

NATIONAL PHYSICAL LABORATORY

Centre for Materials Measurement and Technology

**EC SMT Project, 'CERAMELEC'
WP10 - Electric Strength**

Final Report (August 1999)

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SUMMARY

This report covers the activities within WP10, which had the objective of reviewing electric strength tests for modern ceramic materials. The background to undertaking the work is described, together with the experimental approach employed. Using an alumina substrate product and two PZT piezoelectric materials, various factors associated with the geometry and production method for the specimen were investigated. Statistical evaluation of breakdown data using the well-known Weibull distribution was made to establish the usability of the method for electric strength. The following conclusions have been reached.

- Thin planar test-pieces ranging in thickness between 1.0 and 0.25 mm and with electric strengths exceeding 100 kV/mm can be electroded and tested without difficulties of edge tracking or flashover when tested in transformer oil;
- Differences in electric strength can be systematically resolved using sufficient test-pieces, and examples have been obtained indicating electrode area effects, thickness effects, and machining/annealing effects;
- Dimpling test-pieces is unnecessary to prevent edge flashover; producing small dimples in thin test-pieces is not a simple task and requires specialised precision equipment;
- Test results from nominally identical samples have been found to possess a variation in electric strength which is expressible in terms of the two-parameter Weibull distribution;
- The quality of thin evaporated electrodes must be such that breakdown positions are uniformly distributed over the surface of the electrode;
- Concerns that breakdowns will occur preferentially around the periphery of the electrodes in planar test-pieces as a result of electric stress concentrations have proved to be unfounded.

For testing advanced ceramic products, it is possible to devise a suitable standard with greater flexibility than the existing method given in IEC 60672-2 for traditional ceramics. A draft standard will be submitted to CENELEC.

Areas that have been identified for further work include needs for quantification of the quality of applied electrodes and of the role of electrode area, and for identification of whether the Weibull distribution concept can be extended to electric withstand tests.

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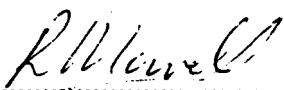
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1. INTRODUCTION

This report covers the results from Work Package (WP) 10 of an EC Standards, Measurement and Test (SMT) project 'CERAMELEC'. This workpackage had the purpose of evaluating approaches to electric strength measurement that are more relevant to modern uses of advanced ceramic insulating materials, specifically substrate and piezoelectric materials, than accounted for in IEC standard 60672, Part 2. This standard was originally devised in the 1970s to determine material properties for the purposes of applications as high voltage insulators, strictly as a means of product classification and minimum specification. The electric strength test incorporated into this standard involved the use of a 5 to 7 mm thick plate of ceramic material into which a spherical cap-shaped depression was ground using diamond tooling, reducing the thickness at the pole of the depression to 1.5 to 2 mm. The test-piece is coated with an electrically conducting layer inside the depression and also on the back face, and is then sandwiched between spherical electrodes. Two types of testing regime can be applied. Either the applied voltage is ramped until the test-piece conducts ('breakdown test'), or a given voltage can be applied for a given period of time ('withstand test'). The former is used to determine the ultimate performance of the material, the latter maybe used as a pass/fail test for a specification.

A number of problems arise with this test:

1. Machining a depression can introduce flaws which may influence the result of the test compared with an annealed or green-state formed test-piece;
2. Green-state direct forming the test-piece by pressing may result in different properties in the material beneath the depression than surrounding it as a result of pressure gradients in pressing, so testing specially shaped test-pieces may not be realistic;
3. Many modern materials are not produced in a sufficient thickness to machine such a test-piece; for example, one of the most widespread products is thin ceramic substrates, which may be as thin as 0.2 mm or even less for some applications. It is well known that electric strength is a function of test-piece thickness (Moulson and Herbert (1990)), but no studies have been performed to compare the performance of such materials with conventionally sized test-pieces to guide the user;
4. Dielectric breakdown is a statistical event; breakdown is thought to be initiated at strong electric field concentrations, such as those occurring within pores or cracks, or even grain boundaries. The tested volume of the material will play some role in determining the risk of breakdown, but existing tests use only 3 to 5 test-pieces for general quality purposes. Statistical assessment is therefore impossible from such QA tests. It is however desirable to make such assessments if the resulting information is to be used for design purposes.

Alternative test methods are available for 'sheet material' (generally interpreted as being for compliant polymeric materials), including IEC 243, which requires dissimilar sized (25 mm and 75 mm diameter) solid coaxial cylindrical electrodes to be applied to opposite faces of the test material, and ASTM D149, which employs similar sized (25 mm diameter) solid cylindrical electrodes (see for example the discussion by Nelson (1983)). These methods are not

completely suitable for thin ceramic substrates since these are neither flat nor compliant, and might readily be cracked simply by squeezing between cylindrical electrodes.

The purpose of WP10 is thus to examine the behaviour of typical advanced ceramic materials in different thicknesses and to evaluate the role of various test-piece preparation, coating and testing parameters on a statistical basis in order to provide background information for the preparation of a standard more appropriate to substrate-like products than the existing method in IEC 60672.

2. TEST EQUIPMENT

2.1 Facility

In order to perform electric strength measurements safely at the voltages, a testing device is required which is designed to operate within an interlocked cabinet, such that no connections can be made with the internal wiring of the cabinet. NPL had previously purchased such a facility from SEFELEC, France, through the UK agents, Wessex Electronics Ltd, Bristol, UK. This equipment comprised a rampable high-tension (HT) DC or AC voltage generator beneath an insulated interlocked cabinet into which live and earth terminals protruded. The ramp function was controlled by a low voltage control signal from an external programming unit. The maximum rated voltage was 50 kV. The instrument was designed automatically to switch off the applied voltage when the breakdown current exceeded either 1 mA or 3 mA (3 mA was selected). The peak breakdown voltage level was retained on a display.

For making the breakdown test, a test-jig was built in polymethylmethacrylate which enabled a test-piece to be inserted between two spring-loaded opposing spherical metal electrodes, which were connected to the cabinet electrodes using metal braiding. This arrangement was immersed in transformer oil (Shell Diala BG, supplied by Senator Lubricants Ltd of Crawley, W Sussex, UK).

2.2 Measurement and calibration

The design of the commercial instrument had some shortcomings, some of which were only discovered during the course of this work:

1. The voltage level was indicated on a conventional front panel meter, which could not be read with accuracy. A digital voltmeter was integrated with the meter in order to provide a digital read-out and a permanent record through data-logging.
2. The instrument had no direct means of traceable calibration of the HT voltage. This problem was not solved during the course of the project. There is implicit reliance on the relationship between HT voltage and the external low-voltage meter reading provided by the manufacturer. It would have been unsafe (as well as invalidating health and safety legislation) to have brought HT leads out from the sealed environment in order to make such measurements with a high-voltage calibrated meter, or a low-voltage meter connected via a high calibrated resistance chain. The possibilities of using a battery operated meter viewed through the glass window of the cabinet were investigated, but time and cost prevented this approach from being pursued.

- 3 The ramping mechanism did not give a consistent or constant voltage ramping rate to breakdown. This was discovered by linking a data logger to the voltage indicator and reviewing its output during a test. Depending on the maximum voltage setting, at a given fraction of this setting, the ramp rate changed automatically. It was understood from the manufacturers that this was a safety precaution to avoid overshoot. Occasionally this interfered with a test series, some test-pieces breaking down while ramping fast, and others after the change of rate. Since the breakdown voltage under ramping conditions usually depends on the ramp rate, and hence the time under electrical stress, this could interfere with the statistical description of the results. The problem was not directly soluble, but when realised, its effect was minimised by setting the upper limit breakdown voltage at level such that the change in ramp rate was mostly clear of actual breakdown values.

2.3 Breakdown jig design

In order to apply a high voltage across the test-piece thickness, a test jig as shown in Figure 1 was designed and manufactured. The PMMA frame supported the two opposed spherical electrodes, which were 10 mm diameter. One of these electrodes was spring-loaded against the other so that a force was applied to the test-piece between them. This arrangement was immersed in a beaker containing transformer oil. All tests were performed under oil in order to prevent arcing around the test-piece edges.

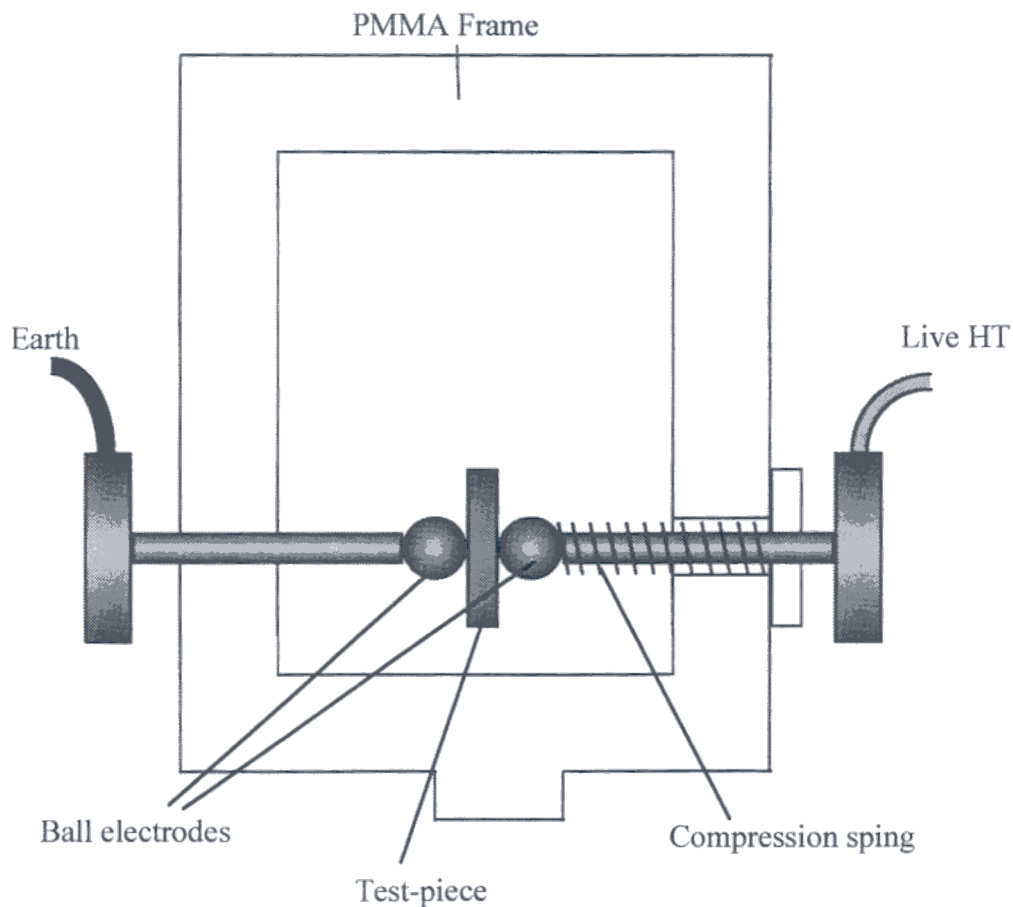


Figure 1 Test jig for applying high tension voltage to test-pieces.

3 TEST MATERIALS AND TESTPIECE PREPARATION

3.1 Test material

A variety of ceramic materials have been employed for this work, summarised in Table 1

Table 1 - Ceramic materials

No	Material	Code	Supplier	Dimensions, mm		No
				Lateral	Thickness	
1P	Alumina	?	Agate and General	20 Ø	1	14
1A				20 Ø	1, dimpled to 0.3	5
1B				20 Ø	1, dimpled to 0.2	5
1C				20 Ø	1, dimpled to 0.1	5
1D				11 Ø	1	?
2	88% alumina ⁺	Regalox	Royal Worcester	24 Ø	0.8	40
3	99.5% alumina ⁺	Deranox 995	Morgan Matroc	25 Ø	0.8	40
4	96% alumina	Rubalit 708 substrates	CeramTec UK	25 x 25	1.0	100s
				25 x 25	0.635	100s
				25 x 25	0.500	100s
				25 x 25	0.380	100s
				25 x 25	0.250	100s
5	PZT-5A*	KXB (NPL)	Morgan Matroc Transducer Products	10 Ø	1	100
6	PZT-4D*	KXC (NPL)		10 Ø	1	100
5U	PZT-5A**	KZAU (NPL)		10 Ø	1	20
6U	PZT-4D**	KZBU (NPL)		10 Ø	1	20

⁺ = machined from rod by sawing and lapping flat

* = supplied electroded and unpoled, ** = supplied unelectroded and unpoled

Initial tests were made on thin discs supplied by Agate and General in flat and dimpled condition, which were prepared from bulk material. Additionally, test-pieces were cut from 25 mm diameter 88% and 99.5% alumina ceramic rods made by extrusion, and then lapped flat. The 88% alumina behaved quite well, but gave a significant scatter in results. The 99.5% alumina was found to have internal cracks, and these samples broke down at very low voltages. In addition, a series of tape-cast and laser diced 25 mm square, 96% alumina substrates from CeramTec (UK) Ltd, of Colyton, Devon was obtained in a variety of thicknesses, which were tested with as-fired surfaces.

In order to apply the test method to a high permittivity material, some hard lead zirconium titanate (PZT) discs were purchased from Morgan Matroc Ltd, Transducer Products Division, UK, in both electroded and unelectroded conditions.

3.2 Test-piece preparation

Individual pieces were either used in the as-received condition, or after grinding a spherical cap depression, the latter with and without subsequent annealing at high temperature in order to heal grinding damage.

In order to machine the depression, 10 mm diameter ball-ended electroplated diamond grinding tools were purchased from Diagrit Ltd of Stapleford, Kent. The mounting of the tool was specially designed to be accommodated in the high-speed horizontal spindle attachment of a Jones and Shipman toolroom grinder, operating at typically 10 000 rpm. In order to rotate the test-piece under the grinding ball, a milling machine rotary dividing head placed on the surface grinder bed was used as the test-piece support. The axis of the dividing head was visually aligned with the centre of the grinding ball using a lathe centre placed in the centre of the dividing head bed as a marker. The grinding machine bed was then clamped into position. A special jig was made to permit substrate test-pieces to be clamped onto the dividing head (Figure 2). The required depth of depression was achieved by slowly lowering the wheel-head of the grinding machine down into the test-piece while this was rotated using the hand-wheel. Using this arrangement, specimens could be quickly machined to reasonably repeatable depths. Initially, this procedure led to the possibility that a pip was left in the centre of the ground depression because of difficulties in accurately positioning the axis of rotation of the dividing head. Consequently, for later batches the dividing head was not rotated, at the risk that the dimple was not perfectly round.

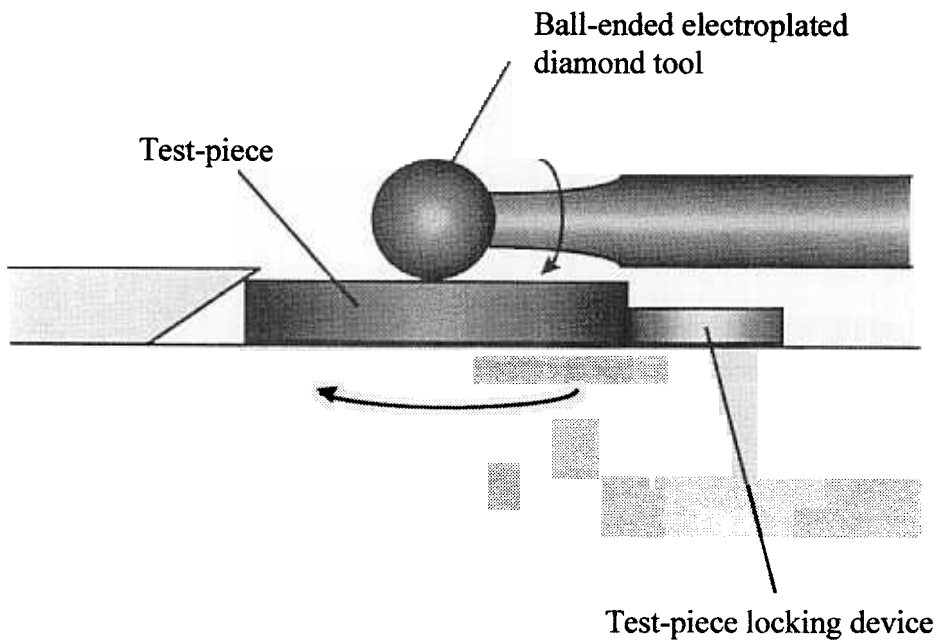


Figure 2 Arrangement for grinding depressions into test-pieces.

All test-pieces, whether machined or not, were cleaned in solvents (5 min ultrasonic cleaning immersed first in acetone and then IMS) and dried in a vacuum oven before applying the conducting coating. In order to apply the coating to a limited area of each test-piece yet process a large number of test-pieces in batches, a method of masking was required. A polymethylmethacrylate jig was designed to accommodate a number of specimens. Each

specimen position incorporated a mask on each face with holes in them of the required electrode size, typically 5 to 20 mm in diameter (Figure 3). The holes were well countersunk in order to prevent shading near the hole edges, which would otherwise mean variable thickness coatings. Coatings were of aluminium applied by evaporation. The jig was double sided, permitting test-pieces to be coated on both faces while ensuring that the electrodes were coaxial, which was achieved simply by turning the jig over and repeating the evaporation process.

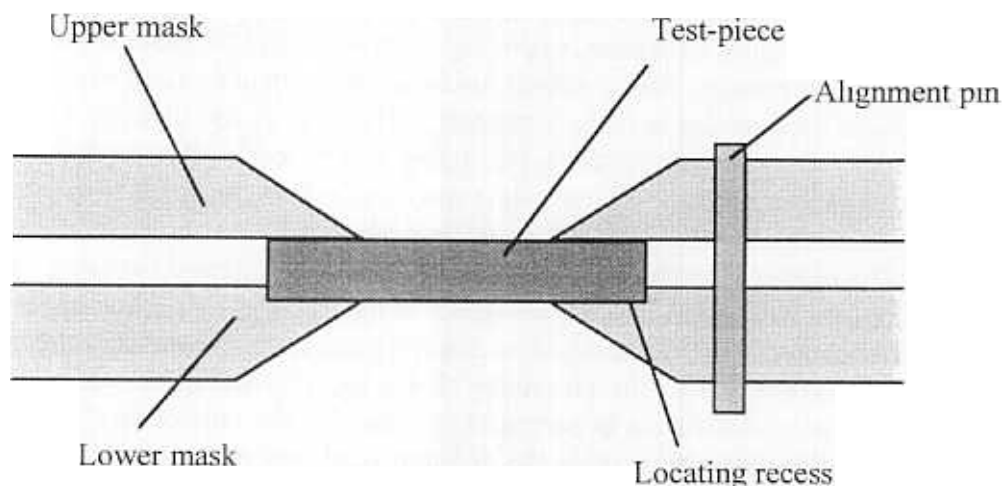


Figure 3 Schematic of mask for defining the electrode areas on the test-piece faces.

Test-piece thicknesses were measured using a digital dial gauge reading to 0.001 mm and equipped with a ball-ended sensor in order to locate the bottom of the depressions in dimpled test-pieces.

3.3 Analysis of data

3.3.1 Weibull analysis

It is well recognised that dielectric breakdown is a stochastic process that has some parallels with mechanical strength testing in brittle materials, i.e. the weakest link is the one that ultimately fails. In the case of mechanical strength, the largest flaw in the highest stressed position is the most likely site of failure. A key element of this research was to investigate the applicability of Weibull statistics to electric strength measurements, as suggested by Wilkens (1987).

The Weibull distribution, in its simplest two-parameter form is given by:

$$P_f = 1 - \exp\left(-\left(\frac{\sigma}{\sigma_0}\right)^m\right)$$

where P_f is the cumulative probability of ranked results, σ is the strength, σ_0 is the characteristic strength, and m is the so-called Weibull modulus, which is a characteristic of the width of the distribution. To visualise the distribution, the series of test results are ranked in

ascending order, assigned a probability of failure P_f (see below), and are plotted as $\ln \ln (1/(1 - P_f))$ vs. $\ln \sigma$. The distribution should then approximate to a straight line. The fitting parameters m and σ_0 can be obtained either by a least-squares fitting routine of a regression line to the plot, or by the preferred maximum likelihood method (as used in ENV 843-5 for mechanical strength testing). For simplicity, this report uses primarily the least squares method using the probability ranking parameter:

$$P = \left(\frac{i - 0.5}{n} \right)$$

where i is the rank number in ascending order and n is the total number of test data. Other ranking parameters are available with varying degrees of complexity, but the one given above has only a small bias, and is recommended in ENV 843-5.

In order to compute Weibull parameters for the batches of test results, a routine was developed using an Excel spreadsheet designed for the purpose. The parameters were fitted as a straight line relationship (i.e. two-parameter). In some cases parallel calculations were made using the maximum likelihood method. Often the results were rather different, because this second method places less emphasis on the shape of the extremes of the distribution.

3.3.2 Reliability and significance of Weibull parameters

The consistency of determining Weibull parameters depends on the sample size. The larger the number of test data, the more reliable the fit. For small numbers of test data, the computed parameters become much less reliable. The 90% confidence interval on m becomes quite large, e.g. about $\pm 20\%$ for $n = 20$, and $\pm 40\%$ for $n = 10$. In contrast the computed values of mean strength $\bar{\sigma}$ and characteristic strength σ_0 have much smaller widths of confidence interval, typically less than 5% for small numbers of test-pieces ($10 < n < 25$)¹.

3.3.3 Breakdown position

After breakdown, breakdown positions were clearly visible, and were measured relative to the centre of the electrode. It was of particular interest to evaluate whether the breakdown occurred predominantly around the edge where a field concentration might be expected, or randomly over the area of the electrode. If the breakdown positions are random, it would be expected that there would be a single value of probability of breakdown per unit area, p . The cumulative probability of failure P in an annulus of radius r and width dr is thus $2\pi pr dr$. Integrating this over the area of the electrode, the total probability of failure to radius r is thus:

$$P = \int_0^r 2\pi pr dr = \pi pr^2$$

Since $p = 1$ for $r = a$, the radius of the electrode, this reduces to:

¹ Confidence intervals have been computed using Monte Carlo simulation techniques with repetitive random sampling of an infinite population of Weibull distributed data, evaluation of apparent Weibull parameters for each sample, and evaluation of the resulting distribution of estimated values.

$$P = \left(\frac{r}{a}\right)^2$$

Thus, plotting the cumulative probability of failure against (normalised radius)² should produce a straight line through the origin with a slope of unity. Failure to approximate to this would imply that the breakdown positions are not randomly distributed, but are concentrated in some way indicated by the plot.

4 TEST RESULTS

4.1 Introduction

The full series of tests undertaken in this project is summarised in Annex A. They are roughly in the sequence undertaken but are grouped for comparative purposes.

4.2 Initial experiments with electroding

The objective was principally to determine how large the electrodes could be without edge flashover being incurred. The Agate alumina, Material 1, was employed for this work. A small series of tests was performed using various sizes of adhesive tape mask applied to both sides of the test-pieces before evaporating a thin layer of aluminium. The results of breakdown tests (Annex A, A1) are shown in Figure 4. Within the scatter of results there is a clear trend of declining breakdown voltage gradient with increasing electrode area (as might be expected from a probabilistic viewpoint), but in no cases did edge flashover occur. This implies that for a 1 mm thick test-piece, electrodes can be placed at least to within 2.5 mm of the edge of the test-piece.

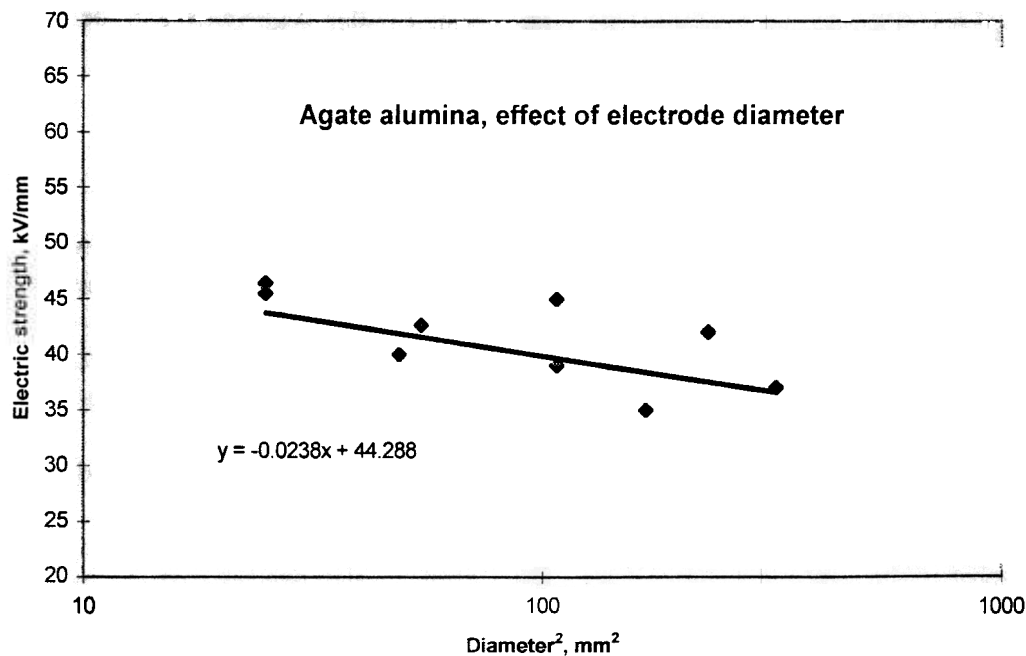


Figure 4 - Effect of electrode area on electric strength for individual test-pieces of Agate alumina.

A larger set of test-pieces with and without dimpling was then tested using the largest electrode size (15 mm diameter). The results are summarised in Annex A, A.2, and are plotted in Figure 5 as Weibull distributions. It can be seen that there is a systematic slope to each of the plots, and, despite the small number of test-pieces with various depths of dimpling, there is a clear increase in breakdown voltage gradient with decreasing residual thickness of material.

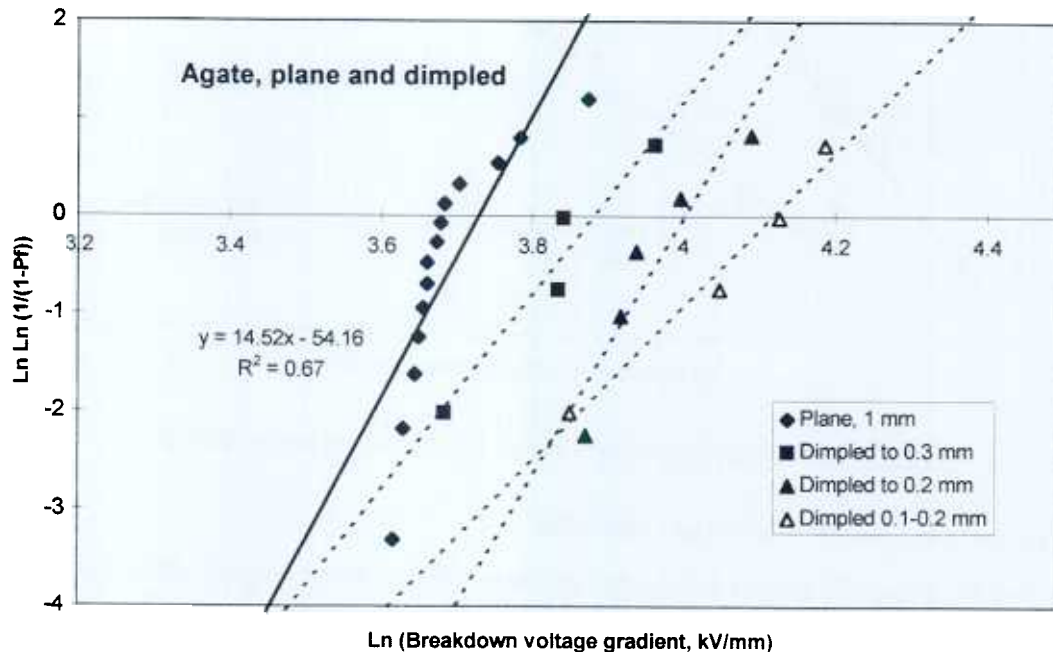


Figure 5 - Weibull distributions for the Agate alumina with and without dimpling. Note that no attempt has been made to fit two separate lines to the plane test-piece results because within the random nature of results the presence of the apparent inflection cannot be taken to imply a genuine bimodal distribution.

4.3 Tests on Royal Worcester alumina, material 2.

Like the Agate alumina, these test-pieces were machined from solid rod by slicing and grinding. Preliminary tests showed that there were no problems in using 15 mm diameter electrodes, and subsequently, batches of test-pieces before and after annealing at 1000 °C were electroded and tested. The objective of this study was to establish whether the grinding routine reduced electric strength through the damage introduced.

The results are summarised in Annex A, A.3, and are plotted as Weibull plots in Figure 6. It is immediately clear that, contrary to expectations, the annealed test-pieces have rather lower electric strength than the as-machined test-pieces, and show a much wider distribution of strengths. Despite this it is clear that the test can readily distinguish the performances of two batches of differently treated materials. No edge breakdowns occurred.

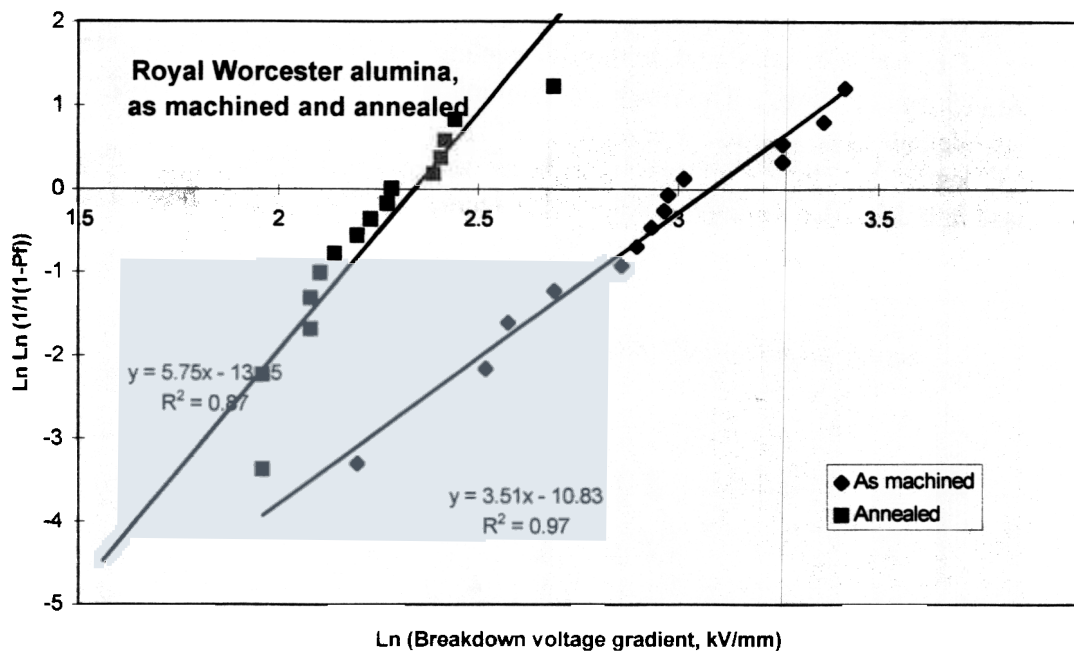


Figure 6 - Weibull distributions for Royal Worcester material

4.4 Tests on Deranox 995 alumina, material 3

The first few tests on this material showed very low breakdown strengths. On examination of the test-pieces it was found that there were cracks in the central region. These must have originated from the original ceramic processing, which like material 2 was by extrusion. No further tests were made.

4.5 Tests on CeramTec alumina

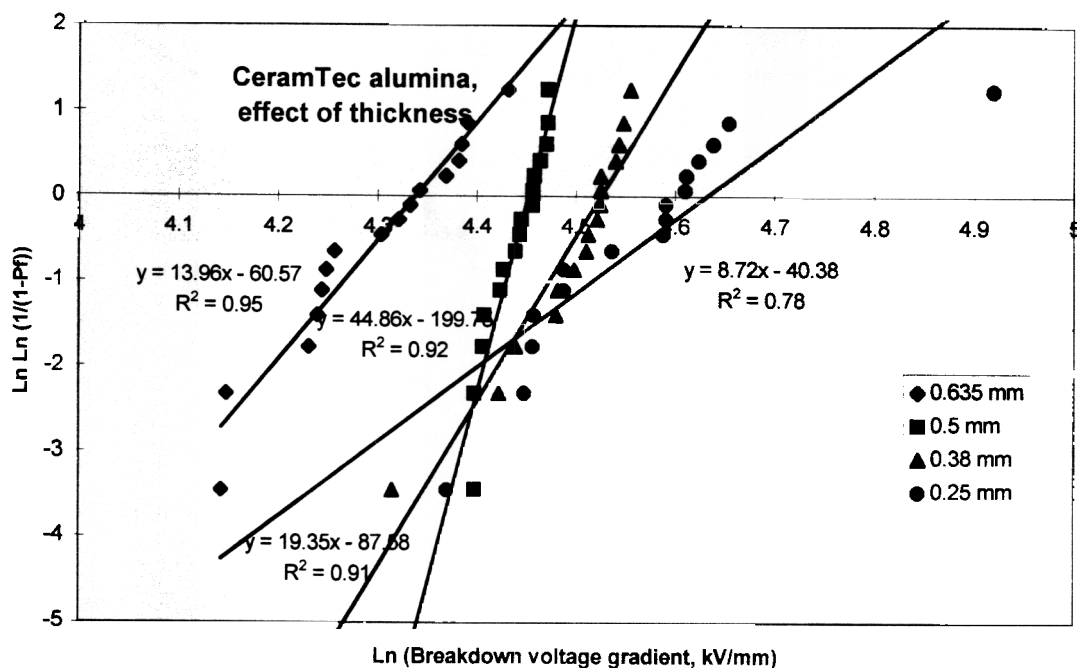
4.5.1 Effect of test-piece thickness

A series of batches of test-pieces manufactured in different thicknesses by tape casting were coated with 14.5 mm diameter electrodes, and tested under the same conditions (Annex A, A.6, and summarised in Table 2). The average electric strength systematically declines with increasing thickness, but is very high, much higher than for materials 1 and 2. A consequence of this was that the 1 mm thick material was outside the kV range of the Sefelec instrument, and thus could not be tested. The Weibull modulus appeared to vary somewhat from thickness to thickness, but not in any systematic manner (Table 2, Figure 7). It was noted also that there were some large differences between the values computed using the least squares method which gives equal weighting to each point, and the maximum likelihood method, which is rather less biased by the extremes of the distributions. The 0.5 mm thick material was particularly consistent in its behaviour, and the results for this batch lay outside the 90% confidence intervals for the other thicknesses. However, a repeat test (Test 11) gave much lower values (Table 3).

Table 2 - CeramTec alumina, Weibull statistics, effect of thickness

Test no.	Ceramic thickness, mm	No. of tests	Weibull modulus				Electric strength	
			Least squares	Max. likelihood	Upper bound ¹	Lower bound ¹	Mean kV/mm	Char. kV/mm
9	0.635	16	13.96	13.9	17.9	9.0	73.9	75.8
6	0.5	16	44.86	44.4	62.8	31.7	84.8	85.8
10	0.38	16	19.35	26.0	33.6	17.0	89.9	91.8
7	0.25	16	8.72	13.8	17.8	9.0	94.4	97.8

¹ Limits of the 90% confidence interval computed for the maximum likelihood method

*Figure 7 - Weibull plots for tests 6, 7, 9, and 10*

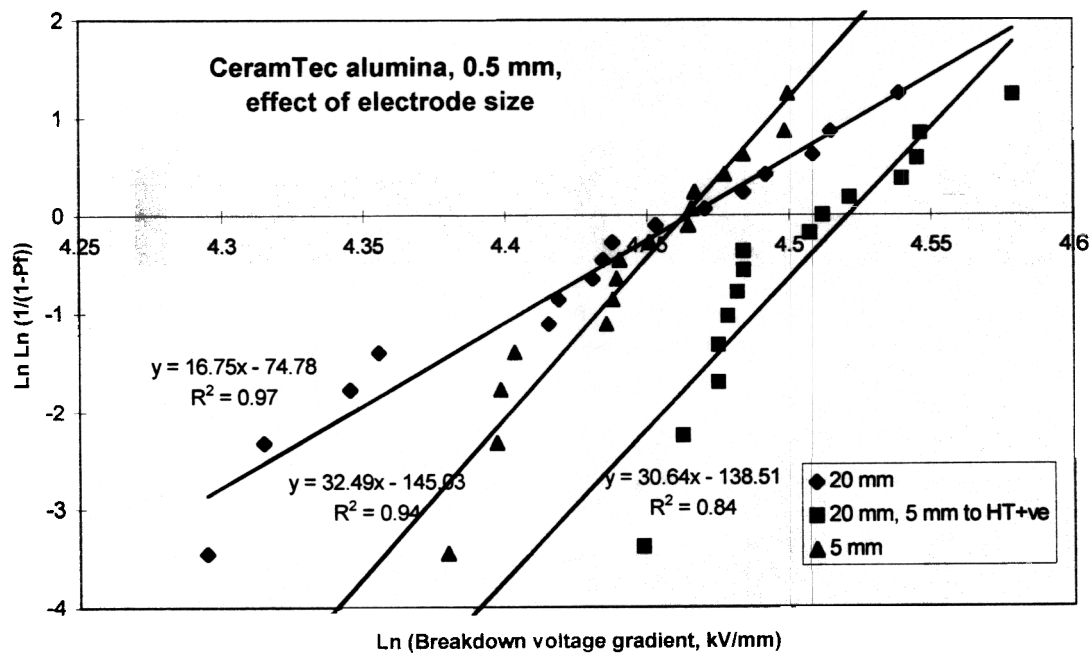
4.5.2 Effect of electrode size and quality

Similar tests were performed on the 0.5 mm thick material, but with different combinations of electrode sizes. The objective was to evaluate the effect of electrode size, and to determine whether one electrode could be much larger than the other. The results are given in Annex A, A.7, and are summarised in Table 3 and Figure 8.

Table 3 - CeramTec alumina, 0.5 mm thick, Weibull statistics, effect of electrode size

Test no.	Electrode diameters, mm	No. of tests	Weibull modulus				Electric strength	
			Least squares	Max. likelihood	Upper bound ¹	Lower bound ¹	Mean kV/mm	Char. kV/mm
6	14.5/14.5	16	44.9	44.4	62.8	31.7	84.8	85.8
11	14.5/14.5	16	18.4	17.8	22.9	11.6	83.7	86.1
12	14.5/5	15	30.2	24.5			90.3	92.0
13	5/5	16	32.6	20.9	27.0	13.6	85.4	87.8

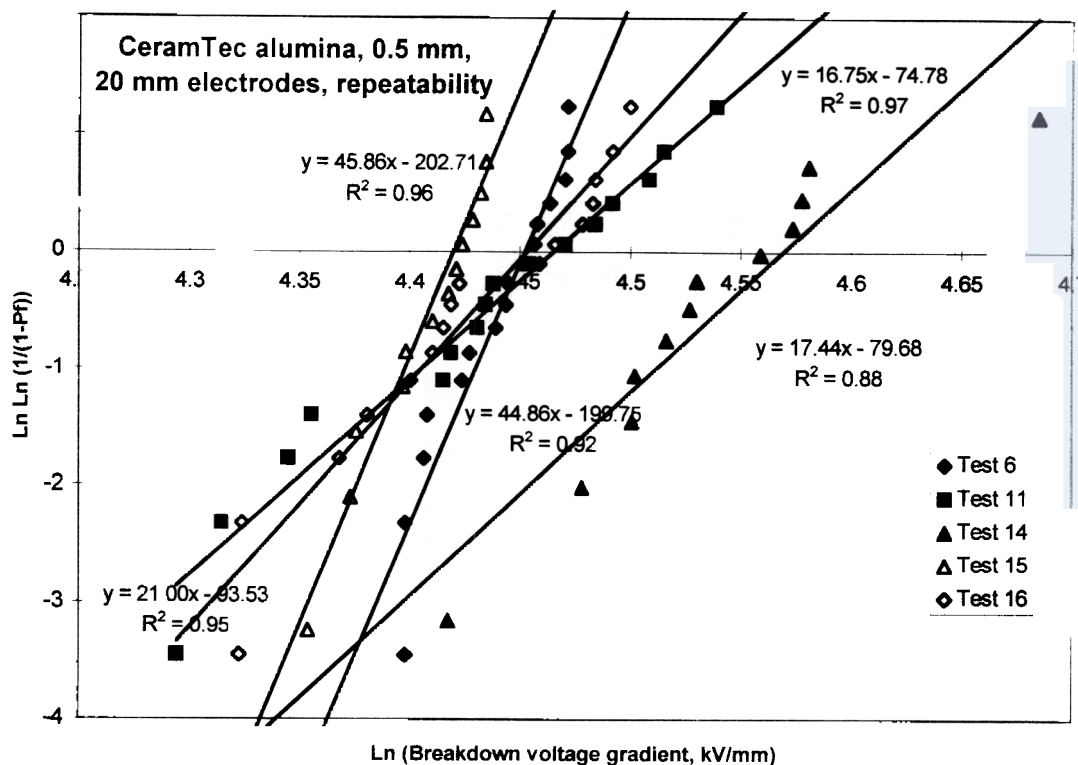
¹ Limits of the 90% confidence interval computed for the maximum likelihood method

*Figure 8 Weibull plots for tests 6, 11, 12, 13*

Because of the apparent differences between the distributions for nominally identical tests 6 and 11, further batches were electroded with 14.5 mm diameter electrodes and tested in the same way. Table 4 summarises the results from five batches, and Figure 9 shows the Weibull plots. In the case of Test 14, the electrodes were rather thin, and it became apparent that most of the failures were very close to the centre of the electrode, unlike the other tests in which there was a wider distribution of failure positions. The mean strength of this batch was also significantly higher than the remainder, which were closely grouped. Following from the results obtained from Test 14, the electrodes were made much thicker in Test 15, and the spheres were repolished to remove accumulated burn marks and to improve contact. Test 16 was then a further repeat.

Table 4 - CeramTec alumina, 0.5 mm thick, Weibull statistics, repeat batches

Test no.	Electrode diameters, mm	No. of tests	Weibull modulus, least squares	Electric strength mean, kV/mm
6	14.5/14.5	16	44.9	84.8
11	14.5/14.5	16	18.4	83.7
14	14.5/14.5	12	17.4	93.6
15	14.5/14.5	13	45.9	82.2
16	14.5/14.5	16	21.0	83.8

*Figure 9 - Weibull plots for tests 6, 11, 14, 15, and 16.*

Comparing the results from Tests 6, 11, 15 and 16, it is noticeable that although the mean strengths were very similar, and well within one standard deviation, the apparent Weibull moduli varied considerably. To ascertain whether this could be purely a result of random sampling, these data sets were combined and plotted as a single Weibull distribution (Figure 10). This distribution from 61 tests appeared to be fairly well represented by a two parameter Weibull distribution, and gave a Weibull modulus of 24.5. There is some curvature to the distribution, but this is considered not to be a significant factor.

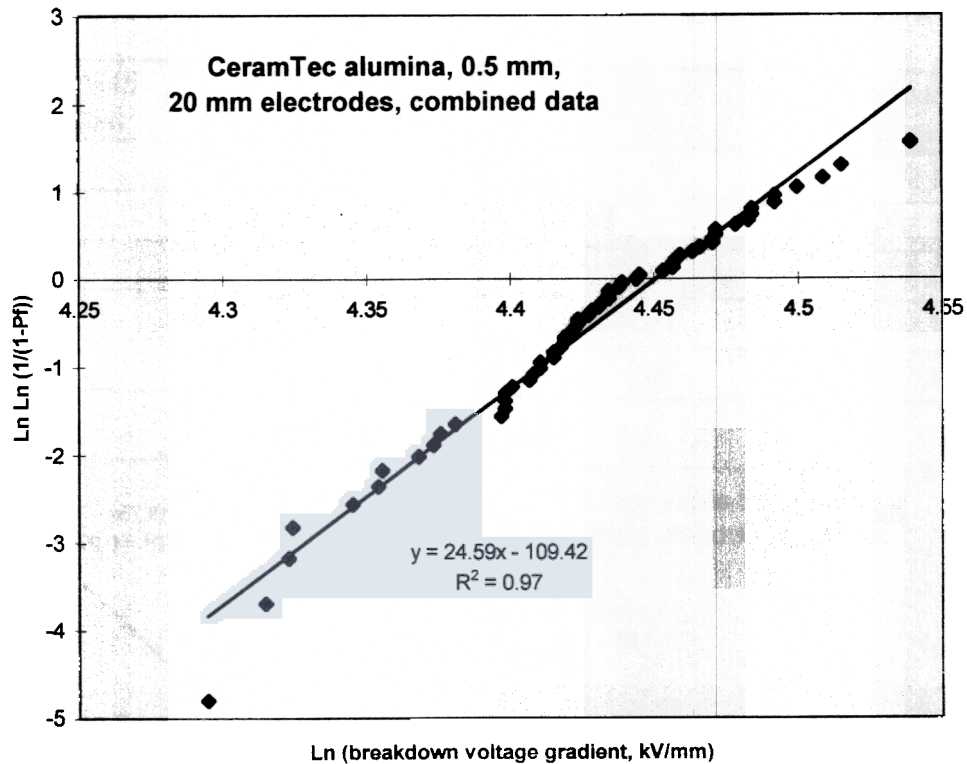


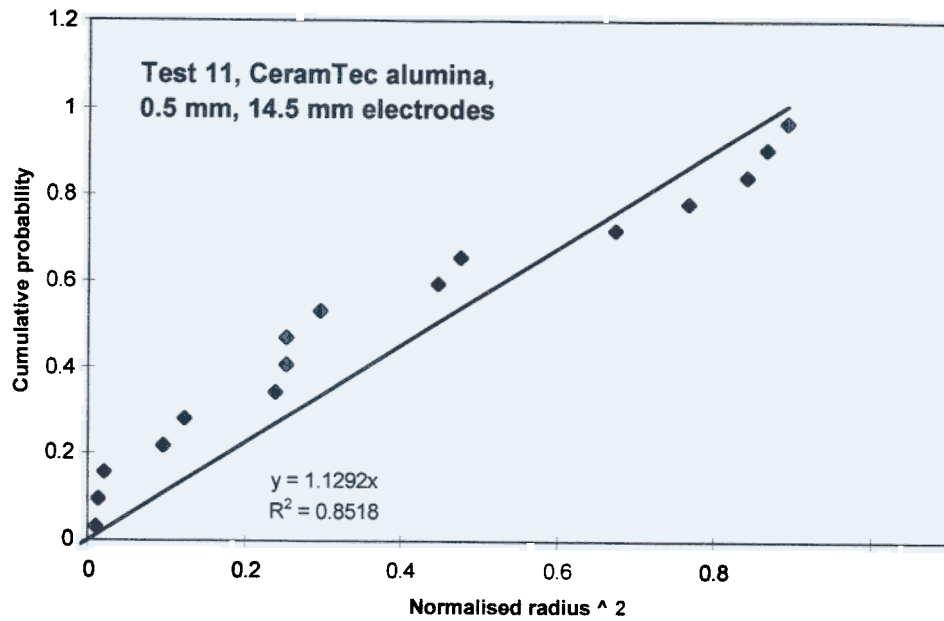
Figure 10 - Weibull plot for combined data set from tests 1, 11, 15, and 16

The distribution of breakdown positions was analysed for this series of tests, and the results are shown in Figure 11, with the numerical data being summarised in Table 5. It is clear that the poor electrodes gave failures highly concentrated near the electrode centre², whereas the remainder of the cases gave slopes close to unity, suggesting that the distributions were uniform. In none of these cases were the results grouped around the periphery of the electrodes, suggesting that the effect of any electrical stress concentration was negligible.

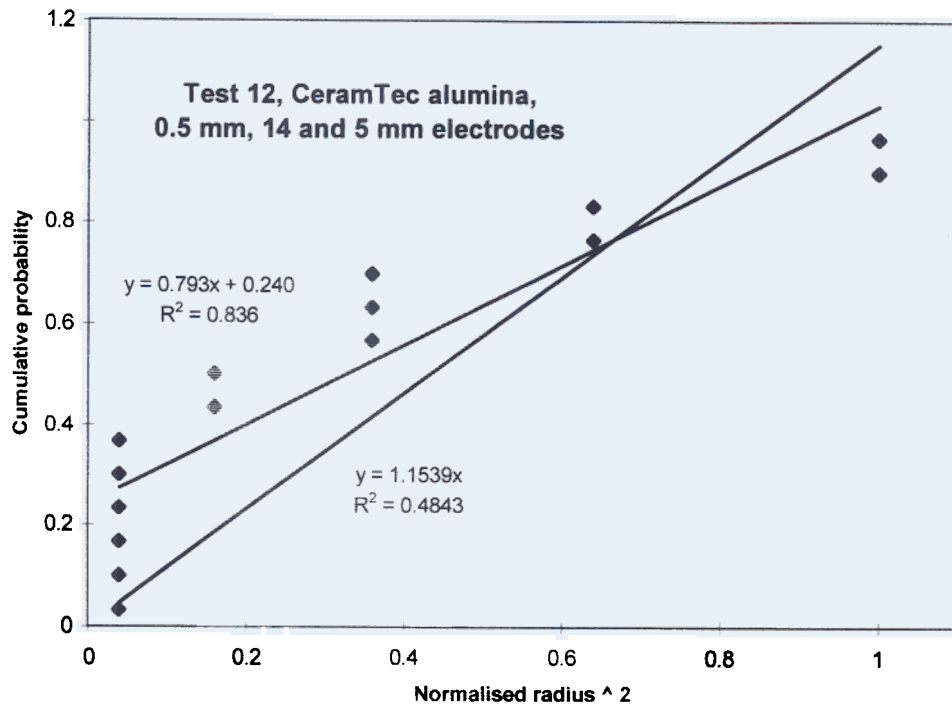
Table 5 - Distribution of breakdown positions

Test no.	Electrodes	Slope of cumulative probability plot	
		Forced through origin	Unforced
11	14.5/14.5	1.129	—
12	14.5/5	1.159	0.793
13	5/5	0.866	—
14	14.5/14.5 (poor)	7.08	—
15	14.5/14.5	0.936	—
16	14.5/14.5	0.908	—

² With plane test-pieces, there is no guarantee that the spherical electrodes were in contact with the centre of the evaporated coating area, nor that the evaporated electrodes themselves were coaxial. The distribution seen in Test 14 may well be controlled primarily by precise position of contact rather than true variations from the centre.

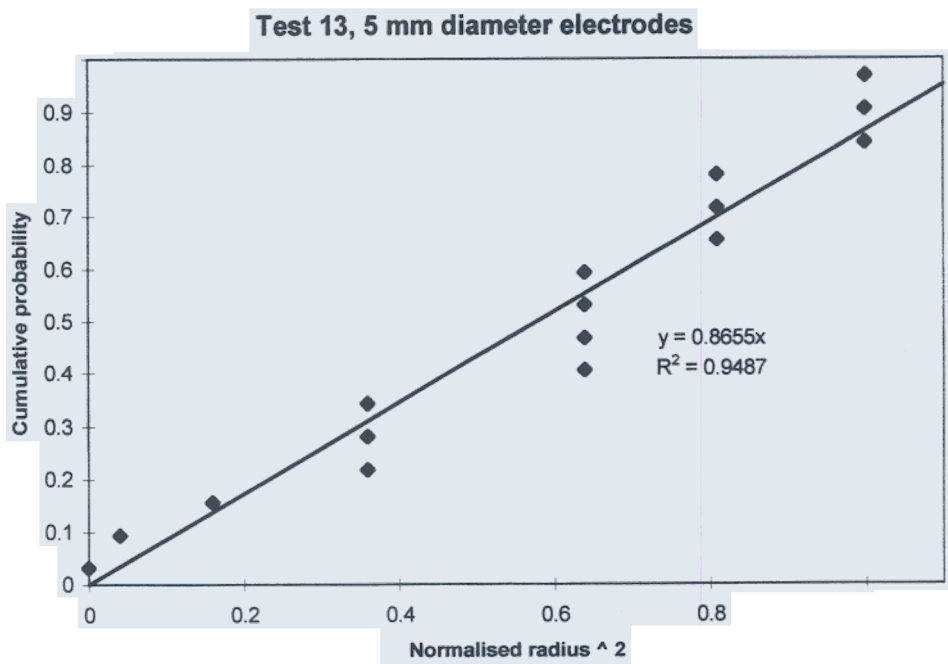


(a)

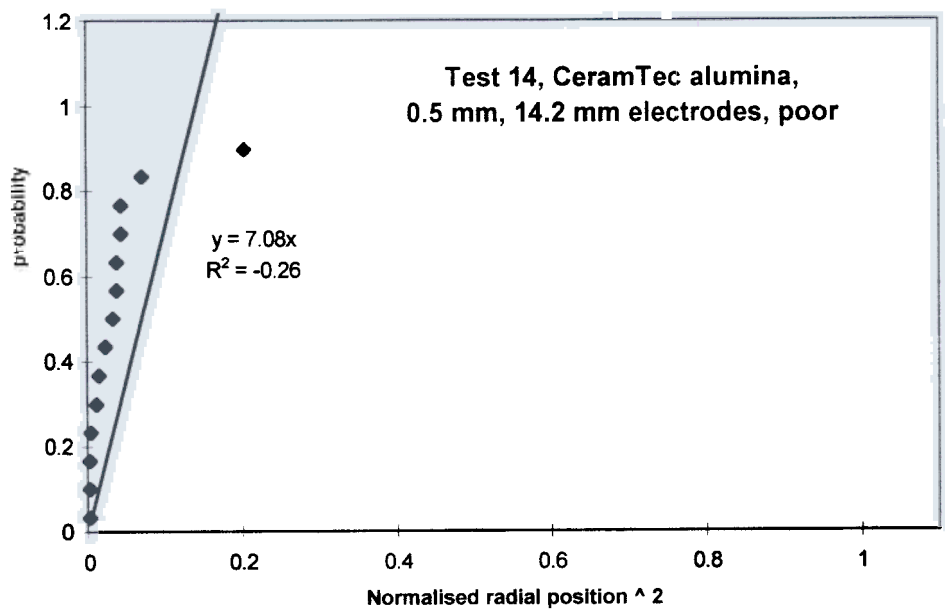


(b)

Figure 11 - Cumulative distributions of breakdown position vs. normalised radial distance squared for (a) Test 11 and (b) Test 12.

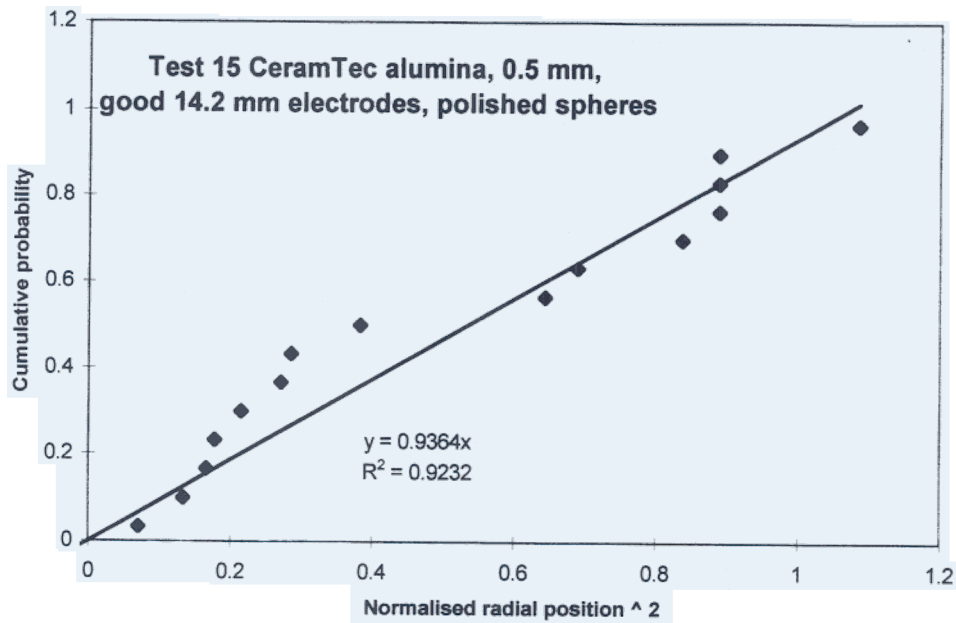


(c)

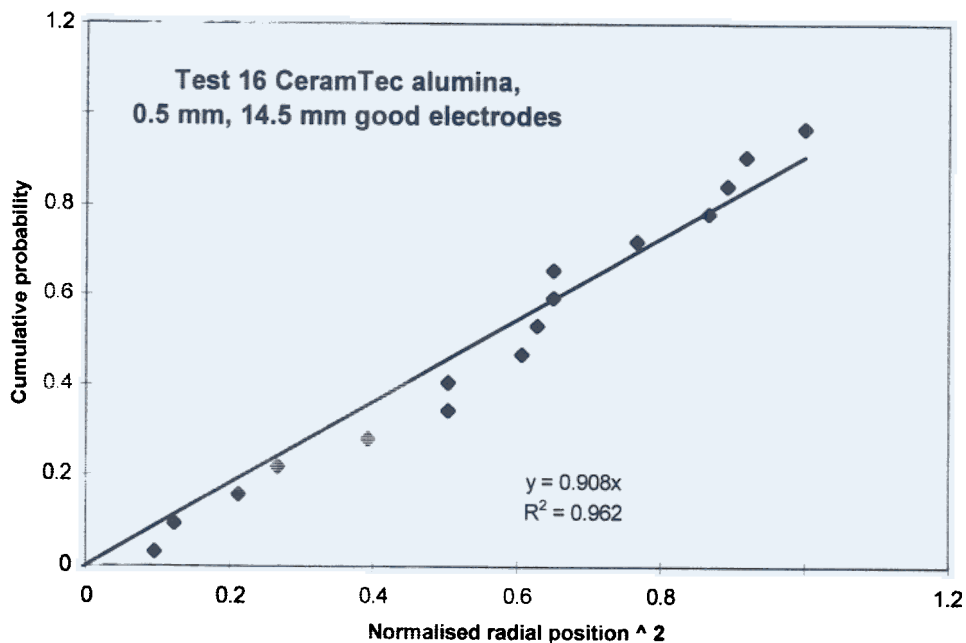


(d)

Figure 11(cont.) – Cumulative distributions of breakdown position vs. normalised radial distance squared for (c) Test 13, and (d) Test 14.



(e)



(f)

Figure 11 (cont.) – Cumulative distributions of breakdown position vs. normalised radial distance squared for (e) Test 15, and (f) Test 16.

4.5.4 Effect of dimpling

The objective of the tests was to ascertain the value of dimpling, whether machining damage incurred during dimpling could be alleviated by annealing, and whether the polarity of the applied voltage was significant or not..

One of the problems with undertaking a comparison of dimpled and non-dimpled samples is the selection of the starting material. In order to preserve the as-fired finish on one side of the samples, it was decided to use the 1.0 mm material, to machine the dimples so that the residual thickness was about 0.38 mm, and to compare these results with the 0.38 mm thick plane material. This means that there is the implicit assumption that the microstructures of the two thicknesses are similar (no visually discernable differences could be seen on polished cross-sections). The electrodes were applied with a diameter of 14.5 mm.

Two sets of tests were performed, 17 and 18 as given in Annex A, A.9 and A.10 and summarised in Table 6. Test 17 was preliminary with small numbers of test-pieces, primarily to discover how best to perform the dimpling, while Test 18 was a systematic series.

Table 6 - CeramTec alumina, Weibull statistics, dimpled and plane

Test no.	Dimpled/ Not dimpled	No. of tests	Weibull modulus, least squares	Electric strength Mean, kV/mm
17A	Dimpled	8	20.16	102
17B	Dimpled and rotated	10	18.3	108.8
17C	Plane	9	8.9	98.3
18A	Plane	29	17.6	92.6
18B	Dimpled, not rotated, +ve HT to dimple	15	16.4	110.7
18C	Dimpled, not rotated, annealed after dimpling, +ve HT to dimple	16	10.6	110.3
18D	Dimpled, not rotated, -ve HT to dimple	17	19.5	97.5
18E	Dimpled, not rotated, annealed after dimpling, -ve HT to dimple	15	15.0	96.5

Both test series showed that results from dimpled samples were of significantly higher electric strength than plane samples with approximately the same thickness (minimum thickness in the case of dimples. This is presumably because the effective electrode area at the minimum thickness was much smaller in the case of dimpled test-pieces than with plane test-pieces. Test 18 also shows that the polarity of the applied HT voltage is significant, with the dimpled side grounded relative to positive HT live giving a lower breakdown voltage than with the dimple to positive HT. The reasons for this are not clear, but since annealing, by comparison, produced very little effect, it has to be presumed that the proximity of the dimpled side to the grounded source of electrons is the main factor. Figure 12 shows the Weibull distributions for the five sets of data in Test 18.

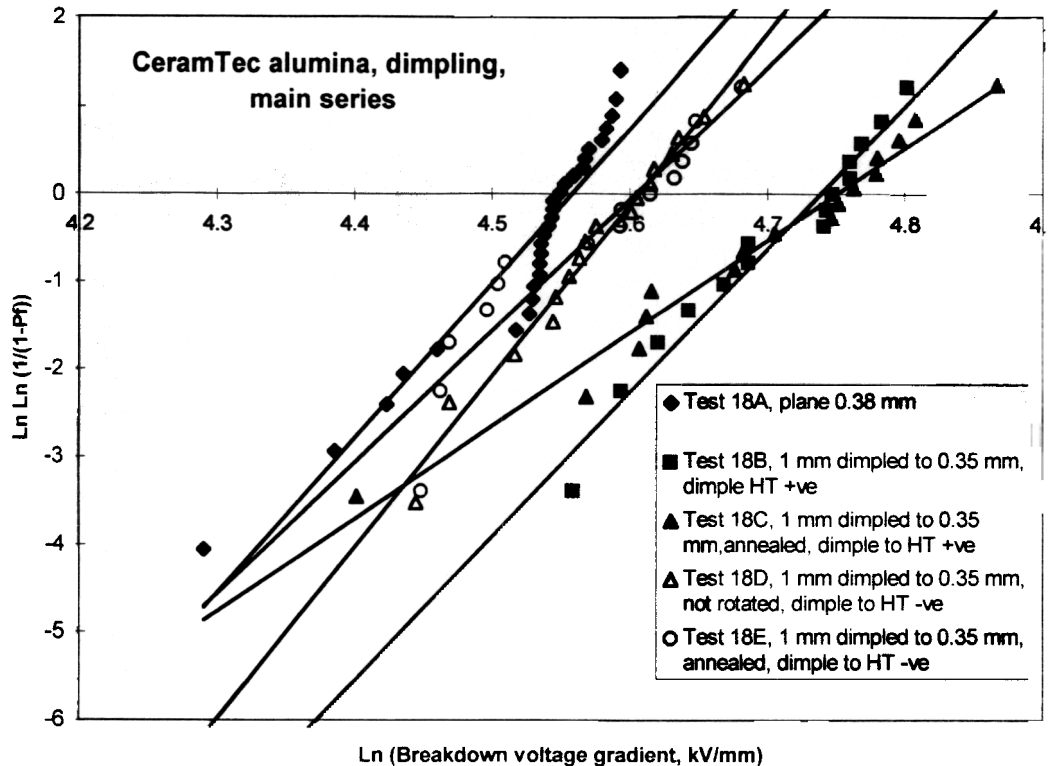


Figure 12 - Weibull plots for tests 18A, 18B, 18C, 18D and 18E.

4.6 Tests on PZT materials

Test 5 was made on the 10 mm diameter, 1 mm thick disc samples after removal of the supplied electrodes which covered the entire disc area, and application of evaporated aluminium electrodes of 5 mm diameter. Tests 5A (PZT-5A), and 5B and 5C (PZT-4D) were made on small numbers of test-pieces. It was found that the electric strength was typically 10 kV/mm, and no edge tracking breakdowns occurred. Test 5C used 1200 grit grinding on one surface before evaporating electrodes, but the results cannot be considered to be any different from those in the as-fired condition, from which it was deduced that internal defects controlled the electric strength. Subsequently, Test series 8A and 8B were made with larger numbers of test-pieces, and the Weibull distributions are plotted in Figure 13. For both materials, the Weibull modulus is rather low, about 5, compared with the CeramTec alumina, which is presumably a consequence of the manufacturing process.

Later, a further batches of the same materials were received from the supplier, this time without electrodes. They were electroded and tested in the same way (Annex A, A.12), and the Weibull distributions are shown in Figure 14. The Weibull moduli are slightly greater than obtained in the earlier tests, but the mean electric strengths are significantly lower. PZT-4D is still electrically stronger than PZT-5A.

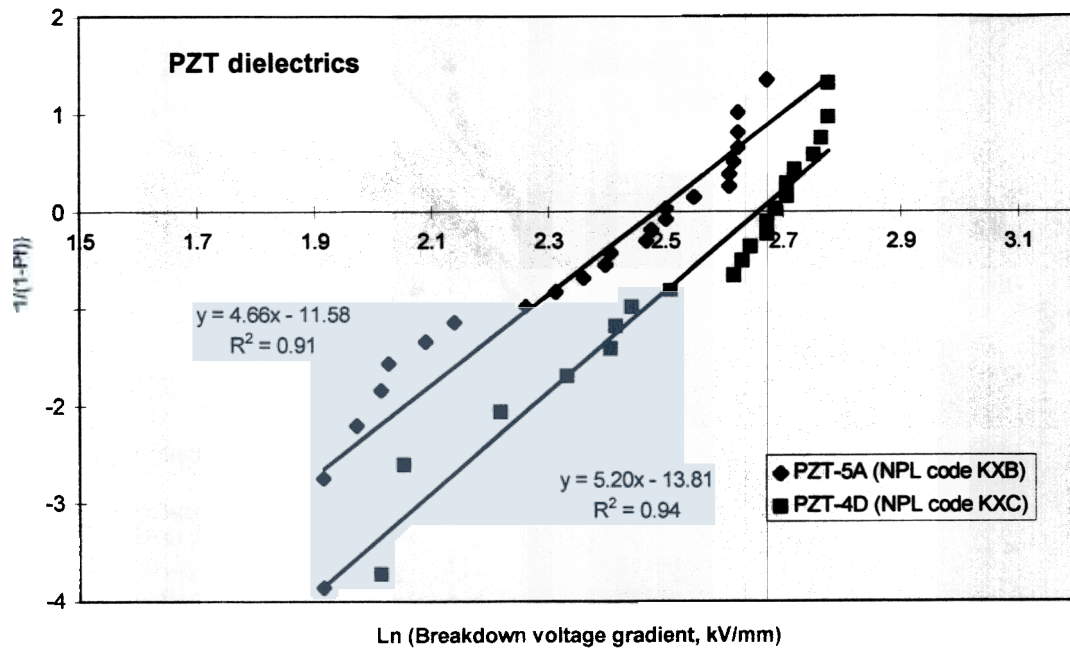


Figure 13 - Weibull distributions for PZT-5A and PZT-4D after removal of supplied electrodes.

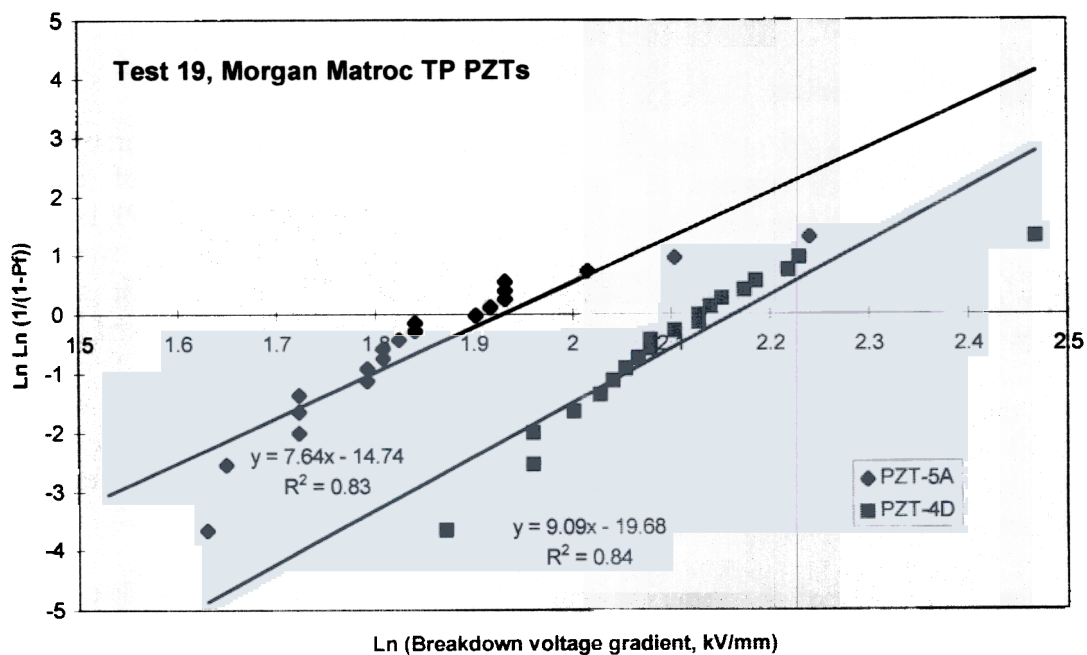


Figure 14 - Weibull distributions for PZT-5A and PZT-4D supplied without electrodes.

5. DISCUSSION

5.1 General conclusions

Overall, the series of tests have shown that electric strength measurements can have some meaning when applied to thin ceramic test-pieces (<1 mm) with permittivities between about 8 and several 1000. When undertaken in an oil environment, the tests can be performed without risk of edge discharge even when the electrode extends as close as 2.5 mm from the edge of the test-piece. This work has shown that meaningful differences can be obtained between material types, and even between similar materials treated in different ways, e.g. with different electrode sizes, or with and without annealing treatments.

The use of Weibull statistical analysis has shown that in most cases, the distribution of electric strength data can be represented by a two-parameter Weibull distribution. However, the sizes of the test batches used, typically 16 test-pieces per condition, were too small for consistency of determination of Weibull modulus. As the analysis within ENV 843-5 for mechanical strength demonstrates, the limits of, for example, the 90% confidence interval on Weibull modulus are large for small sample populations, and the experimental results were scattered even outside these limits, despite good consistency on mean electric strength.

The effect of electrode area is marked, and in both of the cases examined, the electric strength increased with decreasing electrode area. In one extreme case with poor quality very thin electrodes, it became apparent that failure was primarily between the spherical electrodes, and the evaporated coatings played no role. The cumulative probability plot concept developed in this project has value in demonstrating whether a set of results is the consequence of random puncture position, or whether the position is biased in some way. The effect of poor electrode coating produced a very marked example of the value of the analysis, and underlined the need to ensure that the applied coating is continuous with low resistance, i.e. is thickly enough applied. It is believed that should the electrodes be too thin, there is the possibility that localised breakdowns could be 'self-isolating' by, for example, volatilisation or oxidation.

Further, the analysis has also shown that the edge of the electrode is not a preferred site of breakdown. Electric stress concentrations derived in analyses of fields associated with electrode edges do not therefore appear to dominate the site of failure. This is in contrast with the situation when testing plastic materials which, compared with ceramics, are essentially essentially defect-free.

The traditional method of undertaking electric strength and withstand tests on technical ceramics described in IEC 60672-2 involved the use of dimpled test-pieces. Dimpling has the potential advantage of restricting the thickness in the test area, and thus minimising the risk of flash-over, but it requires a thick test-piece. Pressing the dimple into a specially made test-piece may well produce different results from machining the dimple into an otherwise flat test-piece. The present work has shown that dimpling does indeed restrict the location of the breakdown, but gives a higher result for a given thickness than plane electrodes of the same total area. This can be explained as being a consequence of the reduced volume under electric stress. Furthermore, the direction of application of the voltage appears to influence the result. The origins of this behaviour are not clear, but must be related to the direction of current electron flow when breakdown starts. The results of the present study thus show that dimpling

is not necessary, and that tests can be readily performed on plane samples with symmetrical disc electrodes.

5.2 Problems and errors in testing

Although the test data are presented and analysed without reference to errors of measurement, a number of issues arose during testing which may be relevant to setting experimental procedures for testing. These factors are summarised here.

5.2.1 Electroding

Application of coaxial electrodes on opposite sides of planar test-pieces requires care and attention to the mounting and masking arrangements employed. It is most convenient if an arrangement can be made which positively locates the test-piece in a mount with coaxial apertures, as indicated in Figure 3. After coating one side, the mount is turned over to coat the other. However, generally this requires precision in machining the mount to suit a given size of test-piece, and thus leads to lack of flexibility in coping with different test-piece sizes. Clearly, mounts for routine testing of particular sizes of test-piece can be made cost-effectively, but a more complex arrangement may be needed to cope with variable-sized components. The electrodes should also have well-defined edges. Intimate contact between the test-piece and the masking aperture is required to avoid tapering the coating excessively at the edges.

The surfaces must be clean and grease-free before electroding. Thin evaporated coatings can be damaged fairly easily, but thicker coatings are more resilient towards accidental handling damage. The current work used aluminium coatings, and some problems arose with poor evaporation conditions resulting of inadequate thickness which may have been resistive, perhaps even 'self-isolating', giving rise to non-random position of breakdown. Aluminium does have the advantage of easy evaporation at fairly low temperatures, but other types of coating, such as sputtered Au/Pd, may be preferable for continuity and resistance to oxidation.

5.2.2 Calibration of HT voltage

It is highly desirable to be able to calibrate the HT source to provide traceability of measurement. However, the system employed gave no direct access to the HT connections which were sealed inside an interlocked cabinet for safety reasons. It is understood that a LT lead system connects the sealed system to a meter on the control panel, which is somewhat inadequate as a means of reading. As supplied, the instrument relies completely on internal correlation of meter reading and HT voltage.

Consideration was given to three means of direct calibration without compromising safety:

- (i) the use of a battery-energised, separately calibrated digital voltmeter contained completely within the interlocked cabinet. For safety reasons this could not be digitally connected to an external recording device other than through an optical link;
- (ii) the use of a calibrated HT resistor chain within the interlocked chamber, the voltage over part of which is measured using a low-tension battery energised voltmeter. The same problems with recording would be encountered;
- (iii) using a pair of polished spheres with a variable but measured gap between them, the HT breakdown voltage could be compared against well-known tables for breakdown of

air gaps. However, the difficulty with this method is the need to control the atmospheric temperature, pressure and moisture content.

In view of the time and effort in setting this equipment up, no actions were taken during the course of the project. The data reported are therefore nominal, and reliant on the pre-set relationship between the HT source and the meter reading.

5.2.3 HT source control and measurement

The particular instrument employed in the current work generated a ramped voltage at a nominal pre-set rate based on the set peak and a rise time to that peak. On approaching the set peak, integral/derivative type control slowed down the rate to rise to avoid overshoot. This would be important for a withstand test, but for a breakdown test it is highly desirable to maintain the set rate up to failure. With many of the tests requiring voltages approaching the machine limits, some uncertainty in voltage rise rate conditions was introduced. This has been ignored in the analysis. However, it could mean that the duration under electrical stress was longer in some test-pieces than the nominal values. The parallel situation would be to vary the loading rate or displacement rate in mechanical testing, which is never normally undertaken. Correcting for such effects would be impossible without detailed knowledge of the effects of time under electrical stress on breakdown values.

The use of a data-logger measuring the LT response shown on the meter in parallel with the meter was very useful in this work, but highlighted a further potential problem. As breakdown was approached, the apparent voltage rise rate appeared to jump. The effect was variable, but did not seem to occur to any significant extent on the meter. Consequently it was unclear what the true breakdown voltage was. A number of possible approaches to the logged data were evaluated:

- (i) to use the peak hold value from the peak hold circuit;
- (ii) to take the jump as an indication of the initiation of breakdown, and to use a value which was extrapolated from below the jump to the centre of the logger time interval when the jump was first detected;
- (iii) to take the value immediately after the jump and before any drift in the plateau representing peak hold.

The principal difficulty was in deciding whether the effects seen were entirely instrumental in origin, resulting from noise or spikes in the supply system or from changes in HT circuit impedance as current began to flow in the test-pieces, or were due to the behaviour of the test-piece itself, in particular the way in which breakdown currents accelerated with time. It was suggested by the instrument manufacturer that the earthing of the instrument needed to be improved, but after installation of a separate ground to outside the laboratory, there was little improvement. After considerable discussion and analysis it was decided to rely exclusively on the peak hold values, so all data are reported in this condition, with the proviso that there may be an error of several hundred volts in the recording of each break down voltage level. It was concluded that the whole system needed redesigning in a manner which was suitable for proper control of voltage rise rate and for accurate recording of the voltage levels at breakdown.

5.2.4 Dimpling

Although dimpling was found not to be a necessary part of test-piece preparation for the tests, it actually proved to be quite a difficult operation. In particular, setting the position of the dimpling tool such that its centre was on the line of rotation of the test-piece proved quite difficult, with the result that many samples had a slight pip in the centre of the dimple, making thickness measurement difficult. Further, dimpling tools have to be specially made, and the grit size employed in the present case tended to leave grinding marks on the dimpled surface. If the test-piece was not rotated under the tool, the dimple was not round, but adopted the shape of the tool, and grinding marks were strongly directional. If the test-piece was rotated, the dimple was round and there was less directionality to the grinding marks, but the centre pip tended to occur. If dimpling is essential to reduce the thickness of the test-piece to a level capable of being tested with a given instrument, it is important therefore to pay particular attention to the set-up employed and the dimpling procedure adopted.

5.3 Breakdown mechanisms

5.3.1 Optical microscopy of punctures

It was not the purpose of this work to study the mechanisms of breakdown, but some of the test-pieces were examined under the optical microscope after breakdown. Most examples examined showed a single breakdown position, with damage visible on both surfaces. The size of the damaged region tended to be larger on the grounded side than on the HT side, which is likely to be a result of the more severe heating likely at the source of electrons causing the discharge. In some examples, however, there was evidence of two breakdown positions, one usually much more severe than the other. Figures 15 and 16 show two examples from Test series 18, from a plane test-piece and a dimpled test-piece. In the later case, the larger, presumably primary, failure has occurred in the centre of the dimple as expected, but the secondary site is on the parallel part of the test-piece which is 1 mm thick, thicker than can normally be broken down by the HT limits of the apparatus. The reasons for the double event are completely unclear and can only be speculated about. However, the possibility remains that they may be associated with the 'self-isolating' phenomenon due to the electrodes being too thin to support the breakdown current.

5.3.2 Optical microscopy of polished cross-sections

Sections of some of the test materials were made with a view to determining the distribution of porosity, which might explain the values of dielectric breakdown measured in the tests. Examples of the CeramTec substrates of different thicknesses and Morgan PZTs were sectioned using a diamond saw, mounted in metallographic mounting resin and subjected to a grinding and polishing routine. After a satisfactory polish had been achieved, the surfaces were coated with a thin sputtered gold-palladium layer to improve reflectivity, and were examined in an optical microscope. Photographs are shown in Figures 17 (alumina) and 18 (PZTs).

It is clear from Figure 17 that the alumina ceramic possessed a low level residual porosity, but this was on a very fine scale, typically 2 – 5 μm , was homogeneously distributed, and was similar for all the thicknesses evaluated. In particular, no significant difference could be seen between the 1 mm material and the 0.38 mm material used for the comparison of dimpled and planar test geometries in Test 18.

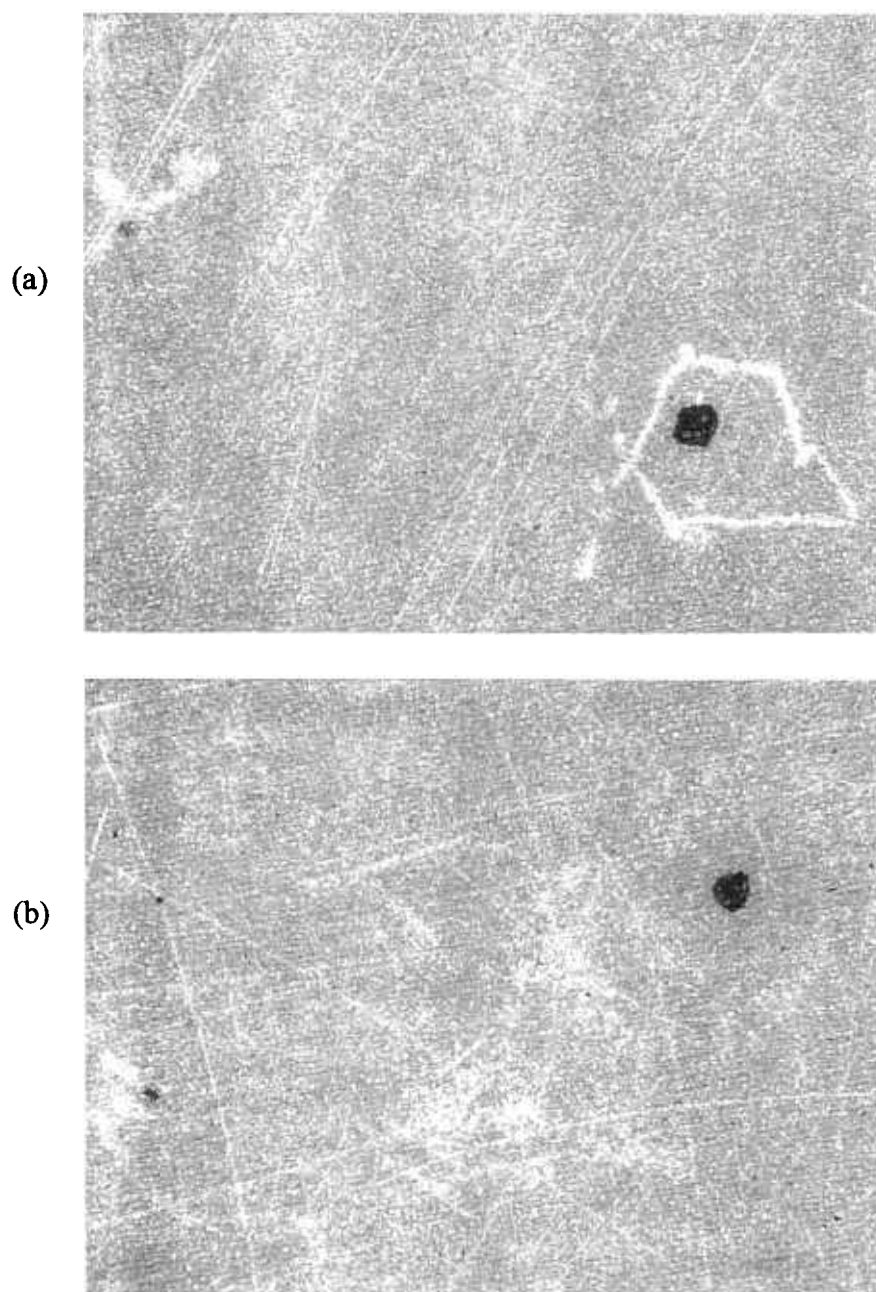


Figure 15 - Optical microscope images of the double breakdown sites in a plane sample from Test series 18 as seen from the respective faces .

In Figure 18 it is clear that the porosity levels in the PZTs are similar, but on a much coarser scale and to a higher level than in the alumina substrates. PZT5A has finer pores than PZT-4D. Many of the pores appeared flattened in the direction of pressing of the discs, and therefore clearly originate from the state of granulation of the precursor powder and the inability of the compaction process to eliminate gaps between granules, or the holes within them³.

³ In formulating a ceramic powder for commercial manufacture of pressed shapes, it is desirable to achieve a free-flowing granulate which permits automatic die filling. Such granulates are typically made by spray drying a powder/binder slurry. A balance has to be struck between crushability of the granulate in pressing of

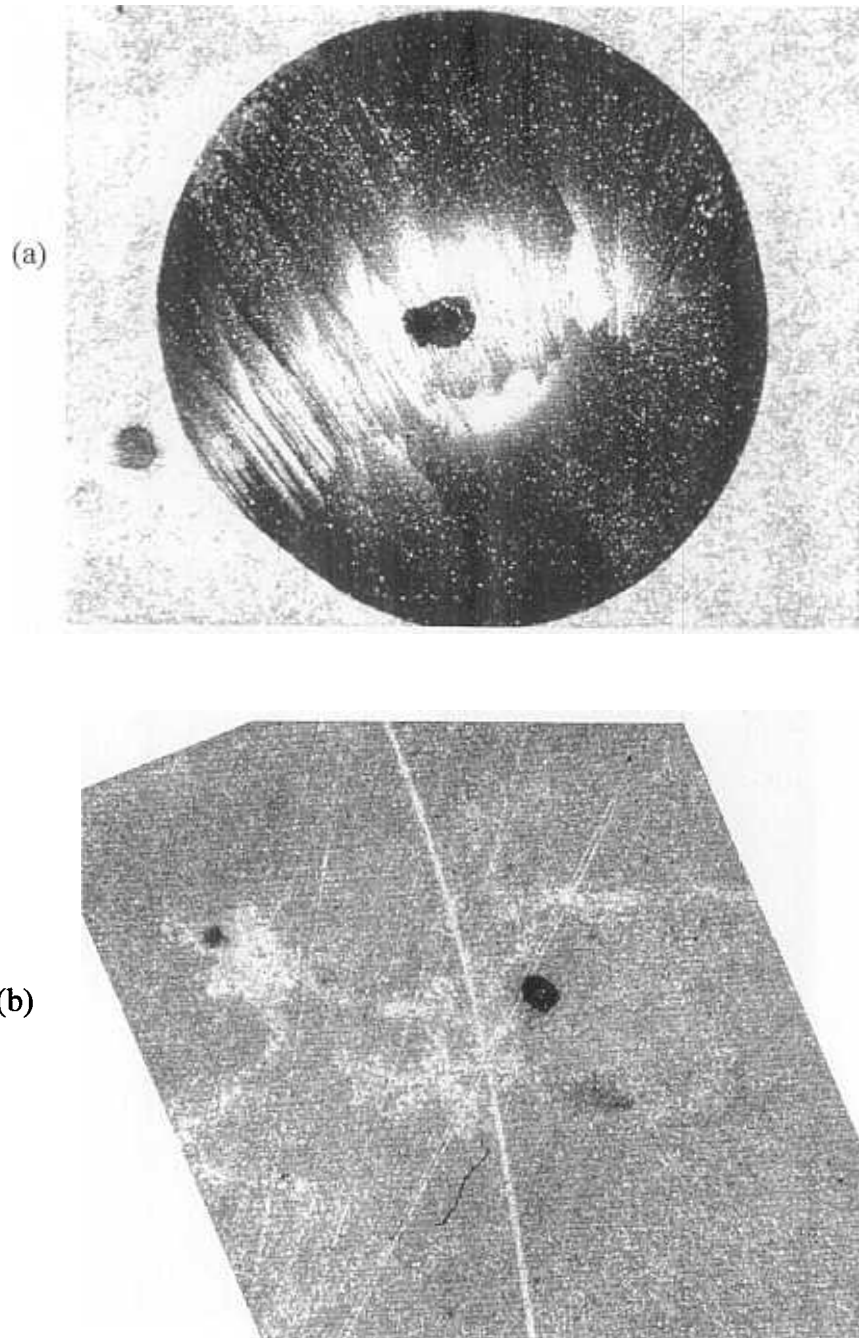


Figure 16 - Optical microscope images of the double breakdown sites in a dimpled sample from Test series 18 as seen from (a) dimpled side and (b) plane side.

5.3.3 Breakdown voltage gradient levels

The results for alumina vary between less than 10 kV/mm for the Royal Worcester 88% up to 100 kV/mm for the CeramTec alumina. These latter results are very high compared with most other data available on aluminas, but the authors think that this is probably due to significant advances in ceramic processing technology. Thus, the minimum values appearing in the

flowability, represented by hardness. If this balance is inadequate, cavities within and between granules may not be eliminated in pressing.

materials specification in IEC 60672-3 for alumina, originating unchanged from an earlier DIN standard formulated in the 1970's, are typically 10–15 kV/mm depending on alumina type, and are typical of the level found for the older Royal Worcester material.

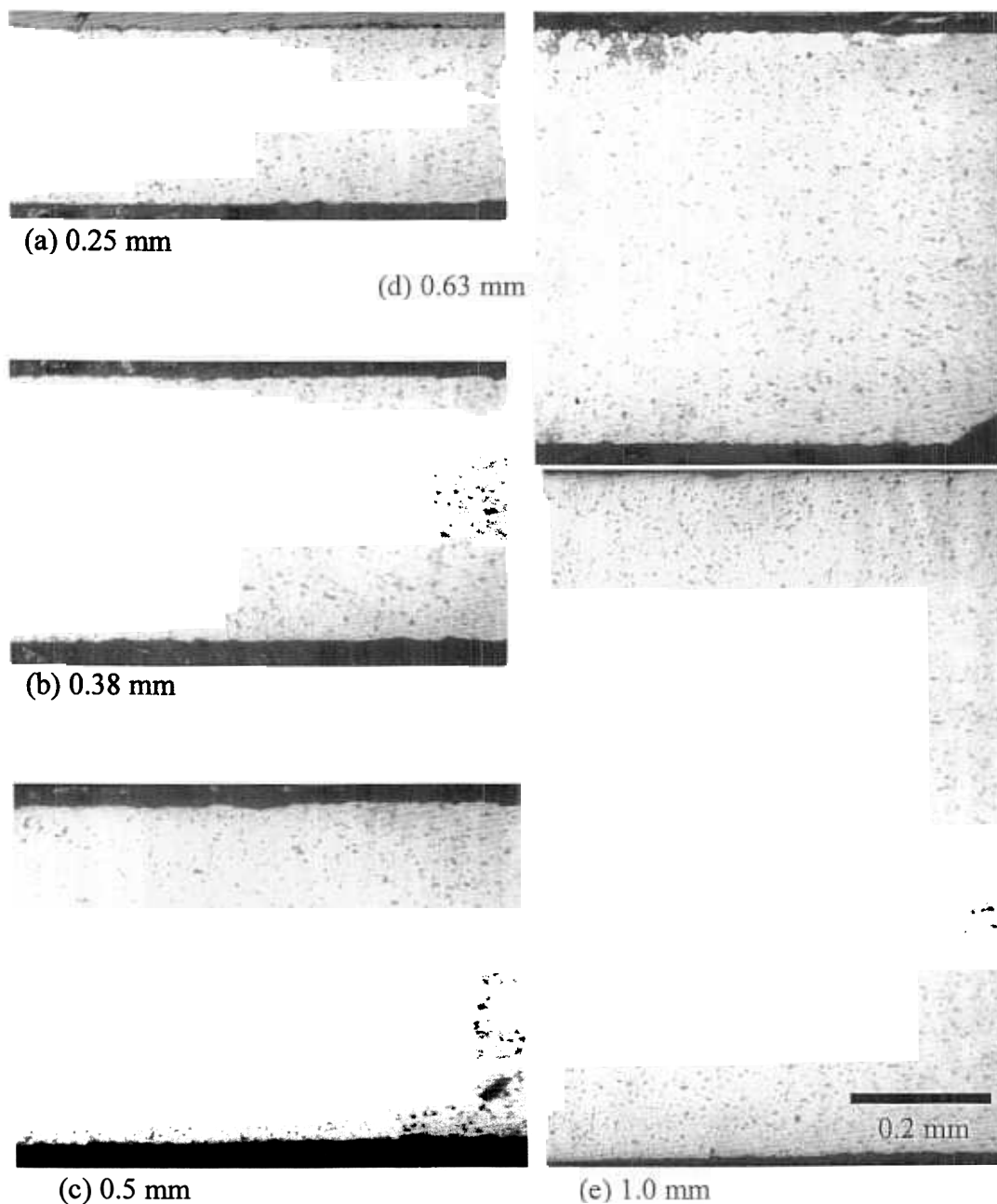
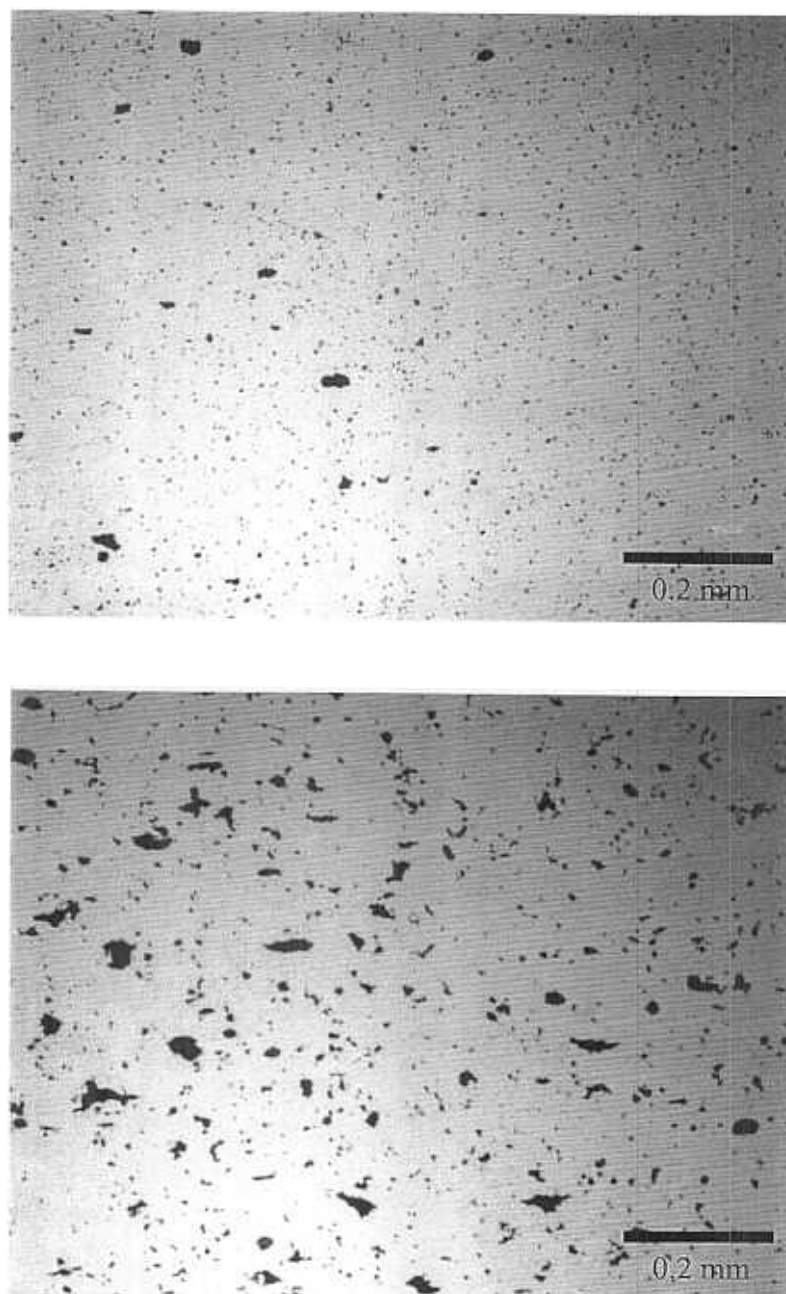


Figure 17 - Polished cross-sections of CeramTec alumina substrates of different as-fired thicknesses showing a low level of fine-scale porosity, and similar appearance irrespective of thickness.



(b)

Figure 18 Optical microscope images of cross-sections of PZT samples, (a) PZT-5A (material 5, NPL code KXB) and (b) PZT-4B (material 6, NPL code KXC).

The newer alumina materials have much improved dielectric breakdown resistance, especially in the thin sections tested. Values of similar order to those found for the substrate alumina in this work are reported in the recent scientific literature (Ishida *et al.* (1991), Liebault *et al.* (1998)), and for hot-pressed material in older literature (Morse and Hill (1970)), while older references employing conventional coarse-grained sintered material seldom exceed 30 kV/mm (e.g. Owate and Freer (1988, 1992)),

In the PZTs, the larger scale and higher volume fraction of pores are consistent with the low electrical strength obtained, with the larger-pored PZT-4D being electrically weaker than the

finer pored PZT-5A. The values obtained are typical of those claimed for such materials by the manufacturers.

6. CONCLUSIONS

An evaluation has been made of the possibilities for developing electric strength test procedures appropriate for modern high-strength and high permittivity ceramic materials used for passive electrical purposes. It has been shown that:

- Thin planar test-pieces ranging in thickness between 1.0 and 0.25 mm and with electric strengths exceeding 100 kV/mm can be electroded and tested in transformer oil without difficulties of edge tracking or flashover;
- Differences in electric strength can be systematically resolved using sufficient test-pieces, and examples have been obtained indicated electrode area effects, thickness effects, and machining/annealing effects;
- Dimpling such test-pieces other than for the purposes of bringing the breakdown voltage within instrumental range has little value, and is unnecessary to prevent edge flashover; producing small dimples in thin test-pieces is not a simple task and requires specialised and precision equipment;
- Test results from a given batch of nominally identical samples have been found to possess a variation in electric strength which is expressible in terms of the two-parameter Weibull distribution analogous to procedures developed to analyse mechanical strength of brittle materials;
- On planar test-pieces, the quality of thin evaporated electrodes must be such that breakdown positions are uniformly distributed over the surface of the electrode, and are not concentrated either at the edge or in the centre; a statistical test for the area distribution has been devised and demonstrated effectively on batches of the same materials with good and bad electroding;
- Concerns that breakdowns will occur preferentially around the periphery of the electrodes in planar test-pieces as a result of electric stress concentrations have proved to be unfounded;
- Concerns that have not been satisfactorily dealt with in this project are:
 - Traceable calibration of HT voltage sources contained within safety enclosures;
 - Obtaining consistency in the ramp rate of HT voltage in the instrument employed, in particular whether the response of the LT data-logger added to the test machine was a true reflection of the HT voltage being applied to the test-piece;
 - Explaining the occasional occurrence of double puncturing of the test-piece;

- For testing advanced ceramic products, it is possible to devise a suitable standard with greater flexibility than the existing method given in IEC 60672-2 for traditional ceramic materials, and which is appropriate to the high levels of electric strength achievable with modern processing technology. Such a document is in preparation in preliminary draft format.

FURTHER RESEARCH POSSIBILITIES

Within the confines of the project it has not been possible to explore some technical aspects of electric strength which have the potential for providing methodologies for life prediction which would have particular relevance to industries working in multilayer technology. These are:

- To evaluate statistically the effects of electrode area over a wide range of areas (e.g. 1 to 1000 mm²) and to correlate the obtained relationship with that expected on the basis of the Weibull weakest link theory, and thereby establish whether Weibull parameter manipulation is feasible.
- To analyse whether the well-known thickness effect can also be expressed in terms of the Weibull weakest link theory on the same basis as the area effects, or whether a different basis is required.
- To evaluate withstand time (the electrical equivalent to static fatigue life under mechanical load) as a function of applied voltage gradient in order to establish the nature of the ageing phenomenon, and whether this can be described on the same basis that slow crack growth limits long-term mechanical strength. This could be done for both d.c. and a.c. conditions.

In each case, the numbers of test-pieces for each set of conditions need to be larger than those employed in the current work in order to reduce the width of the confidence interval on the fitted Weibull parameters.

A successful outcome to the above studies could provide:

- 1) A methodology for predicting performance based on a limited number of tests;
- 2) A method for characterising the ageing of electric strength in insulating and dielectric materials;
- 3) An improved standard for withstand testing which is more meaningful than the existing short-term pass/fail test incorporated into IEC 60672-2.

ACKNOWLEDGEMENTS

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ANNEX - TEST SUMMARY**A.1 Initial trials with electroding**

TEST No.1	Material: 1	Agate Alumina
Test-piece dimensions, mm		
Test-piece condition	Plane	
Electrodes	Al, various electrode sizes see supplemental table	
HV ramp rate	2 kV/s	
Breakdown voltages, kV	40.0, 35.0, 39.0, 45.5, 45.0, 42.6, 46.4, 42.0, 37.0	
Breakdown stress levels, kV/mm	38.8, 33.6, 37.6, 44.1, 43.6, 42.6, 46.4, 40.8, 35.7	
Average breakdown stress level, kV/mm	40.4 ± 4.2	

Test No.	Electrode sizes, mm	Breakdown voltage, kV
1.1	7 Ø	40.0
1.3	13 Ø	35.0
1.4	10.4 Ø	39.0
1.5	5 Ø	45.5
1.6	10.4 Ø	45.0
1.7	7.4 Ø	42.6
1.8	5 Ø	46.4
1.9	15.2 Ø	42.0
1.10	18 Ø	37.0

TEST No.1A	Material: 1P	Agate, Alumina
Test-piece dimensions, mm	11 Ø x 1	
Test-piece condition	Plane	
Electrodes	Al, 6 mm Ø	
HV ramp rate	2 kV/s	
Breakdown voltages, kV	28 - flashover suspected, but sample punctured	
Breakdown stress levels, kV/mm		
Average breakdown stress level, kV/mm		

TEST No.1B	Material: 1A, 1C	Agate, Alumina
Test-piece dimensions, mm	20 Ø x 1	
Test-piece condition	Dimpled, 300 µm and 100 µm residual thicknesses	
Electrodes	Al, 6 mm Ø	
HV ramp rate	2 kV/s	
Breakdown voltages, kV	28 - flashover suspected, but sample punctured	
Breakdown stress levels, kV/mm		
Average breakdown stress level, kV/mm		

A.2 Further systematic tests with Agate material

TEST No. 2A	Material: 1P	Agate, Alumina
Test-piece dimensions, mm		20 Ø x 1
Test-piece condition		Plane, as received
Electrodes		Al, 15 mm Ø
HV ramp rate		2 kV/s
Breakdown voltages, kV		39.6, 38.7, 41.3, 42.7, 42.0, >50*, 39.5, 39.7, 41.3, 41.2, 40.3, 40.8, 44.7, 40.5
Breakdown stress levels, kV/mm		38.3, 37.2, 39.6, 44.0, 40.6, >48.1*, 37.7, 38.5, 39.8, 38.9, 38.9, 39.4, 42.7, 38.7
Average breakdown stress level, kV/mm		40.0 ± 2.9

* Exceeded machine capability

TEST No. 2B	Material: 1C	Agate, Alumina
Test-piece dimensions, mm		20 Ø x 1
Test-piece condition		Dimpled to 100-180 µm
Electrodes		Al, 15 mm Ø
HV ramp rate		2 kV/s, dimple to +ve HT
Breakdown voltages, kV		6.4, 6.1, 8.5, 10.5
Breakdown stress levels, kV/mm		62.0, 57.3, 47.0, 65.8
Average breakdown stress level, kV/mm		58.2 ± 8.1

TEST No. 2C	Material: 1B	Agate, Alumina
Test-piece dimensions, mm		20 Ø x 1
Test-piece condition		Dimpled to 200 µm
Electrodes		Al, 15 mm Ø
HV ramp rate		2 kV/s, dimple to +ve HT
Breakdown voltages, kV		12.5, 11.4, 9.8, 10.3, 10.8
Breakdown stress levels, kV/mm		59.7, 54.5, 50.3, 48.0, 51.3
Average breakdown stress level, kV/mm		52.8 ± 4.6

TEST No. 2D	Material: 1A	Agate, Alumina
Test-piece dimensions, mm		25 Ø x 1
Test-piece condition		Dimpled to 300 µm
Electrodes		Al, 15 mm Ø
HV ramp rate		2 kV/s, dimple to +ve HT
Breakdown voltages, kV		15.7, 16.7, 14.2, 16.9
Breakdown stress levels, kV/mm		46.6, 52.6, 39.8, 46.3
Average breakdown stress level, kV/mm		46.2 ± 5.4

A.3 Trials with other materials

TEST No. 3A (preliminary) Material: 2	Royal Worcester, 88% Alumina
Test-piece dimensions, mm	25 Ø x 1
Test-piece condition	Plane, as machined
Electrodes	Al, 15 mm Ø
HV ramp rate	2 kV/s
Breakdown voltages, kV	15.2, 24.6, 27.9
Breakdown stress levels, kV/mm	20.0, 32.2, 35.7
Average breakdown stress level, kV/mm	

TEST No. 3B Material: 2	Royal Worcester, 88% Alumina
Test-piece dimensions, mm	25 Ø x 1
Test-piece condition	Plane, as machined
Electrodes	Al, 15 mm Ø
HV ramp rate	2 kV/s
Breakdown voltages, kV	15.7, 12.4, 15.1, 21.5, 15.9, 14.9, 17.3, 11.1, 15.4, 7.7, 24.4, 10.1, 21.6, 24.1
Breakdown stress levels, kV/mm	19.6, 14.7, 18.1, 26.1, 18.8, 17.4, 20.4, 13.1, 19.4, 9.0, 28.9, 12.4, 26.1, 30.5
Average breakdown stress level, kV/mm	19.6 ± 6.4

TEST No. 3C Material: 2	Royal Worcester, 88% Alumina
Test-piece dimensions, mm	25 Ø x 1
Test-piece condition	Plane, annealed
Electrodes	Al, 15 mm Ø
HV ramp rate	2 kV/s
Breakdown voltages, kV	7.6, 7.1, 7.3, 7.5, 9.3, 8.1, 6.6, 9.2, 9.3, 9.6, 12.2, 6.6, 7.3, 6.0, 6.8
Breakdown stress levels, kV/mm	9.8, 8.5, 8.0, 9.0, 11.1, 9.7, 10.9, 11.2, 11.5, 14.7, 8.0, 9.3, 7.1, 8.2
Average breakdown stress level, kV/mm	9.6 ± 2.0

A.4 Tests with Deranox 995 alumina

TEST No. 4	Material: 3	Morgan Matroc, Deranox 995 alumina
Test-piece dimensions, mm	25 Ø x 1	
Test-piece condition	As machined	
Electrodes	Al, 15 mm Ø	
HV ramp rate	2 kV/s	
Breakdown voltages, kV	Very low - samples appeared to contain cracks - tests abandoned	
Breakdown stress levels, kV/mm	-	
Average breakdown stress level, kV/mm	-	

A.5 Tests on PZT materials

TEST NO. 5A	Material: 5	Morgan Matroc TP, PZT-5A (NPL code KXB)
Test-piece dimensions, mm		10 Ø x 1
Test-piece condition		Supplied electrodes removed
Electrodes		Al, 5 mm Ø
HV ramp rate		0.8 kV/s
Breakdown voltages, kV		11.2, 6.8, 7.3, 12.5, 12.3, 7.7, 13.0, 11.3
Breakdown stress levels, kV/mm		11.1, 6.8, 7.2, 12.2, 12.2, 7.6, 12.8, 11.0
Average breakdown stress level, kV/mm		10.5 ± 2.4

TEST NO. 5B	Material: 6	Morgan Matroc TP, PZT-4D (NPL code KXC)
Test-piece dimensions, mm		10 Ø x 1
Test-piece condition		Supplied electrodes removed
Electrodes		Al, 5 mm Ø
HV ramp rate		0.8 kV/s
Breakdown voltages, kV		11.3, 11.5, 7.5, 14.9, 16.1
Breakdown stress levels, kV/mm		11.1, 11.5, 7.5, 14.7, 16.1
Average breakdown stress level, kV/mm		12.2 ± 4.7

TEST NO. 5C	Material: 6	Morgan Matroc TP, PZT-4D (NPL code KXC)
Test-piece dimensions, mm		10 Ø x 1
Test-piece condition		Supplied electrodes removed, surfaces ground with 1200 grit SiC paper
Electrodes		Al, 5 mm Ø
		0.8 kV/s
Breakdown stress levels, kV/mm		
Average breakdown stress level, kV/mm		

TEST NO. 8A	Material: 5	Morgan Matroc TP, PZT-5A (NPL code KXB)
Test-piece dimensions, mm		10 Ø x 1
Test-piece condition		Supplied electrodes removed
Electrodes		Al, 5 mm Ø
HV ramp rate		0.8 kV/s
Breakdown voltages, kV		6.8, 8.5, 10.3, 12.2, 14.0, 9.7, 13.9, 12.0, 14.9, 13.8, 10.8, 14.2, 7.6, 8.2, 14.2, 14.0
Breakdown stress levels, kV/mm		6.8, 8.5, 10.1, 11.9, 13.7, 9.6, 13.6, 11.8, 14.5, 13.6, 10.6, 13.8, 7.5, 8.1, 13.8, 13.8
Average breakdown stress level, kV/mm		11.4 ± 2.6

TEST NO. 8B	Material: 6	Morgan Matroc TP, PZT-4D (NPL code KXC)
Test-piece dimensions, mm		10 Ø x 1
Test-piece condition		Supplied electrodes removed
Electrodes		Al, 5 mm Ø
HV ramp rate		0.8 kV/s
Breakdown voltages, kV		14.1, 13.9, 15.4, 15.1, 15.9, 15.4, 14.7, 7.9, 16.3, 14.3, 9.3, 16.2, 11.3, 10.4, 14.6, 12.5
Breakdown stress levels, kV/mm		13.9, 13.7, 15.2, 15.0, 15.7, 15.0, 14.5, 7.8, 16.1, 14.1, 9.2, 15.9, 11.2, 10.3, 14.5, 12.3
Average breakdown stress level, kV/mm		13.4 ± 2.5

A.6 Tests on CeramTec substrate alumina in different thicknesses

TEST NO. 6	Material: 6	CeramTec, 96% alumina
Test-piece dimensions, mm		25 x 25 x 0.5
Test-piece condition		As received
Electrodes		Al, 20 mm Ø
HV ramp rate		2 kV/s
Breakdown voltages, kV		44.0, 43.4, 44.0, 41.7, 44.5, 41.9, 44.7, 41.4, 42.9, 41.4, 41.9, 43.9, 44.5, 43.3, 42.3, 43.4
Breakdown stress levels, kV/mm		86.3, 85.1, 86.2, 82.0, 86.8, 81.3, 87.5, 81.3, 85.2, 82.1, 83.4, 87.4, 87.5, 84.7, 83.7, 86.2
Average breakdown stress level, kV/mm		84.8 ± 2.2

TEST NO. 7	Material: 6	CeramTec, 96% alumina
Test-piece dimensions, mm		25 x 25 x 0.25
Test-piece condition		As received
Electrodes		Al, 20 mm Ø
HV ramp rate		2 kV/s
Breakdown voltages, kV		27.1, 22.8, 25.9, 22.7, 21.7, 24.9, 25.4, 25.7, 26.2, 22.1, 26.6, 34.9, 25.8, 22.0, 20.6, 23.8
Breakdown stress levels, kV/mm		105.0, 89.0, 101.9, 89.0, 85.5, 98.3, 98.6, 98.6, 100.6, 86.2, 103.4, 136.9, 100.5, 86.3, 79.1, 93.4
Average breakdown stress level, kV/mm		94.4 ± 7.9

TEST NO. 9	Material: 4	CeramTec, 96% alumina
Test-piece dimensions, mm		25 x 25 x 0.635
Test-piece condition		As received
Electrodes		Al, 20 mm Ø
HV ramp rate		2 kV/s
Breakdown voltages, kV		42.9, 43.0, 43.5, 49.4, 46.9, 49.6, 43.0, 46.3, 43.6, 40.5, 50.0, 53.5*, 50.4, 39.1, 50.1, 48.1
Breakdown stress levels, kV/mm		69.4, 68.8, 70.0, 76.9, 76.2, 80.2, 69.7, 74.0, 70.6, 63.3, 80.8, 84.1*, 79.0, 63.0, 80.7, 75.3
Average breakdown stress level, kV/mm		73.9 ± 6.3 (73.2 ± 5.9 excluding *)

* = exceeded machine limit - no breakdown

TEST NO. 10	Material: 4	CeramTec, 96% alumina
Test-piece dimensions, mm		25 x 25 x 0.38
Test-piece condition		As received
Electrodes		Al, 20 mm Ø
HV ramp rate		2 kV/s
Breakdown voltages, kV		33.2, 28.8, 37.2, 35.4, 36.1, 36.2, 34.9, 35.9, 34.8, 36.6, 34.3, 36.3, 36.6, 35.2, 35.8, 36.1
Breakdown stress levels, kV/mm		83.3, 74.8, 92.3, 91.0, 93.7, 89.9, 91.2, 88.3, 92.0, 94.4, 84.7, 94.0, 95.1, 92.3, 92.3, 88.4
Average breakdown stress level, kV/mm		89.9 ± 5.2

A.7 Tests on CeramTec substrate alumina with different electrode sizes

TEST NO. 11	Material: 4	CeramTec, 96% alumina
Test-piece dimensions, mm		25 x 25 x 0.5 (repeat)
Test-piece condition		As received
Electrodes		Al, 20 mm Ø
HV ramp rate		2 kV/s
Breakdown voltages, kV		42.7, 44.4, 38.2, 44.7, 41.4, 37.3, 45.4, 42.2, 45.8, 42.3, 39.8, 42.5, 43.3, 42.9, 39.9, 44.4
Breakdown stress levels, kV/mm		84.0, 88.6, 74.8, 87.4, 82.7, 73.3, 90.8, 83.0, 91.4, 84.3, 77.9, 93.6, 84.6, 85.9, 77.7, 89.3
Average breakdown stress level, kV/mm		83.7 ± 5.4

TEST NO. 12	Material: 4	CeramTec, 96% alumina
Test-piece dimensions, mm		25 x 25 x 0.5
Test-piece condition		As received
Electrodes		Al, 20 mm and 5 mm Ø
HV ramp rate		2 kV/s, 5 mm electrode to +ve HT
Breakdown voltages, kV		45.8, 46.5, 49.2, 45.1, 44.3, 44.4, 44.5, 46.0, 44.5, 47.1, 44.6, 43.8, 47.5, 47.3, 44.9
Breakdown stress levels, kV/mm		91.1, 92.0, 97.4, 88.6, 88.4, 86.7, 87.8, 90.7, 87.8, 94.2, 88.1, 85.5, 94.3, 93.7, 88.6
Average breakdown stress level, kV/mm		90.3 ± 3.4

TEST NO. 13	Material: 4	CeramTec, 96% alumina
Test-piece dimensions, mm		25 x 25 x 0.5
Test-piece condition		As received
Electrodes		Al, 5 mm Ø
HV ramp rate		2 kV/s
Breakdown voltages, kV		44.0, 44.3, 43.6, 41.6, 40.7, 46.0, 45.0, 45.3, 45.8, 44.3, 42.9, 44.3, 41.6, 45.0, 42.8, 41.3
Breakdown stress levels, kV/mm		85.7, 87.0, 84.6, 81.7, 79.8, 90.0, 88.6, 84.7, 89.9, 86.9, 84.4, 87.1, 81.3, 88.0, 84.8, 81.2
Average breakdown stress level, kV/mm		85.4 ± 3.1

A.8 Tests on CeramTec substrate alumina

TEST NO. 14	Material: 4	CeramTec, 96% alumina
Test-piece dimensions, mm		25 x 25 x 0.5 (repeat)
Test-piece condition		As received
Electrodes		Al, 20 mm Ø
HV ramp rate		2 kV/s
Breakdown voltages, kV		49.5, 54.5, 46.3, 46.8, 14.7*, 49.3, 45.9, 45.6, 46.8, 44.4, 48.3, 48.4, 41.6
Breakdown stress levels, kV/mm		97.3, 108.3, 91.5, 92.5, 29.0*, 97.6, 90.2, 90.1, 92.8, 88.1, 96.9, 95.5, 82.9
Average breakdown stress level, kV/mm		93.6 ± 6.3

* excluded - tested in air. NB: all breakdowns occurred at spherical electrode contact point suggesting poor coating quality.

TEST NO. 15	Material: 4	CeramTec, 96% alumina
Test-piece dimensions, mm		25 x 25 x 0.5
Test-piece condition		As received
Electrodes		Al, 20 mm Ø
HV ramp rate		2 kV/s, spheres polished to remove burn marks
Breakdown voltages, kV		42.4, 39.5, 41.5, 41.3, 42.5, 40.6, 42.3, 42.5, 42.3, 42.6, 41.5, 43.1, 40.4
Breakdown stress levels, kV/mm		82.9, 77.8, 81.3, 81.2, 83.2, 79.5, 84.1, 83.8, 83.4, 84.3, 82.3, 84.3, 79.3
Average breakdown stress level, kV/mm		82.2 ± 2.2

TEST NO. 16	Material: 4	CeramTec, 96% alumina
Test-piece dimensions, mm		25 x 25 x 0.5 repeat
Test-piece condition		As received
Electrodes		Al, 20 mm Ø
HV ramp rate		2 kV/s
Breakdown voltages, kV		38.6, 44.1, 42.3, 45.6, 45.1, 44.4, 42.1, 37.4, 40.3, 38.9, 46.4, 43.9, 40.8, 42.5, 45.2, 42.6
Breakdown stress levels, kV/mm		81.5, 86.4, 82.7, 89.3, 88.5, 87.0, 82.3, 75.4, 78.9, 75.5, 90.0, 88.1, 79.9, 83.0, 88.6, 83.3
Average breakdown stress level, kV/mm		83.8 ± 4.8

A.9 Tests on CeramTec substrate alumina with and without dimpling

TEST NO. 17A	Material: 4	CeramTec, 96% alumina
Test-piece dimensions, mm		25 x 25 x 1.0
Test-piece condition		As received, dimpled to 0.35 mm, not rotated
Electrodes		Al, 15 mm Ø
HV ramp rate		2 kV/s, dimple to +ve HT
Breakdown voltages, kV		35.8, 36.8, 35.0, 37.3, 41.4, 38.9, 44.9, 36.0, 41.2
Breakdown stress levels, kV/mm		93.7, 111.8, 96.1, 96.3, 98.6, 81.8, 123.3, 114.2, 112.3
Average breakdown stress level, kV/mm		102 ± 7.0

TEST NO. 17B	Material: 4	CeramTec, 96% alumina
Test-piece dimensions, mm		25 x 25 x 1.0
Test-piece condition		As received, dimpled to 0.35 mm, rotated
Electrodes		Al, 15 mm Ø
HV ramp rate		2 kV/s, dimple to +ve HT
Breakdown voltages, kV		37.2, 36.1, 35.6, 36.4, 30.5, 36.3, 36.3, 34.0, 34.6, 35.0
Breakdown stress levels, kV/mm		108.5, 104.1, 109.6, 117.0, 119.5, 105.4, 114.0, 95.0, 108.3, 106.7
Average breakdown stress level, kV/mm		108.8 ± 7.0

TEST NO. 17C	Material: 4	CeramTec, 96% alumina
Test-piece dimensions, mm		25 x 25 x 0.38
Test-piece condition		Plane, as received
Electrodes		Al, 15 mm Ø
HV ramp rate		2 kV/s
Breakdown voltages, kV		38.3, 34.6, 36.4, 41.8*, 41.8, 34.1, 37.1, 37.2, 37.7, 38.6
Breakdown stress levels, kV/mm		101.1, 90.6, 94.9, 81.5*, 108.0, 90.5, 97.4, 99.9, 101.0, 101.4
Average breakdown stress level, kV/mm		98.3 ± 5.6

* 0.5 mm thick test-piece in error, result ignored in averaging.

A.10 Second test series on CeramTec substrate alumina

TEST NO. 18A	Material: 4	CeramTec, 96% alumina
Test-piece dimensions, mm		25 x 25 x 0.38
Test-piece condition		Plane, as received
Electrodes		Al, 15 mm Ø
HV ramp rate		2 kV/s
Breakdown voltages, kV		35.2, 32.9, 30.5, 35.5, 35.5, 34.2, 34.2, 35.9, 34.9, 35.8, 36.1, 35.3, 35.0, 35.2, 36.6, 31.2, 27.2, 35.8, 29.9, 35.0, 35.3, 36.0, 35.6, 36.4, 35.0, 36.7, 36.6, 35.2, 35.2
Breakdown stress levels, kV/mm		94.0, 86.5, 83.4, 96.6, 96.2, 91.6, 93.3, 92.7, 94.1, 97.8, 96.3, 94.6, 93.8, 92.5, 97.5, 84.4, 73.0, 94.9, 80.3, 93.2, 92.8, 93.2, 93.5, 98.2, 94.1, 98.8, 98.5, 95.5, 93.3
Average breakdown stress level, kV/mm		92.6 ± 5.8

TEST NO. 18B	Material: 4	CeramTec, 96% alumina
Test-piece dimensions, mm		25 x 25 x 1.0
Test-piece condition		As received, dimpled to 0.38 mm, not rotated
Electrodes		Al, 15 mm Ø
HV ramp rate		2 kV/s, dimple to +ve HT
Breakdown voltages, kV		35.1, 37.3, 34.9, 39.4, 35.0, 37.6, 36.7, 37.2, 36.1, 34.7, 38.2, 37.5, 32.1, 35.7, 37.8
Breakdown stress levels, kV/mm		103.9, 106.6, 108.5, 101.6, 114.6, 119.5, 121.7, 117.8, 116.8, 98.9, 95.5, 108.5, 116.8, 114.8, 115.3
Average breakdown stress level, kV/mm		110.8 ± 8.1

TEST NO. 18C	Material: 4	CeramTec, 96% alumina
Test-piece dimensions, mm		25 x 25 x 1.0
Test-piece condition		As received, dimpled to 0.38 mm, not rotated, annealed after dimpling
Electrodes		Al, 15 mm Ø
HV ramp rate		2 kV/s, dimple to +ve HT
Breakdown voltages, kV		36.3, 35.6, 38.1, 35.4, 37.1, 37.0, 34.8, 36.7, 35.0, 24.8, 37.9, 35.1, 30.3, 37.5, 36.0, 35.6
Breakdown stress levels, kV/mm		96.4, 117.1, 115.2, 101.1, 121.0, 119.0, 110.5, 119.1, 129.8, 81.6, 108.1, 100.2, 100.7, 122.4, 115.8, 107.4
Average breakdown stress level, kV/mm		110.3 ± 12.1

TEST NO. 18D	Material: 4	CeramTec, 96% alumina
Test-piece dimensions, mm	25 x 25 x 1.0	
Test-piece condition	As received, dimpled to 0.35 mm, not rotated	
Electrodes	Al, 15 mm Ø	
HV ramp rate	2 kV/s, dimple to -ve HT	
Breakdown voltages, kV	42.9, 35.6, 39.2, 38.2, 34.7, 39.4, 36.7, 39.4, 37.7, 40.3, 42.1, 39.0, 37.8, 40.1, 39.2, 36.3, 40.3	
Breakdown stress levels, kV/mm	101.0, 101.3, 103.1, 99.6, 96.3, 91.5, 87.3, 97.1, 94.1, 102.6, 95.2, 95.9, 105.0, 108.1, 85.2, 94.3, 100.0	
Average breakdown stress level, kV/mm	97.5 ± 6.0	

TEST NO. 18E	Material: 4	CeramTec, 96% alumina
Test-piece dimensions, mm	25 x 25 x 1.0	
Test-piece condition	As received, dimpled to 0.35 mm, not rotated, annealed after dimpling	
Electrodes	Al, 15 mm Ø	
HV ramp rate	2 kV/s, dimple to -ve HT	
Breakdown voltages, kV	38.6, 39.1, 40.7, 37.4, 37.1, 34.0, 37.5, 39.8, 41.9, 39.2, 40.0, 40.0, 39.6, 38.9, 40.6	
Breakdown stress levels, kV/mm	89.7, 90.9, 104.4, 101.0, 90.4, 87.3, 86.7, 103.4, 107.9, 98.9, 102.8, 98.8, 104.1, 96.5, 85.5	
Average breakdown stress level, kV/mm	96.5 ± 7.5	

A.12 Tests on unpoled PZT materials

TEST NO. 19A	Material: 5U	Morgan Matroc TP, PZT-5A (NPL code KZAU)
Test-piece dimensions, mm	10 Ø x 1	
Test-piece condition	As received	
Electrodes	Al, 5 mm Ø	
HV ramp rate	0.8 kV/s	
Breakdown voltages, kV	6.9, 5.6, 6.8, 5.7, 6.2, 5.1, 6.0, 6.9, 5.2, 7.5, 6.2, 6.8, 6.9, 5.6, 6.1, 6.0, 6.3, 9.4, 6.3, 8.2	
Breakdown stress levels, kV/mm	6.9, 5.6, 6.7, 5.6, 6.1, 5.1, 6.0, 6.8, 5.2, 7.4, 6.2, 6.7, 6.8, 5.5, 6.1, 6.0, 6.3, 9.4, 6.3, 8.2	
Average breakdown stress level, kV/mm	6.4 ± 1.0	

TEST NO. 19B	Material: 6U	Morgan Matroc TP, PZT-4D (NPL code KZBU)
Test-piece dimensions, mm	10 Ø x 1	
Test-piece condition	As received	
Electrodes	Al, 5 mm Ø	
HV ramp rate	0.8 kV/s	
Breakdown voltages, kV	7.2, 8.6, 8.8, 7.1, 7.8, 8.1, 8.0, 7.5, 6.6, 9.0, 7.7, 8.4, 8.5, 8.3, 8.9, 8.1, 12.0, 8.1, 9.4, 9.4	
Breakdown stress levels, kV/mm	7.1, 8.4, 8.6, 7.1, 7.7, 8.0, 7.8, 7.4, 6.5, 8.9, 7.6, 8.4, 8.5, 8.2, 8.8, 8.0, 11.8, 7.9, 9.3, 9.2	
Average breakdown stress level, kV/mm	8.7 ± 1.1	