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# The dynamic method of determination of the piezoelectric hydrostatic coefficients

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#### Abstract

The dynamic method of the determination of the piezoelectric hydrostatic coefficients,  $d_h$ , was improved. We have constructed new equipment for more exact measurement by means of the direct dynamic method. The piston for the high-pressure mechanical excitation of pressure changes was used. The advantage of the proposed method is a low frequency (about 1 Hz) pressure excitation. The lever hydraulic press is able to create the hydrostatic pressure inside the pressure chamber up to 70 MPa. The temperature control is realised by the PID temperature controller with a resistivity heater and by the compressor cooler. © 2003 Published by Elsevier B.V.

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## 1. Introduction

The static method of measurement has been used in our laboratory. This method of determination of the piezoelectric hydrostatic coefficient  $d_h$  was presented [1,2,5,6]. This simple method is based on the formula (3) for evaluation of the static (integral) value of hydrostatic piezoelectric coefficient  $d_{h,int}$ . The electric charge induced across samples by the change of pressure was measured by Keithley 6517 electrometer. The hydrostatic pressure inside the pressure chamber was changed by the hydraulic press in the range from 0 to 70 MPa. The pressure dependence of hydrostatic coefficients was measured in several cycles. A pressure hysteresis was observed.

The dynamic measurement of the piezoelectric hydrostatic coefficient was already presented. For example, the piezoelectric coefficients were measured by pistonphone method. The piston sound source was driven at frequencies ranging from 100 Hz to 1.5 kHz, producing effectively a uniform dynamic pressure on the sample and the standard [3]. The other technique, so called the dynamic ac technique, was used to determine the hydrostatic voltage

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coefficient  $g_h$ . The electromagnetic driver was used as an ac stress generator that applies pressure waves to the sample and a PZT standard. Both were kept under a static pressure in the hydraulic press [4]. A common disadvantage of all these dynamic methods is a need of using standards, usually samples with known exact value of piezoelectric hydrostatic coefficient  $d_h$ . These methods suppose that the piezoelectric hydrostatic coefficient  $d_h$  value does not depend on the pressure.

### 2. Theory

The derivation of theoretic relations is realised for the sample poled along *z*-axes under hydrostatic pressure p ( $T_{ii} = -p$ , all other components of stress tensor are equal to zero). Simultaneously, the applied electric field equals zero ( $E_3 = 0$ ). It means a short-circuit measurement is used. The polarisation  $P_3$  on the sample induced by hydrostatic pressure p is given by

$$P_3 = (d_{31} + d_{32} + d_{33})(-p).$$
(1)

The induced charge Q on the sample is

$$\frac{Q}{A} = d_{\rm h} p, \tag{2}$$

where  $d_h$  is the hydrostatic piezoelectric coefficient and A the area of surface electrode.

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The integral (static) hydrostatic piezoelectric coefficient  $d_{h,int}$  can be calculated from the expression

$$d_{\rm h,int} = \frac{\Delta Q}{\Delta p A}.$$
(3)

The dynamic (differential) method is described by the equation

$$\frac{\mathrm{d}Q}{\mathrm{d}t}\frac{1}{A} = d_{\mathrm{d,diff}}\frac{\mathrm{d}p}{\mathrm{d}t},\tag{4}$$

where  $d_{h,diff}$  is the differential hydrostatic piezoelectric coefficient.

We can suppose a sinusoidal shape of the pressure excitation

$$Q = Q_0 \sin \omega t, \qquad p = p_0 \sin \omega t + p_S, \tag{5}$$

$$I = \frac{\mathrm{d}Q}{\mathrm{d}t} = Q_0 \,\omega \cos \omega t = I_0 \cos \omega t, \tag{6}$$

$$\frac{\mathrm{d}p}{\mathrm{d}t} = p_0 \,\omega \cos \omega t. \tag{7}$$

From Eq. (4) and equations for harmonic Eqs. (5)–(7) we can express coefficient  $d_{\rm h}$  as

$$d_{\rm h} = \frac{I_0}{p_0 \omega A},\tag{8}$$

where  $d_h$  is the differential hydrostatic piezoelectric coefficient ( $d_{h,diff}$ ),  $I_0$  the amplitude of measured current and  $p_0$  an amplitude of a pressure excitation.

Measurement of the hydrostatic voltage coefficient  $g_h$  is the second possibility to measure piezoelectric behaviour under hydrostatic pressure:

$$g_{\rm h} = \frac{d_{\rm h}}{\varepsilon_0 \varepsilon_{33}}.\tag{9}$$

The electrical displacement on the sample is given by

$$D_3 = \varepsilon_0 \varepsilon_{33} E_3 + d_{\rm h}(-p). \tag{10}$$

From definition (9) and Eq. (10) we get

$$g_{\rm h} = \frac{u_3}{pl} - \frac{D_3}{\varepsilon_0 \varepsilon_{33} p}.$$
 (11)

In conditions of the open-circuit, the second term in Eq. (11) can be neglected. We had to use impedance transformer to reach high impedance input. The problem can cause a high voltage output of samples with high piezoelectric voltage coefficient  $g_h$  (tens of volts).

### 3. Temperature control

The temperature of the sample is set by direct contact with thick copper plate heated by resistivity heater. The metal

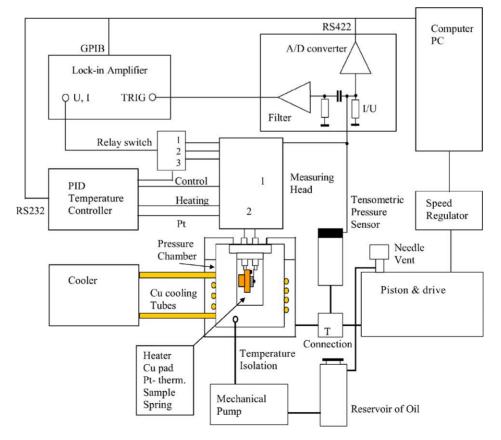


Fig. 1. Block diagram of high-pressure system for dynamic method of measurement.

plate also serves as the ground electrode. The temperature range of stabilisation is approximately -10 to 250 °C with absolute accuracy  $\pm 0.1$  °C and with resolution better than  $\pm 0.01$  °C.

The oil cannot serve as the temperature transmitter because of its low temperature conductivity and high viscosity. Other more important problem follows from a compressibility of the pressure medium that causes adiabatic heating or cooling during changes of the pressure. The temperature is changed in the whole volume of the silicon oil. The undefined flow of the oil can cause the step change in temperature up to 20 °C. This behaviour in principle excludes using silicon oil as temperature mediator and stabilises temperature in the whole volume of the chamber without forced circulation.

#### 4. The design of dynamic hydrostatic method

The simplified block diagram of our equipment for high pressure and temperature measurements is in Fig. 1. The "relay switch" was used for switching between output from tensometric pressure sensor and signals on samples. This setup makes reaching of the maximum accuracy of measurement possible. The "measuring head" enables us to measure the voltage signal (high impedance transformer 1), current signal and switch between two spring sample-holders (relay 2). The pressure signal is subsequently used for external triggering of the selective amplifier. The advantage of this method is the direct evaluation of the value of piezoelectric hydrostatic coefficients  $d_h$  in Eq. (8) and the piezoelectric voltage hydrostatic coefficients  $g_h$  using simplified Eq. (11)

$$g_{\rm h} = \frac{u_3}{pl}.$$

The realisation of the system is shown in Fig. 2. The piston (position 3) for the high-pressure mechanical excita-



Fig. 2. Construction setup: (1) the lid of the pressure chamber with the high-pressure bushings; (2) the temperature isolation; (3) the lever of the pressure pump; (4) the oil reservoir; (5) the piston driver; (6) the engine with a transmission and a cam; (7) the speed regulator; (8) the needle valve; (9) the pressure tensometer; (10) the PID temperature regulator.

tion of pressure changes is used. The advantage of such low frequency excitation method (frequency about 1 Hz) is the constant pressure change in the whole volume of the pressure chamber (positions 1 and 2). The wavelength of sound spread in the medium (silicon oil) is much longer than the dimensions of pressure chamber. The lever hydraulic press (position 9) is able to create the hydrostatic pressure inside the pressure chamber starting at 0 up to 70 MPa. Lock-in amplifier SR830 (Hewlett-Packard) measures the low magnitude current signal on the sample. Selective amplifier separates dc component of a pressure signal, suppresses the disturbing high frequency signals (f > 5 Hz) and detects the amplitude of low frequency pressure excitation.

#### 5. Conclusions

Different materials were studied. Examples are given in Figs. 3–6. The graph of piezoelectric hydrostatic coefficient  $d_h$  versus pressure is given in Fig. 3. In the measurement

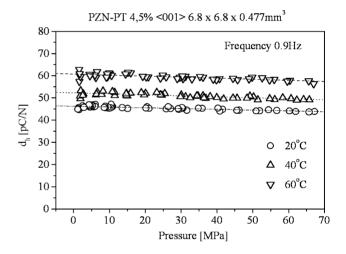


Fig. 3. Variation of hydrostatic piezoelectric coefficient  $d_h$  with static pressure for different temperatures, PZN: 4.5% PT (001) single crystal.

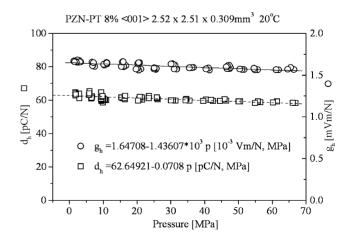


Fig. 4. Variation of hydrostatic piezoelectric  $d_h$  and voltage  $g_h$  coefficient with static pressure for PZN: 8% PT single crystal (001), 20 °C.

Soft PZT type ceramic EPC856 disk diameter 10 mm

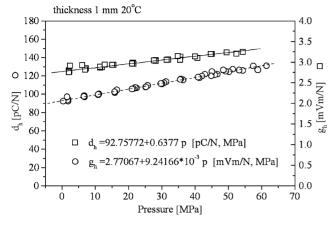


Fig. 5. Variation of hydrostatic piezoelectric  $d_h$  and voltage  $g_h$  coefficient with static pressure for soft PZT type ceramic, 20 °C.

of PZN, 4.5% PT of (001) cut single crystal for different temperatures was obtained.

Variation of hydrostatic piezoelectric  $d_h$  and voltage  $g_h$  coefficient with pressure for PZN: 8% PT single crystal (001) at 20 °C is given in Fig. 4. We can also see in Fig. 5 variation of piezoelectric hydrostatic  $d_h$  and voltage  $g_h$  coefficients with pressure for soft doped PZT ceramic (type EPC 856) at 20 °C. We can observe different behaviour of piezoelectric hydrostatic  $d_h$  and voltage  $g_h$  coefficients as function of pressure. Ceramic materials exhibit increased values of both coefficients instead of small decrease for single crystals. The observed increase of coefficients for ceramic materials can be caused by grain structure of this material.

The graph of variation of hydrostatic piezoelectric voltage  $g_h$  coefficient and pressure changes  $\Delta p$  with pressure for PZN: 8% PT single crystal (011) at 20 °C is given

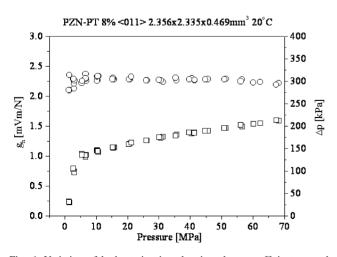


Fig. 6. Variation of hydrostatic piezoelectric voltage coefficient  $g_h$  and pressure changes  $\Delta p$  with static pressure for PZN: 8% PT single crystal (011), 20 °C.

in Fig. 6. We can observe non-linear variation of pressure excitation  $\Delta p$  with pressure. It causes a decrease of accuracy of hydrostatic measurement for low applied static pressure (<5 MPa). The advantage of low frequency excitation method (frequency about 1 Hz) is the constant pressure change in the whole volume of the pressure chamber. The wavelength of sound spread in the medium (silicon oil) is much longer than the dimensions of pressure chamber.

The above-presented dynamic method makes possible the exact measurement of piezoelectric hydrostatic coefficients as well as their variation with pressure and temperature in a wide range.

The method seems to be promising for investigation of intrinsic and extrinsic contribution to piezoelectric behaviour of single crystals and ceramic materials. We can also study phase transition induced by hydrostatic pressure.

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