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A Capacitive Dilatometer for Measuring the Magnetostriction, Piezoelectric Effect, and Linear Thermal-Expansion Coefficient

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Abstract—We describe a capacitive dilatometer for measuring the magnetostriction, piezoelectric effect, and linear thermal-expansion coefficient in the temperature range from 1.85 to 350 K in external magnetic fields of up to 90 kOe under a voltage of 1 kV, which operates on the basis of a Quantum Design PPMS commercial facility for studying the properties of solids.

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The straining of a substance upon variation in environmental conditions reflects numerous processes occurring at the level of a unit cell and interatomic interactions. Therefore, the list of publications on measuring minor strains is still growing [1], although such methods have been known for a long time. The importance of designing new experimental facilities is also caused by new problems that need to be solved using measurement equipment. An example of this is the study of multiferroics, which exhibit bright magnetostriction and piezoelectric properties. The interplay between the magnetic subsystem and electrical properties opens wide opportunities for application of multiferroics [2].

To study such samples, it is important to measure the strain caused by temperature and electric and magnetic fields. In addition, it is of interest to investigate the behavior of the strain of multiferroics in magnetic and electric fields applied simultaneously. Since there has been a lack of works in this area, the development of such measuring setups is of great importance.

This Letter reports on the development of a capacitive dilatometer for operation on the basis of a Quantum-Design Physical-Property Measurement System (QD PPMS) and intended for measuring the magnetostriction, linear thermal-expansion coefficient, and reverse piezoelectric effect.

Dilatometric measurements using the capacitive technique are performed using a measuring capacitor with one movable plate, which changes its position relative to another (fixed) plate under sample straining. Capacitance change ΔC can be interpreted as a variation in interplate spacing Δh.

We developed a dilatometric cell (Fig. 1) adapted to the QD PPMS facility. The measuring-cell design is similar to the constructions proposed in [3, 4]. However, there are some differences, the main one of which is additional conducting contacts for measuring sample straining under the action of an external electric field and a specific galvanic isolation between the movable plate, its suspension spring, and cell casing. The cell design is illustrated in Fig. 1b.

The sample is put on table. The table is connected with measuring-cell casing *2* by an M5 × 0.5 screwed joint. The optimal table height (the gap between the table and movable capacitor plate *3*) is attained by transmitting rotation through a reducing-gear meshing with gear wheel *4*. Nut *5* fixes the gear wheel. After tuning the height of the table, its position is fixed with locking nut *6*. Movable plate *3* is hung on membrane spring *7* fixed with nut *8*. The electric contact between the movable plate and measuring cell casing is prevented by polyimide gaskets *9* placed inside ring *10*. Fixed plate *11* is glued in ring *12* of the fixed plate. The initial gap between the two plates of the measuring capacitor is ensured by gasket *13*. Transition plate *14* fastens electrical connector *15* adapted to the QD PPMS electrical connector.

The main measuring-cell material is M1 copper, which was used in fabrication of most components. Spring *7* is made of BrB2 beryllium bronze. Fixed plate *11* is glued into ring *10* with Stycast 2850 FT glue. Gear wheel *4* is made of plastic.

During the measurements, a sample is pressed between table and movable plate *3* at a certain tension of membrane *7* until the state is achieved at which the

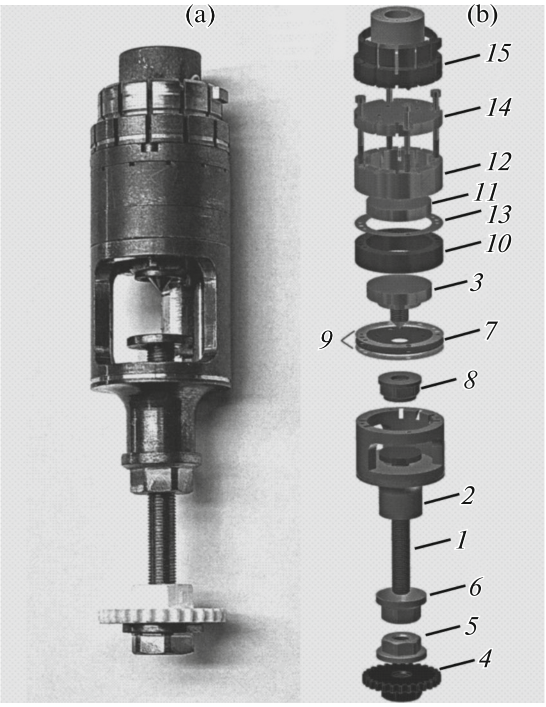

Fig. 1. (a) General view and (b) composition of the measuring dilatometric cell: (*1*) table with a sample, (*2*) measuring-cell casing, (*3*) movable plate, (*4*) gear wheel, (*5*) gear-wheel nut, (*6*) locking nut of the table with the sample, (*7*) membrane, (*8*) movable-plate nut, (*9*) polyimide gasket, (*10*) movable-plate ring, (*11*) fixed plate, (*12*) fixed-plate ring, (*13*) copper gasket, (*14*) transition plate, and (*15*) electrical connector.

capacitance of the measuring capacitor appears to be in the range suitable for measurements.

Since the capacitance of the measuring capacitor can deviate from the parallel-plate capacitor law, it is reasonable to experimentally determine the coupling coefficient between the capacitance of the measuring capacitor and interplate spacing. The results of the calibration measurements of the capacitance as a function of the movable capacitor plate shift are presented in Fig. 2. It can be seen that the dependence of the reciprocal capacitance on the movable plate shift slightly deviates from the linear law in the upper and lower ranges, but obeys this law in the intermediate range. Thus, the working range of the measuring cell is the capacitance-variation range from 13.5 to 32 pF, which corresponds to a sample strain of 110 μm. The measuring-cell sensitivity is 1.85 Å.

In the measurements of the linear thermal-expansion coefficient (LTEC), both the investigated sample and measuring cell are strained, since the cell material is also strained upon temperature variation; therefore, the measured capacitance reflects the sum of the sample and cell strains. To calibrate the LTEC measurements, we, as did the authors of [3, 5], performed temperature measurements of the measuring cell capacitance on pure metal samples (silver 99.99%, aluminum 99.995%, and copper 99.97%), the LTEC values of which are well known.

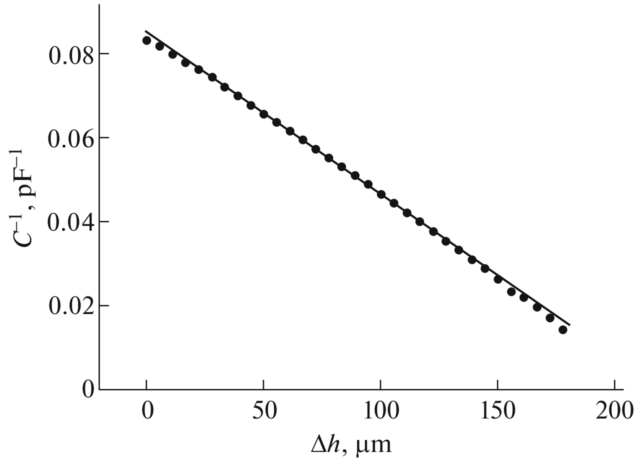

Fig. 2. Dependence of the reciprocal capacitance of the measuring capacitor on the movable-plate shift. Dots show the experimental data, and the straight line shows the linear approximation.

The reverse piezoelectric effect consists in straining a sample by means of an applied electric field. This effect is, as a rule, very weak; therefore, we failed to find studies in which such measurements performed on a capacitive dilatometer were reported. However, we observed this effect using the developed device. The sample used was a holmium alumoborate $HoAl_3(BO_3)_4$ single crystal, which exhibits a giant magnetoelectric effect and is attracting much attention of researchers [6–8].

The electric field was applied to the crystal along the *a* crystallographic direction, and the strain was also measured along this direction at temperature $T = 4.2$ K. To provide uniformity of the electric field, we glued copper-foil plates with a size larger than that of the crystal to the sample (Fig. 3a).

Figure 3a shows the time dependence of the capacitance of the measuring capacitor. Each step in the curve corresponds to the change in an applied electric field by 100 V (the crystal has a thickness of 1 mm along the applied-field direction). Figure 3b shows relative and absolute sample strains calculated from the calibration data (Fig. 2).

It can be seen that the fixed absolute strain was merely ~1.5 nm in an applied electric field of 6 kV/cm, which is indicative of the high sensitivity of the designed dilatometer.

In addition, we measured the reverse piezoelectric effect on a $Bi_{12}GeO_{20}$ single crystal at a temperature of 300 K. Experimental piezoelectric moduli d_{14} for this single crystal were reported in [9]. The authors of [10] investigated the change in constant d_{14} at the manifestation of the photoconductivity effects in the range

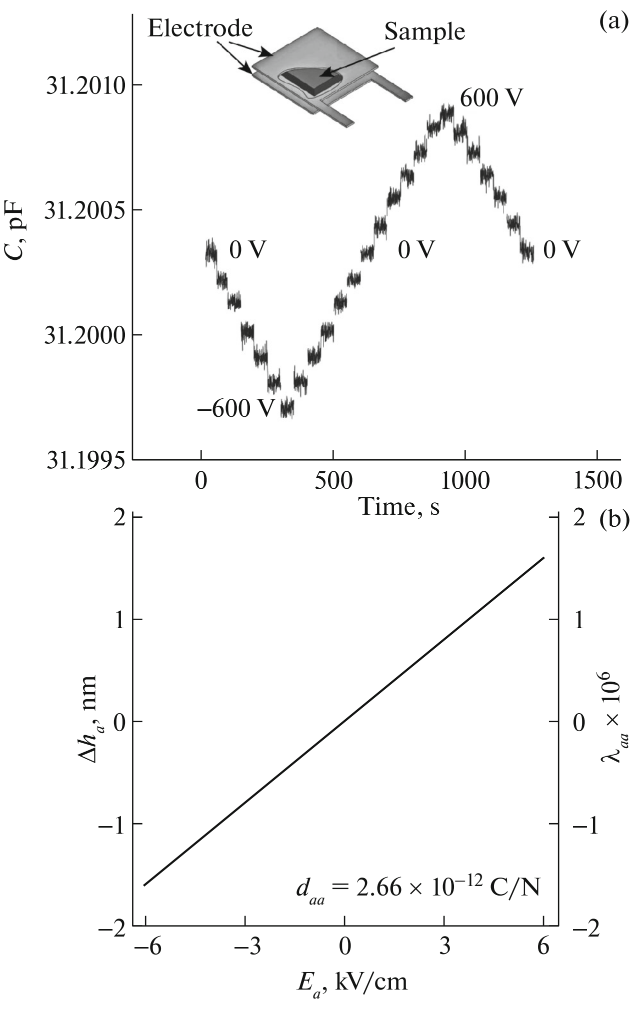

Fig. 3. Experimental data on the piezoelectric effect in $HoAl_3(BO_3)_4$ at a temperature of T = 4.2 K. (a) Time dependence of the capacitance of the measuring capacitor upon stepwise variation in the electric field applied to the sample. (b) Absolute (Δh_a) and relative (λ_{aa}) strains of the single crystal. Inset: schematic of sample preparation.

from 1.5×10^{-11} to 4.2×10^{-11} C/N. The relative strains of the crystal at the longitudinal piezoeffect [11] along the third-order axis for given piezoelectric moduli d_{14} in an electric field of 10 kV/cm lie between $(0.8–2.4) \times 10^{-5}$. The absolute strains of the 1-mm-thick sample are 8–24 nm. The experimental value is 13 nm.

Thus, we have developed and fabricated a capacitive dilatometer for operation on the basis of the QD PPMS facility, which makes it possible to measure the magnetostriction, piezoelectric effect, linear thermal-expansion coefficient, and reverse piezoelectric effect; this last was implemented first on a capacitive dilatometer. Our dilatometer can operate at temperatures from 1.85 to 350 K in magnetic fields of up to 90 kOe under voltages of up to 1 kV on the sample.

We measured for the first time the piezoelectric effect in holmium alumoborate $HoAl_3(BO_3)_4$ at a temperature of 4.2 K. Piezoelectric modulus d_{11} was found to be 2.66×10^{-12} C/N. The obtained data will allow the magnetoelectric effect caused by the interaction of elastic strains (magnetostriction and piezoelectric effect) to be estimated, which can help explain the nature of the magnetoelectric effect in the family of rare-earth paramagnetic borates on the macroscopic scale.

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