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**EFFECT OF CYCLIC ELECTRIC FIELDS ON CRACK GROWTH
IN A MODERATELY HARD PIEZOELECTRIC**

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ABSTRACT

Pre-existing cracks introduced by a Vickers diamond indenter in the moderately hard PZT BM400 (Navy Type I) do not exhibit visible crack growth when subjected to low frequency electric fields with amplitudes up to 1.84 times the coercive field of $E_c = -1.63$ MV/m. This behaviour, contrasts with that observed in soft piezoelectrics, which exhibit crack growth in electric fields as low as $0.9 E_c$. but confirms previous observations that field-induced crack growth does not occur in hard PZT piezoelectrics in applied fields up to 1.63 - $1.68 E_c$. The minimum applied field to induce crack growth in piezoelectrics is considered to be a direct function of the coercive field and an indirect function of the piezoelectric strain constant and the mechanical compliance of the ceramic. However, as piezoelectrics are not used at fields above $0.5E_c$, the minimum fields to induce crack growth in these hard piezoelectrics will not be encountered in practice.

INTRODUCTION

Cracks generated by a Vickers hardness diamond indenter have been observed to grow in a direction normal to a low frequency applied electric field in relatively soft piezoelectrics, such as 58/42 lead zirconate titanate (PZT) [1], 8/65/35 lead lanthanum zirconate titanate (PLZT) [2] and BM532 (Navy Type VI) PZT which is doped to create vacancies at Pb^{2+} sites, in order to increase the mobility of domain walls [3]. Similar crack growth has also been observed in BM660 [4], which is a 70/30 lead magnesium niobate-lead titanate (PMN-PT) ceramic that exhibits piezoelectric, as opposed to electrostrictive, behaviour on cooling

through a Curie temperature, T_c [5]. The crack growth observed in these piezoelectrics, in cyclic electric fields up to three times the coercive field, E_c , is quantitatively different, however, since some investigators report that field induced crack growth is proportional to crack length [1], while others report that pre-existing cracks only grow up to a limiting size, for a given applied field [2,3,4].

PZT piezoelectrics such as BM800 (Navy type III) and BM200 have additional doping to generate internal biased fields that restrict domain wall movement [6,7]. These hard piezoelectrics have been shown to be resistant to crack growth when subjected to cyclic electric fields with magnitudes up to 3.15 MV/m [8], which is the safe upper limit to avoid dielectric breakdown in PZT samples with a thickness of 1.27 mm [9]. This absence of visible crack growth may be an artifact of the experiment, however, since the maximum permitted field to avoid dielectric breakdown across the narrow thickness of the specimens is only 1.63-1.68 times the coercive fields of these piezoelectrics, which range from 1.88-1.94 MV/m [8].

Additional field-induced crack growth experiments now have been conducted on BM400 (Navy Type I) PZT, which is doped with acceptor-type additions that generate space charges to restrict domain boundary movement. As shown by the data in Table 1, the physical properties of BM400 are close to those of the previously examined PZT BM800, which has the relatively high Curie temperature (T_c), coercive field (E_c), piezoelectric voltage constant (g_{33}) and mechanical quality factor (Q_M) typical of a hard piezoelectric, but are further removed from the properties of BM532 which has the relatively high dielectric constant (K_{33}), piezoelectric coupling factor (k_{33}), charge constant (d_{33}) and mechanical compliance (S_{33}) typical of a soft piezoelectric. On the basis of these comparative properties, BM400 may thus be regarded as a moderately hard piezoelectric.

Table 1. Dielectric, Piezoelectric and Mechanical Properties of Industrial PZTs

| Property | BM532 | BM400 | BM800 | Units |
|----------------------|--------------|--------------|--------------|---------------------------------|
| T_c | 210 | 350 | 325 | $^{\circ}\text{C}$ |
| K_{33} | 3250 | 1350 | 1000 | -- |
| $\text{Tan } \delta$ | 2.5 | 0.8 | 0.4 | % |
| E_c | 0.88* | -- | 1.88** | MV/m |
| | | | | |
| k_{33} | 0.75 | 0.70 | 0.64 | -- |
| d_{33} | 630 | 300 | 225 | 10^{-12} C/N |
| g_{33} | 20 | 25 | 26 | 10^{-3} Vm/N |
| Q_M | 70 | 500 | 1000 | -- |
| N_3 | 1850 | 1900 | 2000 | Hz.m |
| | | | | |
| S_{33} | 20.0 | 15.0 | 13.5 | $10^{-12} \text{ m}^2/\text{N}$ |

Data published by SensorTechnology [10].

* Ref. [3]. ** Ref. [8].

EXPERIMENTAL METHODS

Powder of BM400 was 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 12.7 mm x 3 mm faces of the specimens were screen printed with silver paste to form electrodes for applying electric fields for poling the specimens and for the crack growth experiments. The narrow 1.27 mm x 12.7 mm faces were polished with silicon carbide and then lapped with 6 μm diamond paste, to obtain a smooth flat surface, which was indented with a Vickers diamond pyramid, by applying a load of 20 N for a period of 10 sec. During this operation, the samples were oriented in the hardness tester so that one set of the corners of the diamond indent was aligned parallel, and one set normal, to the silvered electrode faces. Two specimens were not indented, to provide reference standards for comparative resonance frequency measurements.

The coercive field of the ceramic specimens of BM400 was determined from polarization vs. electric field plots using an SS05 polarization system, which has been described elsewhere [11]. An Agilent 4294A precision impedance analyzer was used to measure the length thickness extensional (LTE) mode resonance, which is the lowest frequency resonance dictated by the geometry of the specimens, to within an accuracy of ± 1 kHz.

For studying the effect of cyclic electric fields on the pre-induced cracks, the samples were mounted in a specially designed jig, which was placed on the translational stage of an optical microscope, as described previously [4]. Cyclic fields with a frequency of 5 Hz and amplitudes up to 3.8 kV, were applied for successive cycles using a Trek 609E-6 high voltage amplifier, controlled by a computer via the SS05 polarization meter. With the narrow specimen width of 1.27 mm the maximum selected output of 3.8 kV from the Trek instrument generated a maximum field of 3.0 MV/m across the electrode faces of the specimens. The samples were thus not vulnerable to dielectric breakdown, which in PZT typically occurs in the range of 4-8 MV/m [9]. To prevent possible arcing at high electric fields, the sample and contacts were immersed in insulating oil. After each application of a selected number of voltage cycles, changes in the length of pre-existing cracks oriented normal to the field were determined in situ to ± 50 μm by traversing a cross hair in the objective lens from one end of the crack to another at a magnification of 40x, as described previously [4]. At the termination of the cyclic field experiments, the cracks were re-measured in a higher power microscope at a magnification of 400x.

RESULTS AND DISCUSSION

Polarization vs. Field Measurements

The polarization versus field plot obtained for a reference sample of BM400 is given in Figure 1. The ceramic exhibits a remanent polarization of more than 95% of the saturation polarization of 0.53 C/m² and the square P-E hysteresis loop indicates sharp switching of the polarization on passing through the coercive field, E_c . The hysteresis loop is distinctly asymmetrical about the zero field point, due to an internal bias field, E_i , which was calculated

to be -0.42 MV/m, from the horizontal displacement of the loop centre from the $E = 0$ axis [12]. A coercive field of $E_c = -1.63$ MV/m (for an equivalent symmetrical hysteresis loop) was obtained by subtracting E_i from the value of the field at the inflexion point of the loop in negative field. This coercive field is slightly less than the value of $E_c = -1.88$ MV/m obtained for the hard piezoelectric BM800, but it is almost twice as large as the value of $E_c = -0.88$ obtained for the soft piezoelectric BM532 (see Table 1), confirming that BM400 is indeed a moderately hard piezoelectric. The impedance and the capacitance of the samples were also measured by the precision impedance analyser, but no significant changes were observed between as-indentured specimens and those subjected to various cycles of electric fields.

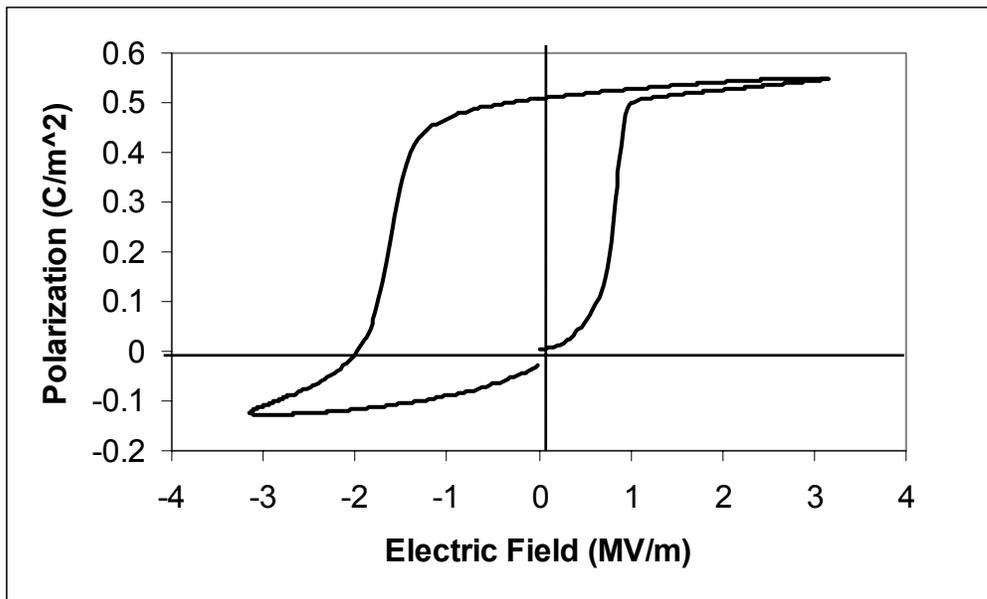


Figure 1. Polarization vs. field plot for a reference sample of BM400.

Effect of Applied Electric Fields on Pre-existing Cracks

Microscopic examination of the specimens at a magnification of 400x showed that the Vickers indentation generated cracks 100-150 μm in length, that emanated from opposite corners of the indents.

The in situ microscopic examination of eight samples of each BM400 after the successive application of 500 cycles of 5 Hz electric fields with progressively increasing strengths up to 3.0 MV/m showed no measurable increase in the length of pre-existing cracks oriented normal to the applied field. This finding was confirmed by the microscopic examination at a magnification of 400x at the termination of the cyclic field tests. The absence of crack growth in BM400 in applied electric fields up to $1.84 E_c$ is in sharp contrast to the behaviour of the soft piezoelectric PZT BM532, in which crack growth occurs in fields as low as $0.9 E_c$ [3], but confirms the absence of crack growth reported previously for pre-cracked samples of the hard piezoelectrics BM200 and BM800 when subjected to fields up to 1.63 -

1.68 E_c [8]. Since the coercive fields of BM400 and BM800 are more than twice as great as the coercive field of BM532, and their piezoelectric charge constants are less than half the charge constant of BM532 (see Table 1), it follows that these hard piezoelectrics are subjected to considerably lower electric stress at any given applied field. In addition, since their elastic compliance is less ≤ 0.75 times the compliance of BM532 (Table 1) the hard piezoelectrics will develop considerably less strain for a given degree of stress. This combination of electromechanical properties means that the hard piezoelectrics would be expected to show a much smaller degree of crack growth for a given applied field. Further, since a minimum applied field is necessary to induce crack growth in soft piezoelectrics [1-4], it follows that a significantly greater field will be necessary to induce crack growth in a hard piezoelectric. It is considered that the minimum field to initiate crack growth in a particular piezoelectric will be a direct function of the coercive field, and an inverse function of the piezoelectric charge coefficient and the mechanical compliance of the ceramic. This minimum field may not be achieved in experiments on hard piezoelectrics, however, because fields must be kept below 4-8 V/m to prevent dielectric breakdown in [9]. On the positive side, it can be concluded that, in common with the hard piezoelectrics BM200 and BM800, the moderately hard piezoelectric ceramic BM400 is also effectively resistant to electric field-induced crack growth, since industrial applications of PZT sensors and actuators are commonly restricted to applied fields with magnitudes $< 0.5 E_c$.

SUMMARY AND CONCLUSIONS

The overall observations and conclusions of the present experiments are:

1. The PZT BM400 exhibits an asymmetrical polarization hysteresis loop that is displaced towards negative electric fields by an internal bias field, $E_i = -0.42$ MV/m. The coercive field is thereby increased, to give an E_c of 1.63 MV/m.
2. In common with previous findings for the hard piezoelectrics BM200 and BM800, the moderately hard piezoelectric BM400 does not exhibit field-induced crack growth in applied fields up to 3 MV/m, or $1.84 E_c$.
3. Within the accuracy of the experiments, no impedance changes, or resonance shifts, were observed in BM400 samples after the application of cyclic electric fields up to 3.0 MV/m.
4. It is concluded that a minimum field, directly related to the coercive field and indirectly related to the piezoelectric charge coefficient and mechanical compliance, is necessary in order to induce crack growth in piezoelectrics.
5. It is further concluded that, in the hard piezoelectrics BM200, BM400 and BM800, this minimum field is above the maximum safe operating field to prevent dielectric breakdown.

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