

REPORT

Reference Materials for measurements on piezoelectric ceramics

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REFERENCE MATERIALS FOR MEASUREMENTS ON PIEZOELECTRIC CERAMICS

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ABSTRACT

Three materials have been examined as potential reference materials for piezoelectric measurements, in particular for displacement-field measurements. Single crystal quartz has limited use as a highly stable resonance frequency generator. It was not possible to detect displacement-field loops on single crystal LiNbO_3 samples using a fibre optic displacement sensor, however this should be possible with higher resolution sensors. LiNbO_3 also has potential use as a reference material for dielectric displacement hysteresis measurements. Measurements on a fully dense hot pressed soft PZT material have shown that although repeatable measurements can be made the material could easily be damaged by mishandling.

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**Approved on behalf of Managing Director, NPL, by Dr M K Hossain,
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1 INTRODUCTION

The properties of piezoelectric materials are described by a multitude of constants and parameters, and there are a number of experimental methods involved in determining these¹. The methods described in the IEEE standard are measurements based on the low field excitation of these materials and these may be very different from the properties at the high fields seen in today's applications. The AM4 project of the DTI programme on Advanced Materials has addressed the problem of developing measurement methods at high fields, in particular measurement of displacement under high stress levels. Many of the measurements both high and low field are complex, and there is a potential for errors to occur. It is for this reason that reference materials are needed to check the consistency of results both within a laboratory over time, and also between laboratories.

Of course the use of traceably calibrated measurement equipment should mean that results concur, however the traceability of the equipment does not mean that whole experiment is traceable. For instance in the case of capacitance measurements the equipment is calibrated with a standard capacitor, which has soldered leads to make the contacts, whereas most piezoelectric ceramics are tested in the form of electroded discs in some sort of sample holder. The effect of this sample holding arrangement could be simply investigated by the use of a reference material.

The goal of a reference material therefore is to produce a behaviour similar to the materials under investigation, and this behaviour should either not change with time, temperature etc. or the change should be well defined and measurable.

2 MATERIALS

Three piezoelectric materials were examined for their potential use as reference materials. Single crystal quartz has already been identified as a potential reference material². The material is readily available and most of the properties are well defined and understood. It is a particularly stable material with respect to time and temperature and does not suffer from ageing as the poled electroceramics do. Single crystal X-cut disk shaped samples 10mm diameter, and thicknesses of 1mm and 0.2mm with evaporated gold electrodes were used in this study.

Lithium niobate, LiNbO_3 , is one of the few piezoelectric electroceramics readily available in the form of single crystals, indeed it is difficult to manufacture by traditional powder sintering methods. The lithium niobate used was in the form of slices of C-cut crystal, roughly 8mm square by 0.6mm thick with evaporated gold electrodes.

The final reference material examined was a hot pressed soft PZT. There is a feeling that much of the variability in properties, particularly at high stresses, is brought about by porosity in conventionally processed ceramics. Therefore the soft PZT disks 10mm diameter, 1mm thick were hot pressed to achieve a near 100% dense ceramic.

3 MEASUREMENTS

The measurements concentrated on assessing the potential of the materials as a reference material for displacement measurements. The simplest and most convenient method for performing displacement measurements on these materials is to use a fibre optic probe. The relative advantages and disadvantages of these systems have been discussed elsewhere³, and they are proving to be a popular choice for these measurements. However using this system it

proved impossible to measure displacement field curves for either of the single crystal materials.

Therefore the d_{33} coefficients for the three materials were measured using a low field Berlincourt type system in order to determine the magnitude of displacements that might be expected at high fields. The average measured d_{33} coefficients for the three materials are listed below, and they are in good agreement with values quoted in the literature.

	X Cut Quartz	C Cut LiNbO ₃	hot pressed soft PZT
$d_{33}/\text{pC/N-1}$	2.23	19.07	289

As the figures show there is at least an order of magnitude difference between each of the three materials and it is not surprising that there is not enough sensitivity in the measuring system to detect the displacement. The fibre optic probe system used has a sensitivity of $0.021\mu\text{m/mv}$ and the peak to peak noise was about $0.2\mu\text{m}$. The technology for the fibre optic systems is relatively new and higher sensitivity probes are available that might be able to detect the LiNbO₃ displacement. However for the quartz, a laser interferometer system would be needed to resolve the small displacements.

The fibre optic probe measurements on the hot pressed PZT was more practicable, and a series of measurements were conducted into the variability that might be expected. Figure 1 shows the displacement field curves for the same sample performed on two consecutive days. This shows the variability due to sample removal and replacement in the experimental set-up. The input voltage was nominally 2kV peak to peak @ 36Hz, across a 1mm thick 10mm diameter disk, giving a displacement of the order of $4\mu\text{m}$ peak to peak. The input voltage was chosen to give a high displacement output without undue damage to the sample, and the frequency was chosen off mains frequency to avoid mains interference. The match in output curves for the two tests is almost perfect.

Differing samples and sample holding conditions can adversely affect the results. The sample is held in place on a brass vacuum holder, and the ground electrode is either soldered or lightly spring loaded on to the top surface. It is not always necessary to have the vacuum pump in operation to hold the sample if the spring loading option is used, however figure 2 shows the difference between holding the sample by vacuum and by spring loading. The point spring loading system means the sample is not necessarily held flat against the bottom electrode, and uneven mechanical and electrical loading occurs. When the vacuum pump is turned on the sample is held flat against the vacuum electrode, and the curve shape is more symmetrical.

The PZT samples have been poled to give the required properties, and this can be seen in figure 3. Here the same sample is turned over in the measurement system, and the sign of the displacement changes with respect to the applied voltage. This demonstrates that the samples are going through minor hysteresis loops.

To examine the effect of accidental damage to the sample by applying higher voltages, the driving voltage was increased to 3kV peak to peak, and held for several minutes. Figure 4 shows the displacement-field behaviour changes with increasing time at this voltage, as the sample heats up due to dielectric losses. However after the sample has had time to cool down the behaviour has returned to the initial state (figure 5).

Figure 6 shows the effect of increasing damage on a sample, the input voltage was increased to such a level that breakdown of the air occurred near the ground contact, and the sample electrode was damaged near the point of contact. The shape of the hysteresis loop has changed permanently as the sample has been mishandled.

The variability from sample to sample is shown in figure 7, for four different samples. It is interesting to note that the samples seem to come from two different populations, however the batch numbers suggest that the variability occurs in one batch. Of course the absolute value of the reference material is not that important since it will always be independently measured. What is important is that the behaviour with respect to time, temperature and sample mishandling is consistent.

4 CONCLUSIONS

Of the three materials examined as a reference material for piezoelectric displacement measurements only the hot pressed PZT has any real potential. The material can easily be measured using a fibre optic probe system, however the high dielectric loss of the material means that at the voltage levels needed to give a reasonable displacement sample heating is likely. It might be possible to use the material if the temperature was monitored, however there is also the possibility that the sample could undergo a permanent change if accidentally overloaded. Again the material could still be used if this change could be detected by some other independent means such as change in capacitance or DC resistance.

Although it was not possible to detect the displacement of the LiNbO_3 using the present system, probes with increased resolution are available and should be able to detect the movement. However since it is easier to detect small changes in current or charge than mechanical displacement, the LiNbO_3 would also be a candidate as a dielectric displacement reference material.

For the quartz material there is little hope of its use as a mechanical displacement reference material. In addition although it is piezoelectric, it is not ferroelectric, therefore dielectric-field displacement measurements would only show capacitive current, and not the polarisation switching current seen in ferroelectric materials. The quartz materials potential use could be in resonance measurements where the high temperature stability provide stable resonance peaks.

5 ACKNOWLEDGEMENTS

This work was carried out within the DTI Programme on Advanced Materials.

6 REFERENCES

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2. J A Close and R Stevens, *Ferroelectrics*, 134, 181, (1992)
3. M Stewart, N J McCormick, and M G Gee, *Ferroelectrics*, In Press.

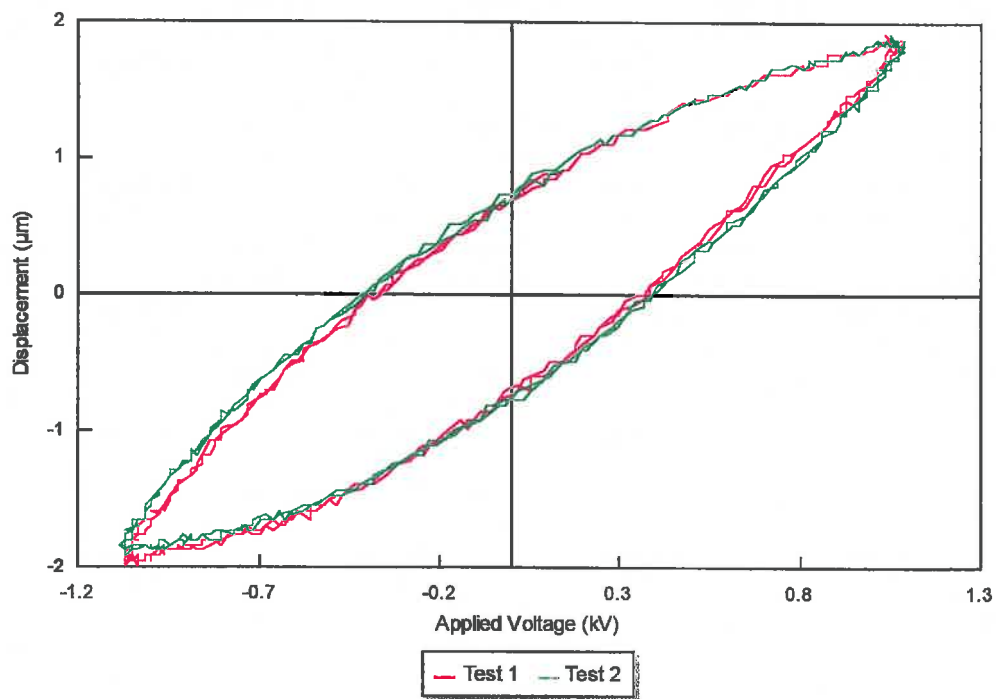


Figure 1. Displacement field loops on the same soft PZT sample, taken on two different days, removing and replacing the sample between tests.

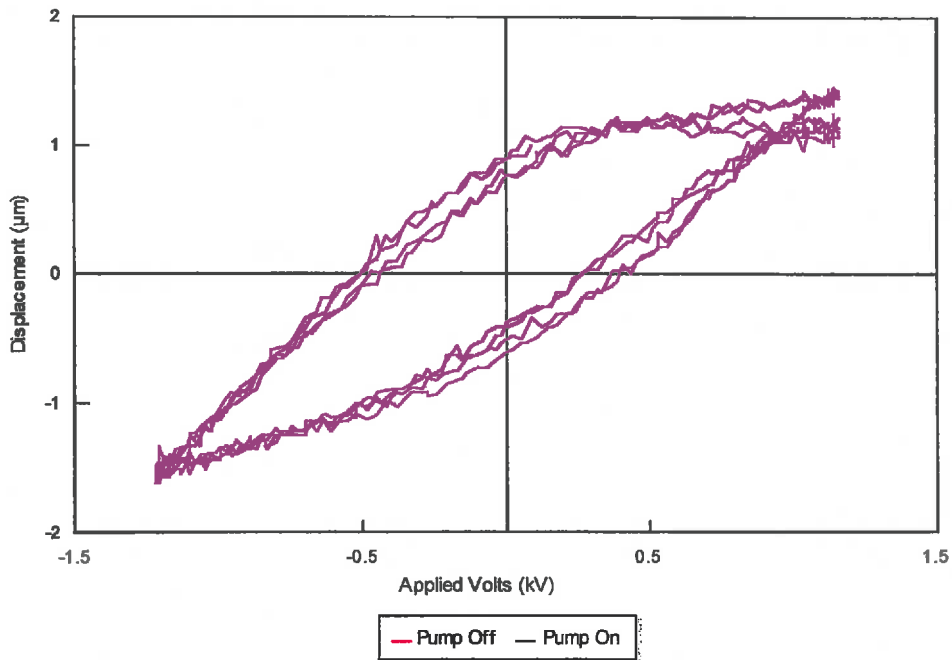


Figure 2. Displacement field loops on a soft PZT sample, showing the effect of sample holding without the vacuum pump switched on, and then with the vacuum pump turned on.

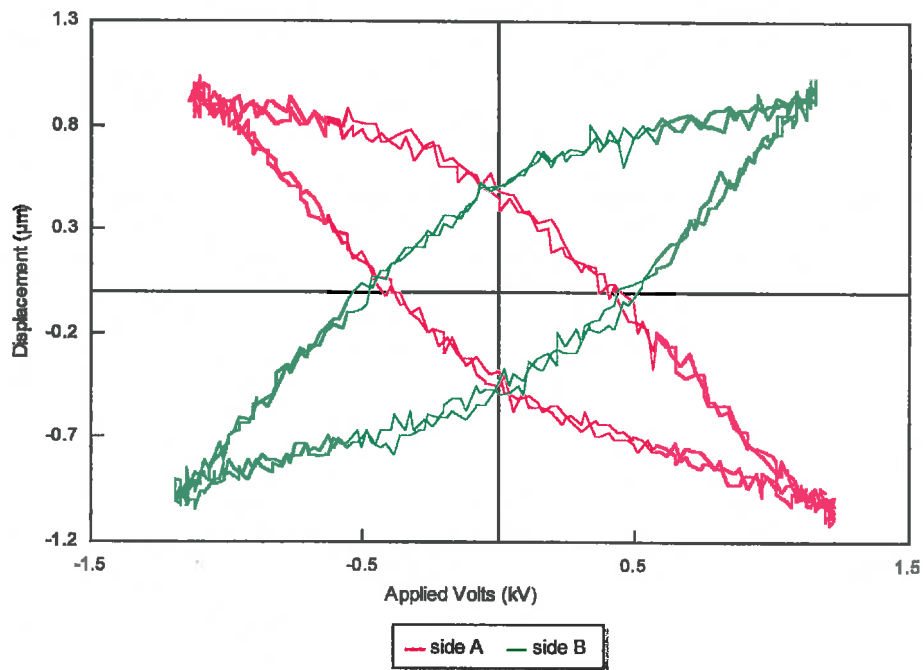


Figure 3. Displacement field loops on a soft PZT sample, showing the effect of turning the sample over in the measurement set-up.

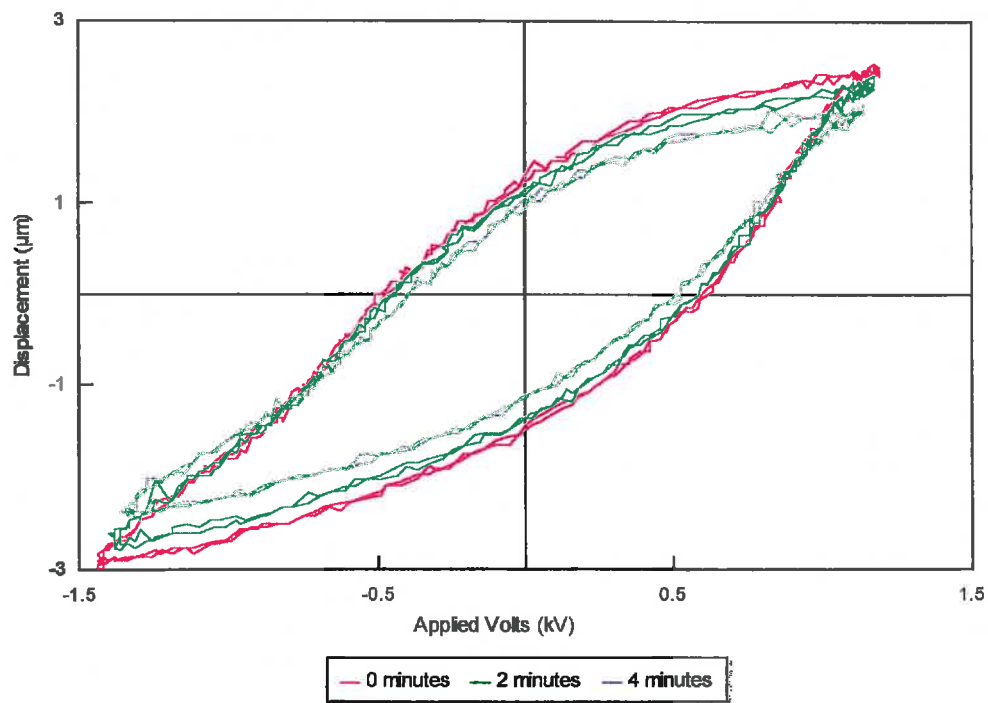


Figure 4. Displacement field loops on a soft PZT sample, with a higher than usual 3kV peak to peak load @ 36Hz applied for different lengths of time.

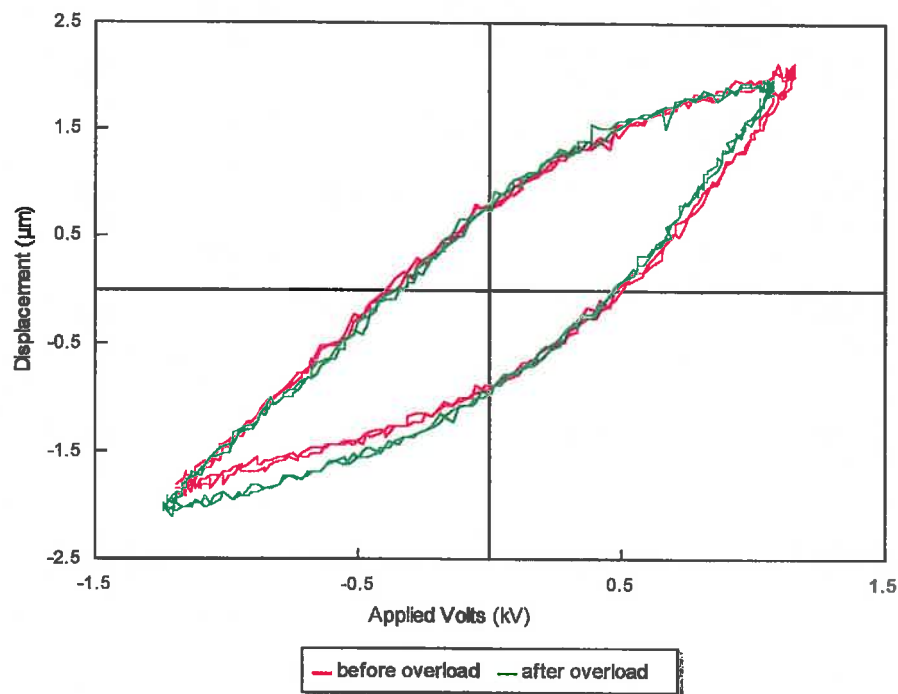


Figure 5. Displacement field loops on the same soft PZT sample from figure 4, before and after overloading with 3kV peak to peak @ 36Hz applied for 10 minutes.

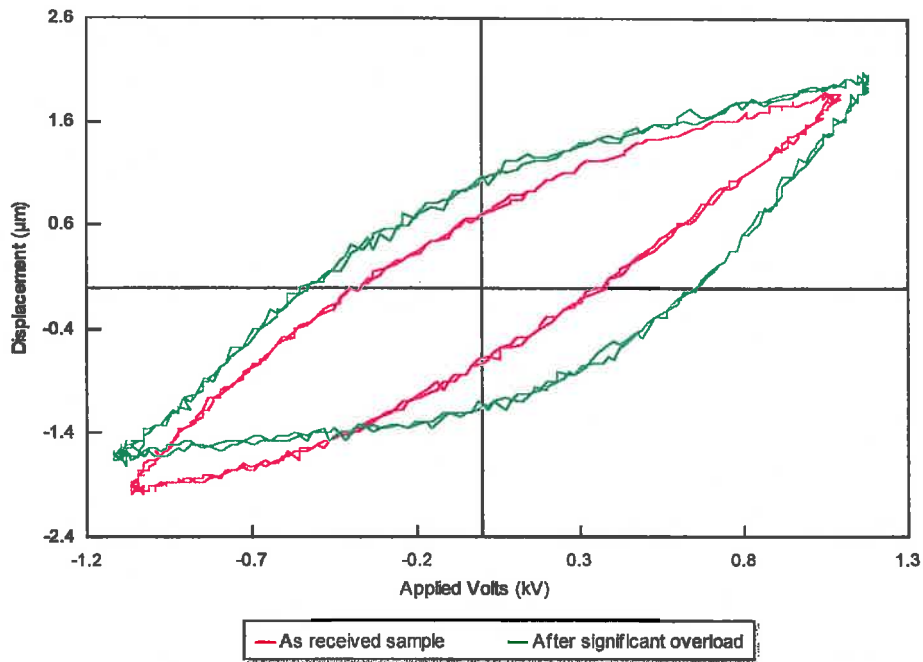


Figure 6. Displacement field loops on a soft PZT sample, before and after overloading with voltages up to 5kV peak to peak @ 36Hz. There were signs of damage to the electrode surface due to flashovers.

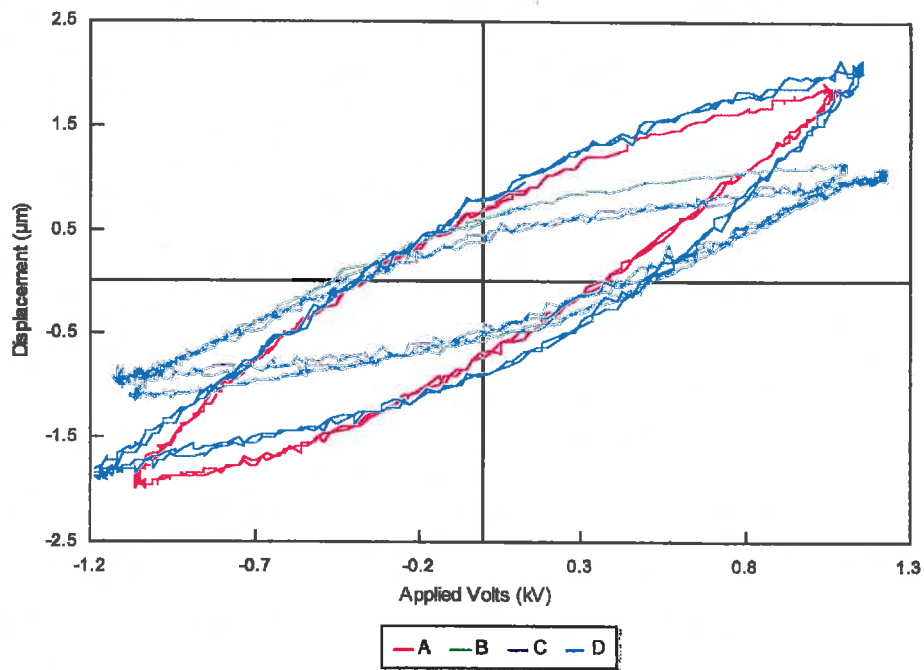


Figure 7. Displacement field loops on four different soft PZT samples. The samples would appear to come from two different populations, however the batch numbers do not support this.

