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CHARACTERISATION OF PC5K HIGH STRAIN PZT CERAMIC AND ESC1 ELECTROSTRICTIVE CERAMIC MADE BY MORGAN MATROC LTD. \(UNILATOR DIVISION\), UK

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CHARACTERISATION OF PC5K HIGH STRAIN PZT CERAMIC AND ESC1 ELECTROSTRICTIVE CERAMIC MADE BY MORGAN MATROC LTD. (UNILATOR DIVISION), UK

S. Sherrit, R.B. Stimpson, H.D. Wiederick, and B.K. Mukherjee

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Abstract

This report presents our characterisation of PC5K high strain lead zirconate titanate (PZT) ceramic and ESC1 electrostrictive ceramic samples manufactured by Morgan Matroc Ltd. (Unilator Division) of the UK. The radial and thickness extensional mode resonances of the PZT material have been analysed to derive the relevant material constants and the hydrostatic properties of the material have been evaluated. The dielectric properties of the electrostrictive material have been determined. The direct strain-voltage relationship has been studied in the case of both materials.

Résumé

Dans ce rapport, nous présentons notre caractérisation du zirconate titanate du plomb(PZT) PC5K et de la céramique électrostrictive ESC1 développé par la compagnie Morgan Matroc Ltée (Division Unilator) du Royaume Uni. Pour la céramique PZT nous avons analysé les résonances dans le mode radial et le mode en épaisseur pour trouver les constantes caractéristiques et nous avons évalué les propriétés hydrostatiques. Les propriétés diélectriques de la céramique électrostrictive ont été déterminé. La dépendance de la déformaton sur la tension appliquée à été étudié pour les deux matériaux.

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Introduction:

In September 1995 we received samples of two types of transducer materials from Morgan Matroc (Unilator Division) in the UK. The materials were:

PC5K: A high-strain lead zirconate titanate (PZT) piezoelectric ceramic composition, and
ESC1: An electrostrictive ceramic composition.

This report presents our characterisation of the above materials.

Samples:

We received sixteen electroded disc samples of the PC5K high-strain PZT and ten electroded disc samples of the electrostrictive ceramic ESC1. The discs had three nominal thicknesses: 0.5 mm, 1.0 mm and 1.5 mm, and they were all approximately one inch in diameter. Unfortunately the 0.5 mm. electrostrictive samples arrived damaged: one had a broken edge held by an electrode and the other was almost broken in two. Table 1 shows the mass, the dimensions and the density for each sample. The samples are identified by their nominal thickness as a prefix and by the suffix "pzt" for the high-strain PZT and the suffix "ele" for the electrostrictive material. In addition, samples of the same thickness are designated numerically for the PZT specimens and alphabetically for the electrostrictive specimens. The diameters marked by asterisks represent values measured by holding two broken pieces together.

Impedance Measurements:

PC5K HIGH STRAIN PZT:

The geometries of the PZT specimens were suitable for analysing their radial and thickness extensional modes of resonance. The experimental resonance data was obtained using a Hewlett Packard Model 9194A Impedance Analyser. The thickness extensional mode resonance

Table 1: Samples received from Morgan Matroc Ltd.

Sample Name	Diameter (m)	thickness (m)	mass (kg)	density (kg/m^3)
.05mm1pzt	0.024900	0.000495	0.001887	7828
.05mm2pzt	0.024910	0.000518	0.001990	7883
.05mm3pzt	0.024920	0.000510	0.001964	7896
.05mm4pzt	0.024900	0.000508	0.001942	7850
.05mm5pzt	0.024870	0.000500	0.001943	7999
1mm1pzt	0.024900	0.001010	0.003883	7895
1mm2pzt	0.024900	0.001010	0.003856	7840
1mm3pzt	0.024915	0.001010	0.003893	7906
1mm4pzt	0.024890	0.001030	0.003925	7832
1mm5pzt	0.024910	0.001030	0.003963	7895
2mm1pzt	0.024880	0.002030	0.007826	7930
2mm2pzt	0.024950	0.002030	0.007851	7910
2mm3pzt	0.024910	0.002040	0.007872	7918
2mm4pzt	0.024920	0.002040	0.007889	7929
2mm5pzt	0.024900	0.002010	0.007704	7871
2mm6pzt	0.024930	0.002000	0.007727	7915
.05mmaele	0.024930	0.000540	0.001987	7538
.05mmb1ele	*0.024920*	0.000530	0.001204	7538
.05mmb2ele	*0.024920*	0.000530	0.000778	7538
1mmaele	0.024940	0.001050	0.003891	7586
1mmbele	0.024930	0.001060	0.003924	7584
1mmcele	0.024930	0.001030	0.003844	7646
1mmdele	0.024940	0.001020	0.003783	7592
2mmaele	0.024960	0.002040	0.007616	7630
2mmbele	0.024930	0.002030	0.007543	7612
2mmcele	0.024950	0.002030	0.007554	7611
2mmdele	0.024980	0.002040	0.007597	7599

data was analysed using Smits' method[1] to determine the complex values of the elastic compliance c_{33}^{D}, the dielectric constant ε_{33}^{S}, the piezoelectric coefficient e_{33} and the thickness mode coupling coefficient k_t. Average values of these material constants are shown in Tables 2 to 4 for the three different thicknesses and an example of the fit to the resonance data is shown in Figure 1.

The radial mode resonance data was analysed using Sherrit et al's method[2] to determine the elastic compliances s_{11}^{E} and s_{12}^{E}, the dielectric constant ε_{33}^{T}, the piezoelectric voltage coefficient d_{13}, the planar electromechanical coupling constant k_p and the Poisson's ratio σ_P. Average values of these material constants are shown in Tables 5 to 7 for the three different thicknesses and an example of the fit to the resonance data is shown in Figure 2.

Both Figures 1 and 2 show that the material constants derived at the fundamental resonance frequency do not fit the data at higher frequencies which shows the presence of dispersion in the material. Thus Figure 2 shows dispersion from around the sixth radial resonance frequency which was typical for the 0.5 mm samples. The 1.0 mm. thick samples showed dispersion from around the third resonance frequency and the 2.0 mm. thick samples showed dispersion even from the second resonance frequency. It should be pointed out that, depending on the frequency used in a given application, any of the higher order resonances may be analysed in order to obtain the material constants at the corresponding frequencies.

Table 2: Thickness mode results for 0.5 mm samples. Results are an average of 5 samples.

Constant	Real	Imaginary
c_{33}^{D} (N / m^2)	1.58 x 10^{11}	1.9 x 10^{9}
ε_{33}^{S} (F / m)	1.69 x 10^{-8}	-1.4 x 10^{-9}
e_{33} (C/m^2)	24.6	-1.6
k_t (#)	0.476	-0.014

Table 3: Thickness mode results for 1.0 mm samples. Results are an average of 5 samples.

Constant	Real	Imaginary
c_{33}^{D} (N/m^2)	1.58×10^{11}	2.5×10^{9}
$\varepsilon_{33}^{S} (F/m)$	1.93×10^{-8}	-6.5×10^{-10}
e_{33} (C/m^2)	28.0	-0.061
k_t (#)	0.517	-0.0056

Table 4: Thickness mode results for 2.0 mm samples. Results are an average of 6 samples.

Constant	Real	Imaginary
c_{33}^{D} (N/m^2)	1.57×10^{11}	1.3×10^{9}
$\varepsilon_{33}^{S} (F/m)$	1.96×10^{-8}	-5.7×10^{-10}
e_{33} (C/m^2)	27.1	-.084
k_t (#)	0.489	-0.0098

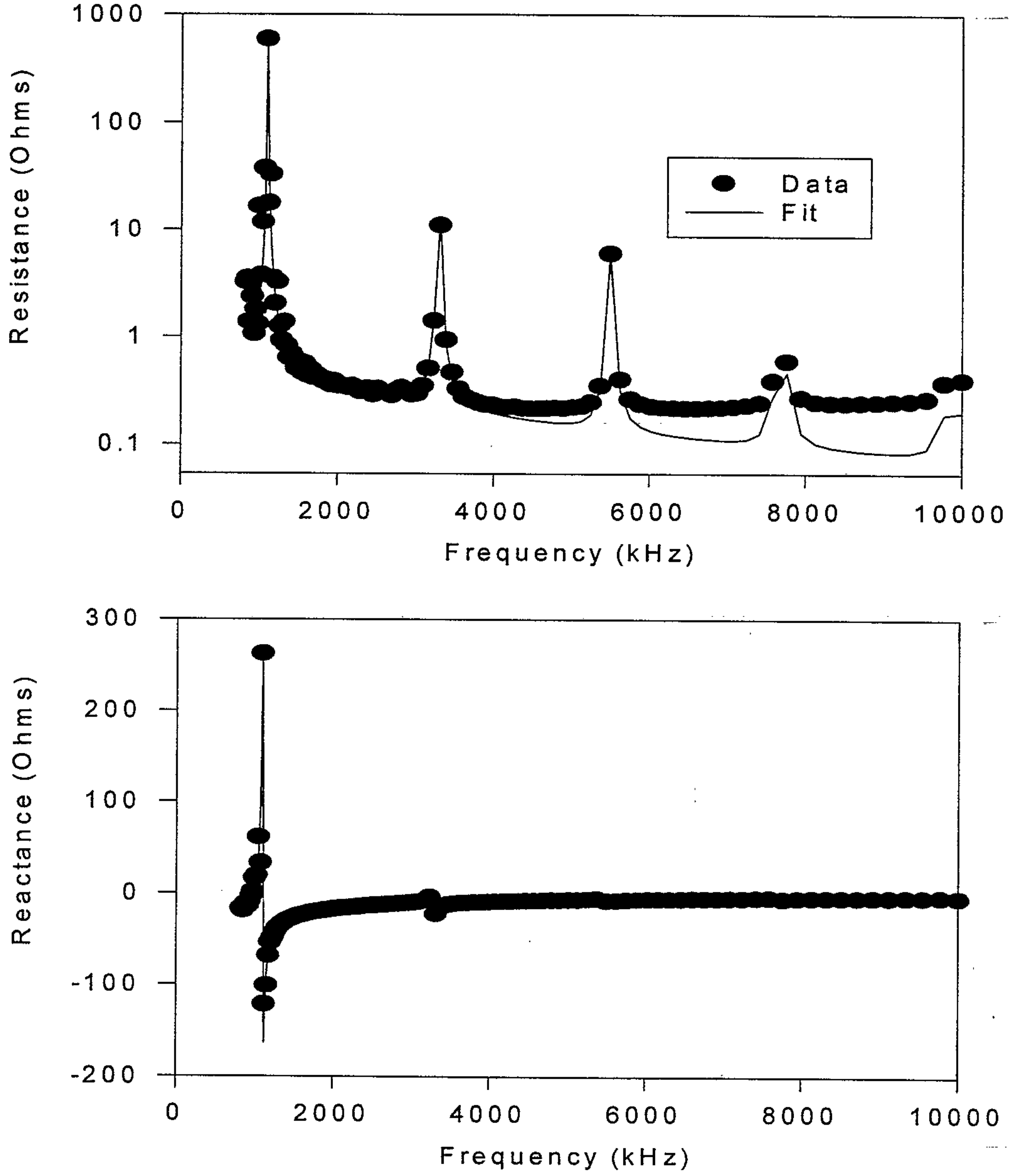

Figure 1: The thickness mode impedance spectra for a 2.0 mm thick high-strain PZT sample produced by Morgan Matroc.

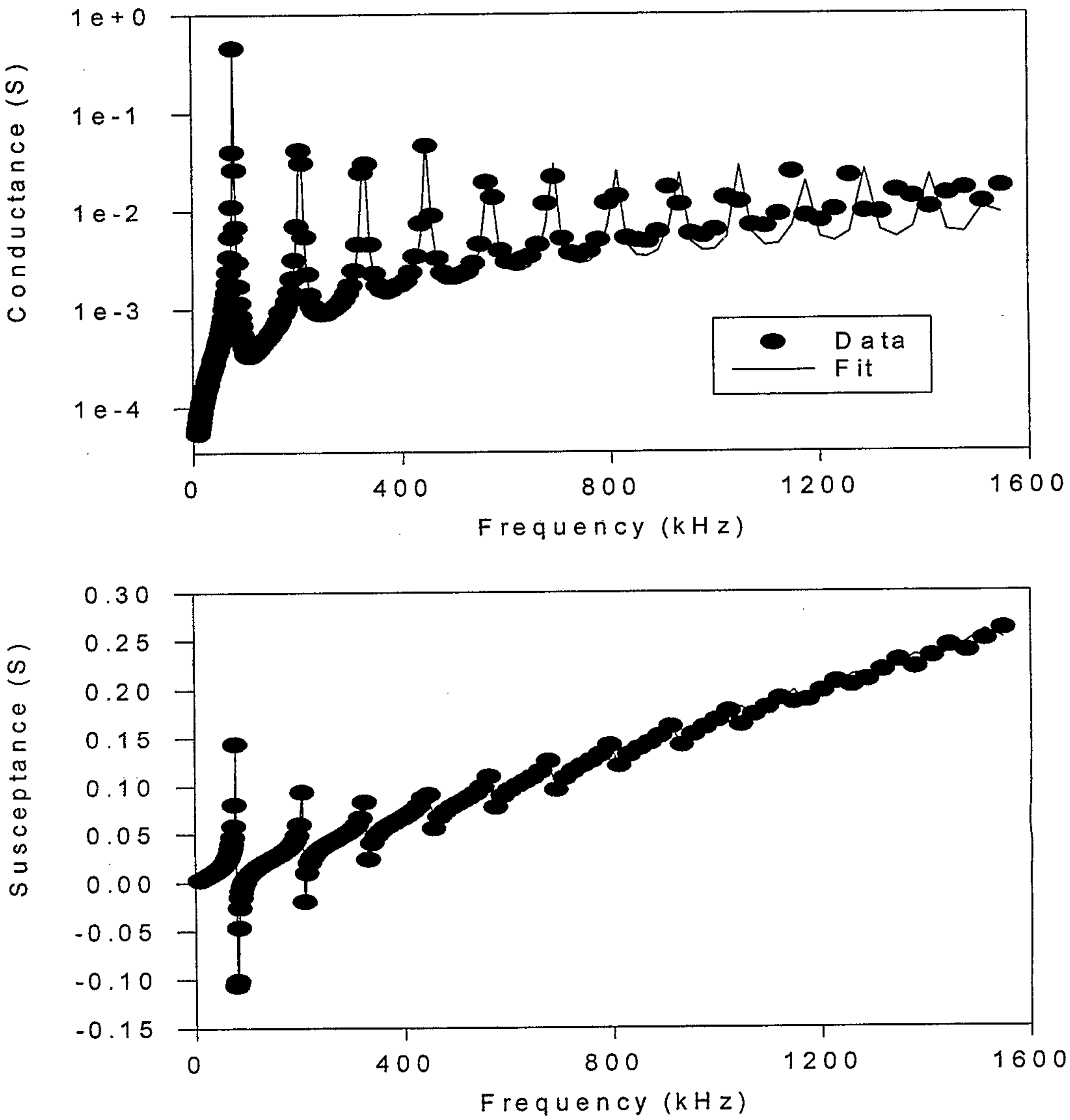

Figure 2: The radial mode impedance spectra for a 0.5 mm thick high-strain PZT sample produced by Morgan Matroc.

Table 5. Radial mode results for 0.5 mm samples. Results are an average of 5 samples.

Constant	Real	Imaginary
s_{11}^E (m^2/N)	1.58×10^{-11}	-2.0×10^{-13}
s_{12}^E (m^2/N)	-5.26×10^{-12}	7.2×10^{-13}
ε_{33}^T (F/m)	4.31×10^{-8}	-9.3×10^{-10}
d_{13} (C/N)	2.90×10^{-10}	-6.2×10^{-12}
k_p (#)	0.609	-.0029
σ_p (#)	0.333	-.00040

Table 6. Radial mode results for 1.0 mm samples. Results are an average of 5 samples.

Constant	Real	Imaginary
s_{11}^E (m^2/N)	1.62×10^{-11}	-2.1×10^{-13}
s_{12}^E (m^2/N)	-5.29×10^{-12}	7.5×10^{-14}
ε_{33}^T (F/m)	4.63×10^{-8}	-1.2×10^{-9}
d_{13} (C/N)	3.18×10^{-10}	-7.8×10^{-12}
k_p (#)	0.633	-.0037
σ_p (#)	0.326	-.00035

Table 7. Radial mode results for 2.0 mm samples. Results are an average of 6 samples.

Constant	Real	Imaginary
s_{11}^{E} (m^2 / N)	1.73×10^{-11}	-2.5×10^{-13}
s_{12}^{E} (m^2 / N)	-6.35×10^{-12}	1.3×10^{-13}
ε_{33}^{T} (F / m)	4.69×10^{-8}	-1.1×10^{-9}
d_{13} (C/N)	3.25×10^{-10}	-7.4×10^{-12}
k_p (#)	0.644	-.0036
σ_p (#)	0.371	-.019

ESC1 ELECTROSTRICTIVE MATERIAL

The Impedance Analyser was used to determine the permittivity and the dissipation of the electrostrictive samples as a function of frequency. The results are shown in Figure 3 and values of the permittivity and dissipation at 10, 100, 1000 and 10000 kHz are shown in Tables 8 to 10 for the three nominal sample thicknesses. The values given in the tables represent average values for the various specimens of the same nominal thickness and the standard deviations of the values are also indicated.

Figure 3 shows that the dielectric permittivities are a function of specimen thickness. This suggests that the materials are not identical and that the thickness of the discs may have affected the processing of the material. Figure 3 also shows that, at higher frequencies, the dissipation increases with decreasing thickness. This is likely to be due to electrode effects being more important in the case of thinner specimens. At higher frequencies, the electrode resistance will be of similar magnitude to the resistance of the ceramic itself and it will therefore cause a part of the dissipation. For thicker specimens this effect becomes negligible. Thus the results for the thickest specimen are more likely to truly represent the dissipation of the ceramic itself.

Table 8: Permittivity and Dissipation of the three 0.5 mm thick electrostrictive samples (one almost full disc and two pieces).

frequency (kHz)	Dissipation (#) (average)	Dissipation (#) (std. dev.)	Permittivity (F/m) (average)	Permittivity (F/m) (std. dev.)
10	0.0482	0.0043	5.85×10^{-8}	6.3×10^{-9}
100	0.0638	0.0081	5.40×10^{-8}	5.7×10^{-9}
1000	0.125	0.045	4.84×10^{-8}	5.3×10^{-9}
10000	0.446	0.15	1.83×10^{-8}	7.4×10^{-9}

Table 9: Permittivity and Dissipation of the four 1.0 mm thick electrostrictive samples.

Frequency (kHz)	Dissipation(#) (average)	Dissipation (#) (std. dev.)	Permittivity (F/m) (average)	Permittivity (F/m) (std. dev.)
10	0.0609	0.0025	8.31×10^{-8}	3.0×10^{-9}
100	0.0773	0.0033	7.47×10^{-8}	2.3×10^{-9}
1000	0.123	0.025	6.51×10^{-8}	1.8×10^{-9}
10000	0.378	0.096	2.28×10^{-8}	1.7×10^{-9}

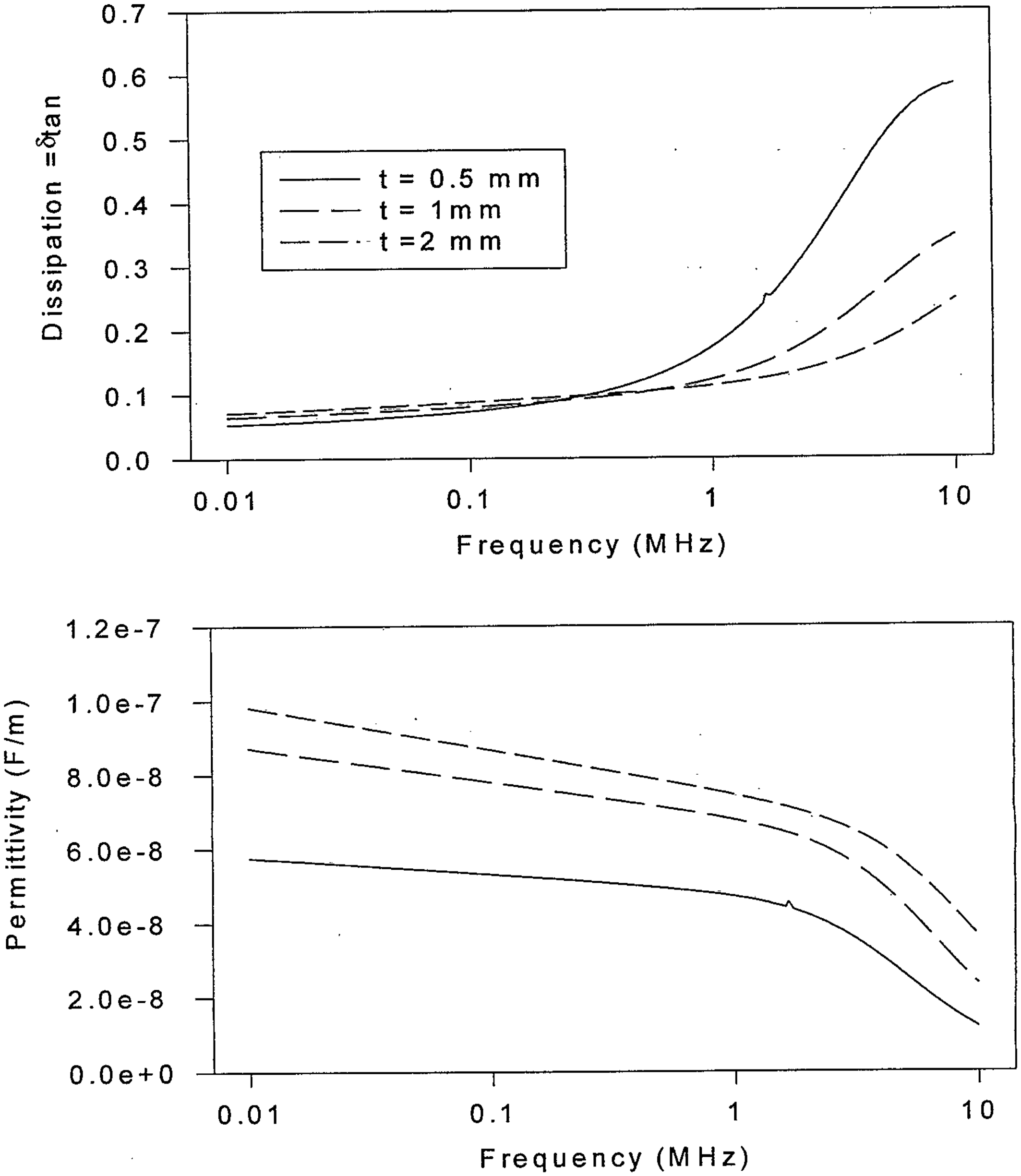

Figure 3: The dielectric permittivity and dissipation for 0.5mm , 1.0 mm and 2.0 mm thick disks of ESC1 electrostrictive ceramics produced by Morgan Matroc.

Table 10: Permittivity and Dissipation of the four 2.0 mm thick electrostrictive samples.

frequency (kHz)	Dissipation (#) (average)	Dissipation (#) (std. dev.)	Permittivity (F/m) (average)	Permittivity (F/m) (std. dev.)
10	0.0724	0.0015	9.89×10^{-8}	1.6×10^{-9}
100	0.0889	0.0017	8.71×10^{-8}	1.4×10^{-9}
1000	0.116	0.0030	7.47×10^{-8}	1.3×10^{-9}
10000	0.269	0.020	3.62×10^{-8}	7.2×10^{-10}

Direct Measurement of Strain as a Function of the Electric Field

PC5K HIGH STRAIN PZT:

The curves in Figure 4 show the strain as a function of the electric field for the 0.5 , 1.0 and 2.0 mm PZT samples using the optical lever technique[3]. The voltage on the sample was cycled three times to ±1500 volts for the 0.5 mm, 1.0 mm and 2.0 mm thick discs. The 0.5 mm thick disc was affixed to a one inch brass flat using silver filled epoxy adhesive in order to eliminate sample bending. The material shows the typical ferroelectric switching that is common to PZT as the coercive field of the material is exceeded. The coercive field is estimated to be between 0.5 and 0.6 MV/m. Below the coercive field the strain of the material is linear in field with hysteresis present. The low field (below the coercive field) strain versus field curves for the samples are shown in Figure 5 for three different thicknesses of the material.

ESC1 ELECTROSTRICTIVE MATERIAL

We have also used the optical lever experiment to measure the strain as a function of an electric field applied to the electrostrictive discs. The curves in Figure 6 show examples of the results of the optical lever displacement measurements cycled three times to ±1500 volts for 0.5 mm, 1.0 mm and 2.0 mm thick plates cut 2.0 mm square from samples provided. The curves in Figure 7 show displacement results cycled three times to ±1500 volts for 0.5 mm, 1.0 mm and 2.0 mm thick discs as received. Once again the lack of symmetry in the hysteresis curves of the larger diameter discs is symptomatic of bending of the discs. Indeed square plates of sides larger than 2.5 mm show such asymmetry and bending. It is possible that the bending of the samples can be reduced by changing the processing of the ceramic material, for example, (a) by improving the tolerances on the thickness of the electrodes and by making both electrodes of the same thickness, or (b) by improving the uniformity of the ceramic itself.

The Piezoelectric Charge Coefficient, d_{33}, of the PC5K High Strain PZT

A Berlincourt type d_{33} meter was used to measure the piezoelectric charge coefficient of the three thicknesses of the PC5K high strain PZT specimens. Since we had already used the specimens for making the high field strain-voltage measurements described in the previous section, the specimens had been depoled. They were therefore repoled at 1500 V dc and aged for about 3 days. An average nominal d_{33} of about 800 pC/N was obtained.

The Hydrostatic Voltage Coefficient of the PC5K High Strain PZT

A comparative technique was used to measure the hydrostatic voltage coefficient, g_h, of the PC5K high strain PZT, up to pressures of 14 MPa. Our results are shown in Tables 11, 12 and 13.

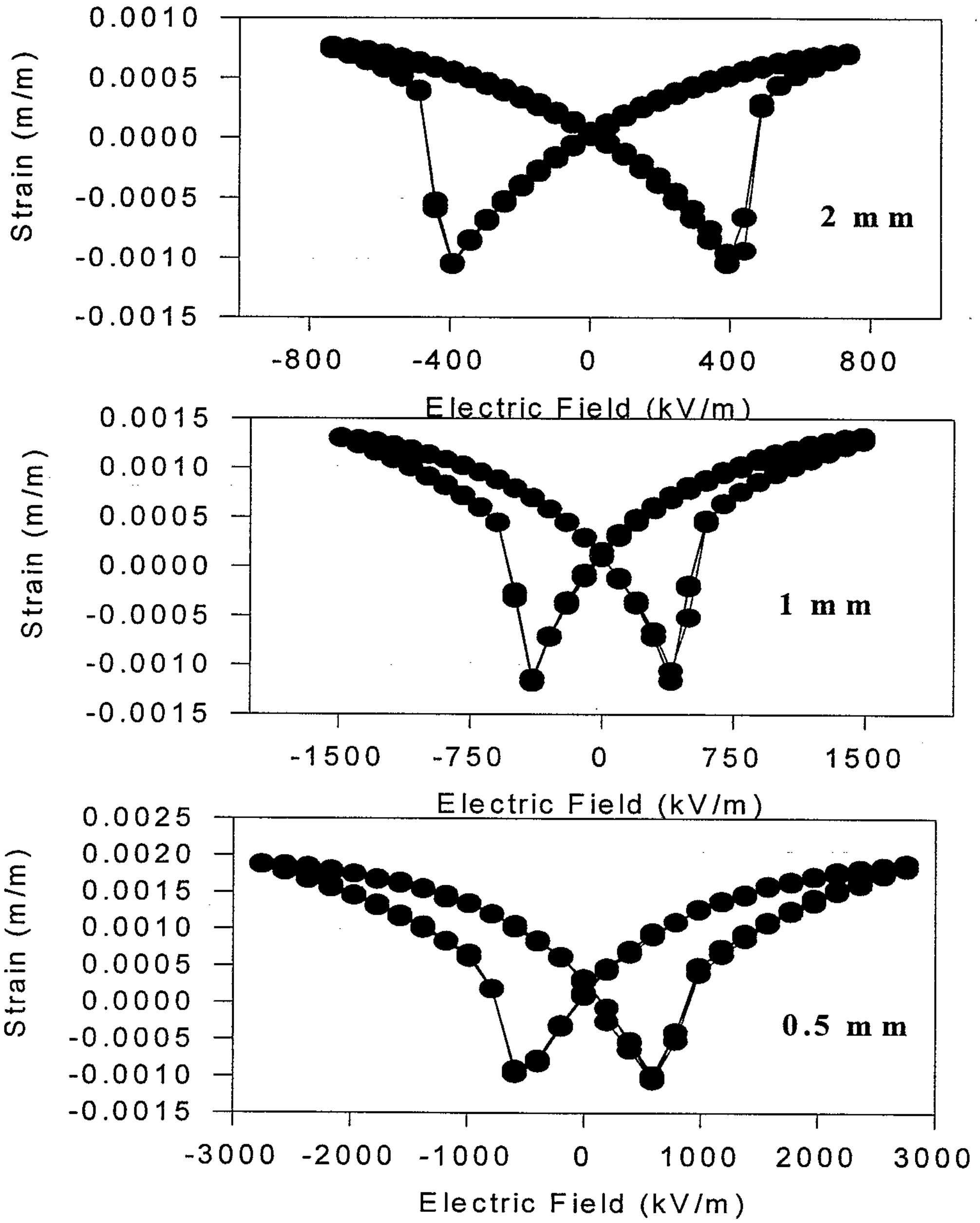

Figure 4: The strain as a function of the electric field for 0.5 mm , 1.0 mm and 2.0 mm thick disks of high strain PZT produced by Morgan Matroc. The field is cycled above the coercive field.

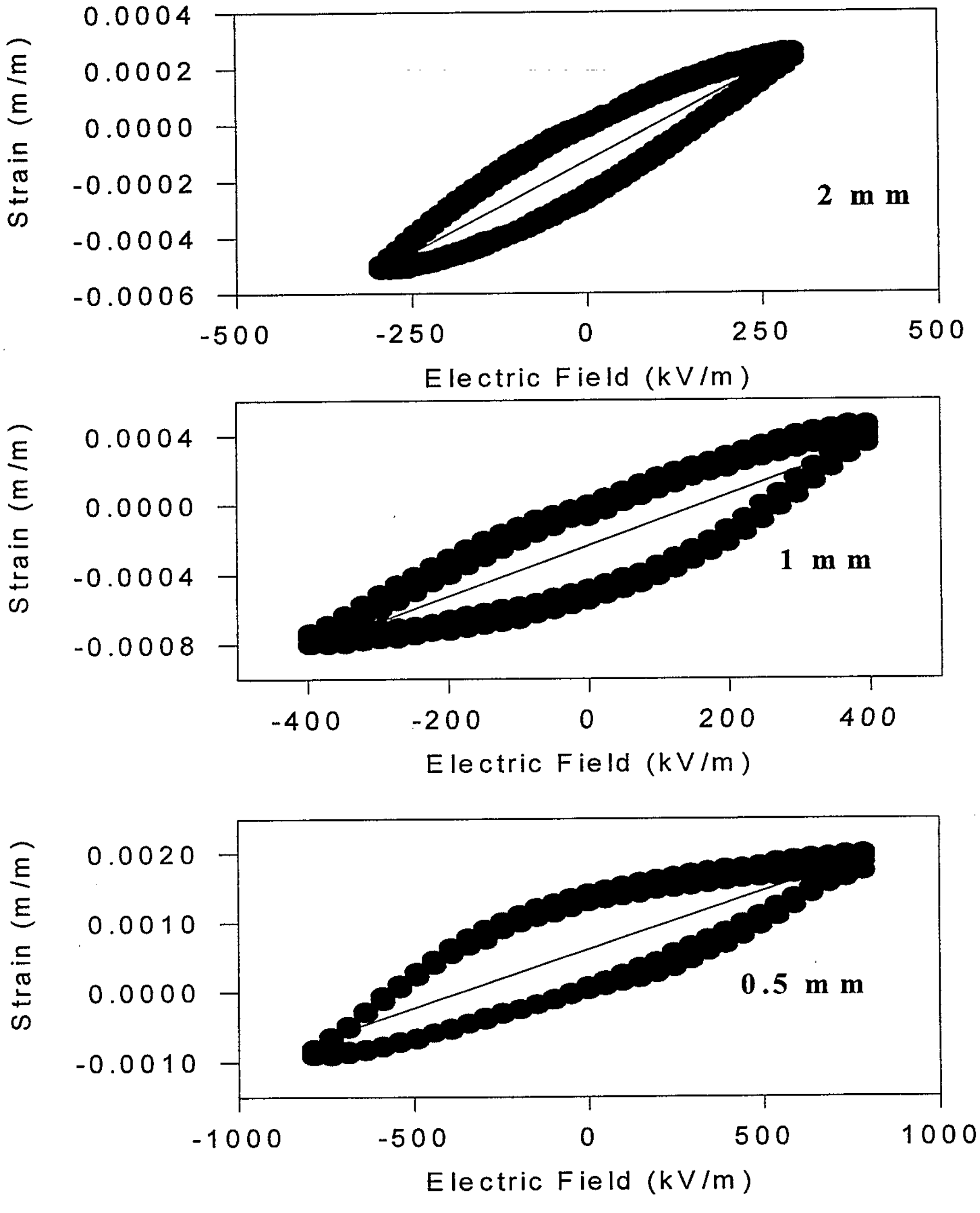

Figure 5: The strain as a function of the electric field for 0.5 mm, 1.0 mm and 2.0 mm thick disks of high strain PZT produced by Morgan Matroc. The field is cycled below the coercive field.

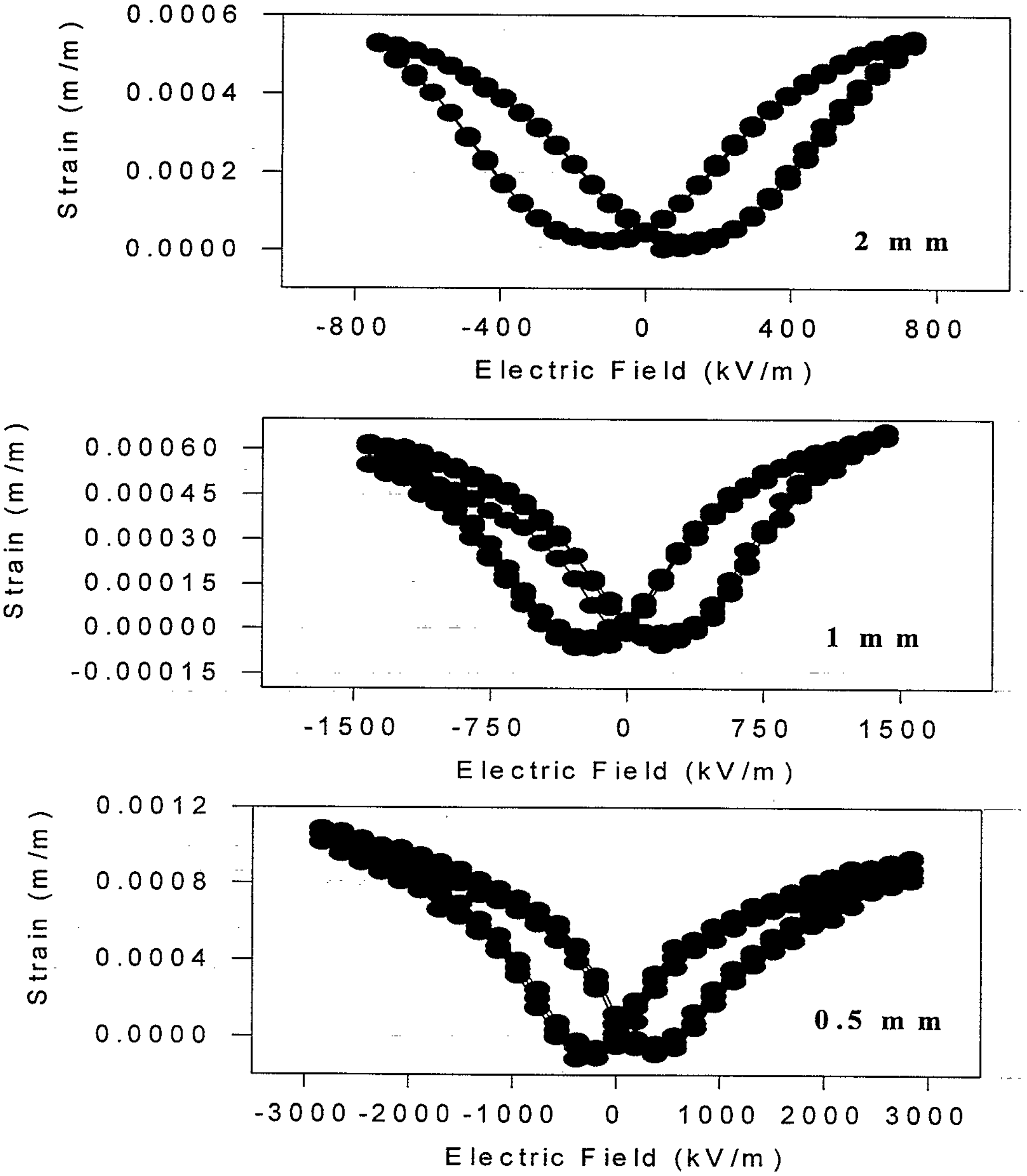

Figure 6: The strain as a function of the electric field for 2.0 mm square plates cut from 0.5 mm, 1.0 mm and 2.0 mm thick discs of electrostrictive material produced by Morgan Matroc. The symmetry is more pronounced due to a reduction of bending modes in a small plate.

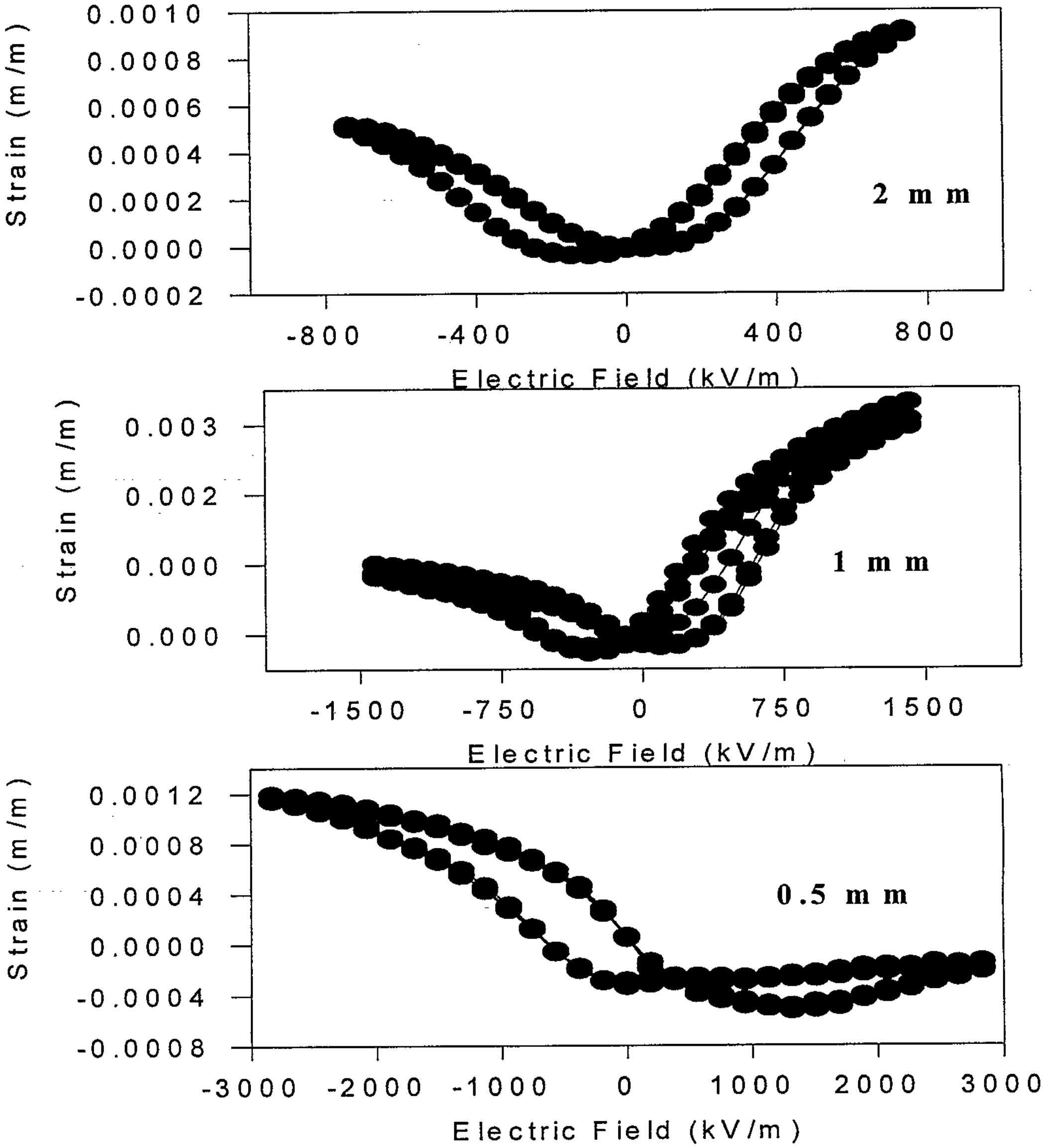

Figure 7: The strain as a function of the electric field for 0.5 mm, 1.0 mm and 2.0 mm thick 1 inch diameter discs of electrostrictive material produced by Morgan Matroc. The asymmetry is due to bending modes in the disc.

The tables show that the hydrostatic coefficient is relatively independent of pressure up to a pressure of 14 MPa. There appears to a slight tendency for g_h to increase slightly at pressures above about 10 MPa.

Table 11: Measured values of the hydrostatic voltage coefficient for one 0.5 mm thick PC5K PZT specimen tested twice.

Pressure (MPa)	g_h (10^{-3} Vm/N) - run 1	g_h (10^{-3} Vm/N) - run 2
2	1.30	1.31
4	1.30	1.28
6	1.30	1.29
8	1.34	1.47
10	1.30	1.31
12	1.28	1.46
14	1.46	1.47

Table 12: Measured values of the hydrostatic voltage coefficient for two 1.0 mm thick PC5K PZT specimens tested twice each.

Pressure (MPa)	g_h (10^{-3} Vm/N) sample 1 run 1	g_h (10^{-3} Vm/N) sample 1 run 2	g_h (10^{-3} Vm/N) sample 4 run 1	g_h (10^{-3} Vm/N) sample 4 run 2
2	1.26	1.47	1.23	1.20
4	1.22	1.28	1.27	1.40
6	1.22	1.28	1.27	1.25
8	1.33	1.47	1.42	1.41
10	1.23	1.30	1.27	1.26
12	1.23	1.47	1.28	1.43
14	1.41	1.49	1.45	1.44

Table 13: Measured values of the hydrostatic voltage coefficient for two 2.0 mm thick PC5K PZT specimens tested twice each.

Pressure (MPa)	g_h (10^{-3} Vm/N) sample 1 run 1	g_h (10^{-3} Vm/N) sample 1 run 2	g_h (10^{-3} Vm/N) sample 2 run 1	g_h (10^{-3} Vm/N) sample 2 run 2
2	1.31	1.37	1.32	1.49
4	1.34	1.37	1.33	1.31
6	1.54	1.36	1.34	1.49
8	1.33	1.55	1.53	1.32
10	1.36	1.59	1.32	1.49
12	1.53	1.58	1.55	1.51
14	1.42	1.57	1.52	1.51

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[2] S. Sherrit, N. Gauthier, H.D. Wiederick and B.K. Mukherjee, "Accurate Evaluation of the Real and Imaginary Material Constants for a Piezoelectric Resonator in the Radial Mode", *Ferroelectrics*, **119**, 17-32 (1991).

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