

# The control of mechanical strength by an electric field in ceramic composites dispersed with piezoelectric particles

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**Abstract** The control of mechanical strength in structural ceramic composite has been proposed and demonstrated by dispersing piezoelectric particles. The change in strength of barium titanate/zirconia composite on polarization treatment was evaluated, and the mechanism of this phenomenon is discussed based on the previously proposed mechanism for monolithic piezoelectric ceramics. The bending strength of BT/8YSZ composite increased upon longitudinal poling and decreased upon transverse poling compared to the untreated samples. Both the strengthened and weakened specimens tended to return almost to their original strength by heating over the  $T_c$  of barium titanate. Scanning electron microscopy was then used to examine the crack propagation after the polarization treatment, and cracks going in the poling direction were found to detour around the barium titanate grains, which was attributable to the increase in strength in the poling direction.

**Keywords** Ceramics · Barium titanate · Zirconia · Poling · Strength

## 1 Introduction

Ceramics have several advantages, for example, the ability to maintain their function even under severe conditions such as high temperatures or highly corrosive environments, exhibiting excellent resistance to wear. These advantages, however, prevent them from being easily

recycled, which is a major disadvantage from the resource conservation point of view.

To break down and recycle ceramics, ways to degrade their strength must be established. A method to recycle zirconia ceramics has been recently proposed. By heating zirconia at 200°C to 500°C, a phase transformation from tetragonal to monoclinic, accompanied by specific volume changes, results, thus reducing its strength [1, 2]. This shows that the strength can be controlled by heat treatment, but this method is only applicable to zirconia.

Ceramics are often strengthened by dispersing another material within itself to form composites [3]. The resulting particle-dispersed composite sometimes exhibits nonlinear effects such as crack deflection and residual internal stresses, since the internal compressive stress is a result of the difference in the thermal expansion coefficients between the components. If the internal residual stress is the main factor that causes the strengthening, the final strength is determined by the difference in the thermal expansion coefficients between the matrix and the dispersoid. Once fabricated, modifying the strength of these composites is difficult.

Therefore, to facilitate recycling, even though the materials have to maintain their improved mechanical properties when in use, reducing their strength for disposal is necessary. When using piezoelectric ceramics as a structural component, its mechanical strength can be controlled by an electric field. However, typical piezoelectric ceramics have small mechanical strengths that render them unsuitable for structural components. We previously reported that the strength of dielectric material (8 mol% yttria-stabilized zirconia, 8YSZ) dispersed with piezoelectric particles ( $\text{BaTiO}_3$ ) can be controlled by polarization treatment [4–6], and this idea has been used in other studies [7, 8]. The cause of the change in strength of this system is

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thought to be the generation of internal stresses at the interface between the matrix and dispersoid, but specific details of the mechanisms involved are still unknown.

Since a piezoelectric material is able to show relatively large strains under an electric field, large internal stresses would be generated in a piezoelectric ceramic with a large elastic modulus. Various attempts have been made to improve the mechanical strength of piezoelectric ceramics, and various strengthening methods that maintain the piezoelectric properties have been employed [9–12]. Understanding the fatigue properties is especially important to prevent fatigue degradation because cyclic stresses are generated when an electric field is applied [13–16].

Apart from these mechanical properties, the stress response of crack propagation in monolithic piezoelectric ceramics is known to be influenced by the applied electric field [17, 18], which has not been reported in most ceramics that show marginal electrostrictive strain. Numerous phenomena concerning the differences of mechanical strength or fracture toughness in piezoelectric ceramics, with and without polarization treatment [19–27] or horizontal or vertical to the poling direction [28–31], have been reported.

If the change in strength is caused by the internal stresses resulting from the residual strain, any change in strength would be recovered by relaxing the internal stresses. However, the strength degradation by transverse poling can also be ascribed to a crack that has been generated, which means the strength would not be recovered upon post-annealing.

In this review paper, a mechanism of the electric control of strength is proposed based on the release of the residual stress caused by the polarization treatment and on the observation of anisotropic crack propagation in connection with the poling direction. We consider the explanations given in the reports (described previously) concerning the electric field response of mechanical strength in monolithic piezoelectric ceramics to explain the strength change in the piezoelectric particle-dispersed ceramic composite.

## 2 Experimental procedure

The experimental procedures to produce our strength-controllable composites are summarized as follows [4–6]. Commercial yttria fully stabilized zirconia, 8YSZ (Toso Co., Ltd., Japan TZ-8Y and barium titanate, BT (Central Glass Co., Ltd., Japan) powders were used as the starting raw materials. Powder mixtures of 8YSZ and BT in predetermined ratios ( $BT/(BT + 8YSZ) = 5$  and 10 mol%) were mixed by ball-milling with ethanol for 2 h, and calcined at 700°C for 1 h. After calcination, the resulting powder mixture of BT/8YSZ was sieved using a 75  $\mu\text{m}$  mesh. They were then pressed into rectangular bars with

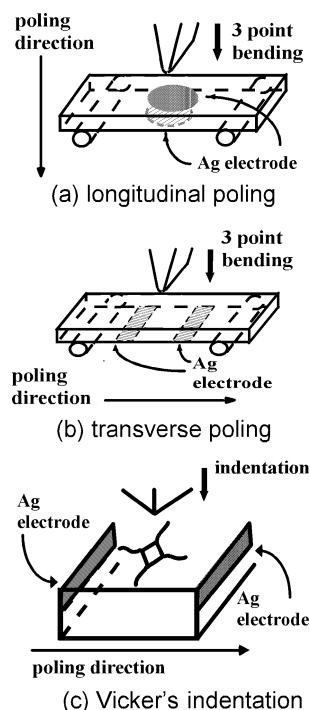
dimensions of  $5 \times 8 \times 15 \text{ mm}^3$  below 100 MPa and subjected to cold isostatic pressure (CIP) at 140 MPa. The pressed powder compacts were sintered at 1400°C for 4 h in an alumina boat in air. The sintered specimens obtained had relative densities over 95%. XRD analysis of the specimens confirmed that they were composites consisting of barium titanate and zirconia.

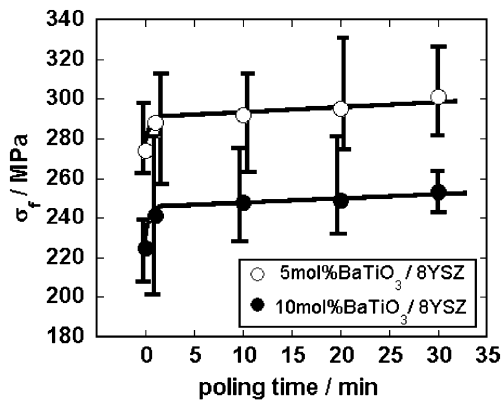
From the larger sintered BT/8YSZ specimens, thin plates were cut with a 2.5 mm width and 0.5 mm thickness, so that they could be subjected to the three-point bending test to measure their strength. To examine the effect of the poling time and field strength on the bending strength, the specimens were prepared as follows. Silver electrode was vapor-deposited on both sides of the specimens that were to be treated with poling before the bending test. Twenty samples were used. The polarization treatments were performed in silicone oil at 80°C. The schematic drawing of the setup is shown in Fig. 1(a).

First, to examine the mechanism of strengthening by poling, poling time and applied electric field intensity were varied, then the applied electric field and poling time were fixed as 60 kV/cm for 30 min. The untreated samples were also held in silicone oil for comparison.

Moreover, the specimens after poling were heated at 200°C, which is over the  $T_c$  ( $\sim 120^\circ\text{C}$ ) of barium titanate transforming from ferroelectrics to paraelectrics, because the residual polarization, namely, the deformation of barium titanate after poling, is expected to be released by heat treatment over  $T_c$ . The change in strength due to the polarization treatment was then evaluated by comparing the

**Fig. 1** Schematic view of sample setup in three point bending test and Vicker's indentation test [4–6]





**Fig. 2** Poling-time dependence of bending strength in BT/8YSZ. The applied electric field is 80 kV/cm [5]

bending strength of ten poling-treated samples with the results from ten untreated samples.

The transverse poling test was applied to other BT/8YSZ samples. Parallel silver electrodes were evaporated at the tension side as shown in Fig. 1(b). Following this, the tests were conducted in the same way as after the longitudinal poling, in which the bending strengths of unpoled samples, poled samples, and those that were heat-treated after poling were compared.

To investigate the effect of poling on the crack propagation direction, Vickers' indentation was performed after poling and the cracks generated parallel or perpendicular to the direction of poling were noted. The relationship between the direction of crack propagation and the poling direction is shown in Fig. 1(c). The crack length was measured under an optical microscope, and the behavior of crack propagation was observed under scanning electron microscopy (SEM).

### 3 Results and discussion

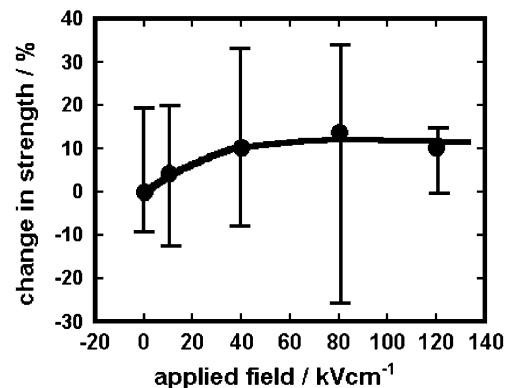
We previously reported that the obtained sintered specimens have relative densities of about 95% and that they are composites consisting of two phases, zirconia and barium titanate, according to the XRD analysis [4]. Moreover, these specimens show a ferroelectric property confirmed by D–E hysteresis. The results suggested that the ferroelectricity is derived from the tetragonal barium titanate phase in this composite. Therefore, the applied electric field would be expected to cause a distortion in barium titanate accompanied by the generation of internal stress.

We first measured the effect of the poling time on the change in strength for the two types of composite samples with 5 or 10 mol% of barium titanate. The results for ten specimens are shown in Fig. 2, where the minimum and maximum values are shown for each measurement point as error bars. Within a few minutes of poling, the mechanical

strength of both types of composites rose abruptly compared to the untreated samples. After this sudden increase in strength, the rate of the strength increase slowed down drastically. Furthermore, as the poling time increased, the bottom of the error bars gradually rose and the distribution width of the strength tended to decrease [5].

Such a sudden increase in strength within the first few minutes of poling is believed to be due to the fast ferroelectric domain switching in barium titanate within the composite, since the dipoles in the barium titanate crystal would be immediately aligned with the poling direction upon polarization treatment. Internal stresses in the poling direction would also be generated in the zirconia matrix because barium titanate extends in the poling direction, while zirconia maintains its crystal structure and orientation. The internal stress, which is reversible, is one possible explanation for the strength change. However, this hypothesis alone cannot explain the tendency of the width of the strength distribution to become smaller with a longer poling time. The possibility exists that a change in the microstructure would occur with several minutes of poling, but this seems to be quite unlikely due to the small strength increase ratio.

We then measured the effect of the applied electric field intensity on the change in strength for the composite with 5 mol% barium titanate [5]. The results are shown in Fig. 3, in which the filled circles represent the mean values at each poling time. The rate of change in the mechanical strength first increased with an increase in the applied field. It then remained constant from around 40–60 kV/cm, and the strength did not change after applying electric fields larger than 40–60 kV/cm. When the electric field is removed after the polarization treatment, the distortion in the crystal structure that remains is comparable to that caused by the electric field [32]. Therefore, the result from Fig. 3 indicates that the residual stress influenced the increase in strength.

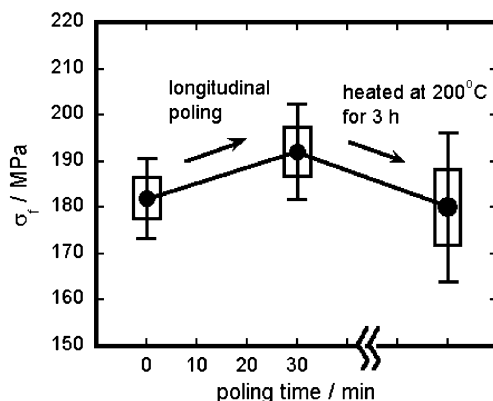


**Fig. 3** Dependence of the strength on applied electric field. The *open symbols* are the raw data, and the *closed ones* are the averages [5]

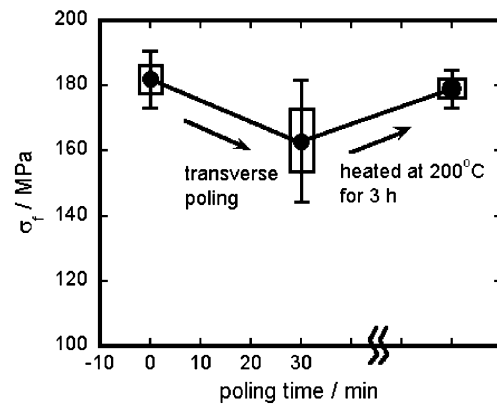
If the increase in strength is really due to the distortion of barium titanate and the presence of internal stresses upon the application of an electric field, then the strength will revert to its original value when the distortion is removed. When barium titanate is transformed from the ferroelectric phase to the paraelectric phase upon heat treatment, the spontaneous polarization disappears. When it is transformed back to the ferroelectric phase, the spontaneous polarization will show random orientations and the residual polarization will not exist. Therefore, if the increase in strength is caused by the residual distortion affects, the strength of the specimen could return to the original value by means of the phase transformation of barium titanate from the ferroelectric phase to the paraelectric one. To evaluate this possibility, experiments were performed as follows.

The strengthened specimens were heated to 200°C, a temperature that is above the transition temperature of approximately 120°C, to remove the spontaneous polarization. Following this, they were cooled down to room temperature. The treated specimens were then compared with the original specimens and strengthened specimens, and the results are shown in Fig. 4. The results for transverse poling followed by heat treatment are presented in Fig. 5. The error bar (=average $\pm$ 1.96 $\times$ SE) indicates the 95% confidence limit of the average mechanical strength, and the width of the box (=average $\pm$ SE) indicates the 70% confidence limit. Compared to the untreated samples, the mechanical strength was found to decrease upon transverse poling (Fig. 5). Whether the strength change is statistically meaningful is uncertain because in both cases, the boxes do not overlap each other [5].

The poled specimens were then heated at 200°C to remove the spontaneous polarization. The strength of both the strengthened and weakened specimens decreased to a value close to the original strength. This result provides further support for the explanation that the residual



**Fig. 4** Change in the mechanical strength of 10 mol% BT/8YSZ on longitudinal poling and on heat-treatment after poling [5]



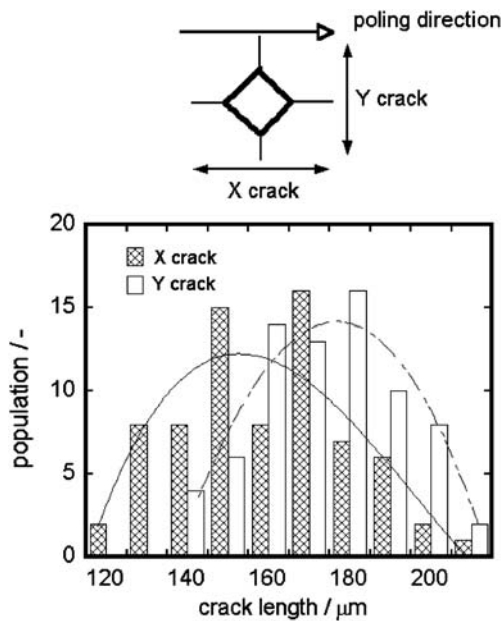
**Fig. 5** Change in the mechanical strength of 10 mol% BT/8YSZ on transverse poling and on heat-treatment after poling [6]

distortion accompanied by residual polarization is the origin of the change in strength.

Another piece of evidence found in Figs. 4 and 5 in support of this explanation is the overlapping of the boxes in both the longitudinal and transverse cases. In addition, irreversible changes such like crack formation would not likely occur upon polarization treatment. Therefore, we can conclude that the orientation of polarization is the main cause of the increase in strength.

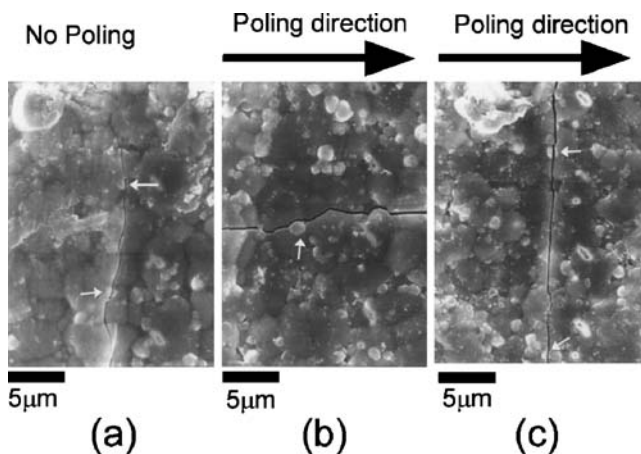
We next investigated the effect of the generated internal compressive stress on crack propagation. Vickers' indentation was performed with load of 1 kgf (i.e., 9.8 N) after the polarization treatment as shown in Fig. 1(c), and the generated crack length was measured in directions parallel and perpendicular to the applied field. In monolithic ferroelectric materials, the indentation crack has been reported to propagate anisotropically according to the direction of the applied field [18, 19, 25, 33]. However, for the composite materials in this paper, no difference was observed in the crack length between the parallel and perpendicular directions.

The distribution of the crack length in both directions was investigated by measuring the crack length on 80 samples poled in the same manner. The results are shown in Fig. 6. Two sets of histogram are presented for the crack length, and the peak of the distribution of crack length in the poling direction ( $X$  crack) is at the left of the peak for the crack perpendicular to the poling direction ( $Y$  crack). The difference between the length of the  $X$  crack and  $Y$  crack are statistically different because the bottom of the error bar of the  $Y$  crack is significantly higher than the top of the error bar of the  $X$  crack [5]. This was probably due to the crack propagation being obstructed along the poling direction and the crack propagation perpendicular to the poling direction being facilitated by the residual stress, after taking into account of the difference in bending strengths for anisotropic poled composites [4–6].



**Fig. 6** Indentation crack Observation: Distribution of crack length in the parallel and perpendicular direction to poling [5]

To examine in detail the behavior of crack propagation, images of the samples were taken by SEM. The crack (a) in the polarization-untreated specimen (b) parallel to the poling direction and (c) perpendicular to the poling direction were observed. The SEM micrographs of these cracks are shown in Fig. 7(a–c), respectively. In most of these cases, the cracks are seen to propagate mainly in the grains of the zirconia matrix. However, the crack in the poling direction [Fig. 7(b)] is different, as it is seen to avoid the barium titanate grains (which have a lower strength than the zirconia matrix) and make a detour around them. In contrast, the other two SEM micrographs (representing the cracks in the untreated specimen and cracks perpendicular

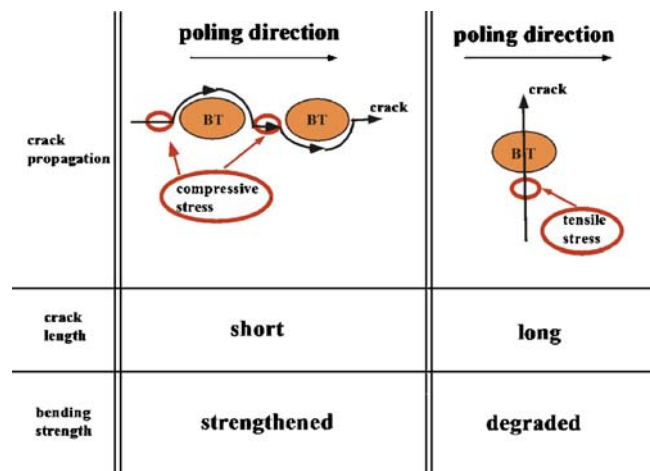


**Fig. 7** SEM micrographs of crack propagation in 10 mol% BT/8YSZ; (a) no poled, (b) the parallel direction to poling and (c) the perpendicular direction to poling [5]

to the poling direction) show the cracks to propagate mainly in the zirconia matrix and penetrate the barium titanate grains. Detours of the cracks around the barium titanate grains were not observed. From our previous studies [4–6], the relationship between crack propagation, crack length, and bending strength versus the poling direction is summarized in Fig. 8.

As noted in the introduction, many interpretations have been proposed for monolithic piezoelectric ceramics. Some of them are now outlined. Many studies have examined the change in fracture mode upon polarization treatment. Kim et al. [16] reported the change in the fracture mode of PZT ceramics and explained this phenomenon in connection with the domain structure. Fu et al. investigated the effects of an applied electric field on the fracture toughness of PZT ceramics [25]. They showed that the electric field affected the fracture toughness negatively, a result that was similar to their previously reported results on the mechanical strength [34]. Their results are also in agreement with our own, in both the mechanical strength aspect and the crack propagation aspect [4–6]. They suggested that the electric field-dependent mechanical response in monolithic PZT ceramics could be due to many reasons, such as the piezoelectric effect, electrical yielding at the crack tip, domain switching, internal stress, or partial discharge [25]. Han et al. [26] also examined the change in fracture toughness due to an electric field. They reported both negative and positive effects on the fracture toughness, depending on the direction of the electric field. To explain their results, they made reference to the domain switching at the vicinity of the crack tip that was influenced by both the mechanical stress and the electric field.

Lucato et al. [28] measured the *R*-curves of PZT ceramics for different poling directions. They observed a steep rise at the beginning of the *R*-curve for all poling



**Fig. 8** Relation between crack propagation, crack length and bending strength against poling direction

directions, but the plateau values were different. The poling in the thickness direction resulted in the highest fracture toughness, while those parallel to the loading direction led to the lowest value. They attributed the toughening of this poled PZT to the domain switching.

Fett et al. employed a statistical approach to analyze the effect of the poling direction on the tensile [29] and bending [30] strengths of PZT ceramics. They showed the increase in the scattering of strength data versus polarization field intensity, which could be connected to the domain switching variation.

The investigations on the polarization-dependent mechanical properties described above were all carried out on monolithic piezoelectric ceramics. We went one step further by fabricating ceramic composites dispersed with piezoelectric particles and demonstrated the toughening and weakening by polarization treatment in different directions [4–6].

Following our study, Hwang et al. [7] fabricated a MgO/10 vol% BaTiO<sub>3</sub> nanocomposite. Liu et al. [8] also fabricated a similar Al<sub>2</sub>O<sub>3</sub>/15 vol% LiTaO<sub>3</sub> composite and demonstrated an improved flexural strength and fracture toughness by applying a polarization treatment along the width of bending specimens. However, both of these properties decreased along the thickness direction. The strength gain by the polarization treatment was as small as 1.6% of the original bending strength; however, the strength value was maintained or slightly improved by dispersion compared to the monolithic alumina. The toughening mechanism in structural ceramic composites containing ferroelectric particles was attributed to domain switching [35].

As reviewed above, almost all of the mechanisms explaining mechanical properties on polarization treatment are based on the domain structure leading to an anisotropic distribution of internal stress, even in the piezoelectric particle-dispersed composite. Liu et al. [8] also observed a detour in the path of crack propagation around the piezoelectric dispersoids in the case of improved mechanical properties. This was attributed to the internal stresses induced by residual polarization or domain formation (which was similar to what we observed), while other mechanisms such as crack branching or domain switching might also have contributed to the improvement in the property. They also attributed the penetration of the crack into the piezoelectric dispersoids to the arrangement of domains when poled vertically to the propagation direction.

Kroupa et al. [36] proposed a theoretical model to describe the anisotropic distribution of internal stresses on poled PZT ceramics based on Eshelby's solution of the "elliptical inclusion" problem [37]. They assumed a situation in which one spherical ferroelectric grain was surrounded by material with different relative orientations

of spontaneous polarization. Their model can be applied to our results with a limited degree of success because the surrounding matrix of the composites in our study consisted of paraelectric ceramics.

Their result indicated that the stresses in the surrounding matrix are very complicated and decays at a rate that is inversely proportional to the third power of the distance from the particle. This suggests that the strengthening should be based on the short-range interactions that exist between the particle–matrix interface and the cracks. The detour taken by the crack path is an indication of the internal tensile stress that is generated in the matrix at the vicinity of the dispersoid in the poling direction, and this phenomenon is thought to cause the increase in strength shown in Fig. 4 or Fig. 5.

To realize the concept regarding the strength control of structural ceramic composites, the combination of the materials used for the matrix and dispersoid is important. The piezoelectricity of the dispersoid and the mechanical property of the matrix should be maintained separately by avoiding any chemical reaction between the two components. It is also important to employ a fabrication atmosphere that does not affect the properties in both components. An inert or reducing atmosphere is usually required to fabricate structural ceramics that contain carbon or nitrogen. Furthermore, special attention must be paid toward retaining the electric insulation of piezoelectric dispersoids during the fabrication process in such environments.

Next, the appropriate combination should exhibit mechanically useful properties. A composite dispersed with piezoelectric particles tends to exhibit poor mechanical properties since typical piezoelectric materials have weaker mechanical strength. In addition to choosing piezoelectric dispersoids with a relatively large mechanical strength, optimizing the mechanical properties of ceramic composites must take into account other properties such as the coefficient of thermal expansion and the elastic modulus. If a nano-sized composite can be realized by reducing the size of the dispersoid, a small amount would improve the mechanical properties of the composite [7].

Finally, the electrical properties are also an important factor. Larger piezoelectric properties are not always desirable. To maximize the change in the mechanical strength, detailed quantitative analysis such as predicting the stress field distribution should be conducted to identify the best piezoelectric dispersoid. The permittivity is another important factor because the electric field that effectively acts on a component in a composite is inversely proportional to its permittivity. Since the permittivity of piezoelectric materials is usually large, enhancing the permittivity would be helpful as far as the mechanical properties are maintained.

In conclusion, the present concept can be realized in ceramic composites with a variety of material combinations. In addition to strengthening by polarization and in situ strengthening by an applied field, strength degradation by a cyclic electric field could also be useful to recycle a ceramic composite that contains piezoelectric dispersoids.

#### 4 Conclusions

The control of mechanical strength in structural ceramic composites was reviewed, with the main focus on our previous studies. In the system of zirconia ceramics dispersed with barium titanate particles, an increase in bending strength is observed when an electric field is applied in the longitudinal direction. The strengthened specimen by polarization treatment returns to almost the original state by heating above the  $T_c$  of barium titanate. These results indicate that internal stress is generated in the zirconia matrix at the vicinity of barium titanate in the poling direction because barium titanate deforms upon polarization treatment. Furthermore, a dependence of the crack propagation on the direction of the applied electric field was observed, and several detours of cracks around barium titanate grains were seen in the poling direction. Considering the increase in strength in the poling direction, this is thought to be influenced by the internal stress generated, which then causes the increase in strength. The present concept can be realized in ceramic composites with a variety of material combinations.

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