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CHARACTERIZATION OF NEW PIEZOELECTRIC MATERIALS FOR SENSING APPLICATIONS

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A mis padres

TRABAJO FIN DE GRADO

“CHARACTERIZATION OF NEW PIEZOELECTRIC MATERIALS FOR SENSING APPLICATIONS”

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ABSTRACT

This research is conducted in order to characterize a new material for its use in pressure sensors. The material to analyze is the **PVDF**, a semicrystalline polymer source with excellent physical and electrical properties which is emerging as a good substitute for other piezoelectric materials used today in sensors and actuators for various applications.

For this purpose a **pressure sensor** is created in a metal flexible beam in which a force that will deform the material longitudinally is going to be exhorted. A **strain gauge** with the objective of monitoring the mechanical strain applied on the piezoelectric system and the response of the material will also be installed.

The obtained results show, on the one hand, the importance of the molecular structure of the material as this only generates electricity in a particular **molecular phase** and, on the other hand, the results highlight the good response to the applied stress, reaching in some cases a voltage generation of 3 volts, making the electrical response of PVDF one of the highest compared to other responses coming from **piezoelectric** ceramic materials such as PZT.

RESUMEN

Esta investigación se realiza con el objetivo de caracterizar un nuevo material para su aplicación en sensores de presión. El material que vamos a analizar es el **PVDF**, un polímero de origen semicristalino con excelentes propiedades físicas y eléctricas que lo perfilan como un buen sustituto de otros materiales piezoeléctricos que se utilizan hoy en día en sensores y actuadores para diversas aplicaciones.

Para ello se crea un **sensor de presión** en una viga flexible metálica donde después se ejercerá una fuerza que deformará longitudinalmente el material y unas **galgas extensiométricas** que sirven para monitorizar la deformación mecánica ejercida en el sistema y la respuesta piezoeléctrica del material.

Los resultados obtenidos muestran, por una parte, la importancia de la **estructura molecular** del material pues éste solo genera respuesta eléctrica en una de sus fases y, por otra, resaltan la buena respuesta ante la deformación aplicada, llegando a generar en algunos casos un voltaje de hasta 3V convirtiendo la respuesta eléctrica del PVDF en una de las mayores en comparación con otras respuestas de materiales **piezoeléctricos** de origen cerámico como el PZT.

CONTENTS

LIST OF FIGURES

LIST OF TABLES

I. CHAPTER ONE

i. Introduction to the characterization of piezoelectric materials

A couple of days ago on the TV news it was discussed how a father had created a prosthetic hand for his child using a 3D printer: the child was born with a malformation and therefore he had not developed the limb. A little research on the topic found that this is not the first case in which someone, using a 3D printer, is able to build a hand or a prosthetic arm. The price of these prostheses can reach in the American market is around 42,000 dollars; the price of a printed 3D prosthesis is between 20 to 300 euros [1]. But what if, in addition, that father could be possible to complement the 3D prosthesis for his kid with some kind of mechanism that imitates the functions of the receptors on the skin? What if some kind of sensor, flexible, lightweight, low cost and very thin was able to send an electrical signal when placed on the tip of a finger, detecting the pressure by it?

The tactile sensing operation is defined as the perception and recognition when there is a contact between surfaces in a deformable medium. The advanced system used by the human body takes place in the skin itself, a natural sensor that converts a mechanical strain or temperature into an electrical signal that reaches the brain. With the idea of imitating the behavior of human skin a sensor could be built with the purpose of reacting to different stimuli by sending electrical responses just as it does the skin of our fingers [2]. A sensor that is able to combine the properties of capacitive sensors - high sensitivity, low power consumption - and those characteristics of the piezoresistive sensors - mechanical stability - among others, could be a breakthrough in applications like robotic hand prosthesis or in clinical fields such as medicine where, for example, highly compact pressure sensor would allow development of an interventional catheter that includes multiple points of pressure measurement, including inflation pressure as well as blood pressure upstream and downstream [3].

The PVDF or polyvinylidene difluoride is a semi-crystalline thermoplastic material with good physical and electrical properties and whose study is progressing for its use in sensors, actuators or transducers in fields such as the mentioned above. Because it is a relatively new material is normal to find studies analyzing its phases or how to use it to increase their electrical properties in composite materials researches with carbon nanoparticles [4].

The objective of this study is, using the advantages of the PVDF material, creating a pressure sensor on a flexible beam, analyzing and characterizing the electrical properties possessed by the material in this class of designs.

ii. Motivation of the investigation about PVDF

This is the case of the Department of Materials Science and Engineering and Chemical Engineering at the Universidad Carlos III of Madrid. Recently, his research team has performed various experiments with piezoelectric materials and composite materials. The results achieved help us to understand the advantages of using this type of materials and open the doors to other investigations in the field.

Specifically, the material based on the study of the department of the Universidad Carlos III of Madrid is the PVDF: a thermoplastic material with high dielectric constant, k, easy to process and low cost, which is normally used as a sensor or actuator in various applications such as robotics and medicine. Thanks to its piezoelectricity, ferroelectricity and pyroelectricity properties and its high sensibility as well as its wide frequency response, it has become a popular sensing material against other piezoelectric materials frequently used [5]. For example, this material was used in piezoelectric sensors allowing detecting the damage in a blade of a wind turbine [6]; also PVDF sensors for biomedical applications are used on the inside wall of the grasper, the pressure generated by the endoscope grasper on tissues could be monitored [7]. The same idea was also applied in lithotrities surgery [8] and catheters [9]. The intensity of shock waves generated in the surgery was fed back by a PVDF shock-wave hydrophone to guarantee the destruction of stones and the safety of benign tissues [8], and pressure could be determined in real time flow measurements [9].

A simple research on Google enables us to realize that most studies are based on the creation of the material and its applications as sensors, transducers and actuators. This leaves a lack of information on their characterization, which is a difficult issue because the material is subject to many factors that modify its properties, from preparation to environmental conditions such as temperature.

The PVDF is a highly non-reactive and pure thermoplastic fluoropolymer produced by the Polymerization of vinylidene difluoride [10]. In the PVDF four phases can be identified: α, β, γ and δ [11, 12], where α and β are the most studied and used. The α phase is more stable while the β is showing greater piezoelectric response [13, 14]. When Kawai discovered in 1969 the piezoelectric properties of this material when studying the piezoelectric coefficient of the PVDF – from 6 to 7 pCN^{-1} – he observed that this value was 10 times bigger than other values obtained from any polymers. Since then this material has continued to be studied and used, evolving from inert lining or pipework material in chemical industries to nowadays where it can be found in advance and modern sensors and actuators where, due to its flexibility and relatively low cost, it has emerged as a useful option for MEMS.

This experiment proposes a study about properties and behavior as a pressure sensor of PVDF in a design similar to a Cantilever beam. A physical sensor is one that measures physical variables as force/load, pressure, temperature or acoustic variations and they can be based on different principles including electrostativity, piezoresistivity and piezoelectrivity. Moreover, physical sensors can be distinguished because of its applications therefore it is necessary to differentiate between the different most important characteristics whichever the application of the sensor will be. Some sensor features are sensitivity, linearity, responsivity, dynamic range, among others. For this reason the characterization of a sensor must include the study and definition of one or more of its characteristics parameters [15].

Within the fundamental principles of sensors, this analysis is going to focus on the piezoresistivity and piezoelectricity criterion. The piezoresistive principle is used when the strain gauges were added to the system because they, as it is going to be explain in the next chapters of this essay, are based on the piezoresistive effect; the strain gauges are placed on a cantilever beam, creating which can be considered as a pressure or force sensor as the main objective with its use is to monitored the beam deformation when a load is exhorted. The piezoelectric principle is going to be studied by means of the PVDF material, piezoelectric polymer that is going to be the central piece of the electric characterization of the pressure sensor designed.

iii. Aim of the experiment

As a polymer based, the PVDF is an excellent combination of physical properties of polymers as its mechanical properties where flexibility is highlighted, and the piezoelectric properties of the material, replacing other piezoelectric ceramic materials such as PZT in various applications as, for example, pressure sensors.

The above mentioned Department of Materials Science and Engineering and Chemical Engineering at the Universidad Carlos III of Madrid, in the development of their studies about PVDF material and composite materials, performed the following experiment: circular samples are placed in between two circular aluminum electrodes of 10mm of diameter, supported by a structure made of two thick plates of Poly (methyl methacrylate). The entire system is connected to the measurement equipment using wires soldered to plates touching the stems of electrodes. The system is then closed with a pair of wing nuts to prevent the PVDF samples to move, avoiding measurement errors [16].

The piezoelectric characterization of the samples was performed measuring the electrical response of the material when a mechanical force was applied by the impact of a stainless steel cylinder of 2 mm of diameter and 2 mm height. The electric response of the material was measured with an oscilloscope without any further conditioning of the signal [16].

With the obtained results, it was proposed to the Department of Electronics Technology at Universidad Carlos III of Madrid the idea of using the PVDF material to characterize it as a pressure sensor.

<u>General objective</u>: creating a piezoelectric pressure sensor using pure PVDF material manufactured in the Department of Materials Science and Engineering and Chemical

Engineering at the Universidad Carlos III of Madrid, in a homemade installation created specifically for the characterization of the sensor.

Specific objectives:

II. DC analysis: creating a weight assembly, a conditioning circuit with the aim of amplifies the signal obtained from the piezoelectric material, and a strain gauge conditioning circuit, used to compare the response of the PVDF piezoelectric pressure sensor.

III. AC analysis: studying the response of the material to a frequency variation stimulus, covering a wide frequency range from low to high frequency or ultrasound, and using a Lock-in Amplifier when necessary to filter the signal obtained from the sensor.

IV. Studying the results obtained in both DC and AC analysis, testing the suitability of the material and the system as a pressure sensor by comparing the results obtained with other similar experiments. In addition, performing again the first experiment consistent of the original assembly, checking that the material response is similar in both experiments and that it is possible to conclude that the material response, in similar situations of mechanical deformation and ambient conditions, is always the same therefore lineal.

II. CHAPTER TWO

i. Deformation measurements: strain gauges

The structure designed in this analysis is similar to a cantilever beam: a flexing structural element attached on one side and subjected to lateral loads. When a force is exerted on a beam, stresses and strains are created in the material. The load, either if it is concentrated in one point or distributed through the length of a region, causes the beam to flex making the beam to deform its straight axis into a curve: when the beam is being deformed, assuming that its cross section remains the same, the lines in the lower part of the beam are elongated while the lines in the upper part are shortened, being this phenomenon called tension and compression respectively [17].

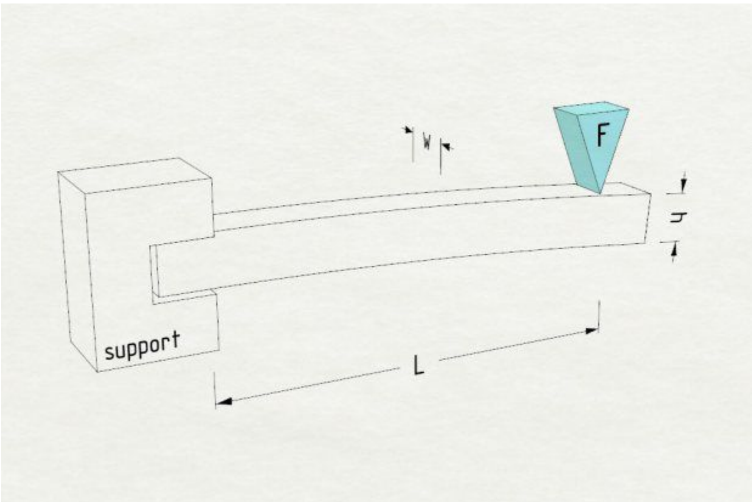


Figure 1: Example of Cantilever beam when a force is applied

Whenever a flexible beam is deformed, either with tension or with compression, there is a change in length along the longitudinal axis due to the mechanical stress performed by the load. The variation in length is a useful effect used frequently in microelectromechanical systems, specifically in piezoresistive sensors.

The piezoresistive effect, firstly discovered in 1856 by Lord Kelvin, is a widely used sensor principle: a change in an electrical resistor is produced when this is under a mechanical deformation or strain [18]. This effect provides an easy and direct relation between the mechanical force and the electrical domain and is widely used in applications as accelerometers, pressure sensors [19], tactile sensors [20] and chemical/biological sensors [21].

Therefore, by definition, a piezoresistor is a resistor whose resistivity changes with an applied strain. Metal resistors change their resistance in response to strain because of the shaping deformation mechanism, being this the case of strain gauges. The resistance of a resistor is usually measured along its longitudinal axis although the strain can have two different components, longitudinal and transverse: the longitudinal gauge factor is the relative change in resistance measured due to the longitudinal strain while the transverse gauge factor exists when the change in resistance is produced by the transversal component of the strain [21].

Strain gauges do generally have the form of a metal-clad plastic patches that can be adhered to the surface where the mechanical strain is going to be produced. It usually consists of a very thin wire arranged in a grid pattern with the objective of maximizing the amount of metallic wire subject to strain in the parallel direction and to minimize the effect of the strain components in other directions. The grid is bonded to a thin support called carrier that is fixed directly to the surface so that the mechanical deformation is transferred directly to the gauge that will be able to respond with a linear electrical resistance change [22]. The basis for choosing the most suitable strain gauge for our system is to take into account some characteristics as the long-term stability, the range of temperature where the gauge can operate correctly, the tolerable amount of elongation, among others [21]; although the most fundamental parameter of the strain gauge is its sensitivity to strain also called Gauge Factor, defined as the ratio of fractional change in electrical resistance to the fractional change in length, expressed as:

$$GF = \frac{\Delta R/R}{\Delta L/L} = \frac{\Delta R/R}{\varepsilon}$$

Due to the small change in resistance usually produced by the mechanical strain, there is a need of using a configuration with a voltage excitation source: resistance changes are often read using a Wheatstone bridge configuration consisting of four resistive arms connected in a loop with an excitation voltage applied across the bridge.

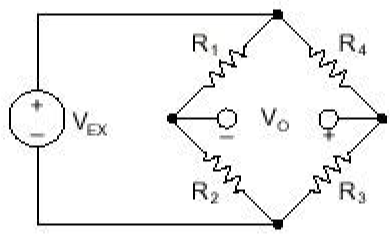


Figure 2: Example of a Wheatstone bridge

The output voltage of the system is defined according to the following relationship:

$$V_o = \left[\frac{R_3}{R_3 + R_4} - \frac{R_2}{R_1 + R_2}\right] \cdot V_{EX}$$

The bridge is balanced when the output voltage is equal to zero, hence, when R_1/R_2 is equal to R_4/R_3. Any change in the value of the resistances composing the bridge will lead to a nonzero output voltage. Theoretically the resistances have an ideal nominal value – usually from 30 to 3000 Ω- but in reality these ideal nominal value have a resistance variable part; the existence of a potentiometer that equilibrates the variable part of the resistances from the bridge is needed.

The frequency response of the strain gauges depends on the mechanical parts of the properties of the structure to which they are bonded but, generally, spans from DC through audio frequency range [23].

ii. Piezoelectricity

History of piezoelectricity and main characteristics

Piezoelectricity was discovered in the latest decades of the nineteenth century, when it was observed that certain materials were able to generate voltage when they were subjected to mechanical stress. Likewise, these materials that were able to produce an electrical response when they were deformed were also mechanically deformed when an electric field was applied to them.

In 1880 the brothers Pierre and Jacques Curie executed the first experiments with piezoelectric materials such as quartz and Rochelle salt. A year later, in 1881, Hermann Hankel coined the term "piezoelectricity" from the Greek word "*piezen*" which means "to press". William Thomson, in 1893, characterized mathematically and then proved experimentally, that a material which exhibits what is known as direct piezoelectric effect - a material generates an electric charge when under mechanical stress - also show the inverse effect - the material under an electric field is deformed mechanically - [24].

The first application of a piezoelectric device occurred in France in 1917, during the First World War: an ultrasonic submarine detector consisting of a transducer made of quartz crystals [25].

Many features of piezoelectric materials come from their crystal structures: of the 32 kinds of crystal classes only 20 have piezoelectric properties; ten of them represent the polar crystal classes [26] which have a spontaneous polarization without mechanical deformation due to a non-vanishing electric dipole moment associated with their unit cell that exhibit pyroelectricity [25]. A crystal can be made piezoelectric by poling, which includes the application of an electric field at an elevated temperature. When the electric field is removed the material dipoles remain aligned, giving the substance a permanent polarization and deformation due to the elongation of the material in the direction of the magnetic field [24]. In this case, it is said that the material is ferroelectric [25].

The polarization of the material is usually the last step while processing the crystal since this could be depolarized and therefore lose its piezoelectric effect. Triggers of the depolarization can be: mechanically (this occurs when a high mechanical stress is exerted destroying the orientation of the dipoles), electrically (when the material is subjected to a static electric field high up to 200 or 500 V / mm) or thermally (the material is heated to such a high temperature that the vibration of the crystal makes its molecules disorder). The material can also be degraded over time and thus lose their ability to generate electricity [24].

Mathematical description of piezoelectric effect

Direct piezoelectricity occurs by the displacement of ionic charges in the crystal structure. These charges, that generate an electric field when in movement, are produced when a stress is applied to the crystal changing the spacing between centers of positive and negative charges in each cell domain. Conversely piezoelectricity occurs when an electrical field applies a force between the centers of the positive and negative charges, also producing an elastic strain and a change of dimensions [27].

Thus, the relationship between polarization (D) and mechanical stress (T) can be described as:

$$D = d \cdot T + \varepsilon \cdot E$$

Where "d" is the piezoelectric coefficient matrix, epsilon is the dielectric permittivity and "E" is the field electric. If "E" is zero then the second term of the equation could be eliminated. The general constitutive equation can be written as:

$$\begin{bmatrix} D_1 \\ D_2 \\ D_3 \end{bmatrix} = \begin{bmatrix} d_{11} & \cdots & d_{16} \\ \vdots & \ddots & \vdots \\ d_{31} & \cdots & d_{36} \end{bmatrix} \cdot \begin{bmatrix} T_1 \\ T_2 \\ \vdots \\ T_6 \end{bmatrix} + \begin{bmatrix} \varepsilon_{11} & \cdots & \varepsilon_{13} \\ \vdots & \ddots & \vdots \\ \varepsilon_{31} & \cdots & \varepsilon_{33} \end{bmatrix} \cdot \begin{bmatrix} E_1 \\ E_2 \\ E_3 \end{bmatrix}$$

Here, the terms T1, T2 and T3 represent the normal stresses along the axes whereas T4, T5 and T6 are the shear stresses. Units of d_{ij} can be obtained as follows:

$$[d_{ij}] = \frac{[D]}{[T]} = \frac{[E] \cdot [\varepsilon]}{[T]} = \frac{\left(\frac{F}{m}\right) \cdot \left(\frac{V}{m}\right)}{\left(\frac{N}{m^2}\right)} = \frac{C}{N}$$

The inverse effect of piezoelectricity can be described with the following expression:

$$S = s \cdot T + d \cdot E$$

$$\begin{bmatrix} S_1 \\ \vdots \\ S_6 \end{bmatrix} = \begin{bmatrix} S_{11} & \cdots & S_{16} \\ \vdots & \ddots & \vdots \\ S_{61} & \cdots & S_{66} \end{bmatrix} \cdot \begin{bmatrix} T_1 \\ \vdots \\ T_6 \end{bmatrix} + \begin{bmatrix} d_{11} & \cdots & d_{31} \\ \vdots & \ddots & \vdots \\ d_{31} & \cdots & d_{33} \end{bmatrix} \cdot \begin{bmatrix} E_1 \\ E_2 \\ E_3 \end{bmatrix}$$

Here we can see how the total strain is related to "E" and the mechanical stress "T". If there is no mechanical stress then is related to the strain E as follows:

$$\begin{bmatrix} S_1 \\ \vdots \\ S_6 \end{bmatrix} = \begin{bmatrix} d_{11} & \cdots & d_{31} \\ \vdots & \ddots & \vdots \\ d_{31} & \cdots & d_{33} \end{bmatrix} \cdot \begin{bmatrix} E_1 \\ E_2 \\ E_3 \end{bmatrix}$$

It should be taken into account that the d_{ij} relating the strain component with the applied voltage is equal in the direct and indirect effect.

Among other applications piezoelectric sensors, actuators and transducers can be found in microphones, phonograph cartridges to translate the grooves in vinyl phonograph records into sound, measurement of minute pulsations on body surfaces, measurement of surface roughness, measurement of acceleration [28], pumps and valves for liquid and particles ,and mirrors or chemical sensors [27].

iii. PVDF

Polyvinylidene fluoride or polyvinylidene difluoride (PVDF) is a highly non-reactive and pure thermoplastic semi-crystalline polymer produced by the Polymerization of vinylidene difluoride. The PVDF comes from the fluoropolymer family and among its main features is its proper piezoelectric and pyroelectric behavior [16, 29 and 30].

This semi-crystalline polymer shows a complex molecular structure and these properties are highly dependent on its polymorphism [16]. The characterization of the crystalline structure of PVDF using X-ray diffraction techniques has been well documented and four polymorphs have been identified: α-phase, β, γ and δ are its most common crystalline phases, sometimes referred to as phases II, I, III and IV, respectively based on the order in which they were discovered [31].

Specifically the α phase, with no piezoelectric response, is the lowest energy conformation and is formed when the polymer is crystallized after melting. The space-filling model is based on a distorted trans-gauche-trans-gauche' (TGTG') conformation [31]. In this phase the molecular chains are places two by two but in opposite ways, so the dipole moments of the two molecular chains have opposite signs and therefore cancel each other, making the unit cell actually non polar [16]. The crystalline phase of interest for its ferroelectricity properties is the polar β phase [31]. In this phase, the space-filling model has a (TTTT) conformation, resulting in a net dipole moment which produces the highest electroactive properties. Phase γ has a conformational structure defined as (TTTGTTTG ') [29] and is considered the intermediate stage between α and β phases.

Many of the interesting properties of PVDF, particularly related to its use as a sensor and actuator are associated with strong electric dipole moment of the monomer unit of PVDF caused by the high electronegativity of fluorine as compared with those of the hydrogen and carbon atoms [25, 26]. Because of its stability in a thermodynamic point of view, α phase is the dominant phase. However, the β phase is the most suitable for this study because it is the one that has higher piezoelectric and pyroelectric properties [16, 24 and 32].

Thin-stretched PVDF films are flexible and easy to handle as ultrasonic transducers. The material is carbon based, and it is usually deposited as a spin cast film from a dilute solution in which the PVDF powder has been dissolved [30]. As with most piezoelectric materials, process steps after deposition greatly affect the behavior of the film. For example heating, mechanical constraint (stretching) and high electric poling fields [28] can modify the polarization of the film, increasing or decreasing the piezoelectric effect of the PVDF. The d matrix of PVDF is the following [30]:

$$d = \begin{bmatrix} 0 & 0 & 0 & 0 & <1 & 0 \\ 0 & 0 & 0 & <1 & 0 & 0 \\ 20 & 2 & -30 & 0 & 0 & 0 \end{bmatrix} pC/N$$

In recent years, research and study fields such as biology and medicine as well as more technical fields like aerospace engineering have begun to focus on the study of the properties of PVDF and other polymers to design a new generation of sensors and actuators that are lighter, more flexible and easier to handle [17, 18, 21 and 28]. For instance, we find examples of its use in robotics domains where actuators are using PVDF films for tactile displays [21,19] and micromanipulation [18,20]; biomedical applications and specifically surgical purposes where sensors are places on the inside wall of the grasper so the pressure generated by the endoscope grasper on tissues could be monitored [33]. The same idea was also applied in lithotrities surgery [32] and catheters [9]. Further example of the use of PVDF actuators will be scanning systems as laser beam scanners or laser beam steering in optics [22, 23]. Another potential field recently studied is in the energy harvesting domain due to the electromechanical coupling and mechanical strength of the PVDF [34, 35].

III. CHAPTER THREE

i. Introduction to the experiment

The aim of the experiment is to characterize a piezoelectric material called PVDF for sensing applications. The polyvinylidenfluoride or PVDF is a synthetic fluoropolymer with monomer chains of (-CH2 –CF2-)$_n$. It exhibits piezoelectric, pyroelectric and ferroelectric properties; excellent stability to chemicals; mechanical flexibility and biocompatibility are other of its main characteristics [36]. Due to the high sensitivity that PVDF presents, its easy fabrication and other properties as its light weight, it has been developed lately for its use in sensing applications [37].

Therefore, because of its piezoelectric property, the material must be subjected to a mechanical effort that creates a deformation with respect the axis of the material to be able to generate electric charge. In this case the mechanical stress is exerted, using a cantilever beam mounting, with weights or with a speaker that will transfer a modulation in frequency. In addition, the electric charge of the material as a function of the deformation will be measured and monitored using a piezoresistive device.

In order to study the reaction of the material to different stimuli, two areas of study were created: DC analysis and frequency modulation analysis. The following sections describe in detail the setup followed for each experiment.

ii. DC Analysis

1. Introduction to DC experiment

Some types of piezoelectric materials can serve, among other applications, as mechanical input transducers: measuring pressure, force, displacement and other physical phenomena which can be related to them. These piezoelectric materials, when mechanically strained in a preferred way, generate an open circuit EMF. Indeed, the mechanical strain causes an unidirectional separation of electric charges that component the interior of the piezoelectric crystal structure. These displaced charges form an effective net charge on a capacitor formed by the join of metallic electrodes on the surface of the piezoelectric crystal.

The transducer material itself, independent of its piezoelectric activity, is theoretically an insulator with a very low conductivity and a high dielectric constant. Although, in practice, the situation is not that simple: the piezoelectric transducer material is not a perfect dielectric and the charges leak off through the exceedingly small volume conductance. Of course, any voltage measuring system attached to the transducer will have finite input impedance and perhaps a DC bias current, altering the stress-caused EMF.

In addition, the transducer has mechanical mass, stiffness, and damping, giving it mechanical resonance properties which can further complicate the transfer function of the transducer when the frequency of the mechanical input approaches the mechanical resonance frequency of the transducer [38]. In the discussion below, it is assumed that the piezoelectric transducers are operated at frequencies below their mechanical resonant frequencies.

For mechanical input frequencies below the own piezoelectric transducer mechanical resonance, the equivalent circuit shown in Figure 3 can be used to model the electrical behavior of the material [38]:

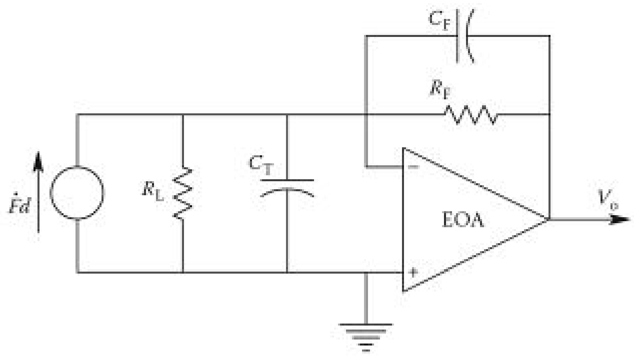


Figure 3: Equivalent circuit for piezoelectric transducers

Although charge is displaced internally by strain, it is necessary to use a current source in the electrical model. Since current is the variation of flow of charge, the equivalent current source is:

$$i_{eq} = \frac{\delta Q}{\delta t} = \delta\left(\frac{\delta F}{\delta t}\right) = \delta\dot{F}$$

Applying Ohm's law, the open-circuit voltage across the transducer is given by the following transfer function that seems to belong to a simple high-pass filter:

$$\frac{V_o}{F}(s) = \frac{s \cdot F \cdot d \cdot R \cdot L}{1 + \left(\frac{sCT}{GL}\right)}$$

2. <u>Brief description of the assembly</u>

In order to examine the behavior of the material in a DC environment, two different conditioning circuits were designed. The first one, consisting of an instrumentational AD620 amplifier, is a simple circuit that amplifies the electrical signal producing by the PVDF when this is subjected to mechanical deformation. The second design is based on a circuit with a charge amplifier or operational amplifier, TL081.

In both circuits a pre-assembly consisting of a wooden frame where a metal plate is placed is constructed. In the metal plate the PVDF sample and a strain gauge are attached, this last one with the objective of monitoring the material deformation. Finally the mechanical deformation is performed using a set of weights hanging from the metal plate.

The following figure shows the final setup where it can be observed some of the components of the assembly:

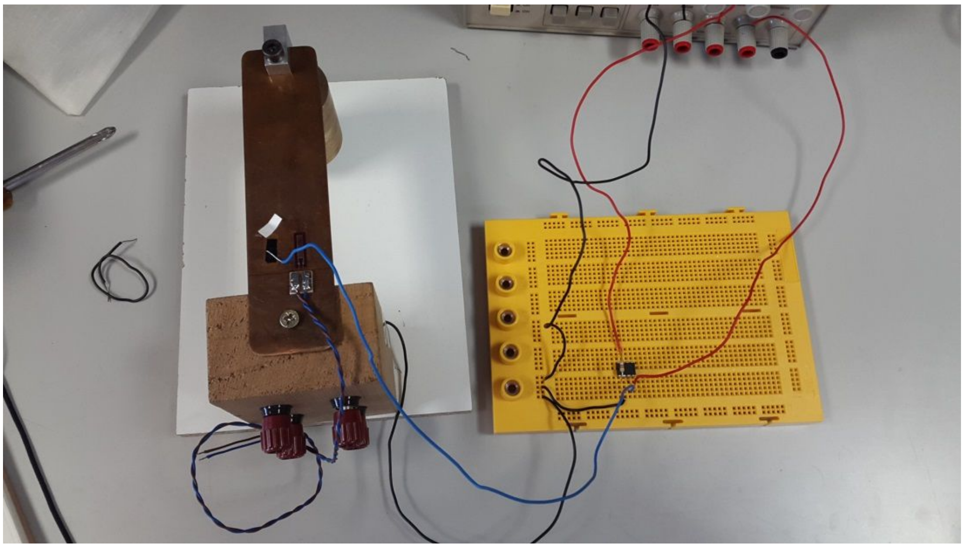


Figure 4: Wood assembly with metal plate, AD620 circuit and set of weights

3. Components of the assembly

Below it is presented a description of the process followed for the design and assembly of all the components in the system. Some of the following components were provided by the Universidad Carlos III of Madrid such as the wooden assembly, loaned from the Department of Electronics Technology or the PVDF samples kindly provided by the Department of Materials Science and Engineering and Chemical Engineering; while others were made in a workshop as the metal plates.

Metal plates

To perform the experiment, four types of metallic plates were used: copper, brass, steel and aluminum. Thus various responses from the PVDF material can be obtained according to the deformation that the metal plate undergoes by exerting a force on it, or as the conductivity and electrical resistance of the material itself.

For the preparation of the metal plates the next procedure was followed:

i) The metal was cut into rectangles plates of dimensions 15 x 3.5 cm with an Electric Metal Shears 18 Gauge from Eastwood Company. Then the plates were sanded to eliminate any imperfection that the material surface may have with a Black + Decker BDER0100 5 in Random Orbit Sander Disc.

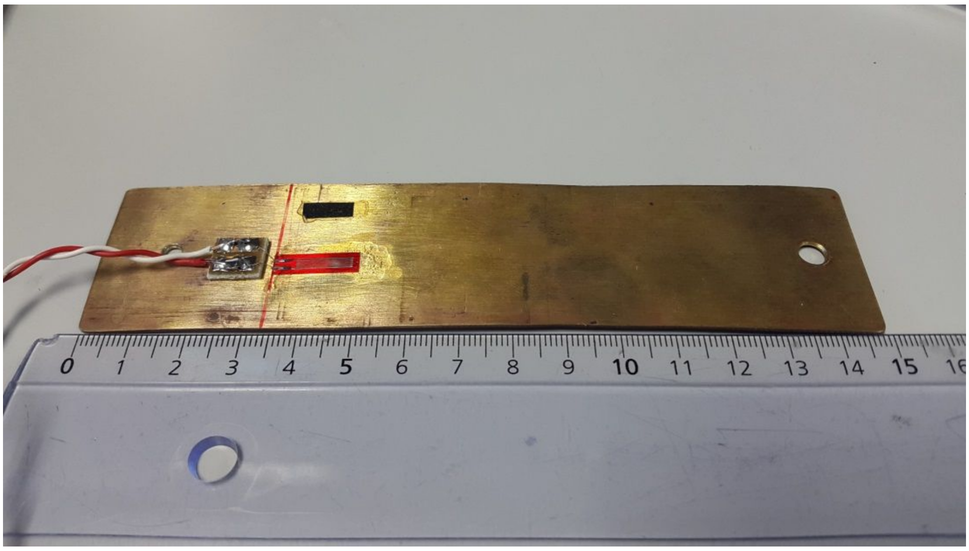
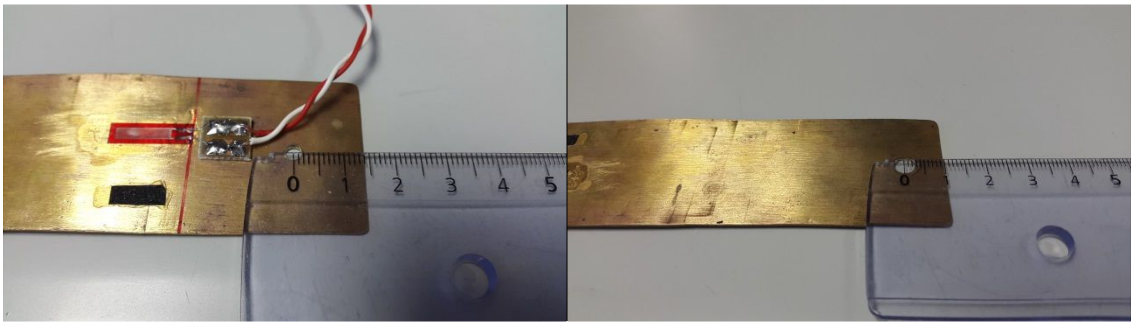


Figure 5: Brass metal plate with dimensions, PVDF sample and strain gauge installed

ii) In the already cut and sanded metal plates two points were marked with a metallic punch. These points mark the place where the two holes must be drilled: the first hole of 0.4 cm of diameter and the second hole of 0.6 cm of diameter. The holes were made with a 120-Volt ½ in Corded Hammer Drill model HD19-2B by Bosch.



Figures 6 and 7: Dimensions of the holes drilled in the metal plates

iii) Once the holes were drilled, the plates were sanded with an electric sander to remove the excess material in the holes. Furthermore, the plates were pressed to ensure a flat surface for the PVDF sample.

Material

The Polyvinylidene fluoride (PVDF) material used was supplied by Sigma-Aldrich (Mn ~ 10,700; Mw ~ 27,500 g/mol and density 1.78 g/cm3) in samples of 100% PVDF composition.

PVDF films were prepared following several processing steps:

i) PVDF pellets were ground using a MF 10 basic Microfine grinder. About 15 g of pellet were immersed in liquid nitrogen for 15 minutes and subsequently milled at 3000 rpm for 5 minutes to obtain a kind of flakes.

ii) A hot plate press Fontijne Presses TPB374 was used to process the powders in film, with thickness obtained around 100 μm. The powders were placed in two polished aluminum plates of 10 x 10 cm, coated with Kapton sheets. Then they were heated to 200 ° C and pressed by applying a load of 50 kN.

Figure 8: Samples of PVDF material

iii) The circular samples of about 15 mm of diameter were cut into rectangular shape samples with dimensions of 5 x 10 mm and then placed in the metal plates at 2 cm distance from the holding hole [16].

On behalf of the PVDF material to adhere to the metal is necessary to follow this procedure:

i) The PVDF material, previously located in its proper place in the metal plate, has to be heated in a JP Selecta 2005163 - Digitronic-TFT oven up to 200 ° C. This process takes approximately one hour. Then, the metal plate with the already adhered material must be cooled to reach 50° C to be removed from the oven.

ii) A double side conductive tape Nisshin-Em is placed above the PVDF material. The dimensions of the conductive double-sided tape must be slightly smaller than those of the material so there is no contact between metal and conductive tape which would produce contact between two conductors and thus cause no voltage effect from the PVDF.

Strain Gauges

A strain gauge is a type of piezoresistive sensor used to measure strain, pressure, force or weight based on the piezoresistive effect, which is a property of certain materials to change the nominal value of its resistance when being deformed due to stress in the direction of its mechanical axes. The deformation in the strain gauge produces a variation in its electrical resistance: this variation may be produced by a change in length, a change in the section or a change generated in resistivity [39].

The strain exhorted on a surface is caused by an external influence usually motivated by forces, pressures, moments, heat or structural changes of the material. If certain conditions are fulfilled, the value of the force performed can be derived from the measured strain value. In experimental stress analysis this feature is widely used: the strain values measured on a surface are used to state the stress that the material is suffering which will be an useful property, for example, safety analysis [40].

For the next experiment a gauge resistance is used: this type of gauge is an electrical conductor that increases its resistance as being deformed. By the use of a Wheatstone bridge, it's possible to convert this variation of resistance in voltage variation, linearly related by a factor called gauge factor.

The strain gauges from Tokyo Sokki Kenkyujo Co. Ltd model PF Gauge-Series were placed at 2 cm approximately from the top hole. This gauges have a nominal value of 120 Ω and they have a temperature range of operation from -20 to 80 degrees; also this gauge has a pre-attached vinyl lead wire therefore soldering are not required and its dimensions of 10 mm of length make them suitable for the experiment [41]. The installation procedure of the strain gauges on metal plates is required a number of distinct steps that must be followed to obtain the desired result [42]:

i) First degrease the surface where the gauge is going to be installed using a small amount of isopropyl alcohol and a KIMTECH SCIENCE KIMWIPES Delicate Task Wipers. Avoid touching the already cleaned surface for the rest of the steps.

ii) Apply one or two drops of M-prep Conditioner A to the cleaned surface and sand it to remove the paint and any other imperfection.

iii) Again, use a Kim Wipe to remove any particles left after the sanding and keep cleaning the surface until no imperfection can be seen.

iv) Wet the exposed area with one or two drops of M-prep Conditioner A and scrub the area with a Kim Wipe. After this, use a fresh wipe and clean the surface.

v) Imprint with one or two drops of M-prep Neutralizer 5A the exposed area and cleanse the area with a Kim Wipe.

vi) If it is required, draw a pencil line on the exterior to proper alignment of the strain gauge in the following steps.

vii) Dispose the gauge and strain relief terminals bonding side down on a clean surface, leaving a 1.5mm gap between them.

viii) Place 100mm of M-LINE PCT-2A Cellophane tape over the clean surface and anchor one end of the tape to the slide, aligning the gauge in the center of the tape. Then, arrange the gauge in the appropriate location and anchor one side of the tape to the desired surface. Wipe the tape down to place the gauge and reposition it if it is needed.

ix) Raise the tape from the end opposite the strain relief terminals till the gauge and terminals are clear of the surface, fold the tape under and tack it behind the gauge. The area where the gauge is to be bonded should now be clear.

x) Apply M-bond catalyst sparingly in a thin and uniform coat following the next indications:

   a. Wipe the catalyst brush on the bottle 10 times to remove excessive product.

   b. Brush the surface with single strokes, covering the entire length of the gauge.

   c. Wait for at least a minute until the catalyst is dry.

xi) Attach the gauge to the desired area. Take into account that this step must be done within 5 seconds if the gauge is to properly bond to the surface, consequently:

   a. Un-tack the end of the tape farthest from the bonding place.

   b. Apply one drop of M-bond 200 Adhesive to the join of the tape and the area nearest the boding site, about 13mm.

   c. Rotate the tape towards the bonding place until it forms an angle of 30° with respect the surface. This way the gauge will be above the desired position but not in contact with the surface.

   d. Use a clean gauze sponge to bond the gauge to the surface with a single movement, starting from the terminal end to the un-tacked end and quickly apply firm thumb pressure directly over the gauge. Maintain the pressure for at least 1 minute considering that the warmth of the thumb helps the adhesive to set correctly. Then wait two more minutes for the adhesive to fully dry. When it is completely dried, remove the tape from the gauge carefully.

   e. If in the previous step, the adhesive leaks from the sides of the tape, remove them with a clean wipe.

xii) In the event of attaching the lead wire, the most important factor is to prevent overheating the gauge as this will melt the gauge and therefore be useless.

   a. Mask the strain gauge with tape, leaving only the solder tabs exposed.

b. Clean the tip of the soldering iron with a wet sponge pad.

c. Apply the iron tip onto the solder and use a firm pressure for no more than one second. Remove both the solder and the iron simultaneously.

d. Repeat the above procedure until a nice and even mound of solder is created on each solder pad and each strain relief terminal.

e. Bend the ends of the wire so there is a small bend between the strain relief and the gauge when the wire is soldered to the gauge. Press the soldering iron to the wire over a pad while feeding a small amount of solder between the iron and the wire. The wire should slide into the melting solder. Cool before handling.

f. Repeat for every solder tab and strain relief terminals.

g. Eliminate any leftover from the solder with a gauze sponge soaked in rosin solvent and use a dabbing action to prevent the gauge to be damage.

xiii) Last, apply a coating of M-Coat A over the entire gauge and terminal area.

Once the gauges are attached to the metal plate, their conditioning design is implemented, consisting mainly of a Wheatstone bridge: an electrical circuit used to measure an unknown electrical resistance by balancing two legs of a bridge circuit, one leg of which includes the unknown component. The primary benefit of a Wheatstone bridge is its ability to provide extremely accurate measurements [43].

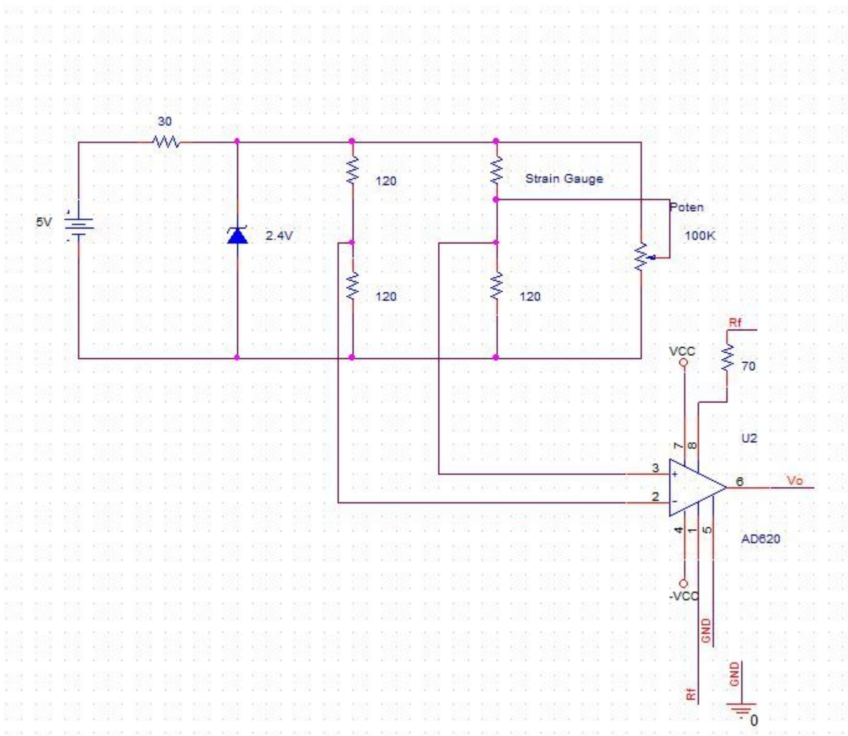


Figure 9: Orcad design of Wheatstone bridge conditioning circuit

As it can be seen in the picture above, the 5 volts supply is divided into a fixed voltage drop in the Zener diode –which has a value of 3.7 V– and the voltage drop in the resistance of 30 Ω, adjusting the value of the electric current through the bridge. The Wheatstone bridge is formed ideally by four resistors whose nominal value is maintained, so that ratio of the two resistances R_2 / R_1 is equal to the ratio of R_4 / R_3, obtaining a value of 0 volts between the midpoints of the bridge [43].

However, the value of the three resistors forming the bridge cannot be considered ideal and fixed; similarly the value of the strain gauge is considered variable in function of the deformation to which the plate is subjected. Therefore the presence of a potentiometer between the strain gauge and the resistor R_4 is required and the first step to prepare the analog conditioning circuit of the strain gauges is to set the value of this potentiometer. For that purpose the voltage difference between the midpoints of the bridge must be measured, moving the position of the potentiometer until a value of 0 volts is obtained. At that point the variation in voltage at points A and B can be characterize depending on the change in the deformation of the plate. In order to facilitate the measurement of voltage between these points an instrumentational AD620 amplifier is placed, with a gain of 706 V / V achieved by using a resistance of 70 Ω value.

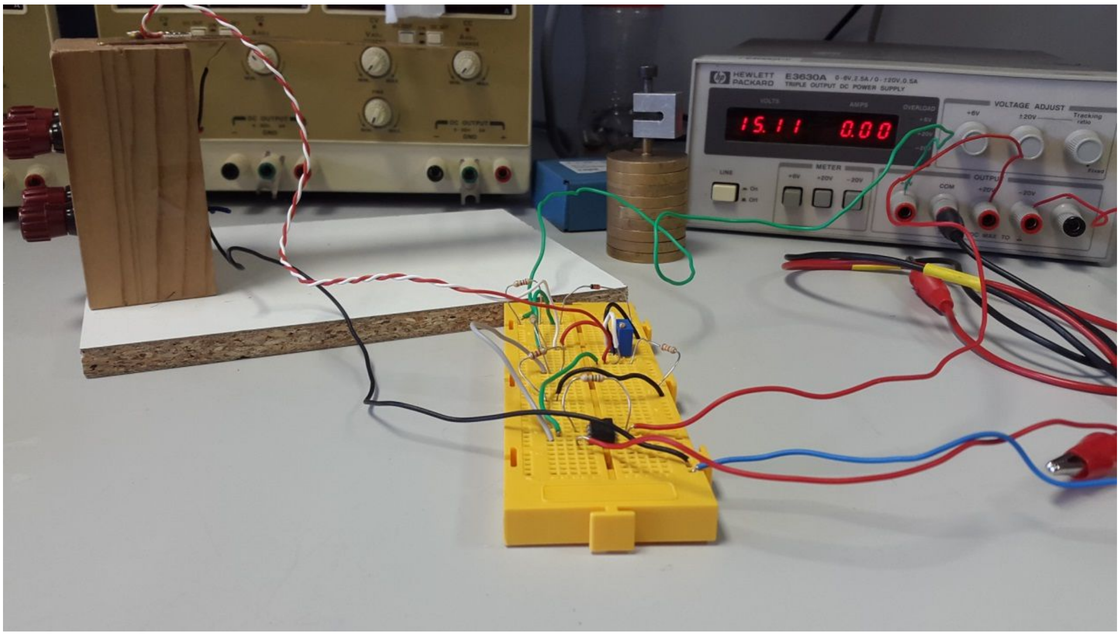


Figure 10: Image of strain gauge conditioning circuit when the assembly is already connected

All the measurements taken during the DC and AC experiments were taken using a 506 Digital Multimeter provided by Protek. Among its main features it can be highlighted its dual display for frequency, AC voltage and temperature measurements; the 10 MHz frequency counter and decibel measurements and the true RMS AC voltage measurements [44].

Signal conditioning circuit

i. Conditioning circuit based on an instrumentational amplifier AD620

Usually the voltage signal response obtained from these piezoelectric materials when under deformation, is on the range of microvolts. These values, although could be seen in an oscilloscope, can also be confused with noise; for this reason the desired value measured is around 3 to 5 volts. The key to achieve this range of voltage would be, of course, to amplify the signal with the suitable amplifier. Some amplifiers may also involve linear filtering in the frequency domain, such as band-pass filtering to improve signal-to-noise ratio at the amplifier's output [45].

By definition, instrumentation amplifiers are described as direct-coupled, low-noise, differential amplifiers with a very high input impedance, high common-mode rejection ratio and a user settable gain from 0 to 60 dB, and bandwidth to the hundreds of KHz [46]. Despite the fact that the instrumentation amplifier is represented identically to an operation amplifier, this is composed by three operation amplifiers: two arranged so that there is one op-amp to buffer each input and the third one to produce the desired output, as can be seen in the figure below [47].

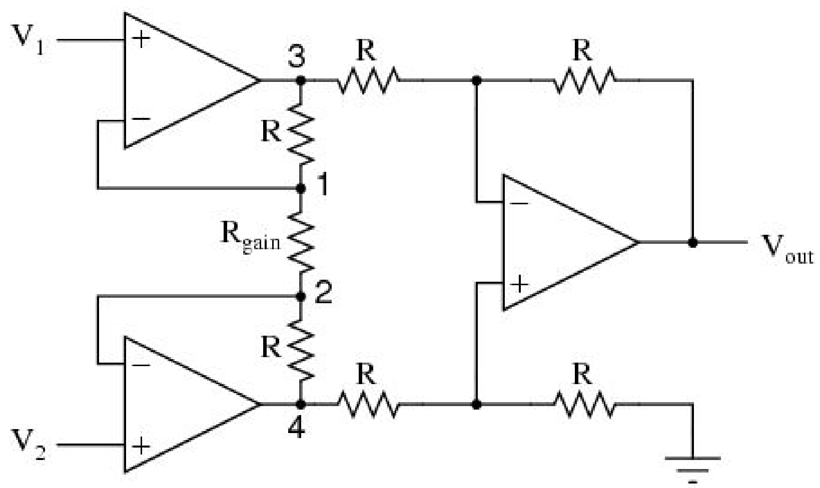


Figure 11: Schematic of an Instrumentation Amplifier

Therefore the gain of these types of amplifiers is defined as [70]:

$$V_{out} = (V_2 - V_1) \cdot \left(1 + \frac{2 \cdot R}{R_{gain}}\right)$$

For signal conditioning in this part of the experiment it is decided to use an AD620 amplifier. Said amplifier, proportionated by the company Analog Devices, is a low cost and high accuracy- 40 ppm maximum nonlinearity- instrumentation amplifier. Other characteristics of the AD620 are its low noise – around 9 nV/√Hz at 1 kHz and 0.28 µV p-p in the 0.1 Hz to 10 Hz band – and low input bias current – of about 1.0 nA – [48]. All these qualities make the instrumentation amplifier AD620 a suitable candidate for, among other applications, weigh scales, transducers interface or data acquisition systems.

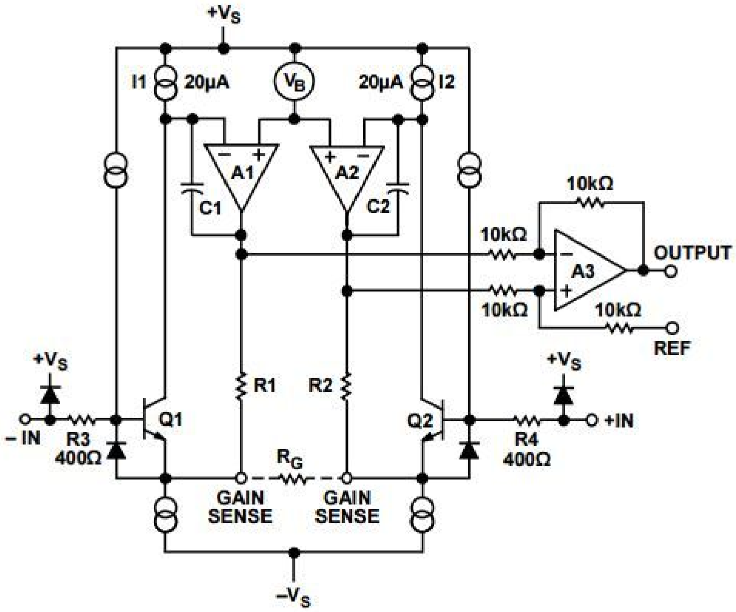


Figure 12: Schematic of an AD620 amplifier

Another quality why the AD620 model was chosen for the experiment is the ease with which the amplifier's gain can be modulated with only a resistor. This is due to a modification of the classic three op amp approach: in this amplifier the input transistors Q1 and Q2 provide a single differential pair bipolar input for high precision while the feedback through the Q_1-A_1-R_1 loop and the Q_2-A_2-R_2 loop maintains constant collector current of the input devices Q_1 and Q_2, thereby impressing the input voltage across the external gain setting resistor RG. This creates a differential gain from the inputs to the A_1/A_2 outputs given by $G = (R_1 + R_2)/R_G + 1$. The internal gain resistors, R_1 and R_2, are fixed to an absolute value of 24.7 kΩ [48], allowing the gain to be entirely set with an external resistor. Accordingly the gain of the AD620 amplifier can be defined as:

$$G_{AD620} = 1 + \frac{49{,}4 \cdot 10^3}{R_F}$$

Towards obtaining the optimal values of voltage desired – from around 3 to 5 volts – the following circuit is designed:

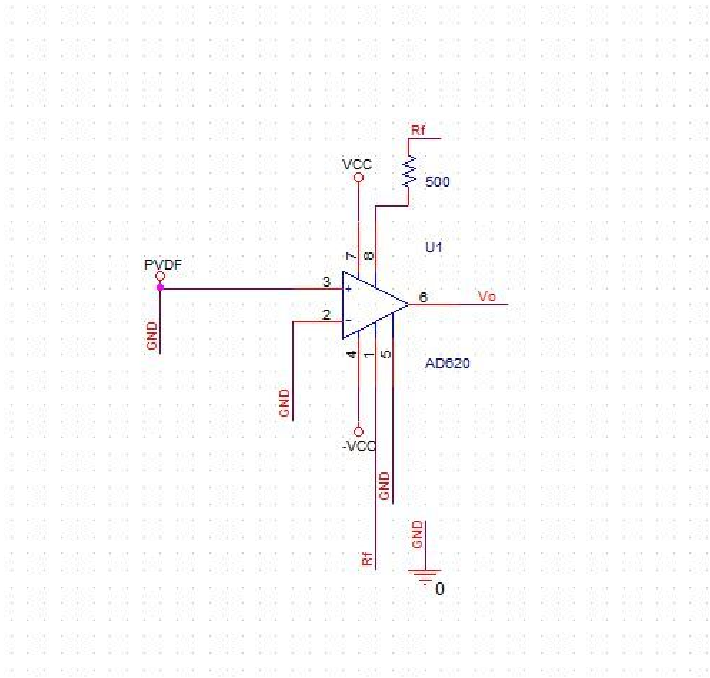


Figure 13: Orcad design of AD620 instrumental amplifier analog conditioning circuit

As it can be seen, it is a very simple analog conditioning circuit where the gain of the amplifier is used in a voltage mode amplifier. In a voltage mode amplifier, the output depends on the amount of capacitance seen by the sensor. Although the capacitance associated with the interface cable could affect the output voltage which could cause variations in the measurements taken [49], this type of configuration is used when the amplifier is very close to the sensor, as it is the case, therefore no variation in the capacitance should be measured.

The gain of the AD620 is set to 100V/V and, as it can be observed in the equation of the gain above, a value of 500 Ω as resistance is obtained. As any other amplifier, the AD620 needs to be electrically powered by a ± 15V voltage source. These cables and the ones to the positive and negative leads of the amplifier are connected in their respectively positions given:

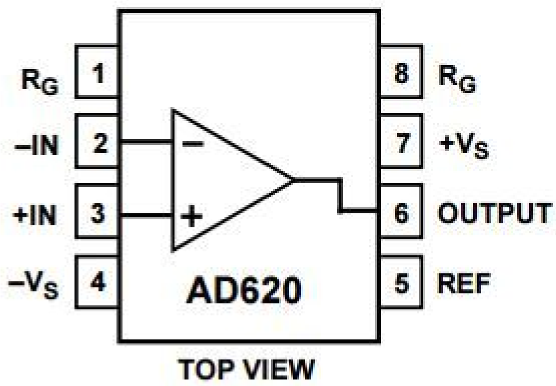


Figure 14: Schematic of AD620 ports connection

The whole circuit already connected with the PVDF material can be seen in the next figures:

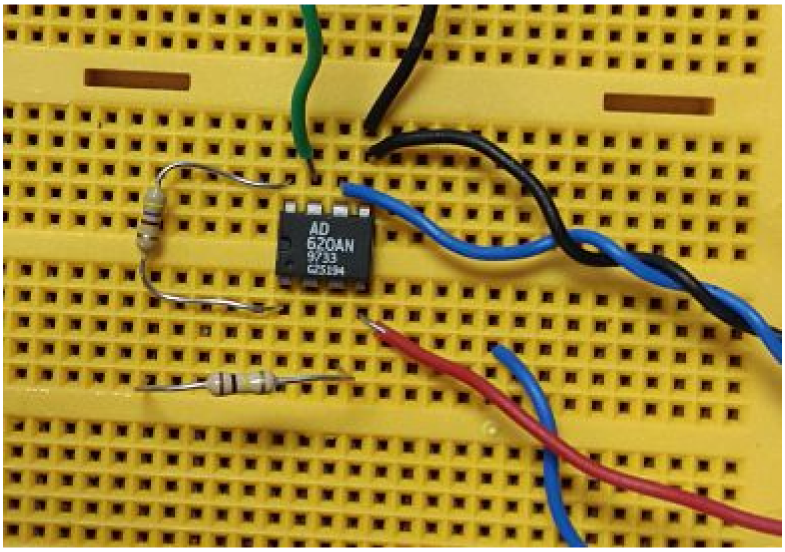


Figure 15: AD620 analog conditioning circuit

ii. Conditioning circuit based on an operational amplifier TL081

As it can be observed earlier in this project, a piezoelectric material is one that emits an electrical signal in response to a stimulus of mechanical deformation, among others types. However, this electrical response, although it varies depending on the type of piezoelectric being analyzed, is located usually in a range of millivolts. For this reason there is a need of designing a conditioning circuit with an amplifier that increases the value of that range at a voltage of up to about 3 or 5 volts, which could be easily recognized with any measuring device.

The first conditioning circuit was based on the voltage mode amplifier and had primarily an instrumental amplifier AD620. In this new section, however, the response of the material will be studied in a charge mode amplifier configuration with a TL081 operational amplifier.

An operational amplifier is typically a differential amplifier with a single output [50]. They usually have a very high DC gain and can be categorized by characteristics and applications. When the weak signal to be amplified is a voltage or a current, it is necessary to take into account the noise and the distortion that can be presented in the system. Another concern while designing the amplifier is the impedance levels: for example, the transducer output may be represented by Thevenin equivalent circuit in which the impedances may not be negligible compared to the signal conditioning amplifier's input impedance [51]. Consequently, the selection of the amplifier and the circuit must minimize all these possible errors in our system.

The TL081 operational amplifier used in the experiment is provided by Texas Instruments and can be defined by some characteristics as its low cost, high speed and high input impedance due to the JFET input state, bandwidth of 3 MHz, internally trimmed input offset voltage and low power consumption, among others [52, 53]. Similarly, the device requires a low supply current which makes the amplifier suitable for the experiment due to the low currents to be measured when using the charge mode amplifier configuration.

Commonly used to amplify signals from piezoelectric sensors, the charge mode amplifier is a current integrator that produces a voltage output proportional to the integrated value of the input current [54]. The amplifier balance the charge through the negative input of the amplifier by charging the feedback reference capacitor, generating an output voltage inversely proportional to the value of the reference capacitor and directly proportional to the total input charge flowing inside the capacitor during that time period. Also a resistance is needed to provide a DC bias path for the negative input [49]. Thus the circuit acts as a charge-to-voltage converter, with a fixed gain of the amplifier depending on the value of the feedback capacitor, and with an output voltage proportional to the pressure of the transducer.

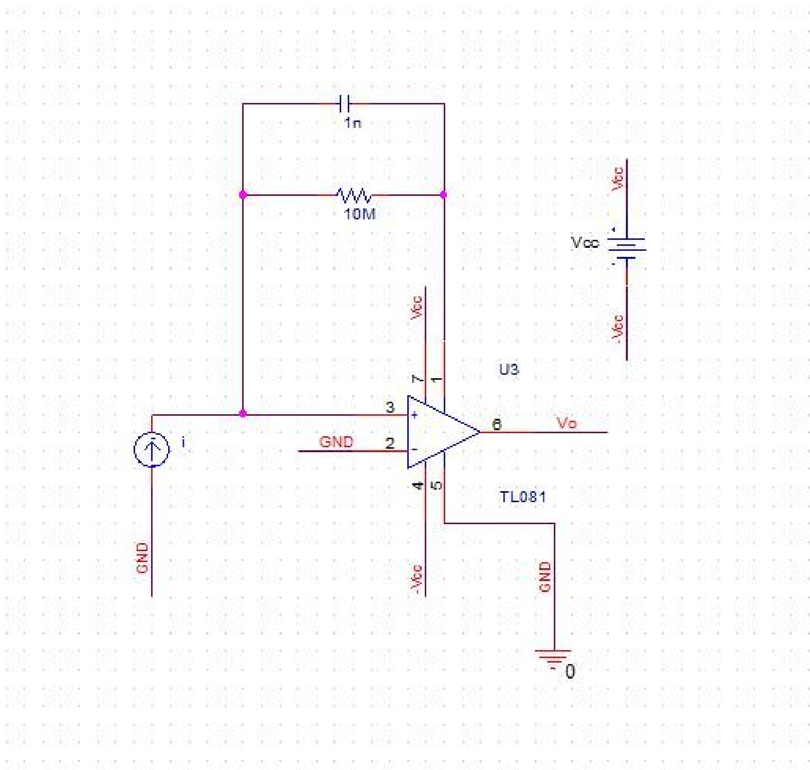


Figure 16: Orcad design of TL081 operational amplifier analog conditioning circuit

The conditioning design of the TL081 amplifier is shown in the figure above. An inverter amplifier where the negative input of the op amp is connected to the PVDF material, where a double side conductive tape is placed, and the positive input is connected to ground. The values of the resistance and the capacitor are 1 nF and 10 MΩ respectively. The high value of the resistance is fixed towards the need of an amplifier with a high gain value; the capacitor is the one in charge of filtering any AC signal that could be filtered in the system from the electric grind. The wires connected to the voltage source fixed at ± 15 V are connected in their respective electrical powered input, just as al the inputs are connected following the following figure:

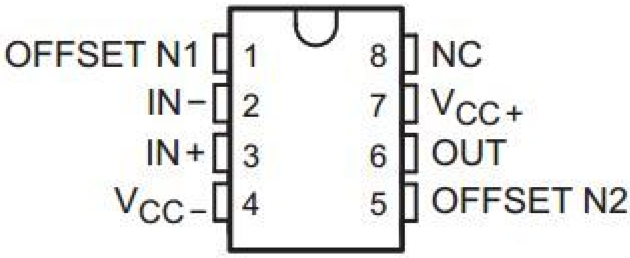


Figure 17: Schematic of TL081 port connection

Subsequently the whole conditioning circuit is shown in the figure:

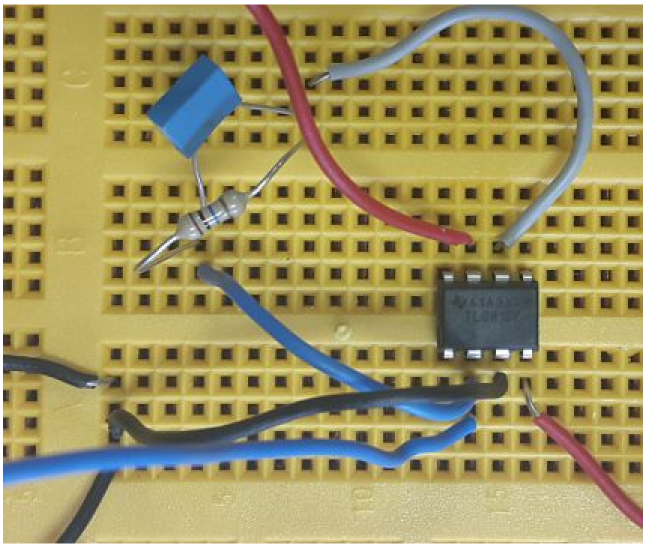


Figure 18: TL081 and some connections of the analog conditioning circuit

iii. AC Analysis

1. Introduction to AC experiment

Recently PVDF thin films have been designed and characterized for quasi-static and high frequency dynamic strain sensing applications. Due to PVDF's advantage of structural and electromechanical capabilities as its light weight and flexibility, it seems like a good alternative to study instead of other piezoelectric which might be thicker and therefore could be degraded by certain resonance patterns [55]. Applications in fields as optics where PVDF based bimorph is ideal for a laser or scanner where high speed laser beam manipulation is feasible [56] or health monitoring where ultrasonic lamb waves propagate in plate like thin structures, are increasing nowadays.

In the DC modulation experiment seen previously, a deformation by a weight was exerted constant over time. This way, by placing the weight set at the end of the metal plate, a mechanical strain or pressure on the plate was performed generating a deformation in the system. This deformation was kept until another mass was placed in the plate causing a new deformation in the system. The DC experiment, therefore, was considered an experiment that studied the electric response behavior of the material due to a slow and prolonged mechanical stimulus over time.

The purpose of frequency modulation is to observe the electric response of a piezoelectric material, in this case PVDF, when exerted on it a dynamic pressure, that is, a modulated frequency mechanical deformation.

2. Brief description of the assembly

In order to study the reaction of the PVDF material to a frequency modulation, a frame was designed and built consisting of a wooden stand on which is secured, with a screw of diameter 0.7 cm, a metal plate specially designed for the experiment. In the plate has been previously adhered a sample of the PVDF material of dimensions 10 x 5 mm with a slightly smaller than the dimensions of the material conductive double-sided tape positioned above it. Furthermore, in the plate, a strain gauge was attached to monitor the response of the material under the mechanical deformation.

An extreme of the plate was fixed to the wooden support while the other end was fixed to a speaker model 007543 provided by WRC of 130 mm of diameter, 100 W maximum of power and an impedance of 40 Ω. To ensure the contact between the support with the plate and the speaker so there were no leaks in the transmission, a small arrangement was installed with a nut screw to the metal plate which is inserted in a metal block attached to the membrane of the speaker using adhesive double layer tape X-Series General Purpose Transfer Tape XG2105 provided by 3M with 2 cm of width.

For the purpose of conditioning the output of the PVDF material as a voltage signal, two conditioning signal circuits were proposed: one formed by an instrumentation amplifier AD620 and another composed mainly of a lock-in amplifier. Furthermore, a strain gauge system with its corresponding analog conditioning circuit is installed to monitor the deformation produced by the loudspeaker.

In principle, there should be no problem with the data acquisition deformation of the plate by the strain gages, being this a system of frequency modulation, because the gauges are capable of measuring deformation up to the range of KHz of frequency: some studies has shown that the frequency response of a strain gauge is determined by the gauge length and the longitudinal elastic wave speed of the test specimen, up to 600 KHz when the gauge is 0.2 mm [57]; while other shown that studying the dynamic response of the strain gauges to ranges of about 300 KHz, the deviations of the static gauge factors do not exceed 5% within the frequency range up from 45 KHz to 300 KHz for gauges of length less than 20 mm [58]. As the strain gauge used in the experiment is about 10 mm of length there should be an adequate dynamic response of the system for a range of 100 KHz.

In the picture below can be seen the full assembly with the speaker and the wooden frame:

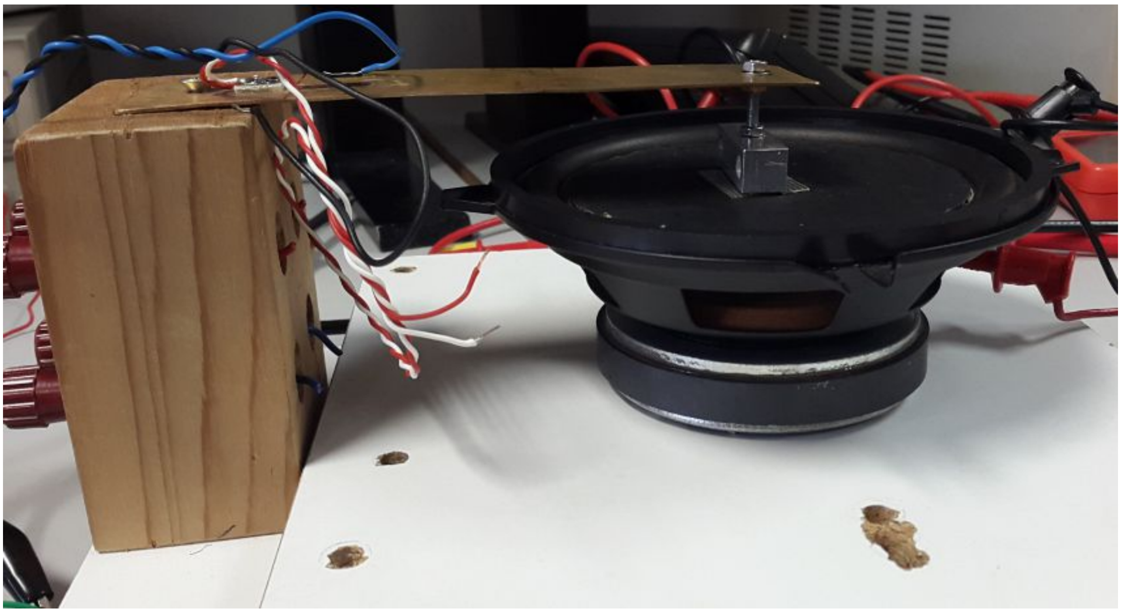


Figure 19: Wood assembly and speaker connected with brass metal plate

3. Components of the assembly

Below it is presented a description of the process followed for the design and assembly of all the components in the system. Some of the following components were provided by the Universidad Carlos III of Madrid such as the wooden assembly, the measurements devices or the speaker were loaned from the Department of Electronics Technology or the PVDF samples kindly provided by the Department of Materials Science and Engineering and Chemical Engineering; while others were made in a workshop as the metal plates.

Metal plates

To perform the experiment, four types of metallic plates were used: copper, brass, steel and aluminum. Thus various responses from the PVDF material can be obtained according to the deformation that the metal plate undergoes by exerting a force on it, or as the conductivity and electrical resistance of the material itself.

For the preparation of the metal plates the next procedure was followed:

i) The metal was cut into rectangles plates of dimensions 15 x 3.5 cm with an Electric Metal Shears 18 Gauge from Eastwood Company. Then the plates were sanded to eliminate any imperfection that the material surface may have with a Black + Decker BDER0100 5 in Random Orbit Sander Disc.

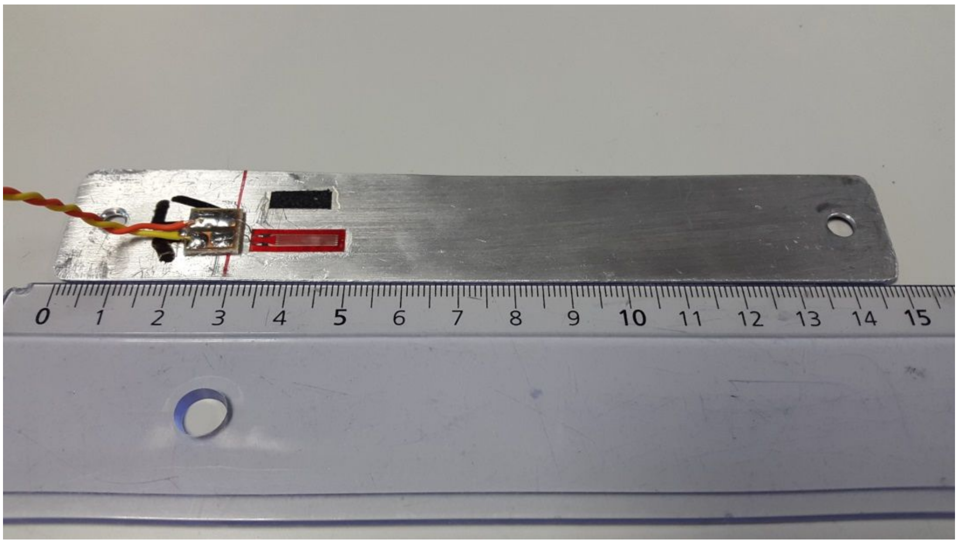
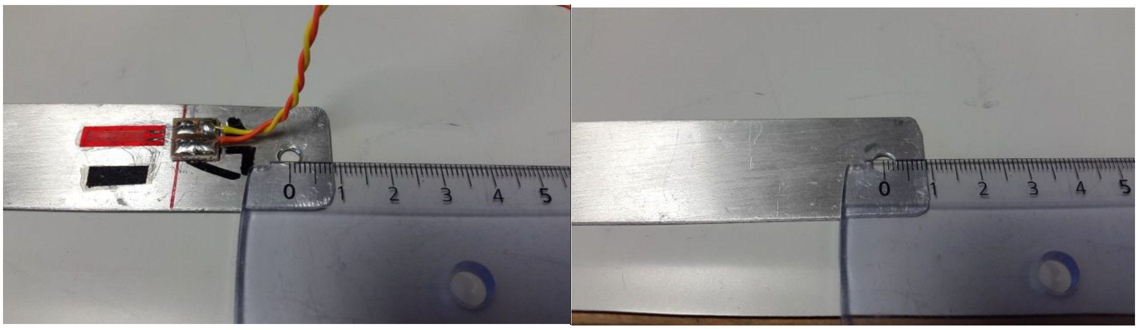


Figure 20: Aluminum metal plate with dimensions

ii) In the already cut and sanded metal plates two points were marked with a metallic punch. These points mark the place where the two holes must be drilled: the first hole of 0.4 cm of diameter and the second hole of 0.6 cm of diameter. The holes were made with a 120-Volt ½ in Corded Hammer Drill model HD19-2B by Bosch.



Figures 21 and 22: Dimensions of the holes drilled in the metal plates

iii) Once the holes were drilled, the plates were sanded with an electric sander to remove the excess material in the holes. Furthermore, the plates were pressed to ensure a flat surface for the PVDF sample.

Material

The Polyvinylidene fluoride (PVDF) material used was supplied by Sigma-Aldrich (Mn ~ 10,700; Mw ~ 27,500 g/mol and density 1.78 g/cm3) in samples of 100% PVDF composition.

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iv) PVDF pellets were ground using a MF 10 basic Microfine grinder. About 15 g of pellet were immersed in liquid nitrogen for 15 minutes and subsequently milled at 3000 rpm for 5 minutes to obtain a kind of flakes.

v) A hot plate press Fontijne Presses TPB374 was used to process the powders in film, with thickness obtained around 100 μm. The powders were placed in two polished aluminum plates of 10 x 10 cm, coated with Kapton sheets. Then they were heated to 200 ° C and pressed by applying a load of 50 kN.

vi) The circular samples of about 15 mm of diameter were cut into rectangular shape samples with dimensions of 5 x 10 mm and then placed in the metal plates at 2 cm distance from the holding hole. [16]

On behalf of the PVDF material to adhere to the metal is necessary to follow this procedure:

iii) The PVDF material, previously located in its proper place in the metal plate, has to be heated in a JP Selecta 2005163 - Digitronic-TFT oven up to 200 ° C. This process takes approximately one hour. Then, the metal plate with the already adhered material must be cooled to reach 50° C to be removed from the oven.

iv) A double side conductive tape Nisshin-Em is placed above the PVDF material. The dimensions of the conductive double-sided tape must be slightly smaller than those of the material so there is no contact between metal and conductive tape which would produce contact between two conductors and thus cause no voltage effect from the PVDF.

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ii) Apply one or two drops of M-prep Conditioner A to the cleaned surface and sand it to remove the paint and any other imperfection.

iii) Again, use a Kim Wipe to remove any particles left after the sanding and keep cleaning the surface until no imperfection can be seen.

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vi) If it is required, draw a pencil line on the exterior to proper alignment of the strain gauge in the following steps.

vii) Dispose the gauge and strain relief terminals bonding side down on a clean surface, leaving a 1.5mm gap between them.

viii) Place 100mm of M-LINE PCT-2A Cellophane tape over the clean surface and anchor one end of the tape to the slide, aligning the gauge in the center of the tape. Then, arrange the gauge in the appropriate location and anchor one side of the tape to the desired surface. Wipe the tape down to place the gauge and reposition it if it is needed.

ix) Raise the tape from the end opposite the strain relief terminals till the gauge and terminals are clear of the surface, fold the tape under and tack it behind the gauge. The area where the gauge is to be bonded should now be clear.

x) Apply M-bond catalyst sparingly in a thin and uniform coat following the next indications:

   a. Wipe the catalyst brush on the bottle 10 times to remove excessive product.

   b. Brush the surface with single strokes, covering the entire length of the gauge.

   c. Wait for at least a minute until the catalyst is dry.

xi) Attach the gauge to the desired area. Take into account that this step must be done within 5 seconds if the gauge is to properly bond to the surface, consequently:

a. Un-tack the end of the tape farthest from the bonding place.

b. Apply one drop of M-bond 200 Adhesive to the join of the tape and the area nearest the boding site, about 13mm.

c. Rotate the tape towards the bonding place until it forms an angle of 30° with respect the surface. This way the gauge will be above the desired position but not in contact with the surface.

d. Use a clean gauze sponge to bond the gauge to the surface with a single movement, starting from the terminal end to the un-tacked end and quickly apply firm thumb pressure directly over the gauge. Maintain the pressure for at least 1 minute considering that the warmth of the thumb helps the adhesive to set correctly. Then wait two more minutes for the adhesive to fully dry. When it is completely dried, remove the tape from the gauge carefully.

e. If in the previous step, the adhesive leaks from the sides of the tape, remove them with a clean wipe.

xii) In the event of attaching the lead wire, the most important factor is to prevent overheating the gauge as this will melt the gauge and therefore be useless.

a. Mask the strain gauge with tape, leaving only the solder tabs exposed.

b. Clean the tip of the soldering iron with a wet sponge pad.

c. Apply the iron tip onto the solder and use a firm pressure for no more than one second. Remove both the solder and the iron simultaneously.

d. Repeat the above procedure until a nice and even mound of solder is created on each solder pad and each strain relief terminal.

e. Bend the ends of the wire so there is a small bend between the strain relief and the gauge when the wire is soldered to the gauge. Press the soldering iron to the wire over a pad while feeding a small amount of solder between the iron and the wire. The wire should slide into the melting solder. Cool before handling.

f. Repeat for every solder tab and strain relief terminals.

g. Eliminate any leftover from the solder with a gauze sponge soaked in rosin solvent and use a dabbing action to prevent the gauge to be damage.

xiii) Last, apply a coating of M-Coat A over the entire gauge and terminal area.

Once the gauges are attached to the metal plate, their conditioning design is implemented, consisting mainly of a Wheatstone bridge: an electrical circuit used to measure an unknown electrical resistance by balancing two legs of a bridge circuit, one leg of which includes the unknown component. The primary benefit of a Wheatstone bridge is its ability to provide extremely accurate measurements [43].

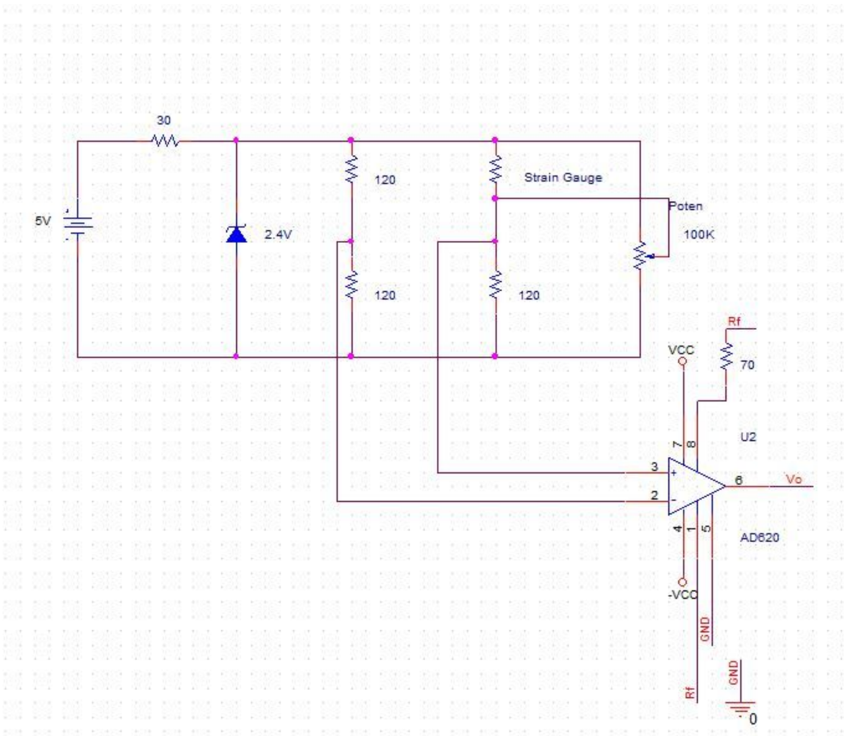


Figure 23: Orcad design of Wheatstone bridge conditioning circuit

As it can be seen in the picture above, the 5 volts supply is divided into a fixed voltage drop in the Zener diode –which has a value of 3.7 V– and the voltage drop in the resistance of 30 Ω, adjusting the value of the electric current through the bridge. The Wheatstone bridge is formed ideally by four resistors whose nominal value is maintained, so that ratio of the two resistances R_2 / R_1 is equal to the ratio of R_4 / R_3, obtaining a value of 0 volts between the midpoints of the bridge [43].

However, the value of the three resistors forming the bridge cannot be considered ideal and fixed; similarly the value of the strain gauge is considered variable in function of the deformation to which the plate is subjected. Therefore the presence of a potentiometer between the strain gauge and the resistor R_4 is required and the first step to prepare the analog conditioning circuit of the strain gauges is to set the value of this potentiometer. For that purpose the voltage difference between the midpoints of the bridge must be measured, moving the position of the potentiometer until a value of 0 volts is obtained. At that point the variation in voltage at points A and B can be characterize depending on the change in the deformation of the plate. In order to facilitate the measurement of voltage between these points an instrumentational AD620 amplifier is placed, with a gain of 706 V / V achieved by using a resistance of 70 Ω value.

Speaker

The DC analysis assembly consisting of weights provided an investigation of the behavior of the material at a slow and prolonged stimulus over time as the metal plate, when deformed by the action of a weight attached to one end, kept said deformation until another weight was repositioned. For this reason it was decided to study the material reaction to another kind of stimulus. In this new experiment, the objective set is to analyze the behavior of the piezoelectric material when a speaker, frequency modulated, mechanically deforms the PVDF material.

The speaker used in the test is a model 007543 provided by the company WRC which was chosen for this purpose because of its next main features: 130 mm of diameter, 100 W maximum of power and an impedance of 40 Ω. Then, it is connected using a cable with two outputs: one to the output of a sine wave generator and the other one to the ground of the function generator.

The method to achieve that the whole wave generated in the function generator is entirely transmitted through the speaker to the metal plate, where the PVDF material is placed, was designing a small assembly that was placed in the system as follows: the speaker is placed under one of the holes in the metal plate where a screw with a nut to hold is settled; after that, a small metal box with a drill of diameter of the screw positioned on the plate, is placed below the drill in the plate. This box is glued to the speaker cone through a double layer of adhesive tape and then the screw with a small nut is adjusted. This way the metal plate is directly connected with the speaker cone responsible for transmitting the generated modulated wave deformation.

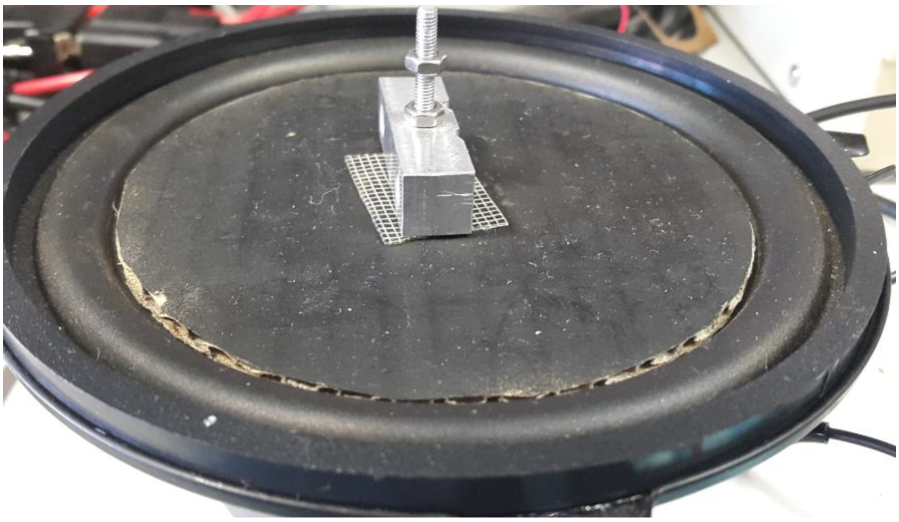


Figure 24: Image of the system created to connect the membrane of the speaker with the metal plate

The function generator used is model G305 from SWEEP. Characteristics as the 9 ranges of frequency capable of reaching (from 0.01 Hz to 10 MHz), the sine wave generation or the gate and trigger oscillation [59], among others makes it a suitable candidate to generate a sine wave within a range from 10 Hz to 1 KHz approximately.

Once connected, the whole system can be observed in the following figure:

Figure 25: Steel metal plate with PVDF and strain gauge connected to speaker

Ultrasonic transducer

An ultrasonic transducer is a device that converts electric signals to ultrasound waves [60]. In this section of the characterization of piezoelectric material, this device is used to test the behavior of PVDF when subjected to deformation caused by ultrasound. As some studies show the ultrasound properties of PVDF materials can be better than other piezoelectric materials. For example, large-area PVDF sensors can produce higher signal amplitude comparable with other piezoelectric sensors therefore making PVDF sensors more accurate under quasi-static, vibration and ultrasonic conditions [61].

The ultrasonic transducer used is the model AT120 from Airmar Technologies which cylindrical design allows its use in installations in different applications. Among its main features it can be highlighted its best operating frequency at 125 KHz, its weight of just 20 g and its typical sensing range from 20 cm to 3 cm [62].

The ultrasonic transducer is placed directly on top of the metal plate where the PVDF is adhered. To help the ultrasonic conduction between the metal plate and the transducer, an ultrasound gel is cast. This ensures that all the ultrasonic waves emitted are collected on the plate and thus on the piezoelectric material.

To condition the electrical signal from the PVDF, an AD620 instrumentation amplifier or Lock-In Amplifier are used, the two designs used for the frequency modulation with the speaker.

Figure 26: Ultrasonic transducer placed in metal plate with PVDF and strain gauge

Signal conditioning circuit

i. Signal conditioning circuit using an instrumentation amplifier AD620

Normally the voltage signal response obtained from a piezoelectric materials when a mechanical deformation is exerted, is on the range of microvolts. These values could be seen and measured in an oscilloscope but they will be covered in noise which will hinder the task of taking measurements or just check that the signal is correct and the whole system is working precisely. In order to achieve a range of voltage of about a few volts, there is a need of amplifying the signal coming from the piezoelectric material, which has to be done using the suitable amplifier. Some amplifiers may also involve linear filtering in the frequency domain, such as band-pass filtering to improve signal-to-noise ratio at the amplifier's output [45].

By definition, instrumentation amplifiers are described as direct-coupled, low-noise, differential amplifiers with a very high input impedance, high common-mode rejection ratio and a user settable gain from 0 to 60dB, and bandwidth to the hundreds of kHz [46]. Despite the fact that the instrumentation amplifier is represented identically to an operation amplifier, this is composed by three operation amplifiers: two arranged so that there is one op-amp to buffer each input and the third one to produce the desired output, as can be seen in the figure below [47].

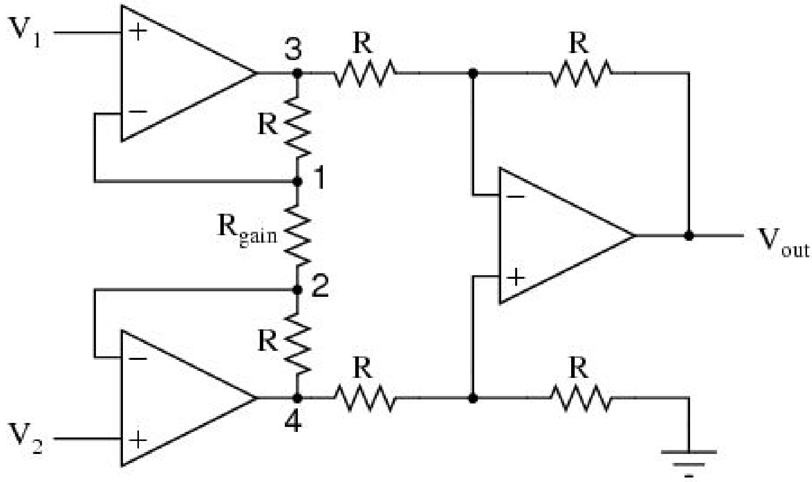


Figure 27: Schematic of an Instrumentation Amplifier

Therefore the gain of these types of amplifiers is defined as [70]:

$$V_{out} = (V_2 - V_1) \cdot \left(1 + \frac{2 \cdot R}{R_{gain}}\right)$$

For signal conditioning in this part of the experiment it is decided to use an AD620 amplifier. Said amplifier, proportionated by the company Analog Devices, is a low cost and high accuracy- 40 ppm maximum nonlinearity- instrumentation amplifier. Other characteristics of the AD620 are its low noise – around 9 nV/√Hz at 1 kHz and 0.28 µV p-p in the 0.1 Hz to 10 Hz band – and low input bias current – of about 1.0 nA – [48]. All these qualities make the instrumentation amplifier AD620 a suitable candidate for, among other applications, weigh scales, transducers interface or data acquisition systems.

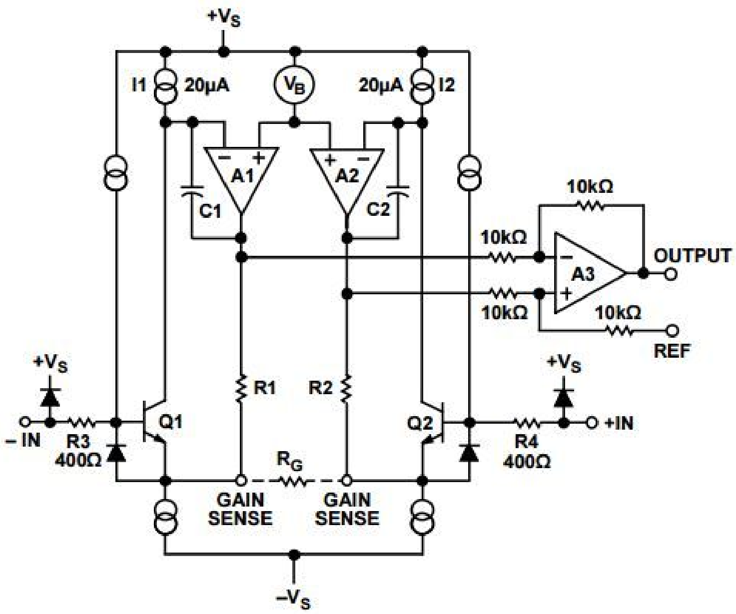


Figure 28: Schematic of an AD620 amplifier

Another quality why the AD620 model was chosen for the experiment is the ease with which the amplifier's gain can be modulated with only a resistor. This is due to a modification of the classic three op amp approach: in this amplifier the input transistors Q1 and Q2 provide a single differential pair bipolar input for high precision while the feedback through the Q_1-A_1-R_1 loop and the Q_2-A_2-R_2 loop maintains constant collector current of the input devices Q_1 and Q_2, thereby impressing the input voltage across the external gain setting resistor RG. This creates a differential gain from the inputs to the A_1/A_2 outputs given by $G = (R_1 + R_2)/R_G + 1$. The internal gain resistors, R_1 and R_2, are fixed to an absolute value of 24.7 kΩ [48], allowing the gain to be entirely set with an external resistor. Accordingly the gain of the AD620 amplifier can be defined as:

$$G_{AD620} = 1 + \frac{49{,}4 \cdot 10^3}{R_F}$$

Towards obtaining the optimal values of voltage desired – from around 3 to 5 volts – the following circuit is designed:

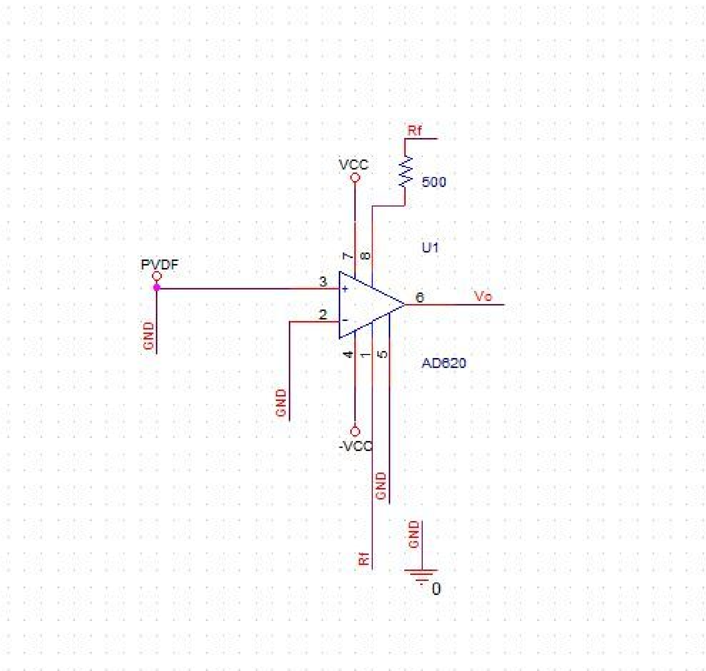


Figure 29: Orcad design of AD620 instrumental amplifier analog conditioning circuit

As it can be seen, it is a very simple analog conditioning circuit where the gain of the amplifier is used in a voltage mode amplifier. In a voltage mode amplifier, the output depends on the amount of capacitance seen by the sensor. Although the capacitance associated with the interface cable could affect the output voltage which could cause variations in the measurements taken [49], this type of configuration is used when the amplifier is very close to the sensor, as it is the case, therefore no variation in the capacitance should be measured.

The gain of the AD620 is set to 100V/V and, as it can be seen in the equation above, a value of 500 Ω as resistance is obtained. As any other amplifier, the AD620 needs to be electrically powered by a ± 15V voltage source. These cables and the ones to the positive and negative leads of the amplifier are connected in their respectively positions given:

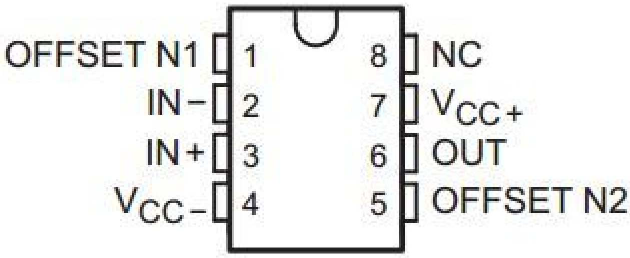


Figure 30: Schematic of TL081 port connection

The whole circuit already connected with the PVDF material can be seen in the next figures:

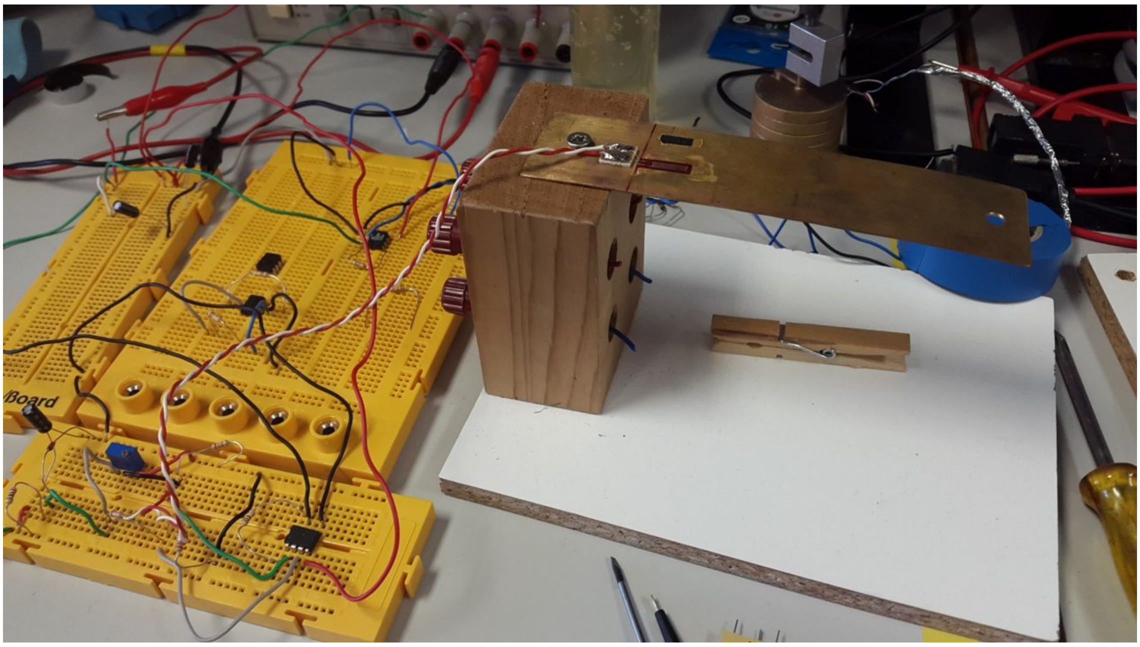


Figure 31: Assembly connected with AD620 conditioning circuit and strain gauge conditioning circuit

ii. Signal conditioning using a Lock-in amplifier

In a system as the one proposed, one of the details that must be considered is the possible noise which may be generated in the signal to be analyzed. An experiment with a signal-to-ratio less than 1 indicates that the noise level relative to the signal level is too high and, therefore, may preclude in the task of taking measurements [63].

We defined noise as a fluctuation of an electric signal characteristic of the electronic circuits [64]. Within the definition of noise this can have different triggers: thermal noise - produced by the agitation of the electric charges in an electrical conductor [65] – or noise generated by the devices themselves called flicker noise caused by material defect and roughness, among others. Noise signal have become an increasingly important issue in modern technologies with reduced supply voltages: noise limits minimum signal that can be processed or detected [66] which could disable an analysis like this based on taking measurements in small circuits.

Thus, a Lock-in amplifier was decided to use for conditioning the signal coming from the piezoelectric material PVDF. Usually Lock-in amplifiers are used to detect and measure very small signals due to the precision of the system that is capable of take measurements even when the small signal is obscured by noise thousands times larger; using a procedure known as phase-sensitive detection, consisting of selecting the component of the signal at a specific reference frequency and phase and then reject all the noise signals at other frequencies than the one of the reference. Therefore, the Lock-in amplifier needs a frequency of reference, that it is usually the frequency at which the experiment is being excited. For example, if our system is being analyzed at 60 Hz, the frequency reference is going to be 60 Hz, thus the Lock-in will detect the response of the experiment at that frequency and will erase all the other signals that are not at 60 Hz [67]. In the next figure it can be observed the functional block diagram of the Lock-In Amplifier:

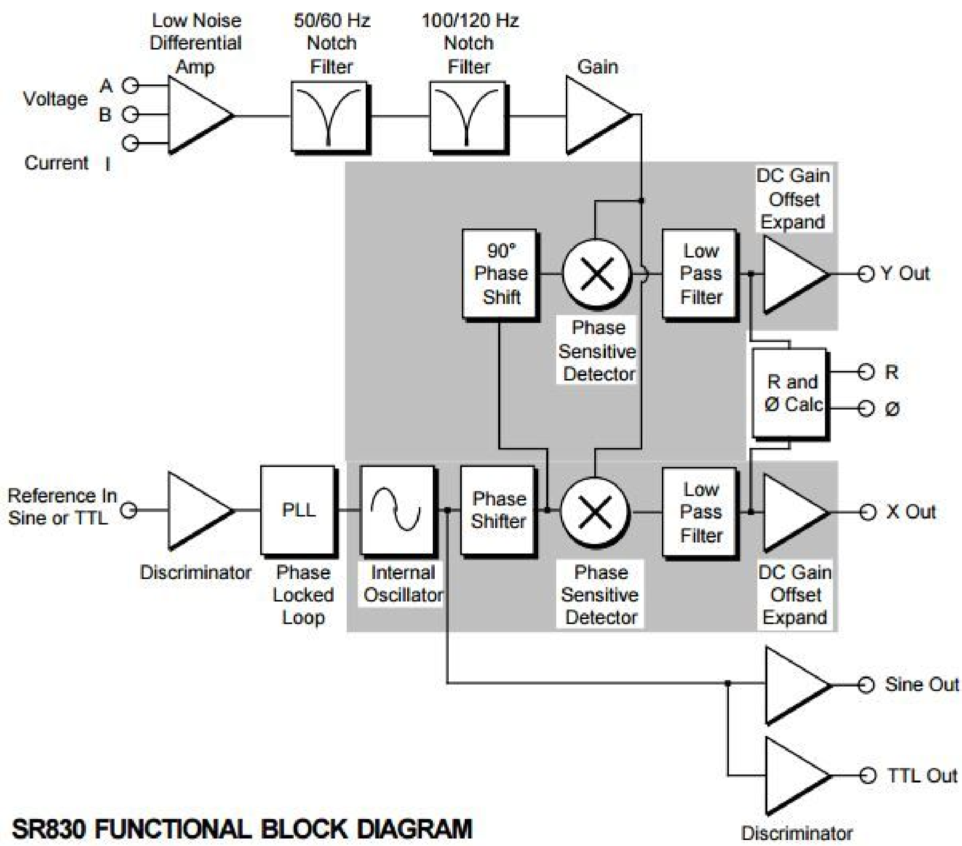


Figure 32: Functional Block Diagram of Lock-In amplifier model SR830

The device used is the model SR830 DSP Lock-In Amplifier provided by Stanford Research Systems. The SR830 displays the magnitude and phase of a signal using digital signal processing (DSP) to replace the demodulators, output filters, and amplifiers found in conventional lock-ins performing with good results within an operating range of 1 mHz to 102 kHz [68].

It was originally intended to use the lock-in amplifier for amplifying and cleaning the piezoelectric response although the idea of using the two amplifiers, both the instrumentation amplifier AD620 as the Lock-In, was also raised. Therefore the main design consisted of connecting the sinusoidal function generator to the speaker attached to the metal plate with the PVDF samples to then, set the reference frequency of the Lock-In in the value of the sinusoidal frequency generator. The piezoelectric output was connected to the input of the Lock-In Amplifier and the oscilloscope Tektronix TDS220 was connected to the output of the Lock-In Amplifier to observe the measured signal.

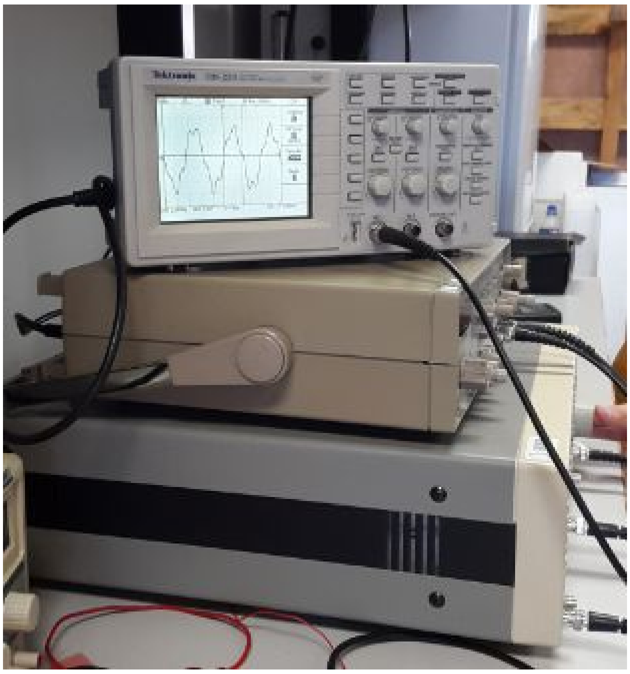


Figure 33: Oscilloscope connected with function generator and Lock-in amplifier

iv. Measurement of piezoelectricity in the PVDF

1. Introduction to the analysis

As it was explained in previous chapters, this study aims to create a system that characterizes the electrical response of a piezoelectric polymeric source material for its use as a pressure sensor. The motivation of the analysis and study was a small experiment that was conducted in the Department of Materials Science and Engineering and Chemical Engineering at the Universidad Carlos III of Madrid, in which the piezoelectric response of PVDF samples that were developed in university laboratories, were tested: the results obtained in that experiment showed an electrical response of the material when, under certain characteristics and in a controlled environment, were subjected to a mechanical deformation as an object fallen from a controlled height.

Therefore it was decided to repeat the original experiment in a similar environment and using the original assembly, monitoring the output signal of the PVDF material and thus obtaining continuity with the data of the first experiment.

2. Brief description of the assembly

The original assembly was used in this case: circular samples were placed in between two circular aluminum electrodes of 10mm of diameter, supported by a structure made of two thick plates of Poly (methyl methacrylate). The entire system was connected to the measurement equipment using wires soldered to plates touching the stems of the electrodes. The system is then closed with a pair of wing nuts to prevent the PVDF samples to move, avoiding measurement errors [16]. Then the system is connecting, using the wires connected to the electrodes that are in contact with the PVDF samples through the aluminum cylinders, to an electronic oscilloscope.

Once the installation has been assembled and placed in a location where it has nothing around, the responses of the material can be measured. For this purpose, a clear plastic cylinder in which a narrow metal cylinder is inserted it is used. This way the transparent cylinder is used as a guide for the weight to fall always on the same point of the assembly, trying to linearize everything as possible results obtained. The guide cylinder also serves to release the object from the same height in all experiments.

3. <u>Components of the assembly</u>

Original assembly

Described above, the system was homemade and can be seen in the following figure:

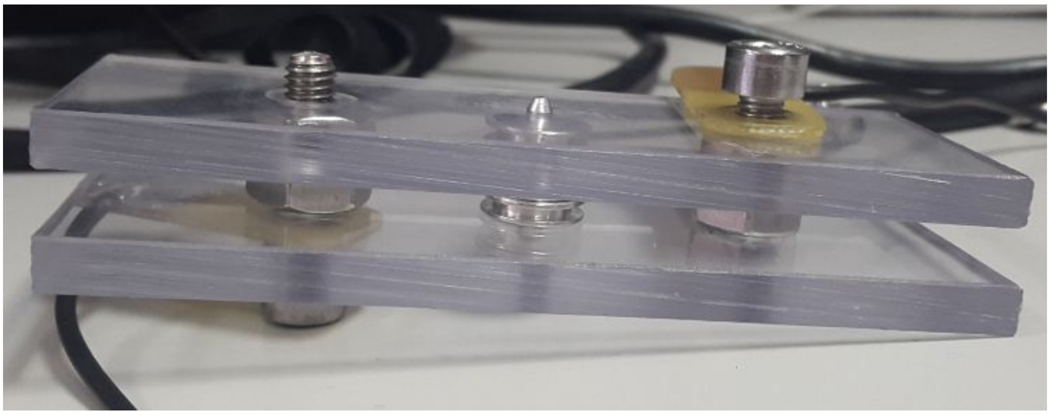


Figure 34: Homemade assembly for PVDF samples

Guide system

Aiming to normalize the results obtained, the following system was designed: using a transparent cylinder of dimensions 2 cm of diameter and 100 cm of height, the height from where to release the weight was fixed so it was also the trajectory of the weight, ensuring that all the measurements of the response of the material were taken in the same conditions of free fall velocity and impact on the same point of the assembly.

The weight hurled was a small metal cylinder of dimensions 7 cm of length and a weight of 14.94 g.

Figure 35: Dimensions of metallic object

Figure 36: Weight of metallic object

Following, a figure of the system:

Figure 37: Image of the whole system including assembly, acquisition data system and computer

Measurement system

An electronic oscilloscope model Tektronix TDS220 was used to measure the electric response of the piezoelectric material. The Tektronix TDS 220 Digital Real-Time oscilloscopes offers uncompromised bandwidth, automated measurement features as period, frequency or RMS, mean, and Peak-to-Peak values and analog-like ease of use, at a cost comparable to analog scopes of equal bandwidth [69].

The oscilloscope was connected using the electrodes from the system, as can be seen in the next figure. There was no need of an amplification system.

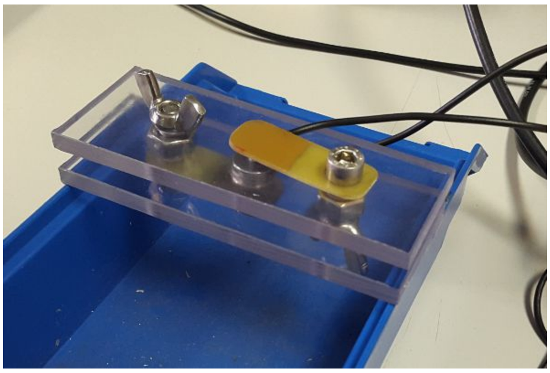


Figure 38: Plastic assembly with the PVDF samples

IV. CHAPTER FOUR

i. <u>Introduction</u>

The starting point of the experiment was the samples of PVDF that were used to analyze the response of the material under different stimuli and under different conditions. With this purpose, three different assemblies, explained in detail in previous chapters, were designed to study the piezoelectric response of the material under DC and AC frequency modulation. Moreover, by installing a strain gauge, it was proceed to model the response of PVDF.

Therefore, there were two objectives in the analysis: the first one was to check if the PVDF material may be suitable for its use as a pressure sensor as other piezoelectric materials which have a ceramic origin, comparing his response to those obtained with the strain gauges; the second one was to analyze the response of the material with respect to its molecular phase.

Regarding the material it must be remembered that the PVDF, a semi-crystalline polymer, can have four main phases: α, β, γ and δ. Of these, the one with a larger dipole moment is the β phase [34]. Therefore, the main issue when using this piezoelectric polymer is ensuring that the material is in β phase, since this is the only one that exhibits the piezoelectric characteristics necessary for it to be used in sensors and actuators. The atomic structure of β phase is one where the dipoles are aligned parallel to each other when a mechanical deformation is applied; in the α phase, the orientation of these dipoles under stress is random, thereby canceling each other and not obtained net surface charges [35].

The most concerning issue with the phases of the material is that, when it has not been treated by what is called poling process in which the PVDF is polarized by using a strong mechanical force producing a deformation in its structure or under the action of an electric field of high value, the material is in the α phase.

The following figure shows a Raman spectroscopy measurement for various PVDF samples in which it can be seen mainly the presence of the β phase in the molecular structure of the crystal; lesser extent α phase can also be seen [3].

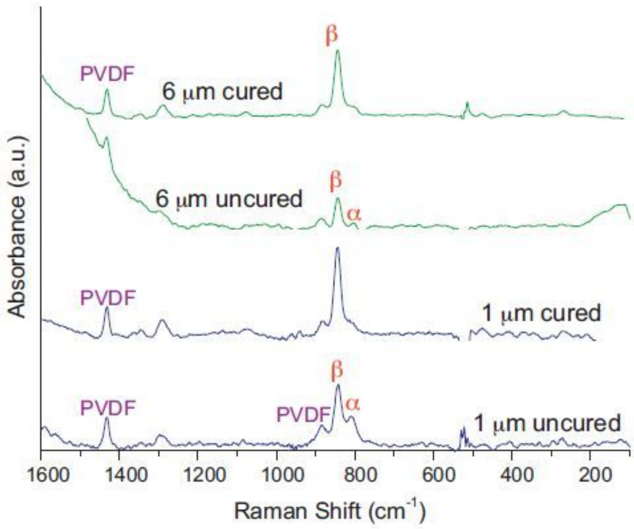


Figure 39: Raman spectroscopy of PVDF material with beta phase predominant

The experiment was started with the assumption that the material samples were in β phase since there an experiment was conducted where the material, encapsulated in a frame, was hit and it was obtained an electrical response as a voltage variation measured with an oscilloscope.

ii. <u>Obtained results from DC analysis</u>

In order to analyze the behavior of the PVDF polymer a system was designed compound metal plates where the material and a strain gauge were adhered; one end of the plates was fixed to a wooden frame while the other was left free, with the intention of creating a cantilever beam. In the free end, a set of eight weights of 25 g each were placed.

After that two signal conditioning circuits were designed: the first was intended to amplify the signal from the piezoelectric PVDF using an AD620 amplifier in voltage configuration mode, where amplified output voltage of the system was the voltage difference between the outputs of PVDF; the second installation contained a TL081 amplifier in charge mode amplifier. In this mode, the charging and discharging of the capacitor attached to the configuration feeding through the signal coming from the material, is the signal that is being amplified.

To connect the sample with the analog conditioning circuits that will be later connected to the measuring devices it was needed to use cables. For this purpose, a small rectangle of double layer conductive tape, slightly smaller than the sample of PVDF, was adhered on the material itself. This way, it was safe to settle the cable above the conductive tape without fear of damaging the material. Then all cables were joined: the output of the material to the input of the amplifiers and the output of the strain gauges to the Wheatstone bridge and it was proceeded to balance the bridge by using a potentiometer of 100 kΩ value. When the output voltage of the bridge was null it was believed that the bridge was balanced and therefore it could begin to take measurements. After that, voltage generators that powered the amplifiers and the Wheatstone bridge were switched on and it was began to take voltage measurements from the gauges and from the PVDF material when a mechanical deformation was exhorted by a weight that was placed on the system. Measurements were performed with a Protek multimeter.

The figure below shows the results obtained from the strain gauges from three of the plates designed for the experiment: steel, brass and copper. The measurements taken with the aluminum plate are not attached because the excessive deformation of the plate under the weight prevented to obtain standardized results. All the magnitudes are measured in volts and are referred in absolute value so the variation of the range can be calculated.

Weights	Steel			Brass			Copper		
0	6,97	6,95	7,06	6,06	6,16	6,18	6,44	6,14	7
Support	6,78	6,85	6,93	5,76	6,1	5,98	6,55	7,17	7,16
1	6,57	6,76	6,74	5,77	5,9	5,92	6,45	7,14	7,15
2	6,51	6,61	6,63	5,64	5,86	5,84	6,38	7,1	7,01
3	6,44	6,54	6,53	5,47	5,77	5,62	6,44	6,99	6,42
4	6,36	6,4	6,31	5,15	5,58	5,48	6,45	7,01	6,4
5	6,21	6,37	6,26	5,2	5,49	5,33	6,24	6,98	6,36
6	6,23	6,32	6,14	5,12	5,39	5,2	6,15	6,97	6,45
7	6,14	6,15	6,04	4,96	5,36	5	6,18	6,93	6,4
8	6,01	6,06	5,98	4,85	5,23	4,88	6,12	6,79	6,37
ΔV	**0,96**	**0,89**	**1,08**	**1,21**	**0,93**	**1,3**	**0,32**	**-0,65**	**0,63**

Table 1: Results obtained from Strain Gauges in DC experiment using different metal plates

If the results obtained from the strain gauge are analyzed it can be concluded that, except one of the measures from the copper metal plate, all results are average from what could be considered as a voltage variation amplified by a design like the one proposed. The results show an increase in the value of the first measurement, probably due to the relocation of the plate in its original position when removing all the weights placed in the previous measurement position.

However, the results measured from the PVDF material were not as positive as it was impossible to take measures of voltage variation coming from the system: when the assembly was connected with the support and the weights included it was observed on the oscilloscope a much higher noise signal with a frequency of 50Hz instead of the output coming from the PVDF even when the signal was supposedly amplified. It was concluded that, as the piezoelectric material was not completely isolated, the weights' support was acting as a noise antenna making the measurement impossible even when the following modifications were tested:

- An attempt of isolation of the material in the metal plate with respect the weight assembly was performed.
- Isolating the wires, braiding them to prevent the parasitic capacitance.
- Connecting capacitors in all the analog conditioning circuits, acting as filters of the sine wave coming from the electrical grind.
- Double side conducting tape was placed also in the ground of the metal plate so the two sideboards of the amplifiers had the same input resistance.
- Another idea was that the impedance of the system composed of the metal plate resistance, the PVDF resistance and the double side conducting tape resistance, was bigger than the impedance of the amplifier – which is normally quite big-. Therefore a small resistance was added in parallel so that this impedance was decreased.

The problem was repeated also with the analog conditioning circuit composed of the TL081 amplifier. In both circuits, all actions previously raised were conducted without obtaining a positive electrical response coming from the PVDF.

iii. Obtained results from AC analysis

In this part of the study on the characterization of a piezoelectric polymer material in order to use it as a sensor, it was proceed to analyze the response when it was subjected to a mechanical deformation produced by a frequency modulated sine wave.

The assembly used is similar to the one used in the analysis of DC, the wood assembly and the metal plate with the strain gauge and the PVDF placed as a cantilever beam is maintained. The difference is that in this part, instead of placing a set of weights on one end of the metal plate, a speaker is attached. Thus an assembly of a small metal box and a screw that is attached to the hole in the metal plate is used. The small metal box is adhered to the speaker cone with a double layer of adhesive tape and the screw is secured using two small nuts. This way it is ensure that all the energy of the sine wave is evenly distributed as mechanical deformation directly to the metal plate and that there are no leaks in the system.

The analog conditioning circuit of the strain gauges and the one designed to conditioning the AD620 amplifier are also used in this experiment; moreover, a new element for the conditioning circuit is included: a Lock-In amplifier. The intention is to adequate the signal coming from the piezoelectric, either using the Lock-in amplifier solely as an amplifier and filter or using it to filter the signal coming from the PVDF being amplified by the AD620.

Once the speaker has been properly positioned in the assembly, it was connected with a function generator model G305 from SWEEP, wherein a frequency modulated sinusoidal function is generated. Before starting the experiment is often necessary to balance the Wheatstone bridge using the potentiometer until the voltage difference between the AD620 inputs is null. It is also necessary to connect the generators feeding the amplifiers used in the conditioning circuits. Then, the PVDF output or the amplifier output is connected to the Lock-In amplifier which, in turn, is connected to an oscilloscope model Tektronix TDS220. Then the deformation of the plate using the modulated frequency function can begin.

The data obtained on the brass plate of the strain gauge response to the deformation produced by different frequencies are shown in the following figures. The signal from the gauge is being amplified by the AD620 as shown in the description of the system and even though the Lock-In amplifier is not used in this part, the signal is being digitized using the computer program Clever Scope. Low frequency values are not altered by any kind of filter in the program; but at higher frequencies the values are averaged.

At 54 Hz:

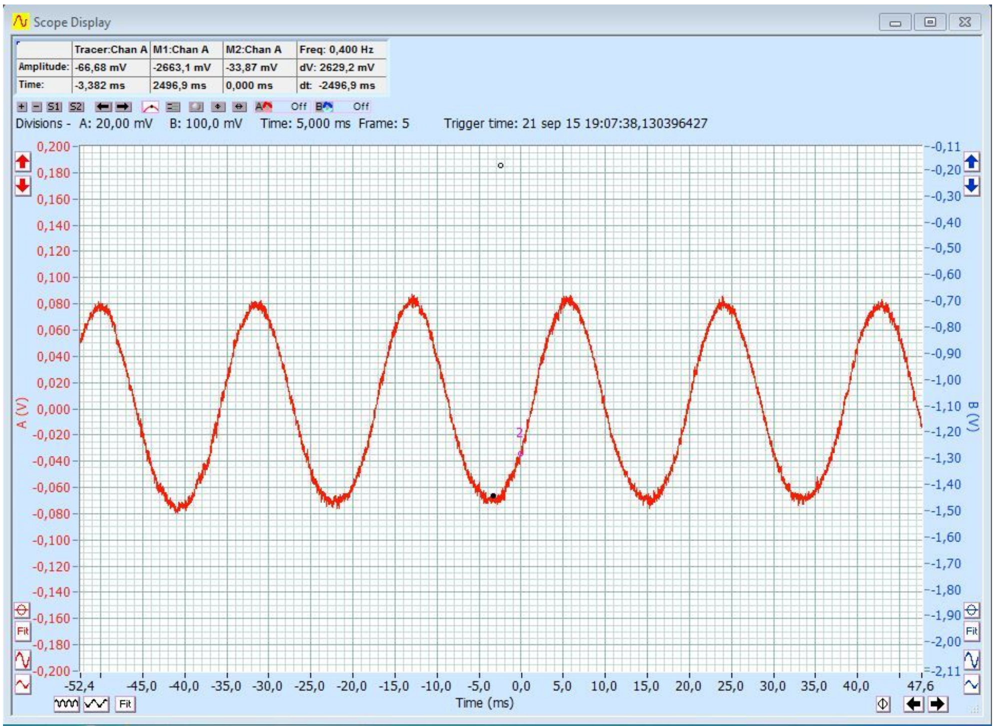


Figure 40: Strain Gauge voltage response in AC modulation at 54 Hz

At 100 Hz:

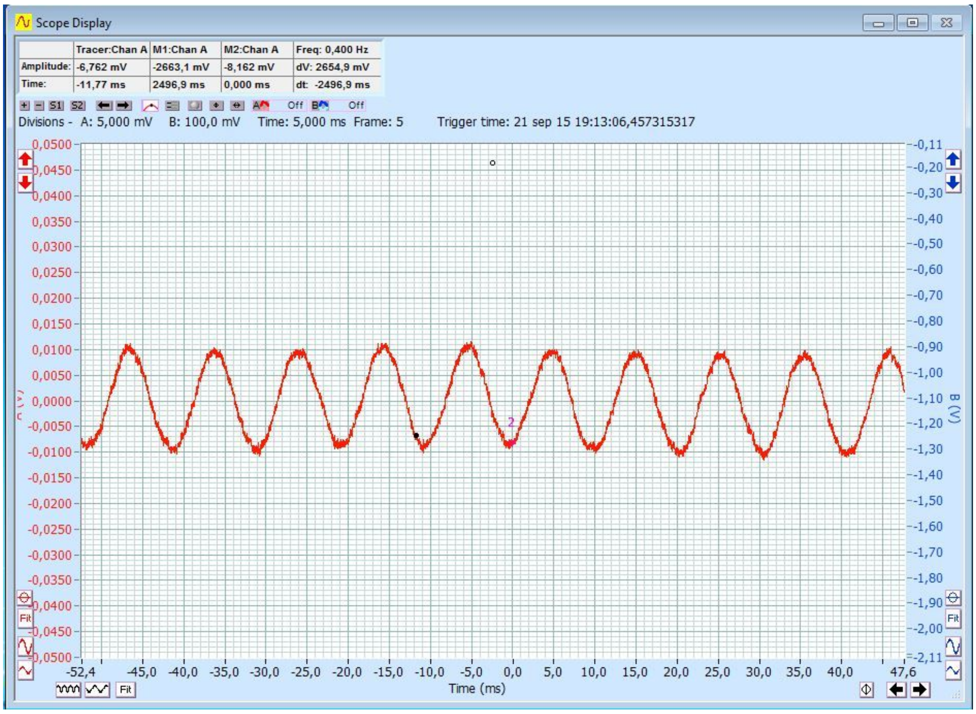


Figure 41: Strain Gauge voltage response in AC modulation at 100 Hz

At 200 Hz:

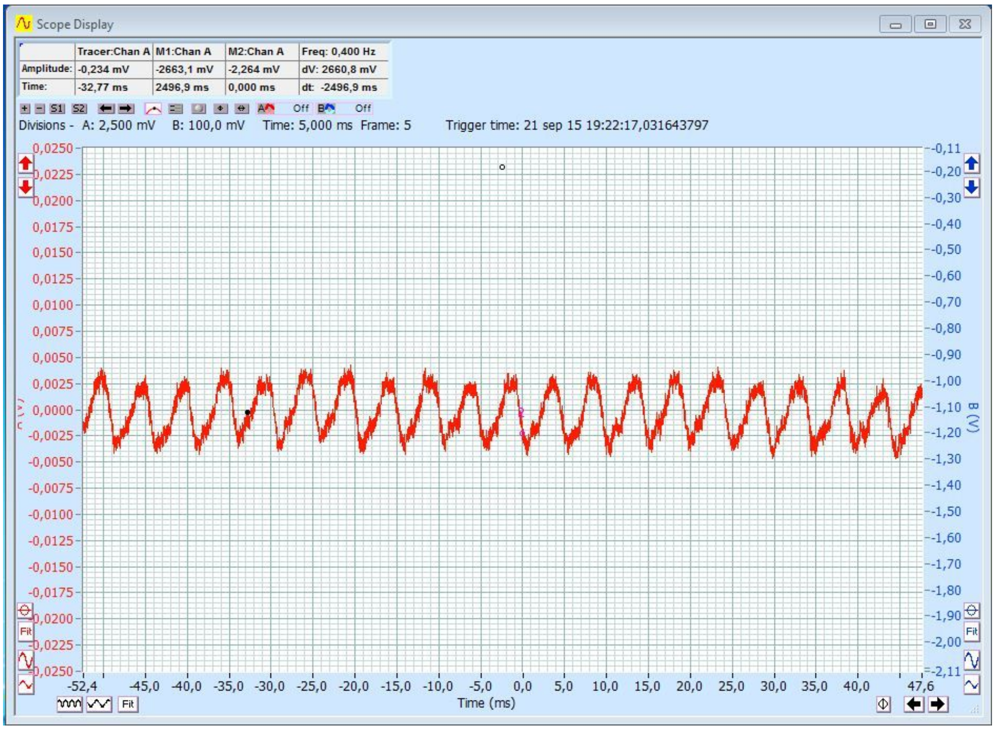


Figure 42: Strain Gauge voltage response in AC modulation at 200 Hz

At 300 Hz:

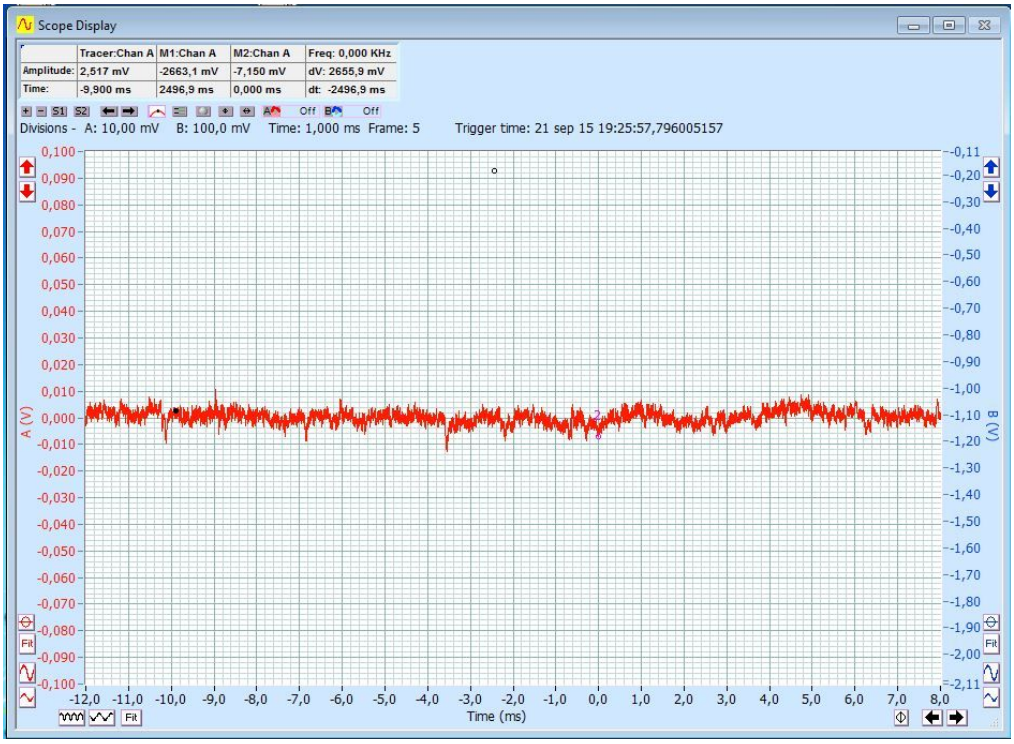


Figure 43: Strain Gauge voltage response in AC modulation at 300 Hz

From the pictures we can observe the following measures voltage variation. The measurements obtained in the last experiment at a frequency of 300 Hz are approximate since, as it can be seen in the image, although the signal is averaged, it is distorted by a larger noise signal and is not easy to evaluate.

	Frequency (Hz)			
Brass	54	100	200	300
ΔV (V)	0,16	0,02	$6 \cdot 10^{-3}$	0,02

Table 2: Representation of the variation of voltage response of strain gauges in AC modulation

However, as it happened in the experiment in DC, it was not possible to obtain an electric response from the PVDF material to a stimulus of mechanical deformation generated by frequency modulation. Again it was tried to enhance the signal from the piezoelectric which, when connected to the oscilloscope, was practically invisible compared with the signal of 50 Hz that returns to seep into the system. To improve the measurement situation, the actions taken in the preceding paragraph are repeated, trying to isolate in the best possible way the system, changing resistances to increase gains and reduce internal impedances, placing capacitors that filter the AC network, among others.

A Lock-In Amplifier, capable of filtering a very small signal using a known reference signal that is generated at the same frequency of the signal to be filtered, with null results was also used.

Likewise the Clever Scope program was used to filter the signal with a high pass filter -preventing the entry of 50 Hz signal- and a Bessel filter, with negative results as well.

Finally it was stated that the double-layered conductive film could have been damaged during the experiments and it was proposed to change it: the resistance of the conductive tape was measured obtaining a resistance in the order of mega ohms. A new sample of conductive double side tape is placed over the PVDF sample and it was checked that the resistance of the tape was still mega ohms.

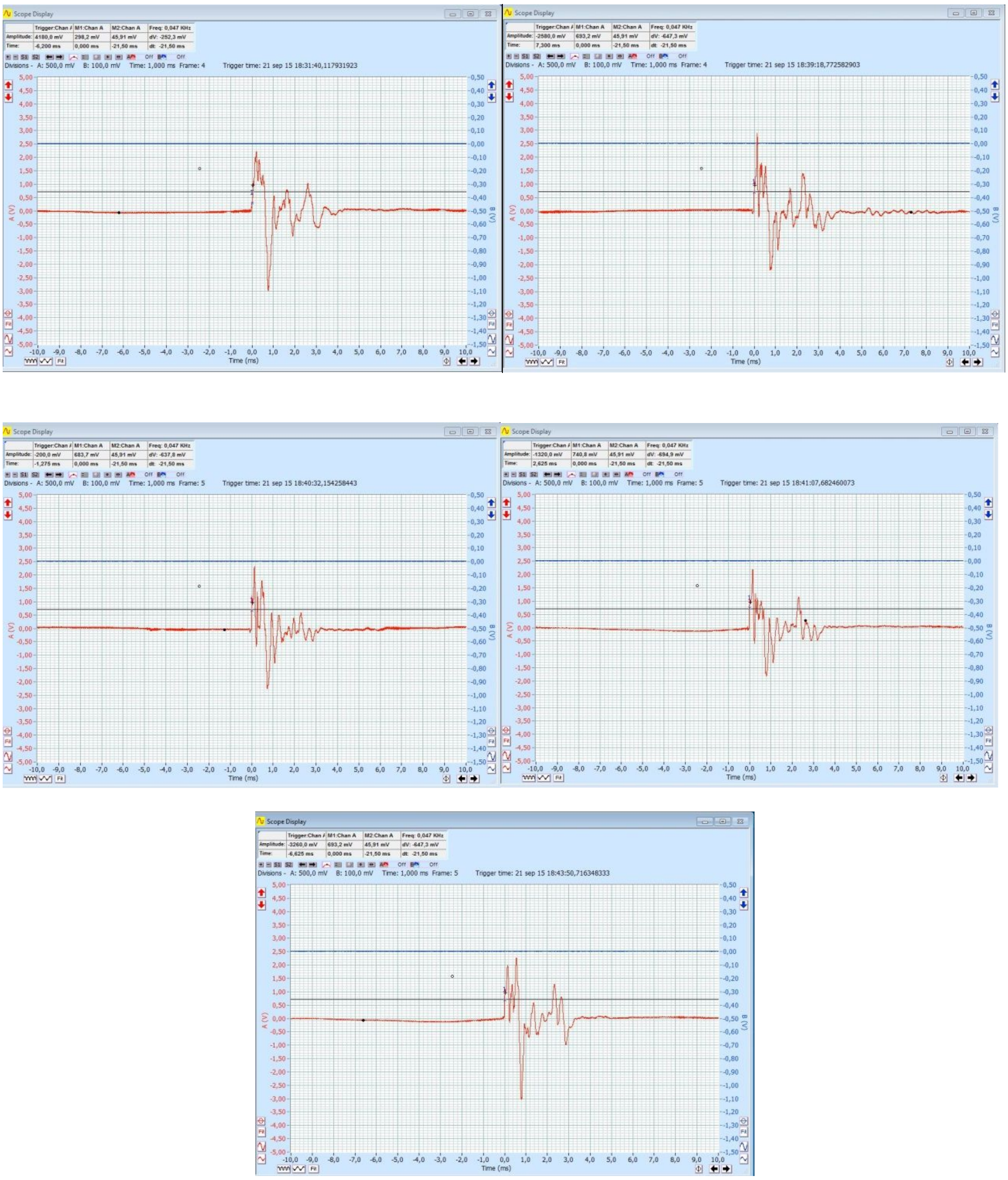
Lastly it was decided to perform the last experiment using the same system; the speaker is substituted by an ultrasonic transducer, thinking that perhaps the material response could only be observed at very high frequencies, taking measurements from 10 KHz to 100 KHz. All wires and function generators are connected and a wave is generated to power the transducer. Again null results are obtained coming from the PVDF.

iv. Obtained results from the original experiment

Due to the poor performance of the PVDF in the experiments of analysis in DC and analysis in frequency modulation or AC, it was decided to make the first experiment conducted by the Department of Materials Science and Engineering and Chemical Engineering at the Universidad Carlos III of Madrid with the PVDF material, including using the original assembly and new samples of the material.

The new PVDF samples were placed between two aluminum cylinders that allow the sample not to move and connect the samples with the electrodes that are then connected to the oscilloscope; a transparent tube of one meter was used as a guide for free fall: a small object is launched from the same height, so that it always impact on the same point of the system. Once the entire system has been arranged, the electrodes that were in contact with the samples, was connected to an oscilloscope that displayed the piezoelectric response when the small object launched from a fixed height hit system.

With the objective of improving and simplifying the data collection in this experiment, the Clever Scope program was used. In the following figures it can be seen the response of the material when a small metal cylinder of weight 15g was thrown from a height of one meter.



Figures 44-48: Representation of signal response of PVDF in the original experiment

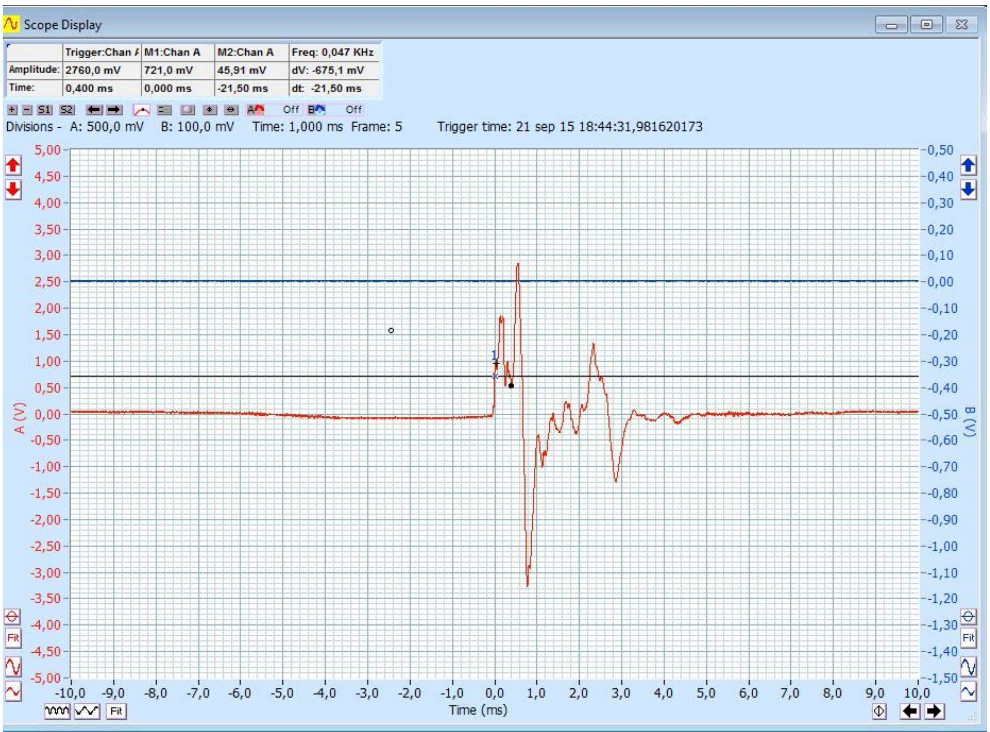


Figure 49: Best electrical response obtained from PVDF

The experiment was conducted six times, obtaining the same electrical response from the material in all of the experiments as it can be seen in the figures above. As it can be observed, the electrical response of the PVDF is clean and clearly seen without amplification. This experiment showed that the material response to a stimulus conducted under the same conditions is similar and therefore linear. As it can be seen in the next figure that represents all the points obtained with a digital acquisition system and the program Clever Scope that graphed 6350 points:

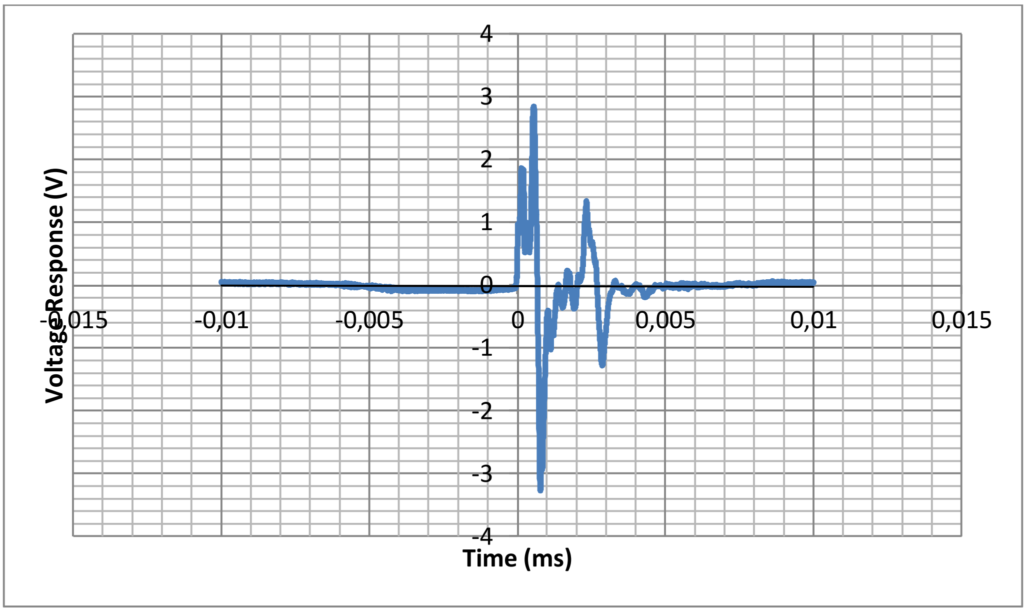


Figure 50: Graphical representation of 6350 points in Clever Scope when measuring the signal coming from PVDF

As it is difficult to observe the maximum values of voltage produced by the signals coming from the PVDF, they have been represented in the following table:

	Signal number					
	1	2	3	4	5	6
V_{max} (V)	2,25	2,8	2,4	2,25	2,25	2,7

Table 3: Maximum voltage obtained from PVDF electrical response

v. Final conclusion

Based on the assumption that the phase of the semi-crystalline material PVDF is the β phase and analyzing the results obtained in the different experiments arranged it can be reached the conclusion that the initial assumption concluded was incorrectly raised.

Therefore, it was analyzed if the molecular phase of the samples is the α phase. To check which is the phase of the PVDF with which it was used a FTIR-ATR spectroscopy experiment was performed.

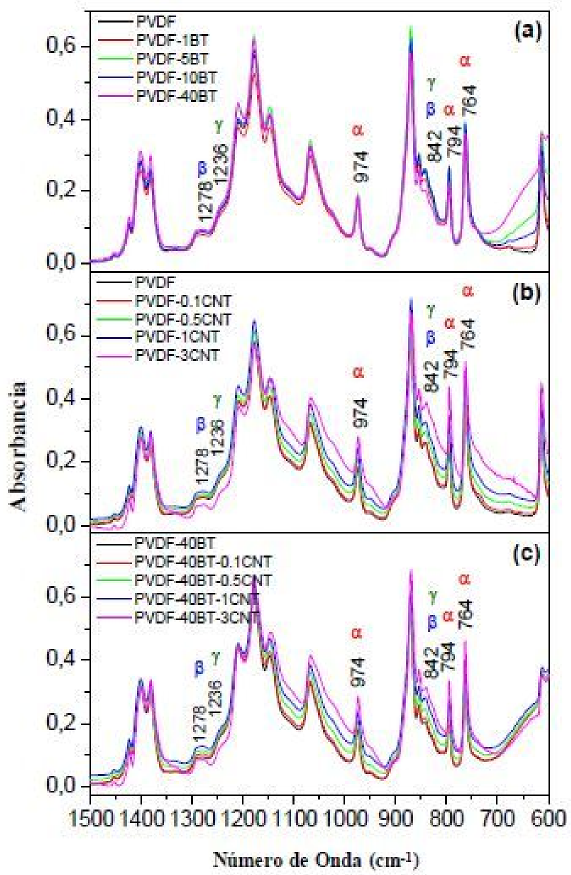


Figure 51: Image from FTIR-ATR spectroscopy experiment performed to PVDF samples used in the analysis

In the picture it can be observed that in all the cases studied in the spectroscopy experiment performed by Freddy Ariel Sanchez Ruiz, it is observed that the values of 1214, 974, 794, 764 cm-1, are the main characteristic absorption bands of the α phase. However it can also be observed a contribution of β and γ phases in small bands at 842 cm -1 and 1278 cm-1, generally assigned to the vibrations occurring in these phases, concluding that the samples are in α phase [16]. Having been proved that the PVDF samples' phase was the α phase, it can be concluded that the results of the system are as expected because the material at this phase has no piezoelectric behavior.

Another topic to be considered is that samples adhered to the metal plates were glued by heating the material above until a temperature value of 200 degrees, exceeding the melting point of the PVDF that is 170 degrees, which may had led to the emergence of impurities in the material when it was re-crystallized. Note that to polarize the PVDF it is needed to subject the material to high temperatures, a large electric field or perform a stress of a very high value on the material; none of the options were held when the material was adhered to the metal plate and therefore the phase of the material is unknown, although with the results obtained could be assumed that the PVDF was not in β phase.

Also there were some other impediments, apart from the phase of the molecular structure, when conducting the experiment as, for example, the case of the conductive double layer tape that had an extremely large resistance; or the filtration in the system of a high signal noise that was attempted to minimize using twisted wire, shielded cables and bypass capacitors on all the connections.

However of all the data obtained from the various experiments performed there was one that stands out as the exception. This is the case of the electrical response measured using the original assembly. In this data it can be observed a constant and linear response of the material in all the experiments executed: the material, without any kind of amplification, it is capable of generating a voltage which reaches 2.5 and 3 volts on average in all the cases. This answer assumes a respectable value comparing with voltage responses obtained with another type of ceramic piezoelectric such as PZT when used under similar conditions to the proposed system: although the piezoelectric coefficient of PVDF material is much smaller than those exhibited by commercial piezo-ceramics, this material represents a good substitute of these materials because of the mechanical strength presented, its biocompatibility, low density, lightness and structural capabilities [56].

The reason why this positive electrical response is generated in the material could be explained by several features of the assembly in comparing with the assembly designed for DC and AC analysis: insulation of the material, no use of analog conditioning circuits that generate noise and interference in the system and the likelihood situation that the free fall impact of the metal object is large enough to generate a temporary alignment in the molecules of the PVDF, making its internal molecular structure β phase for a few seconds.

In the case of this last experiment, if the data obtained from the PVDF is analyzed and compared with, for example, the results obtained from the piezoelectric used as monitoring, in this case measurements taken of the strain gauges in the DC experiment, it can be denoted that the response coming from the PVDF has a higher variation of voltage, approximately of

2.5 V while the electrical response from the strain gauges that has been amplified, has a voltage variation of 1.3 V maximum. This data comparison shows that piezoelectric polymers with semi-crystalline structure as PVDF may be considered good candidates to replace other piezoelectric materials as sensors, actuators and transducers.

vi. Future experiments

After having conducted several experiments in order to obtain the data necessary for the characterization of the material as a piezoelectric sensor, several aspects have to be taken into account when performing new experiments with the material:

- It must be ensured that the phase of the PVDF is the β phase, which has a higher dipole moment; in the event that the samples are not at this phase they must be polarized by any of the methods previously discussed in this description.

- The system must be properly insulated to prevent measurement errors produced by a very high noise signal.

A future study could be, taking into account these aspects, analyzing again the PVDF material in a cantilever beam to characterize it as piezoelectric pressure sensor using a digital system of data collection as Matlab or LabView.

V. ANNEXES

i. Project planning

The project arose when the Department of Materials Science and Engineering and Chemical Engineering performed the first piezoelectric experiment with manufactured samples of PVDF [16], and then presented the idea to my tutor, Pablo Acedo Gallardo, who raised me the idea to characterize the material for its use as a sensor in the latest April of 2015.

From that time meetings began with our liaison at the department, Francisco Javier González Benito with whom it was discussed the approach that it could be given to the project and how it would be the ideal way to prepare it in a system where measurements could be obtained. The idea suggested was a similar system to a cantilever beam. Meanwhile the investigation about the PVDF material started, using various sources as books or Internet.

In early May, it had already been designed the concept to study and the preparation of the installation and preparation of the metal plates where the PVDF samples would be placed started, with the help of Freddy Ariel Sanchez Ruiz; in these same plates the workers at the laboratory of the Department of Electronics Technology put the strain gauges.

During the month of June conditioning circuits of the strain gauges and amplifiers and all the electronic conditioning of the PVDF needed for its use as piezoelectric sensor were designed.

In the months of July and August the whole system was assembled and measurements were taken. After the holiday break in August, measurements were taken until early September. In the middle of this month the results were analyzed and it was decided to rethink the design of the sensor and take further steps in the laboratory with the help of Julio Posada until the results were raised again at the end of September. The memory began in June and is planned to end in late September of 2015.

	Months					
Tasks	April	May	June	July	August	September
Project approaching						
Meeting with the Materials Department						
Knowledge acquisition about PVDF						
Concept design						
Preparation of samples						
Preparation of metal plates						
Samples and strain gauges gluing						
Conditioning circuits design						
Developing of the assembly and circuits						
Measurements acquired						
Re-design of assembly						
Measurements acquired						
Results obtained discussion						
Bachelor Thesis						

Table 4: Planning graph of project

ii. Estimated budget

The following budget is presented in differentiate chapters where it can be seen the different approaches needed in the project, defining: assembly chapter, where all the materials used to fabricate the system are listed; electric components chapter describes all the elements used in the circuits that characterize the material; chapter three is the one that estimated the price of the measurements devices used during the whole experiment and lastly, chapter four groups all the elements that were not included in other chapters.

Chapter 1: Assembly			
Name	Price per unity (€)	Units (u)	Total price (€)
Wood assembly	10	1	10
Metal plates - **Aluminum** - **Copper** - **Brass** - **Steel**	0,25/m	4m	1
Electric metal cutter	50	1	50
Electric metal sander	30	1	30
Power drill - **Drill set**	150 27	1 1	150 27
Screw	0,1	5	0,5
Nut	0,3	5	1,5
Adhesive double layer tape	22	1	22
Screwdriver	5	1	5
Wire cutter	9	1	9
Set of eight weights	10	1	10
		Total Price of Chapter 1 (€)	**316**

Chapter 2: Electric components			
Name	Price per unity (€)	Units (u)	Total price (€)
Amplifiers - **AD620** - **TL081** - **Lock-In**	 4 0,3 4150	 3 2 1	 12 0,6 4150
Wire	0,5/m	4m	2
Resistance	0,1	20	2
Strain gauge	5	4	20
Capacitor	0,25	10	2,5
Potentiometer	0,25	2	0,5
Diode	0,15	1	0,15
Ultrasonic transducer	2	1	2
Function generator	1700	1	1700
Speaker	45	1	45
		Total Price of Chapter 2 (€)	**5936,75**

Chapter 3: Measurement devices			
Name	Price per unity (€)	Units (u)	Total price (€)
Oscilloscope	1500	1	1500
Computer	450	1	450
Acquisition of data card	130	1	130
License of program	50	1	50
Multimeter	106	1	106
		Total Price of Chapter 3 (€)	**2236**

Chapter 4: Other			
Name	Price per unity (€)	Units (u)	Total price (€)
PVDF	62,5	4	250
Laboratory equipment	5000	1	5000
Chemical technician	1200	1	1200
Double layer conductive tape	14,50	1	14,50
Graduate engineer	7700	1	7700
		Total Price of Chapter 4 (€)	**14164,5**

Tables 5-8: Estimated budget represented by chapters

The total estimated price is presented in the following table:

Estimated budge	
Chapter 1	316
Chapter 2	5936,75
Chapter 3	2236
Chapter 4	14164,5
Total estimated price (€)	**22653,25**

Table 9: Total estimated budget of project

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