$) and perpendicular to ($K_{IC}^{\perp}$) the compression direction. Results show that with increasing compressive stress, $K_{IC}^{\perp}$ increases and $K_{IC}^{\parallel}$ decreases in both the MP and MD samples and $K_{IC}^{\perp}$ can reach 2.6–2.7 MPa m$^{1/2}$ at 400 MPa. Such fracture behavior is attributed to the ferroelastic domain switching toughening mechanism in ferroelectric ceramics, and the fracture toughness is proportional to the texture-dependent switchable strain near the crack surface. The obtained results may provide a helpful guidance for improving the reliability and performance of ferroelectric capacitors.

Keywords: Domain texture; Fracture toughness; Ferroelectric ceramics; Domain switching

1. Introduction

As the “smartest” materials, ferroelectric materials have been widely used in modern industries as capacitors, sensors, actuators, etc. due to their high permittivity, peculiar electromechanical properties, and compact size [1,2]. However, because ferroelectric ceramics are very brittle, the fracture problem during operation has severely limited their applications in high reliable devices [3,4]. In order to improve the reliability and extend the lifetime of ferroelectric devices, it is essential to investigate the fracture behavior of ferroelectric ceramics.

During the past decades, intensively experimental works [5–8] as well as theoretical [9,10] studies have been conducted to address the fracture behavior of ferroelectric ceramics. Although debates still exist for some problems [3,4], such as the influence of electric fields on the fracture toughness, it has been widely accepted that domain switching plays an important role in crack propagation [3–10]. Typically, there are two physical mechanisms leading to domain reorientation near the crack surface: one is the Lehovec effect [11], and the other is ferroelastic domain switching caused by the singular stress fields near the crack tip [5–10]. The Lehovec effect means that a very large depolarization field exists within a thin layer just beneath the surface of ferroelectric materials, tending to orient the polar axes of ferroelectric domains perpendicular to the crack surface [11]. As domain switching always dissipates energy, ferroelectric materials usually show fracture toughness enhancement during crack propagation, exhibited as R-curves [5–7]. Furthermore, as the amount of the switchable domains near the crack surface is texture dependent, ferroelectric materials will show fracture toughness anisotropy (FTA) in the pre-poled specimens [12].

The FTA phenomenon in ferroelectric ceramics was first discovered by Okazaki and co-workers in early 1980s [12]. When they used the indentation method to measure the fracture toughness of the unpoled and/or electrically poled $PbTiO_3$ and PLZT ceramics, they found that the crack lengths were the same in the unpoled samples but different in the poled samples, i.e., the crack parallel to the poling direction (hereafter referred as parallel crack) is shorter than that perpendicular to (hereafter referred as vertical crack) it. Anisotropic

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residual internal stress generated during electric poling was suggested to account for the observed FTA. Latter, Pisarenko et al. [13] systematically investigated the FTA in PZT and barium titanate ceramics by using double torsion and indentation methods. They thought the FTA was mainly caused by texture-dependent domain reorientation near the crack tip. In 1990, Mehta and Virkar [14] verified the domain switching toughening mechanism by measuring the domain textures beneath the crack surface using X-ray diffraction. In addition, they found FTA in the unpoled samples after mechanical poling (mechanical poling means that reorienting the domains in an unpoled sample by using a large uni-axial compressive stress. If the unpoled sample is replaced by a poled sample, then the process is called mechanical depoling). This phenomenon was attributed to the anisotropic domain textures after compression. Later the fracture behavior of electrically poled samples had been investigated by Guiu et al. [15], Calderon-Moreno and Popa [16], Fang and Yang [17], dos Santos e Lucato et al. [6], etc. using indentation, three point bending and compact tension methods. It is found that the fracture toughness measured by different methods can qualitatively confirm with each other. However, the FTA ratio measured by the indentation method is larger than that by other methods [16,17]. Recently, a large FTA ratio of 3.82 was found in the mechanically poled PZT samples by Li and Li [18], and they attributed this to that the domain textures in the mechanically poled samples are more saturated than that in the electrically poled samples. So far, studies on the fracture behavior of ferroelectric ceramics have been focused on the electrically poled samples; while the mechanically poled (MP) samples had little been concerned [14,18]. The MP ferroelectric ceramics, although cannot be used in piezoelectric applications, may have special applications in high reliable capacitor areas because of their large fracture toughness perpendicular to the loading direction, and should be intensively studied.

In this paper, the fracture behavior of the partial/complete mechanically poled/depoled Lead Titanate Ziconate (PZT) ceramics was systematically investigated using the Vickers indentation method. The longitudinal strains during mechanical poling/depoling were monitored by strain gauges and the domain textures were characterized by X-ray diffraction. The Vickers indentation results show that in both types of PZT samples, the fracture toughness perpendicular to the loading direction increases and that parallel to it decreases with increasing compressive stress. The domain texture dependent fracture toughness is thought to be caused by the ferroelastic domain switching toughening mechanism in ferroelectric ceramics.

2. Experimental procedure

2.1. Specimen preparation

The material used in this study is soft PZT ceramics, provided by Institute of Acoustics, Chinese Academy of Sciences. Its composition is near the morphtropic phase boundary where the Zr/Ti ratio is at 52/48. Its Curie temperature is at 315.2 °C. The material is cut into blocks with dimension of $6\times6\times12\ mm^3$. Silver paste is sintered onto the two opposite $6\times6\ mm^2$ surfaces of the blocks as electrodes. Electrical poling is conducted above the Curie point using a DC electric field of 500 V/mm along the 12 mm direction. The sample is then gradually cooled to room temperature with the electric field on. After poling, the piezoelectric constant d_{33} was measured to be 495 ± 8 pC/N.

2.2. Mechanical poling/depoling and texture measurement

The testing setup for the mechanical poling/depoling is shown in Fig. 1. The compressive stress is applied to the specimen by a screw-driven testing machine (Shimadzu, Japan), together with a spherical hinge to avoid any bias compression. Strain gauges are glued on the two opposite $6\times12\ mm^2$ surfaces to measure the variations of longitudinal strain during compression. Two pieces of copper wafer are pressed to the sample as electrodes and two alumina blocks are used as insulators to separate the testing machine from the high-voltage loading. The charges released during depolarization is collected by a capacitor of 10 μF and measured by a charge amplifier connected in series. During testing, the signals of stress, strain and the charges are input into an A/D data acquisition card and monitored by a computer.

Before testing, the edges of the specimen were polished to avoid stress concentration during testing. The specimen was then put on the center of the loading equipment and a preload of about 18 N was used to fix it. The strain signals of the two strain gauges are used to adjust the specimen to realize the uniaxial compression without bending. The compression loading rate is set to be 5 MPa/s and unloading starts as soon as the maximum compression reached, at a rate of about 10 MPa/s. For mechanical poling, the compressive stress levels of 50 MPa, 100 MPa, 200 MPa, and 400 MPa were used. As for mechanical depoling, a series of stresses of 50 MPa, 60 MPa, 70 MPa, 80 MPa, 100 MPa, 200 MPa, and 400 MPa were used to capture the point where the fracture toughness anisotropy switches.

After mechanical poling/depoling, X-ray diffraction was explored to detect the domain textures of the samples on the

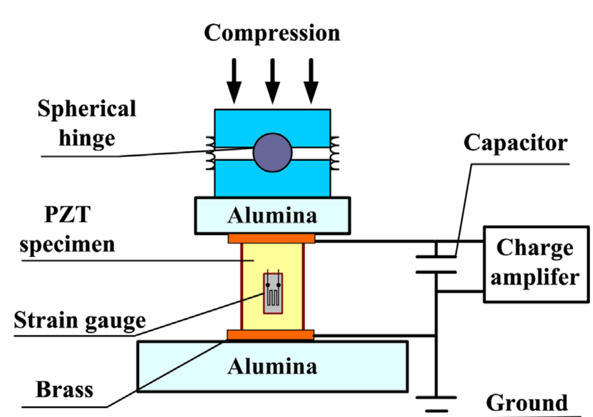

Fig. 1. Testing setup for the mechanical poling/depoling of ferroelectric ceramics.