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Mechanical properties and crack growth behavior in poled ferroelectric PMN–PZT ceramics

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Abstract

Ferroelectric lead magnesium niobate–lead zirconate titanate with the formula $x\mathrm{Pb(Mg_{1/3}Nb_{2/3})O_3}$–$(1-x)\mathrm{Pb(Zr_{0.52}Ti_{0.48})O_3}$ where $x = 0.0$, 0.2, 0.4, 0.6, 0.8 and 1.0 was fabricated using a conventional mixed-oxide method. The densest ceramics were selected and some samples were subjected to electrical poling before mechanical investigation by indentation technique. In the poled ceramics, the growth of radial cracks was found to be dependent on the orientation of ferroelectric domains with respect to the poling direction. The crack length then showed significant anisotropy in the directions parallel and perpendicular to the poling which affected the K_{IC} values. However, the domain reorientation had no effect on H_V values. The hardness tended to reduce with increasing mole ratio of PMN in the ceramics. The microstructure of the fracture surfaces revealed changes mainly of the intergranular fracture mode in the monolithic PMN and PZT to a mixed-mode of inter/transgranular fracture in the PMN–PZT ceramics.

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Keywords: PMN–PZT; Vickers indentation; Hardness; Fracture toughness

1. Introduction

Ferroelectric lead magnesium niobate (PMN) and lead zirconate titanate (PZT) materials are promising candidates for applications in electronic and microelectronic devices [1]. PMN exhibits a high dielectric constant, high electrostrictive strain coefficient, low sintering temperature and a broad dielectric maximum just below room temperature [2–5]. Together with a unique microstructure, PMN ceramics exhibit low loss and non-hysteretic characteristics. PZT ceramic is the most extensively used piezoelectric material because of its high electromechanical coupling coefficient [5,6] which is higher than that of PMN. However, the disadvantages of PZT ceramics are their high loss due to their highly hysteretic characteristics and relatively high Curie temperature (T_c). The latter makes them unsuitable for applications requiring a T_c close to room temperature. To bring together the individual benefits of PMN and PZT, PMN–PZT ceramics have been developed.

The mechanical behavior of ferroelectric ceramics has not been of great relevance to their use because they are not intended for structural applications. However, the structural stability of ferroelectric ceramics has recently attracted more attention due to an increasing demand for applications requiring long lifetimes and large displacements under continuous operation. As with other ceramics, the main disadvantage of these ferroelectric ceramics is their brittleness. They tend to develop cracks due to mechanical and electrical loading which eventually cause fracture. The aim of this research is to explore the effect of electrical poling on the mechanical characteristics of PMN–PZT ceramics.

2. Experimental

Ceramics of composition xPMN–$(1-x)$PZT where $x = 0.0$, 0.2, 0.4, 0.6, 0.8 and 1.0 were fabricated using a

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conventional mixed-oxide method with PMN and PZT powders. To obtain the PMN powder, the first step was to prepare the columbite-like phase $MgNb_2O_6$ (MN). This was synthesized by ball-milling reagent grade $(MgCO_3)_4 \cdot Mg(OH)_2 \cdot 5H_2O$ and Nb_2O_5 in ethanol, drying and calcining at 1150 °C for 4 h with a heating rate of 10 °C min^{-1}. The MN powder was then mixed with PbO, dried and subsequently calcined at 800 °C for 4 hours with a heating rate of 10 °C min^{-1}. PZT powder was prepared by a simpler mixed-oxide method using PbO, ZrO_2, and TiO_2 calcined at 800 °C for 2 h with a heating rate of

Table 1
Physical characteristics of the PMN–PZT ceramics chosen for mechanical investigation

PMN–PZT ratio	Sintering temperature (°C)	Density (g cm^{-3})	Grain size (μm)
1.0PMN	1250	7.91 ± 0.02	2.67 ± 0.21
0.8PMN–0.2PZT	1275	7.58 ± 0.08	1.47 ± 0.17
0.6PMN–0.4PZT	1275	7.64 ± 0.03	1.08 ± 0.03
0.4PMN–0.6PZT	1275	7.60 ± 0.08	2.03 ± 0.09
0.2PMN–0.8PZT	1250	7.73 ± 0.25	1.20 ± 0.01
1.0PZT	1250	7.69 ± 0.04	1.55 ± 0.01

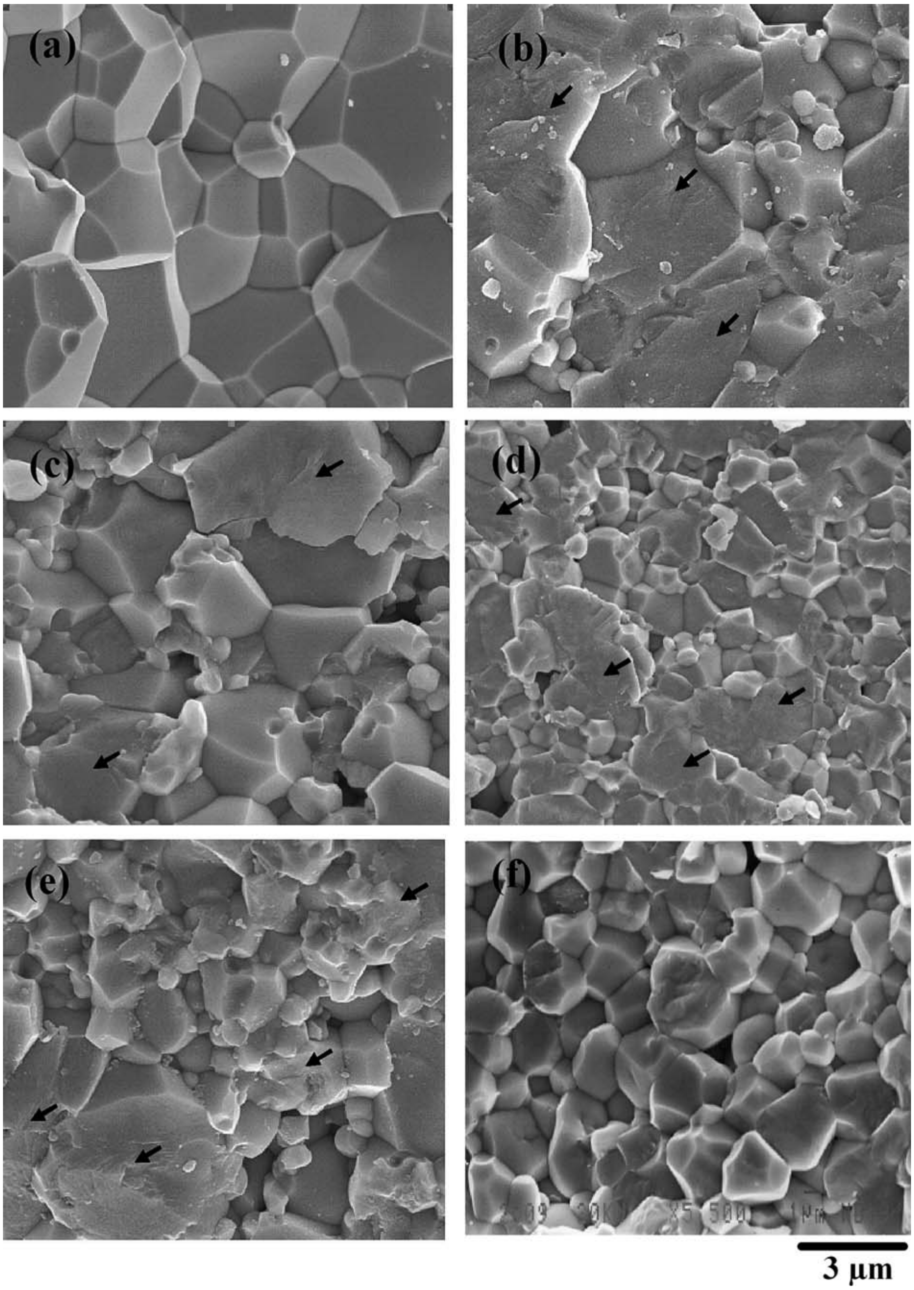

Fig. 1. SEM micrographs of the fracture surfaces of (a) PMN, (b) 0.8PMN–0.2PZT, (c) 0.6PMN–0.4PZT, (d) 0.4PMN–0.6PZT, (e) 0.2PMN–0.8PZT and (f) PZT ceramics. Arrows indicate transgranular fracture grains, but most of the grains show intergranular fracture.