Crack growth in PMN-PT piezoelectric ceramics by cyclic electric fields

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ABSTRACT

Pre-existing cracks introduced by a Vickers diamond hardness indenter in PMN-0.3PT, which displays piezoelectric properties, increase in length under the action of low frequency cyclic electric fields applied normal to the crack. A minimum applied field of 1.1 x Ec is required to cause crack growth. In applied fields of 1.85-5.70 x Ec cracks grow to a common limiting length which is approximately 0.8 times the separation between the electrodes. New cracks are not generated at the corners of a Vickers diamond indent by applied fields up to 5.70 x Ec.

Keywords: piezoelectric ceramics, electrostriction, PMN-PT, cyclic electric fields, fracture, crack propagation

1. INTRODUCTION

Electrically induced fatigue has been reported in lead zirconate-titanate (PZT) and lead lanthanum zirconate-titanate (PLZT)¹⁻⁴. In early experiments, Cao and Evans¹ observed that cracks created by a Vickers diamond hardness indenter in 58/42 PZT grow in a direction normal to applied low frequency cyclic electric fields, with peak to peak amplitudes less than the coercive field, E_c. These authors also report that extensive crack growth, at a rate independent of the crack length, occurs in cyclic fields with amplitudes greater than E_c. In contrast to these results, Lynch et al.^{2,3} showed that pre-existing cracks in 8/65/35 PLZT grow rapidly to a length of the order of the separation of the electrodes, when subjected to fields greater than E_c. They also report that the crack growth observed in unipolar cyclic fields was significantly greater than the crack growth in bipolar fields, indicating that the negative component of a bipolar cyclic field is ineffective for causing crack growth. As part of a study of commercial grades of piezoelectrics, pre-existing cracks in Navy type VI PZT were also found to increase to a limiting length after relatively few cycles of low frequency electric fields applied normal to the crack face⁴. These latter experiments also showed that, at a common frequency of 1.46 Hz, the limiting crack length increased progressively with increasing peak to peak field intensity in the range 0.9-1.8 x E_c. The effect of low cycle fields on the growth of pre-existing cracks in commercial compositions of lead magnesium niobate-lead titanate (PMN-PT) has been investigated, in view of the importance of these materials for applications in smart structures and sonar transducers. Preliminary findings on a ceramic of composition PMN-0.3PT (Sensor Technology Ltd. BM 660 composition) which displays piezoelectric properties⁵, are reported and discussed in this presentation, while subsequent findings on a PMN-PT composition with relaxor properties⁶, will be presented at a later time.

2. EXPERIMENTAL METHODS

Powders of PMN-0.3PT were synthesized at Sensor Technology Ltd., Collingwood, ON, by mixing, grinding and calcining component oxide powders. Sintered ceramics prepared from these powders were cut and ground to dimensions of 12.7 mm x 3 mm x 1.27 mm. The 3-mm faces of the specimens were screen printed with silver paste to

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form electrodes for applying electric fields, as shown in Figure 1. These electrodes were also used to pole the specimens, using standard production procedures. The upper 1.27-mm face of each specimen was polished to a smooth finish with silicon carbide paper and then indented with a Vickers diamond pyramid, using a load of 20 N, applied for a period of 10 sec. The diamond shaped indent was aligned so that one set of corners was parallel, and the other normal, to the electrodes, as indicated in Figure 1. The load applied to the Vickers diamond induced cracks 200-300 μ m in length that emanated from the corners of the indent. Cracks oriented parallel to the electrodes (i.e. normal to an applied field) were used to investigate the effect of cyclic electric fields.



Fig. 1. Specimens used for crack growth studies in cyclic fields.

To determine the coercive field, E_c , of the PMN-0.3PT ceramics, polarization vs. field hysteresis plots of selected specimens were measured (before indentation) with an SS05 polarization measurement system⁷. Crack growth experiments were performed using a specially designed specimen mount, which is shown schematically in Figure 2. The specimen is rigidly clamped in position by two spring loaded pogo pins, which act as electrodes for applying electric fields and ensure that the opposite silvered face of the sample is maintained in good contact with a grounded brass block that forms the other electrode.



Fig. 2. Brass fixture used for crack growth studies in cyclic fields.

For in situ studies of crack growth, the specimen mount was placed on the stage of an optical microscope. The length of the crack was measured before and after the application of an electric field, by aligning one end of the crack with cross-hairs incorporated into the lens of the microscope and then traversing the stage to move the mount from the left tip of the crack, past the indent, to the right tip of the crack. The crack length was obtained from readings of a Mitutoyo model ID-C112E displacement gage which was mounted against the microscope stage. The resolution of the displacement gage was 1 μ m; however, due to uncertainties in detecting the termination of hairline cracks, brought about by the oil immersion, the overall accuracy of the crack length measurements was estimated to be \pm 50 μ m. Cyclic electric fields of amplitude 0.64-5.60 x E_c were applied for various time periods, at frequencies of 2 and 5 Hz. To prevent possible arcing, the sample and contacts were immersed in insulating oil.

3. RESULTS AND DISCUSSION

3.1 Polarization vs. Field Measurements

The polarization vs. electric field plot for a specimen of PMN-0.3PT in the uncracked condition is shown in Figure 3. The plot has a square hysteresis loop with a remanent polarization greater that 90% of the saturation polarization and shows very sharp switching in the sign of polarization on passing through the coercive field, E_c . From measurements made on several specimens prior to indentation, the average value of E_c for the samples of PMN-0.3PT was determined to be 0.37 MV/m, which is less than half the value of 0.88 MV/m obtained previously⁴ in similar experiments on Navy Type VI PZT. The average value of $E_c = 0.37$ MV/m was used to compute the reduced field, E/E_c , which is used to express the magnitude of the applied cyclic electric field in following results.



Fig. 3. Polarization vs. electric field hysteresis plot for PMN-0.3PT.

3.2 Crack Growth in Cyclic Electric Fields

The extension of cracks subjected to various reduced electric fields in the range 2.18-5.60, at a common frequency of 5 Hz, is shown in Figure 4. It is evident that cracks of different initial lengths all extend to a common limiting value, but the progress of the crack extension varies with the magnitude of the applied field. In the sample exposed to the lowest reduced field of 2.18, no extension was observed until the field was imposed for 2000 cycles, after which the crack extended directly to a length of 0.49 mm. At an intermediate reduced field of 3.91, the crack showed a series of discrete intermediate extensions before achieving the same length of 0.49 mm, after 100 cycles. At the highest applied reduced field of 5.60, the crack again displayed an intermediate step and then increased in length to 0.49 mm after only 10 cycles. Increasing the magnitude of the applied field thus achieves the common terminal length in a reduced number of cycles. As these results are analogous to the effect of increasing the applied load in a mechanical fatigue test, it its likely that the multiple stages of crack extension are caused by microstructural features that can act as crack stoppers, so that higher fields, or longer cycles, are required to achieve the common limiting extension. While the effect of microstructural features is beyond the scope of the present project, it will be investigated by subsequent microscopic examination of the tested samples. The analogous behaviour of these results to mechanical fatigue also suggests that



there should be a level of applied reduced electric field below which pre-existing cracks will not grow to the limiting extension over finite cycling times.

Fig. 4. Crack length vs. number of cycles, for $E = 2.18-5.60 E_c$.

The concept of a common limiting crack extension was further examined by plotting the terminal values of crack extension obtained (at various frequencies) against the applied reduced field over the entire experimental range from 0.64-5.60, to obtain the plot shown in Figure 5. The individual extensions observed on each side of the indent are displayed in this plot and show that there are measurable differences between crack growth in the two opposing directions, both of which are normal to the applied field. At reduced fields in the region of 0.64-1.07 the terminal crack length lies between 0.2-0.3 mm, which is the range of values for the pre-existing cracks; i.e. there has been no crack growth. When the reduced field was increased to 1.13, the crack on one side of the indent increased, but no growth was observed on the crack emanating from the opposite corner of the indent. The critical value of reduced field for inducing crack growth in this material is thus close to 1.10.

When the reduced field was increased into the range 1.8-5.7, the terminal lengths of cracks on either side of the indent all lie between 0.41-0.52 mm, while the individual crack extensions conform to an average value of 0.46, within the limit of experimental error of \pm 50 µm. This common terminal crack extension is consistent with the limiting crack lengths obtained for the samples referred to in Figure 4, which were all subjected to the same range of applied reduced fields. The present results also confirm the previous finding of a limiting crack extension length in Navy Type VI PZT when subjected to applied reduced fields in the range 0.9-2.0⁴ and agree with the observation of Lynch et al.², that precracks in PLZT grow rapidly to a common length when exposed to cyclic fields of 1.2-2.0 x E_c. The common overall crack length of 0.92 mm (2 x 0.46 mm) observed in the present results is approximately 0.8 times the separation between the two electrodes which also concurs with the results obtained by Lynch et al.² However, none of these recent

results agree with the earlier finding of Cao and $Evans^1$ that crack growth in PZT at electric fields greater than E_c conform to steady state condition, in which the growth rate is independent of the crack length.

The results in Figure 5 also show that some samples did not display pre-existing cracks after the Vickers indentation. A sample that had no crack at either corner of the diamond indentation did not generate any further cracks when subjected to an applied reduced field of 4.44 x Ec for a period of 6000 cycles. Another sample that had a crack at only one corner of the diamond indent showed growth of this crack to a limiting extension of 0.47 mm after 6000 cycles in a reduced field of 5.7 x Ec, but an additional crack was not generated in the opposite corner of the indent as a result of this treatment. It is thus concluded that applied reduced electric fields up 5.7 are not effective for generating new cracks in otherwise crack-free PMN-0.3PT.



Fig. 5. Terminal crack length vs. reduced field.

The overall conclusions of the present experiments are as follows:

1. A critical minimum applied field, of the order of $1.1 \times E_c$, is necessary to cause the extension of pre-existing cracks in piezoelectric samples of PMN-0.3PT.

2. Applied fields in the range of $1.85-5.60 \times E_c$ cause pre-existing cracks to grow to a limiting size of approximately 0.8 times the separation between the electrodes.

3. Imposed fields up to $5.70 \times E_c$ do not initiate fresh cracks at the corners of Vickers diamond indents in piezoelectric samples of PMN-0.3PT.

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