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# USE OF IMPEDANCE MEASUREMENTS FOR CRACK DETECTION IN MODERATERLY SOFT PIEZOELECTRIC CERAMICS

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# ABSTRACT

Cracks in a BM527 specimen exposed to 1,000 cycles of a low cycle electric field of 1.02 MV/m are qualitatively different from those in specimens similarly exposed to cyclic fields of 1.97 and 3.07 MV/m. These macrostructural defects cause significantly different changes to the resonance spectra of the piezoelectric, and the magnitude of the changes caused by a similar defect are related to its dimensions. These findings indicate that changes in resonance spectra can be effective as a nondestructive test, to determine different types and amounts of macrostructural defects in piezoelectric ceramics.

## **INTRODUCTION**

Measurable changes in the amplitude and frequency of resonance peaks have been observed in lead zirconate titanate (PZT) piezoelectrics that have been subjected to low cycle electric fields, that cause pre-existing cracks generated by a Vickers diamond hardness indenter to be extended in directions normal to the applied field [1]. These observations have indicated that impedance measurements can be used as non-destructive indicators of the presence of macrostructural flaws in piezoelectrics used for sensors and actuators. This potential application is explored by investigating changes in the resonance spectra of samples of BM527 (Navy Type V) PZT, after exposure to low cycle electric fields with various amplitudes, over a common period of 1,000 cycles. The viability and sensitivity of impedance measurements for

identifying macrostructural flaws in piezoelectric ceramics are examined by correlating the form and magnitude of changes in resonance peaks with associated field-induced crack extension and crack widening [2].

#### **EXPERIMENTAL METHODS**

Powder of BM527 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 10 s. 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 electrode faces, as illustrated previously [3].

For studying the effect of cyclic electric fields, the specimens were mounted in a specially designed jig [4], which was placed on the translational stage of an optical microscope. Low cycle electric fields, with a frequency of 5 Hz, and amplitudes of 1.02 MV/m (2.04 x  $E_c$ ), 1.97 MV/m (3.94 x  $E_c$ ) and 3.07 MV/m (6.14 x  $E_c$ ), were applied across the 1.27 mm separation between the electrode faces of the specimen, using a Trek 609E-6 high voltage amplifier, controlled by a computer via a Sensor Technology SS05 polarization meter [5]. To prevent possible arcing at the higher applied fields, the sample and contacts were immersed in insulating oil. After the application of 1,000 electric field cycles, changes in the length of pre-existing cracks oriented normal to the field were measured to an accuracy of  $\pm$  50 µm by traversing a cross hair in the objective lens from one end of the crack to another at a magnification of 40X. At the termination of the cyclic field experiments, the cracks were remeasured at magnifications of 160-400X, using the knife edges in the Vickers hardness microscope.

The impedance spectra of the PZT specimens mounted in the special jig described above were determined with an Agilent 4294A precision impedance analyzer, used in conjunction with the SS05 polarization system. The length thickness extensional (LTE) mode resonance was used for these impedance measurements, as it is the lowest frequency resonance dictated by the geometry of the present specimens.

#### **RESULTS AND DISCUSSION**

No visible extension of the pre-existing crack beyond its initial total length of 200  $\mu$ m was observed after exposure to field of 1.02 MV/m, at a frequency of 5 Hz for a period of 1,000 cycles. In this specimen, the crack in this specimen was in the form of hair lines than emanated from opposite corners of the Vickers diamond indentation. This observation is consistent with previous findings that low cycle field-induced crack growth does not occur below a threshold

field of the order of twice the coercive field,  $E_c$ , which is 0.5 MV/m in BM527 [2]. After exposure for 1,000 cycles in an increased field of 1.97 MV/m, or 3.94 times  $E_c$ , the initial crack emanating from the corners of the diamond indent was extended by 2,000 µm, i.e. by a factor of ten. In addition, the crack was broadened, so that it became clearly visible, in contrast to the hair line crack observed after exposure to the field close the threshold field for crack extension. After 1,000 cycles in an even greater field of 3.07 MV/m, or 6.14 times  $E_c$ , the initial crack was extended by 3,000 µm, to make it 15 times its original length. This crack showed a distinct opening between the edges, i.e. it was clearly increased in both width and length.

The LTE resonance spectra in Figure 1, which refers to a typical BM527 specimen in the as-indented condition, shows distinct peaks in impedance in the frequency regions of 0.12-0.13, 0.5-0.6, 1.6-2.0 and 7-15 MHz. As the peak to peak amplitude of the resonance peaks decreases exponentially with increasing frequency, the relatively shallow peaks observed at frequencies above 2 MHz were not included in the present investigation. In any event, resonance peaks that occur at these high frequencies are more susceptible to glitches caused by incident electric fields from fluorescent lighting and transient fields from the switching in nearby laboratory equipment.



Fig 1. The LTE resonance spectra of a typical BM527 specimen in the as-indented condition.

The effects of cyclic fields on resonance peaks in the frequency region of 0.12-0.13 MHz are shown in Figure 2. After 1,000 cycles in a field of 1.02 MV/m, the resonance peak shows a significant loss of intensity from 15-70 k $\Omega$  and a small but distinct displacement to a higher frequency (Figure 2A). When exposed to a higher field of 1.97 MV/m (Figure 2B), the reduction of the amplitude of the resonance peak is much smaller, i.e. from 15.5-113 k $\Omega$ , and



Fig 2. Resonance peaks in plots of impedance vs. frequency, in the region of 0.12-0.13 MHz
A. After 1,000 cycles in a field of 1.02 MV/m;
B. After 1,000 cycles in a field of 1.97 MV/m;
C. After 1,000 cycles in a field of 3.07 MV/m.

the displacement of the peak to a higher frequency is barely detectable. On further increasing the applied field to 3.07 MV/m (Figure 2C), the reduction of the resonance peak is 15.5-11.5  $k\Omega$ , which is slightly greater than the reduction in Figure 2B, while the peak displacement to higher frequencies is again barely detectable. Hence, the similar resonance peaks for the specimens subjected to the two higher electric fields in Figures 2B and 2C are distinctly different from the resonance peak in Figure 2A, which was obtained after exposure to a cyclic field close to the threshold amplitude for crack extension, which has been identified as approximately twice the coercive field,  $E_c$  [2].

The effect of cyclic fields on resonance peaks in the region of 0.5-0.6 Hz is shown in Figure 3. After 1,000 cycles in a field of 1.02 MV/m, the resonance peak shows a very significant loss of intensity from 7-3 k $\Omega$ , which is accompanied by a distinct displacement to a lower frequency (Figure 3A). After exposure to the higher field of 1.97 MV/m (Figure 3B), however, there was only a relatively small reduction in the amplitude of the resonance peak from 750-620  $\Omega$ , with a barely detectable shift to a lower frequency. A slightly greater reduction in resonance peak intensity, from 780-670  $\Omega$ , with a barely detectable displacement to a lower frequency, was observed after exposure to the higher field of 3.07 MV/m, as indicated in Figure 3C. Once again, the form of the peak change after exposure to the lower applied field differs significantly from the changes observed after exposure to the higher fields, and the changes that occur fields, and the changes that occur in the two higher two fields are similar in form, but different in magnitude.



Fig 3. Resonance peaks in plots of impedance vs. frequency, in the region of 0.5-0.6 MHz. A. After 1,000 cycles in a field of 1.02 MV/m; B. After 1,000 cycles in a field of 1.97 MV/m;

C. After 1,000 cycles in a field of 3.07 MV/m.

The effect of cyclic fields on the resonance peaks that occur in the vicinity of 1.2-2.0 Hz is shown in Figure 4. In this region of the resonance spectrum, the height of the resonance peak is decreased markedly, from 2200-500  $\Omega$  after 1,000 cycles in the near threshold field of 1.02 MV/m (Figure 4A). In contrast to the equivalent resonance peaks observed at lower frequencies, this peak is no longer sharp and its broad maximum is shifted from 1.85-1.75 MHz. A smaller decrease in peak amplitude, from 2.8-1.4 k $\Omega$ , with a similar peak displacement from 1.85-1.75 MHz, but without a loss of peak resolution, was observed after 1,000 cycles in a field of 1.97 MV/m (Figure 4B). A greater peak reduction of 3.4-1.2 k $\Omega$ , was observed after 1000 cycles in field of 3.07 MV/m, which was accompanied by an even greater peak shift from 1.8-1.70 MHz, with a small loss of resolution (Figure 4C).





- B. After 1,000 cycles in a field of 1.97 MV/m;
- C. After 1,000 cycles in a field of 3.07 MV/m.

The reproducibility of the plots in Figures 2, 3 and 4 was checked by examining two further specimens in a field of 1.02 MV/m, one other specimen at 1.97 V/m and two further specimens at 3.07 MV/m. All of these repeated results were practically indistinguishable from the respective plots shown in these figures. The present results thus demonstrate the essential viability of impedance measurements for identifying macrostructural defects in piezoelectrics, since different types of defect (hair line or well defined deep cracks) cause different types of changes to the amplitude and resolution of resonance peaks. The results also show that the sensitivity of impedance measurements for detecting defects depends on the selected resonance frequency, within a given resonance spectrum. The frequency range from 1.2-2.0 MHz is clearly the most sensitive for detecting the cracks induced in the present specimens, but other resonance frequencies may be found to be more sensitive for detecting other types of defects. Having demonstrated the inherent viability of impedance measurements for detecting and the present set of standard specimens in terms of a combination of amplitude changes and resonance frequency shifts, so that different types of defect can be uniquely identified and quantified.

While the prime purpose of this paper was to demonstrate the effectiveness of impedance measurements for detecting macrostructural flaws in piezoelectrics, it is of interest to comment on the changes to the pre-existing cracks that generated the observed changes in the resonance spectra in Figures 2, 3 and 4. Two types of cracks are known to emanate from the corners of a Vickers diamond indent in a brittle material. At relatively low applied loads, separate shallow elliptical cracks emanate from diagonally opposite corners of the indent. Since these surface cracks do not penetrate to the depth of the indent, they are not joined together by passing underneath the indent. They were first identified by Palmquist [6], and are usually referred to by his name. At higher applied loads, the so-called radial cracks that emanate from opposite corners of an indent are joined at a depth below the indent, and thus form a single semicircular crack that extends along the surface and into the body of the specimen. These radial cracks are used as a basis for determining the fracture toughness of brittle materials [7]. The hair line cracks generated in the present specimens, by a Vickers indent at the relatively light load of 20 N, are considered to be of the Palmquist type. This is consistent with observations of indentation cracks that only emanate from one set of diagonally opposite corners, when using the same applied load to generate Vickers indents in hard piezoelectrics [1]. Since the separate fine Palmquist cracks do not penetrate far below the surface, they have to be widened to a relatively large subtended angle, before easy extension can occur. The first action of the initial low amplitude fields will thus be to deepen the cracks so that, while no increase in length is observed at the surface, changes are nevertheless observed in the resonance spectra, as in Figures 2A, 3A and 4A. On increasing the amplitude of the applied field to well above the threshold limit, the cracks are deepened to a depth at which they can join below the indent, to form a radial crack with a significantly lower subtended angle at the surface, and thus be much easier to propagate in length and enlarge in width. The extension of these radial cracks causes a different type of change in the amplitude and frequency of the resonance peaks, and the magnitude of these changes can be correlated to the extension of the crack, as observed in Figures 2 B&C, 3 B&C and 4 B&C.

## SUMMARY AND CONCLUSIONS

The overall observations and conclusions of the present experiments are:

- 1. Cracks in a specimen exposed to 1,000 cycles of a field of 1.02 MV/m, at a frequency of 5 Hz, are qualitatively different from those in specimens similarly exposed to cyclic fields of 1.97 and 3.07 MV/m.
- 2. No change in surface crack length was observed after exposure to a 1,000 cycles in a field of 1.02 MV/m, but progressively increasing crack extensions, and crack widths, were observed after similar exposure to low cycle cyclic fields of 1.97 and 3.07 MV/m.
- 3. Changes in the magnitude and frequency of resonance peaks of a specimen exposed to a cyclic field of 1.02 MV/m differ significantly from those observed in specimens exposed to cyclic fields of 1.97 and 3.07 MV/m.
- 4. Similar types of change in the magnitude and frequency of resonance peaks were observed to increase progressively, after exposure to cyclic fields of 1.97 and 3.07 MV/m.

- 5. On the basis of these results, it is concluded that changes in resonance spectra can be used as a non-destructive test to determine different types and amounts of macrostructural defects in piezoelectric ceramics.
- 6. It is considered that the initial cracks generated by Vickers indentation in the present specimens of BM527, and subsequently exposed to low cycle fields of the order of twice the coercive field, are on the surface Palmquist type, while the wider and more extensive cracks observed after exposure to cyclic fields 3-6 times the coercive field are of the deep radial type, that are joined beneath the point of the indentation.

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