



Comparison of Methods for the Measurement of Piezoelectric Coefficients

FIALKA, J.; BENEŠ, P.

IEEE Transactions on Instrumentation and Measurement

Vol. 62, Iss. 5, pp. 1047-1057

ISSN: 0018-9456

DOI: <https://doi.org/10.1109/TIM.2012.2234576>

Accepted manuscript

©2022 IEEE. Personal use of this material is permitted. Permission from IEEE must be obtained for all other uses, in any current or future media, including reprinting/republishing this material for advertising or promotional purposes, creating new collective works, for resale or redistribution to servers or lists, or reuse of any copyrighted component of this work in other works. FIALKA, J.; BENEŠ, P. „Comparison of Methods for the Measurement of Piezoelectric Coefficients“, IEEE Transactions on Instrumentation and Measurement. DOI: 10.1109/TIM.2012.2234576. Final version is available at <https://ieeexplore.ieee.org/document/6410416>

Comparison of Methods for the Measurement of Piezoelectric Coefficients

Jiří Fialka*, Petr Beneš**

*CEITEC – the Central European Institute of Technology,

**Department of Control and Instrumentation

Faculty of Electrical Engineering and Communication

Brno University of Technology, Brno, Czech Republic

Email: jiri.fialka@ceitec.vutbr.cz, benesp@feec.vutbr.cz

The charge constant of piezoelectric material is one of the crucial constant values. At the present time, the measurement of this value is mostly realized by means of three techniques: the frequency method, the laser interferometer technique, and the quasi-static method. These techniques have been practically applied to piezoelectric ceramic samples. Our paper presents a comparison of the individual methods with regard to their accuracy and the demands placed on the instrumentation and preparation of the piezoelectric material samples. A “soft” ceramic product marketed under the production code of NCE51 was used in the experiments. The methods are described in detail, including the process of sample preparation, description of the experiments, and procedure of calculating coefficients from the measured values.

Index Terms: PZT ceramics; Piezoelectric materials; Piezoelectric resonators; Piezoelectricity; Resonance; Impedance measurement; Laser interferometer.

Introduction

When designing piezoelectric actuators and sensors, it is important to consider the catalogue values of the used material. One of the constants describing the behavior of a piezoelectric material is the piezoelectric charge constant. Multiple measuring methods are used to provide the value of this coefficient. At present, the most common measurement approaches consist in the three following methods:

- the Frequency method
- the Laser interferometry method
- the Quasi-static method

All the aforementioned techniques are used very frequently, mainly because they provide high accuracy results. Their common disadvantage, however, can be identified in the fact that they require specialised and accurate measuring devices [1], [2], [3].

The frequency method is used to obtain the piezoelectric coefficient in cases where the complete matrix of the material coefficients has to be known. The resulting values strongly depend on the accuracy achieved within the reading of the resonance frequencies and on other values required for the calculation of all the material constants. Thus, for this type of measurement, an impedance analyzer is regularly applied as it ensures high measuring accuracy. The most regularly used are the impedance analyzers made by Agilent and Wayne Kerr, namely the Agilent E4294A and the Wayne Kerr 65120B, respectively. The devices differ in terms of sensitivity and frequency range. The greatest disadvantage of the frequency method is the required construction of a complete set of samples made up of a disc, a plate, and a cylinder from a piezoelectric material of one rank. The calculation to be carried out within the frequency method conforms to the European standard EN 50324-2 [4] derived from the world standard CEI/IEC 60483:1976 [5].

The second possible method of measuring the piezoelectric charge constant is laser interferometry. This method is based on measuring the displacement deflection of the sample surface after the connection of voltage to the electrodes of the measured piezoelectric ceramics. High resolution of the interferometer, which should be within units of nanometer, is a crucial precondition here. At present, the described method is most widely used in laboratory conditions. The main reasons for this situation consist in the high price of an interferometer and the intensive requirements placed on the insulation of the measuring centre from any parasitic vibrations. The laser interferometry method is applied for the measurement of the piezoelectric charge coefficients d_{31} and d_{33} . The disadvantage of the method consists in the uncompromising necessity to operate precisely constructed, heavy-duty measuring devices, since any small irregularities or vibrations during the measurement

significantly affect the accuracy of the obtained values. Interferometers with sufficient resolution are produced by manufacturers such as Polytec, Lasertex, Agilent, or SIOS [6], [7].

The last technique applied determines the piezoelectric charge constant through the quasi-static approach to measurement. This method does not involve the costly process of establishing all the material constants concerned and, as in the case of the laser interferometry method, there is no need to provide a complete set of measured samples. The direct and converse piezoelectric effects have to be taken into account within the measurements. To acquire the piezoelectric charge constant, it is necessary to compare the tested sample to the reference sample of a known piezoelectric coefficient. The electric charge on the piezoelectric sample can be measured using charge amplifiers or voltmeters with high input resistance such as the Keithley 6217 [8]. Within the quasi-static method, a “ d_{31}/d_{33} meter” is used. At present, the devices are made by KCF Technologies (type PM350), HC Materials Corporation (type ZJ-6B), and Sensor Technology Ltd. (type SS01-01). All the aforementioned devices can measure both the d_{33} and the d_{31} coefficients, while the other types are designed to measure only the d_{33} coefficient; apparatuses within the former group mainly differ in prices [9].

Measurement

The aim of the measurements was to compare the different methods of establishing the piezoelectric charge coefficient and to evaluate the advantages and disadvantages of the used techniques. The piezoelectric charge constant is a characteristic material constant established during mechanical deformation of the sample. With this constant, the force applied to the sample can be transmitted to the generated electric charge in a desired direction, according to the relation stated below [7].

$$Q = d_{ij} \cdot F, \quad (1)$$

where Q is the charge created through the pressure applied to the sample (C), F is the applied force (N), and d_{ij} is the piezoelectric charge constant (C/N or m/V).

Measured samples

In all the experiments, we used the “soft” piezoelectric ceramics previously referenced as PCM51 (now bearing the ref. code of NCE51) produced by the Noliac Ceramics company. To establish the complete matrix of piezoelectric ceramic coefficients, we need to have the complete set of samples as shown in Figure 2. Particularly suitable in this respect is the process where all the needed samples are obtained from a single disc of the appropriate size, as shown in Figure 1. This production process has already been described in [10] and [11].

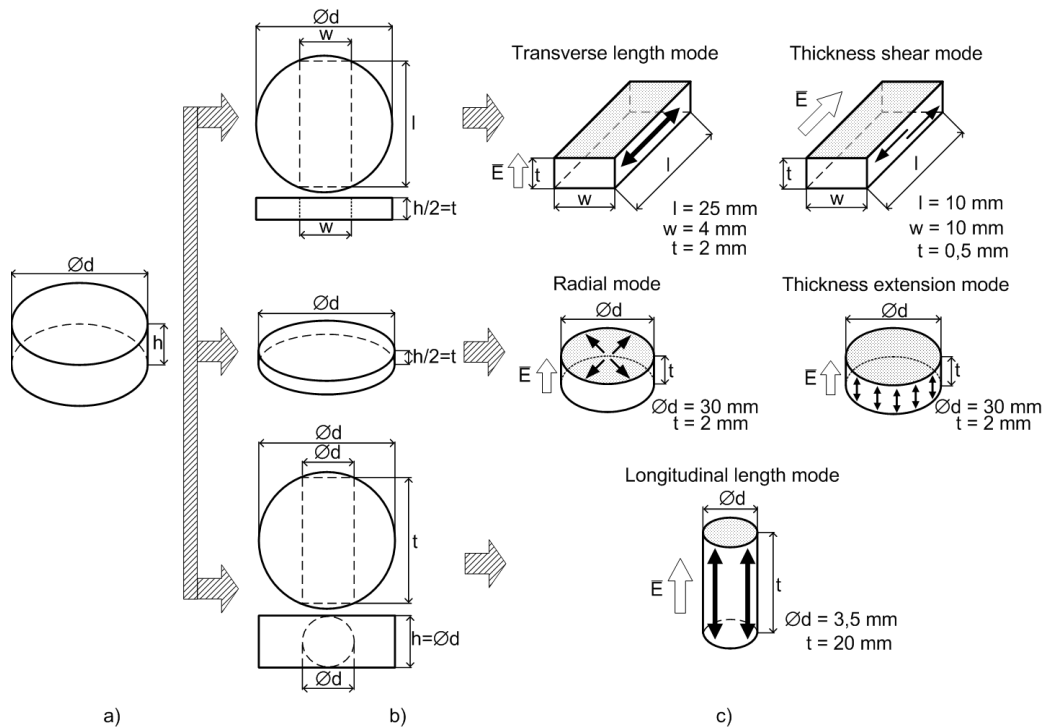


Figure 1. Process of the production of samples for the measurement of parameters. Here, a) is an original disc, b) shows the production of a thin disc, a cylinder, and a prism, c) shows the final production of a thin plate for the transverse length mode and the thickness shear mode, a thin disc for the radial mode and the thickness extension mode, and a cylinder for the longitudinal length mode [14], [15].

The measured samples had to be manufactured before the measurements and calculations were carried out. The sample dimensions have to conform to the European standard EN 50324-1:2002 [12] and the world standard CEI/IEC 60483:1976 [5], [13]. As only a few samples are needed for the measurements, the production of such a small number of pieces would be extremely costly. A disc of $\varnothing d = 30$ mm and $t = 4$ mm was chosen for the manufacturing of the whole set of the measured samples. This disc was first lapped by flat grinding, whereby a thinner disc of the same diameter was produced. Then, a cylinder was made by means of cutting a prism out of the thin disc, which was afterwards abraded cylindrically. For later comparison of the charge constant value d_{33} , it is good to keep some prisms of a square base and of the same height as the cylinder. Samples produced in this way need to be polished on the surface to ensure good contact of the silver electrodes and to provide the best possible conductivity between the ceramics and the metal.

Frequency measurement method

An accurate Agilent 4294A impedance analyzer was used with a Tweezers Contact Test Fixture 16334A, which was directly connected to the impedance analyzer. The entry parameters of the piezoelectric ceramics, resonant frequencies f_s , antiresonant frequencies f_a , impedance, and free capacitance C^T on 1 kHz were measured by the impedance analyser. The dimensions of the piezoelectric samples were measured by callipers, and the related values are shown in Figure 1. The density was the last measured parameter [16], [17].

The Agilent 4294A impedance analyzer facilitates direct displaying of the impedance and phase characteristics on the device screen, and the screen content can be directly saved as an image. Automatic data reading from the device can be used and processed in, for example, an Excel editor: It is possible to connect the device to a personal

computer through the GPIB bus. The direct displaying of the impedance and phase characteristics during this measurement prevents any possible confusion of the double peak regarding the resonance and antiresonance frequency measurement; this problem is a difficulty that can be encountered in devices where it is not possible to directly display the characteristics during the measurements. Within the described context, we used LabVIEW to create a service program enabling the automatization, simplification, and marked acceleration of the search for resonance frequencies. The piezoelectric d , dielectric ε , and elastic s material constants with electromechanical properties are anisotropic and exhibit various values in different directions relative to the direction of polarization. Tensor components of the material coefficients can be written in a simplified matrix form by using symmetry. The complete matrix of the coefficients of electromechanical properties on the piezoelectric ceramics is written as [18], [19], [20]:

$$\begin{bmatrix} S_1 \\ S_2 \\ S_3 \\ S_4 \\ S_5 \\ S_6 \\ D_1 \\ D_2 \\ D_3 \end{bmatrix} = \begin{bmatrix} s_{11}^E & s_{12}^E & s_{13}^E & 0 & 0 & 0 & 0 & 0 & d_{31} \\ s_{12}^E & s_{11}^E & s_{13}^E & 0 & 0 & 0 & 0 & 0 & d_{31} \\ s_{13}^E & s_{13}^E & s_{33}^E & 0 & 0 & 0 & 0 & 0 & d_{33} \\ 0 & 0 & 0 & s_{55}^E & 0 & 0 & 0 & d_{15} & 0 \\ 0 & 0 & 0 & 0 & s_{55}^E & 0 & d_{15} & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 2 \cdot (s_{11}^E - s_{12}^E) & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & d_{15} & 0 & \varepsilon_{11}^T & 0 & 0 \\ 0 & 0 & 0 & d_{15} & 0 & 0 & 0 & \varepsilon_{11}^T & 0 \\ d_{31} & d_{31} & d_{33} & 0 & 0 & 0 & 0 & 0 & \varepsilon_{33}^T \end{bmatrix} \cdot \begin{bmatrix} T_1 \\ T_2 \\ T_3 \\ T_4 \\ T_5 \\ T_6 \\ E_1 \\ E_2 \\ E_3 \end{bmatrix} \quad (2)$$

The complete matrix of piezoelectric constants can be established according to the scheme in Figure 2. The procedure for the calculation of single coefficients is described in detail in the European standard EN 50324-2:2002 [4] and the world standard CEI/IEC 60483: 1976 [5], [13].

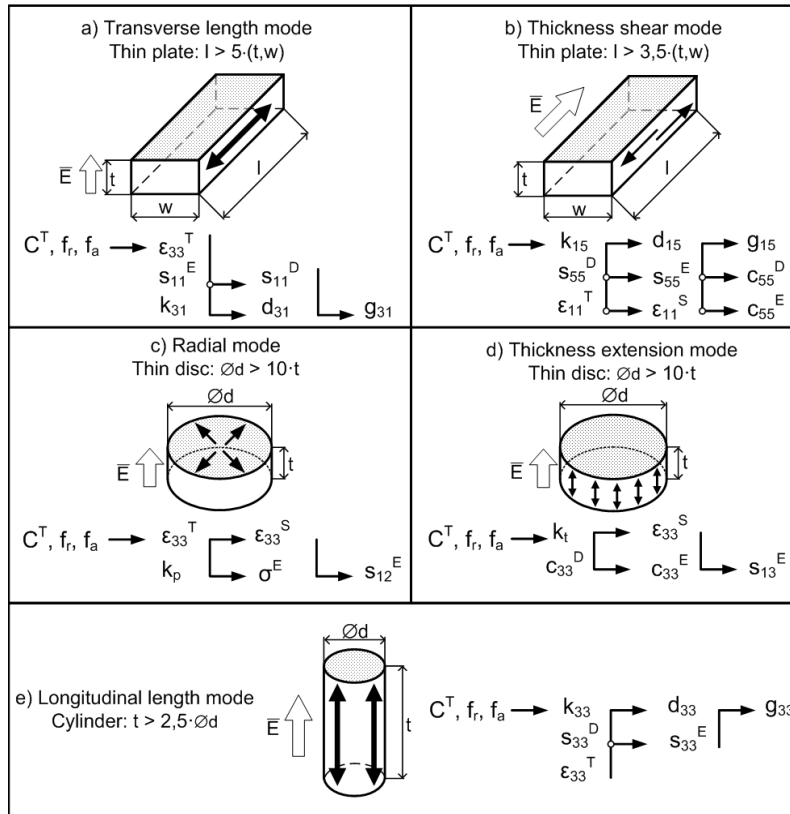


Figure 2. Establishing the complete set of material coefficients defined by the EN 50324-2:2002 [4] and CEI/IEC 60483: 1976 standards [5], [13], [14].

The frequency method was used for the measurements and for the calculation of all the material coefficients [16], [21].

The elastic coefficients $s_{11}^E, s_{12}^E, s_{13}^E, s_{33}^E, s_{55}^E, s_{66}^E$ are calculated from the electromechanical coupling coefficients. The initial parameters are resonant frequencies, density, and the sample dimensions [7].

In the text below, relations for individual material coefficients of the piezomaterials are provided. Applying suitable modification, we derived the relations from formulas presented within standards [4], [5], because direct substitution of the measured parameters (Figures 2, 3, and 4) cannot be realized by means of commonly used relations.

The equation related to the elastic coefficient s_{11}^E for the transverse length mode in the thin plate:

$$s_{11}^E = \frac{1}{4 \cdot \rho \cdot f_s^2 \cdot l^2} \quad (3)$$

The equation related to the elastic coefficient s_{12}^E for the radial mode in the thin disc:

$$s_{12}^E = -s_{11}^E \cdot \left(1 - \frac{2 \cdot k_{31}^2}{k_p^2} \right) =$$

$$= -\frac{1}{4 \cdot \rho \cdot f_s^2 \cdot l^2} \cdot \left(1 - \frac{2 \cdot \frac{\pi}{2} \cdot \frac{f_a}{f_r} \cdot \frac{1}{\frac{\pi}{2} \cdot \frac{f_a}{f_r} - \tan\left(\frac{\pi}{2} \cdot \frac{f_a}{f_r}\right)}}{2,51 \cdot \frac{f_a - f_r}{f_r} - \left(\frac{f_a - f_r}{f_r}\right)^2} \right) \quad (4)$$

The relations between the elastic coefficient s_{66}^E through the elastic coefficient transverse length mode s_{11}^E and the radial mode s_{12}^E :

$$s_{66}^E = 2 \cdot (s_{11}^E - s_{12}^E) \quad (5)$$

The equation related to the elastic coefficient s_{33}^E for the longitudinal length mode in the cylinder:

$$s_{33}^E = \frac{s_{33}^D}{1 - k_{33}^2} = \frac{1}{4 \cdot \rho \cdot f_p^2 \cdot t^2} \cdot \frac{1}{1 - \frac{\pi}{2} \cdot \frac{f_r}{f_a} \cdot \tan\left(\frac{\pi}{2} \cdot \frac{f_a - f_r}{f_a}\right)} \quad (6)$$

The equation related to the elastic coefficient s_{13}^E for the thickness mode in the thin plate:

$$s_{13}^E = \left\{ \frac{1}{2} \left[s_{33}^E (s_{11}^E + s_{12}^E) - \frac{s_{11}^E + s_{12}^E}{c_{33}^E} \right] \right\}, \quad (7)$$

where the elastic stiffness constant c_{33}^E equals to:

$$c_{33}^E = c_{33}^D \cdot (1 - k_t^2) =$$

$$= 4 \cdot \rho \cdot f_p^2 \cdot t^2 \cdot \left(1 - \frac{\pi}{2} \cdot \frac{f_r}{f_a} \cdot \tan\left(\frac{\pi}{2} \cdot \frac{f_a - f_r}{f_a}\right) \right) \quad (8)$$

The equation for the elastic coefficient s_{55}^E for the thickness shear mode in the thin plate:

$$s_{55}^E = \frac{s_{55}^D}{1 - k_{15}^2} = \frac{1}{4 \cdot \rho \cdot f_p^2 \cdot t^2} \cdot \frac{1}{1 - \frac{\pi}{2} \cdot \frac{f_r}{f_a} \cdot \tan\left(\frac{\pi}{2} \cdot \frac{f_a - f_r}{f_a}\right)} \quad (9)$$

The equations for the transverse length mode ε_{11}^T (10) and the longitudinal length mode ε_{33}^T in the cylinder (11) in dielectric coefficients are shown below. The entry parameters are capacitance and the sample dimensions [7].

$$\varepsilon_{11}^T = \varepsilon_{3r} \varepsilon_0 = C^T \cdot \frac{t}{A}, \text{ where } A = w \cdot l \quad (10)$$

$$\varepsilon_{33}^T = \varepsilon_{3r} \varepsilon_0 = C^T \cdot \frac{t}{A}, \text{ where } A = \frac{\pi \cdot d^2}{4} \text{ or } A = w \cdot l \quad (11)$$

The entry parameters for the piezoelectric coefficients are resonant frequency, capacitance, density and dimensions [7]. The equation for the piezoelectric charge coefficient d_{31} for the transverse length mode in the thin plate:

$$d_{31} = k_{31} \left(\varepsilon_{11}^T \cdot s_{11}^E \right)^{\frac{1}{2}} = \left(C^T \cdot \frac{t}{4 \cdot \rho \cdot w \cdot l^3} \cdot \frac{\pi}{2} \cdot \frac{f_a}{f_r^3} \cdot \frac{1}{\frac{\pi}{2} \cdot \frac{f_a}{f_r} - \tan\left(\frac{\pi}{2} \cdot \frac{f_a}{f_r}\right)} \right)^{\frac{1}{2}} \quad (12)$$

The equation related to the piezoelectric voltage coefficient g_{31} for the transverse length mode in the thin plate:

$$g_{31} = \frac{d_{31}}{\varepsilon_{33}^T} = \frac{d_{31}}{C^T \cdot \frac{t}{w \cdot l}} \quad (13)$$

The equation related to the piezoelectric charge coefficient d_{33} for the longitudinal length mode in the cylinder:

$$d_{33} = k_{33} (\varepsilon_{33}^T \cdot s_{33}^E)^{\frac{1}{2}} = \left(\varepsilon_{33}^T \cdot s_{33}^D \cdot \frac{k_{33}^2}{1 - k_{33}^2} \right)^{\frac{1}{2}} =$$

$$= \left(C^T \cdot \frac{t}{\frac{\pi \cdot d^2}{4}} \cdot \frac{1}{4 \cdot \rho \cdot f_a^2 \cdot t^2} \cdot \frac{\frac{\pi}{2} \cdot \frac{f_r}{f_a} \cdot \tan\left(\frac{\pi}{2} \cdot \frac{f_a - f_r}{f_a}\right)}{1 - \frac{\pi}{2} \cdot \frac{f_r}{f_a} \cdot \tan\left(\frac{\pi}{2} \cdot \frac{f_a - f_r}{f_a}\right)} \right)^{\frac{1}{2}} \quad (14)$$

The equation for the piezoelectric voltage coefficient g_{33} for the longitudinal length mode in the cylinder:

$$g_{33} = \frac{d_{33}}{\varepsilon_{33}^T} = \frac{d_{33}}{C^T \cdot \frac{t}{\frac{\pi \cdot d^2}{4}}} \quad (15)$$

The equation for the piezoelectric charge coefficient d_{15} for the thickness shear mode in the thin plate:

$$d_{15} = k_{15} (\varepsilon_{11}^T \cdot s_{55}^E)^{\frac{1}{2}} = \left(\varepsilon_{11}^T \cdot s_{55}^D \cdot \frac{k_{15}^2}{1 - k_{15}^2} \right)^{\frac{1}{2}} =$$

$$= \left(C^T \cdot \frac{t}{w \cdot l} \cdot \frac{1}{4 \cdot \rho \cdot t^2 \cdot f_a^2} \cdot \frac{\frac{\pi}{2} \cdot \frac{f_r}{f_a} \cdot \tan\left(\frac{\pi}{2} \cdot \frac{f_a - f_r}{f_a}\right)}{1 - \frac{\pi}{2} \cdot \frac{f_r}{f_a} \cdot \tan\left(\frac{\pi}{2} \cdot \frac{f_a - f_r}{f_a}\right)} \right)^{\frac{1}{2}} \quad (16)$$

The equation for the piezoelectric voltage coefficient g_{15} for the thickness shear mode in the thin plate:

$$g_{15} = \frac{d_{15}}{\varepsilon_{11}^T} = \frac{d_{15}}{C^T \cdot \frac{t}{w \cdot l}} \quad (17)$$

The resulting courses of impedance characteristics for the transverse, radial, and longitudinal length modes are shown in Figure 3. The resulting courses of impedance

characteristics for the thickness extension and thickness shear modes are shown in Figure 4. These values can be used in the defined relations for the individual coefficients.

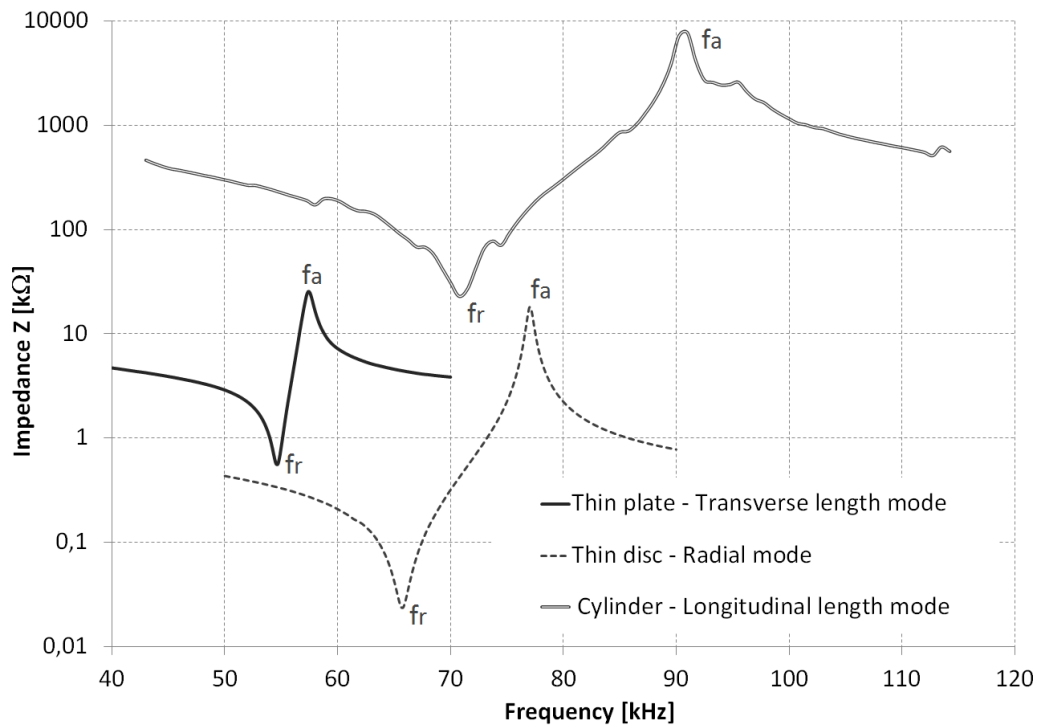


Figure 3. Resonance spectra of the transverse, radial, and longitudinal length modes.

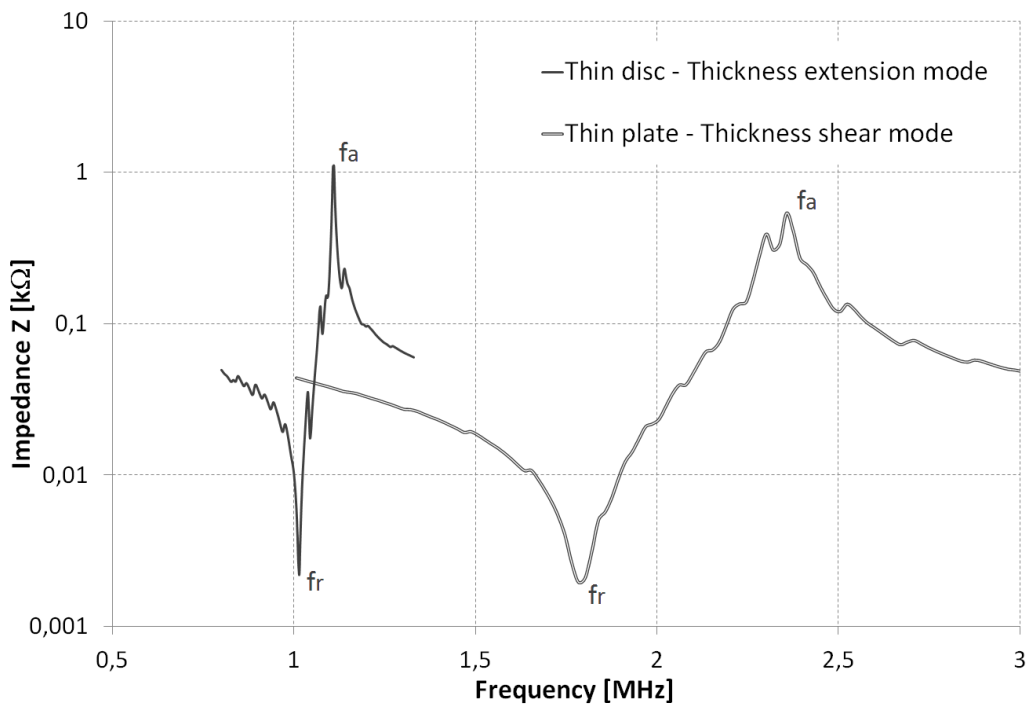


Figure 4. Resonance spectra of the thickness extension and thickness shear modes.

Laser interferometer measuring method

The piezoelectric charge coefficient was measured merely once with this method: on the cylinder and the thin plate of the NCE51 material only. Thus, the measurement was carried out on the same sample as used for the measuring of material constants through the frequency method. The measurement set-up is shown in Figure 5. During the measurements, the measured sample was isolated by a glass plate having the dimensions of $99 \times 99 \times 2$ mm; a reflection label was fixed to the plate for a better quality of the reflected optical signal of the interferometer. The weight of the isolation glass plate was not calculated within the coefficient calculations. The whole measuring set was fixed to an optical table placed on a single base for the maximum elimination of external vibrations. The single base is instrumental towards controlling the vibrations caused by both the evaluation devices and the momentary conditions within the environment, for example movement inside or outside the room. Such interferences can be considered the major error factor influencing the measurements.

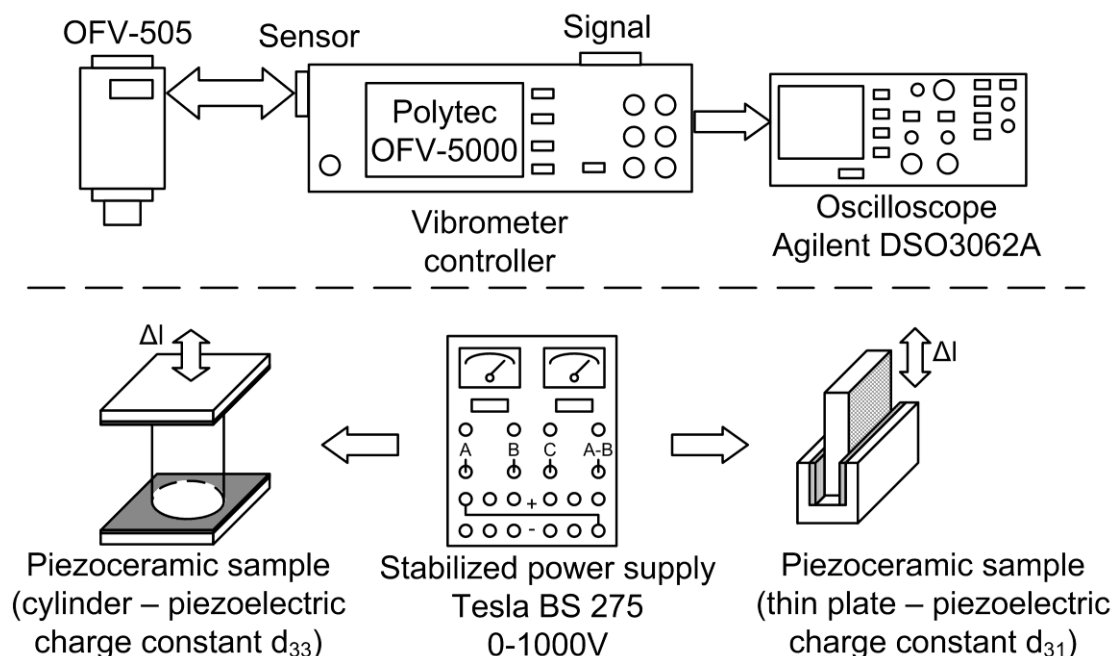


Figure 5. Connection diagram for the measurement of the piezoelectric charge constants d_{33} and d_{31} with a Polytec laser interferometer [14].

The devices used in this experiment can be divided into several groups. Here, the main part is formed by the Polytec OFV-5000 laser interferometer together with an OFV-505 measuring head. A VD-06 velocity decoder with the measuring range of 50 mm·s/V at 350 kHz and a DD-500 displacement decoder with the measuring range of 0.05 μm/V at 350 kHz were used in this measurement. The light source of the OFV-505 vibrometer sensor head is a helium neon laser, and the wavelength is 633 nm. The stand-off distance is measured from the front side of the focusing ring and optimal stand-off distances are at $234 \text{ mm} + (n \cdot l) \text{ mm}$, where $n = 0; 1; 2 \dots$ and $l = 204 \text{ mm} \pm 1 \text{ mm}$. By using short-range OFV-SR front lenses for the sensor head, the vibrometer can be optimally adapted to stand-off distances from 60 mm to 5m.

Further, an Agilent DSO3062A oscilloscope designed to show the resulting signal from the interferometer was used. The level of the measured sample from the IFV-505 measuring head was read on the voltmeter. A Tesla BS 275 stabilized power supply was used to excite the measured sample, allowing us to reach 0 – 1000 V on the output. The used voltmeter, oscilloscope, and stabilized power supply are not the main parts of the connection and can be therefore replaced by other devices with similar characteristics.

The measurement is evaluated through an output voltage signal shown on the oscilloscope. We can read the output voltage ΔU from the process of gained dependence; the output voltage corresponds to the deviation caused by the variation from the voltage applied to the measured sample. The process of the ΔU voltage measurement is shown in Figure 6. The interferometer sensitivity was set to 50 nm/V. The measurement started with the connection of voltage to the sample. The voltage was later disconnected; this is shown as the first drop in the represented process. After the process stabilization, the voltage was connected again, which caused the other

major change. Both the major changes, namely the change initiated by short-circuit of the electrodes and the one achieved by the voltage reconnection, should be equal. With increasing voltage, the deviation increases linearly. The resulting value of the piezoelectric charge constant equals to the relation of the dimension change and the connected voltage. Based on the technique presented within this section, we repeatedly measured all the samples available. The individual measured values of dimension changes in the piezoelectric sample (with identical applied voltage) were averaged.

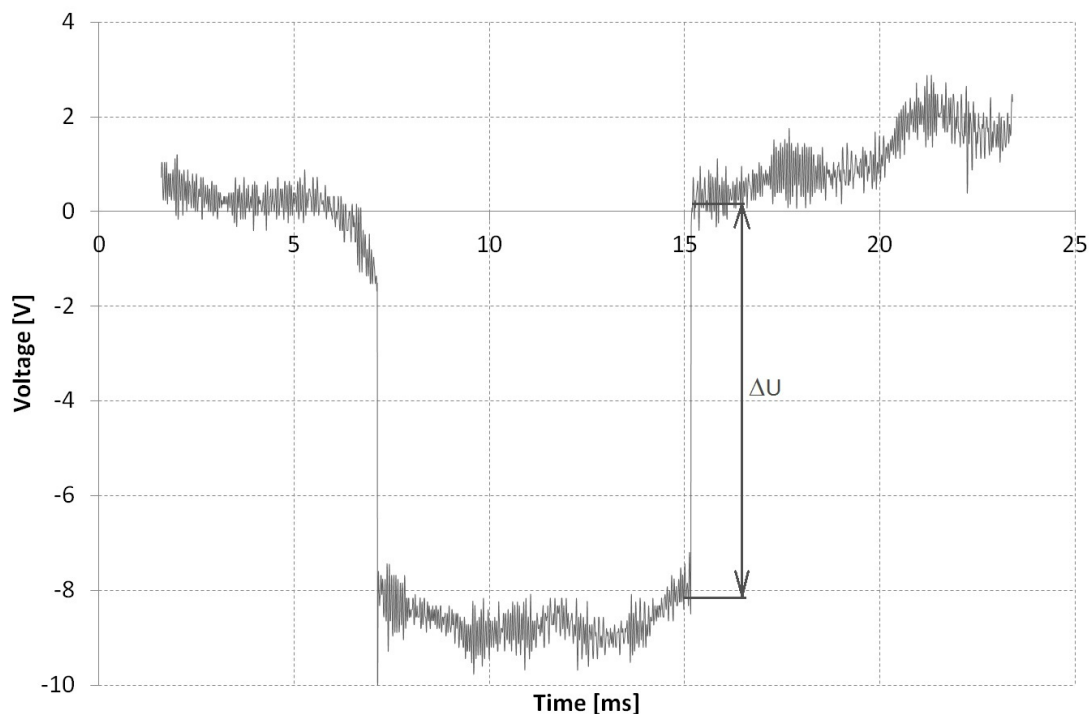


Figure 6. Output signal from the Polytec interferometer shown on the oscilloscope: measured values for the length thickness modes (the cylinder) [14].

The piezoelectric charge coefficients d_{33} and d_{31} are calculated based on the relations shown below, according to which the deviation increases with the gradually increasing voltage, i.e. the height of the cylinder or the thin plate increases or decreases [7].

The piezoelectric charge constant d_{33} for the cylinder is calculated by the following equation:

$$\Delta l = d_{33}U_{in} \Rightarrow d_{33} = \frac{\Delta l}{U_{in}} = \frac{\text{Measurement range (50 nm} \cdot \text{V}^{-1}) \cdot \Delta U(\text{V})}{\text{Supply voltage (1000 V)}}, \quad (18)$$

and the piezoelectric charge constant d_{31} for the thin plate is calculated as follows:

$$\Delta l = d_{31}U_{in} \Rightarrow d_{31} = \frac{\Delta l}{U_{in}} = \frac{\text{Measurement range (50 nm} \cdot \text{V}^{-1}) \cdot \Delta U(\text{V})}{\text{Supply voltage (1000 V)}}, \quad (19)$$

where U_{in} (V) is the power supply voltage connected to the sample, ΔU (V) is the output voltage on the interferometer, Δl (m) is the change of length when the power supply is connected and equals to the piezoelectric charge constants d_{33} or d_{31} (m/V).

Quasi-static method

The quasi-static method is based on the direct piezoelectric effect. The piezoelectric sample is evenly loaded with force F. The charge is generated when the amount of force on the electrodes of the piezoelectric sample changes. The measurement setup is shown in Figure 7. The change of the force is attained by weight placed on one side of the lever. Upon instant unloading of the weight and after amplifying the charge in an AURA (type C4.2) or a Brüel & Kjær (type 2647A) charge amplifier, a voltage pulse equivalent to the applied force is measured on the Agilent DSO3062A oscilloscope. The value of the piezoelectric charge constant is then calculated from the force F and voltage U and is consistent with the change of the force F [8], [22], [23].

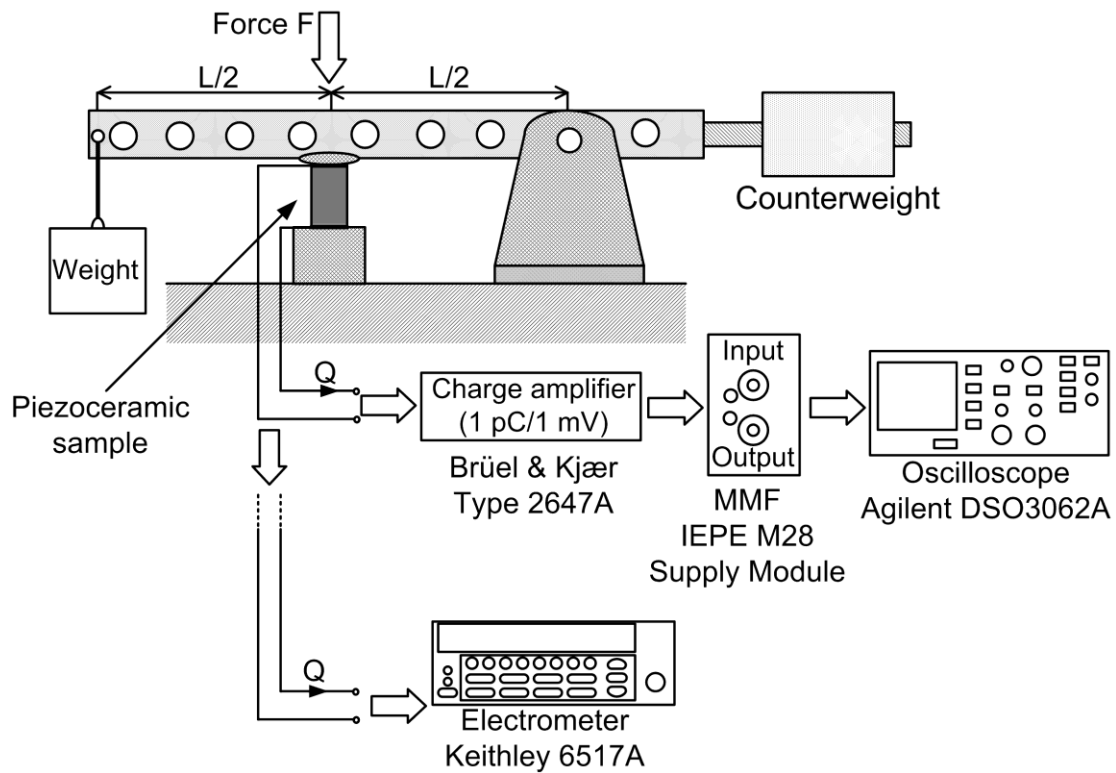


Figure 7. Connection scheme for the measurement of the piezoelectric charge constants d_{33} and d_{31} with the quasi-static and the direct charge measuring methods.

The entire setup comprising a charge amplifier, a supply module, and an oscilloscope can be replaced with an electrometer; for this purpose, a Keithley 6517A is the suitable solution. The connection shown in Fig. 7 remains the same as in the previously described case. Charge Q , generated during the step change of the force F acting on the piezoelectric sample, is measured directly by the Keithley electrometer. The resulting value of the charge Q is directly proportional to the acting force F [8].

It is advantageous to carry out the measurement using the method involving a charge-sensitive amplifier described in [24]; this method of measuring voltage at the capacity eliminates the effect of the measured value of the charge at the capacity of sample C_i , where the measured charge only affects the value of the piezoelectric ceramic charge constant d_{ij} . The measured PZT ceramic sample with the force F acting on it can be imagined as an electric circuit consisting of parallel connection of the current supply i ,

capacity C_i , and resistance R_i , as represented in Figure 8 (the part separated by the broken line).

If the source current i equals to

$$i = d_{ij} \frac{dF}{dt}, \quad (20)$$

the generated charge Q according to the equivalent circuit in Figure 8 is equal to:

$$Q = \int_0^i i \cdot dt = d_{ij} \int_0^F dF = d_{ij} \cdot F. \quad (21)$$

In (7), the influence of the leakage resistance R_i is omitted. The related time constant τ calculated from the magnitude of the resistance R_i and capacity C_i will be smaller than the time t needed to change the force acting on the sample; considering the real preparation, this is the time of lifting the loading lever from the sample. Therefore, $t \ll \tau$ must apply; in this case, the effect of the resistance R_i on the measurement accuracy is negligible.

Figure 8 shows the connection diagram of the charge-sensitive amplifier with a connected equivalent circuit which represents the measured sample of piezoelectric ceramics. The equivalent circuit is separated from the charge amplifier by the broken line [22], [24].

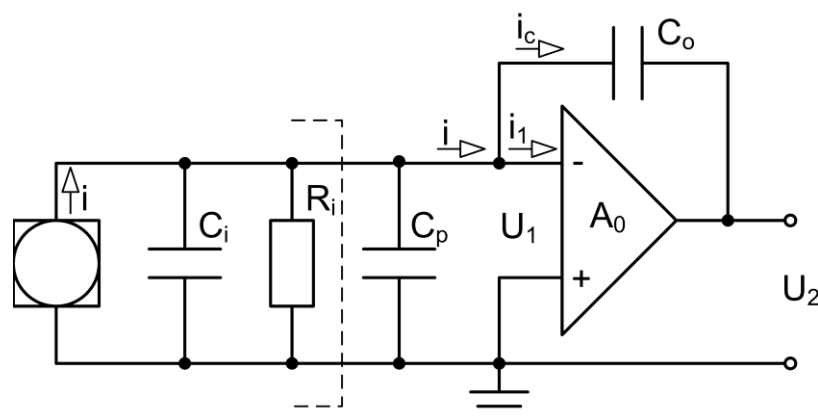


Figure 8. Connection diagram of the charge amplifier with the connected piezoelectric material [22], [24].

The amplification of the charge amplifier A_0 nears infinity. Consequently, the input voltage U_1 almost amounts to zero, and the current i equals to zero for any voltage U_2

on the amplifier output. The current supplied during the loading of the piezoelectric sample then charges the capacities C_i and C_p , where C_p is the parallel capacity of the amplifier power supply conductors and input circuits. The current, at the same time, flows to the capacity C_0 connected in the feedback amplifier. In this case, $i = i_c$ and the voltage U_2 equals to:

$$U_2 = \frac{Q}{C_0} = Q \cdot k_q \quad (22)$$

According to [22], an operational amplifier with amplification greater than 10^4 is used in the connection, whereby the influence of the capacities $C_i + C_p$ on the measurement accuracy is determined by the same relation and is smaller than one ten-thousandth of the relation $C_0/(C_i + C_p)$. The influence of the capacities $C_i + C_p$ on the accuracy of the measurement of the piezoelectric charge constant d_{ij} thus can be omitted.

The piezoelectric charge constant d_{33} for the cylinder is calculated by the following equation:

$$d_{33} = \frac{U_2}{F_3 \cdot k_q}, \quad (23)$$

and the piezoelectric charge constant d_{31} for the thin plate is calculated by the equation

$$d_{31} = \frac{U_2}{F_1 \cdot k_q}, \quad (24)$$

where d_{33} and d_{31} are the piezoelectric charge constants (C/N), U_2 is the output voltage on the oscilloscope (V), F is the stress on an element in the direction x (N), k_q is the charge sensitivity of the piezoelectric sample (pC/mV).

Output voltage from the charge amplifier (2647A) measurement on the Agilent DSO3062A oscilloscope is shown in Figure 9 below. The zero value overshoot in the diagram is induced by the auto-compensation circuit suppressing the unidirectional

drift of the BK2647 amplifier having a time constant of approximately 0.7 Hz. The auto-compensation circuit of the amplifier does not affect the measurement accuracy.

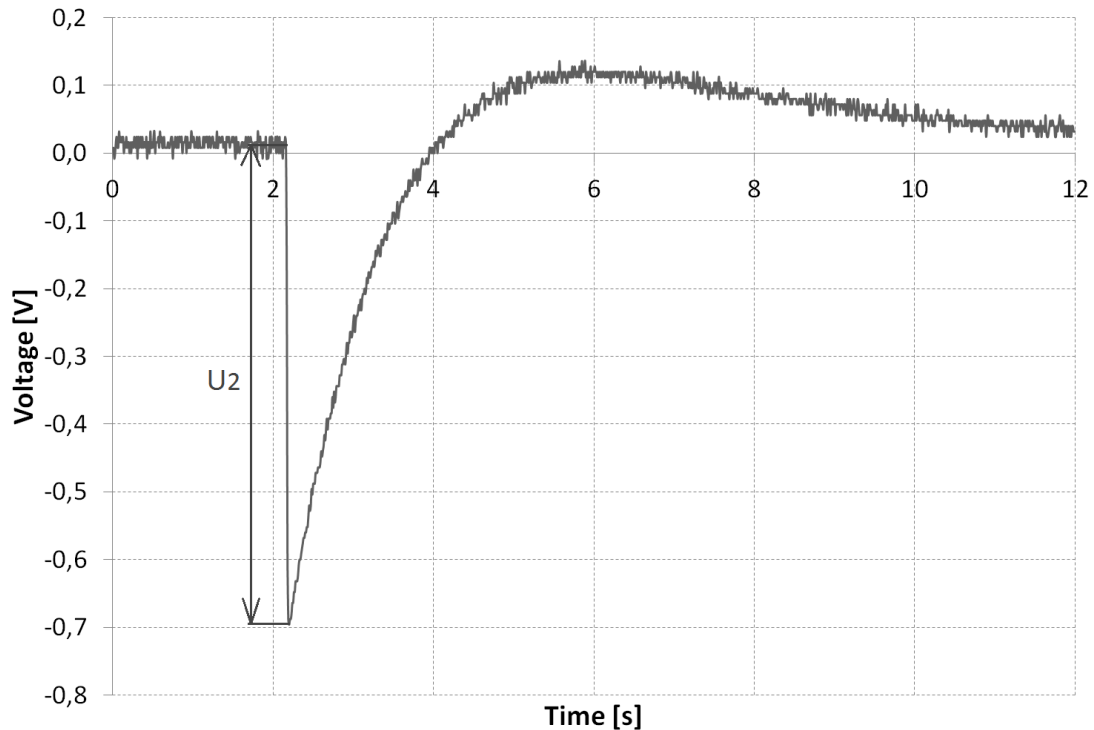


Figure 9. Output signal from the Brüel & Kjær (2647A) charge amplifier shown on the oscilloscope: measured values for the length thickness modes (the cylinder) [14].

Measurement of spurious effects

We measured the influence caused by the clamping of the sample in the measurement jig applied for the frequency method; then, in general terms, attention was paid also to thermal dependence in the coefficients of the piezoelectric samples. In order to verify the influence exerted by the clamping of the sample in the measuring tweezers upon the accuracy of resonant frequency determination within the frequency method, we conducted an experiment using the thin disc for the radial mode and the thin plate for the transverse length mode PZT ceramics (see Figure 1). In successive steps, the measuring tweezers were placed at various points of the sample, and the impact on the resonant and antiresonant frequencies was monitored. The relations between the measured frequencies and the contact point positions are shown in Figures 10 and 11.

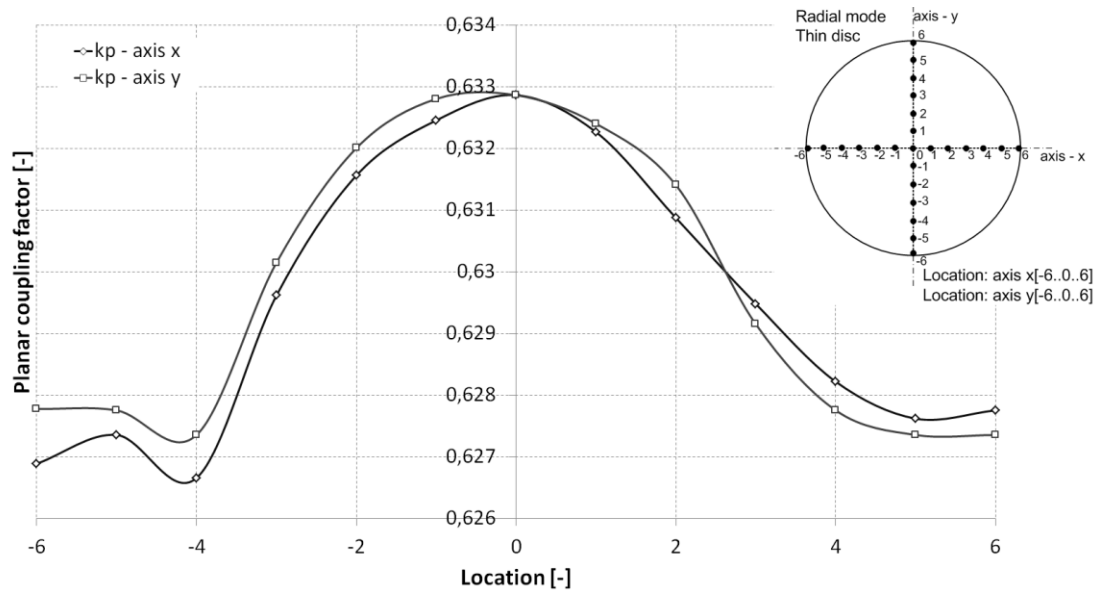


Figure 10. Dependence of the coefficient k_p on the position of the measuring tips in axes x and y: the thin disc.

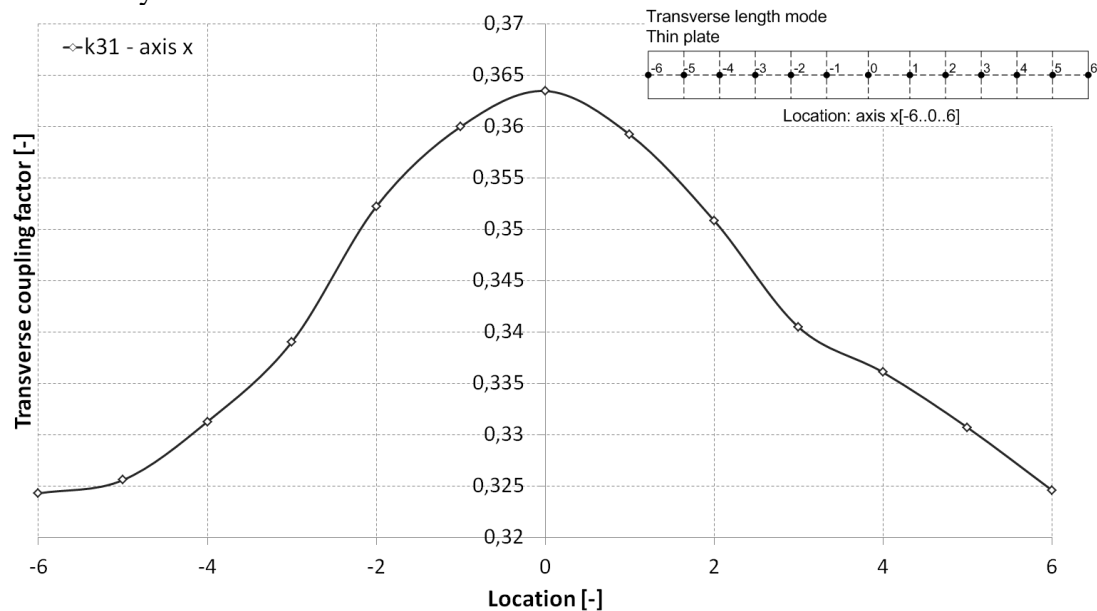


Figure 11. Dependence of the coefficient k_{31} on the position of the measuring tips in axes x and y: the thin plate.

In the thin disc, the influence of the measuring tip position on an electrode of the measured sample did not exceed 1 % of the measured value. In the thin plate, however, the actual tip location is a markedly more significant factor; the difference between the influence detected on the boundaries and that observed in the centre may reach as high as 10% of the measured value. The determined dependence exhibits

very good repeatability and probably could be applied also within the diagnostics of quality and homogeneity of the individual samples.

As the most important spurious effect consists in the temperature, we also measured thermal dependence of the piezoelectric coefficients. The experiment was accompanied by the verification of applicability of the frequency method for thermal dependence measurement within a wide range of working temperatures; within this range, the upper temperature limit approaches the Curie temperature. As a rule, these dependences are not indicated by manufacturers, and available analyses do not include any detailed discussion of the PZT ceramics behaviour at high temperatures [25], [26], [27]. The realized configuration of the experiment is presented in Figure 12.

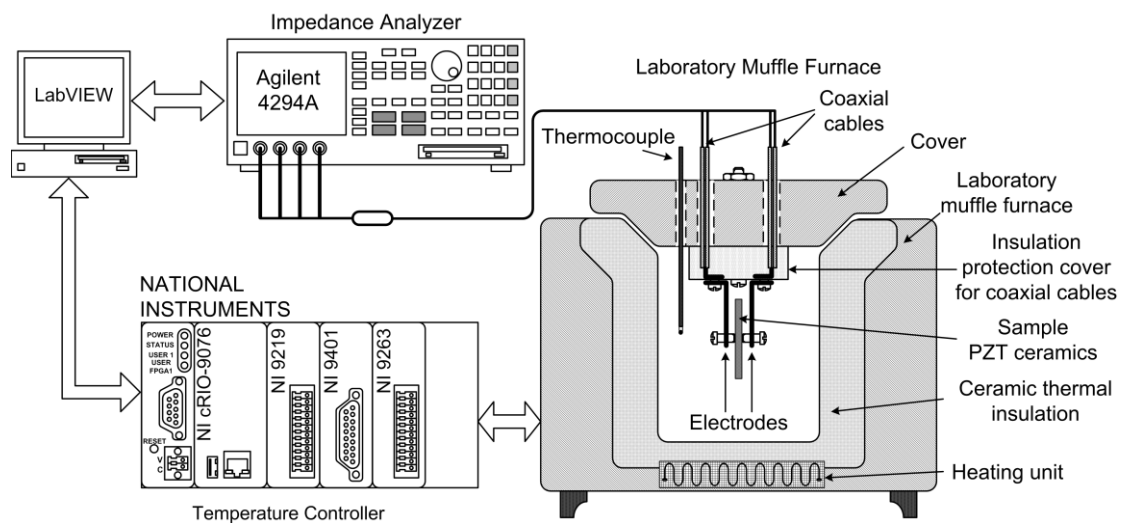


Figure 12. Connection diagram for the measurement of thermal dependences in PZT ceramics.

The clamping of the sample was enabled by a pair of special measuring pincers allowing measurement at high temperatures: the high-temperature conditions exclude the possibility of using the standard measuring tweezers supplied with the analyzer. An example of temperature dependence of the piezoelectric charge coefficient for the

transverse length mode, the thickness shear mode, and the longitudinal length mode is shown in Figure 13.

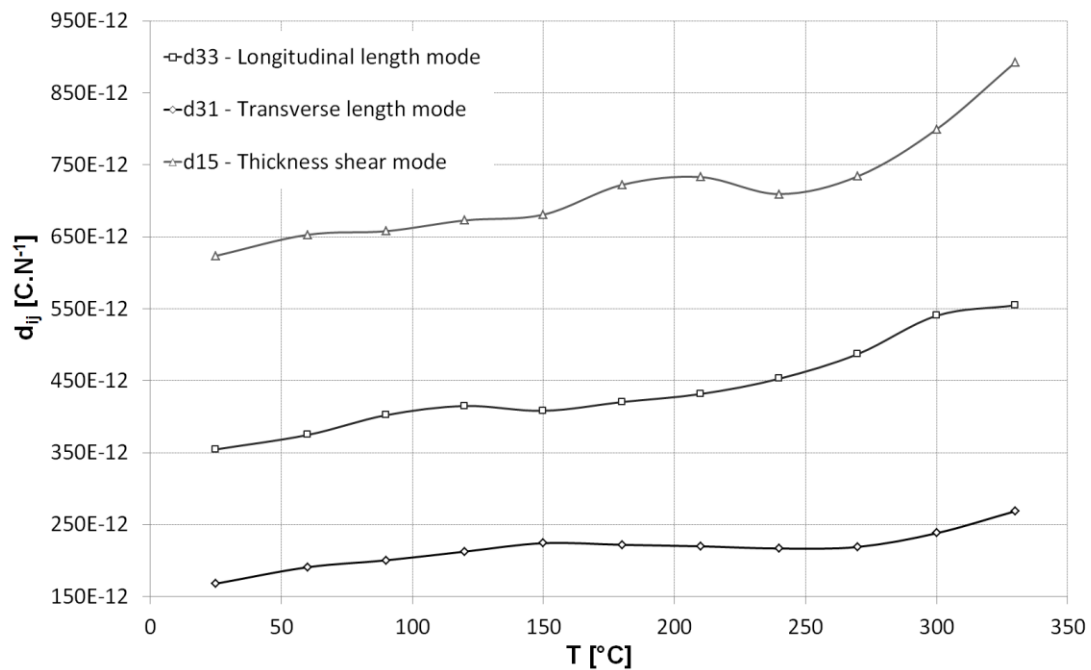


Figure 13. Temperature dependence of the piezoelectric charge constant in the NCE51 PZT ceramics.

Results

To compare all the tested methods for the piezoelectric coefficient measurement, the piezoelectric ceramic charge constant d_{33} was chosen for the thickness longitudinal vibrations and the d_{31} constant was selected for the laterally longitudinal vibrations. To compare the measured and calculated values of the piezoelectric charge constant d_{ij} with those specified by the manufacturer, the PCM 51 (now ref. code NCE 51) piezoelectric ceramics were chosen for the measurement.

The calculated values and the values measured by all the three methods are compared in the table below, where a difference can be seen as compared to the values specified by the producer of the ceramics.

TABLE I. COMPARISON OF MEASUREMENT METHODS

Piezoelectric charge constants d_{33} and d_{31}		
<i>Measurement method</i>	d_{33} [$\times 10^{-12}$ C/N]	$-d_{31}$ [$\times 10^{-12}$ C/N]
Values determined by laser interferometry	448 ± 38	223 ± 31
Values determined by the resonance method	407 ± 25	198 ± 12
Values determined by the quasi-static method	442 ± 31	220 ± 17
Values determined by the charge measurement	404 ± 26	206 ± 8
Value determined by the manufacturer	425 ± 22	195 ± 10

a. The values resulting from the measurements with an interferometer and their comparison with those obtained via the resonance measurement methods and the quasi-static method for the PCM 51 (NCE 51) material (the cylinder where $\varnothing d = 3,5$ mm and $h = 20$ mm and the thin plate where $w = 25$ mm, $l = 4$ mm and $t = 2$ mm)

The elastic s_{ij} , dielectric ε_{ij} , and piezoelectric d_{ij} coefficients obtained by the frequency measurement method appear to be sufficiently accurate in comparison with the NCE 51 catalogue datasheet. This measurement method is useful for applications where a quick measurement of a complete set of material coefficients of the PZT ceramics is needed [21].

TABLE II. PIEZOCERAMICS NCE51 SPECIFICATIONS

Material Coefficients	Symbol & Unit	PZT ceramics NCE 51	
		<i>Datasheet</i>	<i>Calculation</i>
Transverse coupling factor	k_{31} [-]	0,37	0,38
Longitudinal coupling factor	k_{33} [-]	0,72	0,71
Shear coupling factor	k_{15} [-]	-	0,69
Planar coupling factor	k_p [-]	0,65	0,64
Thickness coupling factor	k_t [-]	0,51	0,47
Piezoelectric charge constants	$-d_{31}$ [$\times 10^{-12}$ C/N]	195	198
	d_{33} [$\times 10^{-12}$ C/N]	425	407
	d_{15} [$\times 10^{-12}$ C/N]	-	612
Piezoelectric voltage constants	$-g_{31}$ [$\times 10^{-3}$ Vm/N]	13	12,9
	g_{33} [$\times 10^{-3}$ Vm/N]	27	25,5
	g_{15} [$\times 10^{-3}$ Vm/N]	-	36,9

Material Coefficients	Symbol & Unit	PZT ceramics NCE 51	
		Datasheet	Calculation
Elastic constants (Short circuit)	$s_{11}^E [\times 10^{-12} \text{ m}^2/\text{N}]$	16	15,7
	$s_{33}^E [\times 10^{-12} \text{ m}^2/\text{N}]$	19	19,4
	$-s_{12}^E [\times 10^{-12} \text{ m}^2/\text{N}]$	-	6,5
	$s_{13}^E [\times 10^{-12} \text{ m}^2/\text{N}]$	-	7,1
	$s_{55}^E [\times 10^{-12} \text{ m}^2/\text{N}]$	-	47,3
	$s_{66}^E [\times 10^{-12} \text{ m}^2/\text{N}]$	-	44,2
Permittivity	$\epsilon_{11}^T [\times 10^{-8}]$	-	1,5
	$\epsilon_{33}^T [\times 10^{-8}]$	-	1,7

a. The resulting values of material coefficients of the PZT ceramics of the datasheet and their comparison to the values obtained via the resonance measurement methods for the NCE51 PZT ceramics [21]. (Tolerances $\pm 5\%$, 25°C)

Establishment of accuracy in the individual methods

The complete matrix of material coefficients for the piezoelectric ceramics is measured by means of the frequency measurement method. The overall accuracy of this method depends on the level of accuracy achieved in determining the resonance frequencies via the applied impedance analyzer. We used an Agilent 4294A impedance analyzer with an accuracy precision of ± 40 ppm. The decisive factor impacting the measurement accuracy consists in the manner of fixation of the sample. High repeatability of the measurement is ensured by the use of measuring pincers made by Agilent (type 16334A). With these pincers, the measured samples are fixed always at the same point and compressed with an identical force throughout the whole period of the measurement. In order to ensure repeatability of the measurement, the measuring tips must be placed in the centre of the measured sample. The establishment of measurement accuracy with respect to the optical method depends on the accuracy of deviation measurement, namely on the laser interferometer used and the decoder chosen. A DD-500 displacement decoder within the range of $50\text{nm}/\text{V}$ was used to complement a Polytec OFV – 5000 laser interferometer equipped with an OFV-505 measuring head. The main problem can be identified in the presence of noise in the measurement channel of the interferometer; importantly, this factor

causes reduction of the measurement accuracy. Within the optical method, repeatability largely depends on the structure of the jig enabling fixation of the measured sample. This measurement jig must be efficiently shielded from ambient vibrations; in our case, it was placed on an optical table with active damping capability.

Finally, attention ought to be paid to the quasi-static method, which can be divided into two parts or variants. The first part embodies the measurement of voltage corresponding to the charge generated on the PZT ceramics via the step change of force realized through a charge amplifier. An Agilent DSO 3062A oscilloscope was used to measure the voltage with a $\pm 3\%$ defined accuracy for the vertical axis. The second variant consists in direct measurement of the generated charge using a Keithley 6517A electrometer with an accuracy of $\pm (0.4\% \text{ rdg} + 5\text{counts})$. An important parameter in relation to this method is again the use of an appropriate mechanical structure for the laboratory jig; if this partial precondition is duly observed, equal loading and sufficiently fast unloading of the sample will be ensured. Providing that the conditions described herein are satisfied, adequate repeatability of the measurement can be secured.

TABLE III. COMPARISON OF ACCURACY IN THE APPLIED MEASUREMENT METHODS

Measurement method	Accuracy of measurement	Repeatability of measurement
Laser interferometer measuring method	$\pm 2\%$	Very good
Frequency measurement method	$\pm 2\%$	Best
Quasi-static method:	$\pm 4\%$	Very good
Quasi-static method: charge measurement	$\pm 3\%$	Best

Conclusion

This experiment was conducted based on the need of high-accuracy measurement of the piezoelectric material constants, and its aim was to determine the best method for the realization of such measurement. The resulting values acquired through the three tested methods lead to the following conclusions: The use of the frequency method is advantageous when we need to establish individual coefficients or the complete matrix of the piezoelectric material coefficients. The measurements carried out by means of laser interferometry and the quasi-static methods can be applied when the piezoelectric charge or voltage constants ought to be determined in a short time. The advantage of the latter methods as compared to the frequency method consists in the possibility of using also piezoelectric ceramic samples that do not comply with the requirement for a minimum aspect ratio stipulated by both the European and the world standards. The disadvantages of laser interferometry include mainly the intensive demands placed on the preparation of the measuring equipment and the higher cost of the measuring devices. The method is largely applied in laboratory conditions. The quasi-static method is used predominantly in practice, namely in cases where d_{33} -meters are involved. Both the methods tend to be used for checking the results obtained via the frequency method. As seen in Table I, not a single value of the piezoelectric charge coefficient exhibits a significant difference from the catalogue value of the PCM 51 piezoceramics (now ref. NCE 51). With respect to the measurement accuracy, all the three methods can be regarded as equivalent.

The most advantageous solution for the measurement of thermal dependences consists in the frequency method, which enables us to apply commonly available laboratory equipment. The temperature exerts significant influence on the material coefficients of PZT ceramics; thus, the group of measurement results ought to include working temperature as a relevant aspect.

The determination of the established individual coefficients as well as the complete matrix of the piezoelectric material coefficients measured on samples manufactured from a larger PZT ceramics disc can be used in practice. The measurement results can also be applied within any comparison of the measurement methods as regards achievable accuracy at a later time.

Nomenclature

A	[m ²]	Area
C^T	[F]	Free capacitance
d	[m]	Diameter of a resonator
d_{ij}	[C/N]	Piezoelectric charge constant
E	[V/m]	Polarization vector
F_x	[N]	Stress on an element in direction x
f_r	[Hz]	Resonance frequency
f_a	[Hz]	Antiresonance frequency
g_{ij}	[Vm/N]	Piezoelectric voltage constant
i	[A]	Current
k_{31}	[-]	Transverse coupling factor
k_{33}	[-]	Longitudinal coupling factor
k_{15}	[-]	Shear coupling factor
k_p	[-]	Planar coupling factor
k_t	[-]	Thickness coupling factor
k_q	[pC/N]	Charge sensitivity of the piezoelectric crystal
l	[m]	Length of a resonator
Q	[C]	Charge generated on electrodes
r	[m]	Radius of a resonator
s_{ij}	[m ² /N]	Elastic compliance constant
t	[m]	Thickness of a resonator
w	[m]	Width of a resonator
ε_{ij}	[-]	Absolute permittivity
ε_0	[-]	Permittivity of free space
τ	[s]	Time constant
ρ	[kg/m ³]	Specific density

Acknowledgements

This work is supported by the European Regional Development Fund, project No. CZ.1.05/2.1.00/01.0014 and was realised in CEITEC – the Central European Institute of Technology with research infrastructure supported by the project CZ.1.05/1.1.00/02.0068 financed from the European Regional Development Fund.

References

- [1] International Standard, IRE Standards on Piezoelectric Crystals, “Measurements of Piezoelectric Ceramics,” 1961, vol.41, no.9, pp.1161-1169, ISSN 0096-8390.
- [2] B. Jaffe, W.R. Cook Jr. and H. Jaffe, “Piezoelectric Ceramics,” Academic Press - London and New York, 1971, p. 315, ISBN 0-12-379550-8.
- [3] W.P. Mason, H. Jaffe, “Methods for Measuring Piezoelectric, Elastic, and Dielectric Coefficients of Crystals and Ceramics,” Proceedings of the IRE, 1954, vol.42, no. 6 pp.1731-1737, ISSN 0096-8390.
- [4] European standard, EN 50324-2: 2002, “Piezoelectric properties of ceramic materials and components, Part 2: Methods of measurement – Low power,” CENELEC European Committee for Electrotechnical Standardization, 2002
- [5] International Standard, CEI/IEC 60483: 1976, “Guide to dynamic measurements of piezoelectric ceramics with high electromechanical coupling,” IEE Geneva, Switzerland, 1997, p. 40, ICS 31.140

- [6] L. Burianová, M. Šulc and M. Prokopová, “Determination of the piezoelectric coefficients d_{ij} of PZT ceramics and composites by laser interferometry,” Elsevier Science Ltd., Journal of the European Ceramics Society 21, 2001, pp. 1387-1390, PII:S0955-2219(01)00024-3.
- [7] J.W. Waanders, “Piezoelectric Ceramics: Properties and Applications,” Philips Components, Eindhoven – The Netherlands, First edition 4-91, ISBN 9398 651-80011.
- [8] Keithley Instruments Inc., “Low Level Measurements Handbook, Precision DC Current, Voltage and Resistance Measurement”, 6th edition, 2004, p.239, No. 1559 80440KSL
- [9] J.Erhart, L. Burianová, “What is really measured on a d_{33} -meter?,” Elsevier Science Ltd., Journal of the European Ceramics Society 21, 2001, page 1413-1415, 2001, PII:S0955-2219(01)00030-9.
- [10] S. Sherrit, T.J. Masys, H.D. Wiederick, B.K. Mukherjee “Determination of the reduced matrix of the piezoelectric, dielectric, and elastic material constants,” IEEE Transactions on Ultrasonics, Ferroelectrics and Frequency Control, 2011, vol. 58, no. 9, pp. 1714 - 1720, DOI: 10.1109/TUFFC.2011.2008.
- [11] J. Fialka, “Determination of the piezoelectric charge constant d_{33} measured by the laser interferometer and frequency method,” In Annals of DAAAM for 2010 & proceedings of the 21st international DAAAM symposium, 2010, vol.20, no. 1, pp. 1253-1254, ISBN: 978-3-901509-73- 5.
- [12] European standard, EN 50324-1: 2002, “Piezoelectric properties of ceramic materials and components, Part 1: Terms and definitions,” CENELEC European Committee for Electrotechnical Standardization, 2002
- [13] IEEE Standard, “IEEE Standard on Piezoelectricity ANSI/IEEE 176-1987,” The Institute of Electrical and Electronic Engineers Inc., New York, 1987.
- [14] J. Fialka, P. Beneš, “Comparison of Methods of Piezoelectric Coefficient Measurement“, in Proc. 2012 IEEE International Instrumentation and Measurement Technology Conference (I2MTC 2012), Austria, 2012, pp. 37-42. ISBN: 978-1-4577-1771- 0.
- [15] L. Pardo, A. García, F.M. De Espinosa, K. Brebøl, “Shear resonance mode decoupling to determine the characteristic matrix of piezoceramics for 3-D modeling”, IEEE Ultrasonics, Ferroelectrics, and Frequency Control, March 2011, Vol.: 58, Issue: 3, pp. 646 - 657, ISSN: 0885-3010.
- [16] D. Xiao-Hong, W. Qing-Ming, K. Uchino, “An accurate method for the determination of complex coefficients of single crystal piezoelectric resonators II: Design of measurement and experiments”, IEEE Ultrasonics, Ferroelectrics, and Frequency Control, Feb. 2004, Vol.:51, Issue: 2, pp. 238 - 248, ISSN: 0885-3010.
- [17] D. Xiao-Hong, W. Qing-Ming, K. Uchino, “Accurate determination of complex materials coefficients of piezoelectric resonators”, IEEE Ultrasonics, Ferroelectrics, and Frequency Control, March 2003, Vol.: 50, Issue: 3, pp. 312 - 320, ISSN: 0885-3010.
- [18] N. Pérez, M.A.B. Andrade, F. Buiocchi, C. Adamowski, “Identification of elastic, dielectric, and piezoelectric constants in piezoceramic disks”, IEEE Ultrasonics, Ferroelectrics, and Frequency Control, Dec. 2010, Vol.: 57, Issue: 12, pp. 2772 - 2783, ISSN: 0885-3010.

- [19] S. Sherrit, T.J. Masys, H.D. Widerick, B.K. Mukherjee, "Determination of the reduced matrix of the piezoelectric, dielectric, and elastic material constants for a piezoelectric material with C_{∞} symmetry", IEEE Ultrasonics, Ferroelectrics, and Frequency Control, Sep. 2011, Vol.: 58, Issue: 9, pp. 1714-1720, ISSN: 0885-3010.
- [20] S. Sherrit, H.D. Widerick, B.K. Mukherjee, "A complete characterization of the piezoelectric, dielectric, and elastic properties of Motorola PZT 3203 HD including losses and dispersion", Proceedings of the SPIE Medical Imaging Conference Newport Beach, CA., Sep 1997, SPIE Vol. 3037, 158, pp. 14, DOI:10.1117/12.271326.
- [21] J. Fialka, P. Beneš, "Measurement of Piezoelectric Ceramic Parameters - A Characterization of the Elastic, Dielectric and Piezoelectric Properties of NCE51 PZT". 13th International Carpathian Control Conference (ICCC 2012) - Slovak Republic, Institute of Control and Informatization of Production Processes, 2012, pp. 147-152. ISBN: 978-1-4577-1866-3.
- [22] APC International, "Piezoelectric Theory and Applications, Piezoelectric Ceramics: Principles and Applications," American Piezo Ceramics Inc., 2002, p. 112, ISBN 0-9718744-0-9
- [23] A. Barzegar, D. Damjanovic, N. Setter, "Study of size (aspect ratio) effect on longitudinal piezoelectric coefficient measured by quasistatic technique", 2002 IEEE Ultrasonics Symposium, Proceedings, Oct. 2002, Vol.: 2, pp. 1185 - 1188, ISSN: 0885-3010, ISBN: 0-7803-7582-3.
- [24] G. Gautshi, "Piezoelectric Sensoric: force, strain, pressure, acceleration and acoustic emission sensors, materials and amplifiers", Springer – Berlin, Germany, 2002, pages 269, ISBN 3-540-42259-5.
- [25] C. Miclea, C. Tanasoiu, L. Amarande, M.Cioangher, L.Trupina, C.T. Miclea, C. David, "Temperature Dependence of the Main Piezoelectric Parameters of a Nb-Li Doped PZT Ceramic". International Semiconductor Conference 2006. Bucharest, National Inst. for Mater. Phys., vol. 2, p. 279-282. ISBN 1-4244-0109-7
- [26] W. Hackenberger, E. Alberta, P. Rehrig, S.Zhang, C. Randall, R. Eitel, T. Shrout, "High Temperature Electrostrictive Ceramics for a Venus Ultrasonic Rock Sampling Tool", 14th IEEE International Symposium on Applications of Ferroelectrics - ISAF-04, 2004. p. 130-133. ISBN 0-7803-8410-5
- [27] F. Li, Z. Xu, X. Wei, Xi Yao, "Determination of temperature dependence of piezoelectric coefficients matrix of lead zirconate titanate ceramics by quasi-static and resonance method", J. Phys. D: Appl. Phys, 2009, Vol. 42, 095417, Issue 9.

Biographies

Jiří Fialka received the B.Sc. and M.Sc. degrees in Cybernetics, Control and Measurement from the Faculty of Electrical Engineering and Communication, Brno University of Technology, the Czech Republic, in 2007 and 2009 respectively. He is currently working toward the Ph.D. degree at the Department of Cybernetics, Control and Measurement, Brno University of Technology, the Czech Republic.

Jiří Fialka holds a research position at the CEITEC – the Central European Institute of Technology, Brno University of Technology. At present, his professional interests include the measurement of piezoelectric material, PZT ceramics, acoustic emission, and primary calibration of acoustic emission sensors.

Petr Beneš (M'00) received the M.Sc. and Ph.D. degrees in electronic engineering from Brno University of Technology, Brno, the Czech Republic, in 1991 and 1997 respectively.

He is currently an Associate Professor with the Department of Cybernetics, Control and Measurement at Brno University of Technology, Brno, the Czech Republic, and a senior researcher with the Center for Research and Utilization of Renewable Energy Sources at Brno University of Technology. His research activities are centered on the design and calibration of sensors, flow measurement, and non-destructive testing.