UNIVERSITAT POLITÈCNICA DE VALÈNCIA

Higher Polytechnic School of Gandia

Electrical characterization of piezoelectric ceramics

Master's Thesis

Master's Degree in Acoustic Engineering

AUTHOR: Ibarra Garcia, Diego Alexander

Tutor: Camarena Femenia, Francisco

ACADEMIC YEAR: 2022/2023

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Abstract

For the characterisation of piezoelectric materials, the resonance method, described by the IEEE (Institute of Electrical and Electronics Engineers), is a dynamic non-destructive evaluation in which the material constants can be calculated. Four piezoelectric ceramics were chosen and measured utilising an impedance analyser, obtaining the resonant frequency, the anti-resonant frequency, and the capacitance values. The elastic, piezoelectric and dielectric constants were numerically calculated using the formulas derived from the diverse resonance of longitudinal and radial modes. To verify the accuracy of the resonance method, a comparison with the manufacturer's datasheet values was realised, obtaining satisfactory results.

Keywords: ultrasonics; piezoelectricity; characterization; materials.

Resumen

Para la caracterización de materiales piezoeléctricos, el método de resonancias, descrito por el IEEE(Institute of Electrical and Electronics Engineers), es un método no destructiva dinámica para la obtención de las constantes del material. Cuatro cerámicas piezoeléctricas fueron elegidas y medidas utilizando un analizador de impedancias, obteniendo los valores de la frecuencia de resonancia, la frecuencia anti-resonancia y la capacitancia. Las constantes elásticas, piezoeléctricas y dieléctricas fueron calculadas utilizando las fórmulas derivadas de los diferentes modos de resonancia longitudinal y radial. Para verificar la exactitud del método de resonancia, se realizó una comparación de los resultados obtenidos con los valores del fabricante obteniendo resultados adecuados.

Palabras clave: ultrasonido; piezoelectricidad; caracterización; materiales.

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God for giving me the health to accomplish my objectives and dreams in life.

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“From what once was a seed,
To what we’ve grown to be
Traveled miles,
Across the oceans and seas
Nothing’s out of our reach,
Living positively”
- Through the Darkest Dark and Brightest Bright
by We Came As Romans

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List of Symbols and Units

A	Area	[squared meter]
$c_{\alpha\beta}$	Elastic stiffness constant	[newton per square meter]
C	Capacitance	[farad]
C_h	Capacitance $(2f_p)$	[farad]
$d_{i\alpha}$, $d_{i\beta}$	Piezoelectric constant	[meter per volt]
D	Elastic displacement	[coulomb per square meter]
D_i	Elastic displacement component	[coulomb per square meter]
$e_{i\alpha}, e_{i\beta}$	Piezoelectric constant	[coulomb per square meter]
E	Electric field	[volt per meter]
E_i	Electric field component	[volt per meter]
f	Frequency	[hertz]
f_p	Frequency of maximum resistance	[hertz]
f_s	Frequency of maximum conductance	[hertz]
Δf	$f_p - f_s$	[hertz]
F	Helmholtz free energy	[joules]
$g_{i\alpha}, g_{i\beta}$	Piezoelectric constant	[volt meter per newton]
G	Gibbs free energy	[joules]
G_1	Elastic Gibbs free energy	[joules]
G_2	Electric Gibbs free energy	[joules]
$h_{i\alpha,h_{i\beta}}$	Piezoelectric constant	[volt per meter]
k_{mn}	Coupling factor	
k_p	Planar coupling factor	
k_t	Thickness coupling factor	
l	Length	[meter]
m	Mass	[kilogram]
p_i	Pyroelectric coefficient	[coulomb per square meter kelvin]
Q	Charge	[coulomb]
$s_{\alpha\beta}$	Elastic compliance constant	[square meter per newton]
S	Entropy	[joules per kelvin]
S	Strain	
S_α, S_β	Strain component	
t	Thickness	[meter]
T	Temperature	[kelvin]
T	Stress	[newton per square meter]
T_α, T_β	Stress component	[newton per square meter]
V	Voltage	[volt]
w	Width	[meter]
W^{elec}	Electrical work	[joules]
W^{mech}	Mechanical work	[joules]
α_α	Thermal expansion coefficient	[micro unit per kelvin]
β_{ik}	Impermittivity component	[meter per farad]
ε_{ik}	Permittivity component	[farad per meter]
ρ	Density	[kilogram per cubic meter]

Introduction

Piezoelectricity is a phenomenon in which a material converts mechanical energy to electrical energy; hence, the material becomes electrically polarized due to an applied stress, and electrical energy to mechanical energy, the material changes shape as a result of an applied electric field.

This effect was first discovered by Pierre Curie and Jacques Curie in 1880. In 1910 development of applications for piezoelectric materials started, all thanks to the publishing made by Woldemar Voigt, *Lehrbuch der Kristallphysik* where he described several classes of natural crystal with piezoelectric properties. Crystalline materials like quartz, which produce the piezoelectric effect, were utilized first in developing sonar using ultrasonic transducers for use on submarines.

The concepts behind the piezoelectric effect are complex since many physical theories are involved when analysing this subject. The areas that cover this topic are mechanics, electrostatics, thermodynamics, acoustics, optics, fluids dynamics, circuit theory, crystallography, etc. For this work, the piezoelectric effect will be explained and analysed as a common type of ultrasound source, a plate vibrating in its thickness mode and a radial disk vibrating in radial mode, for the purpose of characterising piezoelectric ceramics. Thus, characterising piezoelectric ceramics by obtaining certain electrical, mechanical and electromechanical constants allows for modelling their operation.

Objectives

General

Develop the resonance method for piezoelectric materials and evaluate the accuracy by characterizing two squared and two circular disks of Lead zirconate titanate (PTZ) synthetic piezoelectric ceramics.

Specifics

1. Describe the piezoelectric effect by summarizing the existing literature.
2. Demonstrate that the piezoelectric electromechanical equations of state can be derived from the thermodynamic potentials.
3. Compare the piezoelectric coefficient values obtained by the resonance method with the manufacturer's values written in the datasheet.

1 PIEZOELECTRICITY

1.1 Piezoelectric Effect

If a stress (T) is applied to a solid, it will deform the material and have a different size and shape. This deformation of a stressed material is called strain (S). The stress and strain are related by the elastic modulus (Y), Young's modulus, and is given by the following equation,

$$T = YS \tag{1.1}$$

Piezoelectricity is the additional creation of an electric charge by the applied stress, and this is known as the direct piezoelectric effect [1]. The charge is proportional to the force, and it is of opposite sign of compression and tension. In terms of dielectric displacement D (charge per unit area) and stress, we may write

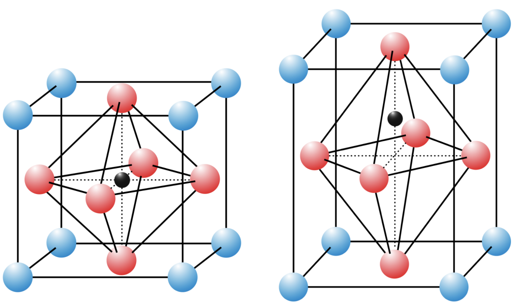
$$D = \frac{Q}{A} = dT \tag{1.2}$$

There is a converse piezoelectric effect, in which an applied electrical field E produces a proportional strain, expansion or contraction depending on polarity.

$$S = dE \tag{1.3}$$

where d, for the direct and converse effect, is a piezoelectric constant with dimensions of coulombs/newton or meters per volt.

The direct piezoelectric effect can briefly be explained qualitatively. The atoms of a solid (and also the electrons within the atoms themselves) are displaced when the material is deformed. This displacement produces microscopic electrical dipoles within the medium, and in particular crystal structures, these dipole moments combine to give an average macroscopic moment (or electrical polarization) [2]. The crystal structure of a traditional piezoelectric ceramic is shown in Figure 1.1.



(a) Cubic. (b) Tetragonal.

Figure 1.1: Cubic and tetragonal lattice of a piezoelectric ceramic. (a) Arrangement of positive and negative charges. (b) Crystal has electric dipole.

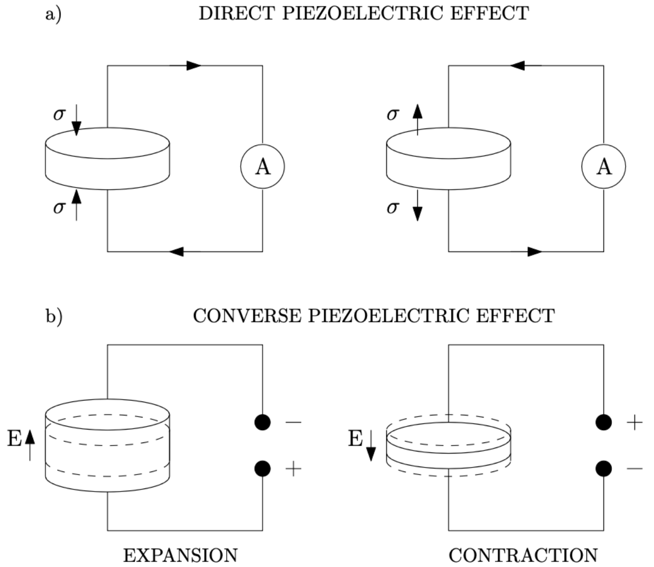


Figure 1.2: Schematic of the piezoelectric effect. a) Direct piezoelectric effect. b) Converse piezoelectric effect.

The direct piezoelectric effect is always accompanied by the converse piezoelectric effect, whereby a solid becomes strained when placed in an electric field. Like the direct effect, this is also linear, and the piezoelectric strain reverses sign with reversal of the applied electric field [2].

The schematic representation of the direct and converse piezoelectric effect is shown in Figure 1.2. Basically, in the direct case, when applied stress occurs, it creates an electrical current; in the converse case, when using an electric field, it creates an expansion or contraction.

In principle, all crystals in a ferroelectric state are also piezoelectric [3]. A ferroelectric crystal exhibits an internal dielectric moment even without an electric field. Above the Curie temperature (Figure Figure 1.1a), the temperature at which certain materials lose their permanent magnetic properties, the crystal loses its ferroelectric state. On the other hand, below the Curie temperature (Figure Figure 1.1b), the crystal structure displays a tetragonal symmetry, where the positive electric charges' centre of symmetry is distinct from the centre of symmetry of the negative charges, causing an electric dipole. Thus, ferroelectric materials have very high dielectric constants and, if available as single crystals, exhibit strong piezoelectric effects [4].

1.2 Piezoelectric Materials

1.2.1 Brief History

After the discovery of the piezoelectric effect by the Curie brothers (1880s) and the publishing of Voigt's first collection of piezoelectric materials (1910), French physicist Paul Langevin used quartz to invent echolocation. Many years later, after the sonar technology appeared, scientists from the United States, Japan, and the Soviet Union noticed that some ceramic materials presented piezoelectric effects just like the natural crystals and that it was quite more easy to manufacture, resulting in developing piezoceramics using barium titanate and lead zirconate titanate.

Since then, plenty of materials, from natural to synthetic origin, have been used. The most commonly used materials are listed and described as follows:

1.2.2 Natural Crystals

- Lead titanate ($PbTiO_3$): Is a single crystal material. Recently, it has become increasingly used because of its excellent mechanical and piezoelectric properties and its very high Curie temperature (1200°C). It is used notably, for instance, in the fabrication of Rayleigh wave filters [4].

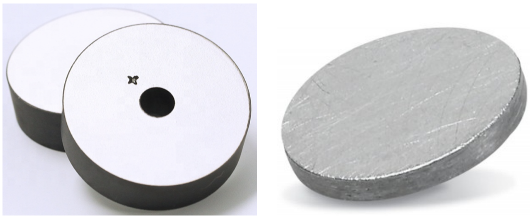
- Quartz: Most popular single crystal piezoelectric material. On account of its excellent mechanical and electrical properties are still firmly established among the practically used piezo materials, especially for frequencies above 10 MHz. Quartz is very resistant to chemical agents and can be used at elevated temperatures. At a temperature of 573°C, however, it is converted into a different modification which is not piezoelectric [4]. Nowadays, quartz is being used in wireless communications and, mainly, in wristwatches and clocks, due to the quartz's stable frequency resonator and stableness against the ambient temperature.

- Topaz: It is a single crystal material that exhibits piezoelectricity, but due to its properties, it is not relatively easy to work with, and instead, quartz is more commonly used.

(a) Quartz.

(b) Topaz.

Figure 1.3: Natural crystals.



(a) Barium titanate. (b) PTZ.

Figure 1.4: Synthetic Ceramics.

1.2.3 Synthetic Ceramics

- Barium titanate ($BaTiO_3$): It is a single crystal piezoelectric material used for Langevin-type piezoelectric vibrators, additionally, can be used for microphones and other transducers.

- Lead zirconate titanate ($Pb[Zr_x \ Ti_{1-x}] \ O_3$ $(0 \leq x \leq 1)$): Commonly known as PZT, it is the most used piezoelectric ceramic due to its high sensitivity, chemically inert, are physically strong, have higher operating temperature, and are inexpensive to manufacture. PZT ceramics are used for soft and hard applications. For soft (sensor) applications, PZT ceramic powders are typically used when high coupling and/or high charge sensitivity are essential, such as inflow or level sensors, ultrasonic nondestructive testing/evaluation or for accurate inspections of automotive, structural or aerospace products. For Hard (high power) applications, PZT ceramic powders are used when high power characteristics are required, including applications such as ultrasonic or high-voltage energy generation in ultrasonic cleaners, sonar devices, etc. [5].

- Potassium niobate ($KNbO_3$): This single crystal lead-free piezoelectric ceramic material has an excellent piezoelectric property and a high Curie point suited for applications in the field of electromechanical conversion.

1.2.4 Other Materials

There are even more piezoelectric materials. Lithium niobate ($LiNbO_3$) is a popular one, Langasite ($La_3Ga_5SiO_{14}$) crystals are piezoelectric material for surface acoustic wave (SAW) filter, Gallium orthophosphate ($GaPO_4$) which is quite similar to quartz, the bone which when a stress is applied produces the piezoelectric effect, and the DNA.

1.3 The Electromechanical Equations of State

The elastic, dielectric and piezoelectric tensor components define the properties of a piezoelectric material. It is important to note that these components are not constants since they depend on the temperature, applied mechanical stress and electric field. The following mathematical description is documented on Heywang W. Piezoelectricity [6].

The energy conservation for linear piezoelectric theory is given by the first law of thermodynamics to obtain the basic piezoelectric equations. Six parameters are used to obtain the properties of such material:

- Temperature T and Entropy S.
- Stress T and Strain S (components T_α and S_β)
- Electric field E and Elastic displacement D (components E_i and D_k)

In general, in a piezoelectric material, the effects of the magnetic field are not considered ($H = 0$).

The thermodynamic potentials must be defined to obtain the equations of state. Additional to the internal energy U, where is a function of strain, electric displacement, and entropy, we must use Helmholtz free energy F, Elastic Gibbs free energy G_1, Electric Gibbs free energy G_2 and Gibbs free energy G.

$$\begin{aligned}
U &= U(S_\alpha, D_i, \mathrm{S}) \\
F &= U - \mathrm{TS} \\
G_1 &= U - \mathrm{TS} - T_\alpha \\
G_2 &= U - \mathrm{TS} - E_i D_i \\
G &= U - \mathrm{TS} - T_\alpha S_\alpha - E_i D_i
\end{aligned} \tag{1.4}$$

The differential equations for these thermodynamic potentials are:

$$\begin{aligned}
dU &= \mathrm{T}d\mathrm{S} + T_\alpha dS_\alpha + E_i dD_i \\
dF &= -\mathrm{S}d\mathrm{T} + T_\alpha dS_\alpha + E_i dD_i \\
dG_1 &= -\mathrm{S}d\mathrm{T} - S_\alpha dT_\alpha + E_i dD_i \\
dG_2 &= -\mathrm{S}d\mathrm{T} + T_\alpha dS_\alpha - D_i dE_i \\
dG &= -\mathrm{S}d\mathrm{T} - S_\alpha dT_\alpha - D_i dE_i
\end{aligned} \tag{1.5}$$

When a particular set of independent variables is held constant, a system will come to thermodynamic equilibrium in such a way that the free energy, for which the constrained variables are the principal ones, is minimized.

Three relations can be obtained using the Gibbs potential if the temperature, stress, and electric field are independent variables.

$$-\mathrm{S} = \left(\frac{\partial G}{\partial \mathrm{T}}\right)_{T_\alpha,E_i}, -S_\alpha = \left(\frac{\partial G}{\partial T_\alpha}\right)_{\mathrm{T},E_i}, -D_i = \left(\frac{\partial G}{\partial E_i}\right)_{\mathrm{T},T_\alpha} \tag{1.6}$$

The linear differential form of these equations, for example, would be,

$$\Delta S_\alpha = \left(\frac{\partial S_\alpha}{\partial \mathrm{T}}\right)_{T_\alpha,E_i}\Delta\mathrm{T} + \left(\frac{\partial S_\alpha}{\partial T_\beta}\right)_{\mathrm{T},E_i}\Delta T_\beta + \left(\frac{\partial S_\alpha}{\partial E_i}\right)_{\mathrm{T},T_\alpha}\Delta E_i \tag{1.7}$$

which we obtain the equations

$$\begin{aligned} \Delta S_\alpha &= \alpha_\alpha^E \Delta\mathrm{T} + s_{\alpha\beta}^E \Delta T_\beta + d_{i\alpha}\Delta E_i \\ \Delta D_i &= p_i^T \Delta\mathrm{T} + d_{i\alpha}\Delta T_\alpha + \varepsilon_{ik}^T \Delta E_k \end{aligned} \tag{1.8}$$

Thus, from the Gibbs free energy, we obtained two equations, where α_α^E are the thermal expansion coefficients, $s_{\alpha\beta}^E$ is the elastic compliances at constant electric field, $d_{i\alpha}$ are the piezoelectric constants, p_i^T the pyroelectric coefficients, and ε_{ik}^T the permittivities at constant stress.

From equations (1.8), in the isothermal form, when $\Delta\mathrm{T} = 0$, we get the piezoelectric constitutive equations:

$$\begin{aligned} S_\alpha &= s_{\alpha\beta}^E T_\beta + d_{i\alpha} E_i \\ D_i &= d_{i\beta} T_\beta + \varepsilon_{ik}^T E_k \end{aligned} \tag{1.9}$$

Nevertheless, the alternate forms of constitutive equations are derived from the other thermodynamic potentials. In their isothermal form, these equations are:

$$\begin{aligned} T_\alpha &= c_{\alpha\beta}^D S_\beta + h_{i\alpha} D_i \\ E_i &= -h_{i\beta} S_\beta + \beta_{ik}^S D_k \end{aligned} \tag{1.10}$$

$$\begin{aligned} S_\alpha &= s_{\alpha\beta}^D T_\beta + g_{i\alpha} D_i \\ E_i &= -g_{i\beta} T_\beta + \beta_{ik}^T D_k \end{aligned} \tag{1.11}$$

$$\begin{aligned} T_\alpha &= c_{\alpha\beta}^E S_\beta - e_{i\alpha} E_i \\ D_i &= e_{i\beta} S_\beta + \varepsilon_{ik}^S E_k \end{aligned} \tag{1.12}$$

This set of equations (1.9, 1.10, 1.11, and 1.12) are called *basic piezoelectric equations* or *piezoelectric constitutive equations.*

It is important to note that the equations are written in matrix form following Voigt's notation[1] and Einstein's summation convention[2] of represented subscripts. Four piezoelectric compliances are defined, the piezoelectric strain coefficients d, the piezoelectric stress coefficients e, the piezoelectric voltage coefficients g, and the piezoelectric h coefficient h.

For equation 1.9, the elasto-piezo-dielectric matrix for a crystal class ∞mm is,

$$
\begin{bmatrix} S_1 \\ S_2 \\ S_3 \\ S_4 \\ S_5 \\ S_6 \end{bmatrix} = \begin{bmatrix} s_{11}^E & s_{12}^E & s_{13}^E & 0 & 0 & 0 \\ s_{12}^E & s_{11}^E & s_{13}^E & 0 & 0 & 0 \\ s_{13}^E & s_{13}^E & s_{33}^E & 0 & 0 & 0 \\ 0 & 0 & 0 & s_{44}^E & 0 & 0 \\ 0 & 0 & 0 & 0 & s_{44}^E & 0 \\ 0 & 0 & 0 & 0 & 0 & 2(s_{11}^E - s_{12}^E) \end{bmatrix} \begin{bmatrix} T_1 \\ T_2 \\ T_3 \\ T_4 \\ T_5 \\ T_6 \end{bmatrix} + \begin{bmatrix} 0 & 0 & d_{31} \\ 0 & 0 & d_{31} \\ 0 & 0 & d_{33} \\ 0 & d_{15} & 0 \\ d_{15} & 0 & 0 \\ 0 & 0 & 0 \end{bmatrix} \begin{bmatrix} E_1 \\ E_2 \\ E_3 \end{bmatrix}
$$

$$
\begin{bmatrix} D_1 \\ D_2 \\ D_3 \end{bmatrix} = \begin{bmatrix} 0 & 0 & 0 & 0 & d_{15} & 0 \\ 0 & 0 & 0 & d_{15} & 0 & 0 \\ d_{31} & d_{31} & d_{33} & 0 & 0 & 0 \end{bmatrix} \begin{bmatrix} T_1 \\ T_2 \\ T_3 \\ T_4 \\ T_5 \\ T_6 \end{bmatrix} + \begin{bmatrix} \varepsilon_{11}^T & 0 & 0 \\ 0 & \varepsilon_{11}^T & 0 \\ 0 & 0 & \varepsilon_{33}^T \end{bmatrix} \begin{bmatrix} E_1 \\ E_2 \\ E_3 \end{bmatrix} \tag{1.13}
$$

1.4 Piezoelectric Constants

The definitions of the most used constants, explained by americanpiezo [5], and the meaning of the superscripts and subscripts will be defined as follows.

- Piezoelectric Charge Constant d: is the polarization generated per unit of mechanical stress applied to a piezoelectric material. The first subscript refers to the direction of polarization generated in the material (at $E = 0$) or the applied field strength. The second refers respectively to the direction of the applied stress or the direction of the induced strain. The constant d indicates a material's suitability for strain-dependent (actuator) applications.
  - d_{33}: induced polarization in direction 3 (parallel to the direction in which ceramic element is polarized) per unit stress applied in direction 3.
  - d_{31}: induced polarization in direction 3 (parallel to the direction in which ceramic element is polarized) per unit stress applied in direction 1 (perpendicular to the direction in which ceramic element is polarized).

[1] Voigt notation: $i, k = 1, 2, 3$ and $\alpha, \beta = 1, ..., 6$.

[2] Einstein's summation convention: $T_\alpha S_\alpha \equiv \sum_\alpha T_\alpha S_\alpha$.

- d_{15}: induced polarization in direction 1 (perpendicular to the direction in which ceramic element is polarized) per unit shear stress applied about direction 2 (direction two perpendicular to the direction in which ceramic element is polarized).

- Elastic and Compliance s: is the strain produced in a piezoelectric material per unit of stress applied and, for the 11 and 33 directions, is the reciprocal of the modulus of elasticity. The first subscript indicates the direction of strain. The second is the direction of stress.
  - s_{11}^{E}: elastic compliance for stress in direction 1 (perpendicular to direction in which ceramic element is polarized) and accompanying strain in direction 1, under constant electric field (short circuit).
  - s_{33}^{D}: elastic compliance for stress in direction 3 (parallel to the direction in which ceramic element is polarized) and accompanying strain in direction 3, under constant electric displacement (open circuit).

- Permittivity Constant ε: The permittivity, or dielectric constant for a piezoelectric ceramic material, is the dielectric displacement per unit electric field. The first subscript indicates the direction of the dielectric displacement, and the second is the direction of the electric field.
  - ε_{11}^{T}: permittivity for dielectric displacement and electric field in direction 1 (perpendicular to the direction in which ceramic element is polarized), under constant stress.
  - ε_{33}^{S}: permittivity for dielectric displacement and electric field in direction 3 (parallel to the direction in which ceramic element is polarized), under constant strain.

- Piezoelectric Voltage Constant g: is the electric field generated by a piezoelectric material per unit of mechanical stress applied. The first subscript to g indicates the direction of the electric field generated in the material or the direction of the applied electric displacement. The second subscript is the direction of the applied stress or the induced strain. The constant g is essential for assessing a material's suitability for sensing (sensor) applications.
  - g_{33}: induced electric field in direction 3 (parallel to the direction in which ceramic element is polarized) per unit stress applied in direction 3.
  - g_{31}: induced electric field in direction 3 (parallel to the direction in which ceramic element is polarized) per unit stress applied in direction 1 (perpendicular to the direction in which ceramic element is polarized).

– g_{15}: induced electric field in direction 1 (perpendicular to the direction in which ceramic element is polarized) per unit shear stress applied about direction 2 (direction two perpendicular to the direction in which ceramic element is polarized).

Because a piezoelectric ceramic is anisotropic, physical constants relate to the direction of the applied mechanical or electric force and the directions perpendicular to the applied force. Consequently, each constant generally has two subscripts that indicate the directions of the two related quantities (e.g. stress and strain for elasticity and electric field and displacement for permittivity).

For example, in the piezoelectric strain coefficient $d_{i\alpha}$, the first subscript i gives the direction of the electric field associated with the applied voltage and the second subscript α gives the direction of mechanical strain. The coefficient d_{31} relates the 3-axis to the electric field and the 1-axis to the strain.

The superscripts denote the context in which the piezoelectric material constants were measured. A superscript E means it was estimated at constant electric field (electrodes short-circuited), T at constant stress (mechanically free), S constant electric field (electrodes short-circuited), and D at constant electric displacement (electrodes open-circuited).

The direction of positive polarization usually coincides with the Z-axis of a rectangular system of X, Y, and Z axes, as shown in Figure 1.5. Direction X, Y, or Z is represented by the subscript 1, 2, or 3, respectively, and shear about one of these axes is represented by the subscripts 4, 5, or 6.

1.5 Electromechanical Coupling Factors

Another set of properties needs to be defined for the characterisation of piezoelectric ceramics, mainly if the small signal method is applied for characterising materials. The *coupling factors* are nondimensional coefficients helpful in describing a particular piezoelectric material under a specific stress and electric field configuration for converting stored energy to mechanical or electric work

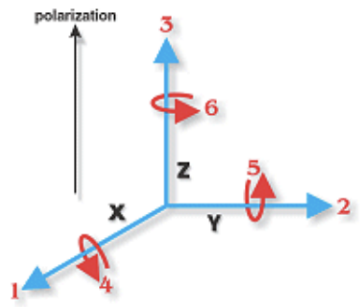


Figure 1.5: Direction of forces affecting a piezoelectric element [5].

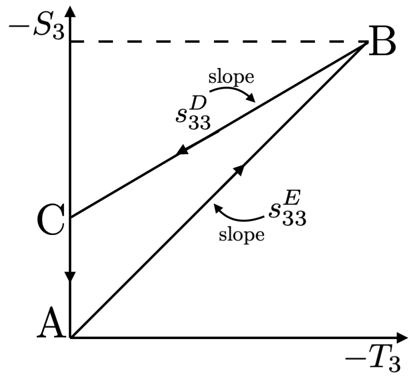


Figure 1.6: Conversion cycle of mechanical to electrical energy.

[7]. These coupling factors are helpful when comparing different piezoelectric materials since they are composed of elastic, piezoelectric and dielectric coefficients.

The coupling factors are also considered as the efficiency of converting energy from mechanical to electrical work or from electrical to mechanical work. To measure the fraction of electrical energy to mechanical energy, the coupling factor k^2 is,

$$k^2 = \frac{\text{electrical energy converted to mechanical energy}}{\text{input electrical energy}} \tag{1.14}$$

The energy conversion does not completely occur, so k^2 is always ¡1. Therefore, also k is less than one.

Figure 1.6 shows the conversion of mechanical work to electrical work. From A to B, increasing stress is applied until it reaches a maximum stress which the material short-circuited. Then, the stress is removed from B to C, and the material is open-circuited. And finally, from C to A, the electrical energy stored in the material is transferred to a connected ideal electric load, where the cycle returns to its initial state.

The mechanical work done from A $\Rightarrow$ B, B $\Rightarrow$ C and the electrical work from C $\Rightarrow$ A, are,

$$W_{AB}^{mech} = \frac{1}{2} s_{33}^E T_{max}^2$$

$$W_{BC}^{mech} = \frac{1}{2} s_{33}^D T_{max}^2$$

$$W_{CA}^{elec} = W_{AB} - W_{BC} = \frac{1}{2} T_{max}^2 (s_{33}^E - s_{33}^D)$$

Then, the coupling factor k_{33}^2 is,

$$k_{33}^2 = \frac{\text{mechanical energy converted to electrical energy}}{\text{input mechanical energy}} = \frac{W_{AB} - W_{BC}}{W_{AB}} \tag{1.15}$$

Finally,

$$k_{33}^2 = \frac{s_{33}^E - s_{33}^D}{s_{33}^E} = \frac{d_{33}^2}{\varepsilon_{33}^T s_{33}^E} \tag{1.16}$$

As we can see from equation (1.16), the coupling factor k_{33}^2 is defined in terms of the elastic, piezoelectric and dielectric coefficients.

In general, the definition of the coupling factor can be used in two forms, to characterize the energy conversion efficiency of a specific piezoelectric transducer (transducer coupling factor) or to characterize the energy conversion efficiency of a material of interest (material coupling factor) [6].

For the characterization of materials, additional coupling factors must be used. These coupling factors are,

$$k_{15}^2 = \frac{d_{15}^2}{\varepsilon_{11}^T s_{55}^E} \tag{1.17}$$

$$k_{31}^2 = \frac{d_{31}^2}{\varepsilon_{33}^T s_{11}^E} \tag{1.18}$$

$$k_p = k_{31} / \sqrt{2/(1 - (-s_{12}^E / s_{11}^E))} \tag{1.19}$$

$$k_t = \frac{e_{33}^2}{c_3^D 3 \varepsilon_{33}^S} \tag{1.20}$$

1.6 Static and Dynamic Methods

1.6.1 Static Methods

Static method measurement techniques for piezoelectric materials involve applying a constant electric field to the material and measuring the resulting mechanical deformation. These methods help characterize the material's piezoelectric properties.

The measurement of d_{33} or other d constants can be made. For direct measurement of the d constant, it is a good practice to have two weights. With the sample connected to a large, high-quality capacitor (to keep a low, almost constant field as specified by the boundary condition), the second weight is removed "without shock". The voltage on the shunt capacitor, measured with a sensitive electrometer, indicates the magnitude of the d constant [1].

1.6.2 Dynamic Methods

Dynamic methods can be employed with excellent accuracy for many crystal orientations and sample geometries. Since elastic bodies show numerous resonances, thus, resonance methods are used to evaluate piezoelectric effects.

The main properties of a piezoelectric vibrator, the frequency and the parameters of the equivalent electric circuit, are expressed in terms of elastic, piezoelectric, and dielectric constants. Therefore, the values for these constants can be derived from measurements of resonance frequency, the dimensions, and the density of a suitably oriented specimen [8]. Simply put, the process evaluates the electrical impedance of the resonator as a function of frequency to measure the capacitance, the serial resonant frequency f_s, and the parallel resonant frequency f_p.

2 EQUIPMENT AND METHODOLOGY

2.1 Equipment

The most relevant equipment for the characterization of piezoelectric ceramics is described below.

2.1.1 Piezoelectric Ceramics

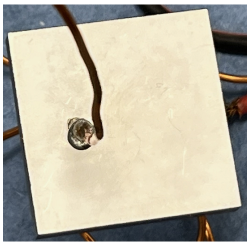
Four piezoelectric ceramics were selected to characterize them—two squared plates of different sizes and two circular disks of different radii. The dimension values of each ceramic are shown in Table 2.1.

Table 2.1: Dimensions of the four piezoelectric ceramics.

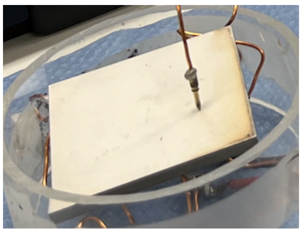
Ceramic	*Length* [mm]	*Thickness* [mm]	*Mass* [g]	*Density* [g/mm^3]
Square 1	$25.0 \pm 0.2^\dagger$	4.0 ± 0.2	20.0 ± 0.1	8.0 ± 0.4 e-3
Square 2	$50.0 \pm 0.2^\dagger$	4.0 ± 0.2	80.1 ± 0.1	8.0 ± 0.4 e-3
Cir. disk B9	$50.0 \pm 0.2^\ddagger$	2.0 ± 0.2	30.7 ± 0.1	7.8 ± 0.8 e-3
Cir. disk C7	$60.0 \pm 0.2^\ddagger$	2.0 ± 0.2	45.1 ± 0.1	7.9 ± 0.8 e-3

† The value is the length of one side of the square.
‡ The value is the diameter of the circular disk.

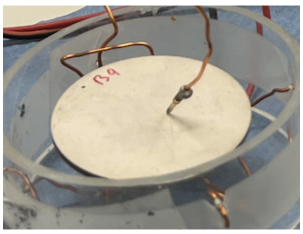
The piezoelectric ceramics are shown in Figure 2.1. The piezoelectric ceramics materials are based on lead zirconate titanate (PZT) and are categorized as hard PZT material. Thus, these ceramics can be subjected to high mechanical and electrical stresses, which makes them useful for high-power applications due to their stability over time.



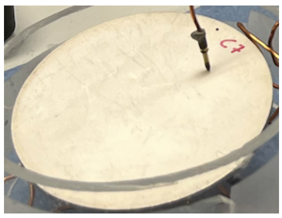
(a) Square 1.



(b) Square 2.



(c) Circular disk B9.



(d) Circular disk C7.

Figure 2.1: Selected piezoelectric ceramics for their respective characterization.

Figure 2.2: Impedance Analyzer IM3570 [9].

2.1.2 Impedance Analyzer

The HIOKI IM3570 impedance analyzer is an impedance measuring instrument which achieves high speed and high accuracy. It has two functions: an impedance analyzer capable of the sweep measurement of frequencies and measurement signals, and an LCR meter capable of simultaneously displaying up to four items under individual measurement conditions. This instrument can be used for a wide range of applications because you can set a wide range of measurement conditions - a measurement frequencies from 4 Hz to 5 MHz and a measurement signal levels from 5 mV to 5 V [9].

The accuracy of the Impedance Analyzer for the measurements items and frequencies are:

- $\pm$ 0.08%: Impedance Z, Admittance Y, Equivalent series resistance R_s, Series equivalent coapacitance C_s, etc.
- $\pm$ 0.01%: Measurements of frequency.

2.2 Methodology

Elastic bodies show numerous mechanical resonances related to half wavelength standing elastic waves. The piezoelectric effect offers a simple method to excite these elastic waves electrically to permit observation of mechanical and electromechanical material properties [6].

The resonance method provides the theory for the mode of motion of a known specimen to obtain the value of the elastic, piezoelectric, and dielectric constants. The IEEE Standard [7] explains and indicates that to measure the materials constants, it is necessary to use samples displaying distinct mode shape, each associated with its unique geometric form.

A brief explanation of the distinct mode shape will be done in the following sections. Related to the measurements, it basically consists of determining the resonator's electrical impedance and admittance as a function of frequency. In principle, measuring the series resonance frequency f_s, the parallel resonant frequency f_p, and the capacitance is necessary.

2.2.1 Thickness Extension (TE)

The polarization and vibration directions follows along the depth (thickness). The conditions must satisfy that the relation between length and thickness $(l/t)^2$ and the length and width $(l/w)^2$ both need to be greater than 10. The following material constants can be calculated,

$$k_t^2 = \frac{\pi}{2}\frac{f_s}{f_p}tan\Big(\frac{\pi}{2}\frac{\Delta f}{f_p}\Big) \tag{2.1}$$

The elastic stiffness c_{33}^D can be determined by the measurement of f_p, thus, another elastic constant, c_{33}^E, can be obtain.

$$c_{33}^D = 4\rho(f_p t)^2 \tag{2.2}$$

$$c_{33}^E = c_{33}^D(1 - k_t^2) \tag{2.3}$$

2.2.2 Length Extension (LE)

in this mode, a thin square column has a vibration and polarization along the direction of the length. The square column must have a ration of length and width greater than 5. The material constants that can be calculated are,

$$k_{33}^2 = \frac{\pi}{2}\frac{f_s}{f_p}tan\Big(\frac{\pi}{2}\frac{\Delta f}{f_p}\Big) \tag{2.4}$$

where Δf is $f_p - f_s$. Other constants can be obtained following the relations between them:

$$s_{33}^D = \frac{1}{4\rho f_p^2 l^2} \tag{2.5}$$

$$s_{33}^E = \frac{s_{33}^D}{(1 - k_{33}^2)} \tag{2.6}$$

2.2.3 Length Thickness Extension (LTE)

The polarization orientation is perpendicular to the vibration occurring along the length. The bars should have (l/t) greater than 10 and $(l/w)^2$ greater than 3. The transverse coupling factor k_{31} can be determined directly from the fundamental frequencies f_p and f_s. Also, other material constants.

$$\frac{k_{31}^2}{(1 - k_{31}^2)} = \frac{\pi}{2}\frac{f_p}{f_s}tan\Big(\frac{\pi}{2}\frac{\Delta f}{f_s}\Big) \tag{2.7}$$

$$s_{11}^E = \frac{1}{4\rho f_s^2 l^2} \tag{2.8}$$

$$s_{11}^D = s_{11}^E(1 = k_{31}^2) \tag{2.9}$$

2.2.4 Radial Extension (RAD)

In this mode, the polarization and vibration of a thin round plate are both oriented along the depth. The circular disk must have a diameter greater than ten times the thickness ($d > 10t$). One of the fundamental measurements for piezoelectric ceramics is determining the planar coupling factor k_p. The term "planar" is used because the stress is two-dimensional (plane) isotropic [1]. The planar coupling factor is related to the conventional piezoelectric and elastic constants by,

$$k_p^2 = \frac{2d_{31}^2}{\varepsilon_{33}^T(s_{11}^E + s_{12}^E)} \tag{2.10}$$

Also, the relationship of k_p with the series and parallel frequencies is,

$$k_p^2 \cong \frac{5}{2}\frac{\Delta f}{f_p} - \left(\frac{\Delta f}{f_p}\right)^2 \tag{2.11}$$

2.2.5 Capacitance

When a piezoelectric ceramic operates at a lower value than the resonant frequency, it behaves as a capacitor. To obtain one of the piezoelectric coefficients, the permittivity ε_{33}^T, the value of the capacitance will be needed, along with the shape dimensions of the ceramic. Since electrical charges will be produced in the surface of the material and if electrodes are connected to the upper and lower surface, the charges generate an electric voltage V and for a dielectric material, there is an electric flux ϕ.

$$V = \frac{Q}{C} = Et \ , \ \phi = \frac{Q}{\varepsilon_m} = EA$$

where Q is the charge, C the capacitance, E the electric field, A is the area, t the thickness, and ε_m is the permittivity of the material. Substituting the charge from one equation into the other, then the capacitance is,

$$C = \varepsilon_m \frac{A}{t} \tag{2.12}$$

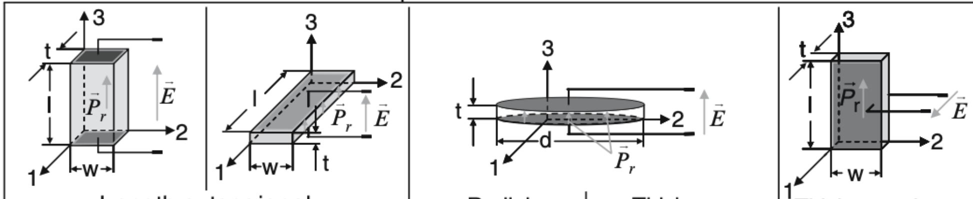
For a plate, with width w and length l, the capacitance results,

$$C = \varepsilon_m \frac{wl}{t} \tag{2.13}$$

For the case of a circular disk, the area is πr^2, accommodating the area variables in terms of the diameter d, the resulting capacitance is,

$$C = \varepsilon_m \frac{\pi d^2}{4t} \tag{2.14}$$

Using equation 2.13 for a plate or equation 2.14 for a circular disk, the piezoelectric coefficient $\varepsilon_m = \varepsilon_{33}^T$ can be obtained.



	Sample/mode: Length-extensional	Length-extensional	Radial	Thickness	Thickness shear
Eq. (1)		$C = \varepsilon_{33}^T \cdot \frac{w \cdot l}{t}$	$C = \varepsilon_{33}^T \cdot \frac{\pi \cdot d^2}{4t}$		$C = \varepsilon_{11}^T \cdot \frac{w \cdot l}{t}$
Eq. (2)	$k_{33}^2 = \frac{\pi}{2} \frac{f_s}{f_p} \tan\left(\frac{\pi}{2} \frac{\Delta f}{f_p}\right)$	$k_{31}^2 / (1-k_{31}^2) = \frac{\pi}{2} \frac{f_p}{f_s} \tan\left(\frac{\pi}{2} \frac{\Delta f}{f_p}\right)$	$k_p^2 \cong \frac{5}{2} \frac{\Delta f}{f_p} - \left(\frac{\Delta f}{f_p}\right)^2$	$k_t^2 = \frac{\pi}{2} \frac{f_s}{f_p} \tan \frac{\pi}{2} \frac{\Delta f}{f_p}$	$k_{15}^2 = \frac{\pi}{2} \frac{f_s}{f_p} \tan\left(\frac{\pi}{2} \frac{\Delta f}{f_p}\right)$
Eq. (3)	$\frac{1}{s_{33}^D} = 4\rho \left(f_p \cdot l\right)^2$	$\frac{1}{s_{11}^E} = 4\rho \left(f_s \cdot l\right)^2$	$s_{11}^E \left(1 - \sigma^{E^2}\right) = \frac{\eta_1^2}{\pi^2 \rho \left(f_s \cdot d\right)^2}$	$c_{33}^D = 4\rho \left(f_p \cdot t\right)^2$	$c_{44}^D = \frac{1}{s_{44}^E} = 4\rho \cdot \left(f_p \cdot t\right)^2$
Eq. (4)	$s_{33}^E = \frac{s_{33}^D}{\left(1 - k_{33}^2\right)}$	$s_{11}^D = s_{11}^E \left(1 - k_{31}^2\right)$	$k_p^2 = \frac{2 d_{31}^2}{\left(s_{11}^E + s_{12}^E\right) \varepsilon_{33}^T}$	$c_{33}^E = c_{33}^D \left(1 - k_t^2\right)$	$c_{44}^E = 1/s_{44}^E = c_{44}^D \left(1 - k_{15}^2\right)$
Eq. (5)	$k_{33}^2 = \frac{d_{33}^2}{s_{33}^E \cdot \varepsilon_{33}^T}$	$k_{31}^2 = \frac{d_{31}^2}{s_{11}^E \cdot \varepsilon_{33}^T}$		$k_t^2 = \frac{e_{33}^2}{c_{33}^D \cdot \varepsilon_{33}^S}$	$k_{15}^2 = \frac{d_{15}^2}{s_{44}^E \cdot \varepsilon_{11}^T}$

Eq. (6)	$k_p^2 = \frac{2}{1 - \sigma^E} \cdot k_{31}^2$ $\sigma^E = \left\| \frac{s_{12}^E}{s_{11}^E} \right\|$	Eq. (7)	$s_{13}^{E^2} = \frac{1}{2}\left(s_{11}^E + s_{12}^E\right)\left(s_{33}^E - \frac{1}{c_{33}^E}\right)$	Eq. (8)	$k_t^2 = \frac{\left(k_{33} - a \cdot k_p\right)^2}{\left(1 - a^2\right)\left(1 - k_p^2\right)}$ $a^2 = 2 \cdot s_{13}^{E^2} / s_{33}^E \left(s_{11}^E + s_{12}^E\right)$

Figure 2.3: Formulas required for the determination of material coefficients. η_1 is the lowest positive root of $(1 + \sigma^E)J_1(\eta) = \eta J_0(\eta)$, J_0 and J_1 are Bessel functions of first kind and of zero and first order, respectively; $\eta_1 = 2.05$ for $\sigma^E = 0.31$ [6].

The compilation of all the formulas needed for obtaining the elastic, piezoelectric, and dielectric constants for different modes are shown in Figure 2.3. For the piezoelectric ceramics in Figure 2.1, the measurements and calculations for the squared ceramics are done using thickness-extensional modes of a plate and for the circular disk ceramics, the measurements and calculations are done in radial mode and thickness-extensional mode.

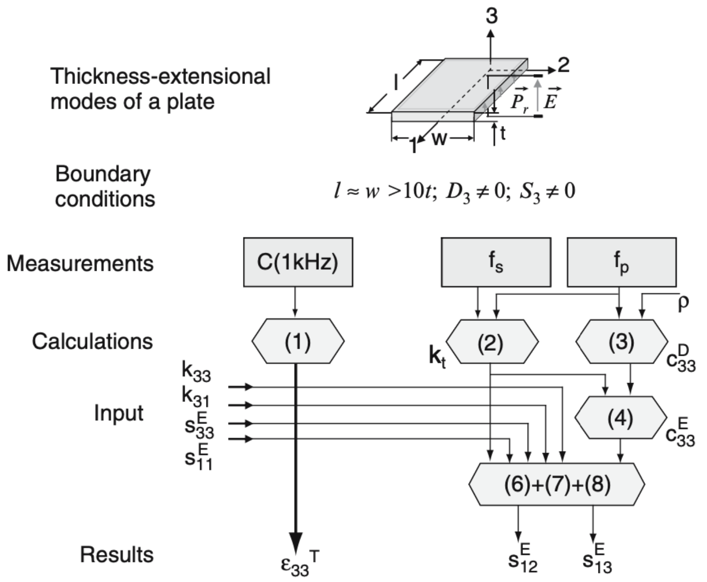


Figure 2.4: Determination of elasticity coefficients s_{12}^E and s_{13}^E using the thickness extensional mode of a plate resonator. Again ε_{33}^T can be obtained from measuring the resonator's capacitance at 1 kHz. The numbers in brackets denote the applied formulas shown in Figure 2.3 [6].

The sequence for determining the elasticity constants using the thickness extensional mode of a plate resonator is displayed in Figure 2.4. The equations for determining the elastic constants using radial modes and thickness extensional modes of a circular disk resonator are shown in Figure 2.5.

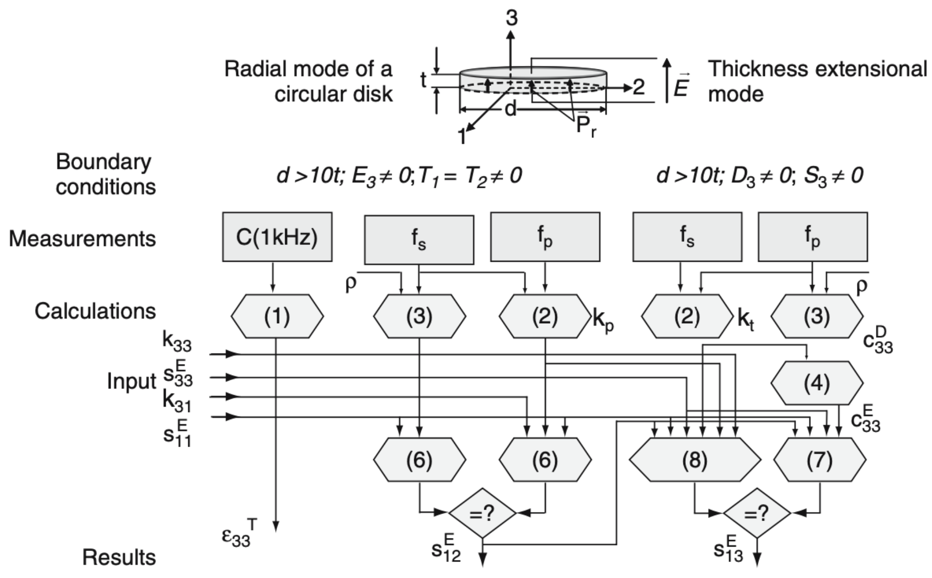


Figure 2.5: Determination of elasticity coefficients s_{12}^E and s_{13}^E using radial modes and thickness extensional modes of a circular disk resonator. Again ε_{33}^T can be obtained from measuring the resonators capacitance at 1 kHz. The numbers in brackets denote the applied formulas shown in Figure 2.3 [6].

Some input coefficients need to be ascertained to determine the coefficients that describe the piezoelectric materials. These input coefficients, k_{31}, k_{33}, s_{11}^E, and s_{33}^E, can be obtained by measuring the resonance frequencies of the radial mode and the thickness-extensional mode of a disk resonator or by measuring the resonance frequencies of the thickness-extensional mode of a thin rectangular plate [6].

2.3 Measurement Procedure

The preparation of the equipment for making the measurements and the acquisition of the data needed to determine the coefficients are described as follows:

1. Place the first ceramic in the base, positioning it without touching the borders or the bottom. In this case, a few cables were positioned below to support the ceramic's weight.

2. Connect the two main cables to the Impedance Analyser. The equipment configuration should be as in Figure 2.6.

3. Turn on the Impedance Analyser and set it in Analyser mode. The impedance (Z) and the admittance (Y) are measured in this mode, showcasing their respective curve. Analysing the peak of the impedance curve, the frequency of maximum resistance f_p is obtained and measuring the peak of the admittance, the frequency of maximum conductance f_s is determined.

4. Change to LCR mode in the Impedance Analyser.

5. Measure the capacitance (C) of the ceramic's resonator at a 1 kHz frequency.

6. Repeat the process 1 to 5 for the other three ceramics.

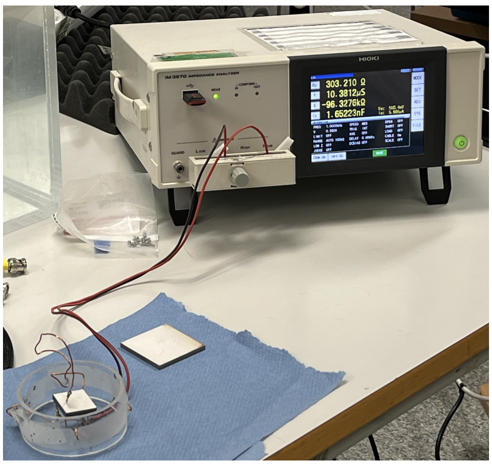


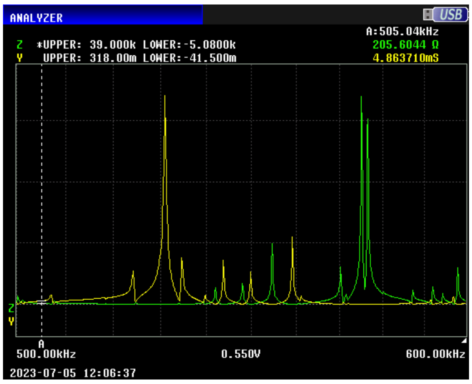
Figure 2.6: Equipment set-up.

2.4 Measurements Data.

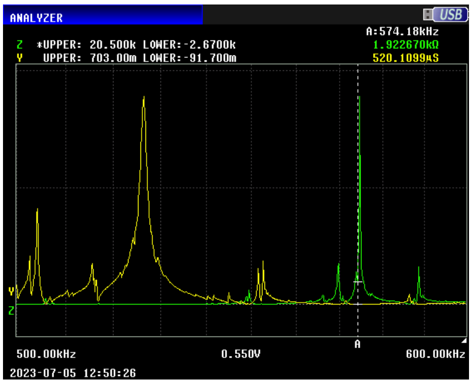
After the measurements, the impedance and admittance curves for the four piezoelectric ceramics, shown in Figure 2.7, were obtained. The values of the resonance frequencies, extracted for these impedance and admittance curves, and the capacitance, measured with the impedance analyser in LCR mode, are presented in Table 2.2.

Table 2.2: Measurements data of the resonance frequencies and the capacitance for the four piezoelectric ceramics.

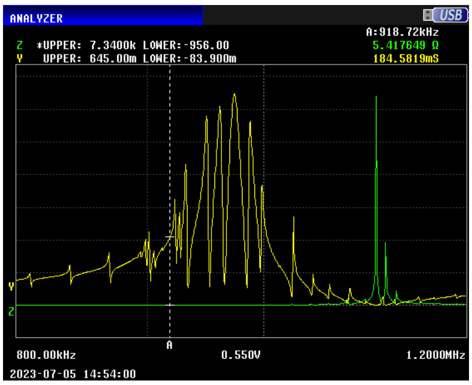
Ceramic	f_p [10^5 kHz]	f_s [10^5 kHz]	C [nF]
Square 1	5.7510 ± 0.0006	5.3101 ± 0.0005	1.6519 ± 0.0013
Square 2	5.7470 ± 0.0006	5.2643 ± 0.0005	6.4322 ± 0.0051
Circular disk B9	11.0650 ± 0.0011	9.7385 ± 0.0010	11.1629 ± 0.0089
Circular disk C7	10.8430 ± 0.0011	9.3421 ± 0.0009	15.6076 ± 0.0125



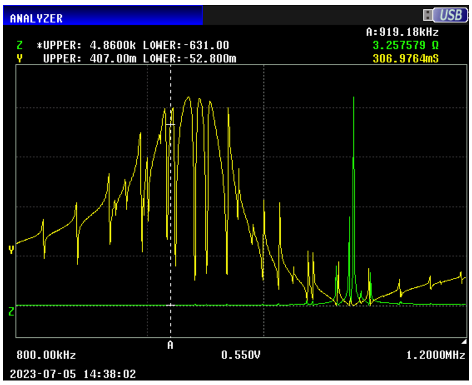
(a) Square 1.



(b) Square 2.



(c) Circular disk B9.



(d) Circular disk C7.

Figure 2.7: Impedance and Admittance curves for the four ceramics.

Following the sequence of Figure 2.4 and Figure 2.5, which references the formulas in Figure 2.3, by operating the measurements data from Table 2.2 determined one permittivity coefficient ε_{33}^T, two elastic stiffness coefficients c_{33}^D and c_{33}^E, and two coupling factors k_p and k_t. Two coupling factors, k_{31} and k_{33}, and two elastic compliance coefficients s_{11}^E and s_{33}^E are assigned as input. These input coefficients relate the coefficients determined by measurements with the last two elastic compliance coefficients s_{12}^E and s_{13}^E, which are obtained using formulas involving the previous coefficients and coupling factors. Additionally, a set of coefficients (s_{11}^D, s_{33}^D, and d_{31}) can be determined by operating the relationships described in the formulas in Figure 2.3. Lastly, additional constants can be known following the equations,

$$\varepsilon_{33}^S = \frac{C_h t}{A} \tag{2.15}$$

where A is the area and C_h is the capacitance at twice the anti-resonant frequency ($2f_p$). From equation (5, Figure 2.3) the piezoelectric stress constant is,

$$e_{33}^2 = kt^2 c_{33}^D \varepsilon_{33}^S \tag{2.16}$$

3 RESULTS

Numerical results of the material constants for the four ceramics are presented, showcasing the accuracy of the resonance method. Each constant and coupling factor's uncertainty is measured using the Joint Committee for Guides in Metrology (JCGM) guideline defined in Appendix A. Also, the Poisson's ratio corresponding to the resonant frequencies, Appendix B, was utilized for the calculations of the constants.

The constants of the four piezoelectric ceramics will be compared to the values given by the manufacturer. The manufacturer of square ceramics is PI Ceramics, and the material Type is PIC 181 [11]. The circular disks are ceramics from the manufacturer Ferroperm, the material is Type Pz26 [12].

The piezoelectric constants for both square piezoelectric ceramics and the comparison with the manufacturer are shown in Table 3.1.

Table 3.1: Square ceramics piezoelectric constants comparison determined by resonance method with manufacturer values.

Parameter	*Symbol & Units*	*Square 1*	*Square 2*	*PI Ceramics*
Permittivity	ε_{33}^{T} (10^{-8} F/m)	1.06 ± 0.05	1.03 ± 0.05	-
R. permittivity	$\varepsilon_{33}^{T}/\varepsilon_0$ †	1194 ± 63	1162 ± 59	1200
Permittivity	ε_{33}^{S} (10^{-8} F/m)	0.53 ± 0.03	0.51 ± 0.03	-
R. permittivity	$\varepsilon_{33}^{S}/\varepsilon_0$ †	597 ± 31	581 ± 29	-
Coupling	k_p	0.53	0.57	0.56
factors	k_t	0.42	0.44	0.46
	k_{31}	0.32	0.33	0.32
Piezoelectric const.	e_{33} (C/m^2)	12.53 ± 1.86	12.89 ± 1.81	-
Elastic const.	c_{33}^{D} (10^{10} N/m^2)	16.93 ± 1.91	16.93 ± 1.90	17
(stiffness)	c_{33}^{E} (10^{10} N/m^2)	13.96 ± 1.58	13.69 ± 1.54	-

† ε_0 is the Vacuum permittivity with value of 8.854×10^{-12} [F/m].

The piezoelectric constants for the two circular disks piezoelectric ceramics and the comparison with the manufacturer are shown in Table 3.2.

Table 3.2: Comparison of piezoelectric constants determined by resonance method with manufacturer values for the two circular disk ceramics.

Parameter	*Symbol*	*Circular disk B9*	*Circular disk C7*	*Ferroperm*
Permittivity	ε_{33}^{T} (10^{-8} F/m)	1.14 ± 0.11	1.10 ± 0.11	-
R. permittivity	$\varepsilon_{33}^{T}/\varepsilon_0$ †	1284.2 ± 129	1246.9 ± 125	1330
Permittivity	ε_{33}^{S} (10^{-8} F/m)	0.568 ± 0.057	0.552 ± 0.055	-
R. Permittivity	$\varepsilon_{33}^{S}/\varepsilon_0$ †	642 ± 65	623 ± 63	700
Coupling	k_p	0.534	0.572	0.568
factors	k_t	0.514	0.507	0.471
	k_{31}	0.314	0.336	0.327
Piezoelectric const.	e_{33} (C/m^2)	15.16 ± 3.62	14.61 ± 3.53	14.7
Elastic const.	c_{33}^{D} (10^{10} N/m^2)	15.32 ± 3.43	15.00 ± 3.36	15.8
(stiffness)	c_{33}^{E} (10^{10} N/m^2)	11.28 ± 2.52	11.14 ± 2.49	12.3

† ε_0 is the Vacuum permittivity with value of 8.854×10^{-12} [F/m].

3.1 Analysis

Analysing the results in Table 3.1, five constants and three coupling factors were calculated for each squared ceramic. Both ceramics present values quite similar to each other, and the uncertainties are also consistent. There is good coherence with the manufacturer values, starting with the relative permittivity at constant stress, the planar and thickness coupling factors are quite close, the coupling factor k_{31} is precisely the same for one ceramic and for the other one is just one value above, and the elastic constant at constant displacement is in the range of the manufacturer's value.

The manufacturer (PI Ceramics) does not provide the values of the relative permittivity at constant strain ε_{33}^{S}, the piezoelectric constant e_{33}, and the elastic constant c_{33}^{E}.

Regarding the circular disks B9 and C7, Table 3.2, five constants and three coupling factors were calculated. Both ceramics present values quite similar to each other which also applies to the uncertainties. Again, there is a good coherence when comparing the results with the manufacturer's values (Ferroperm), beginning with the relative permittivity at constant stress, the relative permittivity at constant strain for the circular disk B9, the planar and k_{31} coupling factors are quite close, also, the piezoelectric constant, and both elastic constants.

The two things to note are that the relative permittivity at constant strain for the circular disk C7 does not reach the manufacturer's value, and the thickness coupling factor for the two ceramics is a little above the manufacturer's value. In this case, all the manufacturer's values were provided.

Concerning the uncertainties, since the permittivity is calculated using the capacitance and the geometry of the ceramic, it is clearly noted that the uncertainties of the radial ceramics are greater than those of the square ceramics. Therefore, the measurements for the circular disk ceramics are less precise.

This work can be extended in future studies by obtaining most of the material's constants. A measurement in the Length Extension mode of a thin square column ceramic with a specification of $l > 5w_1, w_1 > 5w_2$ yields the coupling factor k_{33}, the elastic compliance constants s_{33}^D and s_{33}^E, and the piezoelectric constant d_{33}. Measuring in Thickness Shear Extension mode of a thin square plate with a specification of $l > 20t, w > 10t$, the permittivity ε_{11}^T, the coupling factor k_{15}, the piezoelectric constants d_{15}, the elastic constant s_{44}^E, and the elastic stiffness constants c_{44}^D and c_{44}^E can be obtained. Furthermore, with these measurements and the constants obtained, even more constants can be calculated such as s_{12}^E, s_{13}^E, s_{13}^D, c_{11}^E, c_{12}^E, c_{13}^E, etc.

4 CONCLUSIONS

This work aimed to characterise piezoelectric ceramics by implementing the resonance method. To accomplish the general objective, many important piezoelectricity concepts were summarised, the equipment was described along with the resonance method, and lastly, the materials constants were calculated.

In the first chapter, the piezoelectric effect, materials, the electromechanical equation of state deduced from the thermodynamics potentials, the piezoelectric constants, the coupling factors, and static and dynamic methods were defined, setting the coefficients that needed to be determined by using the dynamic method because of the excellent accuracy and the numerical values for these constants can be derived from measurements of the resonance frequency.

The second chapter described the equipment, the resonance method and the sequence to calculate the piezoelectric constants. The PTZ ceramics were measured with the impedance analyser, obtaining the typical curve of a piezoelectric material ceramic impedance. From the curves, three critical parameters, the series resonant frequency, the parallel anti-resonant frequency and the capacitance, were determined for each ceramic. The resonance method was explained, indicating the resonant modes for thickness extension, length extension, length thickness extension, and radial extension. Consequently, using the formulas for the respective type of mode, the piezoelectric constants for the ceramics were calculated.

Finally, as seen from the results and after analysing them, the resonance method is a practical approach to characterizing piezoelectric materials.

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APPENDIX

A UNCERTAINTY ANALYSIS

The uncertainties will be worked on following the terminology defined by the Joint Committee for Guides in Metrology (JCGM) [10]. This guide provides various definitions for estimating measurement uncertainties in diverse processes.

When calculating the uncertainties of measurements, two classifications are considered for the uncertainty components, generally classified as Type A and Type B, depending on the evaluation method.

- **Type A**: Method of evaluation of uncertainty by the statistical analysis of series of observations.
- **Type B**: Method of evaluation of uncertainty by means other than the statistical analysis of series of observations.

A.1 Combined Standard Uncertainty

A measurement is often not made directly since other n independent quantities determine it with a functional relationship f,

$$y = f(x_1, x_2, \cdots, x_n) \tag{A.1}$$

The standard uncertainty of y is obtained by appropriately combining the standard uncertainties $u(x_i)$ of the input estimates $x_1, x_2, \cdots, x_n$ resulting from a Type A evaluation or a Type B evaluation. This estimate is called the *combined standard uncertainty*, denoted by $u_c(y)$. The combined standard uncertainty $u_c(y)$ is the positive square root of the combined variance $u_c^2(y)$, which is given by,

$$u_c(y) = \sqrt{\left(\frac{\partial f}{\partial x_1}\right)^2 u^2(x_1) + \left(\frac{\partial f}{\partial x_2}\right)^2 u^2(x_2) + \cdots + \left(\frac{\partial f}{\partial x_n}\right)^2 u^2(x_n)} \tag{A.2}$$

$$u_c^2(y) = \sum_{i=1}^{n} \left(\frac{\partial f}{\partial x_i}\right)^2 u^2(x_i) \tag{A.3}$$

Note that the above equations (A.2 and A.3) are equivalent.

Using $u_c(y)$, we can express the uncertainty of the result of a measurement. In some industrial, commercial or regulatory applications, it is necessary to give a value of the uncertainty that defines an interval on the measurement of the result that is expected to cover a significant fraction of the distribution of values that could reasonably be attributed to the measurand.

B POISSONS'S RATIO

The following Table B.1 displays the Poisson's ratio σ^E corresponding to the ratio of the first two resonant frequencies.

Table B.1: Poisson's ratio corresponds to the ratio of the first two resonant frequencies.

σ^E	η_1	η_2	$f_s^{(2)}/f_s^{(1)}$	σ^E	η_1	η_2	$f_s^{(2)}/f_s^{(1)}$
0.00	1.8412	5.3314	2.8956	0.26	2.0236	5.3817	2.6595
0.01	1.8489	5.3334	2.8846	0.27	2.0300	5.3836	2.6520
0.02	1.8565	5.3353	2.8738	0.28	2.0363	5.3855	2.6447
0.03	1.8641	5.3372	2.8632	0.29	2.0426	5.3874	2.6375
0.04	1.8716	5.3392	2.8527	0.30	2.0489	5.3894	2.6304
0.05	1.8790	5.3411	2.8425	0.31	2.0551	5.3913	2.6234
0.06	1.8864	5.3431	2.8324	0.32	2.0612	5.3932	2.6165
0.07	1.8937	5.3450	2.8225	0.33	2.0674	5.3951	2.6096
0.08	1.9010	5.3469	2.8127	0.34	2.0735	5.3970	2.6028
0.09	1.9082	5.3489	2.8031	0.35	2.0795	5.3989	2.5962
0.10	1.9154	5.3508	2.7936	0.36	2.0855	5.4008	2.5897
0.11	1.9225	5.3528	2.7843	0.37	2.0915	5.4027	2.5832
0.12	1.9296	5.3547	2.7750	0.38	2.0974	5.4046	2.5768
0.13	1.9366	5.3566	2.7660	0.39	2.1033	5.4066	2.5705
0.14	1.9436	5.3586	2.7570	0.40	2.1092	5.4085	2.5642
0.15	1.9505	5.3605	2.7482	0.41	2.1150	5.4104	2.5581
0.16	1.9574	5.3624	2.7396	0.42	2.1208	5.4123	2.5520
0.17	1.9642	5.3644	2.7311	0.43	2.1266	5.4142	2.5459
0.18	1.9710	5.3663	2.7226	0.44	2.1323	5.4161	2.5400
0.19	1.9777	5.3682	2.7144	0.45	2.1380	5.4180	2.5341
0.20	1.9844	5.3701	2.7062	0.46	2.1436	5.4199	2.5284
0.21	1.9911	5.3721	2.6981	0.47	2.1492	5.4218	2.5227
0.22	1.9977	5.3740	2.6901	0.48	2.1548	5.4237	2.5170
0.23	2.0042	5.3759	2.6823	0.49	2.1604	5.4255	2.5113
0.24	2.0107	5.3778	2.6746	0.50	2.1659	5.4274	2.5058
0.25	2.0172	5.3798	2.6670				