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Impact Loaded PZT Ceramics**

J A Close, R Stevens, M G Gee

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A Close*, R Stevens* and M G Gee
Division of Materials Metrology
National Physical Laboratory
Queens Road, Teddington,
Middlesex, TW11 0LW

ABSTRACT

Samples of commercially produced, poled lead zirconate titanate (PZT) piezoceramics were subject to longitudinal, compressive impact loading by a free falling striker providing a single, clean stress pulse. Samples were sandwiched in-between a Split Hopkinson Pressure Bar instrumented with miniature foil strain gauges linked to a custom built high frequency amplifier. Values of the piezoelectric charge coefficient, d_{33} were derived and compared with those obtained via the conventional low frequency (100 Hz), low stress resonance method.

The electromechanical coupling factor, k_t , was also derived by correlating the energies produced by the discharging piezoceramic and the stress wave that passed through the sample, these were then compared with values calculated from conventional resonance/anti-resonance measurements. The results obtained from the impact rig gave values for the d_{33} coefficient that are up to twice those gained from the resonance rig⁽¹⁾. It is also apparent that there exists a direct relationship between the impact velocity (and therefore the impact stress) and the piezoelectric charge coefficient of the material.

Ageing of the piezoelectric device under stress was studied by means of plotting d_{33} as a function of the number of impacts suffered. Samples of all the compositions displayed a drop in the values of d_{33} of between 18 and 49% when subject to 5000 impacts of approximately 40 MPa in magnitude.

A sample of X-cut α quartz was subjected to the same tests as the PZT compositions. The quartz showed potential as a reference material for measurements of the piezoelectric response of impact loaded PZT ceramics.

* Division of Ceramics, School of Materials, University of Leeds, Leeds LS2 9JT

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National Physical Laboratory
Teddington, Middlesex TW11 0LW, UK

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Approved on behalf of Chief Executive, NPL, by Dr M K Hossain
Head, Division of Materials Metrology

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INTRODUCTION

In recent years, there have arisen many new applications for piezoelectric devices; impact detonation devices requiring very rapid, large voltage pulses, high speed sensors and actuators such as those utilised in active automobile suspension systems and ignition devices for gas appliances and flash bulbs, to name but a few. In many of these applications, the piezoelectric device is subject to relatively large stresses of short duration, conditions to which conventional metrology techniques do not lend themselves. Currently, standard characterisation methods subject the piezoelectric device to cyclic stresses of very low magnitude and at low frequencies (commonly 100 Hz). Manufacturers data relating the performance of their materials are becoming less relevant as the materials are employed under conditions far removed from those under which they were characterised. It has therefore become necessary to develop a technique whereby piezoelectric device characterisation can take place under conditions relevant to today's demanding applications, but also a technique that in itself does not influence the results obtained. In doing this, new directions are set in the field of piezoelectric coefficient measurement and the limitations of these materials under immoderate conditions can be explored.

EXPERIMENTAL TECHNIQUE

The "drop weight" impact testing rig, illustrated below in Figure 1, comprises a free falling striker bar, running on an air bearing, provides a compressive impact that can be varied between 32 and 81 μ s in duration and of magnitude 2 to 40 MPa, by variation of the drop height. Seven different impact durations are provided by using striker bars of lengths 13 to 104 mm.

The strikers are constructed of 12 mm diameter EN24 steel rods with hardened hemispherical ends. EN24 steel is used since it possesses excellent hardenability and is very resistant to deformation under impact. The striker bar is housed in a perspex sabot that electrically insulates it from the surrounding rig and it also serves to prevent distortion of the ensuing stress wave by virtue of its low elastic modulus. The total mass of the striker bar and sabot is between 60 and 130 grammes, depending on the length of striker bar in use. The striker bar is held in the raised position by a small electromagnet mounted in the top of the rig and its release is initiated by the use of a micro-switch installed adjacent to the magnet.

The piezoelectric device under test (DUT) is situated in a vertically mounted, hardened EN24 steel Split Hopkinson Pressure Bar (SHPB) that consists of a top "impact" bar and a lower "receiver" bar that is 370 mm high in total and 12 mm in diameter. The SHPB arrangement is instrumented with four miniature foil strain gauges that monitor the passage of the impact induced stress wave through the receiver bar following the impact of the striker bar on the impact bar and its passage through the DUT. The strain gauge signal received from the SHPB is sent to a custom made high frequency (approximately 2 MHz bandwidth) virtual-earth pulse amplifier. The amplifier output is then appropriated for analysis by a computer controlled digital storage adaptor (DSA).

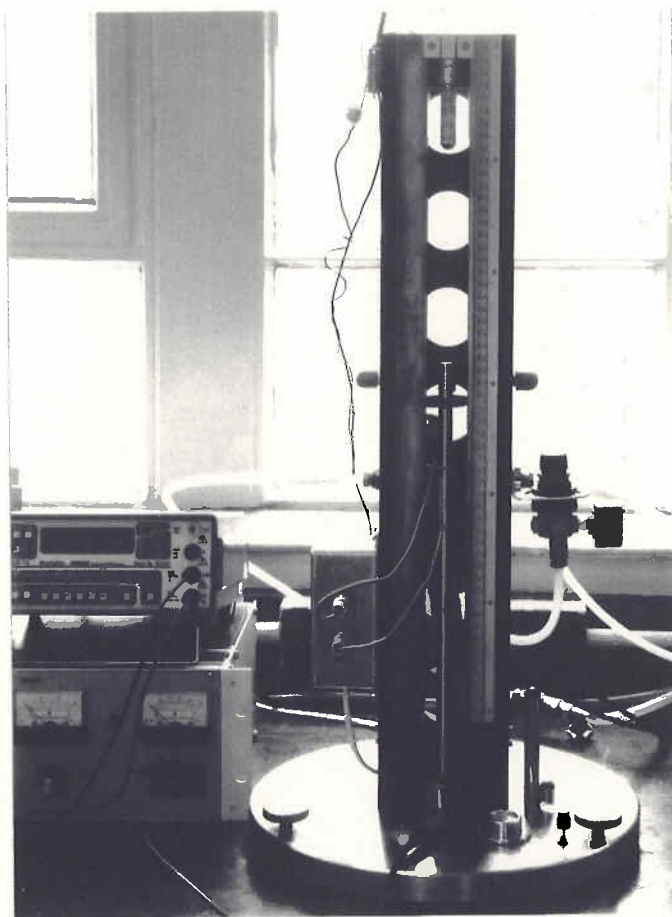
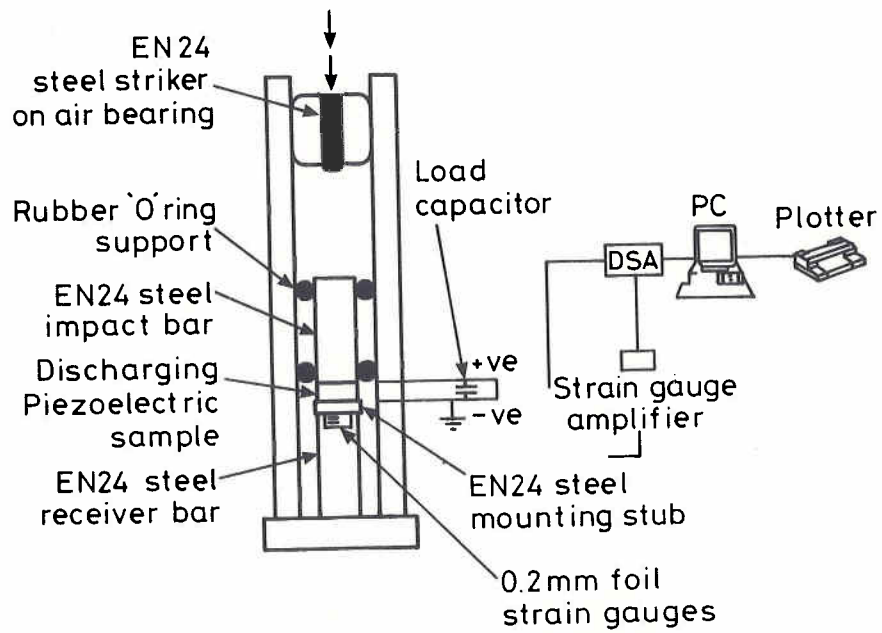


Figure 1. The Impact Testing Rig

Six composites of PZT were tested: PC4*, PC4A*, PC5*, PC5H*, Pz2b** and Pz27**. Two sizes (2 mm thick $\times$ 10 mm diameter and 5 mm thick $\times$ 10 mm diameter) of samples for each composition were tested. The DUT itself was mounted with the poling direction anti-parallel to the direction of wave propagation on a 12 mm diameter, threaded EN24 steel stub by means of a fine layer of silver loaded, conducting epoxy resin. When screwed into the top of the receiver bar, it served as the negative electrode of the sample. The positive electrode of the DUT was made by attaching a small fillet of aluminium foil, 20 μ m thick to the top of the device, again by means of silver loaded epoxy resin. The top surface of the DUT and the face of the impact bar in contact with it were sprayed with a coating of P.T.F.E lubricant to prevent "barrelling" of the sample and to ease the transmission of the stress wave across the interface. In order to maintain a parallel mounting between stub and DUT, which is imperative if micro-cracking in the sample is to be avoided, the mounting process was carried out with the assistance of a microscope slide mounting press.

The electrical discharge from the DUT (this would be of the order of 1-2 kV under open circuit conditions) was transferred onto a load capacitor set in parallel with the DUT that is approximately 1000 times greater in capacitance than the DUT. This arrangement means that the piezoceramic was discharging under short circuit conditions, as is the convention for measurement of the charge coefficient. The exact magnitude of the capacitance required for these conditions to be valid was determined by experimentation: If a significant field exists across the sample whilst it is under stress, a build up of charge on the sample faces serves to maintain the state of polarisation of the ferroelectric domains and therefore by definition, impede the generation of current flow from the piezoelectric device. As a result of this, a falsely low value of the piezoelectric charge coefficient would be recorded. The charge held on the load capacitor is then read into the DSA, simultaneously with the signal from the strain gauge amplifier.

As a control experiment, results taken from the PZT compositions were compared with those from a sample of X-cut α quartz. The quartz gives an accurate reference in that the quartz exhibits the piezoelectric effect by virtue of its asymmetrical crystal structure and not as a result of being exposed to an external poling field. The quartz is not therefore prone to the same depolarising mechanisms ie domain re-orientation, as the ferroelectric materials. The X-cut of quartz is selected as this is the only orientation which will produce an electric current parallel to the applied stress as is the requirement for derivation of the d_{33} coefficient.

INTERPRETATION OF RESULTS

From the captured data, it is possible to correlate both the amplitude and duration of the piezoelectric output from the DUT with that of the stress wave passing through it. A calculation can therefore be made of the piezoelectric charge coefficient d_{33} and also the efficiency of conversion of input mechanical energy into output electrical energy as described by k_t , the coupling coefficient, so that:

* Supplied by Morgan Matrol, Unilater Division, Raubon, Wrexham, Clwyd

** Supplied by Ferroperm, Piezoelectric Ceramic Division, Heijreskovvej 6, Kirstgard, Denmark

$$d_{33} = \left(\frac{dD}{dX} \right)_{E,T}$$

and

$$k_t^2 = \frac{(\text{output electrical energy})}{(\text{input mechanical energy})}$$

where D is the dielectric displacement, X is the stress, the subscripts E and T denote constant (zero) field and constant temperature respectively; whilst the subscripts 33 indicate that direction of the applied stress and that of the current flow from the DUT respectively (conventionally 3 is taken as the polarisation direction in the ceramic device); t denotes the thickness mode.

The coupling coefficient, k_t , is derived by equating the electrical energy held on the capacitor in parallel with the sample ($\frac{1}{2}CV^2$, where C is the value of the capacitive load and V is the voltage across it), with the energy contained in the strain wave that gave rise to the transient electrical signal (found by integrating the area under the strain/time curve). For each composition, two sample thicknesses (2 mm and 5 mm) were tested with a view to verifying suspected thickness effects arising from stress wave reflections within the DUT. An examination of the stress wave and charge/discharge curve was made in order to quantify the finite time taken for the onset of charge flow after the application of stress to the DUT ie the "relaxation time" due to domain mobility effects. Scanning electron microscopy techniques was carried out to determine if there was any microcracking present that would lead to short-circuiting within the DUT and a consequential drop in electrical output.

UNDERLYING THEORIES

The theories presented below are based on the assumption that the system obeys the laws of linear elasticity such that that initial and final body configurations are identical. The duration of the impact is dependent on the time taken for an (acoustic) stress wave, travelling at a speed C_o (where C_o is the **longitudinal** acoustic wave velocity in the material equal to the square root of the material's Young modulus, E , divided by it's mass density, ρ), to traverse the striker bar twice^[2] at which time the tensile release wave extricates the striker bar from the top of the impact bar. The rise time of the stress wave is governed by the geometry of the striker bar facing. A hemispherical facing produces a wave with an almost sinusoidal rise in stress, whilst a flat faced striker will give rise to a wave that in theory should have an essentially instantaneous rise in stress to its maximum value. However, in practice, due to minor misalignments and unevenness of the surfaces between the striker and impact bar, distorted and un-reproducible waves commonly occur. When the impact is served by a hemispherical striker bar, the stress wave originates from point source, producing a radial wave, but as this propagates down an impact bar of specific aspect ratio, the stress wave assumes a planar, uniaxial profile^[3] (this assumption cannot be made when impacting the sample directly).

As already noted, the duration of the stress pulse is dependant on the striker geometry, but the pulse length is intrinsically dependant on the value of C_o in the material used for the SHPB. This means that it is extremely important to construct a SHPB of the correct length

given the value of C_o for its constituent material, in order to avoid the convolution of incident and reflected stress waves that would lead to gross errors in the stress measurements. The stress wave passes through the DUT with minimal reflection at the interfaces and is transmitted into the receiver bar. The DUT is actually 2 mm smaller in diameter than impact and receiver bars for two reasons. Firstly, this configuration negates "edge effects" that arise from surface (Rayleigh) waves travelling down the bars creating distorted stress waves at the bar edges close to the sample and secondly, it allows for ease of sample location in the rig in that slight errors of the piezoceramic positioning on the stub are of little consequence. From the signal picked up by the strain gauges on the receiver bar, a measure of the stress put upon the DUT can be derived. The axial stress on the DUT, X_s , is directly related to the engineering strain, x_r , measured in the receiver bar and is obtained from the balance of the total force between the sample and the receiver bar^[4], thus

$$X_s = \left(\frac{A_r}{A_s} \right) E_r x_r$$

where A_s is the area of the cross section of the sample and A_r is that of the receiver bar whose Young modulus is E_r . The value of x_r is derived from a function involving the peak voltage of the recorded strain wave, the current in the strain gauges, the strain gauge factor and the degree of amplification provided.

It should be noted that the DUT itself cannot be strain gauged directly due to extremely adverse effects arising from capacitive coupling between the discharging piezoceramic and the strain gauge filaments.

RESULTS

Before any results were taken from piezoceramic samples, an experiment was conducted using a single EN24 steel bar, instrumented with ten pairs of equidistantly spaced strain gauges. This served four purposes:

- 1) The stress wave velocity, C_o , was determined by timing its passage past each pair of strain gauges,
- 2) The duration of the stress wave induced by each of the seven different striker bar lengths was accurately measured,
- 3) The length of the impact and receiver bars and the position of the strain gauges on them required to avoid convolution of incident and reflected waves was resolved,
- 4) By using the ten sets of two independently wired strain gauges at precisely known positions on the bar, it was possible to monitor differential behaviour (ie distortion) of the stress wave across the bar as propagation progressed until such time as it had travelled far enough to assume a planar profile.

With this information, the necessary minimum length of impact bar could be determined. The results of these experiments were as follows:

- 1) The stress wave velocity C_0 was found to be 5104 ms^{-1} , which is over 99% in accordance with the longitudinal stress wave velocity calculated from the material's Young modulus and mass density (5122 ms^{-1}). This result indicates that the stress wave is uniaxial and planar as required.
- 2) The duration of the stress waves induced by the seven striker bars are displayed in Table 1.

Table 1. Duration of stress waves induced by striker bars of different lengths

Striker Bar Length, mm	13.1	19.6	26.1	39.2	52.2	78.3	104.4
Impact Duration, μs	32	39	45	56	63	73	81

- 3) The impact bar and the receiver bar should be greater than 172 mm long with the strain gauges at least the same distance from the base of the receiver bar to avoid superposition of the stress waves.
- 4) The impact bar must be at least 196 mm in length for the propagating stress wave to become planar in profile.

Measurements were made of the d_{33} piezoelectric charge coefficient on 2 mm thick samples of each material using the conventional low frequency (100 Hz), low stress method. These data appear in Table 2, together with the value quoted in the manufacturers data tables.

Table 2. Comparison of measured and quoted values of d_{33} for PZT samples

Material Type	Pz-26	Pz-27	PC4	PC4A	PC5	PC5H	Quartz
Quoted d_{33} , pC N^{-1}	275	375	287	296	409	529	2.27
Measured d_{33} , pC N^{-1}	249	398	217	229	340	486	2.52

Values of the electromechanical coupling factor, k_{33} were derived from mechanical resonance and anti-resonance measurements taken on the Hewlett Packard 4194A Impedance/Gain phase analyser, using the following formula^[5]:

$$k_t^2 = \frac{\pi}{2} \frac{f_r}{f_a} \tan\left(\frac{\pi}{2} \frac{\Delta f}{f_a}\right)$$

where f_r and f_a are the resonant and anti-resonant frequencies respectively and Δf is the difference between the two. It is evident that the results shown in Table 3 are substantially lower than those suggested in the manufacturers data. This is a result of the relatively small aspect ratio of the samples ($10 \text{ mm}/2 \text{ mm} = 5$) which gives rise to many interacting modes of vibration in the DUT, leading to low efficiency and hence low k_{33} values. The

electromechanical coupling factor under impact loading has yet to be calculated.

Table 3. Comparison of measured and quoted values of k_{33} from resonance measurements

Material Type	Pz-26	Pz-27	PC4	PC4A	PC5	PC5H	Quartz
Quoted k_{33}	0.65	0.69	0.71	0.68	0.67	0.68	< 0.1
Measured k_{33}	0.28	0.39	0.27	0.28	0.33	0.35	0.04

IMPACT MEASUREMENT RESULTS

In view of the limited space available in this report, the results that appear below are taken from only two of the six PZT compositions along with those of the quartz reference. The two materials selected, Pz-27 and PC5H give a good representation of the results that can be expected from a "soft" and "hard" ferroelectric material respectively. An example of the data acquired by the DSA from the SHPB is shown in Figure 2.

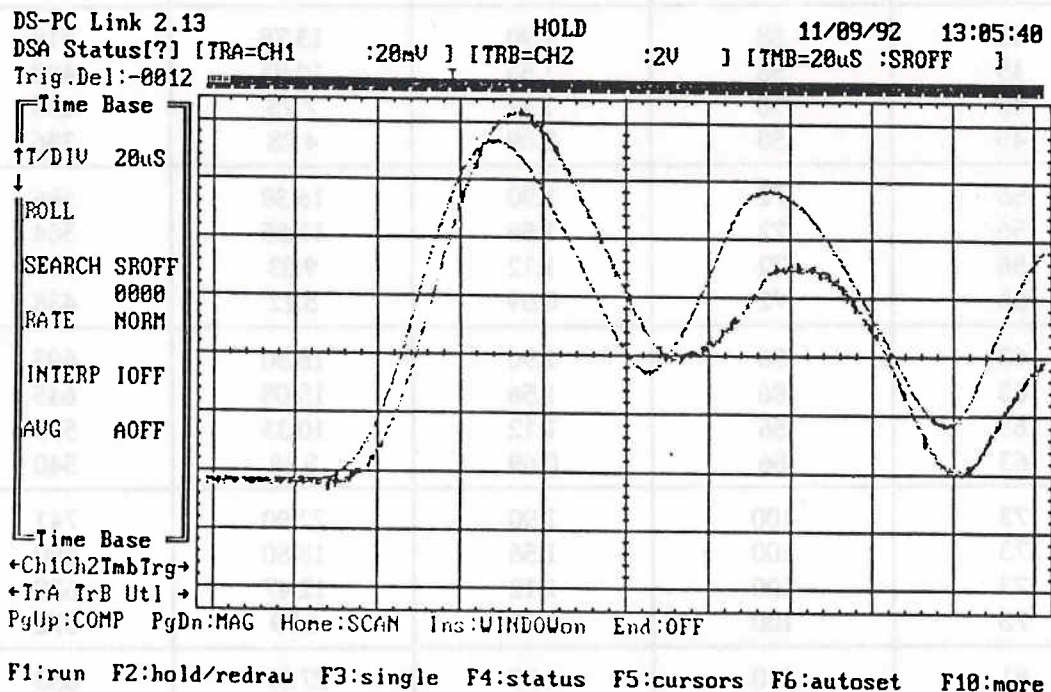


Figure 2. DSA Trace for X-cut α Quartz

The sample tested to give the results in Figure 2 was a piece of 2 mm thick quartz, subject to an impact of 81 μ s. The upper curve is that of strain in the receiver bar whilst the lower is that of the electrical output from the DUT. The evident time difference of approximately 6 μ s between the onset of the stress wave is due to the time taken for it to reach the strain gauge, approximately 30 mm remote from the DUT, on the receiver bar. Both PZT

compositions were subject to a capacitive load of 463 nF whilst the quartz sample required a load of only 92 nF. The results are reported in Tables 4-6.

Table 4. Results of impact measurements for FerroPerm Pz-27

T_i is the impact duration, T_d is the duration of the piezoelectric discharge from the DUT, V_i the velocity of the striker bar at the instant of impact. X_s is the stress imposed on the DUT, and d_{33} was described earlier.

T_i μ s	T_d μ s	V_i ms ⁻¹	X_s MPa	d_{33} pCn ⁻¹
32	46	1.90	8.84	373
32	46	1.56	6.89	350
32	46	1.12	4.93	323
32	46	0.69	2.94	285
39	52	1.90	11.93	442
39	52	1.56	9.68	414
39	52	1.12	6.61	375
39	52	0.69	3.80	326
45	58	1.90	13.78	518
45	58	1.56	10.98	488
45	58	1.12	7.73	435
45	58	0.69	4.28	386
56	72	1.90	16.39	586
56	72	1.56	12.85	564
56	72	1.12	9.03	509
56	72	0.69	5.12	438
63	86	1.90	18.80	693
63	86	1.56	15.08	645
63	86	1.12	10.33	599
63	86	0.69	5.68	540
73	100	1.90	22.90	741
73	100	1.56	18.80	690
73	100	1.12	12.47	629
73	100	0.69	6.79	572
81	110	1.90	27.37	653
81	110	1.56	21.79	595
81	110	1.12	13.78	548
81	110	0.69	7.54	485

Table 5. Results of impact measurements for PC5H

Parameters are described in caption to Table 4.

T_i μ s	T_d μ s	V_i ms^{-1}	X_s MPa	d_{33} pCN^{-1}
32	48	1.90	11.36	509
32	48	1.56	8.84	500
32	48	1.12	5.87	483
32	48	0.69	3.02	469
39	56	1.90	16.57	563
39	56	1.56	13.03	543
39	56	1.12	8.45	530
39	56	0.69	4.37	513
45	60	1.90	20.30	605
45	60	1.56	15.45	592
45	60	1.12	9.68	585
45	60	0.69	4.93	562
56	72	1.90	23.83	683
56	72	1.56	18.44	675
56	72	1.12	11.73	654
56	72	0.69	6.05	634
63	82	1.90	29.42	702
63	82	1.56	23.09	677
63	82	1.12	14.71	662
63	82	0.69	7.26	642
73	97	1.90	32.40	770
73	97	1.56	26.07	731
73	97	1.12	17.50	701
73	97	0.69	9.50	683
81	102	1.90	37.24	786
81	102	1.56	29.42	748
81	102	1.12	19.74	712
81	102	0.69	10.56	698

Table 6. Results of impact measurements for X-cut α Quartz

Parameters are described in caption to Table 4.

T_i μ s	T_d μ s	V_i ms^{-1}	X_s MPa	d_{33} pCN^{-1}
32	47	1.90	7.17	2.58
32	47	1.56	5.68	2.64
32	47	1.12	3.91	2.58
32	47	0.69	2.20	2.62
39	51	1.90	10.06	2.74
39	51	1.56	8.10	2.74
39	51	1.12	5.49	2.73
39	51	0.69	3.13	2.73
45	55	1.90	12.85	2.75
45	55	1.56	10.42	2.76
45	55	1.12	6.98	2.78
45	55	0.69	3.94	2.82
56	68	1.90	16.01	2.99
56	68	1.56	13.22	2.94
56	68	1.12	8.84	3.03
56	68	0.69	4.73	3.03
63	79	1.90	20.20	3.00
63	79	1.56	15.64	3.08
63	79	1.12	10.52	3.09
63	79	0.69	5.77	3.10
73	106	1.90	24.58	3.08
73	106	1.56	19.55	3.14
73	106	1.12	12.29	3.27
73	106	0.69	7.17	3.25
81	112	1.90	30.17	3.09
81	112	1.56	22.53	3.18
81	112	1.12	14.71	3.28
81	112	0.69	8.10	3.34

As can be seen from the plots of d_{33} as a function of V_i (Figures 3,4,5) the PZT piezoceramics exhibit charge coefficients substantially higher than those taken under low frequency, low stress resonance conditions and they show an essentially linear increase in the value of d_{33} with increasing impact velocity (and therefore stress). The "hard" PC5H material exhibits a much lower variation in the value of the charge coefficient with impact velocity than does the "soft" Pz-27 material. It should be noted that when the Pz-27 piezoceramic was impacted with the largest and most massive striker bar, there was a substantial drop in the recorded readings of d_{33} . Subsequent readings re-taken for the striker bars of lower mass all displayed a similar drop in d_{33} values of approximately 20%. This result is indicative of the fact that

the onset of depolarisation had begun in the DUT at this relatively low stress magnitude.

It should also be noted that the duration of the piezoelectric discharge from the samples is approximately 30-40% greater than the impact time. In contrast to the PZT materials, the quartz proved to have a relatively static response under varying impact velocities. An interesting observation from the quartz samples was that the recorded stress waves were much "cleaner" than those taken from the ferroelectric materials. An explanation for this is that the stress wave is subject to distortion by the reorienting domain structure of the ceramic.

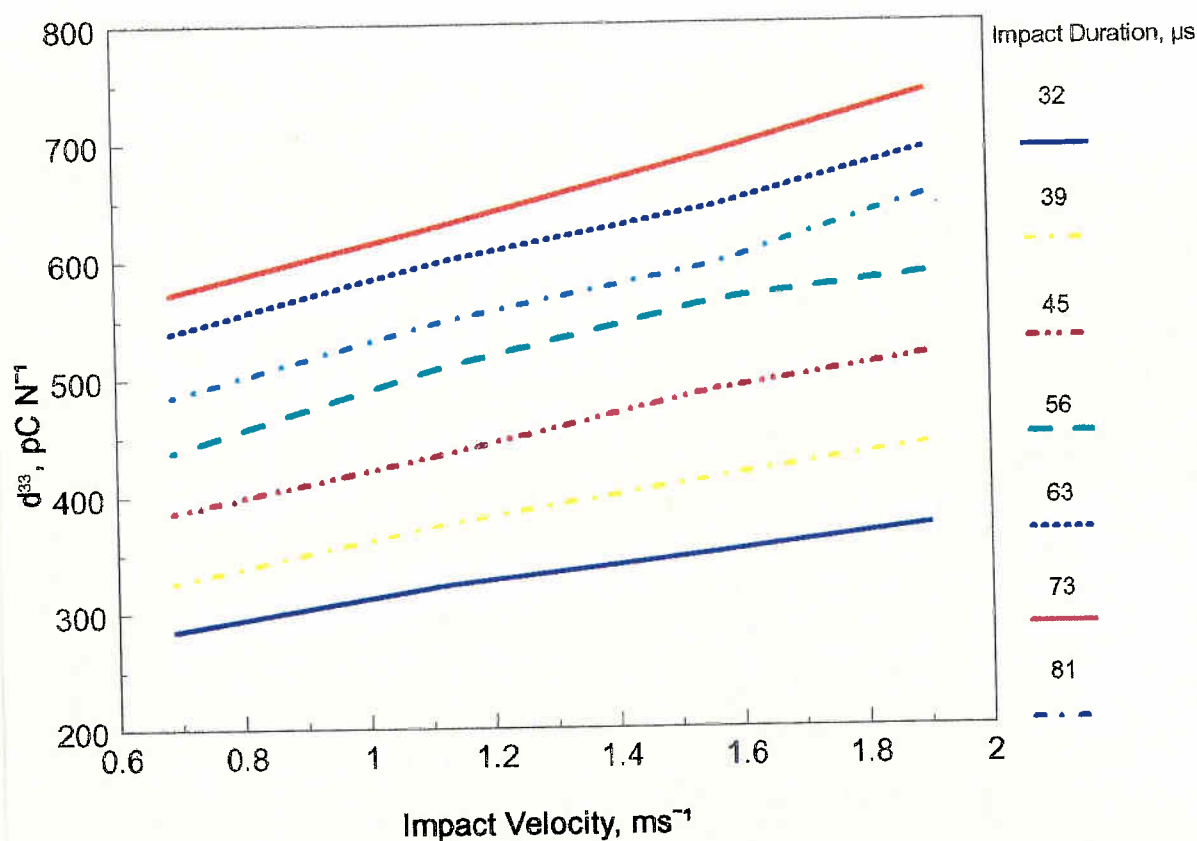
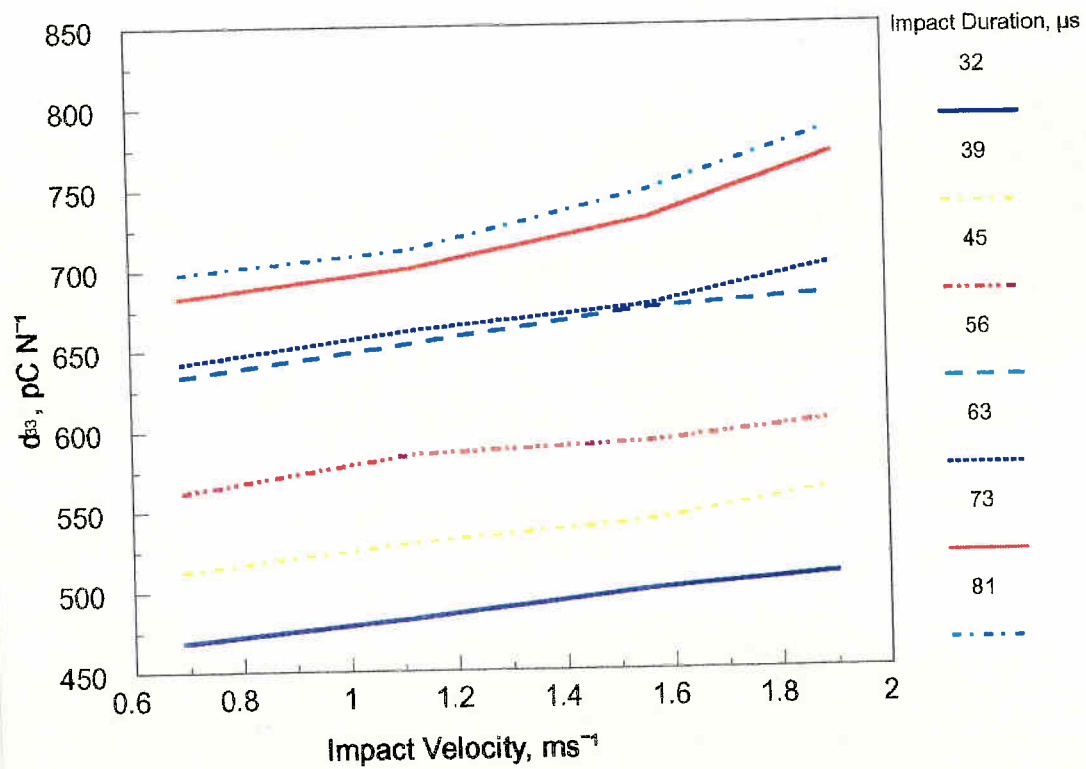
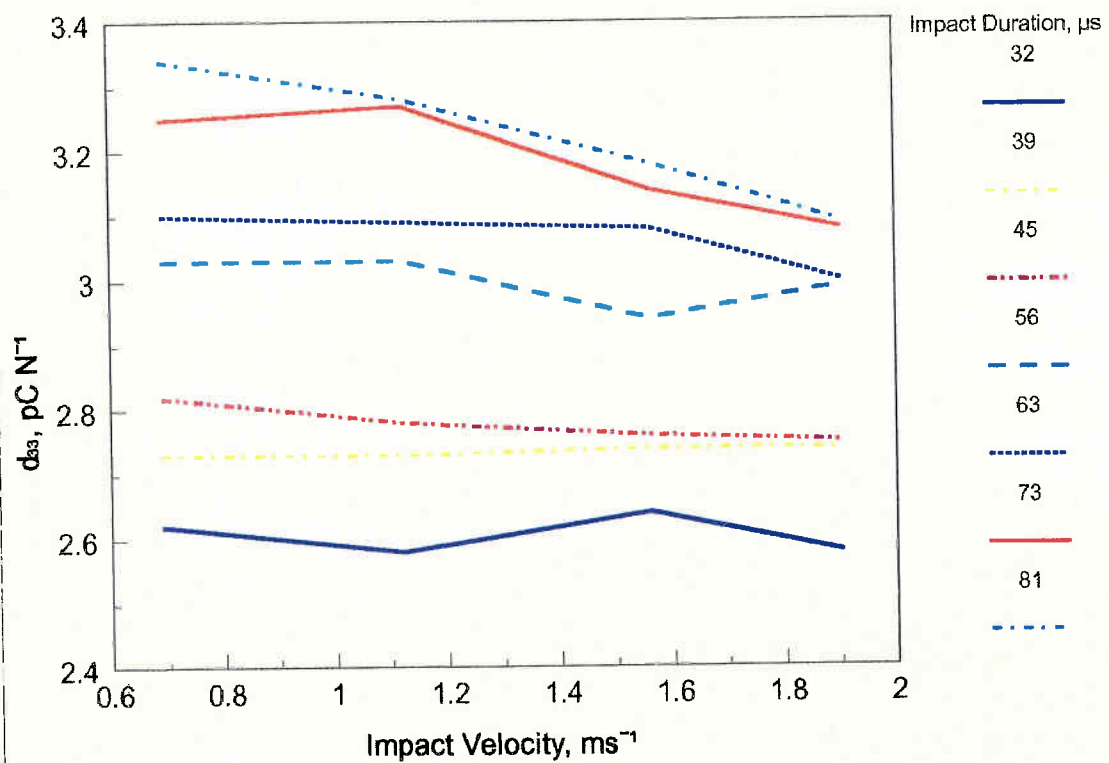


Figure 3. d_{33} as a Function of Impact Velocity for Pz-27

Figure 4. d_{33} as a Function of Impact Velocity for PC5HFigure 5. d_{33} as a Function of Impact Velocity for X-cut α Quartz

For impact repetition tests, only 2 mm thick samples were used as these were expected to have a greater likelihood of microcracking. The samples were subject to the maximum possible stress provided by the impact rig by using the largest striker bar (81 μ s impact duration) and dropping it from the maximum height available, giving rise to an impact stress of approximately 40 MPa for each DUT. Table 7 shows the percentage drop in the value of the piezoelectric charge coefficient, d_{33} for all six PZT compositions and the quartz sample following 5000 impacts.

It should be noted that although the striker bar could easily be caught after each impact to prevent further uncharted impacts, reflected compressive stress waves within the SHPB lead to the DUT being subjected to a small number of further stress cycles after the initial recorded impact.

Table 7. Drop in d_{33} with 5000 impacts for different compositions

Material	Pz-26	Pz-27	PC4	PC4A	PC5	PC5H	Quartz
% drop in d_{33}	19	45	30	36	18	22	2

It is evident from these results and Figures 6 and 7, that all the ferroelectric materials show a distinct and in some cases, extremely large drop in their piezoelectric properties under these conditions (their output voltage drops and there is a slight increase in the measured stress), though there is a tendency for the initially large decrease to flatten off.

These data are in accordance with the behaviour that can be expected from ferroelectrically hard and soft materials, a point that is currently being verified by coercivity measurements on the piezoceramics. The degree of fluctuation in the d_{33} value for quartz at only 2% is almost at the limit of the resolution of the DSA and the average recorded value of 2.54 pC/N⁻¹ is less than 1% greater than the value recorded by the conventional resonance technique. This result is of great significance in that it clearly demonstrates the accuracy and reproducibility of results taken on the impact rig and it also demonstrates that the phenomena of high d_{33} values and the tendency to depolarise in the ferroelectric materials is not a consequence of non-linearity and inaccuracy in the testing rig.

MICROSTRUCTURAL ANALYSIS

Microcracking induced in the test specimens by the impacting process will adversely affect the electrical output from the device as dielectric breakdown occurs across the fissures. It is therefore important that the stress levels resulting in the loss of sample integrity are determined. High localised stresses arising from direct impact by mis-aligned projectiles or which have uneven faces, are a major cause of microcracking which can be alleviated by introducing a smooth, carefully aligned facing in front of the sample receiving the impact, this problem being overcome by the use of the SHPB. It was also evident from early experiments that the alignment of the DUT on its mounting stub was extremely important if it were to remain intact. Figures 8 and 9 show two scanning electron micrographs of the degree of microcracking present in sample of PC5, polished to a 1 μ m finish. In this

particular extreme case, it is evident that the majority of cracking was initiated in or adjacent to areas of porosity or contamination, already present in the sample. Staining with lead nitrate solution has facilitated the detection of microcracks.

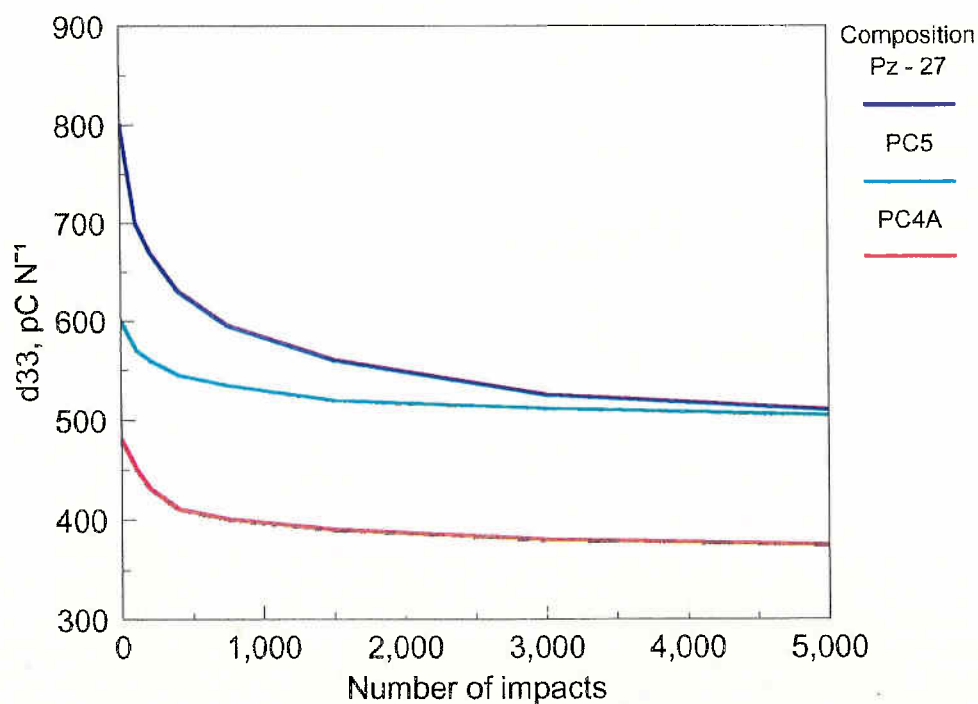


Figure 6. d_{33} as a Function of Impact Repetition: All PZT Compositions

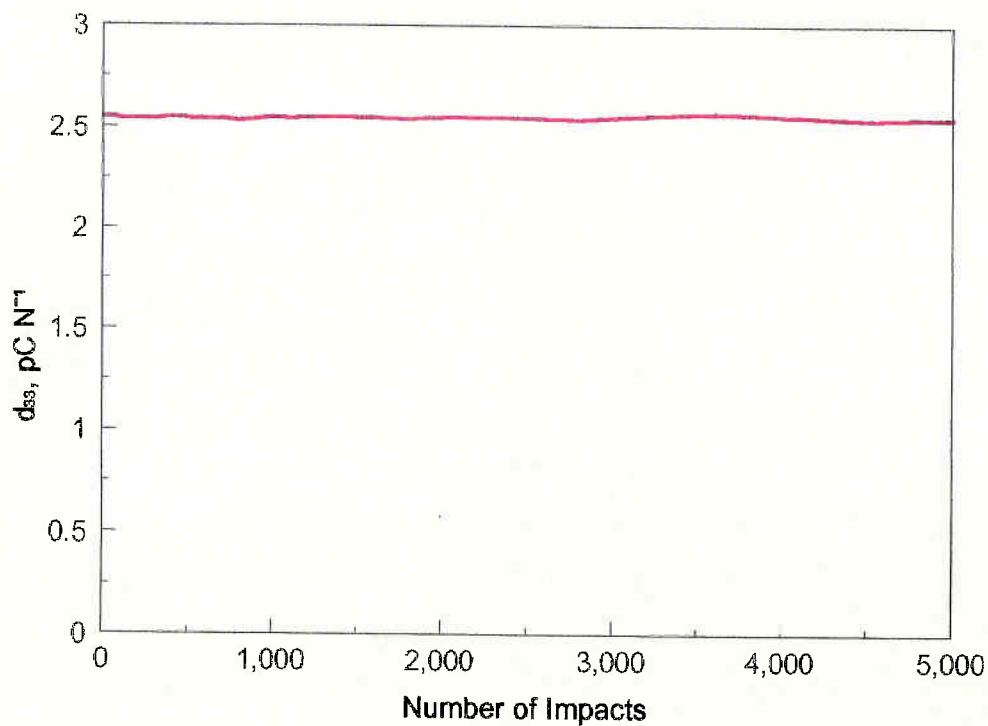


Figure 7. d_{33} as a Function of Impact Repetition: X-cut α Quartz

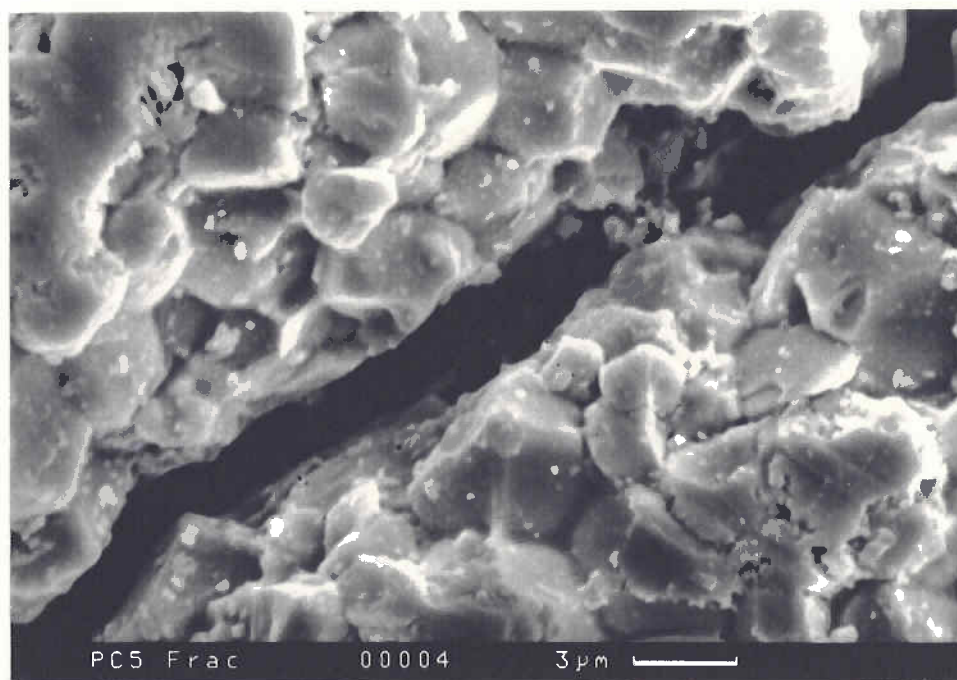


Figure 8. A Typical Fissure Induced by the Impacting Process

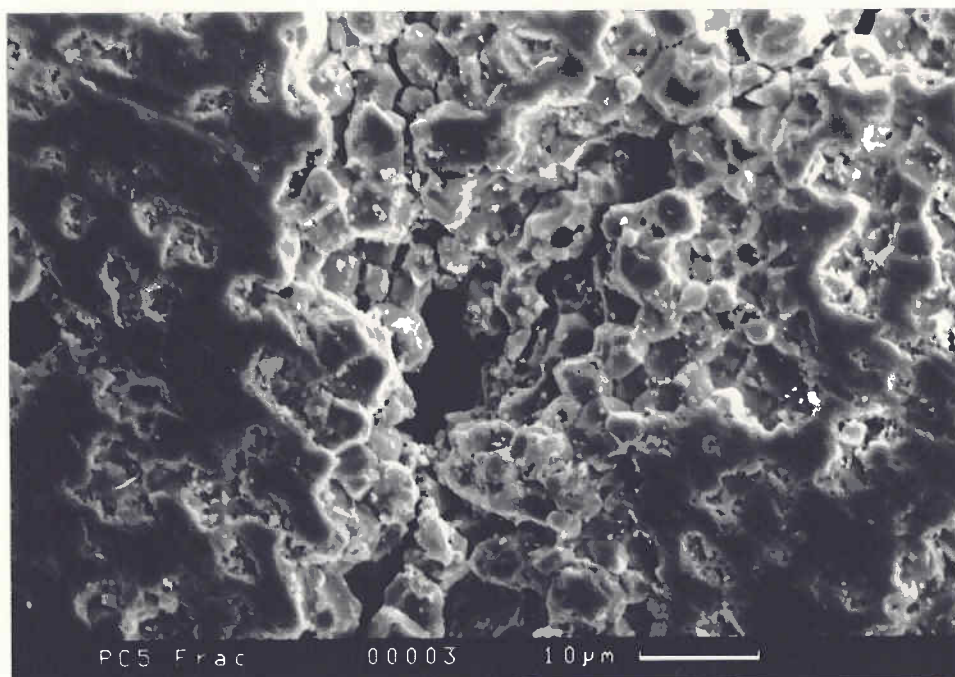


Figure 9. Poor Sample Integrity Leads to Numerous Microcracks

DISCUSSION

The SHB impact testing system has proved to be a practical, repeatable and robust method of making piezoelectric measurements under impact loading conditions. A key to the success of the test method was the development of a strain gauge amplifier of sufficient bandwidth to allow accurate measurement of the stress waves in the bar, and thus the stress wave transmitted into the specimen, by means of readily available strain gauges. This measurement technology may well also have other applications in other mechanical tests where rapid changes in stress and strain are encountered.

The results of the impact tests on the PZT samples show that the charge coefficient d_{33} increases with both the duration and velocity of impact. In addition, the charge coefficient measured in impact tests is always significantly higher than the corresponding value measured in low frequency, low stress resonance conditions. This indicates that the piezoelectric parameters measured by conventional low frequency and low stress methods cannot be used to determine how piezoelectric ceramics will behave under impact conditions. The reasons for the increase in charge coefficient are likely to be related to the mobility of the ferroelectric domains in the materials (its coercivity). This is dependent on the chemical composition and degree of doping in the material.

It also appears that it takes a finite time for the piezoelectric materials to respond to rapid stress pulses, as the electrical discharge times from the samples were always greater than the duration of the impact wave that generated it. This suggests that it would be profitable to study the change in charge coefficient d_{33} and coupling coefficients with frequency by direct measurements of displacement of samples under alternating fields at different frequencies. This information is not normally obtained by either low frequency measurements where there is likely to be little effect of frequency, or by resonance experiments where the piezoelectric parameters are obtained from an analysis of the frequencies of resonance and antiresonance which will be single valued for a particular sample size and material, and thus would be difficult to use in the measurement of frequency dependence of the piezoelectric response.

The drop in charge coefficient as PZT samples were subjected to repeated impacts shows that under high stress conditions there is a major effect of fatigue on piezoelectric properties. The rapid initial drop suggests that an exponential drop in charge coefficient is occurring, and in fact good fits to the data are obtained for exponential curves (illustrated by the straight lines of the experimental points when plotted on a logarithmic time basis, Figure 10). This is similar to the well known exponential aging of piezoelectric materials, and the mechanism, which is thought to be partial depoling of the material, is likely to be similar.

There are considerable practical difficulties in extending the number of impacts to any appreciable extent with the current test system, since it relies on manual replacement of the striker at the top of the impact tester between impacts. It may be possible to automate the impact tester for tests with a higher number of impacts, and the potential for this will be explored.

Another alternative is to carry out more conventional mechanical fatigue of the PZT samples in a mechanical testing system which would also give more control over the stress. This would mean that fatigue tests could be performed for extended periods at relatively low stress levels. These test conditions are likely to be relevant to a wide range of applications for

piezoelectric materials such as actuators and sensors where the materials are subjected to moderately high stress levels, but where information on the long term piezoelectric response of the material is currently lacking.

By contrast to the PZT samples, the X-cut quartz showed a linear response with only a small change in charge coefficient with stress. This is because, unlike the piezoelectric materials which have been given a piezoelectric response by poling, quartz is naturally piezoelectric so that the orientation of the electrical dipole that gives the piezoelectric response to quartz is intrinsic to the crystal, and can only be altered by recrystallisation or severe mechanical damage. This gives quartz potential as a reference material for the calibration of piezoelectric response at high frequencies and under fatigue conditions, but before this goal could be realised it would be necessary to carry out a careful programme of measurements to determine the stability of quartz as a reference material.

The damage that was caused to some of the samples subjected to impact testing show that it is important that these materials have a high microstructural integrity and are free from manufacturing defects if catastrophic failure is to be avoided. This is not only because flaws in the sample act as stress concentrations in a conventional way, but also because flaws are liable to cause internal stress wave reflections which lead to very high stress gradients and a likelihood of crack initiation and propagation.

CONCLUSION

From the results presented above, the following observations can be made:

- 1) The piezoelectric response ie the charge coefficient of the ferroelectric materials, does not show a static value when subject to variations in stress, as one might conclude from conventional, resonant characterisation methods.
- 2) X-cut α quartz shows potential as a reference material for calibrating test systems for the deterioration of the piezoelectric response of ceramics under high stresses.
- 3) It is possible to obtain extremely high values of d_{33} and therefore high output voltages from the ferroelectric materials, particularly those of a "soft" nature, though under repetitive impacting there is a rapid diminution of this phenomena and the charge coefficient tends towards a value more in accordance with that taken under conditions of low stress and low frequency.
- 4) Piezoelectric materials subjected to impact conditions must have high microstructural integrity and be free from manufacturing defects if catastrophic failure is to be avoided.

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