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# Influence of domain orientation on the mechanical properties of lead zirconate titanate piezoelectric ceramics

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**Abstract**

The influence of domain orientation on the mechanical properties of lead zirconate titanate (PZT) piezoelectric ceramics has been investigated using un-poled and poled PZT ceramics. High mechanical properties, e.g., high elastic modulus and compressive strength, were obtained for the polarized PZT ceramics due to strain hardening caused by more severe domain switching during the loading process, while low mechanical properties for the un-poled ceramics. Fracture mechanics of the ceramics were influenced by the direction of the tetragonal lattice structure since cracks propagate along the long axis of the tetragonal structure (*c*-axis). Using X-ray diffraction and electron back scatter diffraction analysis, the domain switching characteristics could be clarified.


*Keywords:* PZT ceramic; PZT; Mechanical properties; Failure analysis; Fracture

## 1. Introduction

In recent years, piezoceramics have been widely studied and utilized in numerous applications, e.g., as displacement transducers, sensors and actuators.[1] In particular, lead zirconate titanate piezoelectric ceramics (PZT) are popular materials, as the PZT ceramics combine several beneficial properties, e.g., high piezoelectric coefficients, high Curie temperature and low sintering temperature.[2] As the PZT sensors and actuator need to become smaller and have higher power generation, the reliability and durability of piezoceramics in engineering application over long periods of use will be significant.[3] In addition, because PZT ceramics in smart structures are required to have high material performance, an examination of the material response to the application is very important. In these applications, the applied electrical and mechanical loadings induce failure and/or crack propagation in piezoelectric ceramics resulting in fracture. It is believed that crack initiation in PZT ceramics results from defects, grain boundaries and the boundary between the electrode and matrix.[4] It also appears that the crack growth rate is dependent on the grain size[5] and/or poling direction.[6] The reason for this is due to the change of crack growth resistance, caused by the domain switching and/or changes in the domain wall direction.[7] Many researchers have investigated the domain switching behavior using various techniques. With a light microscopy, the domain switching characteristic has become clear, and has been shown to strongly affect the crack growth rate and material properties.[8] Jones et al. have examined the influence of the fatigue frequency and loading level on domain switching behavior using X-ray diffraction. In their approach, the relative intensity of the (2 0 0) and (0 0 2) reflections in PZT ceramics was expressed by a domain switching fraction or a multiple of a random distribution (MRD).[9,10] The XRD approach was also used in the study by Hall et al., where a high-energy synchrotron XRD system was used to examine domain switching behavior in bulk PZT ceramics.[11] In the study by Shirakihara, the amount of domain switching was evaluated by the change of the intensity ratio, $I_{222}/I_{22\bar{2}}$, of the (2 2 2) to (2 2 $\bar{2}$) diffraction. The intensity ratio for normal diffraction, i.e., $\psi = 0°$, was reduced with increase of the applied strain, since the spontaneous polarization direction, the (2 2 2) direction, turned to the loading directio.[12] A unique approach was described by Liu et al. They investigated the domain switching behavior of ferroelectric ceramics using a moiré interferometry technique, where in-plane 90° domain

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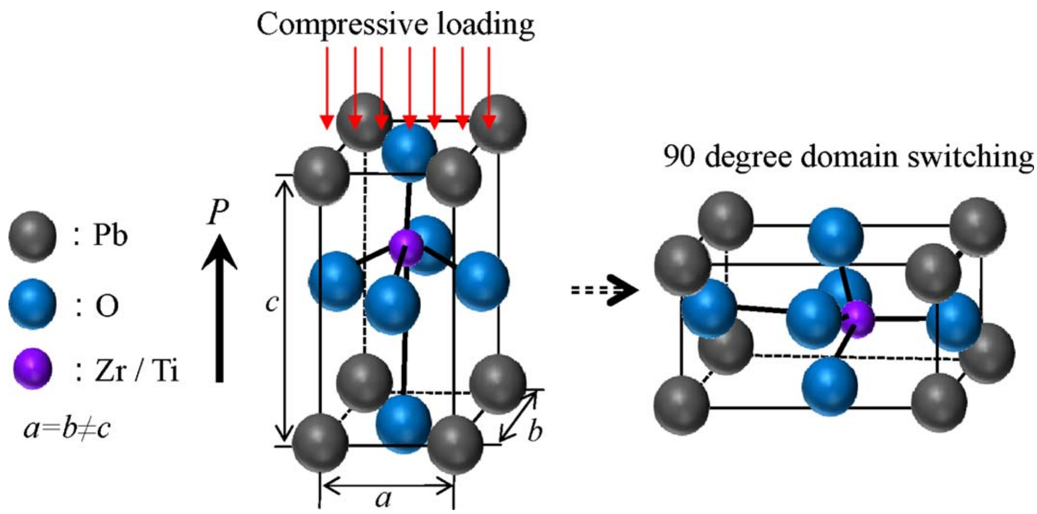


Fig. 1. Domain switching models (90°) by compressive stress on micro-scale.

switching occurs in the region of approximately a 45° band, reducing the fracture toughness and material brittleness in PZT ceramics.[13] Furthermore, several researchers have employed electron back scatter diffraction (EBSD) analysis to examine the domain switching behavior of PZT ceramics.[7,14] With EBSD analysis, the domain orientation can be clearly observed with a color mapping image. From these studies, it has been shown that domain orientation is one of the significant factors which determine the material properties of PZT ceramics. In recent years, a new technique has been proposed for revealing domain switching, where the electric potential is transformed to yield the radial stress and strain distribution in front of the crack tip.[15] To understand the domain switching behavior, several switching models have been proposed. Fig. 1 shows the fundamental tetragonal

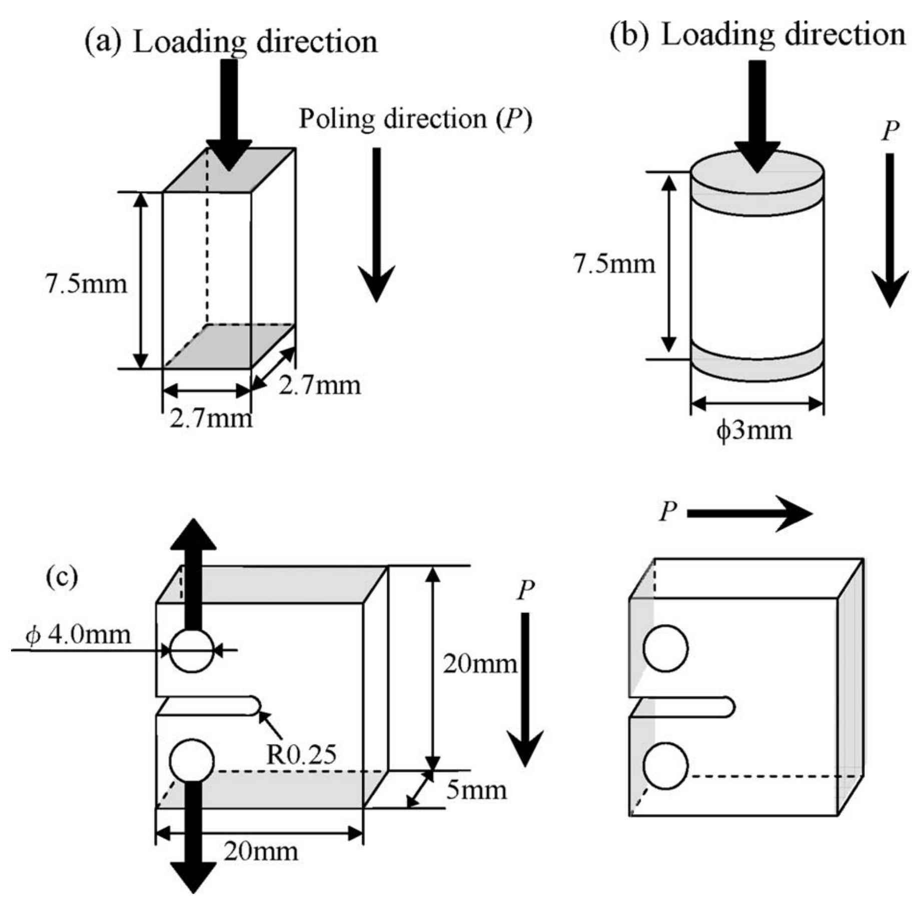


Fig. 2. Schematic illustration of specimen samples (a) rectangular rod; (b) round rod; and (c) compact tension specimen.

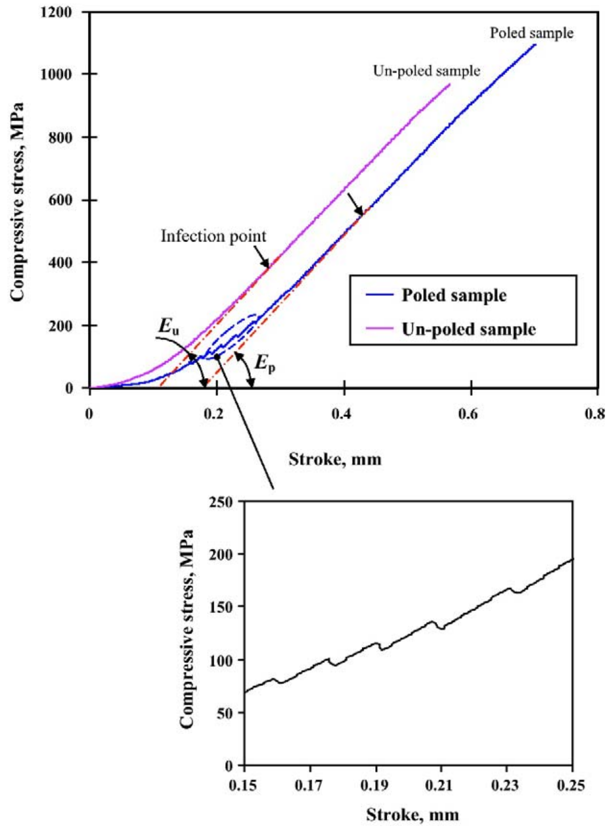


Fig. 3. Relationship between the compressive stress and stroke of rectangular-rod PZT ceramics.

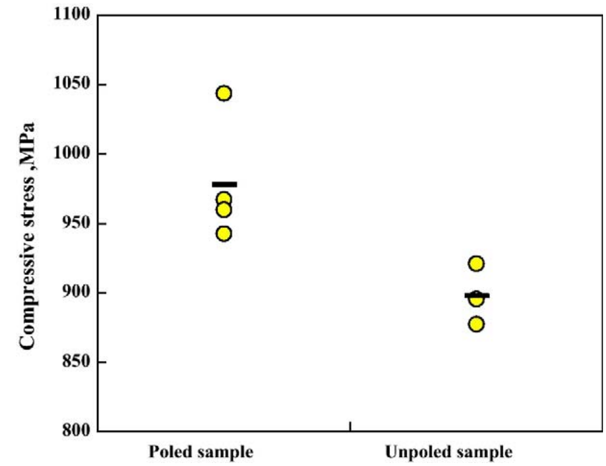


Fig. 4. Compression strength for the un-poled and poled samples.

PZT domain switching models (90°). As can be seen, there is a $Pb^{2+}$ ion at each corner of the unit cell, a $Ti^{4+}$ (or $Zr^{4+}$) ion at the center of the unit cell and $O^{2-}$ ion at the center of each face. With sufficiently large compressive stress squeezing the *c*-axis and hence the unit cell towards the cubic state, the central ion has to move 90° to one of the side sites, changing the direction of polarization so that it is typically aligned almost parallel or perpendicular to the stress.[16,17] Lupascu has proposed a simple fatigue model for perovskite ferroelectics based upon an electric field driven point defect drift, where the case of the oxygen vacancy as an ionic defect is considered.[18] In the study by Li and Fang, a new criterion of domain switching for ferroelectric polycrystals is presented, where a three-dimensional finite element model is developed to simulate domain switching. It is assumed in the model that each crystallite, represented by a cubic element, is a single domain. A domain will undergo 90° and 180° switching when reduction of the Gibbs free energy in a representative element body exceeds the corresponding energy barrier.[19] In recent years, Senousy et al. have further proposed a domain switching model for PZT ceramics. This model considered the changes in potential energy, and accounts for the temperature influence on domain switching.[20]

Although several domain characteristics and domain models have been investigated by a number of researchers, there are no clear reports describing the effects of domain orientation (or domain switching) on the failure and material properties of PZT ceramics. The investigation of these properties is especially important for the design of ceramics for engineering

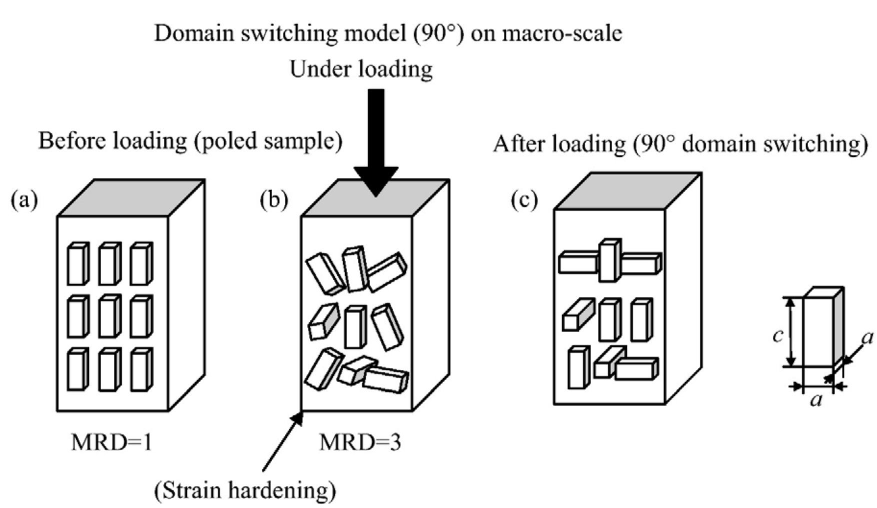


Fig. 5. Schematic illustration showing domain switching behavior.

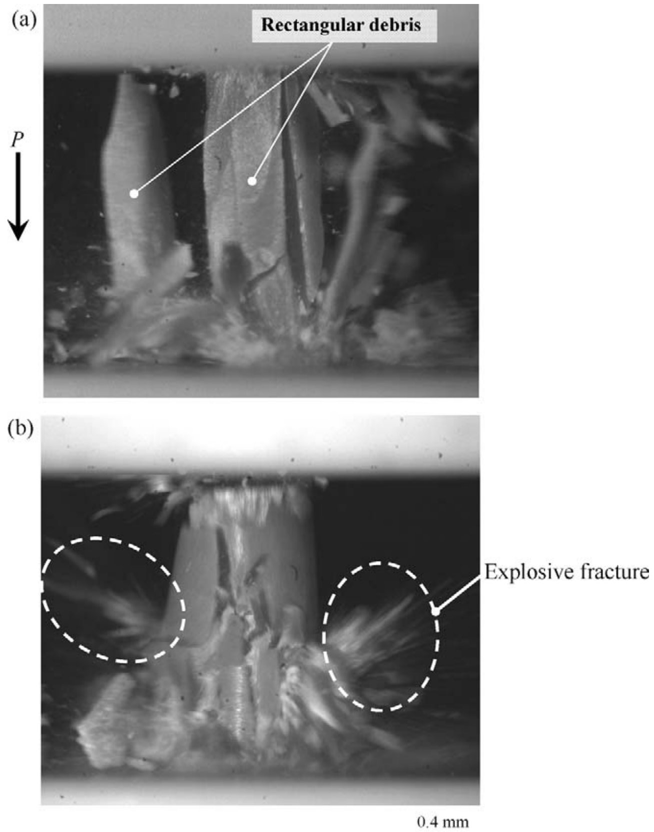


Fig. 6. High speed camera images of the round-bar PZT ceramics just fractured by the compressive loading: (a) poled sample and (b) un-poled sample.

applications. The aim of this work was, therefore, to investigate the influence of the domain structure on the mechanical properties and fracture mechanics of PZT ceramics.

## 2. Experimental procedures

### 2.1. Materials

The piezoceramic selected in the present study was a commercial soft lead zirconate titanate ceramic, $PbZrTiO_3$ (PZT), produced by Fuji Ceramics Co., Japan. The PZT lattice, as examined by X-ray diffraction (XRD) at room temperature, adopts a tetragonal perovskite structure with $a = b = 0.4046$ nm and $c = 0.4103$ nm. Three different PZT specimens with and without poling were used: a rectangular rod (2.7 mm × 2.7 mm × 7.5 mm), a round rod ($\phi 3 \times 7.5\ \text{mm}^3$) and a compact tension specimen (20 mm × 20 mm × 5 mm) as shown in Fig. 2. The average grain size of the PZT ceramic is approximately 5 μm in diameter. For the polarized samples, the silver electroplating was printed on the specimen by firing for several hours in atmosphere at 973 K. After the electroplating process, the polarization was established by applying an electric field 2 kV/mm between two electrodes in silicon oil. The material properties of the PZT ceramics after polarization, as measured by an impedance analyzer (Agilent Technologies, 4294A), were an electromechanical coupling coefficient $k_{33} = 0.76$ and a dielectric constant $\varepsilon_{33}/\varepsilon_0 = 2260$.

### 2.2. Experiments

#### 2.2.1. Compression test

Compression tests were carried out on PZT ceramics using rectangular rod specimens. For these experiments, a screw driven type universal testing machine with 10 kN capacity (Shimadzu EZ graph) was employed. The loading speed for the compression tests was 1 mm/min to failure. The specimens were loaded via insulating ceramic blocks, i.e., open circuit. The stress–stroke relationships were measured by a conventional load cell. During the compression tests, the stress was monitored by a data acquisition system in conjunction with a computer though the load cell. In this test, the mechanical loads were applied along the long axis of the specimen (see Fig. 2).

#### 2.2.2. Fracture characteristic

To examine the effect of domain orientation on the fracture mechanism, the fracture characteristic in the compression test was directly observed using a high speed camera (Photron FASTCAM-SA1.1). In this test, cylindrical rods were used (Fig. 2(c)). The pictures were taken with an image resolution 1024 × 1024 pixels at a frame rate $675 \times 10^3$ Hz. In addition, the interference of domain orientation on the fracture characteristic was investigated, in which crack growth characteristics were examined using the compact tension test specimens with and without poling, as shown in Fig. 2(c).

#### 2.2.3. Domain switching characteristics

Domain switching characteristics were investigated by X-ray diffraction and electron back scatter diffraction (EBSD) analysis. In the X-ray analysis, an X'Pert Pro system (Panalytical Inc.) with a Cu tube source ($\lambda = 0.154$ nm) was utilized. The XRD analysis was done using the rectangular rod specimens after being loaded to various compressive stresses. For the EBSD measurements, an orientation imaging microscopy (OIM) system was employed. In this approach, the rectangular specimens were prepared by first polishing to a mirror finish using colloidal silica after which the surfaces for observation were etched using a solution of 5 ml hydrochloric acid, 2 ml hydrogen fluoride and 250 ml water before coating with carbon on the polished surface. After the sample preparation, EBSD analysis was carried out in the same area of the specimen before and after compressive loading to 50 MPa and 100 MPa.

## 3. Results and discussion

### 3.1. Mechanical properties

Fig. 3 shows a representative compressive stress vs. stroke curve obtained from the un-poled and poled rectangular specimens. The stress–stroke relationships in Fig. 3 can be divided mainly into two major stages on the basis on an inflection point as indicated by the arrows, namely a non-linear region of stress vs. stroke in the early stages of compression and a linear relationship obtained in the later stage, until the final fracture. It is clear from the stress vs. stroke curves that the slopes of the stress vs. stroke in the early stage of loading process are

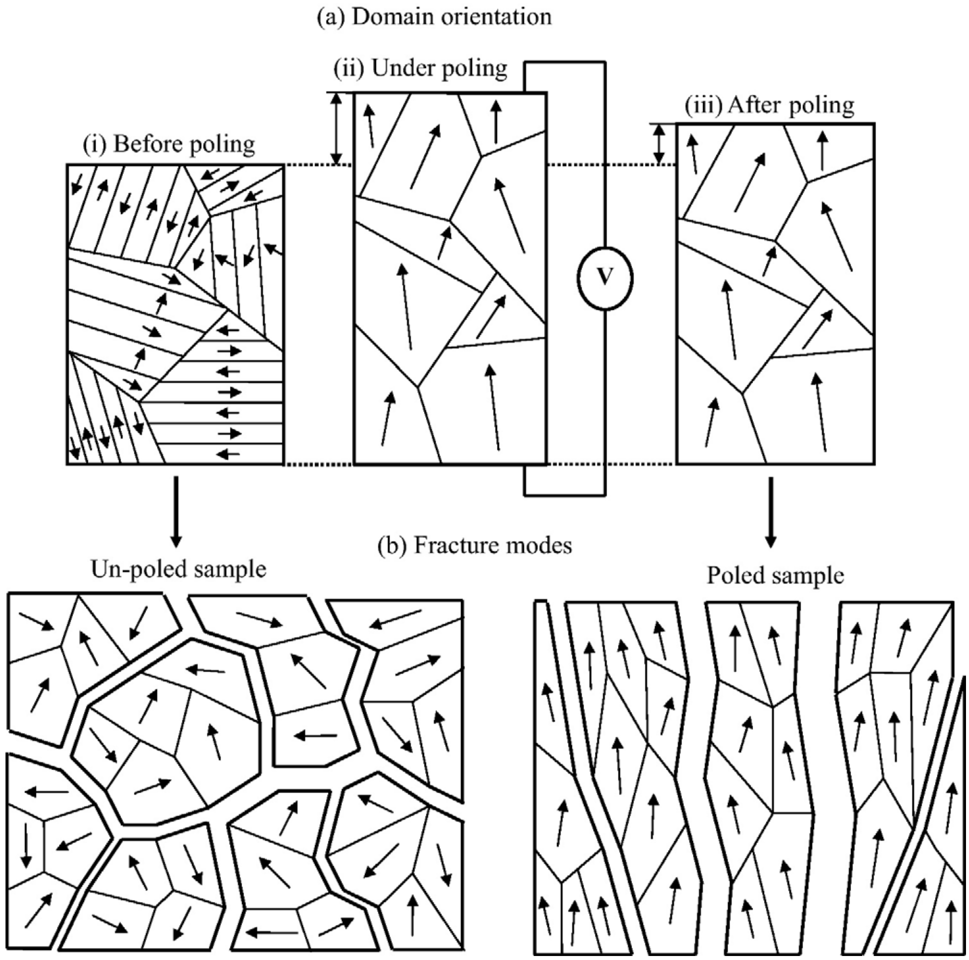


Fig. 7. Schematic illustration of the grains of the PZT ceramics with and without poling: (a) domain orientation and (b) fracture mode corresponding to Fig. 6.

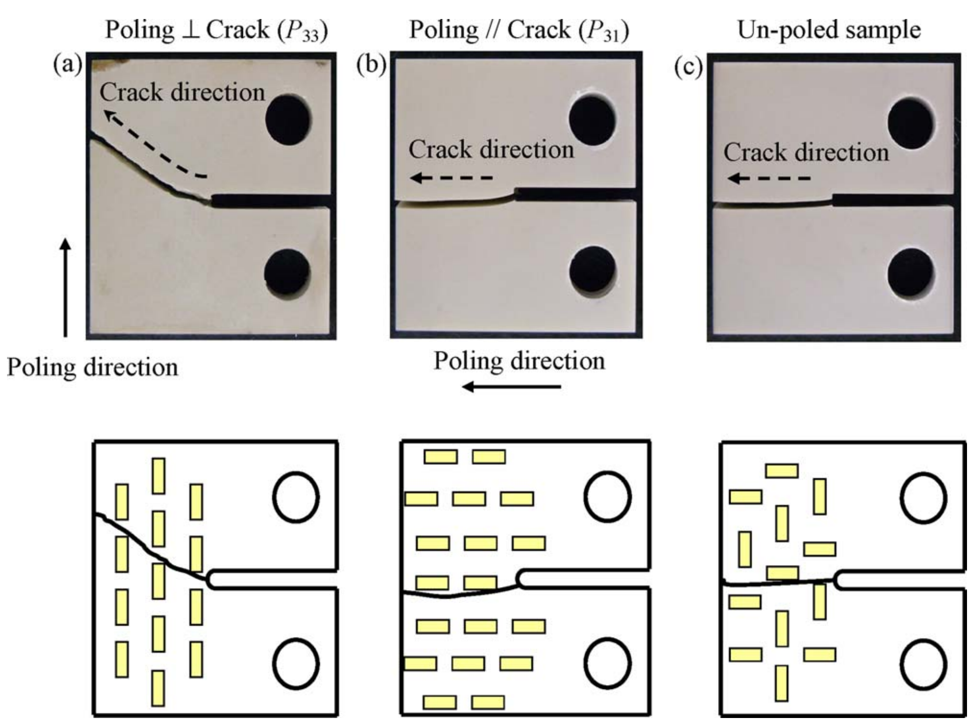


Fig. 8. Compact tension specimens showing crack paths: (a) and (b) poled samples, (c) un-poled sample.

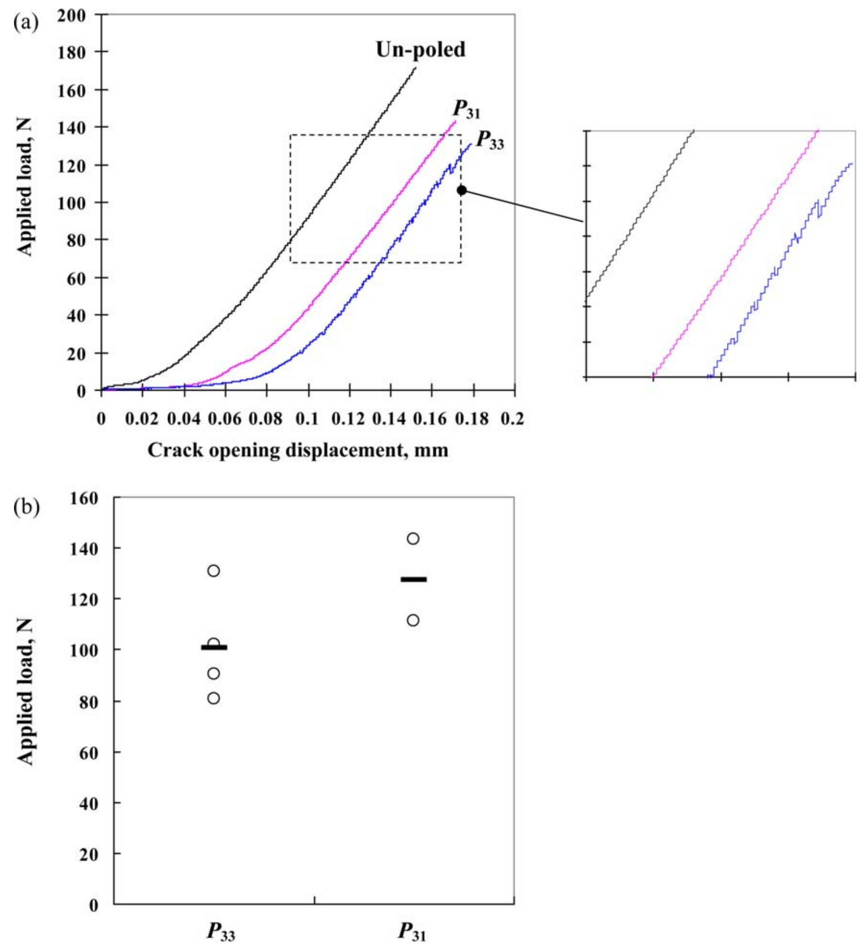


Fig. 9. (a) Relationship between the applied load and crack opening displacement of compact tension PZT ceramics with and without poling processes. (b) Fracture strength of the compact tension specimens.

different; the slope for the un-poled ceramic is slightly higher than that of the poled one. In this case, the change of slope may be attributed to the severity of domain switching, i.e., the lower the slope, the stronger the switching due to accelerated strain. In the latter relationship, a linear applied stress–stroke behavior can be seen, which is mainly related to elastic deformation (or elastic constant). In this case, the elastic constant for both samples is obtained, $E_u = 2120.1$ MPa/mm (for un-poled sample) and $E_p = 2090.6$ MPa/mm (for poled sample).

It is of particular interest to note that the zigzag stress–stroke relationship can be seen clearly only in the poled sample, as outlined by the dashed circle. As severe domain switching occurs for the polarized sample compared to un-poled one, such a zigzag occurrence might be caused by the domain switching.[21] The reason for the zigzag relationships obtained will be further discussed in a later section.

Fig. 4 represents the compressive strength of the rectangular-rod specimens obtained from the stress–stroke curves. As can be seen, the overall compressive strength for the polarized samples is approximately 10% higher, on average, than the un-poled samples. The reason for the high strength of the polarized sample may be related to the material strain due to domain switching,[7] i.e., strain (or work) hardening.

Fig. 5 gives a schematic illustration showing the domain orientation of PZT ceramics: (a) before loading; (b) under loading and (c) after loading. Note that, in this model, only 90° domain switching is considered (Fig. 5(c)), since this type of switching was detected in the related ceramics.[7] From the model, the domain orientation in each grain is altered during the loading process; the poled domains before loading become randomly oriented before 90° domain switching, Fig. 5(b). This occurrence leads to the work hardening phenomenon. Thus, a high material strength can be obtained in the poled samples. In addition, such domain switching might affect the non-linear stress vs. stroke curves, which is shown in Fig. 3. This is because the longitudinal ($c$-axis) tetragonal structure parallel to the loading direction is tilted to 90° (perpendicular to the loading direction), which leads to an acceleration of the macroscopic strain attained during mechanical loading.[10] Such domain switching can also be understood from the micro-scale model shown in Fig. 1.

To understand the effect of domain orientation on the fracture mechanics, direct observation of the fracture behavior was conducted using the high speed camera. Fig. 6 presents images of the poled and un-poled cylindrical rod ceramic samples just after fracture. Note that this experiment was repeated three times to verify the fracture patterns. As seen in Fig. 6(a) the polarized

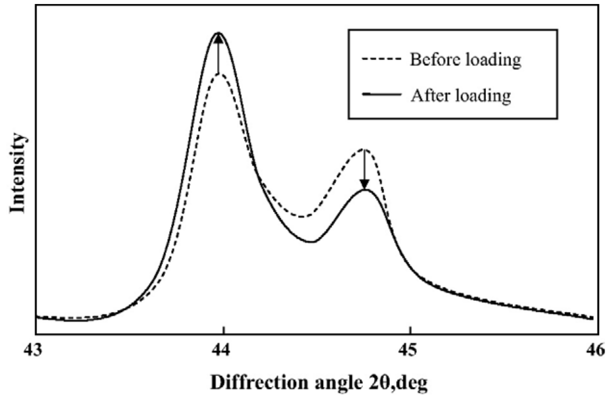


Fig. 10. X-ray diffraction patterns for the poled PZT ceramic.

ceramics were fractured with the formation of many cracks propagating along the direction parallel to the poling direction. Thus, several large rectangular areas of fracture debris were obtained. In contrast, for the un-poled specimen, the sample was fractured completely with little debris arising from the crack propagation in other directions. Moreover, some explosive fractures occurred as denoted by the dashed circles in Fig. 6(b). From Fig. 6, the crack growth direction can be attributed to the domain orientation, i.e., along the *c*-axis, and this can be interpreted using the structural domain models for PZT ceramics. Fig. 7(a) shows the models for the domain orientation of a PZT ceramic before, under and after the poling process,[22] and Fig. 7(b) displays the fracture modes for the un- and poled samples. Because the cracks in the ceramic, observed by the high speed camera, seem to propagate along the domain direction, different fracture characteristics of both un- and poled samples were obtained, as illustrated in Fig. 7(b).

### 3.2. Crack growth characteristics

In order to verify the effect of domain structure on the crack growth characteristics, a crack growth test was carried out using compact tension (CT) specimens. In this approach, two different poled ($P_{33}$: poling direction ⊥ crack path; $P_{31}$: poling direction ∥ crack path) and un-poled samples were employed. Note that each examination was conducted more than three times to confirm the result. Fig. 8 displays representative fractured CT specimens. In this approach, the cracks propagated instantly to final failure for all samples due to their brittleness. Westram et al. have investigated crack growth characteristics in PZT ceramics under electrical cyclic loading, where 0.14 mm/cycle crack growth was obtained at 1.7 MV/m.[23] As seen in the $P_{33}$ polarized sample, Fig. 8(a), the crack growth direction is curved shortly after the crack has been initiated, the crack seeming to turn parallel to the poling direction. Such a crack curvature would be influenced by the domain orientation. In contrast, straight crack paths are obtained for the un-poled and $P_{31}$ poled samples, Fig. 8(b) and (c). Similar crack path characteristics were observed in the study by Dos Santos e Lucato et al. They examined the crack growth characteristics using piezoelectric ceramics with and without poling and showed that two possible crack paths, straight and deflected, were obtained in the poled specimens due to the different poling directions. The crack grows toward to the poling direction.[24] On the basis of the above information, it is considered that the crack growth directions can be attributed to the domain orientation. This can be explained using the schematic illustration shown in Fig. 8 and provides convincing evidence that the crack growth direction is affected by the domain structure (or poling direction). Fig. 9(a) shows the applied load vs. crack opening displacement (COD) curves for the three CT specimens. As can be seen, the slopes of the load vs. COD in the early stage of loading are different; the slope for the un-poled ceramic is higher than that for the poled ones. Furthermore, the slope for the polarized sample $P_{33}$ is low compared to that for the other poled sample, $P_{31}$. Interestingly, the zigzag load–displacement relationship can be seen for the $P_{33}$ specimen, while these steps cannot be detected for the others. This occurrence is similarly observed in the compressive test in Fig. 3. The zigzag relationship is caused by significant domain switching occurring when the sample is loaded perpendicular to the poling direction.[16,21] It should be pointed out that the extent of the zigzag relationship for the $P_{33}$ CT specimen is apparently weak compared to that for the poled rectangular specimen shown in Fig. 3. This can be attributed to different severity of domain switching, e.g., weak domain switching for the CT specimen because of the switching in a limited area due to stress concentration when loaded.

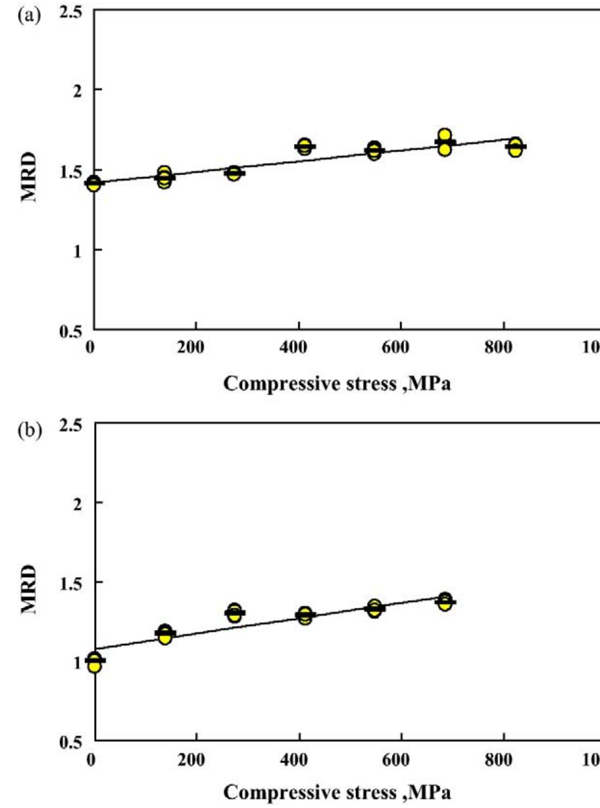


Fig. 11. Variation of MRD values as a function of the compressive stress for (a) poled sample and (b) un-poled sample.

Fig. 9(b) shows the fracture strength of the CT specimens. As shown, a higher average strength of about 170 N, was recorded for the un-poled sample compared to the poled ones, and the strength for the $P_{33}$ polarized sample was about 100 N lower

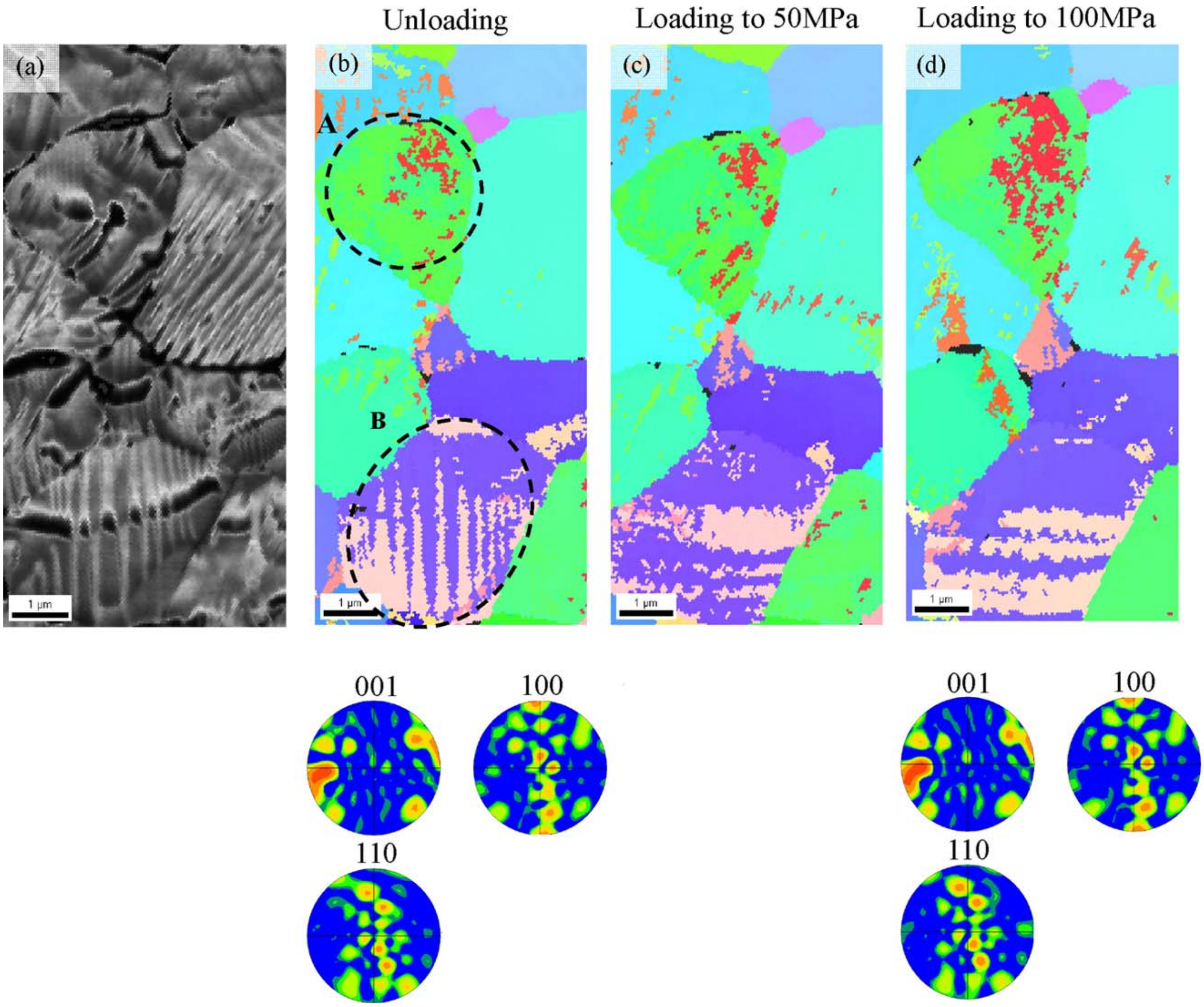


Fig. 12. EBSD analysis of PZT ceramics after etching: (a) SEM image and (b)–(d) crystal orientation maps before and after loading.

than $P_{31}$ (about 130 N). In our previous work, the fracture toughness ($K_{IC}$) of the same PZT was investigated in the $P_{33}$ and $P_{31}$ directions where the crack growth occurs from the edge of the Vickers indentations and the fracture toughness for $K_{IC,33}$ and $K_{IC,31}$ are 0.102 MPa$\sqrt{m}$ and 0.237 MPa$\sqrt{m}$, respectively. This trend is consistent with our fracture strength for the $P_{33}$ and $P_{31}$ samples, shown in Fig. 9(b).

### 3.3. Domain structures

The domain switching behavior during the loading process was investigated by X-ray and EBSD analysis. Because the tetragonal long axis, the *c*-axis, can occur parallel to any of the three ⟨0 0 1⟩ directions, diffraction can be used to measure the proportion of *c*- and *a*-oriented domains parallel to a given sample direction. The reorientation of a domain by 90° domain switching from the *c*- and *a*-direction can be revealed by a change of the diffraction intensity ratio. Several researchers have examined the domain switching characteristics in PZT ceramics using neutron diffraction. In the study by Pojprapai et al., a change in the {2 0 0} reflections provided quantitative measurements of domain switching behavior, domain texture and the strain resulting from domain switching.[10] In their excellent works, the domain switching behavior and domain texture are quantitatively analyzed using the unit multiple of a random distribution (MRD). In this case, the MRD is equal to 1 when a random domain orientation is obtained. On the other hand, the MRD becomes 3 as *c*-domains are all oriented parallel to the poling direction. Their domain orientations are related to the schematic illustration shown in Fig. 5(a) and Fig. 5(b). In the present work, an investigation of the MRD was executed using X-ray diffraction. In this approach, the (0 0 2) and (2 0 0) peaks are related to the angle around $2\theta = 45°$. Fig. 10 displays the representative X-ray diffraction patterns obtained from the rectangular rod samples. The (0 0 2) peak decreases and the (2 0 0) peak increases with increase of the compressive load level. When both peak levels change, the MRD value can be expressed by the following formula:[10]

$$\mathrm{MRD} = \frac{3(I_{002}/I_{002}^{R})}{(I_{002}/I_{002}^{R}) + 2 \times (I_{200}/I_{200}^{R})} \tag{1}$$

where $I_{002}$ and $I_{200}$ are the integrated intensities of the (0 0 2) and (2 0 0) peaks after mechanical loading.[10] Fig. 11 displays the measured MRD variation as a function of the applied compressive load level. For the polarized sample, the MRD level increases with increase of the applied load from about 1.4 to 1.7. On the other hand, the MRD value for the un-poled sample is almost 1.0 without any applied load, and increases with increase of the load level to about 1.4. The trends of MRD variations for un-poled and poled samples are similar to the related experimental results obtained by neutron diffraction.[10]

To understand domain switching characteristic in detail, the crystal structure orientation in the PZT ceramics was further examined using EBSD analysis. In this case, the EBSD was measured on the surface of the rectangular specimen after a compressive load was applied to 50 MPa and 100 MPa. Fig. 12(a) displays the SEM image of the microstructure of the PZT ceramic and Fig. 12(b)–(d) presents the crystal orientation maps before and after the loading process. The color level of each pixel in the crystal orientation map is defined according to the deviation of the measured orientation with respect to the ND direction.[7] As seen in the SEM micrograph (Fig. 12(a)), a rough sample face, showing many grooves, can be observed, which is related to the domain boundary or domain wall. It is clear from the crystal orientation map that the domain direction is different depending on the grain (see Fig. 12(b)). With a compressive load of 50 MPa, the domain direction in some grains changes due to domain switching. The amount of domain switching increases with increasing applied load to 100 MPa. Note the range of compressive load between 50 MPa and 100 MPa is related to the range of MRD of about 0.02 as seen Fig. 11. This result reveals that even a small difference in MRD relates to severe domain switching. It is also clear from the EBSD measurements that a different pattern of 90° domain switching can be obtained, e.g., in grains A and B as enclosed by the dashed circles. In grain A, the domain orientation is tilted about 90° after the loading process, which is similarly observed in Ref. [7]. In contrast, the groove formation in grain B is rotated by about 90°. Such domain switching characteristics may be affected by variations in stress level, but this will be discussed in future work.

## 4. Conclusions

The effects of domain switching on the mechanical properties of lead zirconate titanate (PZT) piezoelectric ceramics were examined using PZT with and without poling. Based upon the results obtained, the following conclusions can be drawn.

(1) The mechanical properties of PZT ceramic are attributed to domain switching. Because of domain switching, good mechanical properties were obtained for the polarized PZT ceramics, where a work hardening-like phenomenon occurred.
(2) Different fracture patterns were influenced by domain orientation in PZT ceramics. The poled PZT ceramics fractured with many cracks propagating along the direction parallel to the loading direction. On the other hand, un-poled PZT ceramics fractured completely with little debris. The crack growth direction is severely affected by the poling direction because of the different domain orientation.
(3) With both X-ray diffraction and electron back scatter diffraction analysis, various types of 90° domain switching were clearly revealed. In addition, the amount of domain switching increases linearly with increasing applied load.

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