Piezoelectricity: Measurement of the resonant response of the radial mode at different temperatures

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(Recibido el 10 de Enero de 2011; aceptado el 19 de Marzo de 2011)

Abstract

A system to experimentally determine the resonant curves of the radial mode of piezoelectric samples at different temperatures is presented. Its design is based on the IEEE standard method for measuring the properties of piezoelectric materials. A description of the assembling, testing and performance of the experimental set up is given. PZT: Gd pellets are used as samples and the experiments are performed at different temperatures. Suitable software is used to obtain the characteristic physical parameters from direct calculations and the fitting of experimental curves. The experiment is intended to be a part of a laboratory course for senior students in physics and engineering.

Keywords: Piezoelectricity, resonant method of radial mode, PZT: Gd ceramics, Physics Education, laboratory experiments.

Resumen

Se describe un montaje experimental para la medición del modo radial en muestras piezoeléctricas a diferentes temperaturas. El diseño del equipo fue realizado en base al método estándar de la IEEE para la medición de propiedades de materiales piezoeléctricos. Se presenta la descripción, la calibración y espectros obtenidos con el montaje experimental. Cerámicas PZT dopadas con Gd son usadas como muestras y las mediciones son realizadas a diferentes temperaturas. Herramientas computacionales adecuadas son usadas para obtener los parámetros físicos y ajustar las curvas experimentales. El experimento descrito forma parte del curso de laboratorio para estudiantes de la carrera de física y de ingeniería.

Palabras clave: Piezoelectricidad, método resonante para el modo radial, cerámicas PZT: Gd, Enseñanza de la Física, experimentos de laboratorio.

PACS: 77.65.-j, 01.50.Pa, 01.40.-d **ISSN 1870-9095**

I. INTRODUCTION

Experimental Methods of Physics is an undergraduate course for senior physics students at the University of Havana. It includes laboratory exercises associated to research projects being carried on by the Faculty of the Physics School as well as by researchers of the Institute of Materials and Reagents for Electronics (IMRE). The experimental determination of the electromechanical parameters starting from the resonance curves of piezoelectric ceramic samples is one of the exercises included in the course. The purpose of this work is to describe how such experiment may be implemented in the laboratory using an experimental set up designed to determine the electromechanical parameters as functions of temperature and using a Gd doped PZT ceramic disk as a representative sample.

II. EXPERIMENTAL

A. Description of the piezoelectric ceramic's characteristics and the resonant method

A modified PZT ceramic sample will be used for the experiments. The $PbZr_{1-x}Ti_xO_3$ (PZT) solid solution with different values of the Zr/Ti ratio have been amply studied mainly due to their remarkable piezoelectric properties and great potential for technological applications [1, 2]. The x = 0.47 composition is particularly important as it is located in the morphotropic phase boundary (MPB) of the phase diagram where some of its properties are magnified. Among the relevant properties of the $PbZr_{0,53}Ti_{0,47}O_3$ (PZT 53/47) ceramic are a strong piezoelectric effect with a large electromechanical coupling factor and a high dielectric permittivity [3]. This ceramic has displaced barium titanate as the basic material for transducers because of its larger electromechanical coefficient (Kp) and higher values of the Curie temperature (Tc) allowing for higher temperatures of operation and device processing. Additionally PZT ceramics are easily poled [4, 5, 6].

The electromechanical parameters of piezoelectric materials are determined through the measurement of the impedance of the transducer as a function of frequency keeping the excitation voltage fixed. The piezoelectric behavior is evaluated by determining the resonant (fr) and antiresonant (fa) frequencies of the fundamental mode as well as the resonant frequency (fr_1) of the first harmonic [7]. The technique consists in exciting mechanical waves in the sample through the application of an ac electric field. Resonance will result when the frequencies of the applied field and the natural frequencies of the sample coincide. Additionally, it is very important to know the dependence and the stability of the electromechanical parameters with temperature as they may be used in applications where conditions may be very demanding.

The methodology for the characterization of the electromechanical parameters was described in the Standards on Piezoelectric Crystals in 1957 [7], and it is known as the transmission or parallel resonant method. It involves the determination of the resonant frequency (fr) at the impedance minimum (Z_{min}), the antiresonance frequency (fa) at the impedance maximum (Z_{max}) as well as the knowledge of the frequency of the first harmonic.

B. Thermodynamics of Piezoelectricity

The thermodynamical analysis of Piezoelectricity may be performed starting from Gibbs potential and the fundamental equations of piezoelectricity, as presented by Mason [8], which is:

$$ds_{ij} = \left(\frac{\partial s_{ij}}{\partial T_j}\right)_{T,E} dT_j + \left(\frac{\partial s_i}{\partial E_m}\right)_{T_i,T} dE_m + \left(\frac{\partial s_i}{\partial T}\right)_{T,E} dT\,, \quad (1)$$

$$dD_m = \left(\frac{\partial D_m}{\partial T_j}\right)_{T,E} dT_j + \left(\frac{\partial D_m}{\partial E_m}\right)_{T_i,T} dE_m + \left(\frac{\partial D_m}{\partial T}\right)_{T,E} dT\,,$$

$$dD_m = \left(\frac{\partial D_m}{\partial T_j}\right)_{T,E} dT_j + \left(\frac{\partial D_m}{\partial E_m}\right)_{T_i,T} dE_m + \left(\frac{\partial D_m}{\partial T}\right)_{T,E} dT\,, \quad (2)$$

$$dS = \left(\frac{\partial S}{\partial T_j}\right)_{T,E} dT_j + \left(\frac{\partial S}{\partial E_m}\right)_{T_i,T} dE_m + \left(\frac{\partial S}{\partial T}\right)_{T,E} dT\,, \quad (3)$$

where $\boldsymbol{S}$ is entropy, $\boldsymbol{D}$ is electric displacement, $\boldsymbol{s}_{ij}$ is strain tensor, $\boldsymbol{T}_j$ is stress tensor, $\boldsymbol{E}_m$ is electric field and T is temperature, then:

$$s_i = s_{ij}{}^{E,T} T_{ij} + d_m{}^{T} E_m + \alpha T\,, \quad (4)$$

$$D_m = d_{mi}{}^{T} T_{ij} + \varepsilon_{mk}{}^{T} E_k + pT\,. \quad (5)$$

It may be observed from Eqs. 4 and 5 how elastic $s_i = s_{ij}{}^{E,T} T_{ij}$, direct piezoelectric $s_i = d_m{}^{T} E_m$, inverse piezoelectric $D_m = d_{mi}{}^{T} T_{ij}$, pyroelectric $D_m = pT$, dielectric $D_m = \varepsilon_{mk}{}^{T} E_k$, and thermal $s_i = \alpha T$ phenomena are present in the expressions. Thermal phenomena are often study at room temperature but it is interesting to show how temperature influences the piezoelectric properties.

III. DESCRIPTION OF THE AUTOMATED THERMO-SPECTRAL ANALYZER. PRINCIPLE OF OPERATION

The measuring system of the response of the electromechanical resonance consists of a dual signal generator with sine and square wave output; a frequency range of 4Hz-4MHz and variable amplitude of 0-10V. The signal from the generator is applied to a voltage divider circuit where the sample is connected and the output signal is measured with an RMS and phase detector (Fig. 1). The experiment consists in sweeping the frequency of the input signal in a predetermined range and amplitude and measuring the RMS output signal, the input-output phase shift and the corresponding frequency value. In the case of poled ceramics and thin films a typical resonance response is obtained, thereby the name of the technique.

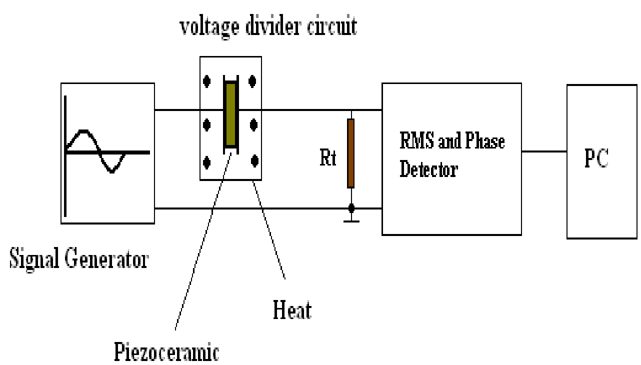

FIGURE 1. Schematic diagram of the experimental set up of the Transmission or Resonant Method.

The electromechanical measurements as functions of temperature were realized using the automated thermo-spectral analyzer described above. The data precision is of 2000 points per decade of frequency. The sample is placed in a heating stage where the temperature may be set in the 25-200°C interval and automatically controlled within ±1°C. Several measurements at the same temperature were performed to test stability. The system is controlled by a personal computer (PC) and software made for this purpose. The data analysis was realized using custom made software, taking into account the expressions for the radial mode and the requirements derived from the ANSI/IEEE Piezolectricity Standards [7].

To obtain good impedance matching the signal generator must have output impedance lower than the minimum impedance of the piezoceramic sample. The resistance Rt, of known value, must also be lower than the minimum impedance (Rt < Z_{min}) of the sample. For the determination of the antiresonance frequency fa, corresponding to the impedance maximum (Z_{max}), a higher value resistance (Rt > Z_{max}) has to be used. In general, there are no problems for ceramics with high dielectric constant except in the case of the determination of the transverse coupling factor k_{33} in very long cylinders.

IV. VIBRATIONS OF THE RADIAL MODE IN DISK-SHAPED SAMPLES

For the analysis of the vibrations in the radial mode [9] a PZT piezoceramic disk poled perpendicular to its plane faces and its center coinciding with the origin of a Cartesian coordinate and cylindrical coordinate systems, as shown in Fig. 2. The rectangular axes are x_1, x_2, and x_3; while the cylindrical axes are r, θ y x_3. The disk faces are located at $x_3 = \pm b$. The disk radius is a. The deformations in the r direction will be labeled u_r . The equation of motion of the system, in cylindrical coordinates is given by:

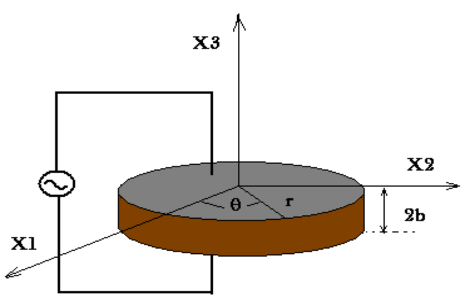

FIGURE 2. Electroded disk positioned in Cartesian and cylindrical coordinate systems.

$$\frac{\partial T_{rr}}{\partial r} + \frac{T_{rr} - T_{\theta\theta}}{r} = \rho \ddot{u}_r . \qquad (6)$$

Substituting the stress components:

$$c_{11}^{p}\left[u_{r,rr} + \left(u_{r,r} / r\right) - \left(u_r / r^2\right)\right] = \rho \ddot{u} . \qquad (7)$$

Since the system is assumed as time independent, Eq. (7) reduces to:

$$u_{r,rr} + \frac{u_{r,r}}{r} + \left[\frac{\omega^2}{\left(\upsilon^P\right)^2} - \frac{1}{r^2}\right] u_r = 0 , \qquad (8)$$

where ω is the angular frequency and $(\upsilon^p)^2 = c_{11}^p / \rho$ gives the speed of the mechanical wave.

For a solid disk, the motion at the center is zero and the general solution is

$$u_r = BJ_1\left(\frac{\omega}{\upsilon^P} r\right) e^{i\omega t} , \qquad (9)$$

where J_1 is the Bessel function of the first order.

Eq. (8) is subject to the following boundary conditions: $T_{rr} = 0$, for $r = a$. That is:

$$c_{11}^{p} u_{r,r} + c_{12}^{p} u_r / r + e_{31}^{p} V / 2be^{iwt} = 0 . \qquad (10)$$

Eq. (10) requires that:

$$c_{11}^{P} B \left.\frac{dJ_1}{dr}\right|_{r=a} + c_{12}^{P} B \frac{J_1}{a} = -e_{31}^{P} \frac{V}{2b} . \qquad (11)$$

The argument of the Bessel function is omitted. At resonance, the applied voltage can be zero and Eq. (11) transforms into:

$$\left.\frac{dJ_1}{dr}\right|_{r=a} + \sigma^P \frac{J_1}{a} = 0 , \qquad (12)$$

where

$$\sigma^P = c_{12}^{P} / c_{11}^{P} , \qquad (13)$$

σ has to be interpreted as a planar Poisson relation if the sample is isotropic in the plane normal to x_3. Before evaluating explicitly the frequency for resonance conditions, the equation for antiresonance will be derived. The total charge on one of the electrodes is:

$$Q = 2\pi \int_0^a (e_{31}^{p}(u_{r,r} + u_r / r) - \varepsilon_{33}^{p} \frac{V}{2b} e^{i\omega t}) r dr . \qquad (14)$$

Where $\eta = \omega a / \upsilon^p$ is the argument of the Bessel function.

From this we can obtain:

$$i = \frac{dQ}{dt} = i\omega \left\{ \frac{2\left(e_{31}^{P}\right)^2}{c_{11}^{P}\sigma_{33}^{P}} \frac{J_1(\eta)}{\left[\left(1-\sigma^P\right)J_1(\eta) - J_0(\eta)\right]} - 1 \right\} \frac{\pi a^2 e_{33}^{P} V}{2b} . \qquad (15)$$

Taking into account that the resonance frequency of a vibration mode depends in general on the material's properties as well as in its dimensions, according to B. Jaffe [3] we can write: $fr \propto \frac{1}{l(T) * \sqrt{\rho(T) s_{ij}(T)}}$ where l, ρ, s_{ij} are the length, the density and the elastic compliance of the material, respectively, all of them dependent on temperature. When a piezoceramic material is embedded in a medium with constant temperature, as this temperature is increased, it dilates making its resonance frequency decrease.

V. ANALYSIS OF RESULTS

The characterization of the PZT53/47: Gd ceramic, synthesized in our laboratory was reported in previous publications [10, 11, 12]. The sample was poled at 20kV/cm. For the characterization, 1 volt amplitude sinusoidal signals with frequency in the 100kHz-4MHz range were selected. Temperature was used as parameter. Each experiment was performed at constant temperature in the 25-150°C interval with steps of 2 and 5°C [8, 9, 10]. Measurements were made at the same temperature to insure stability and to watch for possible degrading effects such as aging and fatigue in the studied samples. Fig. 3 shows the electromechanical resonance in the radial mode and its harmonics at different temperatures in piezoelectric samples 11.29mm in diameter and 0.6mm thickness. A zoom of the region around the peak is inserted to show the shifting of the resonance *fr* and antiresonance *fa* frequencies with temperature for the case of the 5°C steps.

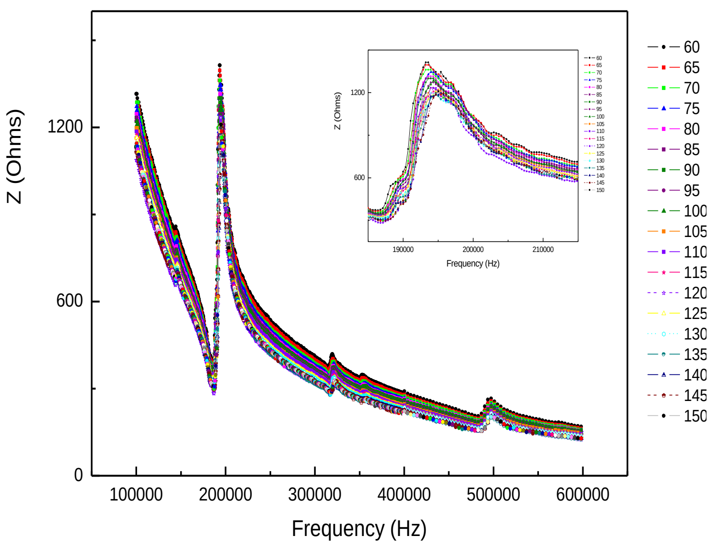

FIGURE 3. *Impedance vs. Frequency* in the radial mode at different temperatures for a PZT57/43: Gd piezoceramic sample.

Fig. 4 shows that the samples are not affected by ferroelectric aging and fatigue and are thermally stable. Three out of the 10 measurements at 100°C were chosen to facilitate visualization and show that no variation with temperature was recorded. The same behavior was observed for the other temperatures.

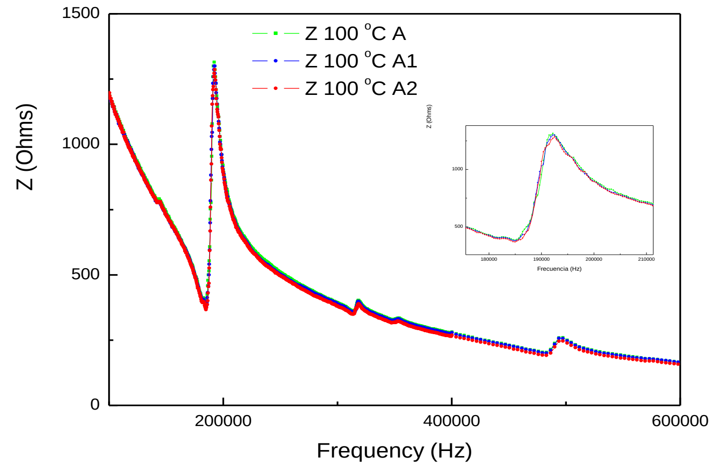

FIGURE 4. *Impedance vs. Frequency* realized with three repetitions at 100°C.

This characterization was a test of confidence for the technique and several of the obtained spectra were selected for the educational experiment.

The determination of the resonance (*fr*) and antiresonance (*fa*) frequencies at the minimum (Z_{min}) and maximum (Z_{max}) impedances, respectively, was made from the experimental curves with the help of a computer program. From these data all the electromechanical parameters were obtained in the 25-200°C temperature range. Fig. 5 shows the experimental curve as well as the calculated theoretical one obtained from expression (15) for the impedance [9, 10]. As can be seen, there is a good agreement in the positions of the fundamental mode and the first harmonic.

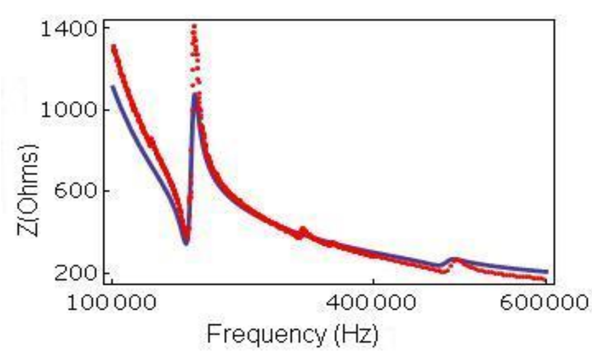

FIGURE 5. Impedance *vs.* Frequency theoretical (blue) and experimental (red) curves.

VI. REALIZATION OF THE LABORATORY EXERCISE

Al leaflet [12] with instructions was provided as a laboratory guide for the students where piezoelectricity is explained in a succinct form and the experimental set up is described. A list of the physical quantities to be measured is presented and some of the most frequently found difficulties when measuring small ac signals are commented. Instructions on how to operate the program to calculate the piezoelectric constants from the three characteristic frequencies and sample properties are provided. A guide on how to write the final report is also included.

VII. CONCLUSIONS

This laboratory experiment gives a useful introduction to the field of piezoelectric materials through the determination of the electromechanical parameters.

The thermodynamics of piezoelectricity is experimentally visualized as the effect of temperature on the characteristic frequencies and the electromechanical properties is evaluated.

We are confident that with the information supplied in this report, undergraduate physics and engineering students will be able to implement the experiment in their laboratories and perform the experiment.

This laboratory exercise has been successfully essayed with senior undergraduate physics students since 2006 and as a result, great interest on piezoelectric materials and their electromechanical properties was generated.

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