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PIEZOELECTRIC MATERIAL CHARACTERIZATION BY ACOUSTIC METHODS

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Two characterization methods of piezoelectric materials based on acoustic measurements are presented. Their main objective is to identify the entire set of electromechanical properties of piezoelectric materials from a single sample. In order to characterize new materials like single-crystals with small dimensions, the first section of the paper presents an identiffication protocol based on the study of the resonance spectra of parallelepipeds. The second characterization method is performed by means of the measurement of the transmission coefficients of plane waves propagating through an immersed plate. It is is suitable for the determination of the properties of bulk piezoelectric ceramics.

Keywords: piezoelectricity, transmission, resonance, spectroscopy.

1. Introduction

Piezoelectric material manufacturers are looking for characterization methods allowing the control of the quality of their production. To design complex devices, the complete set of constants of the constitutive materials is also needed. Classical characterization methods are based on electrical measurements [1], and at least three samples have to be used to identify the entire electromechanical tensors [2] leading to possible inconsistencies in the identified values. The main objective of the methods presented in this paper is to determine the entire set of electromechanical constants of piezoelectric materials from only one sample. To characterize single crystals with small dimensions, an identification protocol based on the study of the resonance spectra of parallelepipeds is first presented [3]. The second method is based on the measurement of the transmission coefficients of plane waves propagating through an immersed plate [4]. Due to the larger dimensions of the tested samples, it is well adapted to the determination of the properties of piezoelectric plates.

2. Resonant spectroscopy

This method was developed to characterize relaxor-based ferroelectric single crystals. These materials can have excellent piezoelectric properties and are very attractive for ultrasonic transducer applications requiring a high sensitivity and a large bandwidth [5]. However, the existing characterization methods have to be adapted to (i) the small dimensions of single crystals, (ii) their anisotropy degree, and (iii) the difficulty in obtaining homogeneous compositions. The study reported in this section is based on Resonant Ultrasound Spectroscopy (RUS) measurements and a new experimental set-up is proposed allowing an electrical excitation of the piezoelectric sample and the detection of its mechanical vibrations through a laser interferometer. The modelling is based on the decomposition of the Lagrangian of the piezoelectric solid on the Legendre polynomials. This leads to the definition of a linear problem whose eigen values and eigen vectors allow the determination of the resonance frequencies and of the corresponding mode-shapes [3].

2.1. Experimental results and discussion

This section presents the experimental set-up developed in [3] to generate and detect the free vibrations of a piezoelectric cube. An electrical excitation is delivered through an impedance analyzer (Agilent 4395A) also allowing the sample electrical resonances and anti-resonances to be measured (Fig. 1a). It has a very large frequency bandwidth (10 kHz – 500 MHz) and the delivered electrical power is set between 0 dBm and 10 dBm depending on the excited modes. The sample is set on a plastic holder and the electrical contact is ensured by a metallic strip fixed on a spring so that the free mechanical boundary conditions at the surfaces of the cube are fulfilled. Velocity measurements at the surface of the sample are carried out through a Laser vibrometer (Polytech OFV-505) to detect resonance frequencies as well as the associated mode-shapes. The interferometer is positioned at 50 cm from the sample leading to a 20 μm focal area. The velocity decoder sensitivity is either 5 (mm·s^{-1})/V or 25 (mm·s^{-1})/V depending on the cut-off frequency, respectively 250 kHz and 1.5 MHz. The sample holder is fixed onto a two-dimensional micrometer computer controlled translation unit. 100 acquisition points are taken on the surface of the sample leading to the representation of the mode-shapes for each resonance. To carry out the electrical excitation, the two surfaces of the sample orthogonal to the x_3 polarization axis are metallized. In order to generate a maximum number of modes, an electrode patterning is used allowing particular mode symmetries to be highlighted. One of the metallized faces is then only partially covered by electrodes (Fig. 1b) in a way to deliver enough electrical power while keeping the external electrical field close to zero.

To identify the material characteristics, a fit procedure has been developed and an identification protocol based on a sensitivity analysis is proposed in [3]. It is applied to the characterization of a $7.55 \times 7.52 \times 7.71$ mm^3 PZN-12%PT single crystal cube. The sample is poled along the $[001]_c$ direction leading to a 4 mm symmetry. It presents inter-

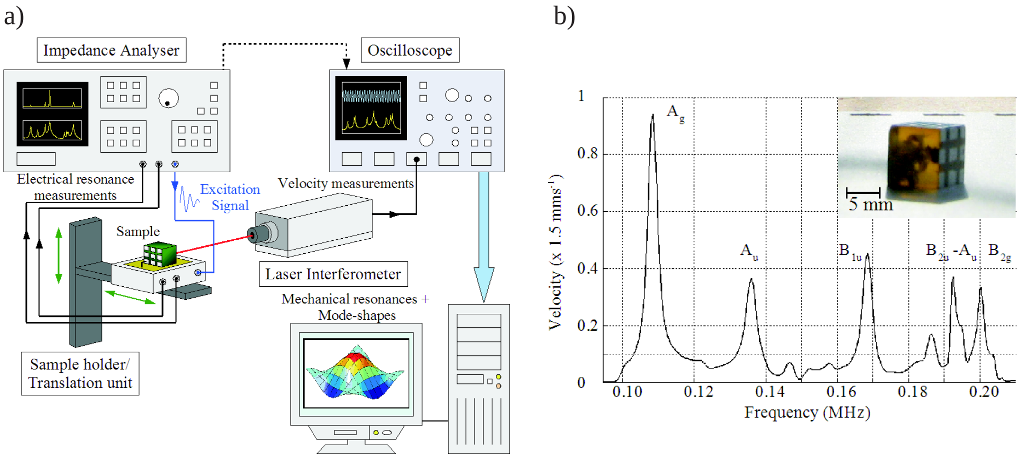

Fig. 1. a) Experimental set-up, b) PZN-12%PT single crystal cube and the corresponding surface velocity spectrum measured on one of the upper corners for frequencies ranging from 94.5 kHz to 210 kHz.

nal defects that might alter its resonant behaviour, but the number of measured modes and resonant frequencies are assumed to be sufficient to identify the electromechanical characteristics of the cube. Figure 1b presents the spectrum of the surface velocity measured by the laser vibrometer on one of the upper corners in the [94.5; 210] kHz frequency range. Six peaks are observed and identified through the measurement of their mechanical displacements. No existing electro-mechanical tensor of a PZN-12%PT single crystal is available in the literature, so the properties of a tetragonal PMN-42%PT single crystal [6] are used as initial guess in the characterization process. The PZN-12%PT single crystal characteristics presented in Table 1 are consistent with tensors of single crystals with similar compositions [5].

Table 1. PZN-12%PT single crystal cube properties identied by acoustic spectroscopy.

C_{11}^E [GPa]	C_{12}^E [GPa]	C_{13}^E [GPa]	C_{33}^E [GPa]	C_{44}^E [GPa]	C_{66}^E [GPa]
152 ± 2	487 ± 1	90 ± 0.5	84 ± 2	37 ± 3	22 ± 1
ρ [kg·m^{-3}]	e_{15} [C·m^{-2}]	e_{31} [C·m^{-2}]	e_{33} [C·m^{-2}]	ε_{11}^S (ε_0)	ε_{33}^S (ε_0)
8380	35 ± 3	-3 ± 0.5	4 ± 1	2420 ± 150	331 ± 50

3. Transmission spectroscopy

The second characterization method is based on the generation of acoustic fields with controlled propagation paths inside plates. The theoretical model describing reflection-transmission of plane acoustic waves through piezoelectric anisotropic plates is based on the octet formalism of piezoacoustics and the concept of the impedance/admittance [4, 7] matrices. Incorporating different types of electrical boundary conditions for an immersed plate, the coefficients are expressed through this admittance in explicitly the same way as in the case of a pure elastic plate [8].

3.1. High permittivity PZT-NN material characterization

This section presents the characterization of a high permittivity material – Pz59 – developed by Ferroperm [9] in the framework of the MINUET European project (no. NMP2-CT-2004-505657). Of $Pb[Zr,Ti,(Ni_{1/3}Nb_{2/3})]O_3$ composition, these materials are meant to be introduced into miniature multi-element devices such as transducer networks, and possess higher piezoelectric properties than classical ceramics.

To infer the material constants, ultrasonic plane wave transmission measurements are performed. The experimental set-up has lately been applied to the characterization of carbon polymer based composite plates [10] and is here adapted to the identification of the entire piezoelastic tensor of piezoelectric materials. As presented in Fig. 2a, the sample is immersed into a water tank. It is fixed on a motorized rotation stage. A 0.375 inch diameter transducer (Technisonic ISL-0303-HR) with a 3.5 MHz central frequency is excited by a pulse centered between 3.5 and 4 MHz. The transmitted acoustic fields are captured for an incident angle ranging from $0°$ to $50°$ every $0.5°$. The reference signal transmitted through water without the sample is also recorded. Fast Fourier Transform is then performed and the transmitted spectra are divided by the reference one, yielding the angular spectrum of the absolute value of transmission coefficient at a given frequency. As in Sec. 2, a fit procedure is then applied to compare the experimental transmission spectra to numerical simulations obtained from a program based on the formalism described in [7] in which the material constants C^E, e and ε are the input parameters. This method has already been validated on a Pz27 ceramic and the effect of the material metallization has been quantified [4]. It is here applied to the identification of the electromechanical constants of a Pz59 plate. The material properties are presented in Table 2.

Figure 2b presents the variation of the transmission coefficients with the incidence angle. The resonance angles are accurately identified, validating the determination of the real parts of the characteristics. However, amplitude discrepancies are observed. They

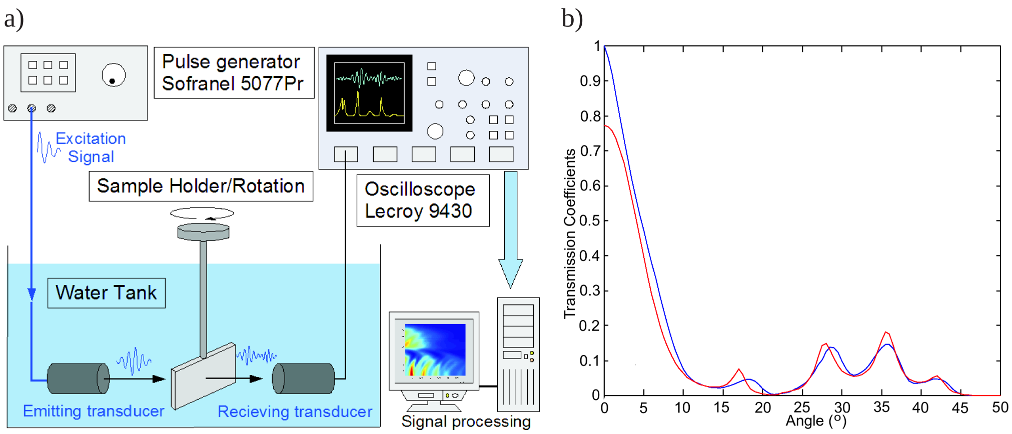

Fig. 2. a) Experimental setup, b) Evolution with the angle of the transmission at $f = 3.11$ MHz. Solid lines: measurements, dashed lines: numerical simulations.

Table 2. Acoustical characterization of a Pz59 plate.

ρ [kg·m^{-3}]	C_{11}^E [GPa]	C_{13}^E [GPa]	C_{55}^E [GPa]	C_{33}^E [GPa]
7850	$137.8 \pm 2.2\%$	$78.8 \pm 1.9\%$	$24.4 \pm 7.7\%$	$116.6 \pm 0.6\%$
e_{15} [C·m^{-2}]	e_{31} [C·m^{-2}]	e_{33} [C·m^{-2}]	ε_{11}^S (ε_0)	ε_{33}^S (ε_0)
$19.5 \pm 9.6\%$	$-7 \pm 58\%$	$25.4 \pm 1.6\%$	$4103 \pm 13.9\%$	$2729 \pm 2.9\%$

can be explained by diffraction losses and/or by a frequency dependence of the material damping. Most of the characteristics presented in Table 2 are identified with an accuracy better than 3%. C_{55}, e_{15} and ε_{11}^S have accuracies of 8%, 10%, and 14% respectively, remaining in the accuracy range of the piezoelectric material characterization methods. However, e_{31} is identified at about 60% accuracy. This can be explained with the help of the Christoffel tensor calculated in the x_1x_3 plane for a propagation angle θ given with respect to the normal of the plate surfaces. It shows that e_{31} always depends on the function sin(θ) cos(θ). This indicates that the e_{31} angular zone of sensitivity ranges between 30° and 70°. This angular region is situated above the longitudinal wave critical angle. Only transverse waves are then transmitted through the plate and their damping is greater than that of compressional waves. The amplitudes of the resulting signals are then small and the accuracy of e_{31} is deteriorated.

4. Conclusions

Two piezoelectric material characterization methods were presented. Resonant Ultrasound Spectroscopy measurements were carried out using an experimental set-up where the electrical excitation of the parallelepiped sample and the laser detection of its mechanical displacements enabled piezo-electrically coupled vibration modes to be studied. The method has been applied to the identification of the full tensor of a PZN-12%PT single crystal. The tested sample was of several hundreds of mm^3 but the instrumentation, and particularly the laser interferometer, should enable the characterisation of smaller samples. The second method is based on the propagation of acoustic waves through immersed plates. It was applied to the characterization of a high permittivity ceramic developed in the framework of the MINUET European Project for high frequency medical imaging. Most of the properties are identified with very good accuracies, but a sensitivity study is currently being carried out to define a new identification protocol and improve the robustness of the characterization method.

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