Evaluation of Piezoelectric Parameters of Several Commercial Thick Film Capacitor Dielectrics

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***Abstract*—Piezoelectric properties of several high-K dielectric pastes from ESL ElectroScience were investigated. The absolute values of measured piezoelectric coefficients d_{31} range from below of 1 pC/N to more than 50 pC/N. The aging rates of piezoelectric response are shown to be substantially higher than of typical piezoelectric materials. Measurements of high-field hysteresis loops together with low-field dielectric parameters and their temperature dependence were performed. Achieved piezoelectric response of samples as function of temperature, electric field strength and duration of polarisation was measured. Results concerning the Curie temperature and the initial polarization of the samples are also presented.**

***Keywords*—piezoelectric effect; capacitor dielectric; thick film paste; screen printing**

I. Introduction

Thick film capacitor dielectric pastes with relative permittivity in the range of up to 12000 are commercially available for about two decades [1]. Most of these pastes are compatible with standard thick film process on alumina and there exist several products which were designed for burying in low temperature cofired ceramic tape (LTCC). To achieve high dielectric constants, substances must be used which are similar in their structure to popular piezoelectric materials like PZT or BaTiO3, so they also show piezoelectric behaviour. The effect of the piezoelectricity is well known for multilayer ceramic chip capacitors, manifesting itself as microphonics on one hand and as sound emission under switching conditions on the other.

In this work piezoelectric properties of several high-K dielectric pastes from ESL ElectroScience (now Ferro Corporation) were investigated. The primary goal was to determine if the substantial piezoelectric effect in the printed high-K dielectrics can be observed and, if applicable, to find the necessary conditions for polarising the film (electric field strength, duration of the process and its temperature). The piezo effect can, on one hand, cause problems in printed capacitors with respect to microphonics and actuator action. On the other hand, it could prove sensible to use these printed films in piezoelectric components: advantages would be that they are readily available, have compatible electrode and sealant pastes and are long proven in production.

In order to estimate whether applications as sensor or actuator films are possible, the aging rate of piezoelectric response must also be known. In the constructed experiments the generated charge in typical sensor configurations (normal load and bimorph bending) was measured. This charge signal can be used directly to find achievable sensor properties like signal amplitude and signal to noise ratio. Additionally, piezoelectric coefficients were calculated from the generated charge using methods described below to allow a better comparison with typical piezoelectric materials.

A preliminary elemental analysis performed on the base of X-Ray fluorescence spectra showed that the dielectrics are built with Bi/Ba/Zn, Pb/Ga and Pb/Nb/Ga systems. It is known from the literature that the Curie temperature of Pb/Nb systems can be as low as –15°C [2], which would prohibit any practical piezoelectric applications. That is why the measurement of the Curie temperature of the dielectrics was one of the goals during the investigations.

II. Sample Preparation

Samples were screen printed on 96% alumina substrates with the size 50.8 mm x 50.8 mm and the thickness of 0.63 mm. Every substrate contained initially four identical test structures in a form of planar capacitors comprising electrode layers separated by two or three layers of dielectric. The electrodes had a diameter of 12 mm, the total thickness of dielectric film ranged from 30 µm to 50 µm. After the last firing step (top electrode) the samples were singulated as shown in Fig. 1.

All layers of the test structure were separately printed, dried and fired; used materials and process parameters are given in Table I. It is known from literature that conductor pastes used for building the capacitor stack can greatly influence relative permittivity and piezoelectric response of the dielectric [1], [3]. For our tests not only the recommended material combinations were used, as indicated in the table.

TABLE I. Thick film pastes used for test structures.

Dielectric	Electrode		Firing Conditions
	Recommended	***Used***	
4113-H	9638	9635-G	930°C / 1 h
4117	9638	9635-G	930°C / 1 h
4163-N	9916	9912-K	850°C / 1 h
4212-C	9916, 9516, 8816	9516	850°C / 1 h

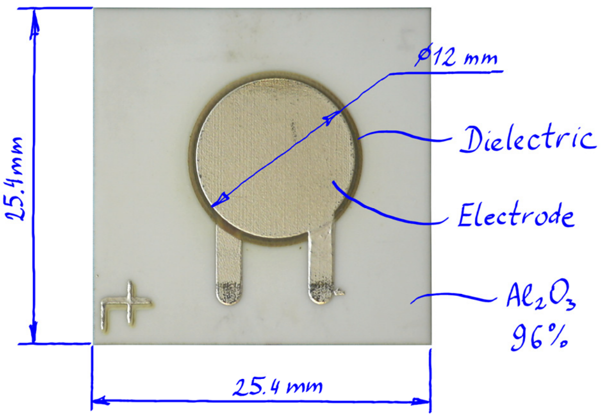

Fig. 1. Measurement sample with the test capacitor structure.

III. Physical Background and Experimental Setup

A. Hysteresis Loops and Relative Permittivity

To acquire hysteresis P-E loops a modified Sawyer-Tower circuit with numerical charge integration was used [4] (Fig. 2). This allowed measuring loops at frequency as low as 50 mHz. Compensation of the circuit was performed digitally; following effects were taken into account: (a) input offset voltage of the acquisition card, which manifests itself as a linear rise (or fall) of the calculated charge on the electrodes, (b) dielectric loss in the material leading to a phase shift of the current in the shunt resistance. The charge offset (integration constant) was defined from the symmetry of the ac signal.

The relative permittivity ε_r was calculated from the slope of P-E curves and, additionally, from the capacitance of the test structure. Measurements of capacitance were carried out using an automatic RLC-bridge at the frequency of 1 kHz.

B. Measurement of Piezoelectric Coefficients

Charge collected on the electrodes of the printed test structure is defined by the electric displacement D_3 in the dielectric material, which is given as (see e.g. [5] or [6]):

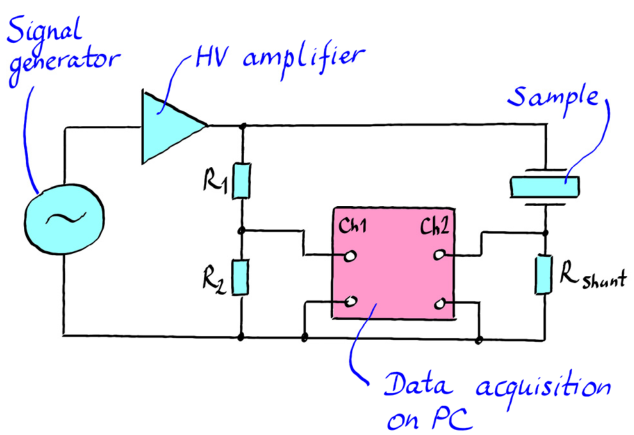

Fig. 2. Modified Sawyer-Tower circuit used for measurement of hysteresis loops.

$$D_3 = d_{31}T_1^f + d_{32}T_2^f + d_{33}T_3^f + \varepsilon_{33}^T E_3, \tag{1}$$

where $d_{31} = d_{32}$ and d_{33} denote piezoelectric coefficients, T_i^f are components of the stress tensor in the dielectric film, ε_{33}^T is the dielectric permittivity at constant stress and E_3 is electric field strength. This equation was used to find the piezoelectric coefficients from the direct piezoelectric effect.

To determine d_{33} the in-plane stress components T_1^f and T_2^f as well as the electric field E_3 were held at zero ("no clamping" and "short-circuit" conditions respectively). The experimental setup was very similar to the standard Berlincourt-type measurement (Fig. 3), which relies on measurement of change in electric displacement due to the introduced change in stress. The sample loaded by a spherical mass was put on the shaker platform, which moved vertically with amplitude 10 µm and frequency 40 Hz. The acceleration of the platform ($a_{ac} = 0.446\ \mathrm{m/s^2}$, rms value) created a sinusoidal force F_{ac} in addition to the static preload due to the weight F_{dc} of the sphere. Given the mass of the sphere $m_{sp} = 70.4$ g the forces can be calculated:

$$F_{dc} = m_{sp} \cdot g = 0.69\ \mathrm{N},\ F_{ac} = m_{sp} \cdot a_{ac} = 3.14 \cdot 10^{-2}\ \mathrm{N}.$$

The coefficient d_{33}^{eff} was then determined as

$$d_{33}^{eff} = \frac{\delta D_3}{\delta T_3^f} = \frac{\delta Q}{F_{ac}}$$

where δQ stands for the rms value of the measured charge. Since a thin dielectric film is used in the test structure, the produced piezoelectric response is described by an effective coefficient d_{33}^{eff} instead of the bulk material coefficient d_{33}. This coefficient is a linear combination of d_{33} and d_{31} which is explained by lateral clamping of the thin piezoelectric layer to the substrate [7], [8].

It is known that d_{33} values measured by the direct (Berlincourt) method depend strongly on sample and loading geometry, working frequency and preload force and have a

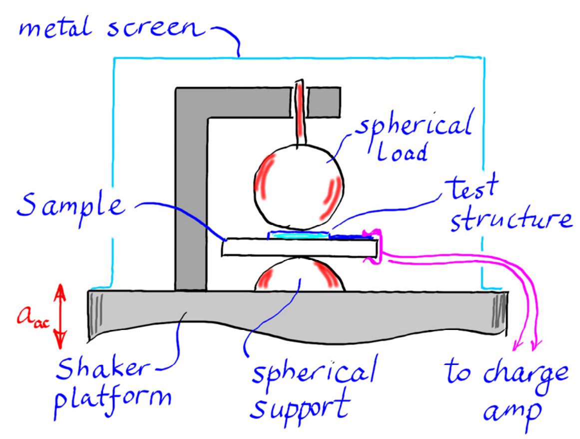

Fig. 3. Principal drawing of the setup for measurement of d_{33} coefficient (Berlincour method). The shaker platform moves with an amplitude of 10 µm at a frequency of 40 Hz. Mass of the spherical load is 70.4 g.

relatively high variability ([9], [10]) but it is suitable to quickly characterise the samples. In our measurements, where the goal was not to determine the exact piezoelectric coefficients but to find conditions for polarising the dielectric and to measure the time dependence of relaxation, this direct measurement of d_{33}^{eff} was absolutely sufficient.

A substantially better sensitivity and a higher signal-noise ratio than for the Berlincourt-like approach was achieved by a direct measurement of d_{31} response. This type of measurement allowed determining piezoelectric coefficients well below 1 pC/N, using experimental setup presented in Fig. 4. Due to the load and to the fixture, the oscillation of the shaker platform introduces a bending stress in the sample (about the y axis), resulting in the normal stress in the dielectric film (along the x axis). Since stresses in y and z directions are not induced, and the electric field in the film is held at zero by the charge amplifier, the piezoelectric response d_{31} can be calculated from (1) as:

$$d_{31} = \frac{\delta D_3}{\delta T_1^f}$$

The electric displacement δD_3 is determined as the ratio of the generated charge to the electrode area; the stress in the film is found from the elastic beam theory (e.g. [11]) in a simplified approach described below. It is assumed, that the printed dielectric and electrodes layers do not influence the bending of the alumina substrate but are deformed with the same strain as the outer substrate fibre. This assumption can be corroborated by the geometry of the samples: the printed layers have a total thickness of about 10% of the alumina substrate and a lower Young's modulus.

The model for calculations is given in Fig. 5, showing the substrate of a mass m_s as a cantilever beam fixed in a wall at its left end. Vertical oscillations of the wall lead to an oscillating bending moment in the region of the printed test structures – they are on the bottom side of the beam at the distance l_1 from the wall. The portion of the beam denoted with l_2 and the load m_l fixed to the right end of the beam at the distance l_3 contribute to the bending moment in the centre of the dielectric film. The bending moment at l_1 is given by

$$\delta M = \left(m_s \frac{l_2^2}{2b} + m_l l_3\right) \cdot a_{ac}$$

where a_{ac} denotes the vertical acceleration of the system due to oscillations of the platform (rms value), b is the width of the substrate in y direction. The outer beam fibre of the substrate with thickness h would experience stress in x direction

$$\delta T_1^s = \delta M \frac{6}{bh^2}$$

Assuming same strains in the printed layer and in the outer substrate fibre adjacent to it, the absolute value of the stress in dielectric film T_1^f can be calculated:

$$\delta T_1^f = \delta T_1^s \frac{Y_f}{Y_s} = \left(m_s \frac{l_2^2}{2b} + m_l l_3\right) \cdot \frac{6a_{ac}}{bh^2} \cdot \frac{Y_f}{Y_s}$$

Here Y_s and Y_f are the Young's modulus values for the substrate and dielectric film respectively. For alumina $Y_s =$ 340 GPa was taken [12], the modulus for the dielectric layer was assumed to be $Y_f \approx 60$ GPa based on literature values for screen-printed PZT layers [13], [14] and [15].

To investigate the variability of results and characterise the setup of Fig. 4 a series of measurements using a sample with a low value of d_{31} was carried out. Between single readings the sample was replaced in order to reproduce all possible placement mismatches. The result of this series are given in Fig. 6: the variation of the measured charge signal lies within $\pm 10\%$ of the average value – this value was taken as the measurement error in further experiments.

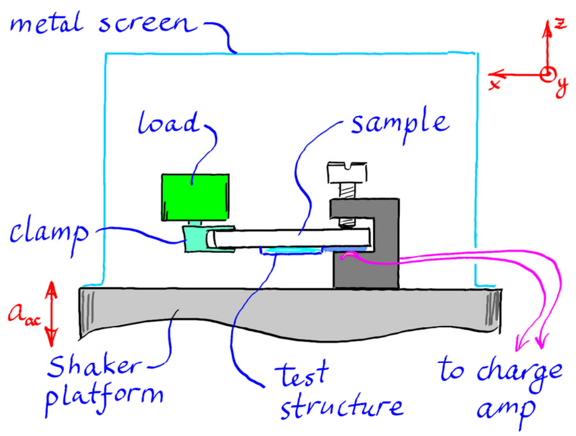

Fig. 4. Principal drawing of the setup for direct measurement of d_{31} coefficient.

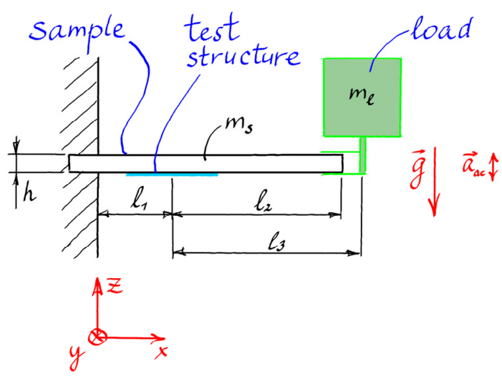

Fig. 5. Measurement setup as an elastic beam model. Dimensions: $l_1 = 7.5$ mm, $l_2 = 11.5$ mm, $l_3 = 14.5$ mm, $h = 0.63$ mm, substrate width $b = 25.4$ mm, $m_s = 1.6$ g, $m_l = 12.0$ g. Acceleration of the platform $a_{ac} = 0.446$ m/s^2 (rms value).

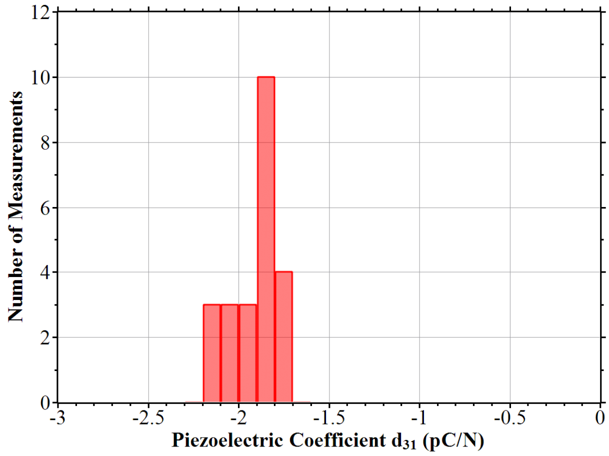

Fig. 6. Reproducibility of results in the direct measurement of d_{31}.

IV. Results and Discussion

As the experiments demonstrated, all tested dielectrics can be polarised under certain conditions and have significant piezoelectric coefficients. Their values and time stability are inferior to PZT ceramics, but are worth considering because of possible negative effects or potential sensor and actuator applications. Two of dielectrics were more extensively investigated: 4212-C, because of the highest measured piezoelectric response, and 4163-N, because of its capability of processing in co-firing with LTCC tapes.

The main goals of the experiments were

- to find conditions which lead to polarising and depolarising of tested dielectric materials,
- to measure piezoelectric properties and
- to determine the aging rate of piezoelectric response.

The results of the investigations are given below alongside with the discussion.

A. Hysteresis Loops

Fig. 7 shows P-E loops measured at the frequency of 5 Hz (the loop for 4212-C dielectric reproduces polarisation value divided by ten for a better overall representation). It is clearly seen, that samples of 4113-H, 4117 and 4163-N dielectrics do not show ferroelectric polarisation effects – the loops represent a linear capacitance with a relatively low loss factor. This result is anticipated because these dielectrics are intended for use in printed capacitors. In the P-E loop of 4212-C dielectric (it has the highest permittivity of all samples) the non-linearity of response is obvious, showing a similarity to the response of typical piezoelectric materials. Hysteresis loops of printed thick PZT films found in the literature [3], [15] and [16] exhibit much more pronounced remanent polarisation at 5 Hz and 50 Hz frequencies. The value of maximum polarisation itself is very close to the literature values for printed PZT films at the same electric field.

The 4212-C dielectric shows a frequency dependency in its hysteresis loops: lower frequencies lead to a higher maximum achieved polarisation (Fig. 8). In the same time the area within the loop rises, giving higher values of the remanent polarisation and coercive field. The rise is very limited, so these loops describe rather a lossy capacitor than a piezoelectric device. On the other hand, the rise can be interpreted as an indication of the possibility to polarise this material if the duration of the electric field action is increased. Indeed, it was found that the field must be applied for at least several minutes to achieve significant remanent polarisation at room temperature (see Fig. 14 below).

B. Relative Permittivity

Relative permittivity ε_r was calculated from the slope of $P(E)$ curves at $E = 0$ (frequency 5 Hz) and from the measured capacitance of the samples (frequency 1 kHz). In the first case expression $\varepsilon_r = \frac{\Delta P}{\varepsilon_0 \Delta E} + 1$ was used, in the second case equation $\varepsilon_r = \frac{C h_f}{\varepsilon_0 A}$ (here h_f denotes the thickness of dielectric film, A is the area of the capacitor). The results are given in Table II in comparison with the manufacturer datasheet values. It can be seen, the measured values are in a good agreement with the manufacturer's data.

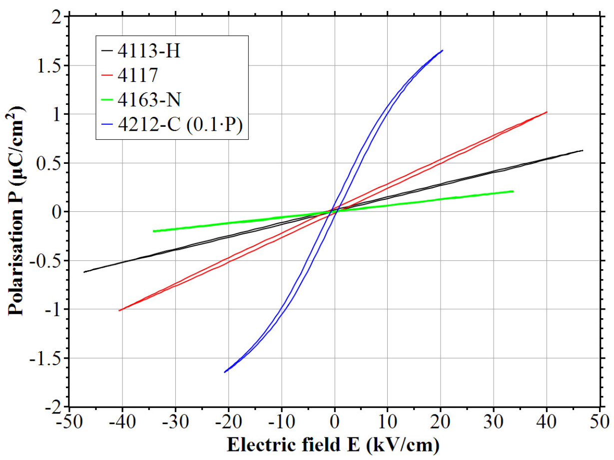

Fig. 7. P-E loops of tested dielectrics at 5 Hz. Note: the curve of 4212-C dielectric is drawn with the polarisation reduced by a factor of 10.

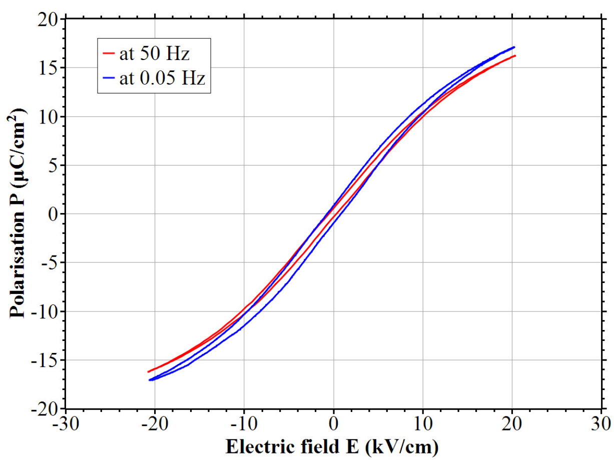

Fig. 8. P-E loops of 4212-C dielectric at different frequencies. At the lower freqency a slight increase in the area inside the loop is seen.

TABLE II. RELATIVE DIELECTRIC PERMITTIVITY OBTAINED FROM P-E HYSTERESIS LOOPS AND CAPACITY MEASUREMENTS IN COMPARISON WITH DATASHEET VALUES.

	Relative permittivity ε_r		
Dielectric	**datasheet**	**hysteresis**	**capacitance**
4113-H	120-160	150	138
4117	270-330	285	242
4163-N	70-130	69	68
4212-C	10500-13500	12500	9900

C. Curie Temperature

Temperature dependence of relative permittivity at low field conditions can be seen from the change of sample capacitance with temperature $C(T)$; as an example temperature dependencies for 4212-C dielectric are presented in Fig. 9 for temperatures between 25°C and 240°C. Up to the temperature of 120°C published manufacturer's data exist [1], in this region the obtained dependence is in a good agreement with the literature.

The temperature region for the measurement of $C(T)$ was chosen based on the anticipated Curie temperature of the material – the relative permittivity (and thus capacitance) should show a distinct peak at this point because of a phase transition taking place. Measured dependencies show, however, smooth curves in the evaluated temperature span, and no direct indication of a phase transition was found. As the Curie point T_{Curie} limits the highest possible temperature for the operation of devices based on piezo effect, knowledge of this point is important.

The approximate value of the limiting temperature was determined observing changes of piezoelectric coefficients after heating. The following criterion was employed: piezoelectric response of a sample heated during 10 minutes at a given temperature (no voltage applied) should fall by at least 95% of the original value. The temperature T_{depol} found in this way would lie below Curie temperature, but it would lead to a rapid depolarisation prohibiting practical application of such devices. Obtained values for the depolarisation temperature are given in Table III.

TABLE III. TEMPERATURE T_{depol} LEADING TO THE DEPOLARISATION OF THE SAMPLES WITHIN 10 MINUTES.

Dielectric	T_{depol} (°C)
4163-N	185
4212-C	115 - 135

D. Spontaneous Initial Polarisation

An unexpected effect with a high potential impact was found for capacitors with 4212-C dielectric: all produced samples possessed initial polarisation directly after the last firing step (top electrode). This spontaneous initial polarisation of the samples was always directed towards the bottom electrode (i.e. as if the samples were polarised with the positive pole of the voltage on the top electrode). The value of initial polarisation did not depend on the thickness of the dielectric (number of printed layers). In Fig. 10 measured values of the dielectric constant d_{31} due to initial polarisation are given for all produced samples. Here the data for samples with two and three printed dielectric layers are combined.

If the samples were polarised by an electric field as described below and subsequently depolarised, they always returned to the initial polarised state with the initial piezoelectric coefficient. It means, heating does not destroy the initial polarisation. This behaviour could be of concern for electronics using such printed capacitors: because of the electric charge generated by the capacitors under action of mechanical forces the circuit could be prone to noise signals due to microphonics. On the other hand, if the reproducibility of the initial polarisation is given in the production, sensor or actuator applications with high time stability could be constructed.

The origin of the described effect is not clear at the moment and must be further investigated.

E. Polarisation of Samples and Aging

Polarisation of the samples for the aging test was carried out under electric field of 20 kV/cm, which was applied during 50 minutes (except for dielectric 4163-N, which was polarised at 15 kV/cm in this experiment). Table IV summarises achieved piezoelectric coefficients measured 20 minutes after withdrawal

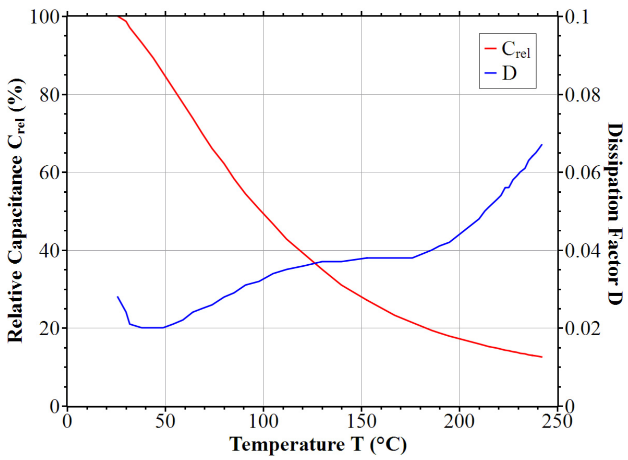

Fig. 9. Temperature dependence of capacitance and dissipation factor for dielectric 4212-C.

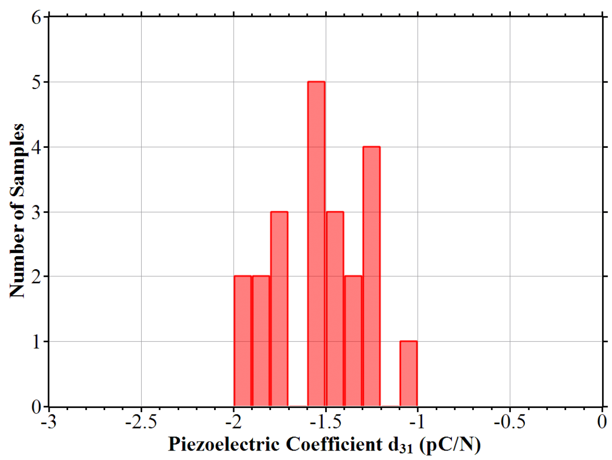

Fig. 10. Spontaneous initial polarisation of samples with 4212-C dielectric.

of the polarising electric field. Most dielectrics required elevated temperature for polarisation, the only one, which was possible to polarise at room temperature, was 4212-C.

The samples were stored at room temperature after polarisation, and their piezoelectric coefficient d_{31} was monitored during 3.5 months. Fig. 11 and Fig. 12 show the results and the approximated time dependence; slopes of the lines are given in Table IV as aging rate in %/decade.

TABLE IV. PIEZOELECTRIC COEFFICIENTS AND THEIR AGING RATE.

Dielectric	d_{31} (pC/N)	d_{33} (pC/N)	Aging rate (%/decade)	T_{pol} (°C)
4113-H	–0.29		11	150
4117	–0.26		11	150
4163-N	–0.17		6	150
4212-C	–56	105	41	100
4212-C	–56		57	25

As it can be seen from Table IV, initial values of d_{31} and d_{33} of 4212-C dielectric are comparable to the coefficients of printed PZT films [13], [17] and of established piezoelectric materials like barium titanate. The aging rate of the piezoelectric response is, however, much higher than for the bulk PZT, for instance, and is dependent on the temperature, at which polarisation was carried out. Notwithstanding the high aging rate, it can be anticipated from Fig. 11 that piezoelectric coefficients of dielectric 4212-C polarized at 100°C would be higher than of quartz ($\approx$ 3 pC/N) for about twenty years. Taking this into account, the piezoelectric response – desired or not – could be noticeable for a long time once polarisation took place.

Other tested dielectrics showed piezoelectric coefficients well below 1 pC/N but a relatively moderate aging rate compared with the typical aging rate of about 1%/decade for bulk PZT.

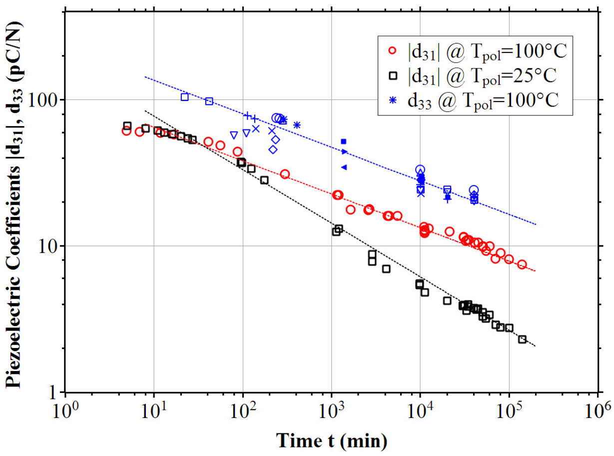

Fig. 11. Time dependence of the piezoelectric response for dielectric 4212-C polarised at 25°C and 100°C. Different plot symbols for d_{33} denote different measurement points on the sample.

F. *Variation of Polarisation Parameters*

Another measurement series was devoted to establishing the dependence of piezoelectric coefficient d_{31} on electric field during polarisation of samples. Here only dielectrics 4163-N and 4212-C were examined. Dielectric 4212-C was polarised at room temperature, dielectric 4163-N – at $T_{pol} = 150°C$. Polarisation lasted 30 minutes in all experiments; the piezoelectric response was measured 20 minutes after polarisation.

Coefficients d_{31} given in Fig. 13 and in the following figures are normalised for an easier comparison of different dielectrics. The curves indicate that the value of polarising field E_{pol} needed for reaching the saturation in piezoelectric response lies at about 20 kV/cm. Breakdown voltage specifications in the manufacturer's data sheet state that an electric field of at least 40 kV/cm should be possible without an overglaze layer. We could not achieve these values with our samples, probably due to not absolutely continuous printed dielectric layers, so further investigations to confirm the value of saturation field must be carried out. For 4212-C in the region of electric field below 15 kV/cm the dependency of d_{31} is approximately linear and indicates that polarisation at room temperature takes place even at low electric fields (Fig. 13).

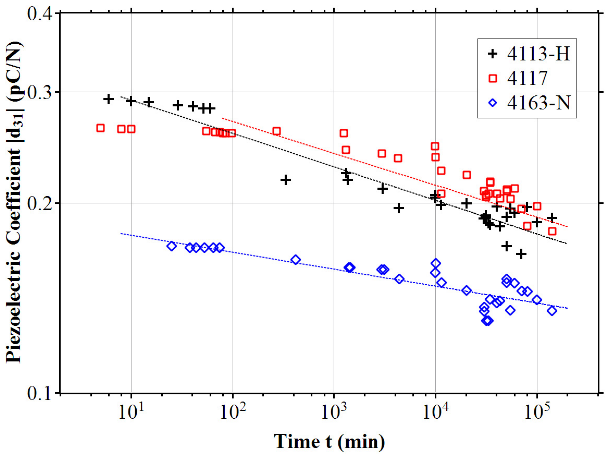

Fig. 12. Time dependence of the piezoelectric response for dielectrics 4113-H, 4117, 4163-N. Slopes of lines are given in Table IV.

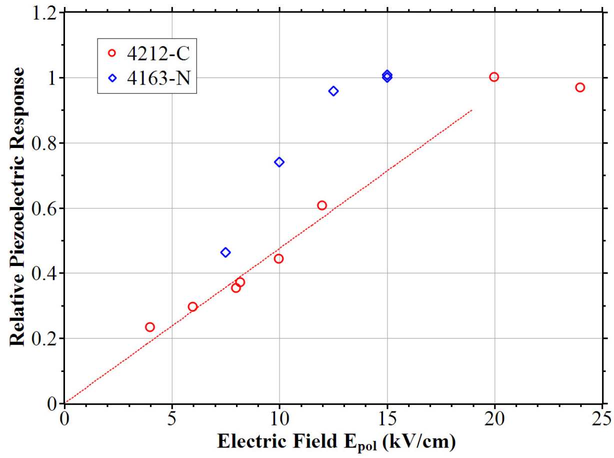

Fig. 13. Piezoelectric coefficients d_{31} obtained at different polarisation fields (normalised).

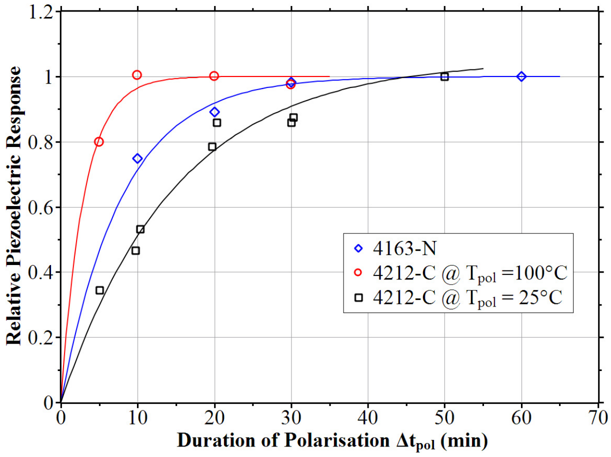

Fig. 14. Piezoelectric coefficients d_{31} obtained for different duration of polarisation (normalised).

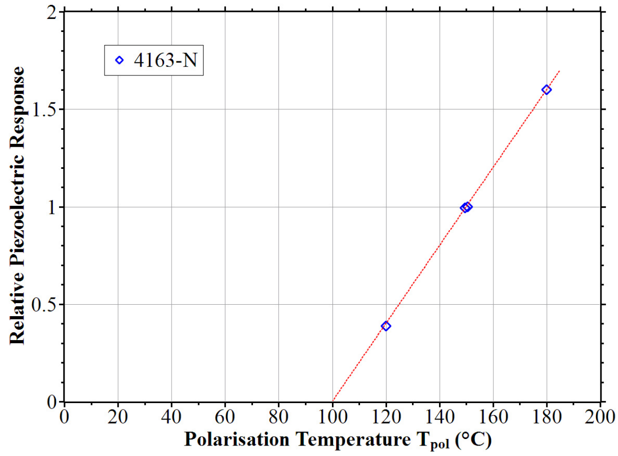

Fig. 15. Piezoelectric coefficients d_{31} (normalised) obtained after polarisation of dielectric 4163-N at different temperatures.

The effect of changing the duration of polarisation process on achieved piezoelectric response is illustrated in Fig. 14. Here the normalised coefficient d_{31} for dielectrics 4163-N and 4212-C is presented. Polarisation was carried out at $T_{pol} = 150°C$ for 4163-N; dielectric 4212-C was polarised at room temperature and at $T_{pol} = 100°C$. Electric field used for polarisation amounted to 20 kV/cm for dielectric 4212-C and to 15 kV/cm for dielectric 4163-N. Readings of d_{31} were done 20 minutes after withdrawal of electric field; the experimental data are approximated by exponential functions. As seen from the figure, polarisation field must be present for at least 10 minutes (dielectric 4212-C at 100°C) to about one hour (the same dielectric at room temperature) to reach the highest possible degree of polarisation.

The temperature dependence of polarisation results was measured for dielectric 4163-N. Fig. 15 shows obtained coefficient d_{31} normalised to its value at $T_{pol} = 150°C$, which was used in the experiments described above. Polarisation lasted 30 minutes and the piezoelectric response was measured 20 minutes after polarisation. As it can be seen from the figure, the temperature must be higher than approximately 100°C to make polarisation effective. Further temperature rise allows to achieve higher piezoelectric coefficients; in the measured range the temperature dependence can be approximated by a straight line.

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