



R-curves in transformation toughened lead zirconate titanate



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ABSTRACT

Composites from lead zirconate titanate (PZT) and yttria stabilized zirconia were manufactured to assess cracking and R-curve behavior in compact tension specimens. Additions of zirconia led to a reduction in PZT grain size and shifted the fracture behavior from intergranular fracture to intragranular fracture. The R-curves exhibited higher starting values, a steeper slope and a higher plateau value with increasing content of zirconia, albeit the effects on the toughness values were not more than 20%. The results are rationalized through increased crack tip toughness, reduced ferroelastic toughening and enhanced transformation toughening.

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1. Introduction

Lead zirconate titanate (PZT) is the main component in multilayer actuator stacks [1]. Their assembly necessitates a combination of electrically active and inactive regions with concurrent strain incompatibility and high tensile stresses at electrode edges [2]. As a consequence, cracks of several hundred micrometers can develop and threaten the functionality of the multilayer stack [1]. According to Munz [3], typical ceramics fail from natural defects or small surface cracks, where toughening is only relevant over a very small distance of about 50 μm . In contrast, PZT is one of the few materials, where both elliptical surface cracks as well as extended through-thickness cracks are of relevance. Ferroelastic toughening in this material is an intricate behavior that leads to R-curves depending on geometry and local stress distribution at the crack tip [4]. This feature provides a further complexity compared to the case of crack bridging due to crack closure stresses and R-curves dependent on crack/specimen geometry as noted by Fett and Munz about 20 years ago [5].

The study of crack extension in PZT first focused on through-thickness cracks [6–9]. Special emphasis was placed on the effect of prior poling [7], influences of composition [8] and subcritical crack growth [9]. Somewhat later, details on the crack propagation of surface cracks in PZT were revealed [10,11]. These, however, had required consideration of the stress distribution in lead zirconate titanate in bend bars as affected by plastic deformation (domain switching) [12]. Domain switching due to deviatoric stresses introduces a further complication, similar to the specimen thickness-dependent plastic zones in metals, which leads to thickness-dependent R-curve behavior [13]. The process zone due to domain switching can be monitored using both diffraction techniques [14–16] and electrooptical methods [17]. A recent review by Schneider [18] summarizes the wide variety of effects in the field of ferroelastic toughening of PZT.

Expanded application of lead zirconate titanate into a number of new applications can be envisaged if the toughness could be increased. Crack bridging by stiff elongated grains to further crack bridging presumably would strongly hamper the excellent piezoelectric properties of PZT. Transformation toughening of a second phase zirconia [19,20], however, may be suitable to increase the fracture toughness of piezoceramics with little reduction in salient actuator properties. This toughening mode requires metastable tetragonal zirconia to undergo phase transformation to monoclinic zirconia with an

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Nomenclature

PZT	lead zirconate titanate
R-curve	resistance curve
PZT- x TZ	lead zirconate titanate with tetragonal stabilized zirconia
d_F	feret's diameter
λ	stretching exponent
K	fracture toughness
K_{ini}	initial fracture toughness
K_{max}	maximum fracture toughness (plateau toughness)
$K_{sh,max}$	maximum shielding toughness
a	crack length
Δa	change in crack length

attendant volume increase of 4% in a process zone around the crack tip due to its enhanced stress field [19,20]. Although PZT is thermodynamically compatible with zirconia, only few studies have been reported so far [21,22]. In order to obtain optimum mechanical and piezoelectrical properties a composition at the morphotropic phase boundary (MPB) has to be obtained. The MPB marks the phase assembly where both tetragonal and rhombohedral symmetries coexist and maximize ferroelectric switching under both electrical and mechanical driving force. An in-depth study of phase composition of lead zirconate titanate with yttria-doped zirconia has recently been performed [23]. While this former work also reported ferroelectric and piezoelectric properties, the current report is the first to describe transformation toughening and R-curve behavior in optimized phase compositions in PZT/zirconia composite materials.

We focus on crack extension in compact tension specimens and correlate it with microstructure and the mode of fracture (intergranular and intragranular fracture). The R-curve behavior is quantified in terms of crack tip toughness, crack tip shielding, plateau value but also in terms of steepness of the R-curve [24].

2. Materials and methods

PZT- x TZ ($x = 0, 5, 10, 20$ vol.%) composites were prepared from Ba and Nb modified lead zirconate titanate solid solution (denoted as PZT) and different amounts of tetragonal stabilized zirconia with 3 mol% Y_2O_3 (denoted as TZ). The powder mixtures with the nominal composition $[Pb_{0.98}Ba_{0.01}][(Zr_{0.53}Ti_{0.47})_{0.98}Nb_{0.02}]O_3$ were prepared by the mixed-oxide method using PbO (Sigma, 99.9% purity, 211907, Steinheim, Germany), TiO_2 (Alfa, 99.8% purity, 042681, Karlsruhe, Germany), ZrO_2 (TZO, Tosoh, Yamaguchi, Japan), Nb_2O_5 (Sigma, 99.9% purity, 208515, Steinheim, Germany) and $BaCO_3$ (Alfa, 99.8% purity, 014341, Karlsruhe, Germany) as precursors. Powder mixtures were homogenized in a planetary mill and calcined twice at 900 °C for 1 h followed by milling after each calcination step. Various concentrations (5, 10 or 20 vol.%) of as-received tetragonal stabilized zirconia with 3 mol% Y_2O_3 (TZ3Y, Tosoh, Yamaguchi, Japan) were added to the prepared powder mixture. The PZT- x TZ ($x = 0, 5, 10, 20$ vol.%) powder mixtures were additionally homogenized in an attrition mill, pressed into larger plates (65 mm in diameter) using first uniaxial pressing (49 MPa) followed by isostatic pressing (300 MPa) and sintered at 1275 °C for 2 h. After sintering the edges of the plates were cut off and the samples were polished. From each sample a compact disk with dimensions of $33.6 \times 35 \times 3.2$ mm³ was cut out using a wire saw. A detailed description of the synthesis is given elsewhere [23]. The grain size for each composition was determined by analyzing the micrographs of polished and thermally etched surfaces. The digitized microstructures were processed with the Image Tool software [25] to obtain the areas of more than 200 grains. The grain size expressed as the Feret's diameter (d_F) is provided in Table 1 [23].

For the fracture experiments, compact tension (CT) specimen with dimensions of approximately $33.6 \times 35 \times 2.8$ mm³ for all compositions were obtained. A 33.6×35 mm² face was polished to a surface finish of 1 m with diamond paste. Two holes with diameter of 7 mm were drilled in the thickness direction and a notch was cut in the middle of a 33.6 mm long edge,

Table 1
Grain size in PZT- x TZ composites as a function of content of ZrO_2 [23].

Material	d_F (st. dev.) for PZT grains (μ m)
PZT	3.3 (\pm 1.6)
PZT-5TZ	1.6 (\pm 0.8)
PZT-10TZ	1.3 (\pm 0.5)
PZT-20TZ	1.0 (\pm 0.4)

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