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METHOD FOR MEASURING PIEZOELECTRIC CHARGE COEFFICIENTS

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AUTHOR'S DECLARATION

I hereby declare that this thesis is the result of my independent work.
On the basis of materials not previously applied for an academic degree.
All materials used in the work of other authors are provided with corresponding references.

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The work meets the requirements for a master's work.

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Eessõna

Lõputöö teema on väljakasvanud projektist „Carbon Nanotube Reinforced Electrospun Nanofibers and Yarns“ ehk „Elektroketruse teel valmistatud süsinik-nanotorudega armeeritud nanokiud ja lõngad“. See projekt viidi läbi Tallinna Tehnikaülikooli Polumeermaterjalide instituudis. Selle projektiga oli seotud mitmeid ettevõtteid: Estfil Tehno AS, Fein-Elast Estonia AS ja Estiko Plastar AS. Projekti eesmärgiks oli leida meetodeid ja tehnoloogiaid nanokiudude elektroketramiseks ning nendes kiududest lõnga ning tekstiilide valmistamiseks. Üks projekti väljund oli piezoelektriliste omadustega nanokiud. See tekitas vajaduse piezoelektriliste omaduste mõõtmemeetodi loomiseks. Katsetused viidi läbi Tallinna Tehnikaülikooli Polumeermaterjalide Instituudis. Autor avaldab tänu Mihkel Viirsalu ning Andres Krummele, kes osalesid lõputöös nõuandjatena.

Foreword

The work of this thesis has arisen from the project „Carbon Nanotube Reinforced Electrospun Nano-fibers and Yarns“. This project was conducted in Tallinn University of Technology Department of Polymer materials. There were several commercial partners from Estonia: Estfil Tehno AS, Fein-Elast Estonia AS and Estiko Plastar AS. The purpose of this project was to find methods and technologies to produce yarns and textiles from electrospun fibers. One of the desired results was a nano-fiber with piezoelectric properties. This generated a need for a measuring method to check if the created fibers had the necessary properties. The experiments for this work have been performed in Tallinn University of Technology Department of Polymer materials. The author thanks Mihkel Viirsalu and Andres Krumme who participated in this work as advisors.

Introduction

This thesis was chosen due to the need for a measuring method for piezoelectric materials that have been created in Tallinn University of Technology's Department of Polymer Materials. The created materials are a result of a research project „Carbon Nanotube Reinforced Electrospun Nano-fibers and Yarns," that is using different substances to increase different properties of electrospun polymer fibres. There are few commercial devices that are designed to measure the piezoelectric properties but they are mainly for hard solids but not for fibers or textiles. Additionally the existing devices cost several thousand Euros which gave rise to the idea of developing a prototype to evaluate the properties of the piezoelectric mats in an economically more viable way. The main providers of quasi-static d_{33} coefficient measurement devices are H. C. Materials Corporation and Piezotest. Both of them offer high quality devices for a wide variety of applications.

Piezoelectric electrospun materials have been progressively more frequent topic for scientific articles and projects all over the world [1]. This is due to the fact that electrospinning is becoming more widely used and researched method for creating nanofibres. The use of these materials in everyday life is limited by the overall productivity of electrospinning processes and the lack of research of the created fibers.

There are three main methods of measuring piezoelectric properties. They are the following: Frequency method measurement, Laser interferometry method and Quasi-static method. All of these methods have their preferred fields of usage and they are briefly discussed in the first part of the work. This thesis is going to focus on Quasi-static method because of the availability of the equipment needed and lower overall cost compared to the other methods [2].

This thesis develops the preliminary method that could be used in Tallinn University of Technology to measure piezoelectric coefficients of piezoelectric materials including the electrospun nanofibre mats in an economical and accessible way.

The first part of the thesis focuses on the theoretical background of the piezoelectric effect. The relatioship between mechanical deformation and electricity in piezoelectric materials is examined. An investigation of the importance of polarization and dipole placement in the material is done. Piezoelectric charge constants d_{ij} and their use in comparing piezoelectric materials are discussed. A short overview of electrospinning's main principle and the piezoelectric effect in electrospun PVDF fibers is given. The main uses of different piezoelectrical materials and their properties are shown. A brief overview of the three main piezoelectric material measuring methods are presented.

The second part explains the use of quasi-static method for measuring electrospun PVDF fiber mats. A functional diagram and the process of measuring with the proposed measuring method are given. Overview of the mechanical components used and their purpose is examined. An extensive study on the electronic components that are to be used in the measuring system is done. Load cell properties and responses to static and dynamic measurements are studied. Description of the charge amplifier and its working principles are discussed. Additionally the electrical circuit that was used to test the measurement method is studied.

The third part explains the LabVIEW™ program used to collect data and control the measuring system. User interface description and the functions of the program are shown.

Fourth part consists of testing and describing how the validation of the measuring method would be done. Material properties and the charge coefficients on the validation material NCE51 are given. The weigths, capacitors and Data Acquisition Device used for the adjustments and characterization of the measuring system are described. The method for adjusting the load cell and characterization of the charge amplifier are explained. Uncertainty analysis is conducted and the measurement uncertainty of the system is estimated.

The fifth part proposes a design for the measuring device that consists of a PCB board and a 3D model for further development processes.

The sixth part shows the cost of the components that are requiered to construct the measurement device proposed in the fifth part.

This work required the use of LabVIEW™ 2011, which is a visual programming language, Eagle Light7.2.0 PCB design software and CAD program SolidWorks®.

1. Measurement of piezoelectric coefficients

1.1 Piezoelectricity

Piezoelectricity is a well-known material property. Most common household item that uses piezoelectricity is the lighter; more specifically the part of the lighter that ignites the gas. When one pushes the lighter button a small hammer is loaded and released with high speed to impact the piezoelectric crystal residing inside of the lighter. Once the crystal is hit with enough speed it produces a charge. Depending on the strength of the collision the charge produce is either bigger or smaller. If the charge is big enough an arc is created between electrodes connected with the piezoelectric crystal and the gas is ignited [3].

Piezoelectric materials are used in many different applications as sensors or mechanical actuators in addition to generating electricity. Some examples of sensors are microphones, sonar and pressure sensors. As mechanical actuators piezoelectric materials are used as ultrasonic wave generators, micro motors and other applications [3].

1.2 Basic principles of piezoelectricity

The word "piezo" is derived from the Greek word ”piezin”, meaning to press. In 1880 brothers Jacques and Pierre Curie reported about an existing relationship between mechanical load and electric polarization in a number of crystals such as quartz and tourmaline. In the year 1881 the now commonly use term “piezoelectricity” was proposed by Wilhelm G. Hankel [3].

Piezoelectricity is present only in materials with special crystalline structures. The piezoelectric effect occurs both in monocrystalline materials and in polycrystalline ferroelectric ceramics. The structure needs to have crystals without a center of symmetry. On Figure 1.1 the non-centrosymmetric atomic structure of the PZT crystal is demonstrated [4].

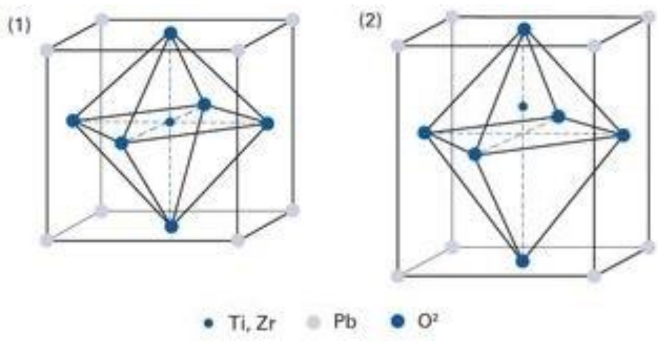

Figure 1.1. Non-centrosymmetric atomic structure of a PZT crystal [4]

Piezoelectricity can be observed in two different ways: the direct and the converse piezoelectric effect. The direct effect is observable when the crystal stricter of the piezoelectric material is mechanically deformed and produces an electric charge on the opposite faces of the material [3]. The converse effect means that when an electric field is present the material is deformed [3].

1.3 Piezoelectric materials

There are many possible piezoelectric materials to choose from but only a number of them have been proven suitable for sensors and actuators. Ideally these materials should have the following properties among others [3]:

- high piezoelectric sensitivity
- high mechanical strength
- high rigidity
- high electric insulation resistance
- linear relationship between mechanical stress and electric polarization
- high stability of all properties
- low production cost

The importance of these individual properties is discussed in the book "Piezoelectric Materials for Sensors" in more detail.

Natural materials tend to have weak piezoelectric properties. That is why materials used in industries today have been developed by researches to exhibit larger displacements or induce larger electric voltages [4]. The most common piezoelectric materials used are Polycrystalline ferroelectric ceramics such as barium titanate ($BaTiO_3$) and lead zirconate titanate (PZT). One can modify PZT to achieve specific properties for a variety of different applications.

PVDF (polyvinylidene fluoride) is used as a piezoelectric sensor or an actuator where softer and more malleable material is required. PVDF could be electrospinned which means it is in nanofiber form and could potentially be fabricated into textiles if the technology evolves further. At the moment there are only mats made of said PVDF nanofibers.

1.4 The piezoelectric effect

This effect is the reason why piezoelectric materials have a relationship between mechanical deformation and electricity. The effect arises from the fact that certain materials have molecules with no center of symmetry. Polarization causes one end of the molecule to have a positive charge and the other end have a negative charge. This type of molecule is called a dipole. The piezoelectric effect is dependent on the polar axis which is an imaginary line that connects the center of both charges in the molecule. In a monocrystal the polar axes in all the dipoles are lined in one direction. The crystal is said to be symmetrical because if you were to cut the crystal at any point, the polar axes of the cut pieces would be identical to the original. However in polycrystals, different regions of the material have varying degrees of polar axes. Therefore polycrystals are called asymmetrical because there is no point at which the crystal could be cut that would leave the two remaining pieces with the same resultant polar axis [5]. In Figure 1.2 the difference between monocrystal and polycrystal polarization is shown.

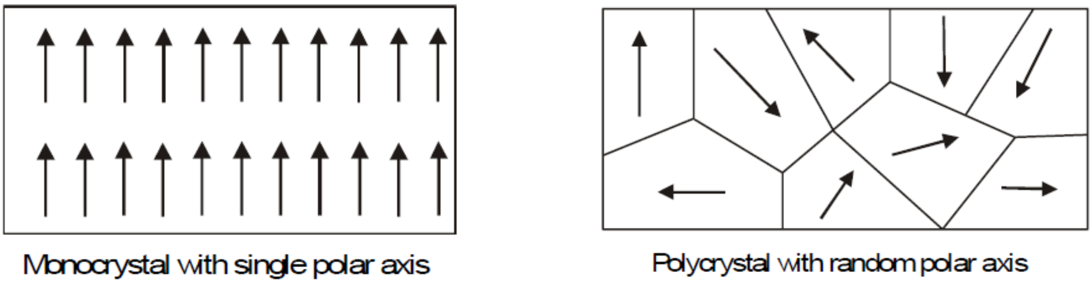

Figure 1.2 Mono vs. Poly Crystal polar axes [5]

Polycrystals with random polar axis are not usable in applications which require piezoelectric effect due to the fact that randomly oriented dipoles tend to eliminate the effect altogether. To achieve high piezoelectric sensitivity the material should be polarized.

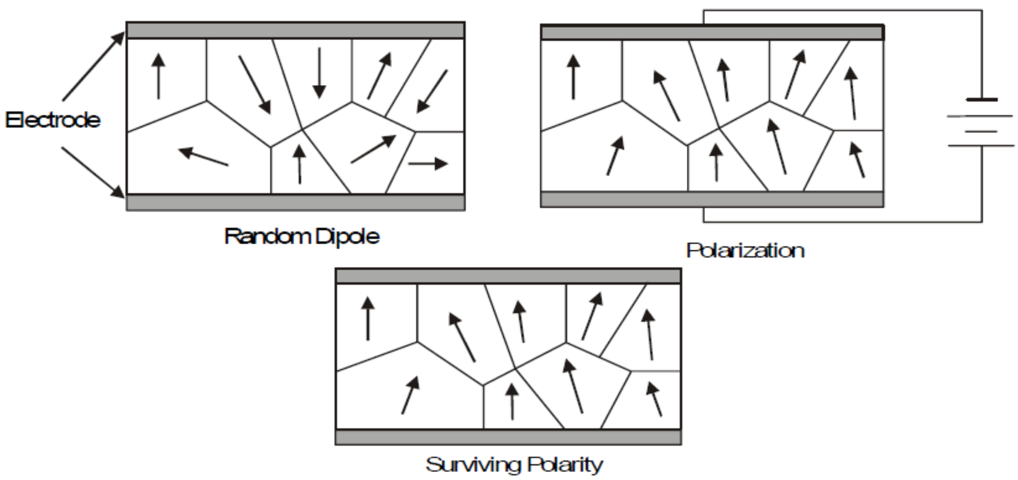

Figure 1.3. Polarization process of polycrystal material [5]

The process of polarizing is as follows (Figure 1.3). First the material is heated to provide enough room for to the molecules so they could realign when a strong electric field is applied over the material. The electric field forces all the dipoles to line and face approximately the same direction [5].

1.5 Piezoelectric charge constants d_{ij}

As described in the Chapter 1.4 the dipoles in piezoelectric materials are arranged to align with the electric field during the poling process. This alignment inside the material will remain constant but the mechanical stress that is applied to the material during its use changes. That is why to compare different piezoelectric materials charge constants are used. Piezoelectric materials are anisotropic which means that their physical constants (elasticity, permittivity etc.) are related to both the direction of the applied stress, electric field etc. and to the directions perpendicular to these [6]. On the Figure 1.4 the piezoelectric material designations and directions are given for axes and deformations respectively.

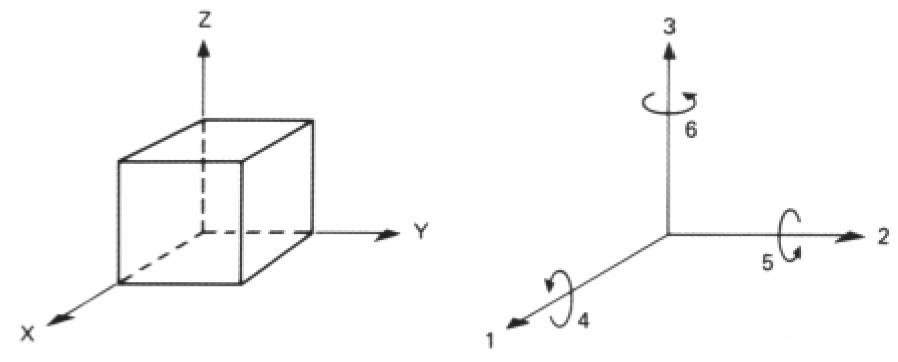

Figure 1.4. Designation of the axes and directions of deformation [6]

These designations and directions are used to mark different charge constants; which are defined as the electric polarization generated in a material per unit of mechanical stress applied to it [6]. This means that the first subscript *i* shows the direction of polarization in the material and the second *j* refers to the direction of the applied stress. For example:

d_{33} – shows that both the polarization and the mechanical stress are aligned in the direction 3 on Figure 1.4.

d_{31} – shows that the polarization is in the direction 3 and the mechanical stress is aligned with the direction 1 on Figure 1.4.

The piezoelectric materials are the most effective when the mechanical deformation occurs in the direction of the aligned dipoles. In general the material should be used in the same direction as the polarization.

1.6 Piezoelectricity of PVDF

1.6.1 Electrospinning history

This work grew out of the research done in TUT's Department of Polymer materials where they are working with electrospinning technologies. One of the outcomes of that research was the use of PVDF to create piezoelectric nanofibre mats and in the future to use PVDF for piezoelectric yarns and textiles created from said fibers.

Electrospinning principles are known for a long time dating back to the 19 century; but have not been used in large scale industries due to the small amount of fibers that can be produced. In 1897 Rayleigh was the first to observe the process of using electricity to induce the movement of polymers. Then in 1914 Zeleny studied electrospraying in more detail. The process of electrospinning was patented by Formhals in 1934. In the year 1969 work done by Taylor who studied electrically driven jets laid the foundation for the electrospinning process known today. The term electrospinning became more frequent in the year 1994 [7].

PVDF has been the object for research extensively due to its electroactive properties. PVDF is semicrystalline polymer with a typical crystallinity of 50% and whose molecular structure is formed by the monomer (-CH_2CF_2-) [8].

1.6.2 Electrospinning principles

Electrospinning uses a variety of different polymers, ceramics and composites in the solute that can be electrospinned [9]. In TUT Department of Polymer materials the research is mainly done with polymers. To electrospin a polymer it has to be solved in a solvent.

In the case of PVDF the solvents most commonly used in TUT are the following: DMF (Dimethyl formamide) and DMSO (Dimethyl Sulfoxide). Once the polymer has been solved in the required ratio with the solvent it can be electrospinned by pressing the polymer solution out of a syringe, while charging the needle with sufficiently high voltage. These voltages could vary from 5 kV to up to 120 kV in big assemblies. The necessary voltage also depends on the properties of the polymer solution used. The created fibers are collected onto the electrically grounded collector for easy removal of the final product. The previously described method is the most basic and simple version of electrospinning. There are ample versions of this method but all of them rely on these simple necessities. The process of electrospinning is illustrated on Figure 1.5.

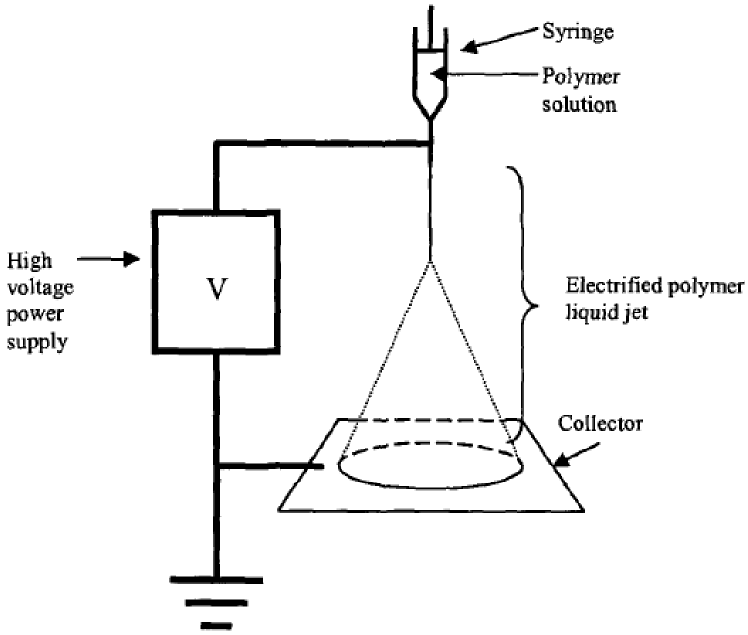

Figure 1.5. Basic setup of elctrospinning process [9]

1.6.3 Piezoelectric effect in electrospun PVDF fibers

As previously explained in Chapters 1.2 and 1.4 common piezoelectric materials need some form of after treatment to give rise to piezoelectric effect in those materials. This is not the case with electrospun PVDF fibers. Thanks to the nature of electrospinning process the poling of fibers is done during the electrospinning. This means all the dipoles in PVDF fibers are aligned with the electric field while electrospinning said fibers. On the right side of the Figure 1.6 the electrospinning of PVDF fibers is shown.

The piezoelectric effect in PVDF is possible due to the distinct crystallite polymorphs this material has. There are five different polymorphs in total, but only the one of them is useful. The most common polymorph is the α-phase. The PVDF that has mostly this phase, has almost no remarkable properties. The second and the most important polymorph is the β-phase. Only this polymorph has the correct crystalline structure where the dipoles are naturally aligned to the chain axis. This generates the largest spontaneous polarization and piezoelectric properties. Unfortunately achieving 100 % of β-phase inside the PVDF is difficult and that is why there are usually at least some parts of the PVDF material that has an α-phase [8].

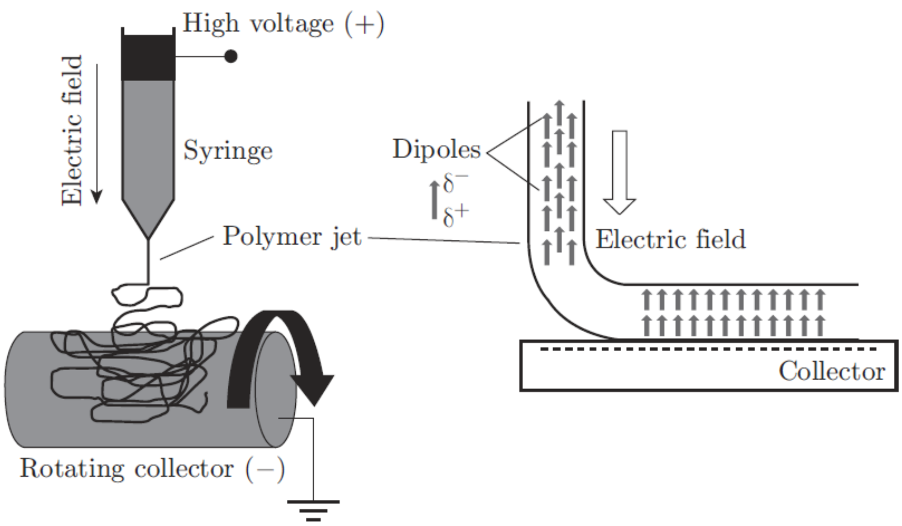

Figure 1.6. Electrospinning PVDF fibers and collecting them on the collector on the right side and on the left the alignment of the dipoles due to the electric field [10]

1.7 Piezoelectric sensors

Piezoelectric sensors are a part of a group of active sensors, which means that they do not need an external source of power to output a signal. One could consider piezoelectric sensor to be an active capacitor that charges itself when mechanically loaded [3].

There are many depictions for the piezoelectric sensor. As shown on Figure 1.7 these models are using a resistor and a capacitor in different configurations depending on whether the piezoelectric sensor is depicted as a charge source or a voltage source [11].

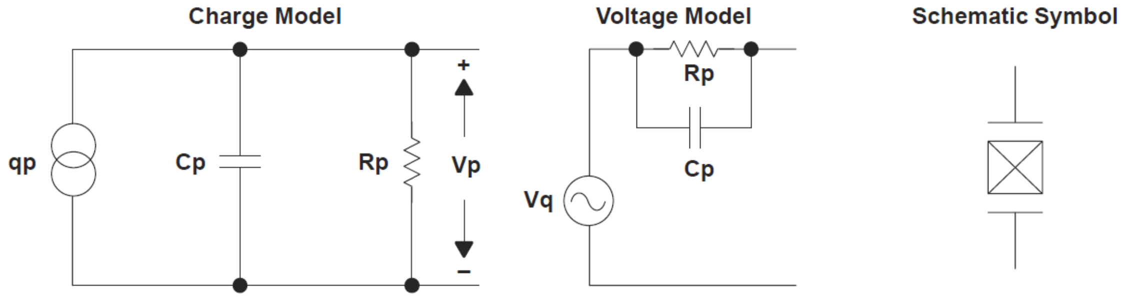

Figure 1.7. Piezoelectric sensor models [11]

One interesting feature of the piezoelectric sensor is the absence of a zero point for measurement. This is due to the fact that the sensor is always loaded with another acting force. To explain, consider a force sensor under a constant load. The charge generated will eventually dissipate because no material has an infinitely high insulation resistance. Now when a new load is applied the measurement will refer to the "new" zero point. This property allows measuring of the constant load after an indefinitely long time by removing the load from the sensor and measuring the negative force step as an output [3].

1.8 Measuring methods for piezoelectric materials

When designing piezoelectric sensors and actuators one usually needs to test and verify the properties of the materials used. For this three different types of measuring methods are considered the most effective. The three most common methods are the following:

- Frequency method measurement
- Laser interferometry method
- Quasi-static method

These methods are prefferred due the their accuracy. The downside is that they are heavily dependant on the expensive measuring devices [2]. The most common materials tested with these methods are of ceramic nature. For example PZT. There are other possibilities aswell but they are not very wide spread at the moment.

1.8.1 Frequency method measurement

Frequency method measurement is the most complicated and expensive but also the most accurate of the three methods. The method requires samples of specific size and shapes to be effective. One has to have a disc, a plate and a cylinder from the piezoelectric material that is tested to have an accurate measuring result. There are commercially available devices for this method as well. The most common ones are made by Agilent and Wayne Kerr [2]. This method of measurement is not suitable for PVDF nanofiber mats because of the required shapes of the samples.

1.8.2 Laser interferometry method

This is the most common measuring method at the moment. This method is based on measuring the displacement deflection of a sample surface after voltage connection to the electrodes of the measured piezoelectric material [2]. The drawbacks of this method are high requirements for the measuring device itself, because any small irregularities or vibrations while measuring will corrupt the accuracy of this method a lot. Interferometers for measuring are made by Polytec, Lasertex, Agilent and many others.

This method was not used because the method requires a high quality reflection surface on the sample [12].

1.8.3 Quasi-static method

This method is the cheapest method of the three. There is no need to establish all the material constants involved and the test can be done with only one sample. To have an accurate result the tested sample needs to be compared to a reference sample of a known piezoelectric coefficient. There are different setups for this method but the general principle remains the same [2, 13]. The method uses charge amplifiers or high resistance input voltmeters to measure the charges created when the sample is mechanically deformed. Commercial measuring devices are offered by companies like Piezotest and HC Materials Corporation [14, 15]. This method is the preferred one because of the simpler design of the measuring device and the necessary knowledge to do the tests.

1.9 Commercial d_{33} meter comparison

In Table 1.1 a comparison between two common quasi-static piezo d_{33} measurement devices is given. The most compelling reason behind trying to build a measurement system instead of purchasing one is the cost of the existing commercial devices.

Table 1.1. Comparison of JZ-6B and PM100 d_{33} meters [14, 15]

Manufacturer	**H. C. Materials Corporation**	**Piezotest**
Model	JZ-6B	PM100
Price	**6087 EUR**	**13090 EUR**
Range	X1 (20…4000) pC/N X0.1 (2…200) pC/N	X1 (10…1000) pC/N X0.1 (1…100) pC/N
Accuracy	±(2 % +1 to 3 count for d_{33})	X1 ±(2 % +1 pC/N) X0.1 ±(2 % +0,1 pC/N)
Sample size	80 mm in polarization direction	50 mm in polarization direction Maximum diameter 136 mm
		

2. Measuring system

2.1 Realization of the quasi-static method

The first goal was to understand how this method works and how it could be realized in TUT. For this the main sources were articles that were discussing the process of measuring electrospun PVDF fiber mats [16, 17]. The main difference between regular piezoelectric materials and mats created in TUT were that the ceramic piezoelectric material has a uniform body of solid material. Electrospun mats are made from randomly oriented PVDF fibers which are physically weak. This creates the need for having fixtures that will not destroy or deform the sample. These fixtures can have many forms as shown in articles [16, 17, 18]. The method of deforming the sample varies also. Ceramic piezoelectric materials could be safely bent or pressed and the sample would stay relatively the same. Fibers will deform with every test and therefore one should design the fixtures in a way where the least amount of permanent damage is done to the samples. One of the safest ways is to place the sample between to metal electrodes while the pressing of the sample is done. This method allows using the sample more than once; when the deforming forces are not damaging.

In the following chapters the choosing of mechanical and electrical components for quasi-static method measuring is conducted.

2.2 Functional diagram of the measuring system

The process of measuring the piezoelectric coefficient with the measuring system in this work is shown as a diagram on the Figure 2.1. Choosing of the sample is the first step and the most important one. Quasi-static method is able to measure the piezoelectric coefficient only on samples of sufficient size and strength because the exerted force will destroy samples that are too fragile or thin. The optimal sample size for the system in this work is based on the size of the PVDF mats that researches are creating in TUT laboratories. The PVDF fiber mats are not big because it takes a lot of time to produce large quantities of electrospun fibers. The average mat is 100 mm by 50 mm in size. From there one could extract 5 samples with sufficient quality. This means the samples used in the measuring system should be between 10 mm and 20 mm in length and 10 mm to 15 mm wide. Thickness could be as thin as 0,3 mm but is entirely depending on the strength of the material. Maximum thickness should be less than 2 mm.

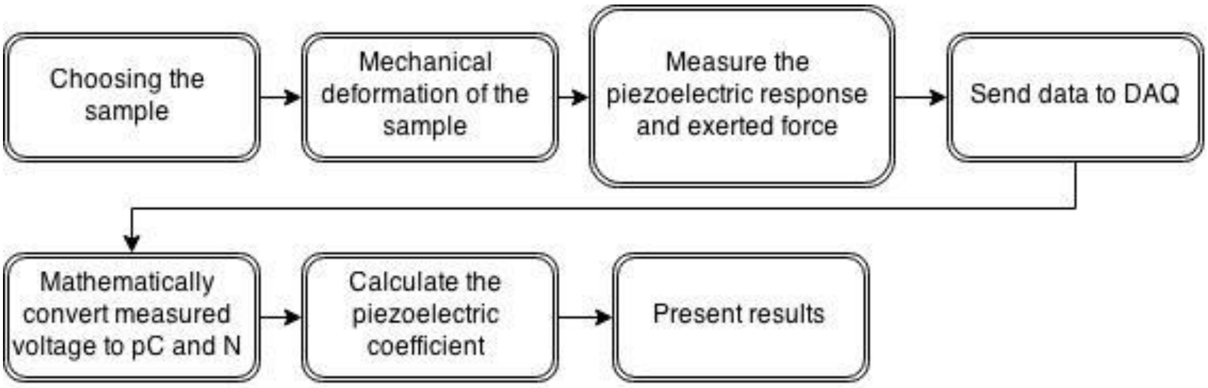

Figure 2.1. Functional diagram of the measuring system

When the sample of sufficient size and shape has been chosen the testing can begin. First the sample is deformed by the movement of the solenoid's plunger. One could use frequencies from 0,5 Hz up to 1 Hz to exert force with the solenoid. A test that controls the load cell capabilities to work with dynamic loading is done in the Chapter 2.4.4.1. The load cell is used to measure the value of the force exerted onto the sample. The forces that could be used vary from 7 N to 12 N.

The piezoelectric response is recorded by the charge amplifier and then sent to the DAQ (Data Acquisition Device) USB-1616-HS-2. This DAQ is connecting the analog circuit of the load cell and charge amplifier to the computer and from there to the LabVIEW program. In the LabVIEW program the measured voltages are represented graphically on the front panel. The program then converts the voltages into nC (nano Culon) and N (Newton) for representing the charge measured by the charge amplifier and the exerted force. With these values one can calculate the piezoelectric coefficient and present the results in an Excel spreadsheet.

2.3 Mechanical components

The measuring method needs a system which can apply pressure to the samples with regulated force and preferably controlled frequency. To achieve this it was decided that a solenoid will be used to exert the force due to the simplicity of the parts necessary for such mechanism. Polymer Material Institute had an existing modular milling machine which had the required dimensions and rigidity to support the solenoid, sample and the load cell. The aluminium frame that holds the solenoid, load cell and the sample, has manual actuators that are used for regulating the system. One actuator is used to regulate the height of the solenoid and is found on the top of the rig shown on Figure 2.2. The load cell can be moved in one fixed plane in X and Y dimension by turning either the bottom left actuator for Y-axis and the bottom right actuator for X-axis manipulation.

To keep the solenoid away from the sample and give more room to operate with the system a pressing tip made out of teflon was added to help to center the exerted force on the top of the electrode. This helps to distribute the force more evenly over the sample tested.

To simplify the fixing of the samples a fixture for the electrodes was also milled. It has a rectangular opening to limit the possibility of the sample sliding and beign torn while testing. The top electrode is hold in place by a leaf spring.

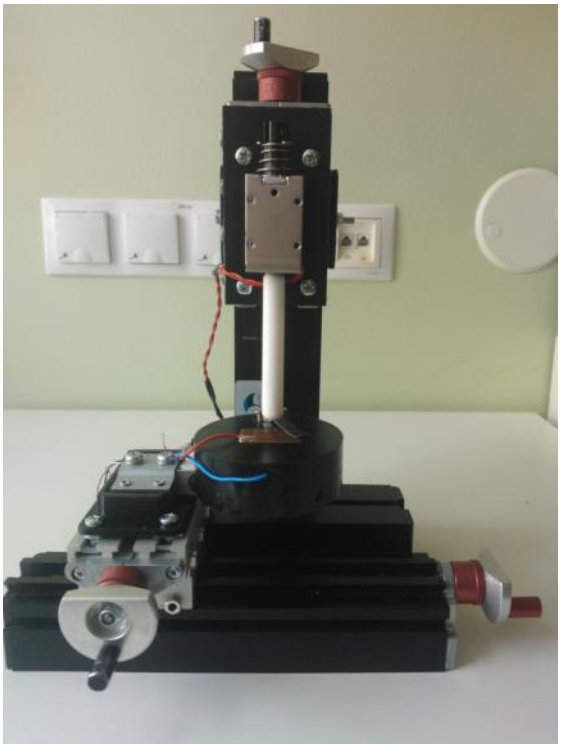

Figure 2.2. Frame of the milling machine for the measuring device

2.4 Electronic circuit

The entire electronic schematic is shown in Appendix A. It depicts the connections between the following microchips: L293B, INA125 and TL071. There are also connections to the power supplies, the load cell and to the DAQ. The connections between the DAQ and the circuit are shown in Table 2.1.

The Piezoelectric sensor is represented on the schematic as a connector. Diode D1 is used to protect the L293B microchip from the current flowing back from the solenoid when it is returning to its neutral position. Resistor R1 is a trimmer potentiometer to adjust the gain of the INA125 amplifier to have the necessary accuracy for the load cell measuring circuit. Resistors R2 and R3 are used as pull-up and pull down resistors respectively to keep the L293B ready for any PWM signal from the DAQ.

Table 2.1. Connections from the circuit to the DAQ

Number on the schematic	DAQ I/O channel
1	Analog ground
2	Analog output 0
3	Analog Channel 0 High
4	Analog Channel 0 Low
5	Analog Channel 1 High
6	Analog Channel 1 Low

Resistor R4, connected to the ground, stabilizes the PWM pin on the L293B when the DAQ is not actively sending signals to the chip. R5 is required to slowly draw current from the capacitor C2 so the piezoelectrically generated charge can be measured. R6 reduces the electrical noise coming from the connections of the sensor and is a part of the high cutoff frequency calculations. Resistors R7 and R8 make up the voltage divider to provide a middle reference point of approximately 2.5 V so that the values coming from the sensor won't saturate the output of the TL071. Capacitor C1 is to remove any disturbances coming from the power supply to TL071. C2 capacitor is the main component of the charge amplifier and is collecting the charges generated by the piezoelectric material.

2.5 Electronic components

All the electronics for this system has been setup on a prototype board. The components were chosen to have TTL-compatible logic inputs which mean that they have the ability to be controlled with microcontrollers by using 5 V outputs. Another aspect of choosing was to make sure all the components are capable of working without a heat sink. These requirements make the building and using of the prototype board much easier and faster.

2.5.1 L293B quad push-pull driver

The purpose of this component is to receive PWM (Pulse Width Modulation) signals from the controller and drive the solenoid or an electric motor with a maximum output current of 1 A per channel [19]. This driver is capable of driving four DC motors in one direction as long as the output current does not exceed 1 A. Alternatively one could drive two DC motors in either direction while staying under the same maximum current limit. These options were important in the prototyping phase when different solutions were explored. The solenoid uses 24 V for operation and this is well under the absolute maximum limit of 36 V that this driver could potentially handle. In the prototype the current does not exceed 500 mA and therefore this driver could be run continuously and without any harm to the driver. The connection diagram of the L293B microchip is given on Figure 2.3.

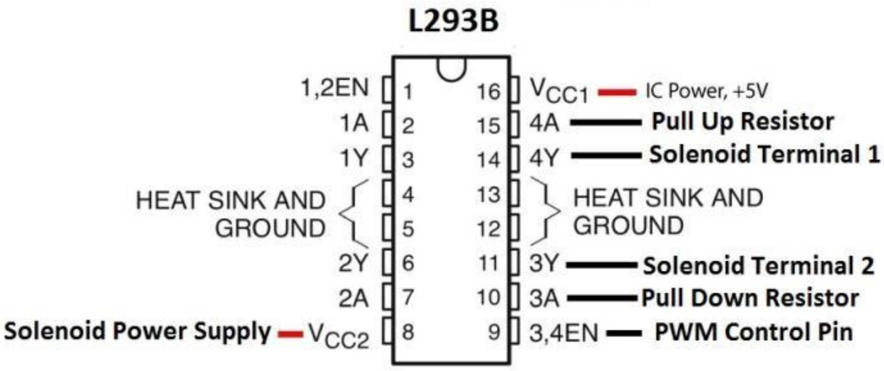

Figure 2.3. Connection diagram of L293B

2.5.2 Force measuring

To measure the force a load cell is used. To excite the load cell a special amplifier is used to provide 5 V input into the load cell and measure the change in output voltage when the load cell is mechanically deformed. INA125 is a low power and high accuracy amplifier. It is used in industrial process control, factory automation and general purpose instrumentation [20]. INA125 in a 16-PIN DIP package; which means it can be easily mounted on the prototype board for quick assembly.

2.5.3 Load cell

Load cells are widely used in digital scales and other devices which require the measurement of force or mass. The measurement is done by a strain gauge which is usually attached to an aluminum fixture which has specially designed dimensions to provide controlled deformation in the required direction. Strain gauge shape changes when the fixture is deformed. This deformation changes the resistance of the strain gauge and could therefore be measured. These strain gauges are usually mounted in groups of four to form a full Wheatstone Bridge [21].

The load cell in use was one from a household scale with a maximum measurable mass of 5 kg. The choice to use a simple load cell was due to the simplicity of the prototype. When the load cell is correctly adjusted it has enough accuracy and reliability to finish our preliminary tests and measurements. The load cell has four wires where two of them are for excitation and two for the signal output. The load cell is shown on Figure 2.4.

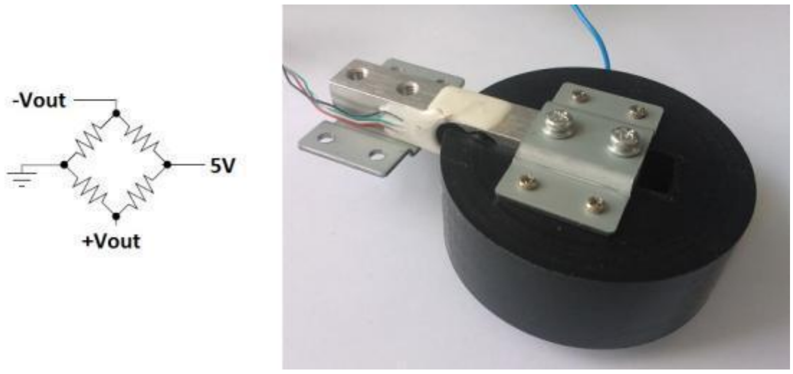

Figure 2.4. Load cell symbol on the left and load cell with the sample holder on the right

2.5.4 Setting the gain for INA125

The gain, G, for INA125 can be set with only one external resistor, R_G, between pins 8 and 9 [20]:

$$G = 4 + \frac{60k\Omega}{R_G} \quad (2.1)$$

G – Gain

R_G – External resistor (Ω)

The gain is adjustable in the range of 4 up to 10000 and is responsible for the amplification occurring inside the amplifier. Adjustment of the prototype can be done by the resistor, R_G, which is a potentiometer with the resistance range from 0 Ω to 200 Ω. Calculating the gain with the above formula 2.1 shows that the available gain is from 304 to 10000. In the process of adjusting the force measurement the potentiometer is used to regulate the output voltage to the desired level to have the required measurement range. The connection diagram for the amplifier INA125 is shown on Figure 2.5.

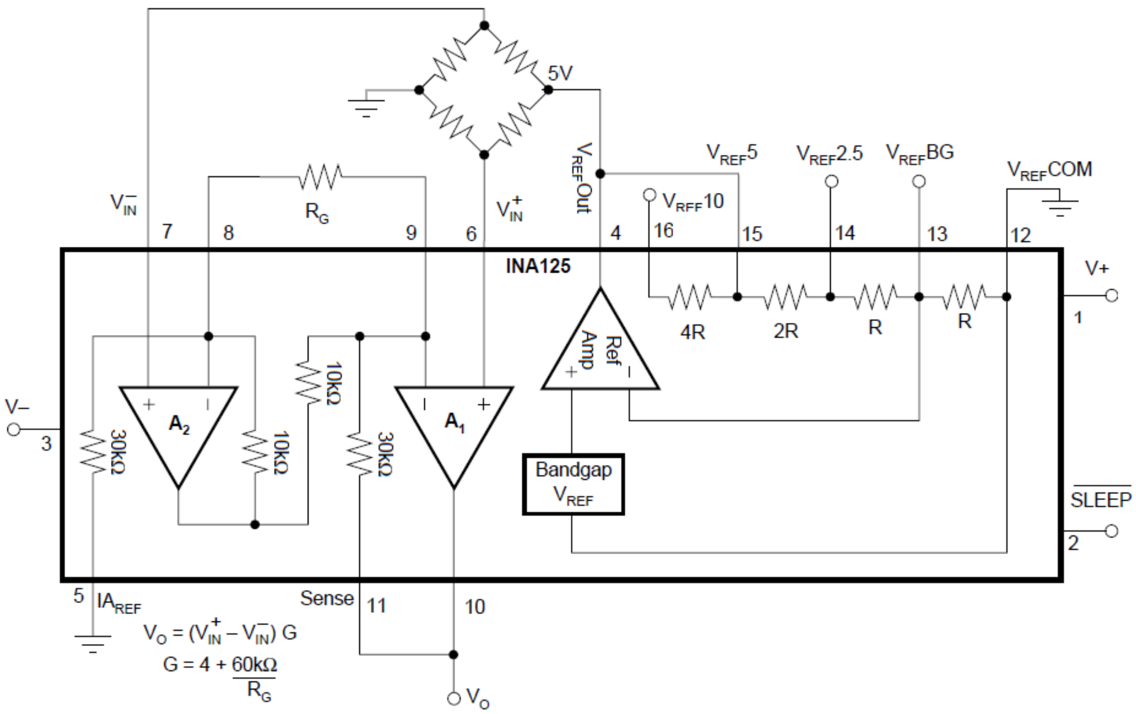

Figure 2.5. INA125 connection diagram [20]

2.5.4.1 Testing the static adjustment in the dynamic mode

The adjustment is done with fixed weights in a static measurement. But to test the piezoelectric material the load cell will work dynamically and therefore oscillate between each push by the solenoid. To make sure that the oscillation has subsided before the next measurement a set of tests was conducted. After adjusting the load cell output with static weights the solenoid is used to press the load cell with increasing frequency. Figure 2.6 depict the output of the load cell amplifier when the solenoid moves at the frequency of 1 Hz. The oscillation that occurs when the solenoid returns to its neutral state below 0,2 V is not affecting the end result. When 7 N of force is used the amplifier's output is 1,5 V.

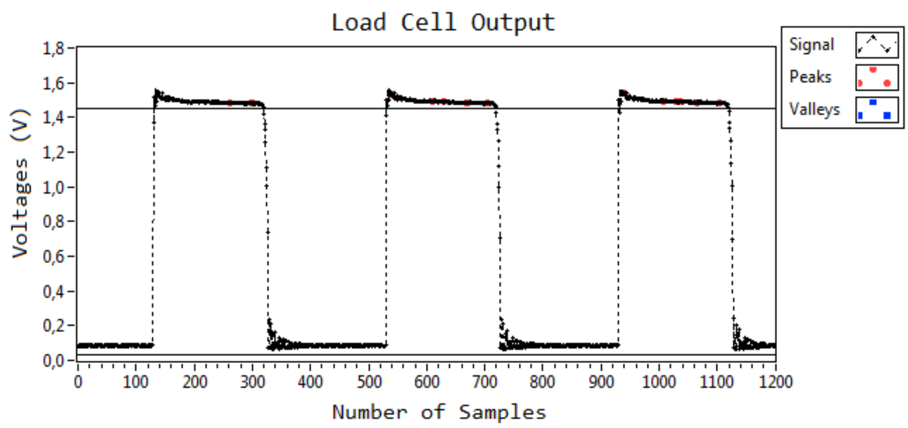

Figure 2.6. Load cell settling at 1 Hz with the testing time of 3 seconds

2.5.5 Charge amplifier

The charge amplifier is used most commonly in applications involving piezoelectric sensors. Although the name charge amplifier suggests that somehow electric charge is being amplified. The truth is that this type of amplifier just converts electric charge into a proportional electric voltage. Piezoelectric sensors output electric charge which's SI unit is the „Coulomb"(C), defined as C=1 A·1 s. This means that 1 Coulomb of electricity is transported by a current of 1 Ampère during one second. Most piezoelectric sensors work in the range of pC (picocoulomb, 10^{-12}) which is also the common accepted working unit for piezoelectric measurements [3].

It is not recommended to measure the output of a piezoelectric sensor with a voltmeter or an oscilloscope because one would get an unreliable result. This is due to differences in cable length which means different capacitance inside the circuit. Also to have reliable measurements once the sensor is calibrated one would need to try to match the calibration lab load conditions when doing measurements somewhere else. This is of course very difficult and that is why for many years piezoelectric sensors were having trouble being incorporated into industries. The charge amplifier was first described by W.P. Kistler in 1950 [3].

The piezoelectric sensor outputs a charge which is difficult to measure; that is why the generated charges are collected by a capacitor and therefore converted into electric voltage that can be measured for an example a regular voltmeter.

2.5.5.1 Electrical connections and components in the charge amplifier

The charge from the piezoelectric material is going to the negative input of the operational amplifier while charging the C_f capacitor. Resistor R_f is there to slowly draw the charges from the capacitor to protect the amplifier from saturation. The schematic for a common implementation of a charge amplifier is shown on the Figure 2.7. To calculate the charge generated by the piezoelectric material one has to use the following formula [11]:

$$qp = C_f \cdot \left(\frac{V_{cc}}{2} - V_0\right) \quad (2.2)$$

qp – Charge generated by the piezoelectric material (C)

C_f – Capacitance of the capacitor (F)

V_0 – Output from the charge amplifier (V)

V_{CC} – Voltage powering the microchip (V)

In this work the differential measurement is used. In this case the formula 2.2 changes due to the fact that the differential input measures only the change of the charge amplifier. The modified formula to calculate the charge in this system is given in the equation 2.3.

$$qp = C_f \cdot (-V_0) \quad (2.3)$$

Resistor R_i is providing ESD (Electrostatic discharge) protection to the TL071P microchip and also controls the high cutoff frequency. The biasing connected to the positive input of the amplifier is there to raise the output voltage to half of the voltage powering the microchip so that the piezoelectric output can swing around this value [11].

Charge amplifiers give a negative output in response to a positive charge from the sensor.

In this work the operational amplifier TL071P is used. It has a low noise JFET input with an input resistance of 1 TΩ [22].

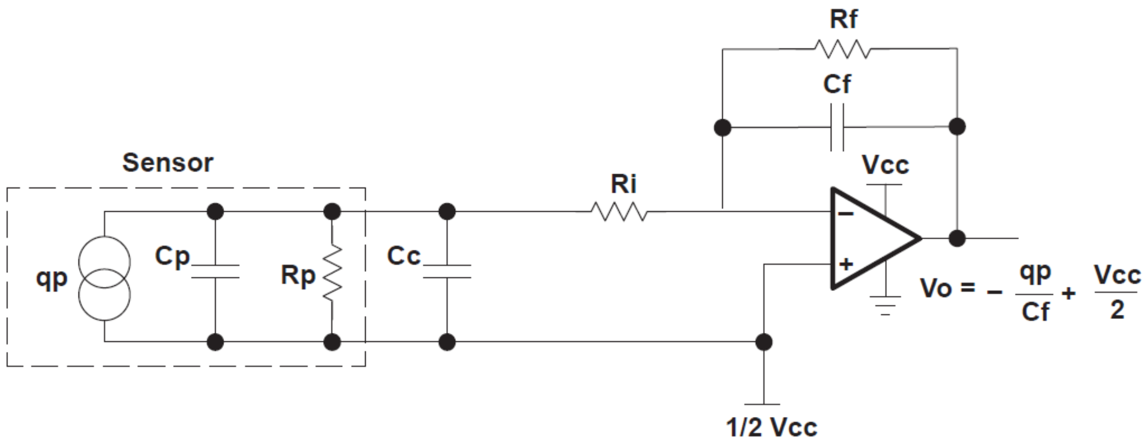

Figure 2.7. Schematic for the most common realization of a charge amplifier [11]

2.5.5.2 Charge amplifier gain and low cutoff frequency

Resistor and capacitor R_p and C_p respectively are the inherent properties of the piezoelectric sensor and in this work considered as unknowns due to the nature of the PVDF mats that are to be controlled in the future. The gain of the charge amplifier is controlled with the capacitor C_f and its value is the inverse of the capacitors value. Resistor and capacitor R_f and C_f are setting the lower limit for the amplification frequency. This is calculated using the following formula [11]:

$$f_L = \frac{1}{2 \cdot \pi \cdot R_f \cdot C_f} \quad (2.4)$$

f_L – Lower frequency limit for the amplification (Hz)

R_f – Resistor in the circuit (Ω)

C_f – Capacitor in the circuit (F)

$$f_L = \frac{1}{2 \cdot \pi \cdot 100 \cdot 10^6 \cdot 1 \cdot 10^{-8}} \approx 0{,}16\ Hz \quad (2.5)$$

The resistor R_f value is 100 MΩ and the capacitor C_f value is 10 nF. This means the gain of the charge amplifier is $1 \cdot 10^8$.

2.5.5.3 Charge amplifier high cutoff frequency

The amplifier maintains 0 V in its input terminals, which means that when cables are under few meters long the capacitance of the cabling does not affect the measurement. This is due to the fact that cables usually have 70 pF/m capacitance [3]. The high cutoff frequency of the amplifier is defined by the resistor R_i and capacitances C_p and C_c. High cutoff frequency is calculated with the following formula [11]:

$$f_H = \frac{1}{2 \cdot \pi \cdot R_i \cdot (C_p + C_c)} \quad (2.6)$$

f_H –High frequency limit for the amplification (Hz)

R_i – ESD protection providing resistor (Ω)

C_p – Capacitance of the piezoelectric sample tested (F)

C_c – Capacitance of the cables or a capacitor used to remove the effect of cables (F)

Capacitor C_c represents the capacitance of the cables which in this work is replaced by a 100 nF capacitor to make sure that with different piezoelectric sensors tested, the high cutoff frequency would stay relatively in the same magnitude. The value of 100 nF was chosen because the piezoelectric material NCE51 sample pieces that were tested, have a capacitance of 616 pF to 3428 pF. The values are shown in Table 2.2 and are from the specification sheets that came with the piezoelectric material. The change in the high cutoff frequency with these samples changes about 3 % which can be neglected due to the low frequencies used during the measurements.

When the values from the circuit are used to calculate the high cutoff frequency the result is the following:

$$f_H = \frac{1}{2 \cdot \pi \cdot 10000 \cdot 100 \cdot 10^{-9}} \approx 159\ Hz \quad (2.7)$$

Table 2.1. NCE51 material sample's properties [Appendix B].

Sample nr	Sample 1	Sample 2	Sample 3
Length (m)	0,015	0,010	0,02
Width (m)	0,015	0,005	0,004
Thickness (m)	0,001	0,001	0,002
Capacitance (F)	$3{,}428 \cdot 10^{-9}$	$8{,}65 \cdot 10^{-10}$	$6{,}16 \cdot 10^{-10}$
High cutoff frequency f_H (Hz)	154	158	158

2.5.6 Data acquisition device USB-1616-HS-2

Polymer Material Institute has a Data Acquisition Device working with LabView software. The DAQ is shown on Figure 2.7. This device has 16 analog inputs, 24 digital input/output channels, which include counters/timers and quadrature encoders. There are up to 4 analog outputs for PWM signals. Measurement computing offers TracerDAQ software with the device but there is also support for software like Visual Studio, DASYlab and NI LabVIEW [24].

This work uses DAQ for generating the PWM signal which is used to control the movements of the solenoid. Additionally two analog channels are used to measure the INA125 and charge amplifier outputs. Analog inputs have a 16-bit 1 MHz Analog to Digital converters.

Figure 2.7. DAQ USB-1616-HS-2 [24]

2.5.7 Power supplies

The power to the electronic circuit is supplied by a laboratory bench supply that has an output from 0 V to 30 V and up to 3 A of current. For the solenoid another supply was used to provide 24 V up to 2 A of current.

2.5.8 Solenoid

To have control over the frequency of the mechanical deformation a solenoid was used to generate the force on the piezoelectric material. The reason why a solenoid is used is due to the simplicity and straightforward use of this device. One just needs to power it to 24 V and it will exert a force on the load cell. The accuracy of the solenoid is not important because the force is measured independently by the INA125 and load cell. The solenoid is used purely as a mechanical motion generator. The symbol of the solenoid and the solenoid itself is shown on the Figure 2.8.

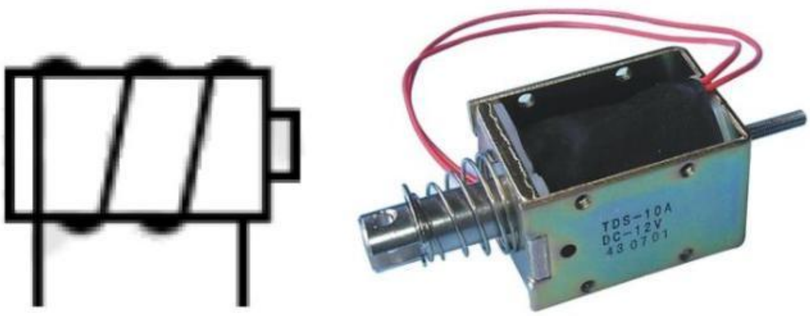

Figure 2.8. Solenoid symbol and solenoid TDS-10A [25]

2.5.9 Choice of cables, electrodes and connections

One of the main problems involving measuring charges generated by the piezoelectric sensors is how to make sure no additonal charges are being picked up by the connections and wires that connect the charge amplifier with the sensor. There are few requierments for the cables. First the should have sufficiently high insulation resistance, have a small capacitance and maintain these properties over a wide variety of temperatures. Also triboelectric effect could have a huge impact on the measurement if correct precautions are not used [3].

Very good choice and most commonly used cables are coaxial cables. Unfortunately having these cables is not enough. One has to take great care to make sure the connectors to the sensor and the amplifier are also very clean. This can be achieved by capping the connetors with a protective cap or cleaning them with special cleaning sprays or benzene. There should not be any oil residues or dirt between the connections [3].

It is important to keep the cables from moving while measuring piezoelectric sensors because moving cables could produce triboelectricity and therefore ruin the result. If measurements are needed to be done whilst moving special “low noise cables” have to be used. These cables have a layer of graphene between the wire and the insulation to remove triboelectric effects [3].

The cables in this work are stable during measurements which means that there is no need for special low noise cables. Ordinary thin coaxial cables have low capacity and low mass and are less prone to produce noise when moved [3].

The electrodes used to transmit the signal from the piezoelectri material to the charge amplifier is a copper plate that has been polished to achieve better contact between the materials. Before any measurements the electrodes are cleaned with ethanol to remove grease and impurities to have a reliable result.

3. Software for the measuring system

3.1 Requirements

The measurement system should be easy to use and simple to configure. System design platform LabVIEW from National Instruments was chosen because of its wide support for DAQ-s and the ability to create a user friendly interface. To be able to effectively adjust the system and in the future test the PVDF mat samples, the user interface needs to be clear, informative and easy to use. Additionally it must be possible to save the measurement data into a separate Excel file for archiving and further study.

3.2 Program flow diagram

The entire measurement system is controlled and monitored through the DAQ by a LabVIEW program. The program flowchart is shown on Figure 3.1.

When the program is started initial values from a data file created when the program was last closed is automatically loaded. One could change the initial values from the front panel to make adjustments to the system. To set up the DAQ channels the user needs to specify the DAQ channels connected to the analog circuit. Before the measurements begin the desired frequency for the solenoid movement has to be selected. The force has to be adjusted to the required level with the manual actuator that controls the distance of the solenoid from the sample while reading the value of the measured force from the front panel indicator.

The program waits until the START button value is changed to TRUE to begin the measurement cycle. In the cycle the PWM signal is generated and sent to the L293B microchip through the analog output channel specified by the user. During the same cycle measurements of the load cell amplifier output voltage and the output voltage of the charge amplifier is collected at the rate of 400 samples per second by the DAQ. This measurement lasts until the specified time has passed from the beginning of the measurement cycle. After the measurement collection the generation of the PWM signal is stopped.

Voltages that have been measured by the charge amplifier are corrected with the value k_c form the characterization (Chapter 4.2.5) of the charge amplifier. Then the data collected is checked for high and low points that correspond to the presses of the solenoid onto the sample. The acquired data is displayed on the charts on the front panel for assessment. The

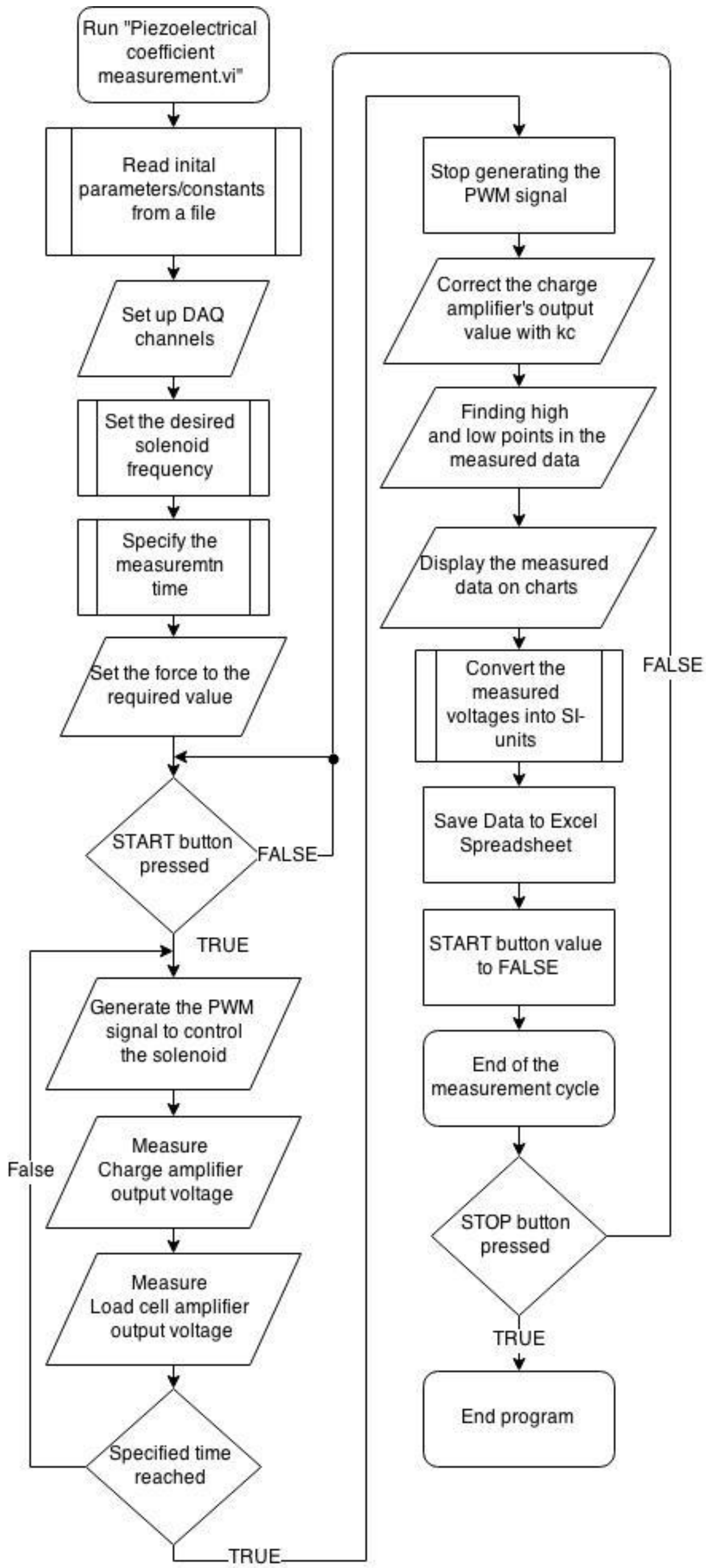

Figure 3.1. Flowchart of the LabVIEW control program

last measured piezoelectric coefficient's value is shown on the front panel indicator. Then all of the data collected is written into an Excel spreadsheet file.

Finally the START button value is changed to FALSE and that concludes the measurement cycle. If the STOP button is not pressed the program waits for the value of START to change to begin a new measurement cycle. The STOP button ends the program.

3.3 Program user interface

The front panel of the program is shown on Figure 3.2. The first things that the user must input into the front panel are the channels that correspond to the connected channels on the DAQ. These channels can be selected from drop-down lists on the left side of the front panel in the area of "DAQ Channel Connections." All the channels have their specific names written on top of the selection list. The channels that have to be chosen are the following:

- Channel that outputs the PWM signal to the L293B
- Channel that measures the output voltage of the Charge amplifier
- Channel that measures the output voltage of the Load cell amplifier

There is additional information about the wires and the DAQ connectors to make choosing the correct channels easier for the user. To change the frequency of the solenoid, one has to write the desired frequency value into the control box labeled "Frequency of the solenoid.“ The available frequencies are between 0,5 Hz and 1 Hz. The measurement time has to be specified also. The default value is set to 6 seconds but could be set up to 29 seconds. There is a text box for comments about the upcoming measurement for the user to elaborate on the measurement being done. The "Change parameters" button gives access to a pop-up window where new parameters can be inserted in the case when there are any adjustments to the parameters of the measuring system. The parameters that can be changed are the minimum and maximum weights and their corresponding values used to adjust the load cell. Additionally the size of the capacitor C_f that controls the gain of the charge amplifier and the values of the correction factor k_c can be adjusted. To start the measurement process the user has to press the START MEASUREMENTS button.

To know before the actual measurement what the exerted force is going to be the "Set-up force Value" can be pressed to show the value of the force on the load cell when the solenoid applies maximum force. To end the force adjustment "Stop force Set-up" is pressed.

The indicator “Measurement in progress” lights up when the measurement has started. During the measurement the solenoid presses with the specified frequency onto the sample until the measurements are finished and the results appear on the charts seen on the right side of the front panel. The top chart will display the voltage output of the load cell amplifier and the bottom chart the charge amplifier voltage output. The user can redo the measurement without changing any parameters by pressing START MEASUREMENT again. Or press the STOP button to close the program.

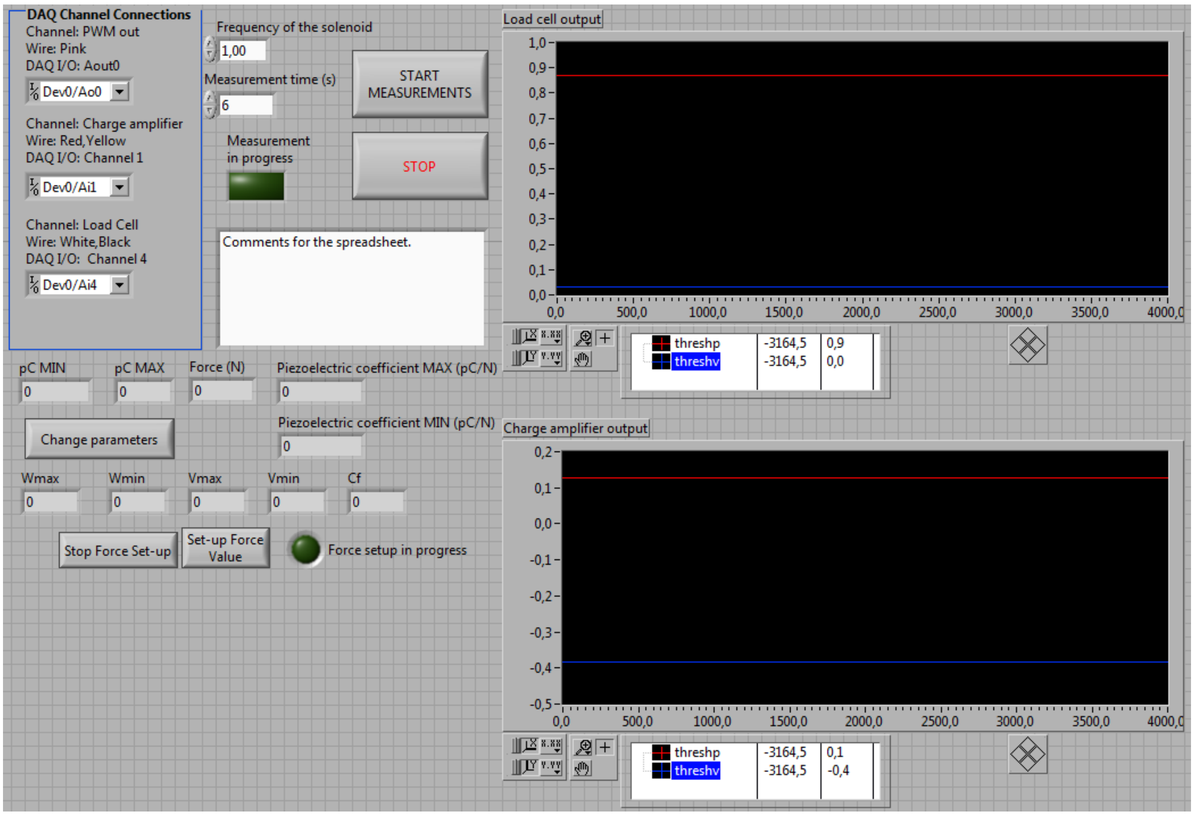

Figure 3.2. Front panel of the LabVIEW control program

4. Test and validation

4.1 Description of the method

The quasi-static method measures the direct piezoelectric effect. This means that the piezoelectric material creates a charge when mechanically deformed by the solenoid. The measuring system provides the means to conduct measurements with frequencies from 0,5 Hz up to 1 Hz with forces varying from 7 N to 12 N. These ranges were determined during the testing of the measurement system. The charge is measured with the charge amplifier and the amount of deformation is registered as force. The testing time is regulated by the user and could vary from 6 seconds up to 29 seconds. The lower limit is set to reduce the error that come from testing samples in as short period of time. The upper limit is just a practical consideration that arouse from the specifics of the program. During this time voltages from the amplifiers outputs are collected and transferred to the measuring computer. The voltages are converted into values of charge and force and then the final result in the form of the piezoelectric coefficient can be calculated using the following formula:

$$d_{33} = \frac{q}{F} \quad (4.1)$$

q- Charge generated (C)

F –Force exerted (N)

d_{33} –Piezoelectric coefficient (C/N)

4.2 Reference material

Before one could use this system to determine the piezoelectric constant of PVDF fiber mats a characterized material has to be used to evaluate the measurement uncertainty of the measuring system. In this work the NCE51 material is used as a reference for the piezoelectric coefficient value. The process that is used in the article “Comparison of Methods of Piezoelectric Coefficient Measurement“ [12] is followed. This article tests different methods of measuring the piezoelectric coefficient using the same type of piezoelectric material during all the measurements. The reference material is from a Danish based company called Noliac A/S and is described as a material suitable to static or semi-static situations. The material name is NCE51, see Figure 4.1.

Figure 4.1. Samples made of the piezoelectric material NCE51

According to manufacturer specifications the piezoelectric charge constant d_{33} of the reference material has a value of 443 pC/N with the expanded measurement uncertainty of 5 % of the measured value [26].

4.2.1 Weights for the load cell adjustment

The weights used to adjust the load cell have a cylindrical shape with the lifting knob and are made of brass. The weights are of class M1. The biggest weight has a mass of 500 g. This means that two 500 g and four 200 g weights are used to make the adjustments to the load cell measurements. The expanded measurement uncertainty for the 500 g weight is 25 mg and the 200 g weight 10 mg, respectively [27]. The weights are shown on Figure 4.2.

Figure 4.2. Weights used at the adjustment of the load cell

4.2.2 Capacitance of C_f and C_{ref}

Capacitor C_f is one of the variables that are used to calculate the charge generated by the piezoelectric sensor. Capacitor C_{ref} is used when the charge amplifier output is characterized. The capacitors have been measured with a capacitance bridge AH2700A at 50 Hz, with the expanded measurement uncertainty of 0,1 % of the measured value. The measured values are given in the Table 4.1.

Table 4.1. Capacitors C_f and C_{ref} values

Capacitor	Capacitance from specifications (F)	Measured capacitance (F)
C_f	$10 \cdot 10^{-9}$	$9{,}23 \cdot 10^{-9}$
C_{ref}	$1 \cdot 10^{-9}$	$9{,}74 \cdot 10^{-10}$

4.2.3 Voltage measurement

The DAQ USB-1616-HS-2 is responsible for measuring the voltages necessary to calculate the force exerted on the piezoelectric sample and the charges generated by the sample. The accuracy given in the specifications of the DAQ in the -5 V to +5 V range is ±(0,031 + 0,009) % for % of reading + % of range, respectively [28]. To provide this measurement accuracy an adjustment of the DAQ should be performed.

4.2.3.1 Adjustment and configuring the DAQ

The measurement adjustments and configuring is done with special software that comes with the DAQ called instaCal. This method of adjustment relies on the stored correction factors at the time of adjustment in the factory [24]. Adjustment for this work is done in the TUT lab while it is installed into the measuring system.

The entire adjustment consists of pressing the calibrate button in instaCal and when configuring the DAQ the “Calibration Coefficients” value has to be changed to Field. The Figure 4.3 shows the configuration window for the USB1616HS-2. The DAQ is working in the differential setting and the ADC has a settling time of 1µs. And the performance test shows that the DAQ is capable of receiving 983391 samples per second.

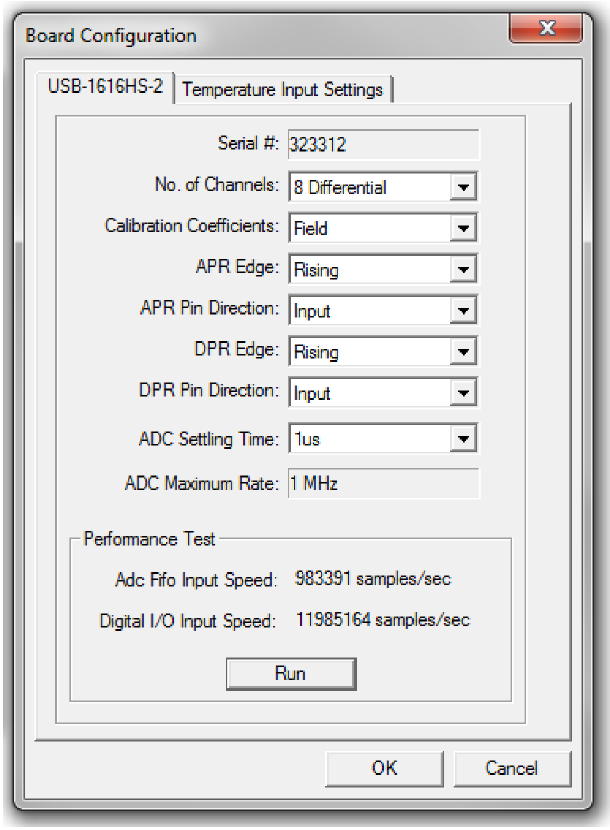

Figure 4.3. Configuring settings for the USB1616HS-2 in instaCal

4.2.4 Adjustment of the load cell

To achieve reliable and accurate results one has to test and adjust the load cell and the INA125 amplifier to the specific need of the device. To do this one needs a set of weights to determine the output voltages when different loads are measured with this setup. This process is necessary to calculate the linear progression to be used for the measuring of the desired range of this load cell. The formula 4.2 is the modified formula for calculating the linear interpolation between two known points [29]. To calculate the output voltage of the amplifier when the load is known the following formula is used:

$$W_L = W_{min} + \left((W_{max} - W_{min}) \cdot \left(\frac{V_f - V_{min}}{V_{max} - V_{min}} \right) \right) \quad (4.2)$$

W_L - Load applied to the load cell (g)

V_f - Output voltage of the amplifier (V)

V_{min} - Output voltage at minimal load (V)

V_{max} - Output voltage at maximum load (V)

W_{min} - Minimum load applied (g)

W_{max} - Maximum load applied (g)

This equation gives the mass in grams placed on the load cell. To convert this value to Newtons, W_L has to be multiplied by 0,009807 which is the value of one kilogram in Newtons. The Table 4.2 shows the mass of the weights used and the corresponding voltage from the amplifier INA125.

Table 4.2. Measured weights and corresponding voltages

Mass (g)	**INA125 output (V)**
200	0,44
1800	3,78

4.2.5 Charge amplifier characterization

4.2.5.1 Testing method

Charge amplifiers need to be tested before one could use them for measurements. This requires having a source of a known amount of charge to feed into the input of the charge amplifier. To do this a sine wave, generated by a signal source, is used to load a capacitor in series with the input. When the capacitor's value is known the generated charge can be calculated with the following formula [30]:

$$q_{ref}(t) = U_{gen}(t) \cdot C_{ref} \quad (4.3)$$

$q_{ref}(t)$ – Charge created by the sine wave (C)

$U_{gen}(t)$ – Amplitude of the sine wave (V)

C_{ref} – Reference capacitor (F)

When the input charge is known the output of the charge amplifier is measured and the output is compared to the input. The difference between the input and output gives the value for a constant that should be included into the calculations when the charge amplifier is used in measurements. The capacitor C_{par} can be neglected due to the reasons discussed in the Chapter 2.5.5.3. The set-up for characterization of the charge amplifiers is shown on the Figure 4.4.

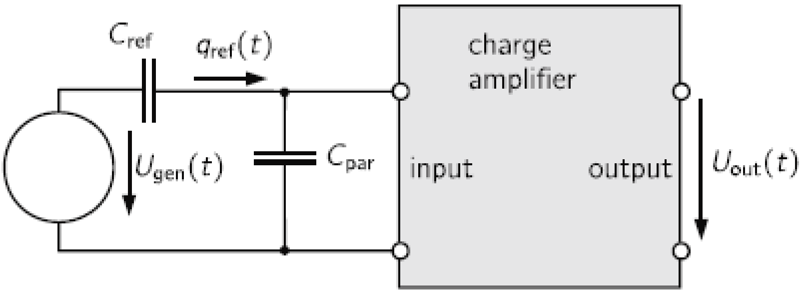

Figure 4.4. Set-up schematic for the charge amplifier's characterization [30]

4.2.5.2 Software for characterization of the charge amplifier

To characterize the charge amplifier a special LabVIEW program was created. This program is used to generate a sine wave which's amplitude is rising in time. The program is also responsible for measuring the output of the charge amplifier and comparing the charge amplifier's output to the generated sine wave. Finally the program saves the collected data into an Excel spreadsheet file, where an approximated equation can be created to use the values in the control program described in the Chapter 3.2. These values correct the output value of the charge amplifier. The front panel of the characterization program is shown on Figure 4.5. To generate and measure the signals the user chooses the channels used during the characterization. Then the value of the reference capacitor's value needs to be specified. There are also controls for different sine wave properties and the amount of samples generated. The generated output signal is shown in the "DAQ Output Waveform" chart and the measured signal form the charge amplifier is shown on the "Charge amplifier Output Waveform" chart. They are the inverse version of each other because the input to the charge amplifier is connected to the inverting input of the operational amplifier. The Excel spreadsheet consists of seven different types of data. This data enables to create a formula to use the correct correction constant for a wide range of different charge amplifier voltage outputs measured with the system.

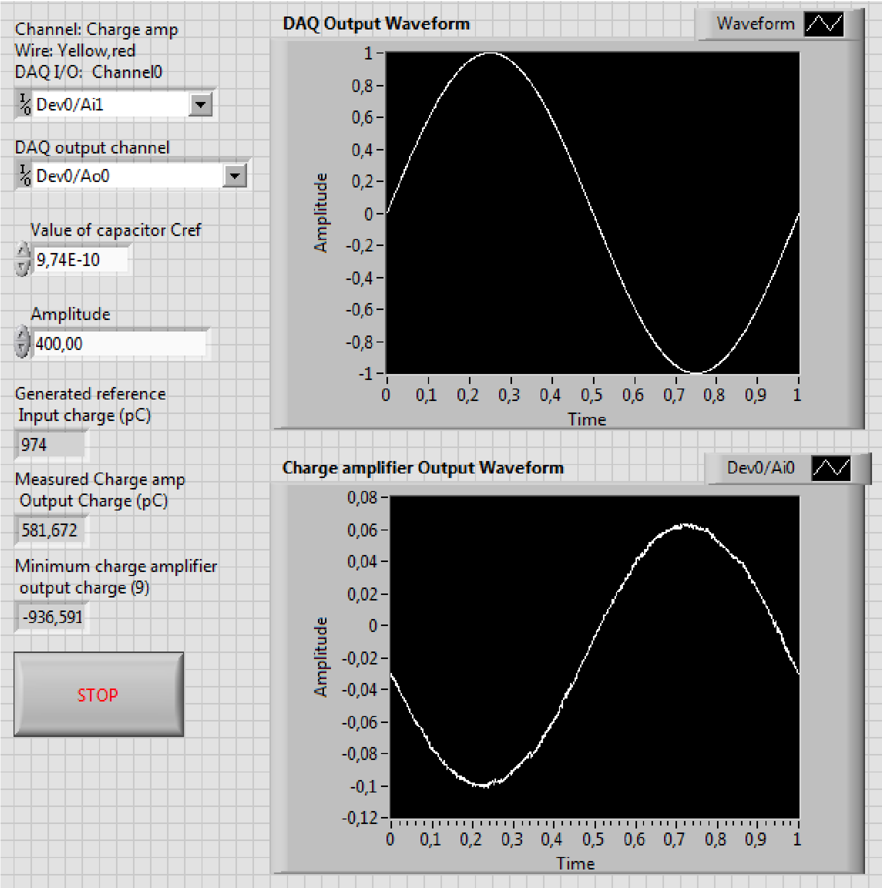

Figure 4.5. The input/output signals and the initial conditions for the characterization process are shown

4.2.5.3 Selection of the reference capacitor

The selection of the reference capacitor is based on the value of the NCE51 piezoelectric coefficient. When the amount of exerted force is known one can calculate the charge that the material could potentially generate. The maximum force generated by the measuring system is 12 N. And the value of the NCE51 piezoelectric coefficient is given in Chapter 4.2. The corresponding calculation is shown in the formula 4.4.

$$Q = d_{33} \cdot F = 443 \cdot 10^{-12} \cdot 12 = 5{,}316 \cdot 10^{-9}\, C \quad (4.4)$$

Q – Charge generated by the exerted force (C)

d_{33} – Piezoelectric coefficient of the NCE51 material (pC/N)

F – The amount of exerted force (N)

To generate this charge, while staying in the limits of 10 V maximum output of the DAQ, a 1 nF capacitor was chosen by using the formula 4.3.

$$q_{ref}\,(10V) = 10 \cdot 1 \cdot 10^{-9} = 1 \cdot 10^{-8}\, F \quad (4.5)$$

$q_{ref}(10V)$ – Maximum generated charge (C)

The capacitor that was used had a capacitance of $9{,}74 \cdot 10^{-10}$ F and gives the potential characterization range of 1168 pC up to 9545 pC. This covers the maximum charge generated by the NCE51 material pieces.

4.2.6 Determining the measurement range for the measuring system

The highest charge that the current measurement system has a known response is 9545 pC and the lowest known measurable charge is 1168 pC. The highest force that could be exerted is 12 N and the lowest force is 7 N. To find out the range of the measurable piezoelectric coefficients the maximum measurable charge is divided by the maximum force that could be produced. This calculation is done with the equation (4.1).

$$Max\ d_{33} = \frac{9545\ pC}{12\ N} = 795\frac{pC}{N} \quad (4.6)$$

The minimal piezoelectric coefficient is calculated by:

$$Min\ d_{33} = \frac{1168\ pC}{7\ N} = 166\frac{pC}{N} \quad (4.7)$$

4.2.6.1 Corrections for the charge amplifier

The spreadsheet file generated by the characterization program is used to create equations that are used in the control program to adjust the voltages of the charge amplifier output. On Figures 4.6 and 4.7 the correlation between the reference charge and the charge amplifier's output voltage is shown.

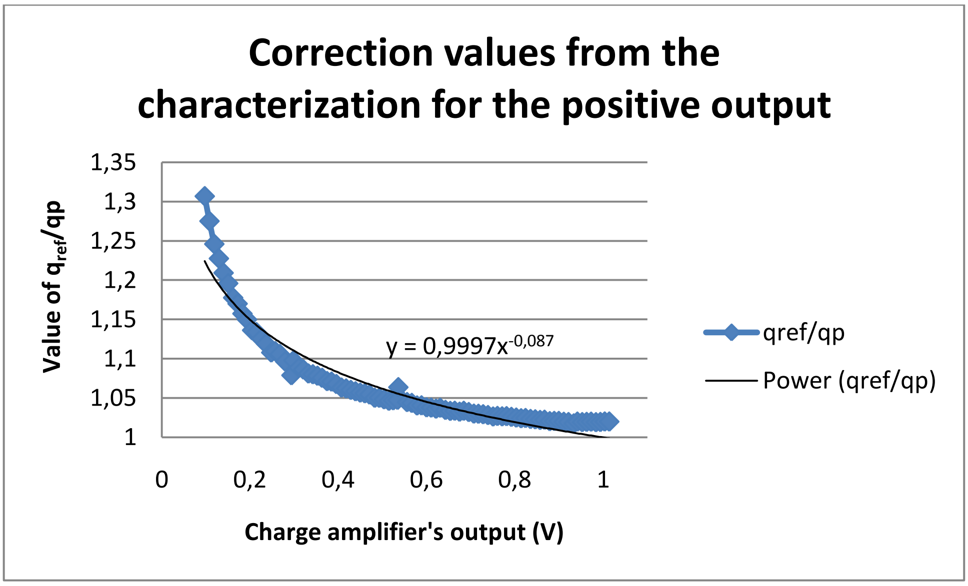

Figure 4.6. Difference between the reference charge (q_{ref}) and the charge measured (qp) by the charge amplifier in correlation with the amplifier voltage output during the characterization. Positive charge amplifier's output.

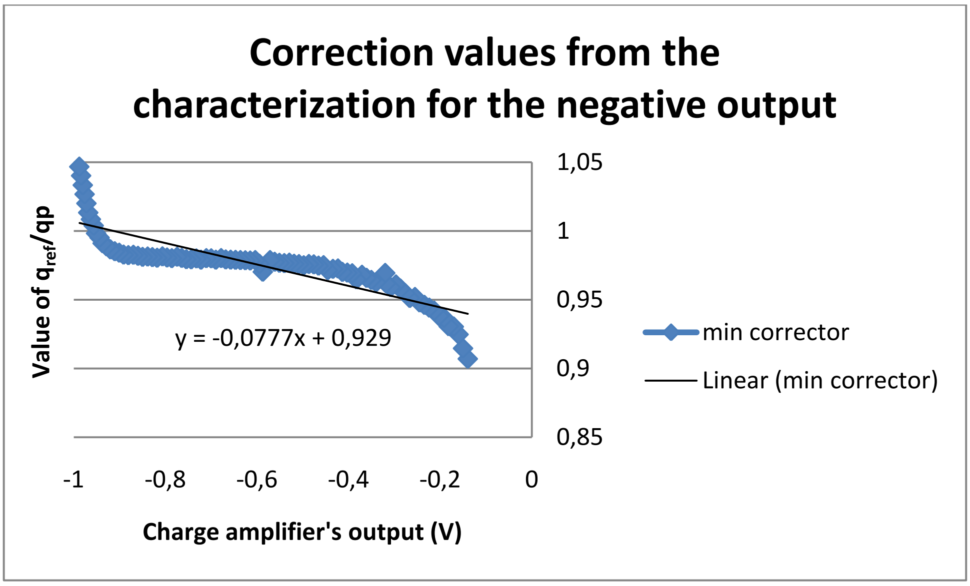

Figure 4.7. Difference between the reference charge (q_{ref}) and the charge measured (qp) by the charge amplifier in correlation with the amplifier voltage output during the characterization. Negative charge amplifier's output.

4.3 Measuring the piezoelectric coefficient

The measurements with the Noliac A/S NCE51 material were done using 0,5 Hz. The sample was placed between the copper electrodes that were cleaned of dirt and grease before the measuring. The measurement results are the individual peaks extracted by the LabVIEW program. When the load cell measures a push from the solenoid a negative response is measured on the charge amplifier. On the Figures 4.8 and 4.9 the outputs of the charge and load cell amplifiers response to one pulse from the solenoid are shown. The positive output of the charge amplifier is lower than the negative output, but this difference is due to the characteristic of the amplifier and it will be corrected with the correction equation for the value k_c that was found in the Chapter 4.2.6.1.

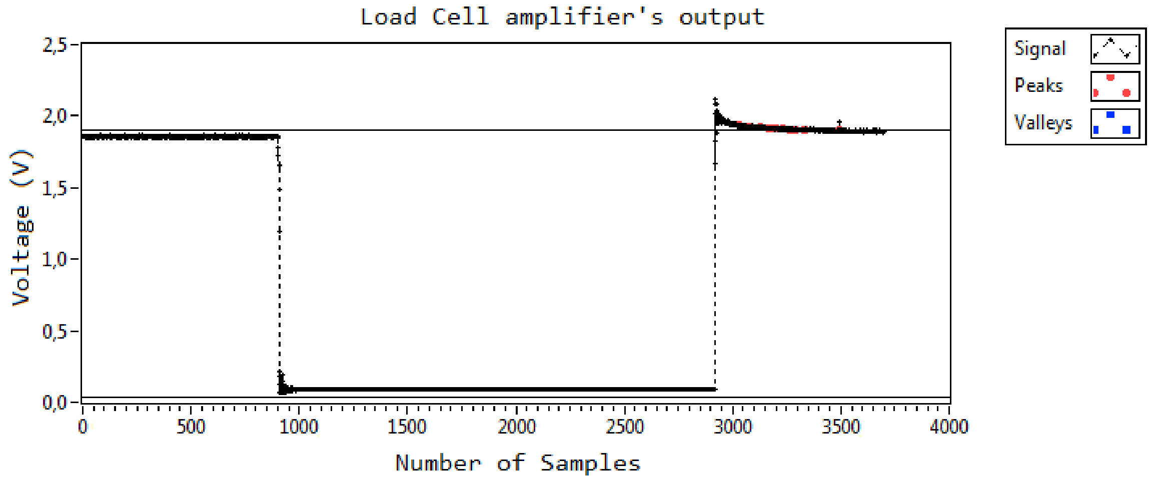

Figure 4.8. The change in the output voltage of the load cell amplifier to a single change in force in respect to the number of samples taken

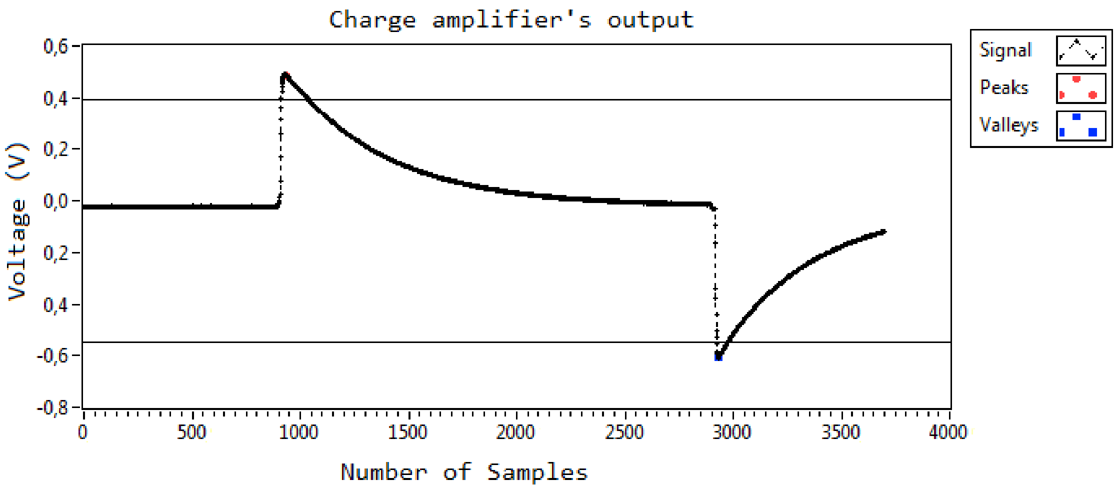

Figure 4.9. The change in the output voltage of the charge amplifier to a single change in force in respect to the number of samples taken

4.4 Measurement results

The force of 10 N was used during the measurement. This force was chosen because between the force values of 7 N to 12 N the measurement results are the most stable. The measured piezoelectric coefficients, which correspond to the release of the solenoid from its maximum position, are given in the Table 4.3.

Table 4.3. Piezoelectric coefficients measured at 0,5 Hz with a force of 10 N

Number of measurement	d_{33m}	**Number of measurement**	d_{33m}
1	466	**11**	470
2	472	**12**	479
3	481	**13**	467
4	484	**14**	482
5	473	**15**	487
6	472	**16**	485
7	464	**17**	469
8	469	**18**	472
9	467	**19**	470
10	477	**20**	480

Equation 4.8 is used to calculate the piezoelectric coefficient from the values acquired by the DAQ.

$$d_{33_m} = \frac{q}{F} = \frac{C_f \cdot (-V_0) \cdot 1 \cdot 10^{12}}{\left(W_{min} + \left((W_{max} - W_{min}) \cdot \left(\frac{V_f - V_{min}}{V_{max} - V_{min}}\right)\right)\right) \cdot 0{,}009807} \quad (4.8)$$

The mean value of piezoelectric coefficients measured with the force of 10 N at the frequency of 0,5 Hz is 474 pC/N and the experimental standard deviation is 2,4 pC/N.

4.5 Uncertainty analysis

4.5.1 Measurement uncertainty of the piezoelectric coefficient

The combined standard uncertainty $u_c(y)$ is calculated with [31]:

$$u_c(y) = \sqrt{\sum_{i=1}^{N} \left(\frac{df}{dx_i}\right)^2 \cdot u^2(x_i)} \quad (4.9)$$

Where *f* is the function which consists of all the variables that make up the value that's accuracy is calculated. Values of $u(x_i)$ are the uncertainties of the variables from function *f*. All the calculations necessary for determining measurement uncertainty are done with Excel and MATLAB.

The first step in the calculation process is the calculation of the partial derivatives. This step is done with a MATLAB code to speed up the process. Partial derivatives are calculated in MATLAB by using the command *diff(f,x)* where the *f* represents the function in the equation

4.8 and the *x* represents the variable that the partial derivative is taken. The individual results will not be presented due to the monotonic nature of the data.

When the partial derivatives have been calculated they can be used to calculate the standard uncertainty of the piezoelectric coefficient. This is done in the MATLAB by the following equation:

$$u(d_{33_m}) = \sqrt{\begin{array}{c}\left((dC_f)^2 \cdot u_{C_f}{}^2\right) + \left((dV_0)^2 \cdot u_{V_0}{}^2\right) + \left((dW_{min})^2 \cdot u_{W_{min}}{}^2\right) + \left((dW_{max})^2 \cdot u_{W_{max}}{}^2\right) + \\ \left((dV_f)^2 \cdot u_{V_f}{}^2\right) + \left((dV_{min})^2 \cdot u_{V_{min}}{}^2\right) + \left((dV_{max})^2 \cdot u_{V_{max}}{}^2\right) + u_s + u_{mat}\end{array}} \quad (4.10)$$

Where dC_f, dV_0, dW_{min}, dW_{max}, dV_f, dV_{min} and dV_{max} are the corresponding values for each partial derivative. Their calculation formulas are written in in the MATLAB code which is in the Appendix C. The u_{Cf}, u_{V0}, u_{Wmin}, u_{Wmax}, u_{Vf}, u_{Vmin} and u_{Vmax} are the uncertainties of the individual variables. The u_s and u_m are the standard deviation of the 20 measurements and the uncertainty of the material NCE51 respectevly. The uncertainty budget for the piezoelectric coefficient d_{33_m} is presented in Table 4.4.

4.5.1.1 Calculating the uncertainty of the piezoelectric coefficient

Table 4.4. Uncertainty budget (d_{33_m})

Quantity X_i	**Estimate** x_i	**Standard uncertainty** $u(x_i)$	**Sensitivity coefficient** c_i	**Uncertainty contribution** $u_i(y)$
C_f (nF)	9,23	$4{,}62\cdot10^{-3}$	52 (pC/N)/nF	$2{,}4\cdot10^{-1}$
V_0 (V)	0,528	$2{,}8\cdot10^{-4}$	898 (pC/N)/V	$2{,}5\cdot10^{-1}$
V_f (V)	2,21	$5{,}5\cdot10^{-4}$	216 (pC/N)/V	$1{,}2\cdot10^{-1}$
W_{min} (g)	200	$5\cdot10^{-4}$	0,213 (pC/N)/g	$1{,}06\cdot10^{-4}$
W_{max} (g)	1800	$2{,}1\cdot10^{-3}$	0,240 (pC/N)/g	$4{,}9\cdot10^{-4}$
V_{min} (V)	0,44	$2{,}7\cdot10^{-4}$	102 (pC/N)/V	$2{,}7\cdot10^{-2}$
V_{max} (V)	3,78	$8\cdot10^{-4}$	115 (pC/N)/V	$9{,}2\cdot10^{-2}$
u_m (pC/N)	474	11	1,0	11
u_s (pC/N)	0,0	2,4	1,0	2,4
d_{33_m}	**474**			**11,3**

The expanded uncertainty of the piezoelectric coefficient is given by:

$$U = k \cdot u(d_{33_m}) = 2 \cdot 11{,}3 = 23\,\frac{pC}{N} \quad (4.11)$$

The coverage factor k =2 is used.

The measured piezoelectric coefficient value of the NCE51 material is 474 pC/N ±23 pC/N.

4.6 Analysis of the measurement result

The developed measurement system has been characterized against the reference material NCE51. The measurement results are shown in Table 4.5. Where, the applied value of the material NCE51, the value measured by the system, deviation of the measured value from the reference value and the expanded measurement uncertainty are given.

Table 4.5. Measurement results

Applied value, pC/N	**Measured value, pC/N**	**Deviation, pC/N**	**Expanded measurement uncertainty, pC/N**
443	474	31	23

The relatively high deviation of around 7 % of the measured value is most likely due to the contact quality of the electrodes and changes in the solenoid movements. These effects should be further investigated and analyzed to increase the accuracy of the measurement system. Additionally calibration of the system components at a calibration laboratory should be conducted before making high-accuracy measurements. In its current state the measurement system can be used for measurements of the piezoelectric coefficients relative to the reference material in the range (166…795) pC/N at frequency 0,5 Hz, at forces of 7 N up to 12 N and the estimated measurement uncertainty of 5 % of the measured value.

5. Design the measuring device

This system was constructed as a prototype which means it is hard to handle and use. To make the everyday usage of the system better, a design solution is offered that could be implemented to improve the robustness of the set-up.

5.1 Design of the PCB board

The most fragile part of the system is the prototyping board that could easily be damaged. The wires connecting the microchips, DAQ, load cell and the power supply should be connected on a PCB board for increased durability and to lower signal errors coming from long wires. The schematic that the board design is based on is shown in the Appendix A. On the PCB a DC/DC converter is added to discard the 5 V power supply, used while prototyping. The board layout is created with the Eagle Light software directly from the schematic. On the Figure 5.1 a design solution is proposed that could be made into a functioning PCB board. The routes connecting the parts are 0,6 mm wide and the routes are on the top and bottom layers of the board because of the complexity of the circuit.

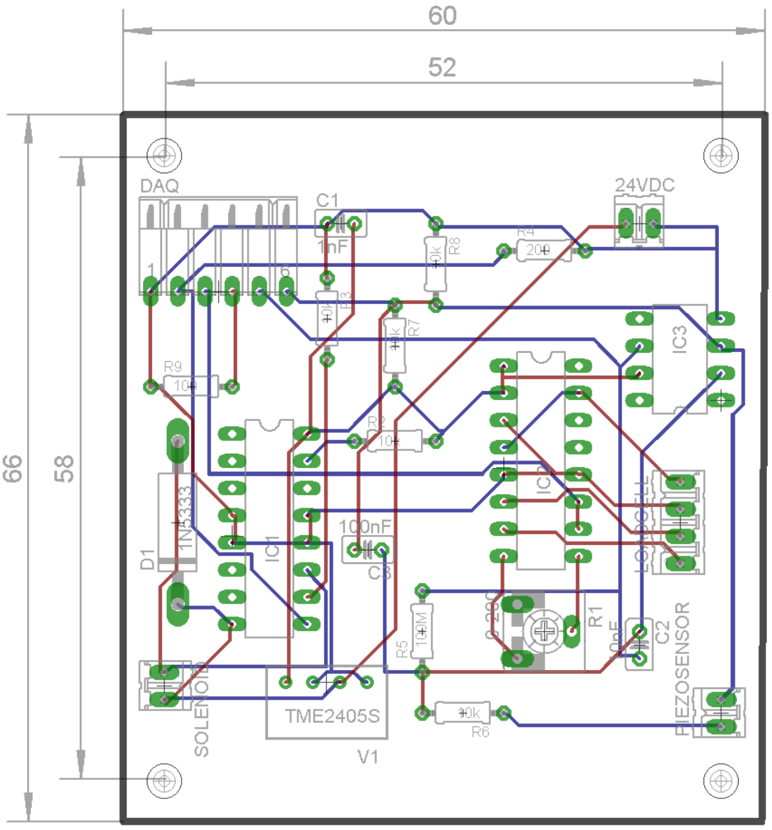

Figure 5.1. Proposed PCB design solution for the measurement system

5.2 Design of the measuring device

The piezoelectric measuring device should be a compact and robust. To protect the PCB and make sure the connections to the power supply and the DAQ are reliable. A design for the casing is proposed. This design uses the same main principle as does the prototype. The solenoid is attached to two rods that act as linear guidance and to a threaded rod that is used to regulate the height of the solenoid from the testing sample. The height is regulated by turning the knob on top of the device. The load cell attached to the base of the device with screws to avoid placement issues. The base of the device holds the PCB, power connector and the D-Sub input/output connector for easy connecting the DAQ to the device. The proposed design for the measuring device is shown on Figure 5.2.

Figure 5.2 Proposed design for the measuring device

This design was created so that most parts could be printed with a 3D printer. This goal was set because the Polymer Material Institute has a 3D printer and this means that the costs of building would be lower compared to constructing this device with more traditional means.

6. Building cost of the measuring device

The cost of the proposed design of the measuring device is shown in the Table 6.1. This listing does not include the Data Acquisition Device or the computer that is used to take the measurements. All of the electrical components are from Elfa Distrelec AS. The PCB board cost is an estimation from the SQP International's price calculator's.

Table 6.1. The cost of components for the proposed design of the measurement device

Product	**Amount**	**Price (€)**
PCB Terminal Block 4P2.54 mm	1	2,00
PCB Terminal Block 2P2.54 mm	3	3,24
Tantal capacitor, radial 1 uF 35 VDC 5 mm	1	0,6
Capacitor 10 nF 50 VDC 5.08 mm	1	1,62
PCB Terminal Block 6P2.54 mm	1	2,95
Capacitor 1 nF 50 VDC 5.08 mm	2	0,94
Resistor 1 MΩ 1 W ± 5 %	1	0,1
High-ohm resistor 100 MΩ 0.5 W ± 1%	1	4,37
Resistor 100 Ω 0.6 W ± 1 %	2	0,14
Resistor 10 kΩ 1 W ± 1 %,	4	0,59
Motor Driver IC DIL-16, L293B	1	3,07
Operational Amplifier Single 3 MHz DIL-8, TL071IP	1	1,03
Instrumentation Amplifier DIL-16, INA125PA	1	4,97
Trimmer Carbon 220 Ω linear 0.15 W	1	0,63
Power Jacks 2.5 mm 5.5 mm	1	1,77
D-Sub Socket, Solder Pins, 9P	1	1,49
DC/DC converter 24 VDC 5 VDC 1W	1	5,20
Ordering the PCB from SQP Inernational	1	55,06
Zener diode 27V 5W 1N5361BG	1	0,64
Ball bearing d = 8 mm D = 16 mm	2	10
Threaded Rod M8, 2 m	1	0,8
3D Printer Filament PLA black 1 kg, PLA, 3 mm	1	51,70
Aluminium rod d=8MM, 2 m	1	0,8
Bolt DIN 7991, M3, L=20 mm	20	4,4
Screw DIN 7982 M3, L=13 mm	100	1,66
Electric hoisting magnet 8 mm 4.2 W	1	27,40
	Total	**191,67**

Summary

The purpose of this thesis was to develop a measurement method to measure the piezoelectric coefficient in a cost effective way. The need for such measurements arouse from the research done in Tallinn University of Technology's Department of Polymer Materials. The piezoelectric materials that need measuring were created during the research project „Carbon Nanotube Reinforced Electrospun Nano-fibers and Yarns."

Due to the high cost of the commercial measuring devices from H. C. Materials Corporation and Piezotest, the incentive to construct a measuring system for these materials was high. Additionally the commercial devices are mainly for solid ceramic piezoelectric materials and not for soft materials, like PVDF fiber mats which are the materials created during the research project.

In the first part of the thesis the theoretical background of the piezoelectric effect was given and the relationship between mechanical deformation and electricity in piezoelectric materials was examined. The importance of polarization and dipole placement in the piezoelectric materials were introduced. Piezoelectric charge constants d_{ij} and their use in the comparison of different piezoelectric materials were discussed. The method of creating the electrospinned PVDF fiber mats was shown and the creation of piezoelectric properties in PVDF was explained. The existing measuring methods for piezoelectric coefficients were studied and the choice to use the quasi-static method was made because of its lower cost, simpler design and better versatility.

The second part described the realization of the quasi-static method and focused on the measuring system's mechanical and electronic components. Description of the used frame where the solenoid, load cell and the sample holder were attached to is given. Individually the importance and the implementation of electronics components such as the L293B driver, load cell amplifier INA125 and the charge amplifier were shown. The frequency limits of the charge amplifier were analysed and calculated. The low cutoff frequency limit was set to 0,16 Hz and the high 159 Hz. The amount of disturbance created by the sample's capacitance was investigated and it was concluded that with the correctly chosen values the capacitance will not have any measurable effect on the frequency range. The importance of connecting and cleaning the measuring system was detailed.

The third part described the requirements for the program that controls the measuring system, explained the flow of the program by detailing the processes executed and finally the user interface in the form of the front panel was examined in detail.

Fourth part consisted of validating the result measured by the system. The material NCE51 was introduced and its properties examined. The adjustment process for the force measurement and the charge amplifier is investigated. A method for testing the charge amplifier is examined. This method was used to characterize the charge amplifier by determining the correction of the output by using a known charge input. To use this method a program had to be composed to provide the means for conducting such test. The analysis of the components required for this test was also done. The results of this characterization were used as correction values in the measuring system.

A deeper description of the method of measurement was given and the parameters for testing were chosen. A set of measurements was conducted at the frequency of 0,5 Hz and with the force of 10 N. The uncertainty analysis was conducted and described.

The measurement of the piezoelectric coefficient of the reference material NCE51 has been conducted. The deviation of the measurement results of the measurement system is less than 7 % with the measurement uncertainty of 5 % of the measured value. The deviation from the reference value is most likely due to the vibrations created by the movement of the solenoid and the contact quality of the electrodes. These effects should be further investigated and analyzed to increase the accuracy of the measurement system.

The fifth part of the thesis proposed a design for the PCB board and the measuring device's fixture.

The sixth part gave an overview of the costs involving the building of the proposed design.

Kokkuvõte

Käesoleva lõputöö eesmärgiks oli välja arendada mõõtemeetod piesoelektrilise konstandi mõõtmiseks lihtsal ning majanduslikult otstarbekal viisil. Vajadus sellise mõõtemeetodi järgi tekkis Tallinna Tehnikaülikooli Polümeermaterjalide Instituudis tehtud teadustööst. Mõõtemeetodit vajavad piesoelektrilised materjalid loodi teadusprojektis „*Carbon nanotube reinforced electrospun nanofibers and yarns*“ e. „Elektroketruse teel valmistatud süsinik-nanotorudega armeeritud nanokiud ja lõngad“.

Ettevõtete H. C. Materials Corporation ja Piezotest poolt müüdavate mõõteseadmete kõrge hind oli määrava tähtsusega punktiks alustada iseseisvalt mõõtesüsteemi arendamist. Lisaks kõrgele hinnale, piirab müügil olevate seadmete Polümeermaterjalide Instituudis kasutamist fakt, et pakutavad seadmed ei ole mõeldud mõõtmist vajavate PVDF nanokiududest loodud mattide mõõtmiseks.

Töö esimeses osas tutvustati piesoelektrilise efekti teoreetilist tausta ja olemust. Uuriti mehhaanilise deformatsiooni ja elektri suhet piesoelektrilistes materjalides ning kirjeldati piesoelektrilise konstandi d_{ij} olulisust erinevate piesoelektriliste materjalide võrdlemisel. Anti ülevaade elektroketruse olemusest ning selgitati kuidas toimub nanokiududest koosneva PVDF matti loomine. Põhjalikumalt uuriti olemasolevaid meetodeid, piesoelektrilise konstandi mõõtmiseks ning otsustati kasutada “*quasi-static*” meetodit - see on odavam, lihtsam teostada ning universaalsem.

Teises peatükis kirjeldati valitud meetodi realisatisiooni ning tutvustati individuaalselt mehhaanilisi ning elektroonilisi komponente, mida kasutati mõõtesüsteemis. Põhjalikum ülevaade anti solenoidi juhtimiseks kasutatud L293B juhtkiibist ja tema ühendustest skeemis. Lisaks uuriti jõu anduri võimendit INA125 ning ka laengu võimendi tööpõhimõtet. Töös kirjeldatakse ka arvutuskäike laengu võimendi töösageduste arvutamiseks. Laengu võimendi sagedusvahemikuks seati 0,16 Hz kuni 159 Hz. Kontrolliti sagedusvahemiku muutust erinevate katsekehade kasutamisel, mille tulemusena selgus, et õigesti valitud laengu võimendi komponentide puhul, ei oma katsekeha mahtuvus märgatavat mõju. Peatuti elektroodide ning katsekehade puhtuse olulisusel ning kaablite võimalikust mõjust mõõteseadmele.

Kolmandas osas kirjeldati programmile esitatud nõudeid ning toimus koostatud kontrollprogrammi ülesannete seletamine. Tutvustati kasutajaliidest ning erinevaid mõõtesüsteemi parameetreid.

Neljas osa koosnes mõõtetulemuste valideerimisest. Kirjeldati piesoelektrilise materjali NCE51 omadusi. Viidi läbi mikrokontrolleri USB1616HS-2 seadistamine. Kirjeldati häälestus protsessi nii jõu andurile kui ka laengu võimendile. Anti ülevaade häälestamisel kasutatud kondensaatoritest ning kaaluvihtidest. Laengu võimendi peamine häälestamine toimus kontroll-laengu suhtes. Kontroll-laeng tekitati kondensaatori laadimisel siinus signaaliga, mis oli genereeritud LabVIEW keskkonnas loodud programmiga. Häälestamise käigus saadud tulemuste põhjal loodi võrrandid, mis iseloomustavad laengu võimendi väljundi erinevust võimendisse sisenevast laengust. Võrrandeid kasutati mõõtesüsteemi programmis mõõdetud laengu väärtuste täpsuse tõstmiseks.

Põhjalik ülevaade anti mõõteseadme mõõtetulemustest ning mõõtemääramatuse arvutamisest mõõtetulemustele, mis tehti sagedusel 0,5 Hz ning deformeeriva jõu 10 N korral. Selgitati parameetrite valikut, mõõtetulemuste tegemiseks.

Teostatud on materjali NCE51 piesoelektrilise konstandi mõõtmine. Mõõtesüsteemi mõõtehälve oli alla 7 % laiendmääramatusega 5 % mõõdetud väärtusest. Mõõtehälve tekib tõenäoliselt elektroodide ühendusest katsekehaga ning lisaks mõjub ka solenoidi poolt tekitatud vibratisoon mõõtetulemusele negatiivselt. Neid mõjureid peab edasi uurima ning analüüsima, et kasvatada mõõtesüsteemi täpsust.

Töö viies osa pakub välja disaini nii trükkplaadile kui ka mõõtesüsteemile, et tagada süsteemi mugavam käsitlemine.

Kuues osa koosneb mõõtesüsteemi omahinna arvutusest.

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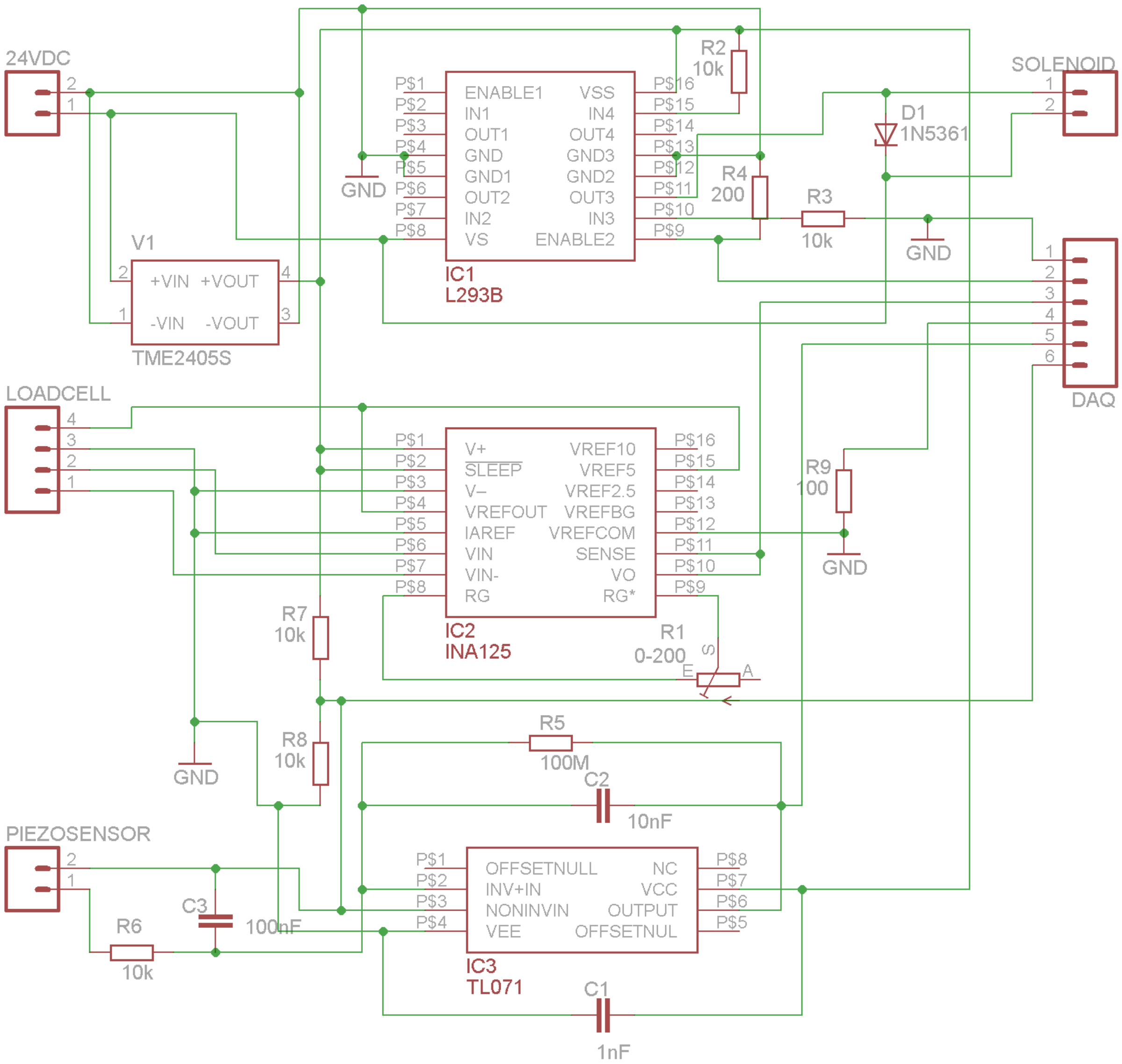
Appendix A


24VDC
V1
+VIN +VOUT
-VIN -VOUT
TME2405S
ENABLE1 VSS
IN1 IN4
OUT1 OUT4
GND GND3
GND1 GND2
OUT2 OUT3
IN2 IN3
VS ENABLE2
IC1
L293B
R2
10k
R4
200
R3
10k
GND
SOLENOID
D1
1N5361
DAQ
LOADCELL
V+ VREF10
SLEEP VREF5
V− VREF2.5
VREFOUT VREFBG
IAREF VREFCOM
VIN SENSE
VIN- VO
RG RG*
IC2
INA125
R1
0-200
R9
100
R7
10k
R8
10k
R5
100M
C2
10nF
PIEZOSENSOR
R6
10k
C3
100nF
OFFSETNULL NC
INV+IN VCC
NONINVIN OUTPUT
VEE OFFSETNUL
IC3
TL071
C1
1nF

Appendix B

noliac
Ceramics

Final test sheet

PLATE	L[mm]	W[mm]	TH[mm]
	15	15	1

Customer:	Tallinn University	Frequency:	
Noliac order:	901150419	Material:	NCE51
Invoice:		Batch:	
Measured by:	MPE	Pol. Date:	11.2.2015
Quantity:	1	Meas. Date:	5.5.2015

Note:	Thickness mode			Measured on 1KHz				
Nr.	Fs	Fp/Fs	Z Ω	C^T pF	tan δ 10^{-4}	ε_r	k_t	Qm
1	2003.1	1.126	2.6	3428.0	143	1722	0.498	
Minimum:								
	2003.1	0.000	2.6	3428	143	1722	0.498	
Maximum:								
	2003.1	0.000	2.6	3428	143	1722	0.498	
Average value:								
	2003.13	**########**	**2.6**	**3428**	**143.0**	**1721.5**	**0.498**	
Sigma:								
	##########	#########	##########	##########	##########	##########	##########	
Variation in %:								
	########	**########**	**########**	**########**	**########**	**########**	**########**	

	Thicknes mode			Measured on 1KHz		
100% inspection:	fs	fp/fs	|Z|	C^T	tan δ	

Approve by:

According to:
Made by: MSU

Date: 3/12/2009
Number of template: B-P6.2-03

noliac
ceramics

Final test sheet

PLATE	L[mm]	W[mm]	TH[mm]
	20	4	2

Customer:	Tallinn University	Frequency:	
Noliac order:	901150419	Material:	NCE51
Invoice:		Batch:	
Measured by:	MPE	Pol. Date:	17.4.2009
Quantity:	1	Meas. Date:	5.5.2015

Note:	Thickness mode			Measured on 1KHz				
Nr.	Fs	Fp/Fs	Z Ω	C^T pF	tan δ 10^{-4}	ε_r	k_t	Qm
1	985.2	1.192	68.0	616.0	155	1740	0.584	
Minimum:								
	985.2	0.000	68.0	616	155	1740	0.584	
Maximum:								
	985.2	0.000	68.0	616	155	1740	0.584	
Average value:								
	985.20	########	68.0	616	155.0	1740.1	0.584	
Sigma:								
	##########	#########	##########	##########	##########	##########	##########	
Variation in %:								
	########	########	########	########	########	########	########	

100% inspection:	Thicknes mode			Measured on 1KHz		
	fs	fp/fs	|Z|	C^T	tan δ	

Approve by:

According to:
Made by: MSU

Date: 3/12/2009
Number of template: B-P6.2-03

noliac
Ceramics

Final test sheet

PLATE	L[mm]	W[mm]	TH[mm]
	10	5	1

Customer:	Tallinn University	Frequency:	
Noliac order:	901150419	Material:	NCE51
Invoice:		Batch:	
Measured by:	MPE	Pol. Date:	27.3.2015
Quantity:	1	Meas. Date:	5.5.2015

Note:	Thickness mode			Measured on 1KHz				
Nr.	Fs	Fp/Fs	Z Ω	C^T pF	tan δ 10^{-4}	ε_r	k_t	Qm
1	2108.0	1.079	16.3	865.0	134	1955	0.409	
Minimum:								
	2108.0	0.000	16.3	865	134	1955	0.409	
Maximum:								
	2108.0	0.000	16.3	865	134	1955	0.409	
Average value:								
	2108.00	########	16.3	865	134.0	1954.8	0.409	
Sigma:								
	##########	########	##########	##########	##########	##########	##########	
Variation in %:								
	########	########	########	########	########	########	########	

	Thicknes mode			Measured on 1KHz		
100% inspection:	fs	fp/fs	|Z|	C^T	tan δ	

Approve by:

According to:
Made by: MSU

Date: 3/12/2009
Number of template: B-P6.2-03

Appendix C

```
%Piezoelectric coefficient uncertainty calculation
syms Cf V0 Vf Wmin Wmax Vmin Vmax

%Equation for piezoelectric coefficient calculation
d33=((Cf*(-V0))/((Wmin+((Wmax-Wmin)*((Vf-Vmin)/(Vmax-
Vmin))))*0.009807))*1*10^12;

%Partial derivatives
dCf=diff(d33,Cf);
dV0=diff(d33,V0);
dVf=diff(d33,Vf);
dWmin=diff(d33,Wmin);
dWmax=diff(d33,Wmax);
dVmin=diff(d33,Vmin);
dVmax=diff(d33,Vmax);

%Uncertainties for voltage measurements by the DAQ
%Voltage range
VRange=5;
%From measurement
Vacc=0.00031;
%from range 5V
Vaccr=0.00008;

%Values
% Capacitor Cf
Cf=9.23*10^-9;
%Charge amplifier's output voltage
V0=0.528;
%Load cell amplifier's output voltage
Vf = 2.21;
%Minimum weight used for adjusting the load cell
Wmin=200;
%Maximum weight used for adjusting the load cell
Wmax=1800;
%Load cell amplifier's output voltage corresponding to the
minimum weight used for adjusting the load cell
Vmin=0.44;
%Load cell amplifier's output voltage corresponding to the
maximum weight used for adjusting the load cell
Vmax=3.78;

%Uncertainties
%Capacitors Cf 5% uncertainty
Cfunc=9.23*10^-12;
Cfunc=Cfunc/2;
%Uncertainty from DAQ measurements
V0unc=(V0*Vacc)+(VRange*Vaccr);
```

```
V0unc=V0unc/2;
Vfunc=(Vf*Vacc)+(VRange*Vaccr);
Vfunc=Vfunc/2;
Vmaxunc=(Vmax*Vacc)+(VRange*Vaccr);
Vmaxunc=Vmaxunc/2;
Vminunc=(Vmin*Vacc)+(VRange*Vaccr);
Vminunc=Vminunc/2;
%1800g uncertainty 41 mg
Wmaxunc=0.0041;
Wmaxunc=Wmaxunc/2;
%200g uncertainty 10 mg
Wminunc=0.0010;
Wminunc=Wminunc/2;
%NCE51 material uncertainty 22/2=11
uNCE51=11;
%Uncertainty calculated for the 20 measurement values
uStat =2,8;

%Standard uncerατinty of the piezoelectric value d33 (pC/N)

d33unc=sqrt((dCf^2*Cfunc^2)+(dV0^2*V0unc^2)+(dVf^2*Vfunc^2)+(d
Wmin^2*Wminunc^2)+(dWmax^2*Wmaxunc^2)+(dVmin^2*Vminunc^2)+(dVm
ax^2*Vmaxunc^2)+uStat^2+uNCE51^2);
%Calculating the value of the equation d33unc
ud33 = eval(d33unc)

%Expanded uncertainty, Ud33
Ud33=ud33*2
```