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A new measurement method of piezoelectric properties of single ceramic fibres

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Abstract

For single piezoelectric ceramic fibres a method was developed to measure the piezoelectric strain loops. In this work we present a measurement method using a capacitive displacement sensor to characterize single fibres with diameters of 100–500 μm. The electric field is applied in the length direction of the fibre and the strain parallel to the field is measured. Strain-electric-field loops of PZT fibres were measured at high electric field strength and frequencies up to 100 Hz. The sensitivity of the measurement method is high enough to determine the low-voltage piezoelectric coefficient d_{33}. The results were compared with other results measured on single fibres. The advantages and drawbacks of the method are discussed.

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Keywords: Fibres; Piezoelectric properties; PZT

1. Introduction

Piezoelectric fibres have a high potential of applications in the field of ultrasonic and micro-electromechanical systems (MEMS).[1,2,3,4,5] Usually, they are embedded in composites for utilization of their sensoric and/or actuator potential. In the last years the preparation technologies of ceramic fibres with diameters of some hundreds down to 10 μm were enhanced and optimized.

An important question for producers is the characterization of the piezoelectric properties. The standard measurement methods are not applicable for single ceramic fibres. A proven method is the measurement of 1–3 composite with embedded fibres. The effective piezoelectric, dielectric and elastic properties of the composite can be determined by conventional dynamic or quasistatic methods. These effective coefficients depend from the properties of the piezoelectric fibres and the polymer matrix as well as from the volume content of the fibres. The properties of the composite can be calculated by different ways depending on the used basic equations.[6,7,8,9]

Steinhausen et al. used such a couple of effective coefficients to recalculate some of the important fibre properties from the effective properties of the composite and the polymer matrix. In addition to the piezoelectric properties d_{33} and d_{31} and the dielectric coefficient ε_{33} [10] also the elastic compliance s_{33} of the fibre can be determined.[11] In this method the polymer matrix is used as a kind of sample holder. Therefore, the exact knowledge of the properties of the polymer plays a major role. In particular the elastic stiffness of the polymer may be changed radically in different regions of frequency. In addition, the preparation of composites needs a lot of time and effort. Bowen et al. used an analogue method to calculate the piezoelectric coefficient d_{33} of high activity piezoelectric fibres.[12]

The investigation of the ferroelectric and electromechanical behavior of single fibres is complicated due to handling, electroding and contacting of the brittle fibres. Yoshikawa et al. describe a measurement method for the polarization hysteresis of single fibres.[13] More difficult is the determination of the piezoelectric properties. Belloli et al. have presented a quasistatic strain measurement method.[14] The piezoelectric strain is measured with a dynamic mechanical analyzer (DMA). The measurement time of one butterfly loop amounts some minutes. The frequency is typically in the region from 2 to 3 mHz. Additionally, the limited displacement sensitivity of the DMA allows only the characterization of the high voltage behavior (butterfly

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loops). Bowen et al. described a method for characterization of the elastic properties of single fibres. A fibre was glued on a special test coupon to determine the tensile strength of the single fibre.[15]

In the present work we describe a novel method for determination of the low and high voltage properties of single ceramic fibres at higher frequencies up to 100 Hz.

2. Measurement method

The measurement method described in this work is based on a capacitive displacement sensor. The piezoelectric specimen (1 in Fig. 1) is build in a mechanical serial connection with a parallel plate capacitor (2 and 3) and a quartz disc (4). Applying an electric field to the specimen the piezoelectric displacement yields to a change of the distance of the capacitor's plates and consequently to a change of the capacitance. This measurement capacitor is part of a serial resonance circuit (HF-circuit with a resonance frequency of about 90 MHz). The change in capacitance results in a frequency shift of the circuit which is detected by a modulation analyzer. The output voltage of the analyzer proportional to the displacement of the specimen is measured by a LockIn-voltmeter. Due to the frequency selected measurement of the LockIn-technique a sensitivity of displacement in the range of 10^{-10} m is possible. The sensitivity is the higher the smaller is the gap of the measurement capacitor. Since it is an air capacitor its capacitance depends on the temperature and humidity of the atmosphere in the lab. Therefore, the voltage-displacement dependence is calibrated immediately after each measurement using the quartz disc. The piezoelectric quartz is cut in x-direction. Thus, a defined displacement can be induced by applying a constant voltage.

For an accurate measurement of small displacements the compensation method is used. Here, two voltages were applied simultaneously to the specimen and the quartz. The phase shift of the quartz voltage is approximately 180° so that the displacement of the quartz compensates the one of the specimen. Amplitude and phase of the quartz voltage is changed until the displacement of the measurement capacitor is zero. In this case, the piezoelectric coefficient d_{33} of the sample can be determined by

$$d_{33} = \frac{U_{\text{quartz}}}{U_{\text{appl}}} d_{\text{quartz}} \qquad (1)$$

where U_{appl} is the voltage applied to the sample, U_{quartz} is the voltage applied to the quartz and d_{quartz} is the piezoelectric coefficient of the quartz.

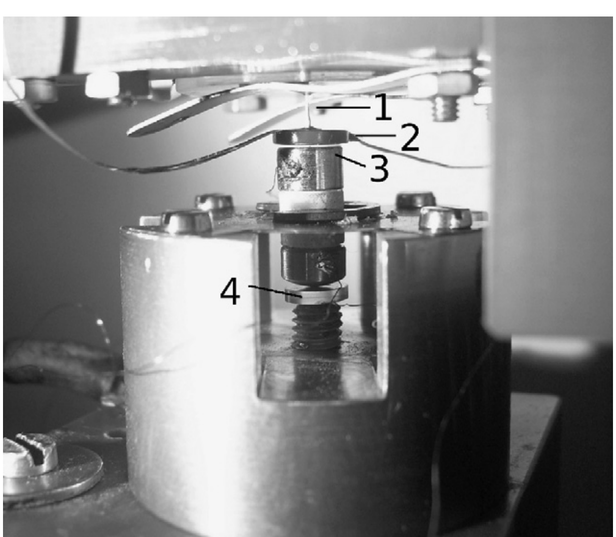

Fig. 1. Experimental setup.

3. Sample preparation

The PZT fibres investigated in this work are developed at Empa Dübendorf. These fibres were produced by mixing a commercial PZT5A type powder (EC65, EDO Corp.) with a thermoplastic binder system, extruding the compound vertically downwards through a die 300 μm in diameter and thermally heat treating the green fibres at 1200 °C in a PbO-enriched atmosphere. The final diameter of the fibres was 250 μm. The production steps are described in more detail in Ref. 16. All fibres were characterized with regard to porosity, grain size, phase composition near the fibre surface, phase composition of the bulk material and ferroelectric properties. The results are presented in Ref. 14.

For the piezoelectric measurements the fibres have to be electroded. A short piece of fibre with a length of approximately 3–4 mm was glued into the sample holder using a conductive glue (conductive silver). The end of the fibre electroded by this way was used as the positive electrode (phase). The other end of the fibre was metallized by dip coating using the same conductive silver. The distance of these ring electrodes was of about 1 mm. The accurate value, i.e. the active fibre length was measured using a microscope (Fig. 2). The free end of the fibre was affixed to the top of a plastic disc and connected there electrically with ground. The disc with a diameter of 5 mm was metallized

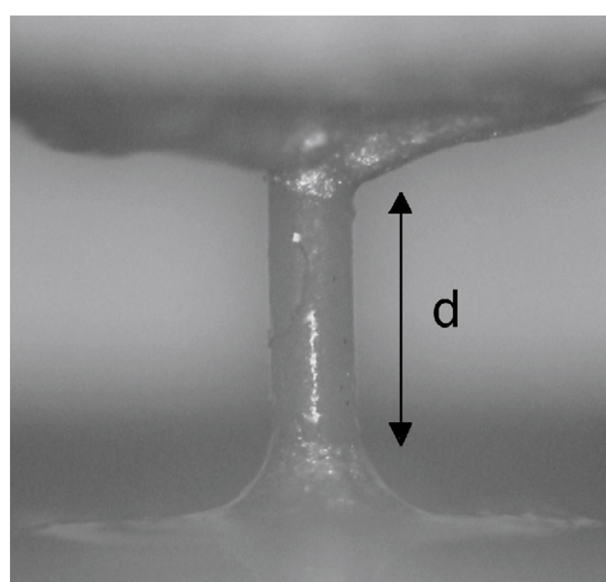

Fig. 2. Fibre glued in the sample holder with affixed capacitor plate: d– distance between the electrodes.

on the bottom side. The electric contact of this electrode was connected also on the top side of the disc. The bottom side of this disc is one plate of the measurement capacity (parallel plate capacitor). Both electric contacts of the fibre and the measurement capacitor (HF resonance circuit), respectively, were separated by an isolating varnish.

4. Electric field displacement in single fibres

The ring design of the electrodes are comparable with interdigitated electrodes (IDE) used in piezoelectric fibre composites. The electric field distribution in such structures can be modeled by finite element method (FEM) or analytical approximations.[17,18] In our case the electrodes are complete ring-shaped. In the region between the ring electrodes the electric field is parallel to the fibre length direction and relatively constant. Under the ring electrode near the electrode edge the electric field distribution is inhomogeneous. Far from the edge of the electrode there is a so-called dead zone where the electric field strength is nearly zero. Fig. 3 shows the electric field distribution in a fibre calculated by a FEM analysis.

This distribution is important not only for the piezoelectric response of the fibre but also for the poling process. In the part between the electrode rings the fibre should be polarized parallel to the fibre length direction. In the near of the edges of the electrodes the polarization should be much smaller (incomplete poling). Additionally, the direction of polarization should be rotate perpendicular to the length direction. Otherwise, the region of inhomogeneous electric field is much smaller than the distance between the electrodes (1 mm). Thus, this effect is neglected and the electric field during poling and measuring the fibres is assumed as homogeneous. The electric field strength is calculated using

$$E = \frac{U_{appl}}{d} \quad (2)$$

where d is the distance between the edges of the electrodes. Consequently, in this work the piezoelectric strain of the fibre is determined by

$$S = \frac{u}{d} \quad (3)$$

where u is the measured piezoelectric displacement of the fibre.

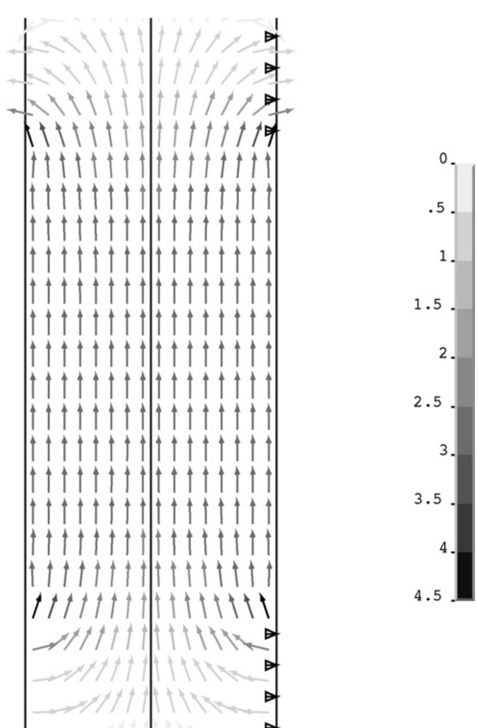

Fig. 3. Finite element analysis of the electric field distribution in a fibre with ring shaped electrodes.

5. Experimental results

5.1. Low voltage measurements

Before starting the measurements the PZT fibres were polarized by applying a DC voltage for 2 min. The electric field strength for poling was 3 kV/mm. The fibre was insulated with silicon oil in order to avoid electrical breakthrough at the surface of the fibre. The low voltage piezoelectric response, i.e. the linear piezoelectric coefficient d_{33} was measured with an applied voltage of around 3 V. The measurement frequency was varied from 0.5 up to 77 Hz. The measurement at lower frequencies using the compensation method described above is difficult due to the necessary long time constant of the LockIn-voltmeter. The measurement time for the compensation at 0.5 Hz is about 2 min. During this time the conditions especially at the air capacitor should be stable (for instance no temperature drift). Measurements at higher frequencies are not done yet. Possible problems could occur due to the natural oscillation of the fibre. A typical frequency dependence of the low-voltage piezoelectric coefficient d_{33} is shown in Fig. 4. The piezoelectric coefficient slowly decreases with increasing frequency. A small *peak* was determined around the supply frequency of 50 Hz. The curve was measured with an applied voltage of 3 V.

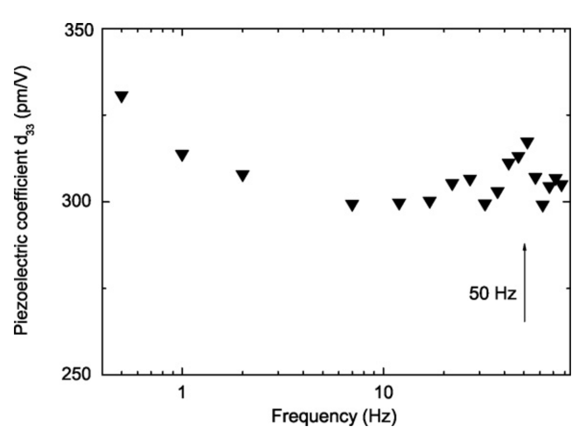

Fig. 4. Piezoelectric coefficient d_{33} vs. frequency measured with an applied voltage of 3 V.

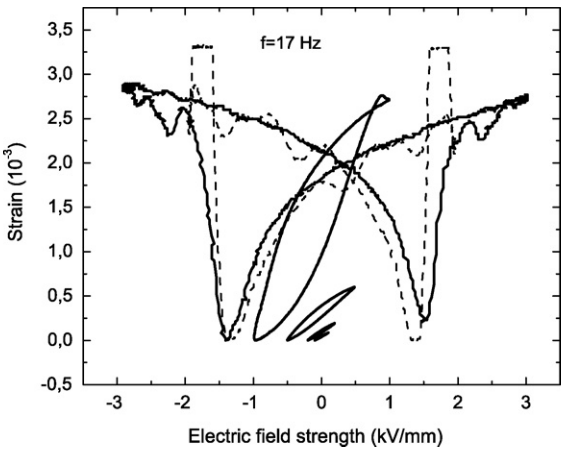

Fig. 5. Strain vs. electric field strength of a single PZT fibre at 17 Hz.

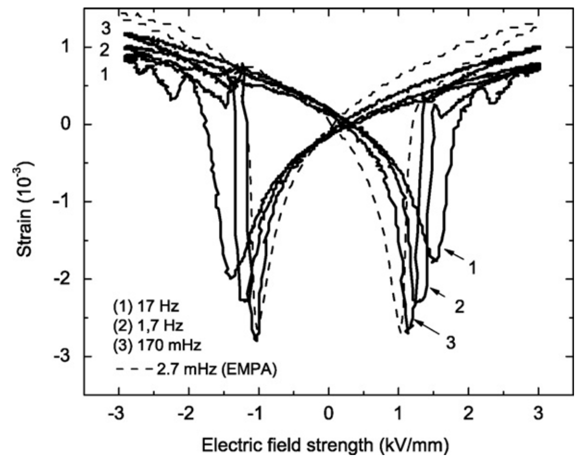

Fig. 6. Butterfly loops at different frequencies in comparison with DMA measurements.[14]

5.2. High voltage measurements

The high voltage behavior of single fibres was characterized by the strain-electric-field dependence, well known as butterfly loops. The piezoelectric strain S_3 increases with increasing electric field E. Up to an electric field of 0.5 kV/mm the loops were relatively linear without hysteresis. Close to the coercive field strength of 1.5 kV/mm switching processes start and the loop becomes a hysteretic shape. In Fig. 5 the curves are plotted so that the minimum of strain was set to zero. At electric field strength of 2 kV/mm and more a butterfly loop was obtained.

The symmetric loops show that the polarization can be completely switched. Some times a deformation of the curves was observed at higher electric fields in connection with the switching of polarization. Close to the coercive field strength the value and direction of the strain change in a very short time. The higher the frequency the higher the acceleration in the material as well as on the glued capacitor plate. Thus, oscillations of the system fibre/capacitor can be induced. We assumed that some times even bending oscillations are excited. That could explain the apparently higher strain values in some curves (dashed line).

At lower frequencies the oscillations are reduced and the butterfly loops becomes more and more smooth (Fig. 6). The maximum value of the strain increases with decreasing frequency. However, the coercive field strength decreases at lower frequencies. The results are in a good agreement with the measurements done by the DMA method at the Empa.[14]

The so-called high voltage piezoelectric coefficient of the nonlinear piezoelectric loop can be determined by

$$d_{33}^{*} = \frac{S_{max} - S_{rem}}{E_{max}} \qquad (4)$$

where S_{max} is the maximum value and S_{rem} is the remnant value of the strain, and E_{max} is the amplitude of the applied electric field. The results are shown in Fig. 7. For comparison the low voltage (linear) piezoelectric coefficient d_{33} is plotted too. The piezoelectric coefficient increases with increasing electric field up to the coercive field and decreases then again. This behavior is similar to bulk ceramics.

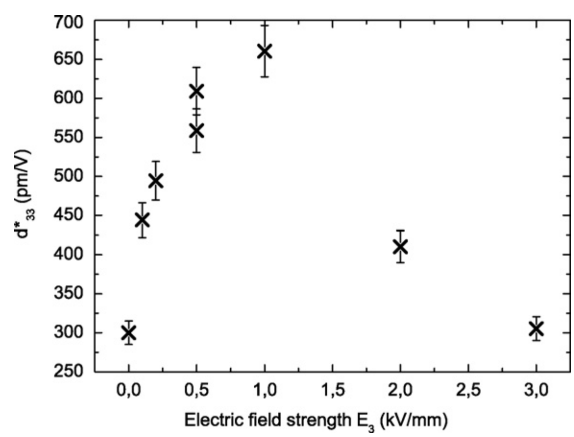

Fig. 7. Low and high voltage piezoelectric coefficients d_{33} and d_{33}^{*} of a single PZT fibre.

6. Conclusions

A capacitive displacement sensor was modified for the characterization of single ceramic fibres. The equipment was tested with PZT fibres with diameters of 200 μm. The low voltage piezoelectric coefficient d_{33} was measured with an applied voltage of 3 V at different frequencies between 0.5 and 100 Hz. In this region the coefficient decreases slowly with increasing frequency. The nonlinear behavior of the strain (butterfly loops) was measured at frequencies from 170 up to 17 Hz. At higher frequencies the strong piezoelectric response during the polarization switching excites oscillations of the mechanical system fibre/sensor. The results from low frequency measurements are comparable with DMA measurement done with the same fibres.

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