

REPORT

Round-Robin exercise on the measurement of electromechanical properties of piezoelectric ceramics

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ROUND-ROBIN EXERCISE ON THE MEASUREMENT OF ELECTROMECHANICAL PROPERTIES OF PIEZOELECTRIC CERAMICS

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ABSTRACT

As part of the DTI programme on advanced materials, project AM4, on high stress testing of piezoceramics, a round robin exercise was conducted by sending piezoceramic samples to various UK laboratories involved with the measurement of their properties. The exercise has demonstrated that there are considerable differences in piezoelectric measurement results between laboratories performing what were thought to be relatively simple measurements. The reproducibility within a laboratory is good, however the poor inter laboratory repeatability questions the validity of comparisons of different manufacturers data.

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Director, Centre for Materials Measurement and Technology

CONTENTS

1 INTRODUCTION	1
2 CAPACITANCE MEASUREMENTS	1
3 RESONANCE MEASUREMENTS	1
4 BERLINCOURT MEASUREMENTS, d_{33}	3
5 CONCLUSIONS	3
6 ACKNOWLEDGEMENTS	3
7 REFERENCES	3
TABLES AND FIGURES	4
APPENDIX	9

1 INTRODUCTION

As part of the DTI programme on advanced materials, project AM4, on high stress testing of piezoceramics, a round robin exercise was conducted by sending sets of samples to various UK laboratories involved with the measurement of their properties. The instructions sent with the samples are listed in the appendix, but essentially a set of two materials were used, a hard and a soft ceramic, and six samples of each material were sent out to each laboratory. There had been some concern during the programme that the results were being affected by the poor quality of materials, and so the soft ceramic used was a hot pressed ceramic which would hopefully reduce some of the variability caused by porosity in previously conventionally sintered ceramics.

2 CAPACITANCE MEASUREMENTS

Most of the participants performed the room temperature capacitance measurements, and the results of these are listed in the tables. Although the inherent material variability and the restricted number of samples examined makes detailed analysis unreliable there are nonetheless some conclusions that can be drawn. Firstly the reproducibility of repeated measurements on the same samples for a single laboratory gave differences of the order of 0.5%. However the repeatability between measurements on the same samples for different laboratories showed significant differences. For example laboratory F results were 5% higher than Lab A for one set of six samples, and Lab F were 2% higher than lab B for another set. Figures 1 and 2 shows that there is a consistency between the measurements, indicating that this is a real measurement error and not simply due to material variability.

There are of course several possible reasons for the discrepancies apart from the obvious possibility of incorrect calibration of the measuring instrument. The results returned did not all state the exact conditions such as frequency of measurement, temperature, applied voltage level, and also the method of contact onto the samples themselves. There is also the possibility that the samples have undergone some transformation due to ageing or thermal cycling. There were also significant differences in sample means of different sets of samples sent out as measured by the individual laboratory. This is assuming that the original set of samples all come from the same population. For a more rigorous test there should be only one set of samples to be measured by each and every participant, with one 'reference' laboratory measuring the samples after each participant has performed the tests. This would sort out the problems of material variability, and also by performing measurements at each stage check for any ageing or other sample degradation.

3 RESONANCE MEASUREMENTS

Most of the participants performed resonance measurements on the samples and found the resonance and anti resonance frequency using one of the Hewlett Packard family of impedance analysers. Figures 3 and 4 show sample resonance curves near the radial resonance peaks for the hard and the soft PZT. The results have been plotted as conductance, and resistance against frequency, and in this form it is easy to see the resonance frequency is the maximum in the conductance curve, while the maximum in the resistance curve corresponds to the anti-resonance frequency. The impedance curves can also be plotted in the form of amplitude and phase, where the resonance and anti-resonance frequency are characterised as the phase angle going through zero, however using computer control it is often simpler to detect the maximum rather than a change in sign. There were two fundamental resonances for the

samples, the radial mode with a frequency near 200kHz, and the thickness mode at around 2MHz. From these values the electromechanical coupling factors, the ratio of output energy to input energy for the material, can be calculated. To a first approximation this coupling coefficient can be expressed as

$$k_{eff}^2 = \frac{f_a^2 - f_r^2}{f_a^2} \quad (1)$$

The difference between the two samples can be seen in figures 3 and 4. For the soft PZT the two peaks are separated further than the hard PZT peaks and therefore has a higher coupling coefficient. The sharper peaks in the hard PZT material, in particular for the anti-resonance peak is an indication of the higher mechanical quality factor Q_m .

However the coupling coefficient can depend on the geometry of the sample, and the poling direction, and the concept has been refined for various geometries. The most common form of resonator geometries are flat discs and tall cylinders or rods. For a flat disc, poled in the thickness direction, the diameter must be at least ten times the thickness (the samples used in this round robin were 10mm diameter 1mm thick). The sample is excited in the radial mode and the resulting coupling coefficient is termed the planar coupling coefficient

$$\frac{k_p^2}{k_p^2 - 1} = f \left[J_0, J_1, \nu \frac{f_a - f_r}{f_a} \right] \quad (2)$$

The terms J_0 and J_1 are Bessel functions, and Poisson's ratio is also included in the formula. The coupling coefficient can either be calculated analytically or there are look-up tables provided in the IEEE standard¹ for various Poisson's ratio. Fortunately the function does not depend strongly on the value of Poisson's ratio and a value of 0.3 is often used for ceramic samples.

For cylinders or rods poled in the thickness direction the sample is excited in the thickness mode, and the sample geometry must be such that the length is greater than 2.5 times the diameter. This gives an electrochemical coupling factor of

$$k_{33}^2 = \frac{\pi}{2} \frac{f_r}{f_a} \tan \left[\frac{\pi}{2} \frac{f_a - f_r}{f_a} \right] \quad (3)$$

Most of the issues raised by the capacitance measurements such as material variability and degradation with time also apply for the resonance measurements, with the added difficulty of the various forms of equations to use in each case. Indeed there are some minor differences in terminology of the coupling coefficients with the various standards bodies^{1,2}. To overcome this the k_{eff} was calculated from the simple approximation in equation 1, using the resonance and anti-resonance frequencies determined by the participants. From the results on the same samples tested by different laboratories it can be seen that the results for laboratory F and B are virtually identical, whereas those of laboratory A are consistently higher than the results of laboratory F (figures 5,6).

Again there is the question as to whether all the equipment used is traceably calibrated, and factors such as the temperature have not been considered, however the most likely cause of these differences is that the samples are held differently. Laboratories F and B used a sample holding jig with one electrode as a flat surface and the other a point contact, whereas laboratory A used a jig with two point contacts. The clamping force used to hold the sample for testing is a variable that has not been quantified for all three cases. Again each participants reproducibility is good since they each show the same trends for groups of samples, however this is because the sample holding conditions are consistent. Figure 7 shows the trend for the

difference in resonance and anti-resonance frequencies for two laboratories and given the formula for k_{eff} it is evident that the differing k_{eff} values come from the change in separation in these frequencies $f_a - f_r$ and not a general shift in the resonance and anti resonance peaks.

4 BERLINCOURT MEASUREMENTS, D₃₃

Although these measurements were not explicitly asked for in the round robin exercise several participants performed these measurements as they are simple to carry out, given the requisite equipment, and are often performed in industry as part of a QA exercise. The Berlincourt test measures the charge output of the sample under a small AC mechanical load. There are several variables in this test that are not always defined, such as the frequency of the load (usually around 100Hz), the magnitude of the clamping load, and the amplitude of the applied alternating load.

Looking at these results on the same samples there is considerable variability between laboratories, depending on the material. For the soft ceramic, laboratory F results were consistently higher than those of laboratory B, however for the hard ceramic the results in general were more variable and the inter laboratory differences not so pronounced (figure 8). It is interesting to note that laboratory B and C used the same type of commercial test equipment to perform the measurements, whereas laboratories D and F used 'home-made' equipment. The results of B and C were similar, whereas laboratory F were generally higher, while those of laboratory D (who used the highest clamping force) were the lowest.

5 CONCLUSIONS

The round robin exercise has demonstrated that there are considerable differences in piezoelectric measurement results between laboratories performing what were thought to be relatively simple measurements. The repeatability of the measurements within a laboratory shows the validity of using these results for internal quality control or material improvement, however the variability between laboratories question the value of comparing manufacturers data to choose materials. It is disappointing that some of the high stress measurements such as the polarisation-field, and displacement-field measurements were not performed by the participants, but is understandable due to their complexity and the need to fit the measurements into a commercial environment. It does however highlight the need to isolate all forms of measurement induced variability, and to advise on how to avoid these in the form of standards or best practice guides. It is obvious that the present standards do not contain sufficient information to prevent the differences found in this round robin.

6 ACKNOWLEDGEMENTS

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

7 REFERENCES

1. IEEE Standard on Piezoelectricity, IEEE Std. 176-1987.
2. Guide to dynamic measurements of piezoelectrics with high electromechanical coupling, IEC 483-1976.

	Cap(pF)	TAN	Fr(kHz)	Fa(kHz)	K _g	d ₃₃ pC/N	Z(ohms)	Q	dielectric constant	current @20V (pA)	resistivity	dielectric loss %	K _g equation	calc k _g , k _t equation(2)
Lab A radial														
KMM/A/1	738.57	0.003	229.87	244.88	0.388		13.386	589.7					0.345	0.377
KMM/A/2	728.87	0.003	229.12	246.75	0.416		45.433	152.7					0.371	0.405
KMM/A/3	748.93	0.003	229.5	245.63	0.402		12.25	595.2					0.356	0.39
KMM/A/4	768.02	0.004	231	244.5	0.368		19.647	425.2					0.328	0.359
KMM/A/5	749.67	0.003	229.5	246	0.404		11.178	638.3					0.36	0.394
KMM/A/6	753.35	0.003	229.5	246.75	0.413		15.603	437.2					0.367	0.401
Lab A thickness k33														
KMM/A/1	737.92	0.003	2,115	2,187.5	0.281		5.134	304.7					0.255	0.281
KMM/A/2	727.47	0.003	2,117.5	2,197.5	0.294		10.687	135.2					0.287	0.294
KMM/A/3	750.07	0.003	2,110	2,192.5	0.299		3.824	358.1					0.272	0.299
KMM/A/4	763.37	0.004	2,155	2,230	0.283		5.037	290.4					0.257	0.283
KMM/A/5	746.75	0.003	2,115	2,197.5	0.299		4.825	283.4					0.271	0.299
KMM/A/6	749.44	0.003	2,105	2,187.5	0.299		5.88	231.8					0.272	0.299
Lab B radial														
KMM/B/1	705.5	0.002	232.8	245.6		170			1,015				0.319	0.35
KMM/B/2	641.4	0.001	233.5	244		154			922.8				0.29	0.319
KMM/B/3	737	0.002	230.25	246.25		195			1,060.3				0.355	0.388
KMM/B/4	729.7	0.002	229.75	246.75		201			1,049.8				0.365	0.399
KMM/B/5	715	0.002	229.75	245		189			1,028.7				0.347	0.38
KMM/B/6	754.2	0.002	230.5	246.75		201			1,085				0.357	0.39
Lab C radial														
KMM/C/1	722					197			1,042.31	9.5	16.4	0.245		
KMM/C/2	721					204			1,048.04	33	4.7	0.225		
KMM/C/3	739					200			1,060.58	39	4.03	0.245		
KMM/C/4	700					188			1,048.25	53	2.84	0.22		
KMM/C/5	705					195			1,068.08	13	11.4	0.21		
KMM/C/6	734					199			1,058.22	14	11.1	0.215		
Lab E radial k13														
KMM/D/1			230.5	244	0.389	159			N3	c0	c0(13)		0.328	0.36
KMM/D/2			230.5	248.5	0.453	162			2,440	4,420	4,880		0.374	0.408
KMM/D/3			228.25	246.25	0.456	163			2,485	4,380	4,970		0.375	0.41
KMM/D/4			228.25	244	0.424	159			2,463	4,393	4,925		0.353	0.387
KMM/D/5			230.238	245.463	0.397	154			2,440	4,408	4,880		0.347	0.379
KMM/D/6			231.225	245.313	0.414	153			2,453	4,352	4,908		0.334	0.366
Lab E thickness kt														
KMM/D/1			2,130.93	2,209.9	0.306				Nt				0.265	0.292
KMM/D/2			2,108.958	2,190.05	0.312				2,210				0.27	0.297
KMM/D/3			2,114.08	2,196.49	0.314				2,190				0.271	0.299
KMM/D/4			2,124.61	2,202.978	0.305				2,197				0.264	0.291
KMM/D/5			2,104.975	2,176.113	0.291				2,203				0.254	0.279
KMM/D/6			2,114.238	2,188.15	0.297				2,176				0.258	0.284
Lab F radial														
KMM/A/1	775.7	0.003	231.75	242.75									0.298	0.327
KMM/A/2	765.3	0.001	233	244									0.297	0.326
KMM/A/3	790	0	230.75	243.25									0.316	0.347
KMM/A/4	790.3	0.002	232.25	242.5									0.288	0.316
KMM/A/5	781.8	0.001	231	244.5									0.328	0.359
KMM/A/6	788.8	0.001	230.5	244.25									0.331	0.363
KMM/B/1	718	0.001	233.25	246		179							0.318	0.349
KMM/B/2	654.8	0.001	234	244.25		135							0.287	0.315
KMM/B/3	748.1	0.001	230.75	247		217							0.357	0.39
KMM/B/4	744.6	0.001	230.25	247.25		178							0.364	0.398
KMM/B/5	727.3	0.001	230.5	246.25		184							0.352	0.385
KMM/B/6	770	0.001	231	247		211							0.354	0.387
Lab F thickness														
KMM/A/1	775.7	0.003	2,128	2,197									0.249	0.274
KMM/A/2	765.3	0.001	2,129	2,202									0.255	0.281
KMM/A/3	790	0	2,123	2,198									0.256	0.282
KMM/A/4	790.3	0.002	2,164	2,235									0.25	0.276
KMM/A/5	781.8	0.001	2,127	2,198									0.252	0.278
KMM/A/6	788.8	0.001	2,118	2,190									0.254	0.28
KMM/B/1	718	0.001	2,160	2,232									0.252	0.278
KMM/B/2	654.8	0.001	1,943	2,042									0.308	0.338
KMM/B/3	748.1	0.001	2,137	2,224									0.277	0.305
KMM/B/4	744.6	0.001	2,121	2,207									0.276	0.304
KMM/B/5	727.3	0.001	2,119	2,194									0.259	0.286
KMM/B/6	770	0.001	2,174	2,274									0.293	0.322

Table 1 Summary of round robin results for Hard PZT

	Cap(pF)	TAN	Fr(kHz)	Fa(kHz)	K _t	d ₃₃ pC/N	Z(ohms)	Q	dielectric constant	current @20V	resistivity	dielectric loss %	K _{eff} equation(1)	calc K _{eff} , K _t equation(2)
Lab A radial														
KMR/182/	1,022.38	0.02	210.38	249.38	0.61		38.49	66.7					0.54	0.58
KMR/182/	940.18	0.03	212.25	250.12	0.6		83.92	34					0.53	0.57
KMR/182/	989.69	0.02	211.88	247.88	0.59		42.19	66.8					0.52	0.56
KMR/182/	972.23	0.02	213	245.63	0.56		49.02	63.2					0.5	0.54
KMR/182/	967.17	0.02	211.88	243	0.55		50.42	64.2					0.49	0.53
KMR/182/	965.04	0.02	203.63	243.38	0.62		114.97	23.5					0.55	0.59
Lab thickness														
KMR/182/	1,014.92	0.02	2,092.5	2,295	0.45		10.42	42.6					0.41	0.45
KMR/182/	942.51	0.03	2,090	2,275	0.43		14.8	35					0.4	0.43
KMR/182/	992.76	0.02	2,082.5	2,272.5	0.44		11.48	41.8					0.4	0.44
KMR/182/	963.78	0.02	2,087.5	2,267.5	0.43		13.71	37.8					0.39	0.43
KMR/182/	962.46	0.02	2,070	2,252.5	0.43		15.92	32.3					0.39	0.43
KMR/182/	954.25	0.03	2,042.5	2,230	0.44		24.02	21.1					0.4	0.44
Lab B radial														
KMR/179/	957.7	0.02	208.5	256		374			1,377.8				0.58	0.62
KMR/179/	896.2	0.02	211.5	256		361			1,289.4				0.56	0.6
KMR/179/	924.6	0.02	212.5	257		368			1,330.2				0.56	0.6
KMR/179/	921.5	0.02	212.5	256		350			1,325.8				0.56	0.6
KMR/179/	905.4	0.02	212.5	256		361			1,302.6				0.56	0.6
KMR/179/	909	0.02	212	256		351			1,307.8				0.56	0.6
Lab C radial														
179/7	942.3					344			1,385.23	8	19.2	2.05		
179/8	906.1					350			1,361.59	10	15	2.03		
179/9	930.8					353			1,367.5	8	19.2	2.02		
183/4	851.1					303			1,277.62	4	37.6	2.35		
183/7	849.46					274			1,267.51	12	12.6	2.38		
183/8	860.46					283			1,299.06	6	24.9	2.3		
Lab D radial														
180/1	975.34		2,080	2,270.5		362			1,402.57	20,800			0.4	0.44
180/2	925.29		2,090	2,283.5		344			1,330.6	20,875			0.4	0.44
180/3	931.12		2,100	2,278.5		341			1,338.98	21,040			0.39	0.42
180/4	928.04		2,090	2,282		353			1,334.56	20,890			0.4	0.44
180/5	937.57		2,090	2,280		353			1,348.26	20,935			0.4	0.44
179/10	922.52		2,110	2,320		370			1,326.62	21,055			0.42	0.45
Lab E radial														
181/3			214.04	242.84	0.61	245			2,264	4,365	4,526		0.47	0.51
181/4			214.75	246.25	0.65	278			2,257	4,390	4,513		0.49	0.53
181/5			214.75	246.25	0.65	253			2,463	4,382	4,925		0.48	0.53
181/8			212.5	241.75	0.62	268			2,418	4,319	4,835		0.48	0.52
181/10			207.86	242.84	0.7	296			2,429	4,377	4,857		0.52	0.56
181/11			205.75	237.25	0.66	303			2,373	4,354	4,745		0.5	0.54
181/11			210.79	252.68	0.77	326			2,527	4,370	5,054		0.55	0.59
181/12			212.5	246.25	0.68	304			2,463	4,399	4,925		0.51	0.54
181/13			208	239.5	0.66	329			2,395	4,279	4,790		0.5	0.54
Lab E thickness														
181/3			2,083.66	2,182.5	0.35				2,183				0.3	0.33
181/4			2,090.63	2,194.77	0.36				2,195				0.3	0.33
181/5			2,091.5	219.9	0.35				2,191					
181/8			2,054.94	2,159.28	0.36				2,196				0.31	0.34
181/10			2,065	2,188.25	0.39				2,189				0.33	0.36
181/11			2,067.75	2,176.88	0.37				2,177				0.31	0.34
181/11			2,079.04	2,185.23	0.36				2,185				0.31	0.34
181/12			2,082.93	2,199.38	0.38				2,200				0.32	0.35
181/13			2,036.55	2,139.56	0.36				2,140				0.31	0.34
Lab F radial														
KMR/182/	1,069	0.02	215.25	244									0.47	0.51
KMR/182/	969.5	0.02	216.25	243.25									0.46	0.5
KMR/182/	1,036	0.02	215.5	242.5									0.46	0.5
KMR/182/	1,004	0.02	216	240.75									0.44	0.48
KMR/182/	988.4	0.02	216.5	242.25									0.45	0.49
KMR/182/	994.4	0.02	213.75	241.5									0.47	0.5
KMR/179/	966.4	0	208.5	256.25		402							0.58	0.62
KMR/179/	911	0.02	212	256.75		397							0.56	0.6
KMR/179/	939.2	0.02	212.75	257.75		399							0.56	0.6
KMR/179/	940.2	0.17	212.5	256.25		398							0.56	0.6
KMR/179/	922.7	0.02	213	256.75		386							0.56	0.6
KMR/179/	932.3	0.02	212	256		387							0.56	0.6
Lab F thickness														
KMR/179/	966.4	0	2,098	2,318									0.43	0.46
KMR/179/	911	0.02	2,082	2,288									0.42	0.45
KMR/179/	939.2	0.02	2,080	2,334									0.45	0.49
KMR/179/	940.2	0.17	2,118	2,296									0.39	0.42
KMR/179/	922.7	0.02	2,088	2,312									0.43	0.47
KMR/179/	932.3	0.02	2,100	2,318									0.42	0.46
KMR/182/	1,069	0.02	2,098	2,278									0.39	0.43
KMR/182/	969.5	0.02	2,100	2,188									0.28	0.31
KMR/182/	1,036	0.02	2,088	2,196									0.31	0.34
KMR/182/	1,004	0.02	2,082	2,196									0.3	0.33
KMR/182/	988.4	0.02	2,084	2,178									0.28	0.32
KMR/182/	994.4	0.02	2,054	2,144									0.29	0.32

Table 2 Summary of round robin results for Soft PZT

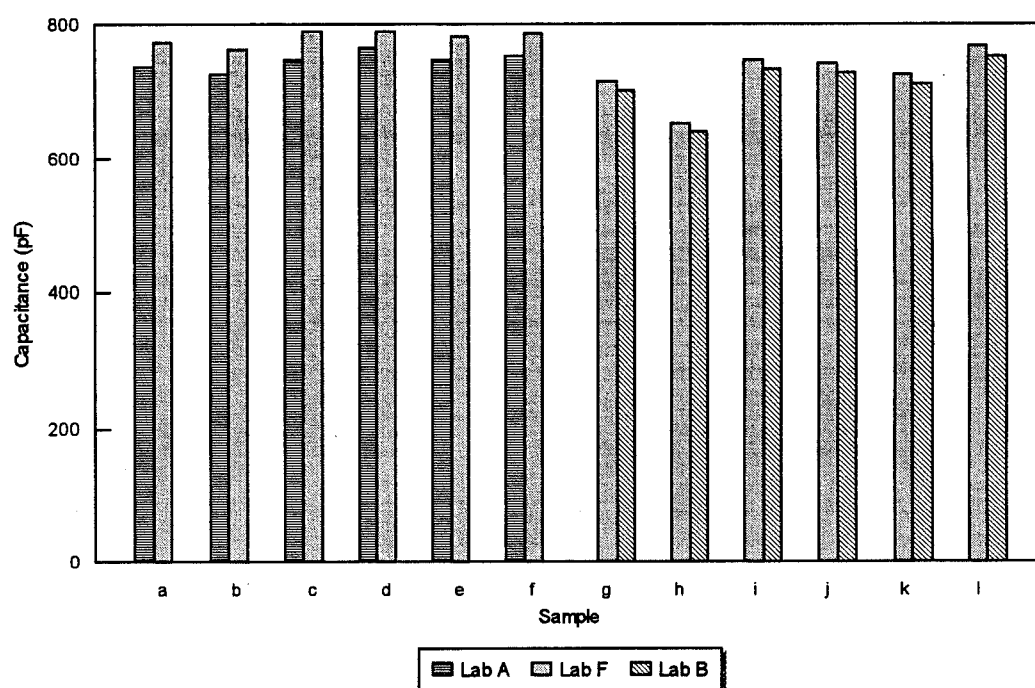


Figure 1. Round robin capacitance measurements for laboratories A, B, and F on two sets of Hard PZT samples.

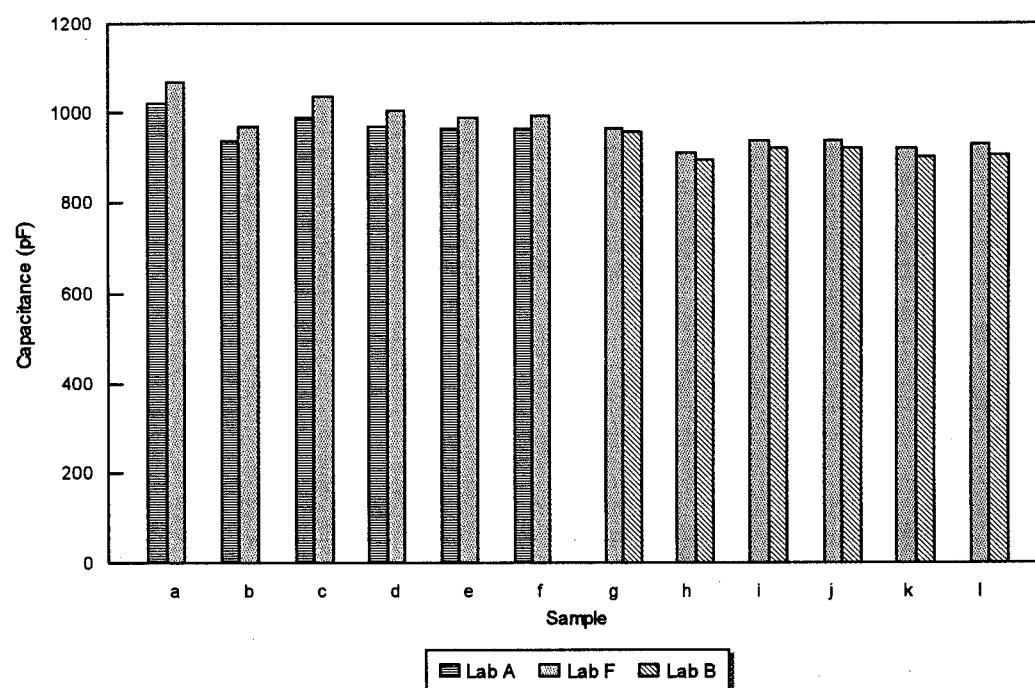


Figure 2. Round robin capacitance measurements for laboratories A, B, and F on two sets of Soft PZT samples.

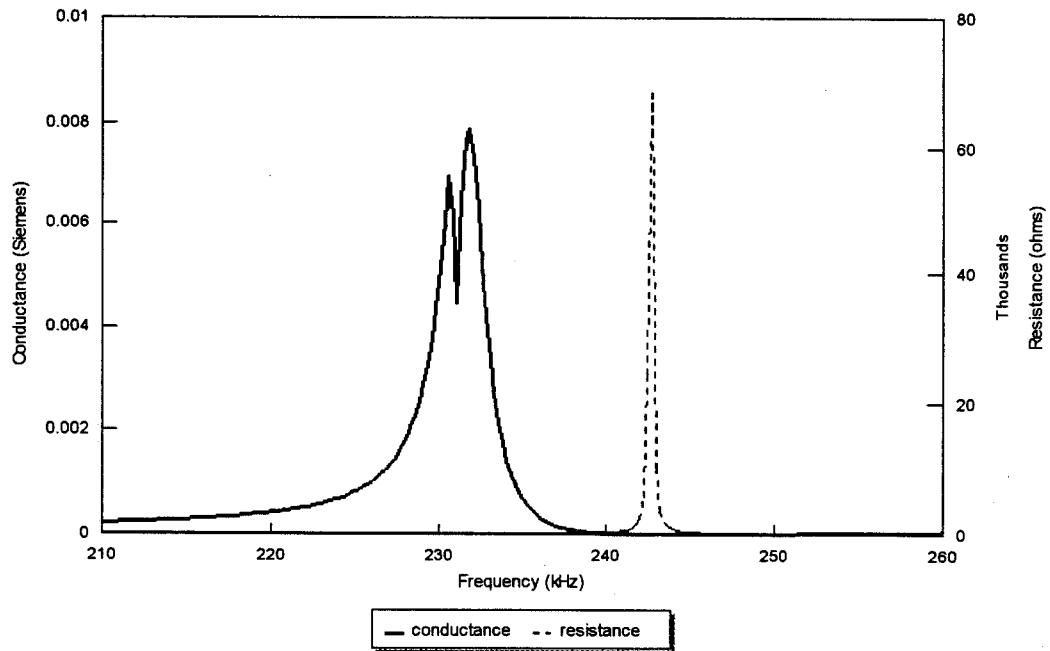


Figure 3. Example radial resonance curve for hard PZT sample KMM/A/1, measurement performed by laboratory F, showing conductance and resistance against frequency.

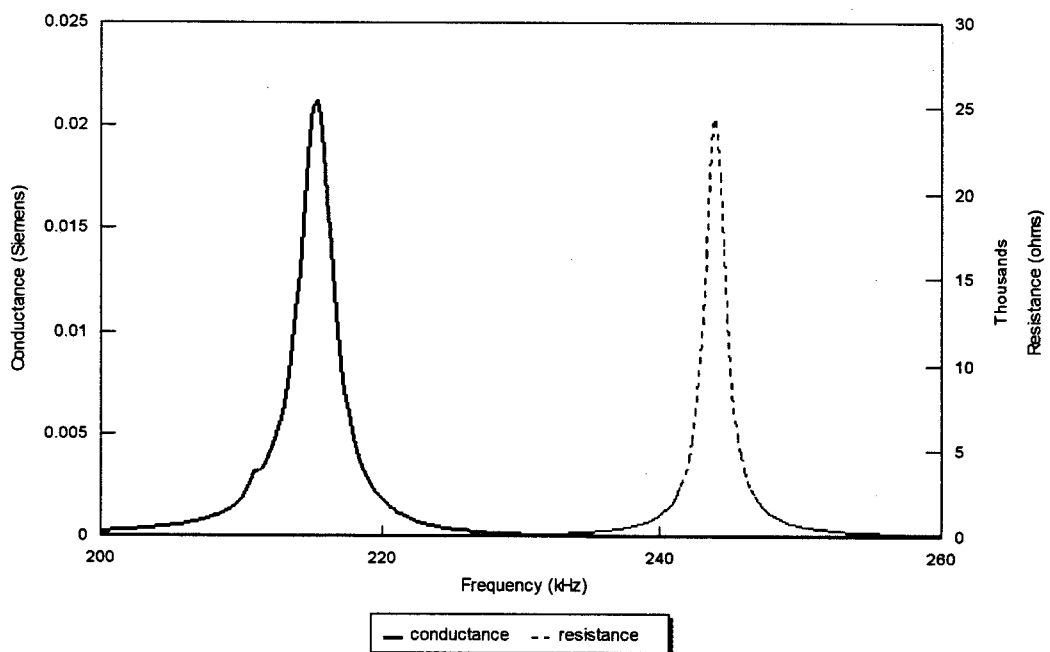


Figure 4. Example radial resonance curve for soft PZT sample KMR/182/2, measurement performed by laboratory F, showing conductance and resistance against frequency.

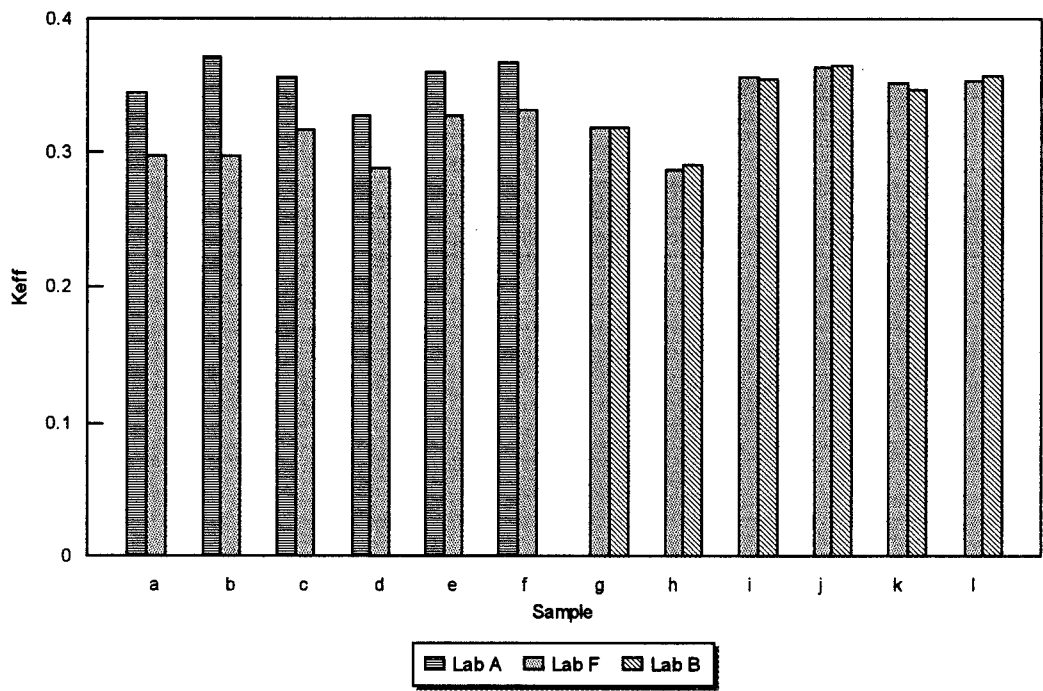


Figure 5. k_{eff} calculated using equation (1) using radial resonance measurements on the Hard PZT samples.

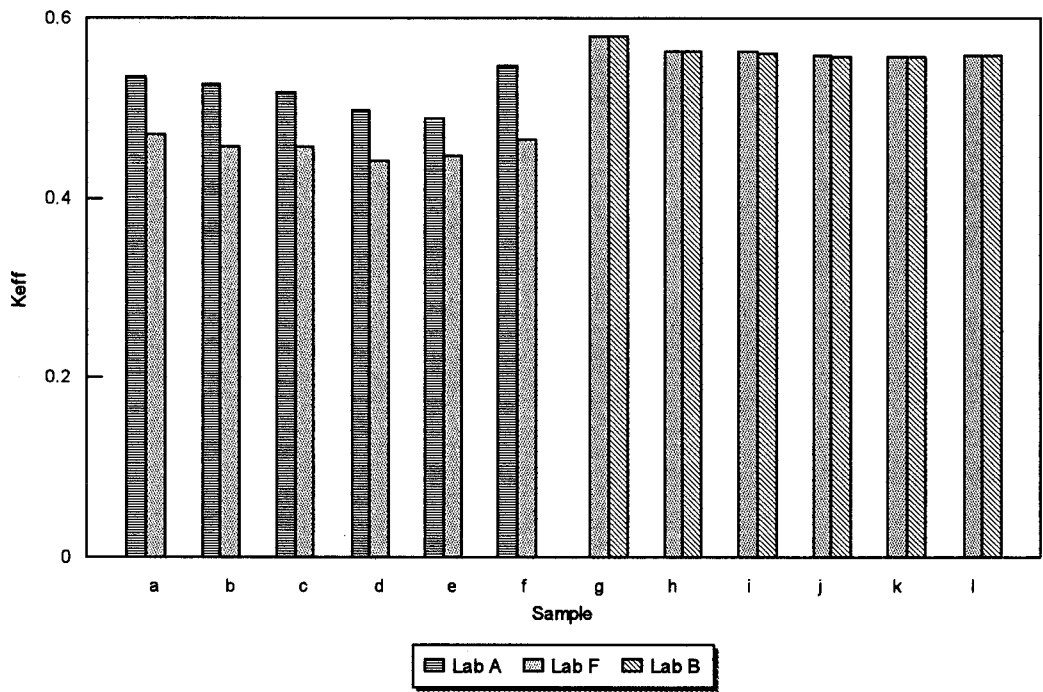


Figure 6. k_{eff} calculated using equation (1) using radial resonance measurements on the Soft PZT samples.

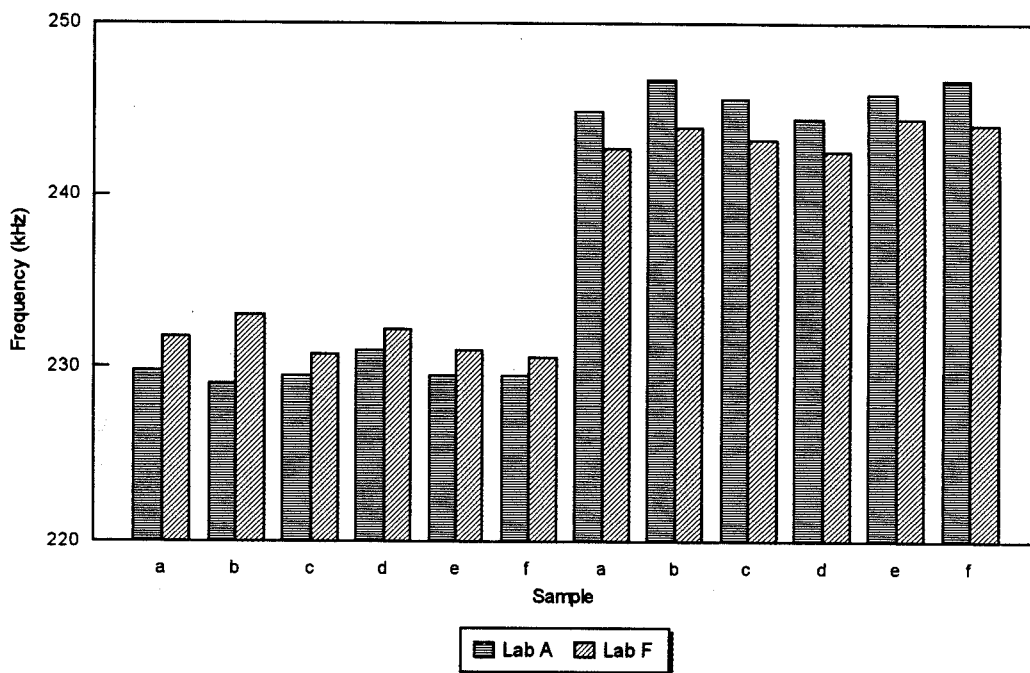


Figure 7. Radial resonance and anti-resonance frequencies for the same hard PZT samples measured by laboratories A and F.

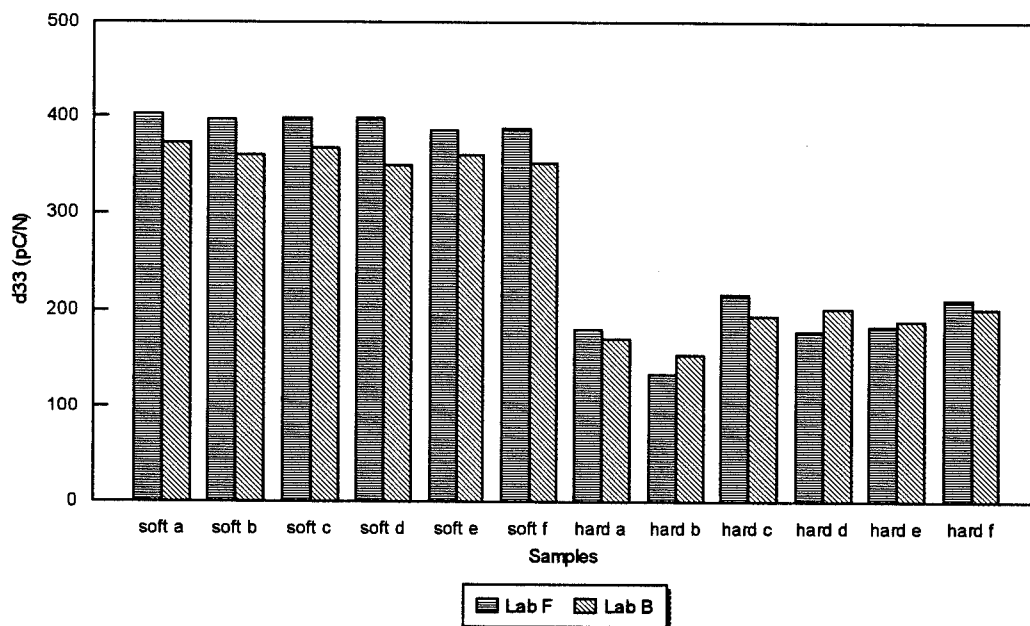


Figure 8. Berlincourt d_{33} measurements on the same hard and soft PZT samples measured by laboratories B and F.

APPENDIX

Piezoceramic Measurement Round Robin Exercise

General

Two materials are supplied for the tests, one a hot pressed soft piezoceramic labelled KMR and the other a conventional dry pressed hard type, labelled KMM. There are six samples of each nominally 1mm thick, 10mm diameter disks.

For each sample measure the thickness and diameter, and then either the mass or density. Also measure the room temperature and humidity at which the tests were performed, and if the sample temperature is significantly different also give the sample temperature. For the low stress level tests, i.e. the dielectric and impedance measurements keep the applied voltage below 4V/mm. Samples that have been used for the low stress level tests can then also be used for a high stress test (displacement-field, or polarisation-field), but not vice versa.

Where possible return the results in the form of computer output in an ASCII format file, on PC format disks.

Dielectric Measurement

Measure the capacitance and dissipation factor at 1kHz, at temperatures from room temperature up to 300 degrees C at intervals of 10 degrees C.

Resonance

The disc shaped samples can be made to resonate either radially or in the thickness mode. Measure the amplitude Z and phase angle θ of the complex impedance at these resonances in order to determine the resonance and anti-resonance frequencies. Please give an indication of the sample holding and contacting methods used in each case. Also what formula (and maybe further tests) would you use to determine the k_t , k_p , and mechanical Q factor from these samples.

Displacement Field

Apply a sinusoidal voltage of 5kV peak to peak across sample @ 5Hz and 500Hz and measure the displacement or strain in the 3-3 direction. For each experiment give the applied voltage and strain or displacement for 1 cycle at 200 equally spaced time intervals. If the acquisition rate is not fast enough for 200 steps then sample as many as possible or return the analogue output graphically.

The voltage reported should be the voltage across the sample and not the input to a voltage amplifier. Also check that this voltage is applied across the sample and that there are no stray contact resistances or short circuit paths. Similarly for the displacement measurement, since it is usual to measure the displacement at one point make sure that this represents the change in length, i.e. the back face of the sample remains stable. The most effective sample holding technique we have found is using a brass plate with holes for a vacuum on the rear face. This provides a stable surface and should leave the sample free to move in the 1-3 direction.

If using a fibre optic probe to measure displacement calibrate the transducer for every sample (i.e. normalise the output at peak position) and ensure the probe is perpendicular to the target.

Polarisation-Field

Apply a sinusoidal voltage of 5kV peak to peak across sample @ 5Hz and 500Hz and measure the current through the sample. For each experiment give the applied voltage and current for 1 cycle at 200 equally spaced time intervals. If the acquisition rate is not fast enough for 200 steps then sample as many as possible or return the analogue output graphically.

Give a description of the measuring circuit and where possible convert the results to charge density and remove the capacitive and resistive terms.