Development of a dynamic mechanical analysis with a vibrating beam method

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Dissertation Thesis for Integrated Master in Mechanical Engineering

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To my parents

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Abstract

Most of metals have mechanical properties that do not vary to a great extent from low to high temperatures. Still, this fact is not true for a series of materials like polymers (which includes the materials studied in this thesis, adhesives) since the glass transition temperature, abbreviated as T_g, is often not too far from room temperature or their expected use temperatures. Below T_g, adhesives have a glass-like behaviour and over T_g they have a rubber-like behaviour. This concept is very important in learning about the properties of the materials, fundamental to all engineering design.

Common methods used to determine T_g are thermal and thermo-mechanical analyses. In the dynamic mechanical thermal analysis (DMTA), the mechanical properties are measured as a function of the temperature. Well below and well above T_g (in cross-linked materials), the damping is often quite low. At T_g, there is a peak corresponding to the maximum energy dissipated hence the maximum damping. Therefore, if the damping peak can be measured as a function of temperature then T_g can be found.

One method is to vibrate a constrained layer at resonance and to record the temperature and the amplitude. From the theory of forced vibration, the damping is proportional to the inverse of the amplitude. In other words, when the amplitude is at its minimum the damping is at its maximum. The exact value of the damping is not needed, but just the temperature at which it peaks. The objective here is to use a method based on that principle. An automatic device to maintain the constrained layer at resonance previously developed in the Department of Engineering of the University of Bristol, is used, optimized and redesigned at a low cost.

In this project, a box with electronic boards was made and which replaces the separate function generator, amplifier, feedback box, filter, DC rectifier and the switches from the T_g measuring system. The main objective was to reproduce the system at a low cost and ensure that it is precise and reliable.

Resumo

Enquanto a maior parte dos metais não apresenta uma varição profunda das propriedades mecânicas devido à temperatura, alguns materiais como os polímeros (grupo que inclui os materiais estudados nesta tese, os adesivos) são muito sensíveis à temperatura. Isto deve-se ao facto da sua temperatura de transição vítrea, abreviada Tg, não estar muito afastada da temperatura ambiente ou das suas temperaturas funcionamento. Abaixo da T_g, os adesivos comportam-se de forma vítrea enquanto acima desta temperatura os seu comportamento assemelha-se ao de uma borracha. Este conceito é de extrema importância no estudo das propriedades dos materiais, fundamental para todo o projecto de engenharia envolvendo adesivos.

Os métodos mais comuns para determinar a T_g consistem em análises térmicas e termo-mecânicas. Na análise termo-mecânica dinâmica (Dynamic Mechanical Thermal Analysis - DMTA), as propriedades são determinadas em função da temperatura. Para temperaturas muito afastadas da Tg, o amortecimento é consideravelmente baixo. Na T_g, surge um pico no amortecimento, correspondente à máxima dissipação de energia. Se o pico do amortecimento for medido em função da temperatura, a Tg pode então ser determinada.

Um método consiste em vibrar uma camada de adesivo, devidamente restringida, à sua frequência de resonância e registar a temperatura e amplitude. Segundo a teoria da vibração forçada, o amortecimento é proporcional ao inverso da amplitude. Por outras palavras, quando a amplitude está no valor mínimo, o amortecimento está no valor máximo. O valor exacto do amortecimento não é necessário, apenas a temperatura à qual ocorre. O objectivo deste trabalho consiste em usar um método baseado neste princípio. Um aparelho capaz de manter um provete de adesivo em resonância, incialmente desenvolvido na Universidade de Bristol, foi adaptado, optimizado e redesenhado com baixos custos.

Neste projecto foram desenvolvidas as necessárias placas electrónicas e respectiva caixa para as conter, substituindo gerador de funções, amplificador, caixa de resonância, filtro, rectificador de sinal e botões que anteriormente estavam montados separadamente. O principal objectivo é a construção de um sistema de baixo custo, mas preciso e fiável.

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Contents

List of Acronyms

BNC - Bayonet Neill Concelman

DMA – Dynamic Mechanical Analysis

DMTA – Dynamic Mechanical Thermal Analysis

DSC – Differential Scanning Calorimetry

EAGLE - Easily Applicable Graphical Layout Editor

FEUP – Faculty of Engineering of the University of Porto

IDMEC – Institute of Mechanical Engineering

IEC - International Electrotechnical Commission

SMPT - Materials and Technological Processes Group

TA - Thermal Analysis

TMA – Thermal Mechanical Analysis

List of Figures

List of Tables

1 Introduction

1.1 Background and motivation

Most of metals have mechanical properties that do not vary to a great extent from low to high temperatures. Still, this fact is not true for a series of materials like polymers (which includes the materials studied in this thesis, adhesives) since the glass transition temperature, abbreviated as T_g, is often not too far from room temperature or their expected use temperatures. Below T_g, adhesives have a glass-like behaviour and over T_g they have a rubber-like behaviour. This concept is very important in learning about the properties of the materials, fundamental to all engineering design **[1]**.

In this thesis is described a method of measuring the glass transition temperature, by dynamic mechanical analysis, in which the glass transition temperature can be obtained by determining the temperature at which the peak value of damping is observed. The method reported here uses a fast heating technique with a feedback circuit that maintains the specimen at resonance. By doing the test in about a minute, polymer cure is not altered significantly by the heating cycle.

1.2 Problem definition

Available devices for measuring T_g are currently expensive and the measurement process is complicated. Their long work cycles might over cure the adhesive or dry a previously wet adhesive. The device presented in this work is faster to use and less complex. This device allows us to determine the T_g with accuracy, is relatively fast and also economic, when compared to the general methods used for measuring T_g.

1.3 Aim of the study

This research aims to develop a reliable device based on previous works from the University of Bristol. This device is optimized so that the apparatus can be used in a easier way to measure T_g.

The main objectives of this study are:
- Develop a device based on the one developed at the University of Bristol,
- Develop electronic boards for power supply, function generator, amplifier, filter, feedback box, DC rectifier,
- Develop a housing for the electronic boards,
- Ensure that it is a low cost and functional device,
- Establish a reliable and simple procedure to obtain data and validate the results.

1.4 Research methodology

In this thesis an T_g measurement system was optimized in order to make it easier to use for the operator. The measurement of T_g is important and to get a good result a reliable, compact and simple to use system is needed. This work resulted in manufacturing an enclosure that contain all the key elements that replaced most of the devices used in the previous system.

This measure was taken because the previous system was occupying a lot of space and errors appeared because of operator mistakes or wrong connections.

The actual system consists of an enclosure which contains six electronic boards, two power supplies, switches, potentiometers and soldered wires which connects the boards. The boards were made to replace the devices that were occupying too much space and that had more functions than needed.

The boards that are inside the enclosure are the following: the power board, function generator, amplifier, filter, feedback box and DC rectifier. The power board was made in order to connect the power supplies to it and to feed power to the other boards. The function generator generates a wave to the specimen, which is amplified by the amplifier board. The feedback box is keeping the system in a closed loop by making the specimen constantly vibrate at the natural resonance frequency. The filter transforms the square wave from the feedback box into an approximately sinusoidal one and the DC rectifier has an output which connects to a data acquisition board in order to find the maximum damping. The T_g is found where the maximum damping appears.

A small experimental procedure was also made to validate the new built system made in this work.

1.5 Plan of the thesis

The structure of the thesis is summarised below:

This thesis is divided into six chapters that describe in detail the theoretical and experimental work developed.

Chapter 1 (Introduction) introduces the dissertation subject and presents the main objectives of this work.

Chapter 2 (Literature review) describes the theory about *Tg* and the general methods used for measuring it, like:

- Thermal mechanical analysis (TMA),
- Differential scanning calorimetry (DSC) and,
- Dynamic mechanical thermal analysis (DMTA).

Chapter 3 (T_g apparatus overview) describes the previous system used for measuring T_g and the operating principle of the system.

Chapter 4 (Improvements brought to the system) presents the process of building the new T_g measuring system. It has a detailed description of each step made during the development of the housing that contains the boards that replace devices from the old system and the power supplies. It also contains information about development and layout of each board. At the end of the chapter a measurement comparison is made between the old and the new system.

Chapter 5 (Conclusions) summarizes the main conclusions of this thesis.

Chapter 6 (Future works) provides some suggestions for future works and improvements.

2 Literature review

As this work describes a method of determining T_g based on a dynamic mechanical thermal type of analysis, it is important to present what T_g is and the main methods currently used for its determination, like thermal mechanical analysis (TMA), differential scanning calorimetry (DSC) and dynamic mechanical thermal analysis (DMTA) **[2]**.

2.1 Glass transition temperature

Knowing the properties of a material is important to all engineering design. Equally, the way in which, and the circumstances whereby these properties change, is also important. In a polymer, the mechanical properties are highly dependent both on temperature and on the time scale of any deformation. Also, polymers are viscoelastic and thus exhibit some of the properties of both viscous liquids and elastic solids.

The glass–liquid transition is the reversible transition from a hard and relatively brittle state into a molten or rubber-like state. The glass-transition temperature T_{g} is always lower than the melting temperature, T_{m}, of the crystalline state of the material, if one exists. Many physical properties change at the glass transition temperature, including coefficient of thermal expansion, heat capacity, refractive index, mechanical damping, and electrical properties **[3]**.

The glass transition can be understood by considering the nature of the changes that occur at the temperature in question. As a material is heated to this point and beyond, molecular rotation around single bonds suddenly becomes significantly easier. A number of factors can affect the ease with which such molecular rotation takes place, and hence influence the actual value that the glass transition temperature takes. The inherent mobility of a single polymer molecule is important and molecular features which either increase or reduce this mobility will cause differences in the value of T_{g}. In addition, interactions between polymer molecules can lead to restrictions in molecular mobility, thus altering the T_{g} of the resulting material **[4]**.

At low temperatures a polymer may be glass-like (glassy), with a value of Young's modulus in the region of 1 – 5 GPa. In this region, only a little intermolecular movement is allowed, and the polymer will break at strains of a few percent. At high temperatures, the polymer may be rubber-like, with a modulus in the region 0.1 – 1 GPa. In this region, it exhibits a large elongation when a load is applied and may withstand large extensions of the order of 100% or more with no permanent deformation.

For thermoplastics, a further increase of temperature will cause large scale molecular movement and the polymer softens. However, thermosets do not generally melt. In the intermediate temperature range, called the glass transition region, the polymer is neither glassy nor rubber-like and has an intermediate modulus and develops viscoelastic properties. The glass transition temperature T_g is related to the molecular motion. Above T_g, the polymer chains move freely which makes the polymer very flexible. Below T_g, the chains have much less mobility and the polymer is effectively 'frozen'. T_g is usually explained by the theory of free volume. A polymer is made up of occupied volume and free volume. As the polymer is heated, the free volume increases, easing the movement of the backbone. In the T_g region, several of the material properties of the polymers are known to change. These properties include: the density, the stiffness or Young's modulus, the damping properties, and the coefficient of linear expansion. It is important to know the temperature at which the glass transition will occur for a particular polymer. For rubbers, the normal operating temperature range should be much higher than T_g; otherwise, the material will have brittle behaviour properties, and will not perform its function. For a structural adhesive, T_g takes on a different role and the normal operating temperature should be more than 20ºC lower than T_g **[5]**.

The most common methods used to determine Tg are thermal and thermo-mechanical analysis: Differential Scanning Calorimetry (DSC), Dynamic Mechanical Analysis (DMA) and Thermal Mechanical Analysis (TMA). Although these measurement techniques are excellent research tools, they are not suitable for situations where the long time exposure used in the test can change the physical state of the studied specimen like the degree of cure, the thermal history or the moisture uptake of the specimen.

As adhesives are polymeric materials, it is very relevant to know their T_g so that we can predict how an adhesive joint will react within a certain temperature range. Polymers used as adhesives have an amorphous or semi-crystalline structure. This means that there is significant degree of randomness in the distribution of the polymeric chains. The motion of the atoms in these chains is the underlying factor for the existence of the T_g. Below the T_g there is a lack of rotational and translational movement of the atoms that consists in the polymeric chain. They are frozen in place and this translates into a stiff adhesive that cannot resist large deformations. Above T_g there is more freedom for this molecular motion to occur, which leads to more flexibility and deformation capability on the adhesive **[6]**.

The T_g of a material can be determined by studying the variation of certain parameters with temperature. The modulus of elasticity, damping, specific volume and enthalpy all suffer noticeable changes around the T_g value. Figure 1 shows schematically how Young's modulus and the damping of a polymer vary with temperature.

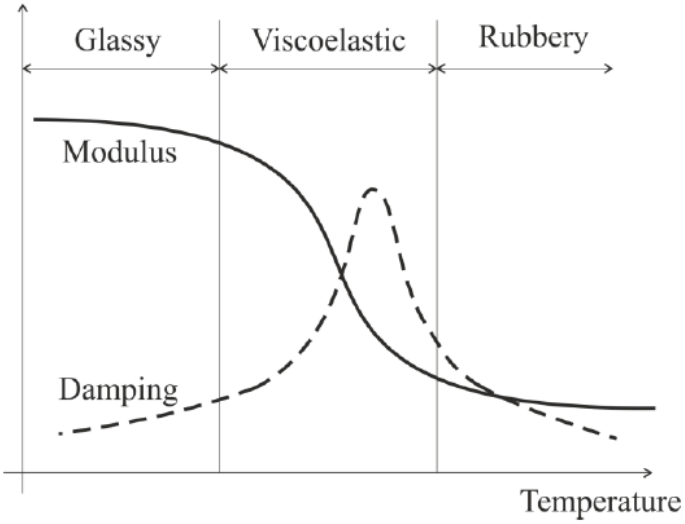


Figure 1 - Young's modulus and damping vs. temperature of a polymer [5]

Different experimental methods study the variation of these basic properties to determine the range of temperatures where there is an important change of behaviour. This permits the precise identification of a T_g value.

2.2 Methods used for measuring T_g

The methods most commonly used for this purpose are:

- Thermal Mechanical Analysis (TMA),
- Differential Scanning Calorimetry (DSC),
- Dynamic Mechanical Thermal Analysis (DMTA).

2.2.1 Thermal mechanical analysis (TMA)

Thermal mechanical analysis (TMA) is a technique used in thermal analysis, a department of materials science which studies the properties of materials as they change with temperature. Mechanical testing is made to measure mechanical properties of materials using different test specimen and fixture geometries using a range of probe types. Measurement is desired to take place with minimal disturbance of the material being measured. Some characteristics of a material can be measured without disturbance, such as dimensions, mass, volume, density. However, measurement of mechanical properties normally involves disturbance of the system being measured.

TMA measurements record the changes made by changes in the free volume of a polymer.

Figure 2 shows a diagram of the equipment that is used in this method. There is a sudden expansion during the change of the polymer state from vitreous to rubber, which allows the measurement of T_g.

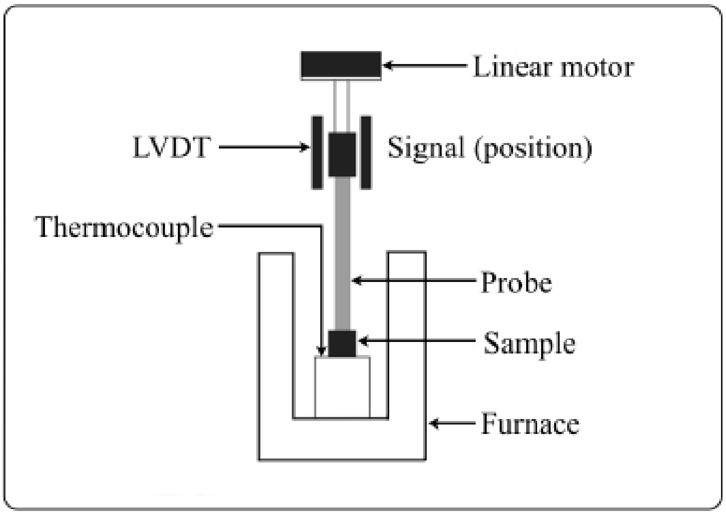


Figure 2 – Vertical TMA instrument [2]

The two most important fields of TMA application in polymers are the determination of the thermal expansion coefficient and the measurement of T_g. Sometimes the melting point (T_m) of semi crystalline polymers is also measured using this method [7].

These quantities can be measured only once in TMA, as shown Figure 3. The glass transition temperature is determined as the point of intersection of the curves of glass and expansion phases of rubber **[2]**.

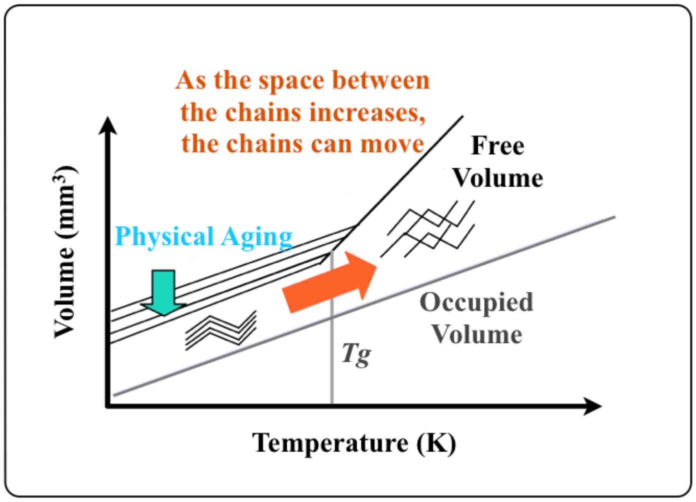


Figure 3 – Display of a TMA curve in expansion [8]

Occasionally a certain dynamic loading is used to study the elastic behaviour of materials. The name for this version is the DLTMA (Dynamic Load TMA). The TMA technique covers many materials such as plastics, composites, ceramics and adhesives. The method is also applicable to the determination of properties such as, for example, the coefficient of thermal expansion, T_g and softening temperature. However, it is a method that has a high dependence on sample preparation and poor repeatability **[9]**.

2.2.2 Differential scanning calorimetry (DSC)

Differential Scanning Calorimetry (DSC) measures the temperatures and heat flows associated with transitions in materials as a function of time and temperature in a controlled atmosphere. These measurements provide quantitative and qualitative information about physical and chemical changes that involve endothermic or exothermic processes, or changes in heat capacity.

The DSC is a measurement method, which is part of a group of techniques called thermal analysis (TA). The TA is based upon the detection of changes in the heat content (enthalpy) or the specific heat of a sample with temperature.

As thermal energy is transmitted to the sample, its enthalpy increases and its temperature rises by an amount determined, for a given energy input, by the specific heat of the sample. The specific heat of a material changes slowly with temperature in a particular physical state, but alters discontinuously at a change of state. As the sample temperature increases, the supply of thermal energy may induce a physical or chemical process in the sample, e.g. melting or decomposition, accompanied by a change in enthalpy, latent heat of fusion, heat of reaction etc. Such enthalpy changes may be detected by thermal analysis and related to the process occurring in the sample. The measuring principle of DSC is to compare the rate of heat flow of the sample to an inert material that are heated or cooled at the same rate **[6]**. The changes in the sample are associated with absorption of heat. By comparing the heat absorption of the sample with that of the inert material, a differential heat flow is measured. The heat flow curves have a peak, where the area under the peak is directly proportional to the enthalpy change and its direction indicates the thermal event is an endothermic process. The analysis of a DSC thermogram enables to determine two important parameters, the transition temperature peak (T_g) and the enthalpy **[10]**. Figure 4 shows a temperature versus heat flow graph, which can be used to identify T_g as described above.

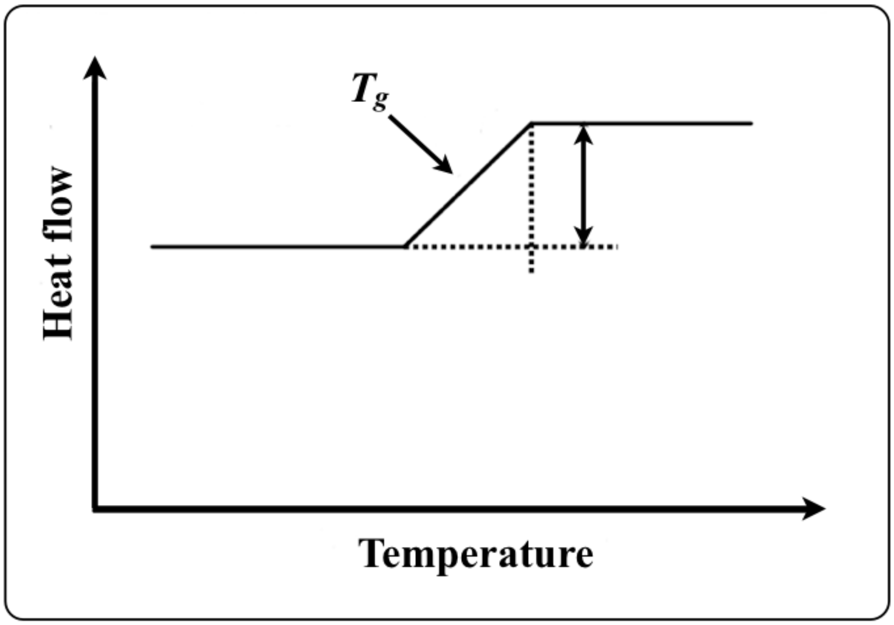


Figure 4 - Determination of Tg from the combination of heat capacity and temperature [11]

The equipment used for this measurement consists in a metal disk, which connects a sample and a reference with low heat flow resistance. This connection is placed inside a heating device. The enthalpy or heat capacity of the sample when experiencing changes causes a differential temperature measured against a reference sample **[12]**. This is then registered and is related to the change in enthalpy in the sample by an experimental calibration system **[11]**.

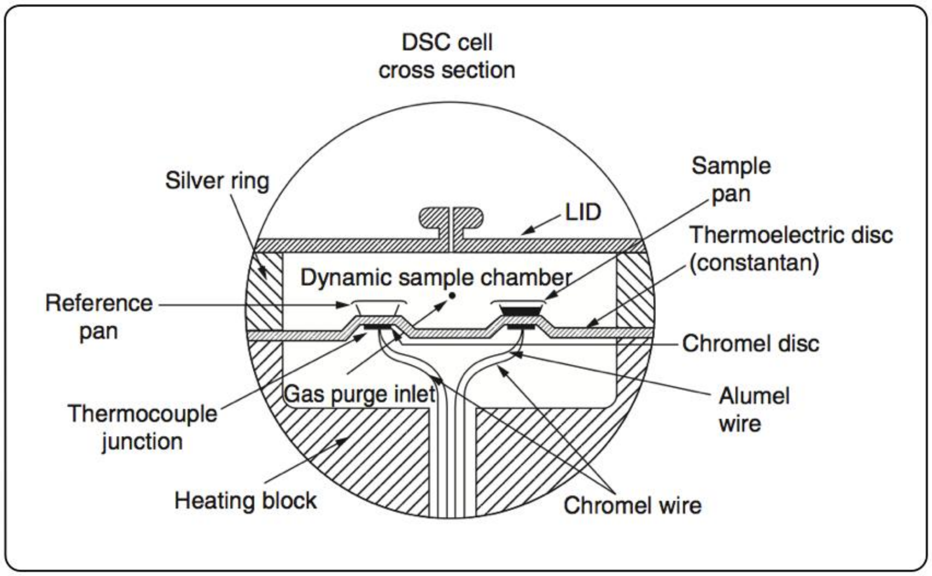


Figure 5 – Cross-section of a Du Pont (now TA Instruments, Pennsylvania , United States) DSC heat flux cell

This system has some advantages such as the easy preparation of the samples, because it makes use of small samples and is an automated procedure **[3]**, applied to solids and liquids, and covers a wide range of temperatures.

With the help of DSC the following can be found:

- Glass Transitions
- Melting and Boiling Points
- Crystallization time and temperature
- Percent Crystallinity
- Heats of Fusion and Reactions
- Specific Heat
- Oxidative/Thermal Stability
- Rate and Degree of Cure
- Reaction Kinetics
- Purity

The most important disadvantages and concerns are that the results are susceptible to systematic errors based on incorrect calibrations by poor packaging and preparation of samples or by an inappropriate reference.

2.2.3 Dynamic mechanical thermal analysis (DMTA)

The dynamic mechanical (thermal) analysis it is abbreviated as DMA, but also called DMTA [3], is one of the common method used to measure the glass transition temperature of polymeric materials. It is most useful for studying the viscoelastic behavior of polymers. DMA measures the viscoelastic moduli, storage and loss modulus, damping properties, and tan delta, of materials as they are deformed under a period (sinusoidal) deformation (stress or strain).

The DMTA apparatus measures the modifications in mechanical behaviour of any material as a function of temperature, frequency, time, stress or combinations of these parameters over the temperatures between -150 ºC and +500 ºC with experimental scanning rates of 0.1 to 5 ºC/minute for heating and cooling. This is a very delicate technique, especially for finding T_g, which give rise to a pronounced maximum damping in a dynamic mechanical experiment.

With the help of DMTA we can determine with accurate information the following:

- Glass Transitions
- Stiffness,
- Cure,
- Ageing,
- Melting behaviour,
- Stress/strain,
- Mechanical hysteresis.

The analyser is utilized to get information regarding the structural properties of materials, product and materials development, process optimisation, quality control and production process support. The materials that can be studied include polymers (both thermoplastics and thermosets), elastomers, adhesives, optical materials and metals ranging from solid bars to coatings, films and fibres **[13]**.

A typical DMA device with grips to hold the sample and an environmental chamber to provide different temperatures is shown in Figure 6. The mounted sample is an environmental chamber and can slide to and encompasses the sample.

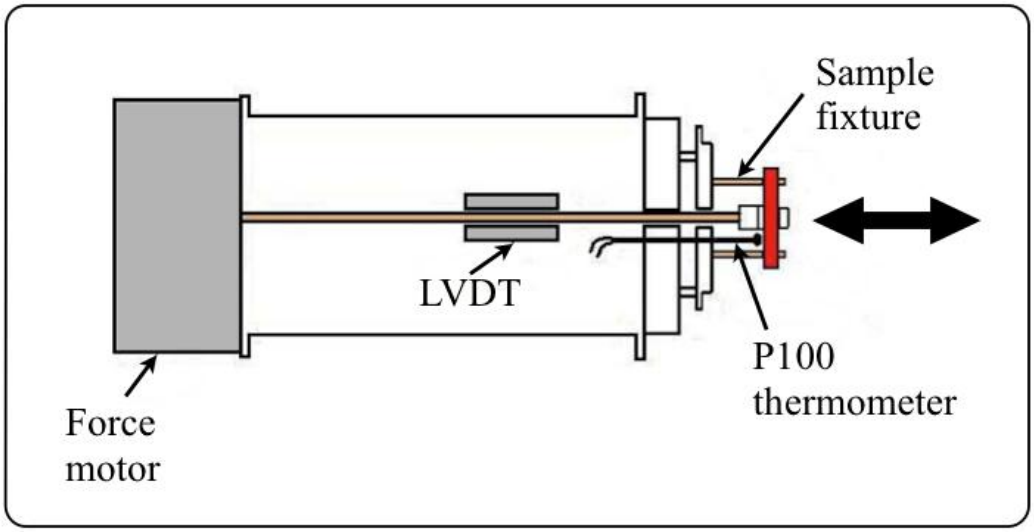


Figure 6 – Sketch of a DMA operation (Perkin Elmer Instruments, Massachusetts, United States of America)

The polymer viscoelastic property is determined by dynamic mechanical analysis, where a sinusoidal force (tension σ) is sent to a material and the resulting displacement is measured **[14]**. For a elastic solid, the resulting stress and deformation will be perfectly in phase. For a purely viscous fluid, there is a phase delay δ of 90 degrees with respect to the deflection voltage.

Polymers have viscoelastic characteristics where the phase delays δ will occur during the DMA testing.

The storage modulus measures the stored energy, which represents the elastic portion, and the loss modulus measures the energy dissipated as heat, representing the viscous portion **[2]**. Figure 7 presents a graph with curves obtained by DMA of an epoxy adhesive. The curves are tan δ, the storage modulus, E' and loss modulus, E", as a function of temperature. The glass transition point (and the corresponding T_g) is observed at the inflection point of E', the maximum peak of E" and tan δ.

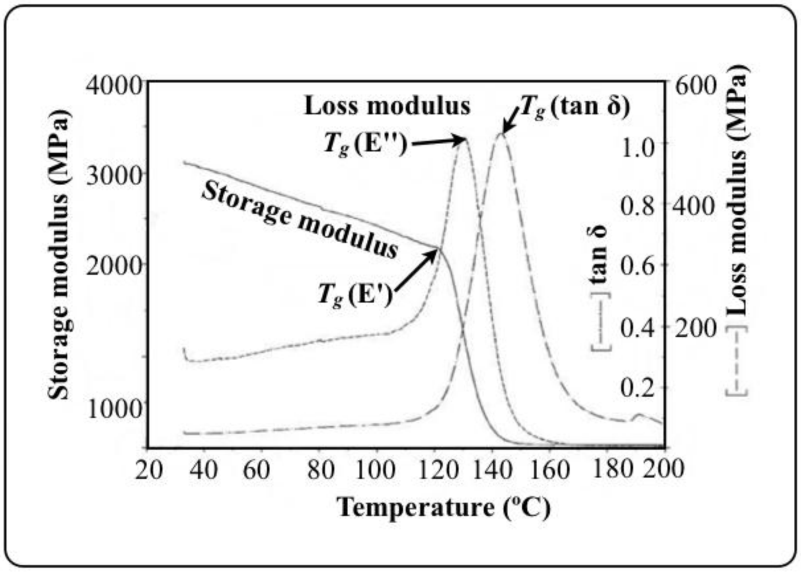


Figure 7 - Curves E', E'' and tan δ of a DMA of an epoxy system [15]

For highly cross-linked polymers, the T_g section is wide and the test depends on parameters such as the frequency of the test and heating rate. By this technique, the T_g range extends from the point of inflection of the curve E' to the temperature of tan δ peak. According to Li et al **[15]**, the standard recommended for reporting T_g is the temperature of the temperature peak of E''. Although DMTA is as powerful as DSC for measuring T_g, it is time-consuming. Moreover, the method may change the curing state of the specimen or dry samples that are initially wet.

2.3 Comparison between TMA, DSC and DMTA

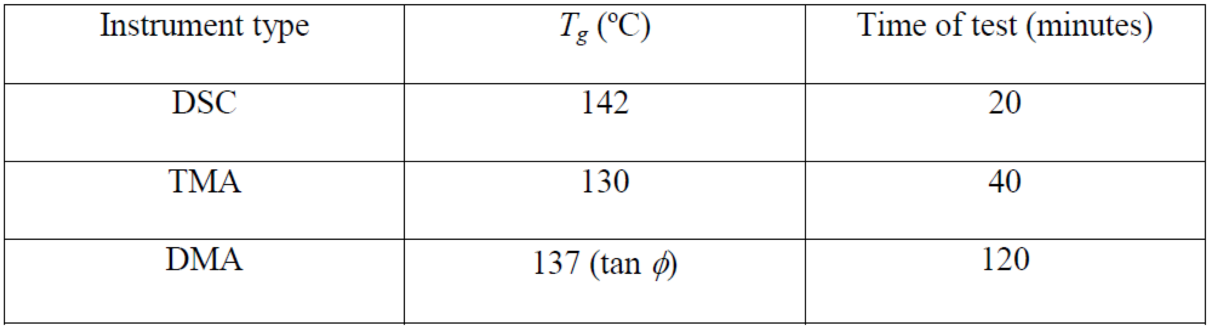
Polymers represent a large study field in which thermal analysis has strong applications. Thermoplastic polymers are usually found in packaging and household items, but for the analysis of the raw materials, effects of the many additive used (including stabilisers and colours) and fine-tuning of the moulding or extrusion processing used can be achieved by using DSC. An example is oxidation induction time (OIT) by DSC which can determine the amount of oxidation stabiliser present in a thermoplastic (usually a polyolefin) polymer material.

Thermal analysis of composite materials, such as carbon fibre composites or glass epoxy composites are often carried out using DMA or DMTA, which can measure the stiffness of materials by determining the modulus and damping (energy absorbing) properties of the material. Aerospace companies often employ these analysers in routine quality control to ensure that products being manufactured meet the required strength specifications. Formula 1 racing car manufacturers also have similar requirements. DSC is used to determine the curing properties of the resins used in composite materials, and can also confirm whether a resin can be cured and how much heat is evolved during that process. Application of predictive kinetics analysis can help to fine-tune manufacturing processes.

The TMA method measures the volumetric expansion of a specimen during heating. By analysing the expansion versus temperature curve, transition temperatures can be identified by changes in the curve slope.

Konarski [16] studied the effect of measuring instruments in the T_g of samples cured at 145ºC for 2 hours. Table 1 shows the glass transition temperature measured using the methods of DMA, DSC and TMA. This study supports the fact that T_g varies as a function of the measuring device, in this case T_g varies from 130 until 146 °C.

Table 1 - Effect of the type of measuring on the value of Tg [16]



Instrument type	T_g (ºC)	Time of test (minutes)
DSC	142	20
TMA	130	40
DMA	137 (tan ϕ)	120

The DSC test is one of the simplest and fastest methods to measure T_g. The method needs extremely small samples (usually 5-20 mg), which do not need special preparation. Unfortunately, this quick and easy to use method is not universally applicable to all materials. High load, high crosslink density, and other processes may mask the displacement due to T_g and make the transition difficult or impossible to identify. This technique becomes costly because of using large quantities of liquid nitrogen for each test to obtain the cooling of the sample.

The TMA analysis consists in simply heating the sample and measuring its dimensional change with a probe. This method also achieves relaxation of the stresses around the glass transition region, which sometimes leads to ambiguity in assigning a specific T_g and can measure a different value for the same sample if measured at a different point. The measured T_g, for instance, can vary when measured near the edge or at the centre of the sample.

The DMA is a technique, which consists on measuring the oscillation energy of bending applied to a cured sample. The stress in the sample is measured versus temperature. The method is very accurate, but T_g may be found in different ways, which will have different values.

3. T_g apparatus overview

The system used in this thesis measures damping changes of a specimen as a function of temperature. To that purpose, a specimen with adhesive is made to vibrate while its temperature changes. When the glass transition temperature is reached, the damping is at its maximum. This can be seen in
Figure 8.

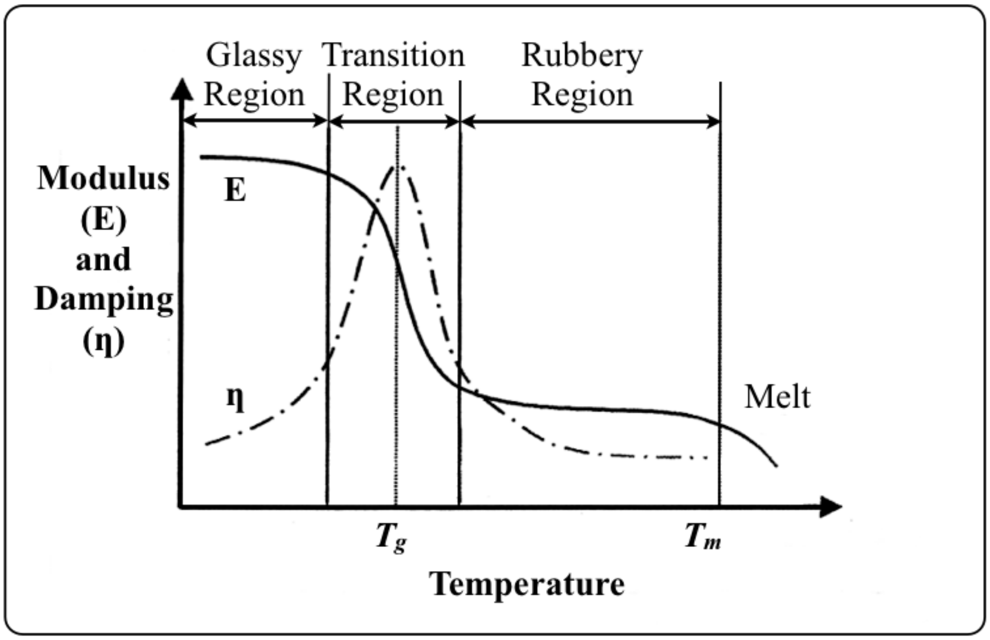


Figure 8 – Graph of damping and temperature for a polymer [17]

By determining this damping peak and the temperature at which it occurs we can accurately find the glass transition temperature of a polymer. This happens when the specimen starts to vibrates less during the experiment.

Damping is a measure of the ratio of energy dissipated as heat inside the material to the maximum energy stored in the material during one cycle of oscillation. There are several different, but related, quantities listed below which are normally used to characterize damping capacity **[7]**.

In its simplest case, vibration can be seen as steady state or freely decaying as shown in Figure 9.

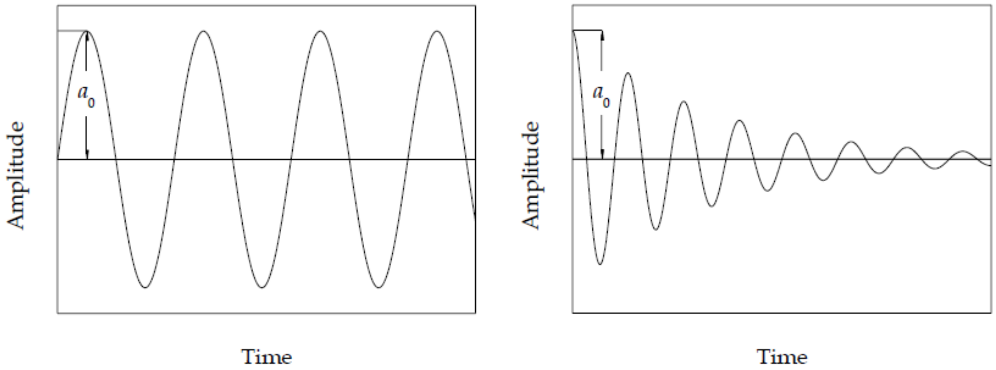


Figure 9 - Steady state under forced vibration, and transient vibration.

If the excitation frequency coincides with a resonance frequency, then the amplitude of vibration can be magnified as shown in Figure 10. If the damping is high, the transient decays more quickly, and the resonance amplitude is smaller.

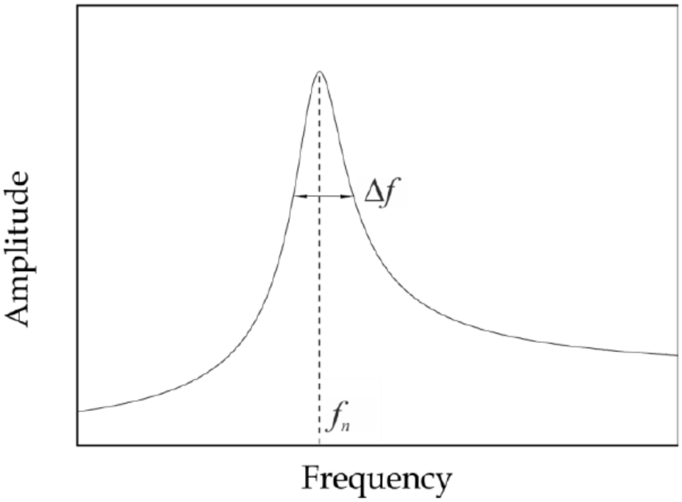


Figure 10 - Change of steady-state amplitude of vibration with the excitation frequency and the resonance frequency

The specific damping capacity is defined as the ratio of the energy dissipated per cycle to the maximum elastic energy stored per cycle, per unit volume. To measure this, the specimen is vibrated at its resonant frequency. The energy input required to maintain this steady state vibration is measured, and the maximum elastic stored energy is calculated from the geometry of the specimen and its elastic modulus.

Damping characteristics are very important in finding the T_g in polymeric materials. As a material reaches T_g, the damping properties will change significantly. The device developed during this thesis subjects a thin layer of adhesive to a constant and cycling oscillation. This oscillation movement is achieved with a pair of coils and magnets. The adhesive layer is mounted on an aluminium beam with a magnet mounted in each extremity of the beam. A coil is placed underneath each magnet. A variable electric current passes through the coils. When the current reaches its highest intensity (positive signal) the coils attract the magnets. When the coils do not have current the magnets go back to their original positions and when the current reaches its lowest intensity (negative signal) the coils push the magnets. If the current is changed cyclically, the beam and the adhesive layer are going to have oscillatory movements.

Figure 11 presents a schematic diagram of the system. In the following sections the importance of each part is explained.

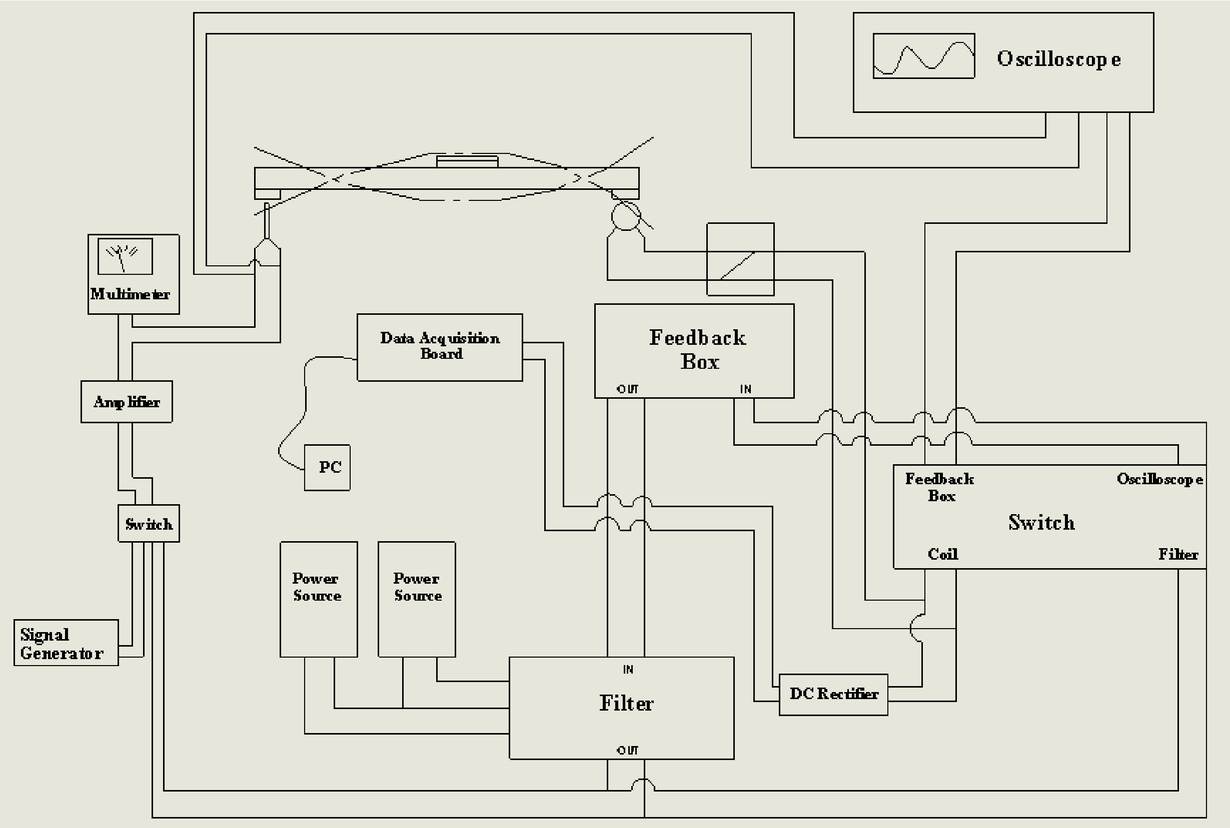


Figure 11 – Schematic diagram of the T_g system

Figure 12 presents the previous Tg measurement system. The purpose of this thesis is to make the system more compact, reliable and easier to use.

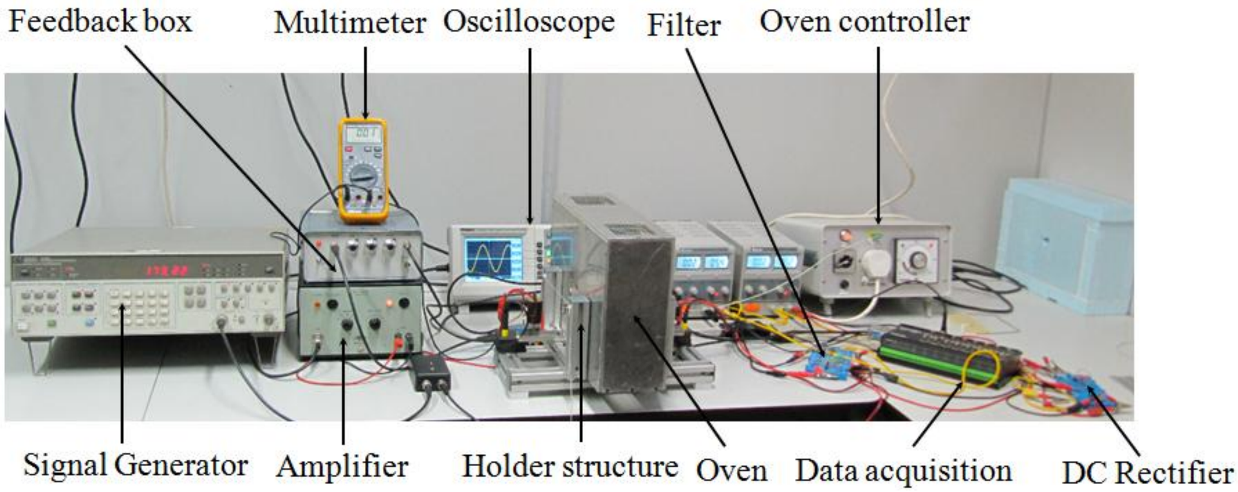


Figure 12 - T_g measurement system

3.1 Specimen

The beams and the upper plate, used in the measurement of $T_{g,}$ were made from aluminium alloy (2024). The beam has the dimensions of 250 x 12.5 x 3 mm^3 and the plate has the dimensions of 30 x 12.5 x 1 mm^3. The material was chosen for its high strength and low specific heat, which makes it suitable to make the beam specimen. More than that, aluminium has a low rigidity and low damping property **[11]**. The adhesive has a similar dimension to the top plate, but with twice the thickness (2 mm).

Figure 13 it is presents a study from which results that the best length for the adhesive is 30 mm. The graph shows that there are diminishing returns when the length of the adhesive layer is bigger. Adhesive layer of 30 mm has very alike performance with the one who has 40 mm and because of that there is no need to increase the adhesive layer length, especially as larger specimens will require bigger equipment to be manufactured.

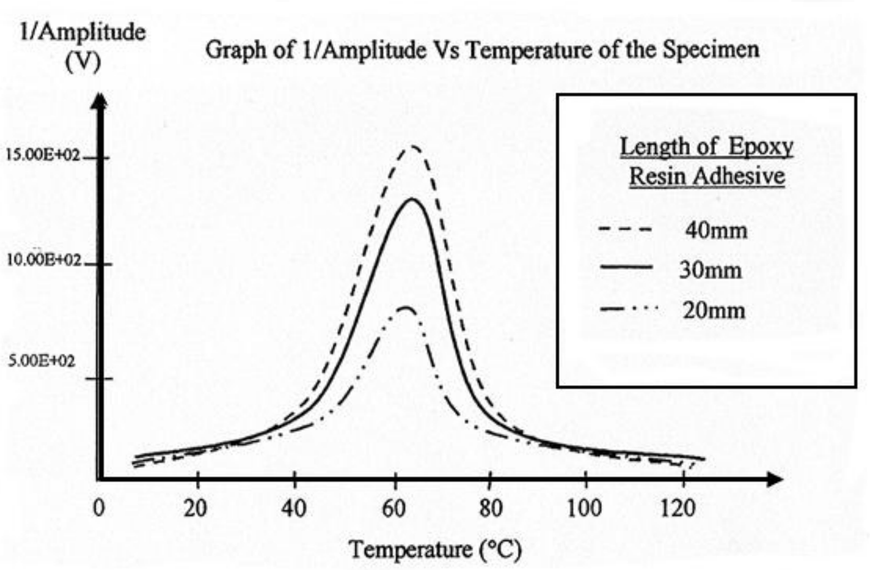


Figure 13 - Effect of the length of the epoxy resin adhesive on the damping properties [17]

Figure 16 presents the mould used to make the specimens. A very significant factor is that the assembly of the mould for specimen manufacture is made from bottom to top. It begins with plate 3 and ends with plate 1. Four screws are needed to tighten all the components to close the mould.

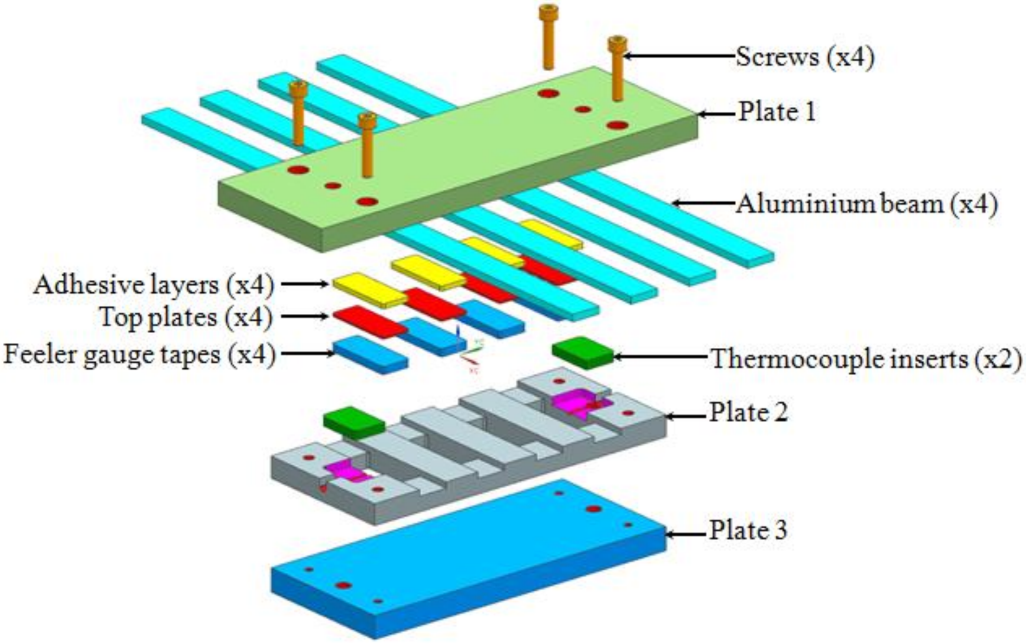


Figure 14 - View of the mould for specimen manufacture [18]

Plate 1 (plate that sits on top of the mould) is 20 mm longer per side than plate 2 and plate 3, allowing using it as two extra handles. Mould for specimen manufacture is easy to use and to handle even with thermal gloves. The total weight is just 1.9kg, which makes it a relatively lightweight mould. This allows easily placing and removing the assembled mould into the heated plates press.

Figure 15 presents an overview of the beam arrangement. The beam is supported by two strings. At those two spots where the strings touch the beam there is no vibration, so the damping measurement will not be affected. On each extremity, under the beam, are placed magnets which are very close to the coils. Above, the adhesive and metal plate can be found. The plate above the beam allows the adhesive to dissipate some energy and amplifies the influence of T_g for a better reading.

Figure 16 shows a thermocouple lead placed on an identical beam, called “dummy beam”, for the temperature of the adhesive measurement.

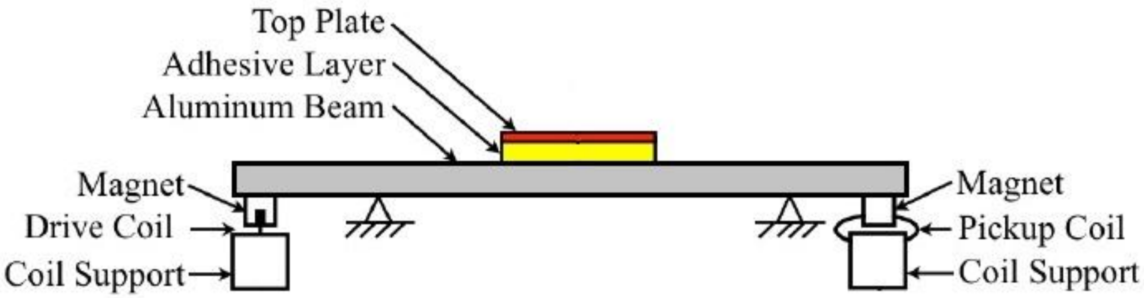


Figure 15 – Vibrating beam configuration

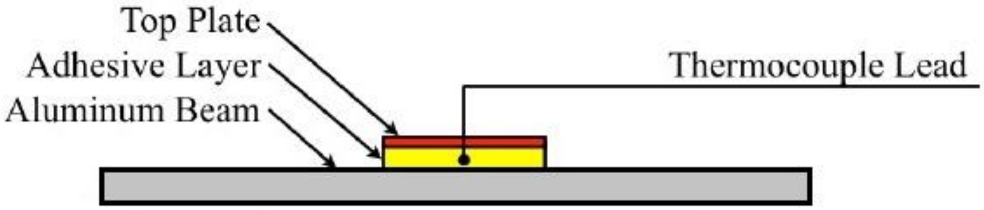


Figure 16 - Dummy beam

Figure 17 shows the polarity change of the coils and the effect they have in the movement of the specimen. With the polarity change, the beam with the adhesive vibrates up and down. The T_g can be observed because the adhesive is acting as a dumper and the beam vibrates less.

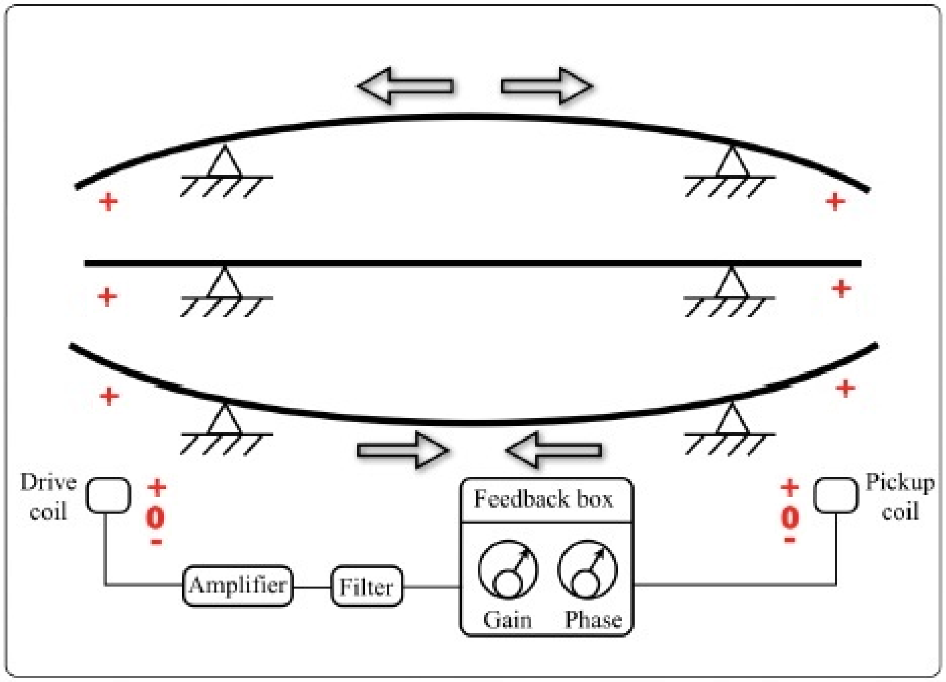


Figure 17 – Working principle of the Tg apparatus

If the temperature of the specimen changes during heating, while the oscillation movement occurs, there will be a temperature value where there is a big increase of damping. This means that the T_g is reached and the adhesive has started to have a rubber like behaviour, resisting the movement. If this increase in damping is detected, the temperature at which it occurs can be classified as being the T_g. The temperature of the specimen is measured with another specimen, called “the dummy”. It is located very close to the test specimen. This dummy specimen has a thermocouple lead put in the adhesive layer. If the thermocouple lead were installed in the real specimen, it would change the vibration behaviour of the specimen during the test and could hide the damping changes caused by the glass transition.

For finding results, the amplitude of the specimen vibration should be as large as possible. The maximum amplitude of vibration is reached when the specimen is forced to vibrate at its resonance frequency. This creates a problem during the heating phase, as the resonance frequency changes with the temperature and if the frequency of the current applied to the coils is significantly different from the resonance frequency the specimen might stop to vibrate. To ensure that the specimen is always vibrating, a feedback system is required. This system is explained in the following section.

An alternative method to produce the specimens can be used. Figure 18 presents a piece of precured adhesive that is fastened by a nut and bolt. For temperature to be measured, thermocouples can be put between the layers of the specimen. This type of specimen construction permits a fast and economic way to produce specimens. It is particularly useful for assessing the effect of environment on T_g as the adhesive sample can be easily removed from the sample and introduced in the environment to study.

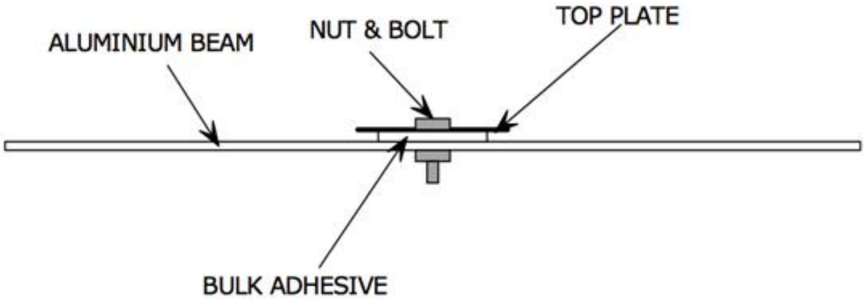


Figure 18 - Specimen made with pre-cured adhesive [11]

Specimens used in this thesis were made using the mould from the previous thesis **[18]**.

3.2 Holder structure

The structure of the oven holder must be practical, stiff, easier to adjust and use and must be cheap. Because of that, aluminium profiles, screws, nuts and fastening elements among others were used to build the holder for the oven and the specimens. A 3D drawing is shown in Figure 19 presenting this structure. This system was developed in the previous thesis **[18]**.

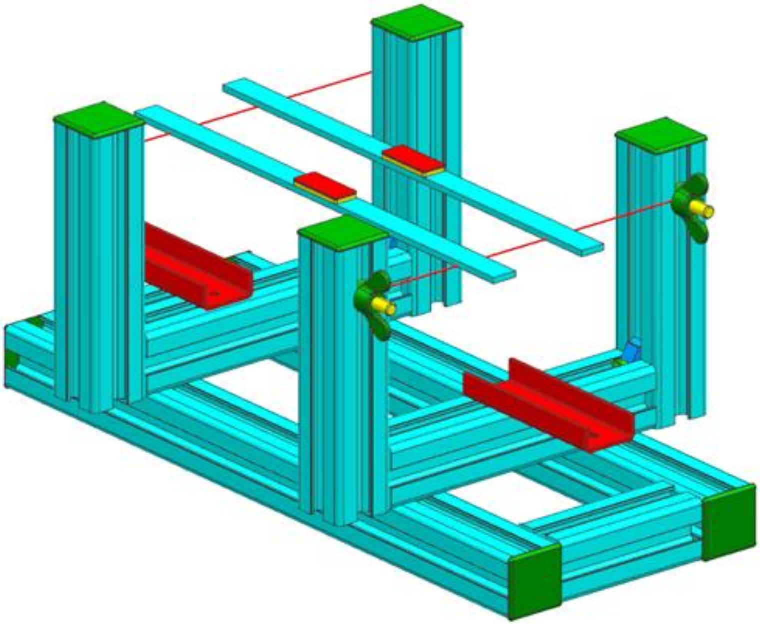


Figure 19 – 3D drawing of the holder structure along with a pair of specimens [18]

Figure 20 presents a picture of the physical holder structure along with the aluminium profiles, screws, nuts and fastening elements.

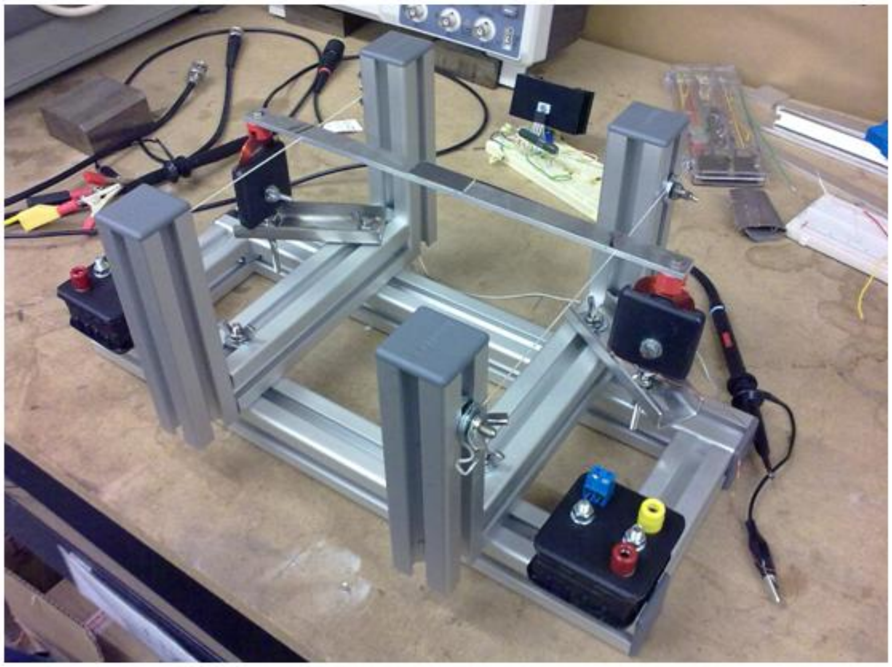


Figure 20 – Holder structure

3.3 Generating the signal

Figure 21 shows the part of the system which can start the test.

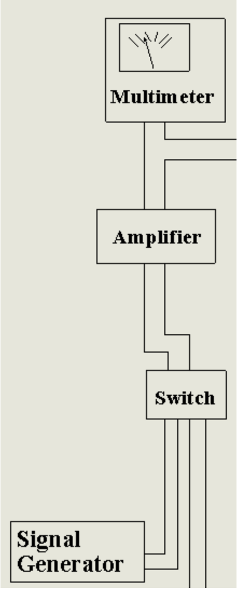


Figure 21 – Connection between Signal Generator, Amplifier, Multimeter and Switch

At the beginning of the T_g measurement, a signal generator transmits a cyclic wave at the resonance frequency of the specimen. The resonance frequency is found by the operator and it can be observed when beam starts to vibrate. The cyclic wave is amplified in a power amplifier and transmitted to the drive coil. This coil is responsible for the movement of the specimen.

The amplifier is used to amplify the frequency for a better observation of the vibration. For the system not to heat up, a multimeter is installed to monitor the amplifier so it won't transmit more than 1 Ampere (the maximum current allowed by the coils).

On the other side of the specimen is the pickup coil. This coil transforms the vertical movement of the magnet bolted to the specimen into a small electric current. This current is directly proportional to the amplitude of movement of the specimen. This can be used as a simple method to identify T_g. However, as the temperature changes, the resonance frequency of the specimen also changes and the frequency provided by the signal generator must be changed frequently to reassure that the specimen keeps vibrating. This is unwanted, as it requires constant human action until T_g is found.

To manage this problem, a device known as a feedback box was created. This system works in a closed loop, with feedback.

3.4 Feedback box

The heating and cooling of the specimen cause changes in properties such as damping, modulus and even dimensions. As a result, the frequency of the input signal needs to be constantly adjusted to keep the system vibrating at resonance. The feedback unit achieves this by first considerably amplifying the sinusoidal wave received from the pick-up coil. Then, this is modified to yield what is almost a square wave.

A phase shift circuit then alters the phase of the signal to correspond with that needed to input the power amplifier to maintain resonance (i.e. it allows for the various phase changes in the electronics between the coil and the input to the power amplifier). Finally, a filter with its central frequency approximately at the resonant frequency of the specimen is used to block frequencies that are outside a select bandwidth bracketing the first resonant frequency of the specimen. This transforms the square wave to a sinusoidal wave which is then amplified and sent via a power amplifier to the driving coil (the theory of feedback to maintain resonance is illustrated in Figure 22).

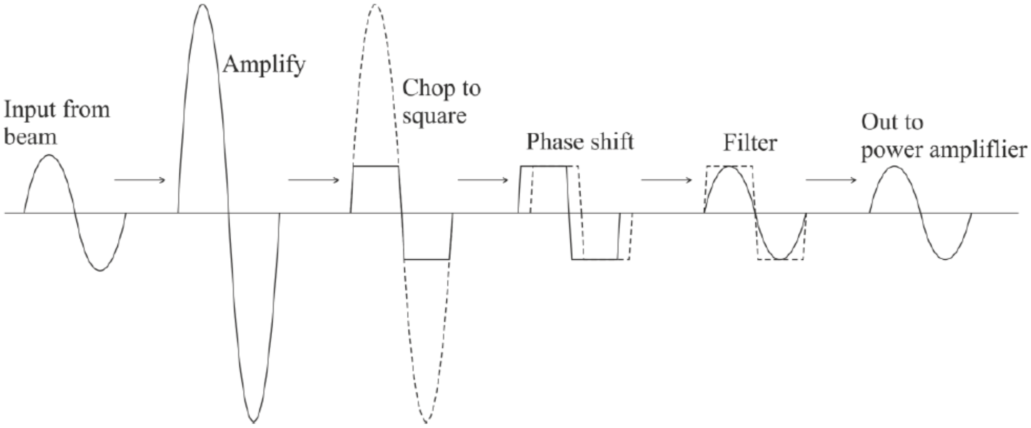


Figure 22 - Feedback circuit theory [5]

The above process follows directly any change in the resonance frequency caused by factors such as temperature. As a quick and continuous method, it completely eliminates the need for retuning and testing for each temperature. It should be noted that the T_g of polymers are frequency dependant. Therefore, the T_g measured with this system is for a particular value to frequency. A DC signal proportional to the resonance amplitude was plotted against time or temperature **[5]**.

Figure 23 presents the system working in a loop with the help of the feedback box and without the function generator.

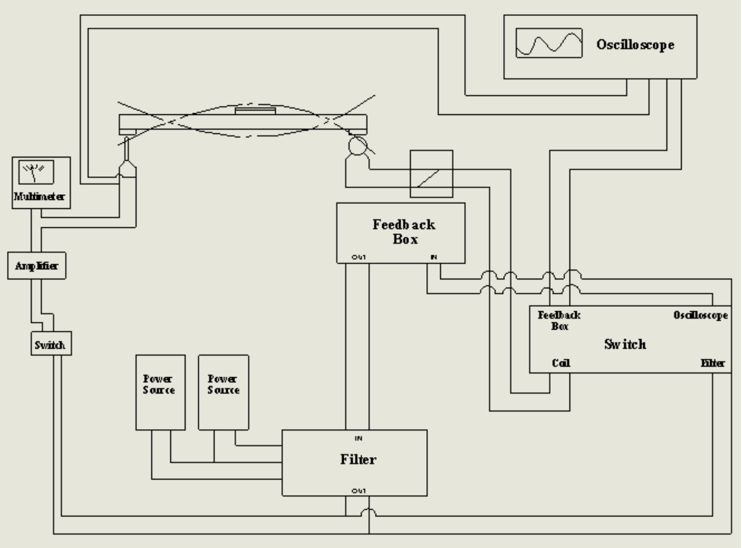


Figure 23 – Schematic of the system working with the help of the feedback box

The feedback box reads the small current induced in the pickup coil, amplifies it and feeds it to the drive coil. It is configured to create a phase change in the signal so that the signal at both coils overlaps. This brings the specimen at its resonance frequency, keeping it vibrating independently of the temperature changes.

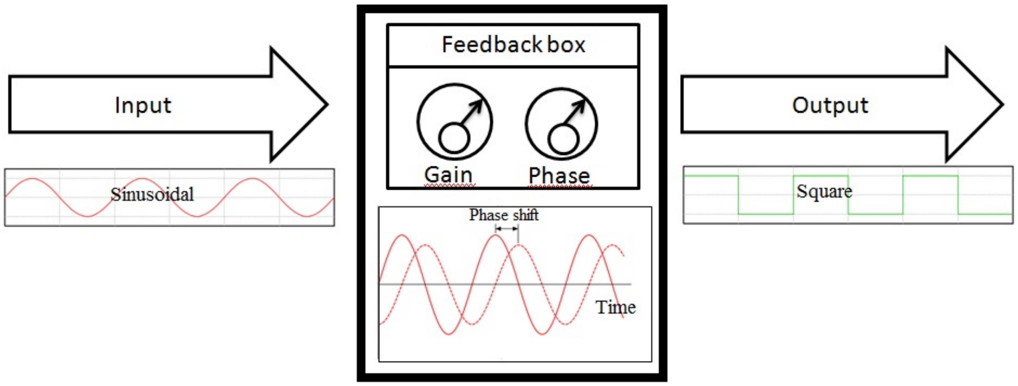


Figure 24 – Feedback box principle

As it can be seen in Figure 24, because of the electronic circuits used by the feedback box, the output signal has a square shape instead of the sinusoidal shape suitable to drive the coils and magnets. To transform this square wave into a sinusoidal wave, a wave filter is installed after the feedback box. The filter can be found in the scheme shown in Figure 23. This feedback box also has an output called "DC level". This DC level output is an electric signal whose voltage is directly proportional to the amplitude of the specimen displacement.

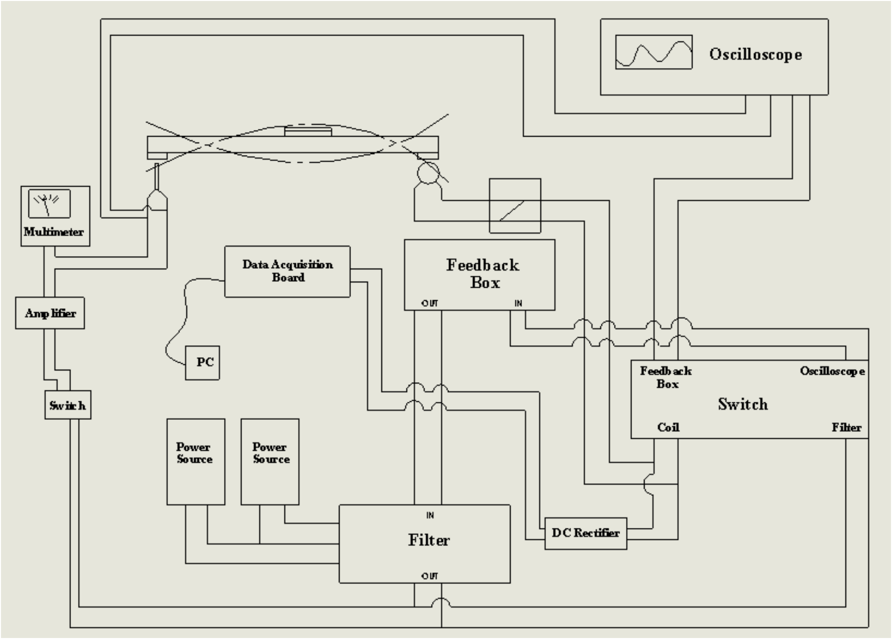


Figure 25 - System with data acquisition system

As it can be observed in Figure 25, with the help of a data acquisition system to register the temperature from this DC level, T_g can be found. The Instrunet, Somerville, United States of America, i100B device, which is connected to a PC, was used as the data acquisition system. Data was registered in two voltage channels, one for the temperature and another for the amplitude. Data was acquired at a rate of 100 Hz, to allow the use of the digital filter provided by the Instrunet i100B. This filter reduced an important amount of noise in the final data.

3.5 Specimen heating system

The oven that is heating the specimen is built with a round heating element closed in aluminium walls. The walls are insulated from inside with boards of glass fiber wool which is a material with extremely reduced thermal conductivity and that can resist up to a temperature of 1000 ºC. The heating source is a ring-shaped Mica heater with 200 mm in diameter and 400 W of power. Figure 26 presents the oven structure along with its dimensions and inside elements.

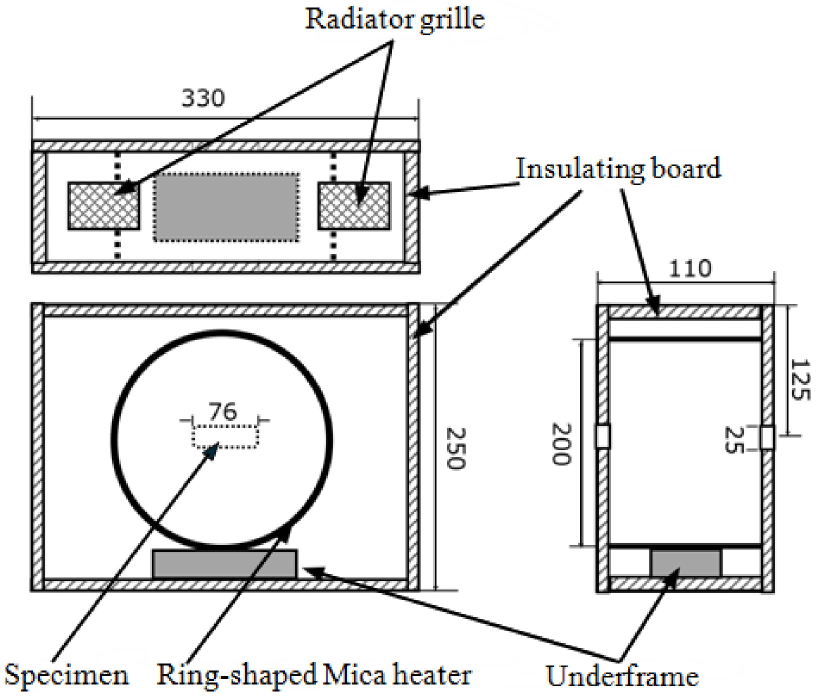


Figure 26 - Oven [11]

Two of the walls from each side of the oven have a small hole. The specimen can go through these holes so that the middle section, containing the adhesive, is placed directly inside the ring shaped heating element. Figure 27 presents the coils, coils supports and magnets of the cast specimen out of the portable oven.

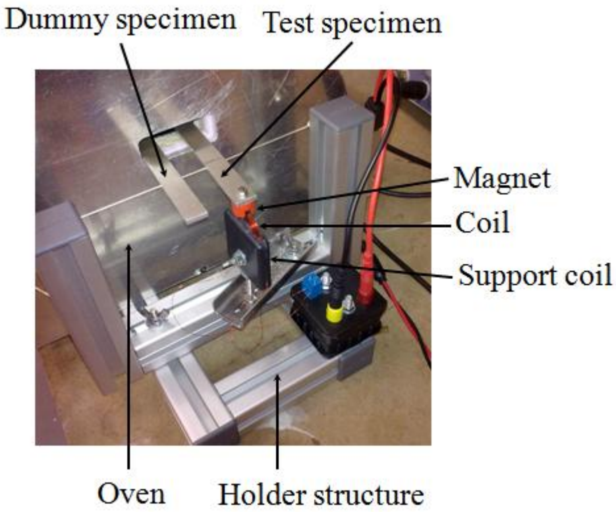


Figure 27 - Coils, coils supports and magnets of the cast specimen out of the portable oven

To avoid temperature fluctuations, an oven controller was built along with a thermostat, which allows controlled heating from room temperature up to 200 ºC. A thermocouple is located inside the oven and it provides the controller with the actual temperature and allows the continuous adjustment of the temperature. Figure 28 presents the heating system with its elements.

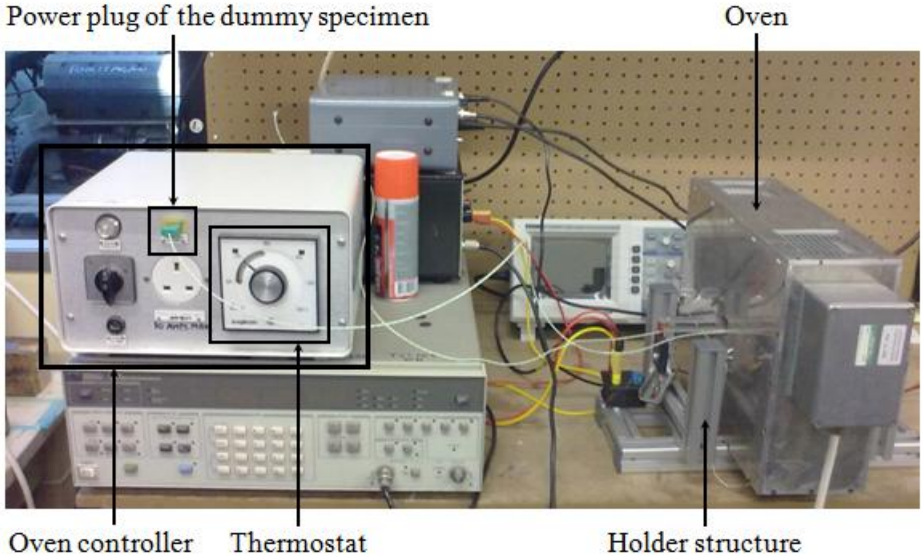


Figure 28 - Heating system

Using a portable oven has the advantage of heating in an easy and controlled way the temperature of the specimen. Because the oven has a small work area that means it performs flash heating and does not over cure the specimen. A test with the Araldite® AV119 damping during heating and cooling process by using the portable oven **[11]** is shown in Figure 29, where it can be seen that the T_g measured in the heating and cooling cycles nearly coincide.

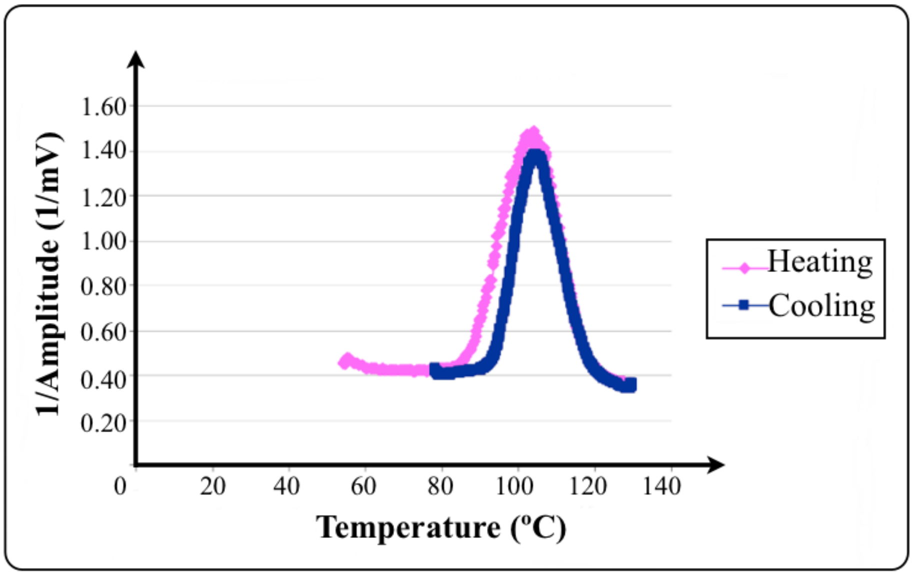


Figure 29 - Araldite® AV119 damping of the heating and cooling process by using a portable oven [11]

4. Improvements brought to the system

4.1 Purpose of the improvements

The current system is difficult to use due to the large space occupied and it has a large amount of components connected with temporary wires. The purpose of this thesis is to reproduce and improve components like the function generator, amplifier, feedback box, filter and DC rectifier as standalone boards which can be fitted in a box that will replace most of the current system. This will make it easy to use for every operator and increase its reliability.

First, each component of the system was studied and a diagram was made of its electronic layout. A temporary test board with electronic components was created after each diagram and with the use of the datasheets and experimentation, improvements were made. After each reproduced component was successfully tested, Cadsoft Eagle, Philadelphia, United States of America, V5.9.0 software was used to design and optimize the circuit boards before their manufacture.

The system will be used in the same way as the previous one. The benefit is that because of its reduced dimensions, it is easy to manoeuvre and because it is compact, no errors will appear due to loose connections or connections made wrong.

4.2 Eagle V5.9.0 software

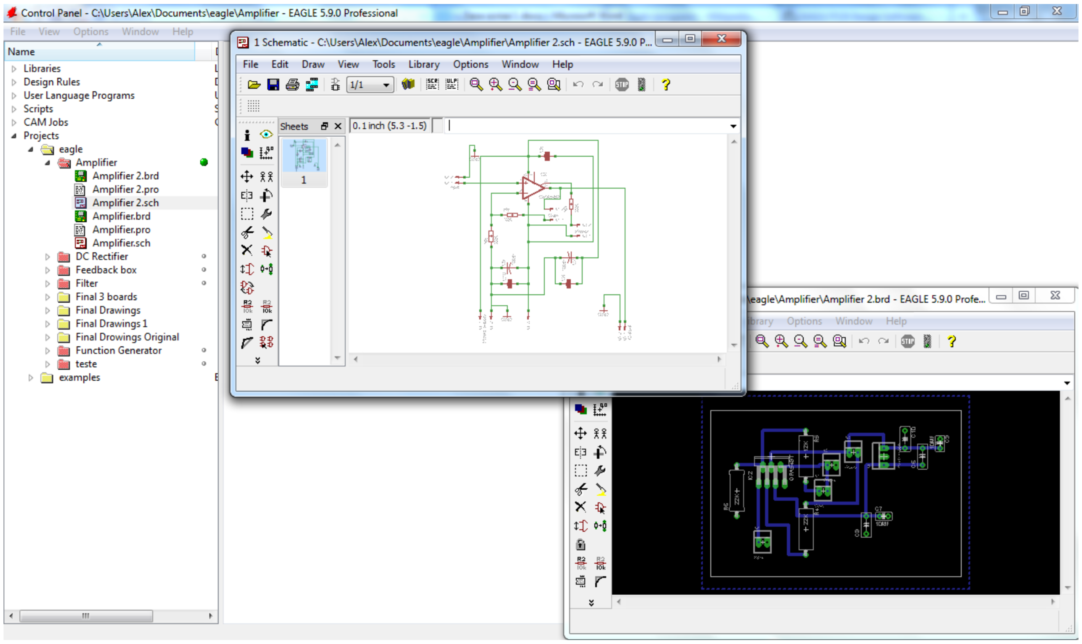


Figure 30 - Eagle V5.9.0 software

Figure 30 presents screenshots of Eagle V5.9.0 (Easily Applicable Graphical Layout Editor) which is the software used to design circuit boards. It is user friendly software which has a library full with a large variety of components from well known part producers. With the help of this software the electronic layout was designed, making all the right connections between integrated circuits, resistors, diodes, capacitors, power supply and connectors. After the electronic layout was designed, the electronic board was created and each component was arranged on the board in order to avoid any short circuits.

4.3 Boards integrated in the system

4.3.1 Function generator

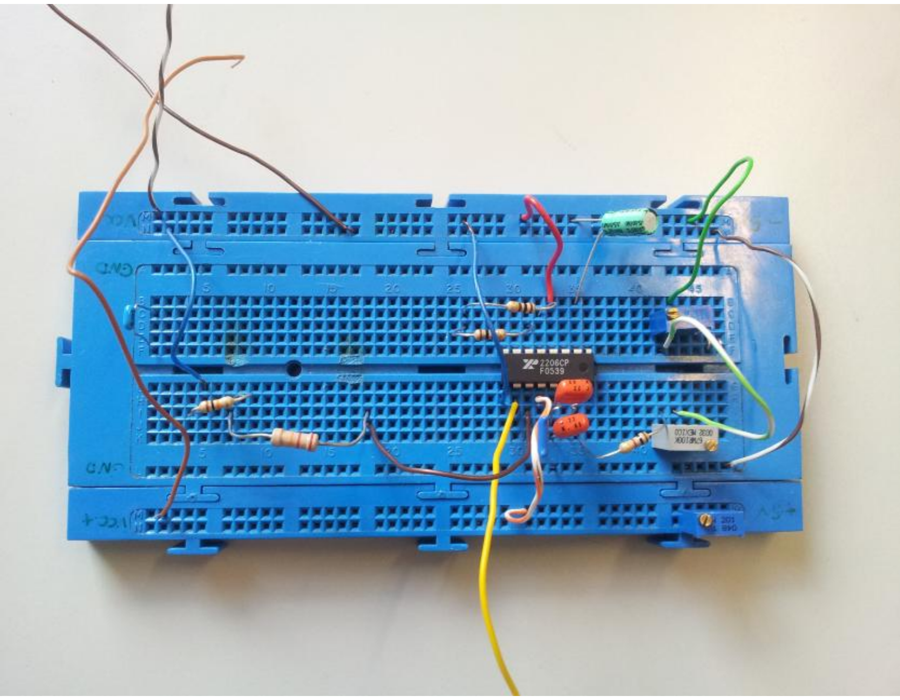


Figure 31 - Function generator test board

Figure 31 presents the test board of the function generator. This is an important part of the system because it is used at the beginning of the T_g measurement. The operator has to find the natural resonance frequency of the beam by adjusting the function generator until the beam is vibrating at a full potential. For doing this, two potentiometers are used. One is used for a coarse tuning and the other one is used for a fine tuning of the frequency.

For the function generator an Exar, Fremont, United States of America, XR2206CP integrated circuit was used and with the help of the examples from the datasheet, an adapted electronic scheme was made.

Figure 32 presents the schematic design of the function generator which was made to generate the board scheme, which allows elements to be arranged in a space efficient and practical way.

In order to reduce the noise, a group of non-polarized and electrolytic capacitors were installed in each of the positive and negative side of the power supply.

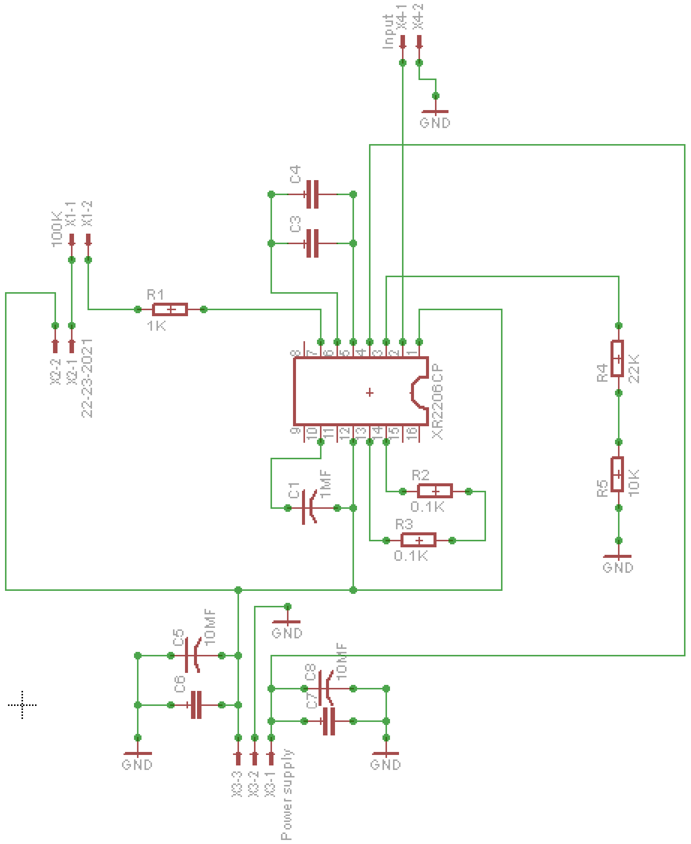


Figure 32 – Electronic schematic of the function generator

For a better noise attenuation, non-polarized and electrolytic capacitors were put closer to the power supply. The function generator was connected to a potentiometer to adjust the frequency and an output which connects it with the amplifier.

Figure 33 shows the scheme of the function generator board.

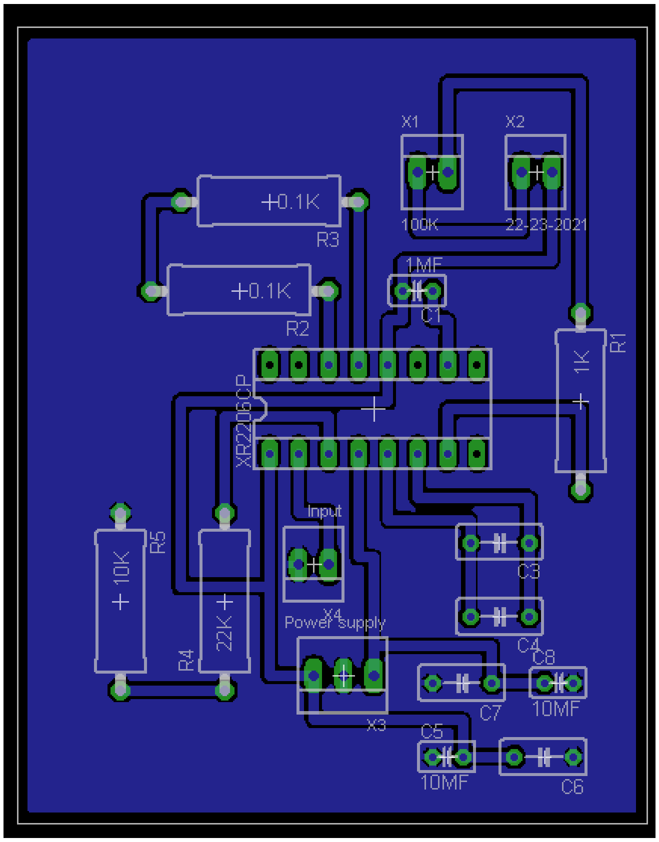


Figure 33 - Function generator board

Figure 34 presents the final printed and soldered version of the function generator board.

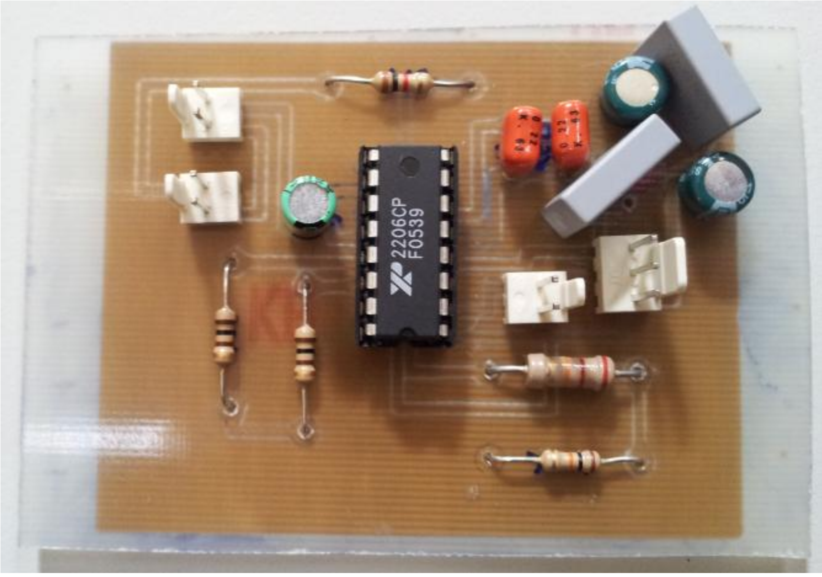


Figure 34 - Function generator printed board

4.3.2 Amplifier

The amplifier is an electronic device that increases the power of the function generator. It does this by taking energy from a power supply and controlling the output to match the input signal shape but with a larger amplitude. The amplifier modulates the power output of the waveform supplied to the drive coil.

Figure 35 presents the testing board of the amplifier.

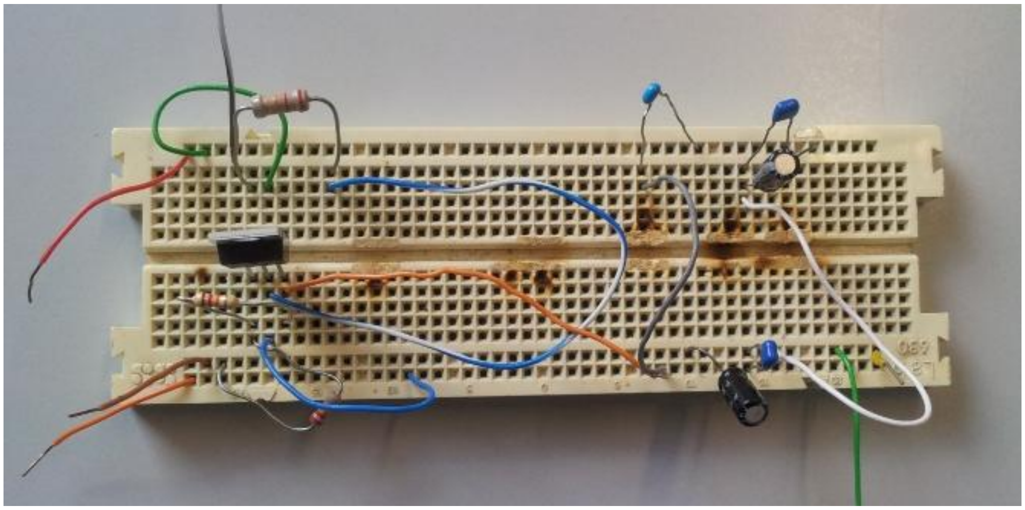


Figure 35 – Amplifier test board

The function generator is generating a signal to the aluminium beam, but the vibration of the beam is not visible. So the amplifier is important to the T_g measurement because when the natural frequency of the beam is reached, the amplified wave will make the beam vibrate more. When this happens, the operator will observe the phenomenon and can precede the test with.

The amplifier has to have its current limited. When it is used, it cannot send to the system more than 1 amp of constant current because otherwise parts of the coil will overheat and become damaged.

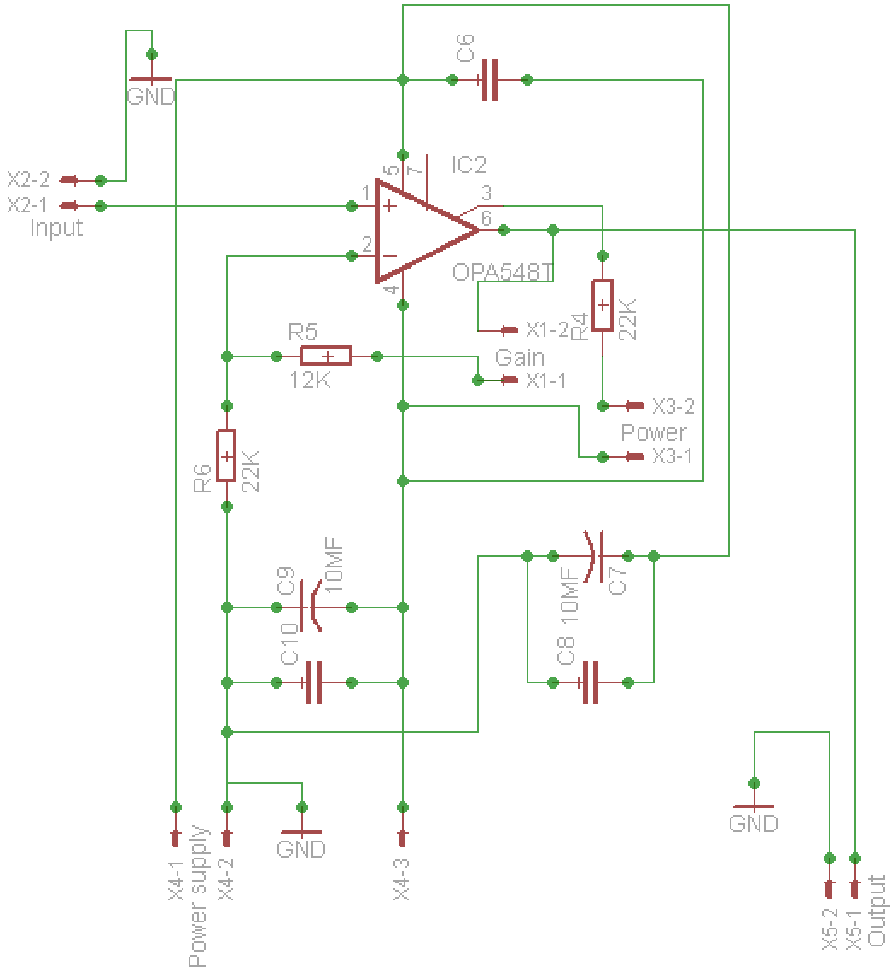


Figure 36 – Electronic schematic of the amplifier

Figure 36 shows the electronic schematic of the amplifier.

With the help of the datasheet of the Texas Instruments, Dallas, United States of America, OPA548T integrated circuit, improvements were made to the amplifier. By installing 22K and 12K resistors near the gain and output potentiometers, the board will be limited and it will not transmit more than 1.5 amps. Also, for noise reduction, capacitors were installed in each of the positive and negative side of the power supply.

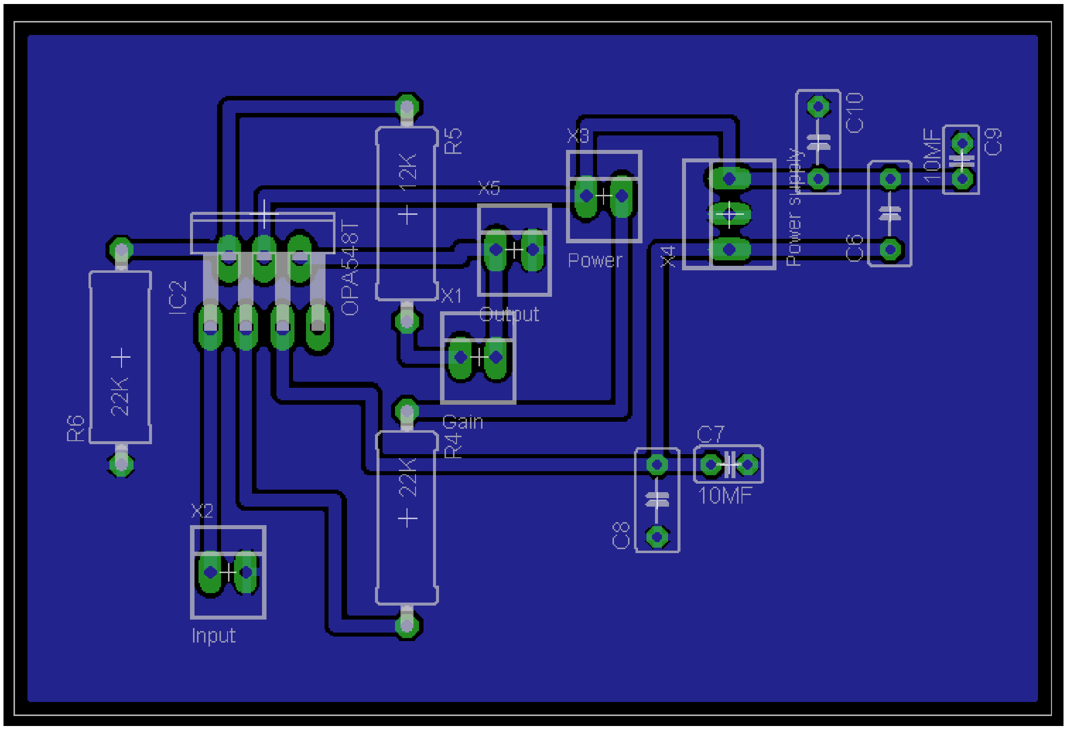


Figure 37 - Amplifier board

Figure 37 presents the optimized board of the amplifier.

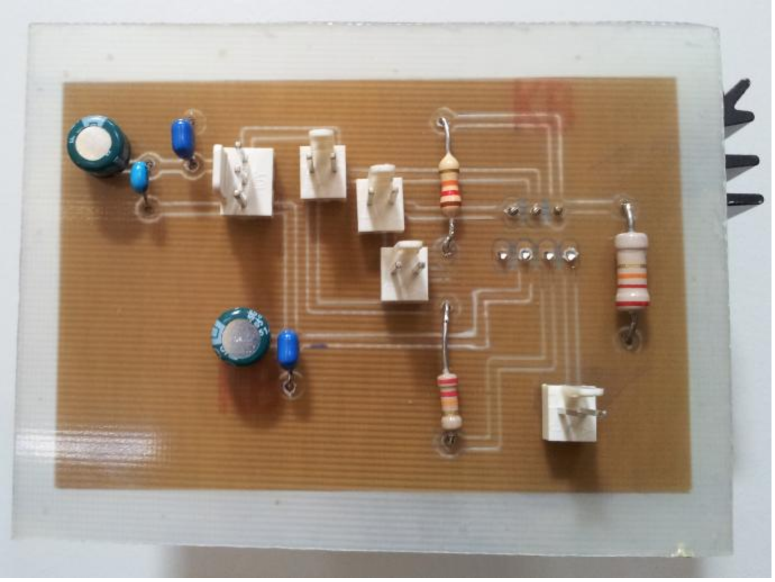


Figure 38 - Amplifier printed board

Figure 38 presents the final printed and soldered version of the amplifier board.

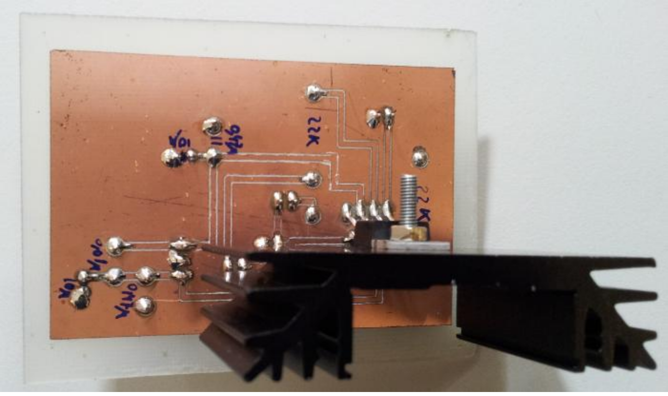


Figure 39 - Back of the printed amplifier board

Figure 39 shows the back of the printed amplifier board, where the integrated circuit is visible with a heat sink that cools it down. Considering the fact that a high current passes through the amplifier integrated circuit, safety measures must be taken. To avoid overheating the system, a fan was installed in the box that host the boards.

4.3.3 Feedback box

Figure 40 presents the feedback box test board. The electronic design of it was based after the original feedback box and it was optimized for smaller size and better range of adjustments. Texas Instruments Dallas, United States of America, LM324N integrated circuits were used and a larger capacitor was used to increase the amount of phase shift that can be adjusted. The feedback box has a switch that can phase shift with 180 degrees and two potentiometers: one for approximately 85 degrees phase shift and one for approximately 5 degrees phase shift.

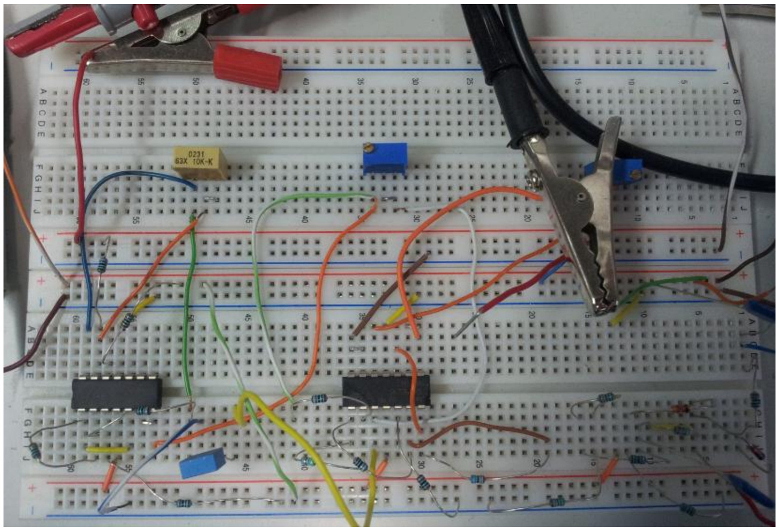


Figure 40 - Feedback box test board

The feedback box has the purpose to keep the system in an autonomous closed loop and it does that by amplifying the sinusoidal wave received from the pick-up coil which is then transformed almost in a square wave. The phase shift circuit alters the phase of the signal to correspond with that needed to input the power amplifier to maintain resonance.

Figure 41 presents the electronic schematic of the feedback box.

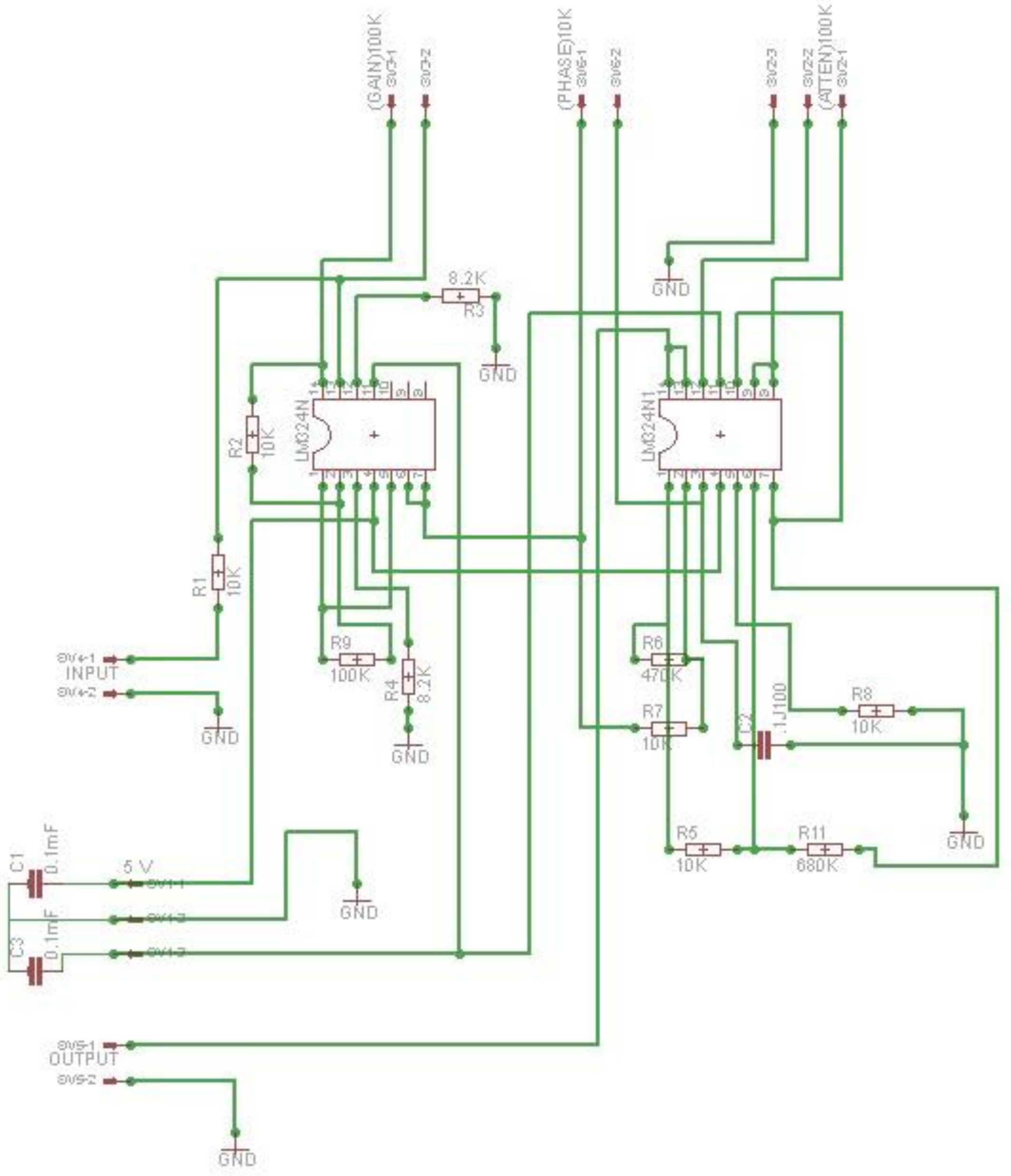


Figure 41 – Electronic schematic of the feedback box

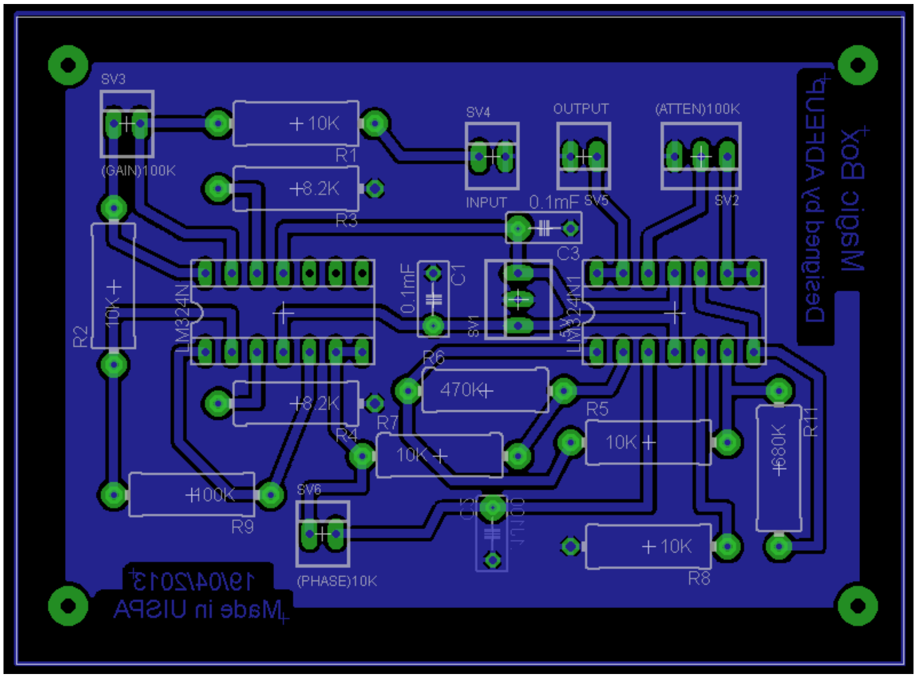


Figure 42 - Feedback box board

Figure 42 presents the optimized version of the feedback box board. On this board the capacitors which are placed in each side of the positive and the negative of the power supply can be seen. They are placed there to reduce the noise and to offer a better reading.

Figure 43 presents the final printed and welded version of the feedback box board.

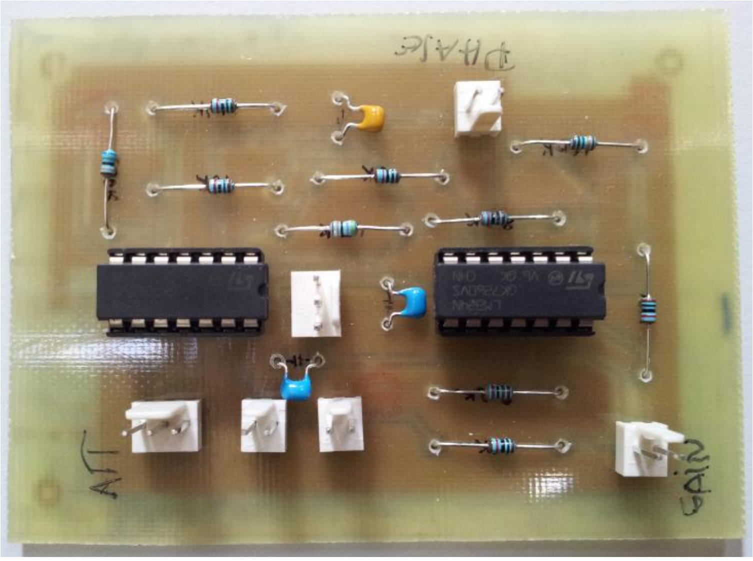


Figure 43 - Feedback box printed board

4.3.4 Filter

The filter is a device that removes from a signal some unwanted features. Filtering is a class of signal processing, the defining feature of filters being the partial suppression of some aspects of the signal.

In the current case, the filter with its central frequency approximately at the resonant frequency of the specimen is used to block frequencies that are outside a select bandwidth bracketing the first resonant frequency of the specimen. This transforms the square wave into almost a sinusoidal wave which is then amplified and sent via a power amplifier to the driving coil.

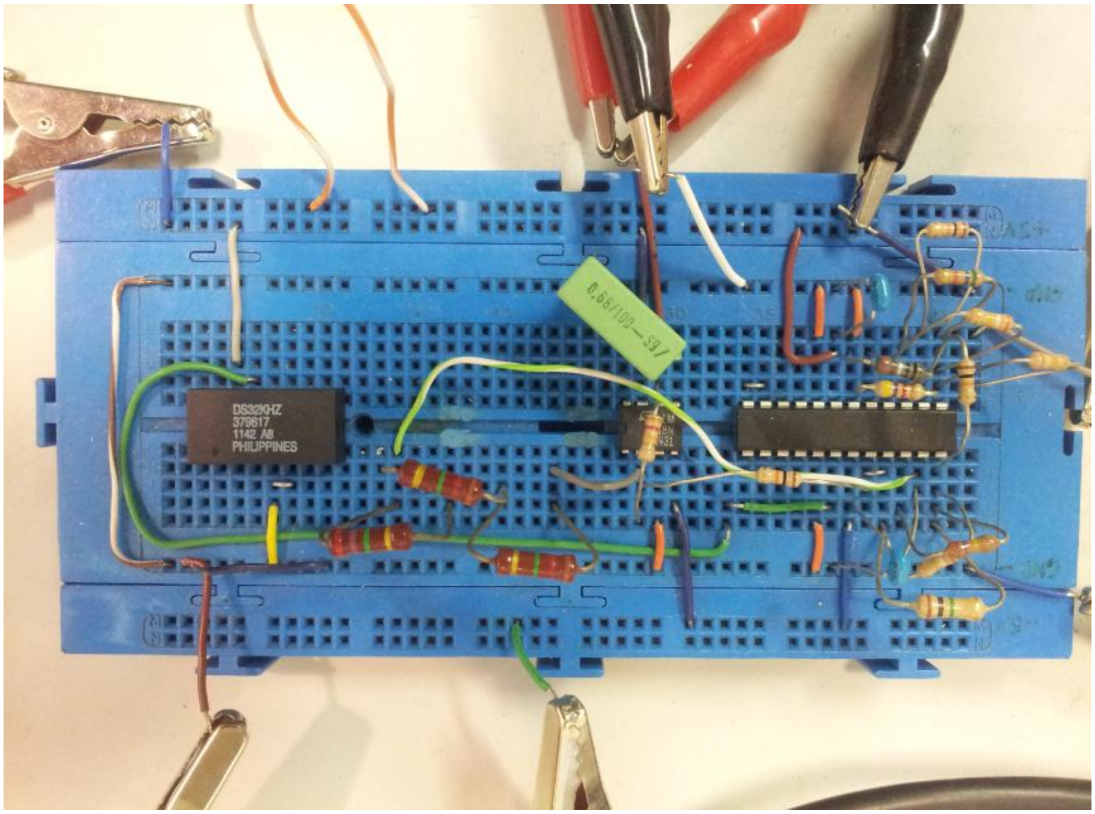


Figure 44 - Filter test board

Figure 44 presents the test board of the filter and Figure 45 presents the electronic schematic of the filter.

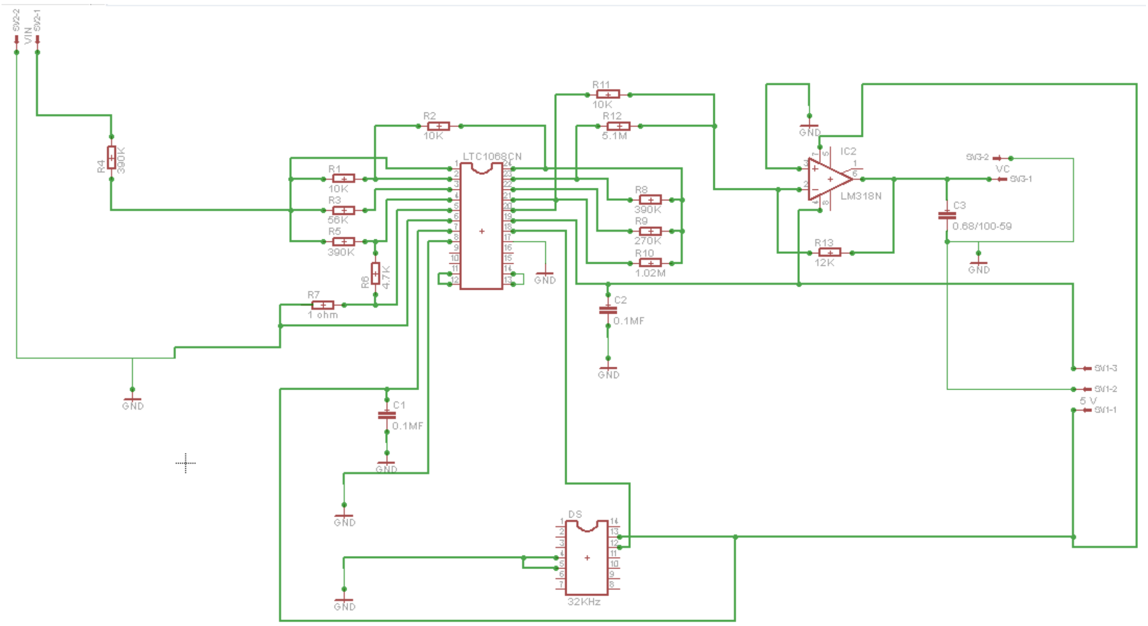


Figure 45 – Electronic schematic of the filter

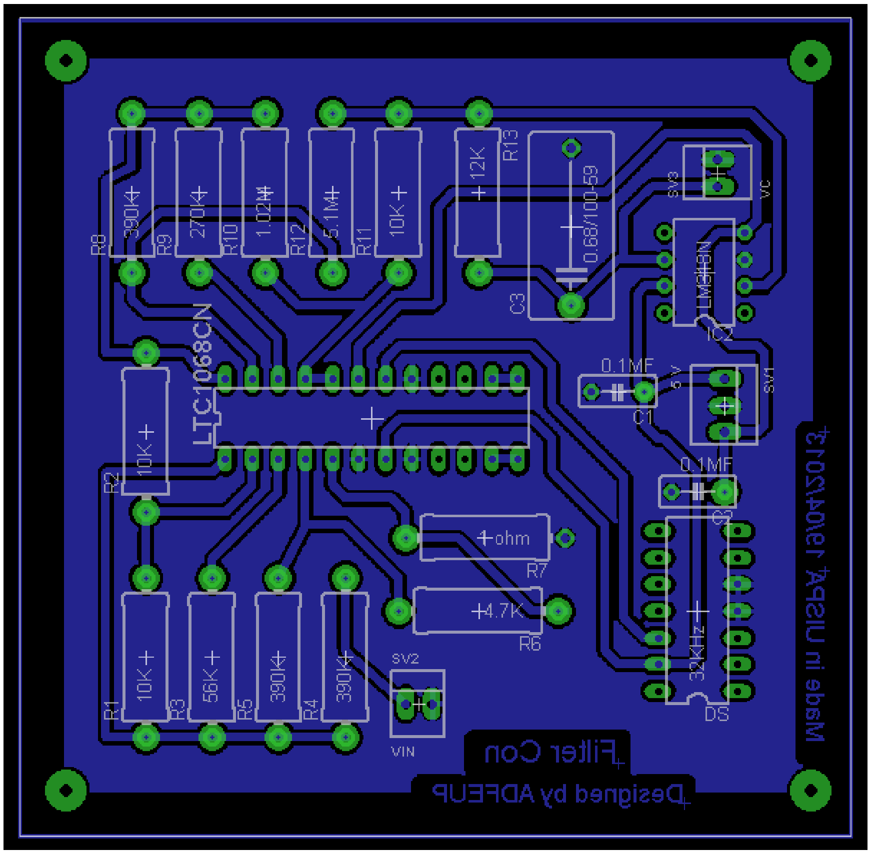


Figure 46 - Filter board

Figure 46 presents the optimized version of the filter board.

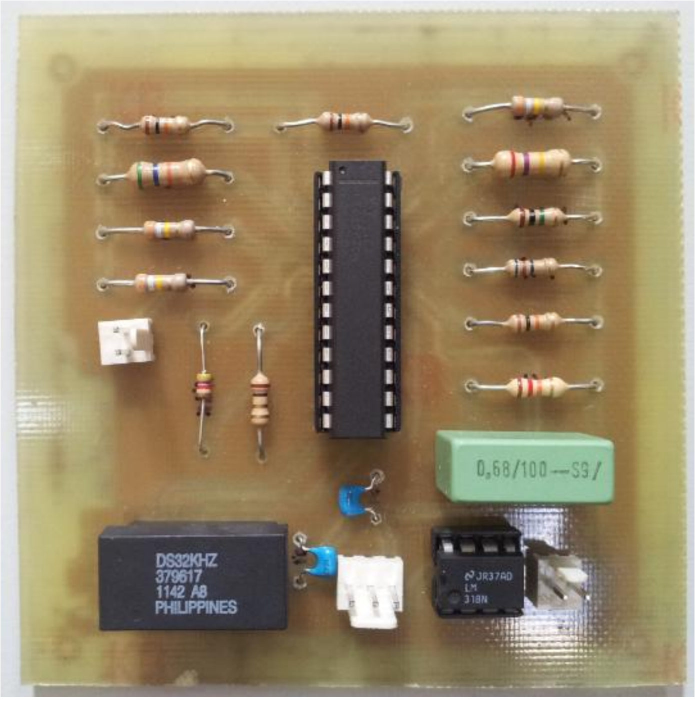


Figure 47 - Filter printed board

Figure 47 presents the final printed and soldered version of the filter board.

4.3.5 DC rectifier

The feedback box has an output which goes to the DC rectifier. This DC rectifier is an electric signal whose voltage is directly proportional to the amplitude of the specimen displacement.

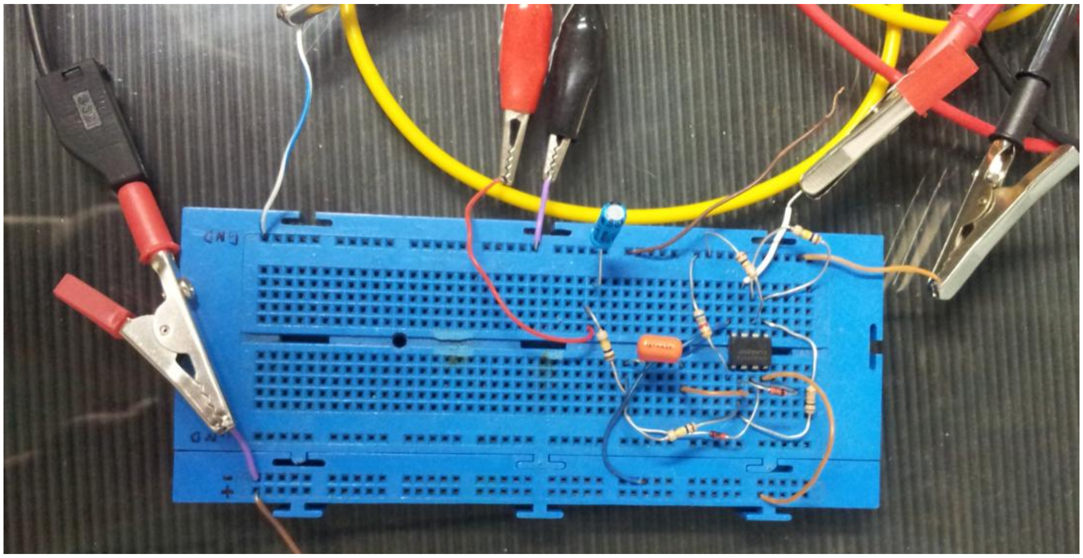


Figure 48 - DC rectifier test board

Figure 48 presents the DC rectifier test board and Figure 49 presents the electronic schematic of the DC rectifier.

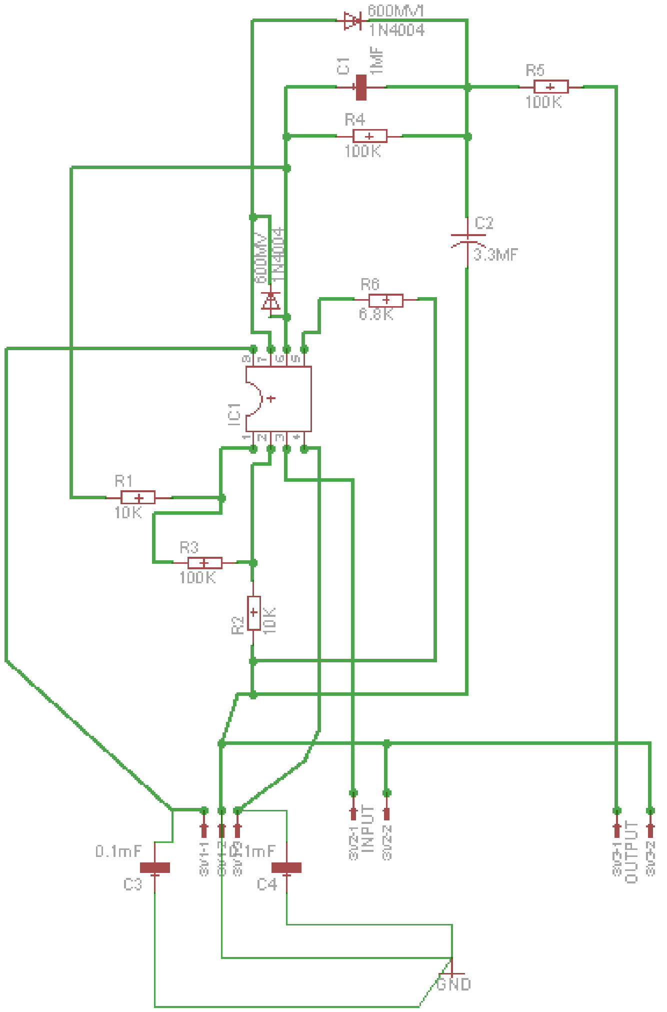


Figure 49 – Electronic schematic of the DC rectifier

Data acquisition system was used to read the temperature from the oven and the displacement from the DC rectifier. Instrunet Somerville, United States of America, i100B device was used as the data acquisition system. Data was registered in two voltage channels, one for the temperature and another for the amplitude with the help of the computer. Data was acquired at a rate of 100 Hz, to allow the use of the digital filter provided by the Instrunet i100B. This filter reduced an important amount of noise in the final data.

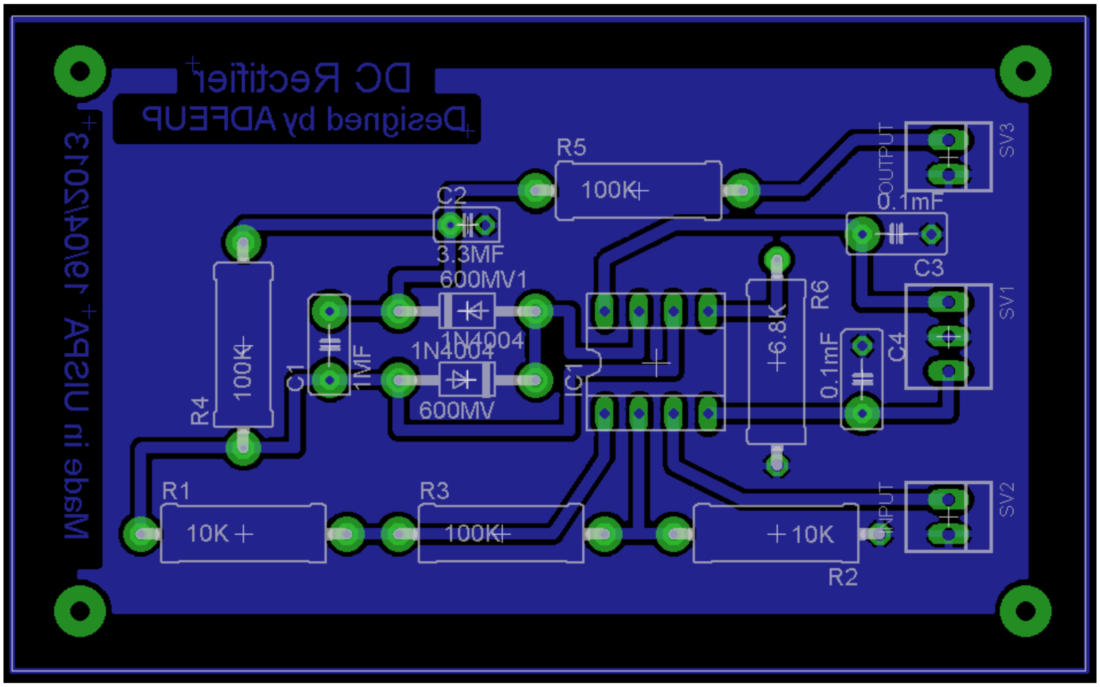


Figure 50 - DC rectifier board

Figure 50 shows the optimized version of the DC rectifier board where, as in the other boards, capacitors are placed in the positive and the negative side of the power supply. To have a better noise reduction, the capacitors were put as near as possible the power supply connector.

Figure 51 - DC rectifier printed board

Figure 51 presents the final printed and soldered version of the DC rectifier board.

4.4 Device enclosure

The purpose of the thesis is to make the T_g measurement system more compact and easier to use by the operator. Therefore all the electronic boards presented above where installed in one enclosure that combine five separate components of the system: the function generator, amplifier, feedback box, filter and the DC rectifier. Also the control switches where installed in the enclosure.

Figure 52 presents the frame of the enclosure with its inside dimensions: 520 mm length, 200 mm width and 150 mm height. The sizes are suitable for all plates along with the power supplies and switches to fit, and there also remains a small space for future improvements. For a better rigidity, the enclosure has a aluminium bottom plate.

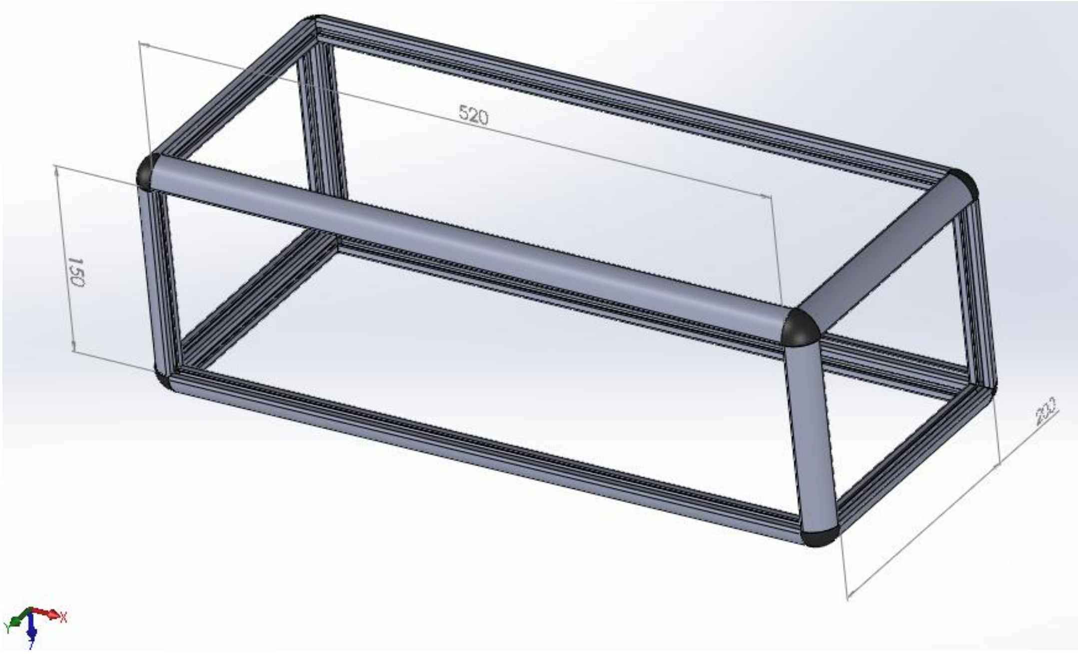


Figure 52 – 3D representation of frame of the T_g apparatus boards enclosure

The enclosure has two power supplies inside. Figure 53 presents the ±12 V power supply to which are connected the function generator, amplifier and the fan (see Figure 54) which has the purpose to cool down the inside of the enclosure. The power supply is connected to a power board with a 3 pin connector.

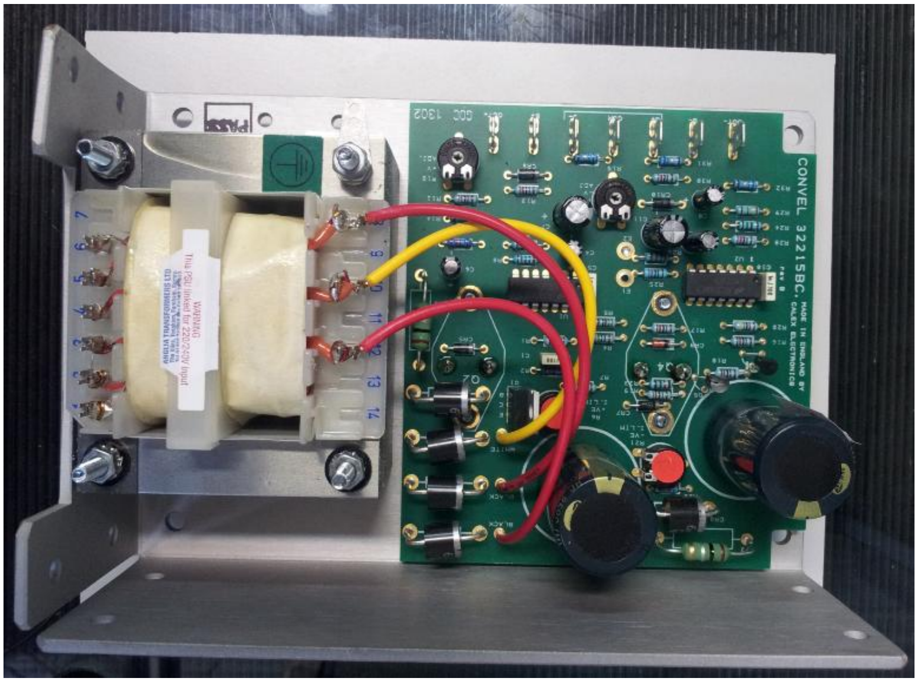


Figure 53 - ±12 V power supply

Figure 54 - 12 V fan

Figure 55 presents a power board on which the ± 5 V power supply is mounted. On the upper part of the board, are connectors for ± 5 V for the feedback box, filter and DC rectifier are present. On the lower part of the board, the ± 12 V power supply is connected and 3 pin connectors are placed for the function generator, amplifier and the fan. For each power supply there is one extra connector to use in case if future upgrade work will be made for the system.

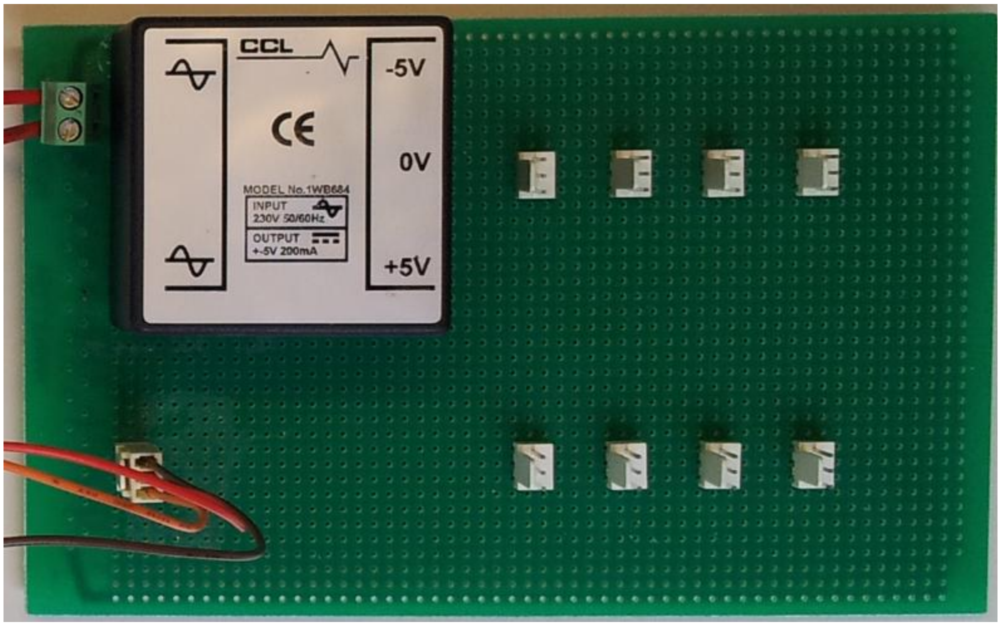


Figure 55 - Power board

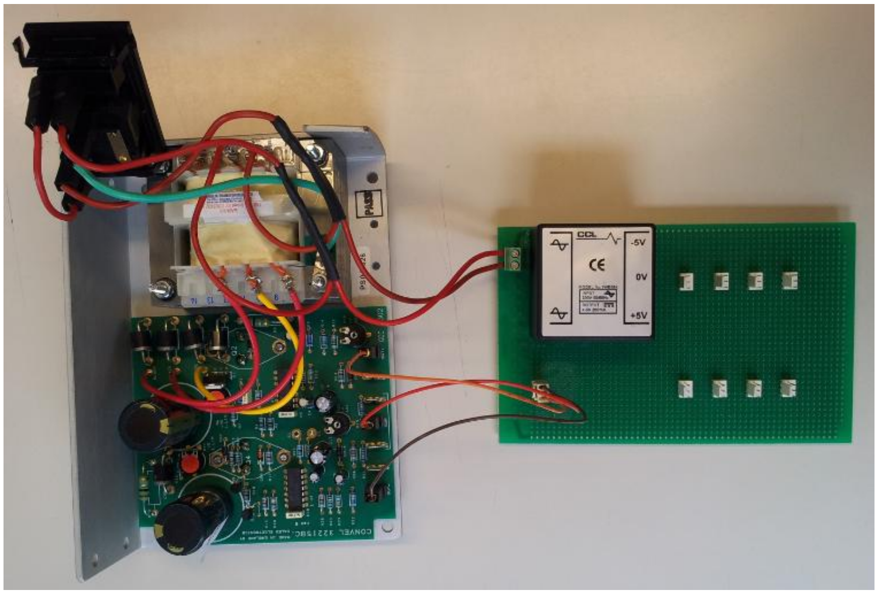


Figure 56 - Connection between power supplies, power board and the fused and switched IEC inlet

Figure 56 presents the connection between power supplies, power board and the fused and switched IEC inlet. The system uses normal 220V current and it uses an IEC 3 pin jack inlet to power it up. The inlet also has a fuse to protect the system in case of short circuit.

During construction of the enclosure, some wires were welded to each other in order to split them, to adjust their length or to attach to them different connectors. Figure 57 presents the protection installed over the welding in order to avoid appearance of outside interference.

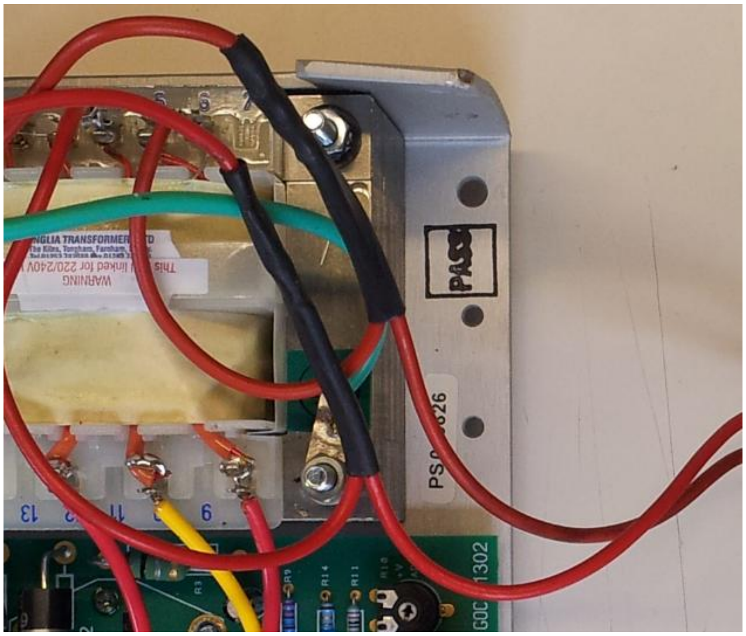


Figure 57 - Wire protection

Figure 58 presents the inside of the box. The boards were positioned in a specific order to be closer to the power board and also to the front panel where they have to be connected to the switches, potentiometers and the BNC outputs and inputs.

The first board from the left is the function generator. It was placed the first because the measurement starts with it when the natural frequency of the beam is found by the operator. The function generator board is next followed by the filter and the feedback box board. After them the DC rectifier board is installed and it has a BNC output which connects to data acquisition board. Tast board is the amplifier.

The fan was placed at the rear of the enclose facing the amplifier because the amplifier is the board which will heat up the most.

The fused and switched IEC inlet was placed in the back of the enclosure. It was installed between the power board and the ± 12 V power supply because it has to be connected to both of them in order to feed the system with current.

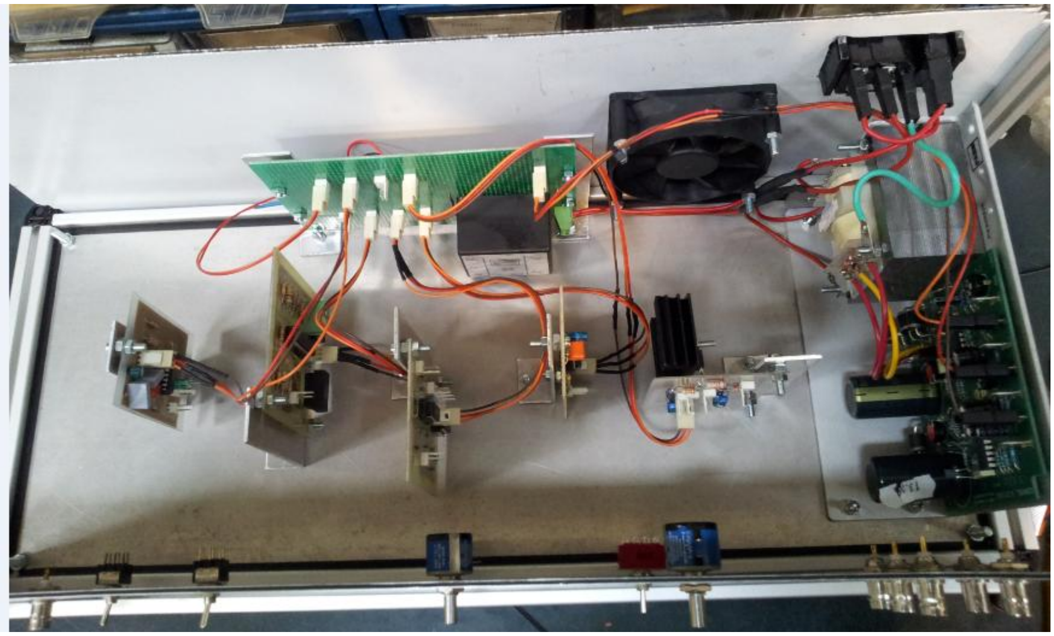


Figure 58 - Enclosure inside

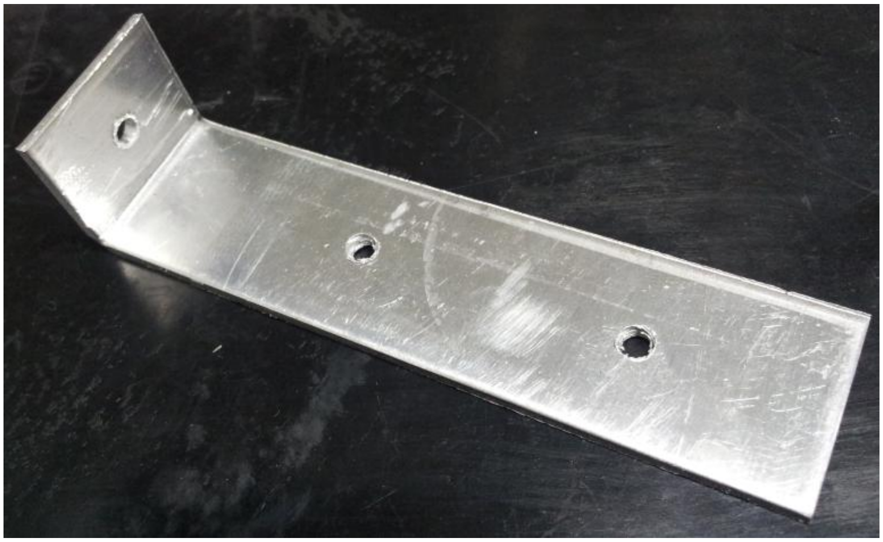


Figure 59 - "L" shape aluminium support

The boards were connected to the bottom plate using "L" shape aluminium supports as presented in Figure 59. This solution was chosen because the boards are lightweight and the support presents enough strength to offer stability for the system. The boards were attached to the supports using 4 mm screws and spacers.

Figure 60 - Front panel

Figure 60 presents the front panel of the enclosure. Potentiometers and switches were installed in sequence of using. In the left two potentiometers were installed for the function generator. One is for rough tuning and the other is for fine tuning. Under them there is a BNC input and a switch. They were installed in order to switch to an external function generator in case more precision regarding the frequency wave is needed.

The next switch is for passing from an open loop, offered by the function generator, to a close loop, offered by the feedback box board. It is followed by two potentiometers that are connected to the amplifier. The first potentiometer is for power and the other one is for gain.

The last two potentiometers are connected to the feedback box board. They adjust the phase change. The first potentiometer adjusts the phase shift by approximately 5 degrees and the other one adjust the phase shift by approximately 85 degrees. In case this adjustment is not sufficient, there is a simple switch installed near the pickup coil which crosses the lines and can phase shift 180 degrees.

On the right side of the front panel, BNC connectors are installed. Two are for the coils, two are for the oscilloscope and one is for the DC rectifier output. The enclosure has four adjustable legs which are connected to the aluminium bottom plate using 5 mm screws.

4.5 Measurements comparison between the previous system and the present system

After assembling the enclosure, comparison measurements were made to observe if the T_g values are the same with both measurement systems.

The previous system and the present system have the same measuring method, so in consequence T_g values should be the same. The only difference between the systems is that the present one is more compact and easier to use by the operator. Because of that, measurements are more exact and they are made faster.

The glass transition temperature was measured for two pairs of specimens with Araldite AV 138M Huntsman, Basel, Switzerland adhesive using the dynamic mechanical test device described in section above. T_g was measured during the heating and cooling stages. The first test was performed with the previous T_g measurement system and the second test was performed with the new improved T_g measurement system.

In the following part, T_g measurements for Araldite AV 138M adhesive are compared.

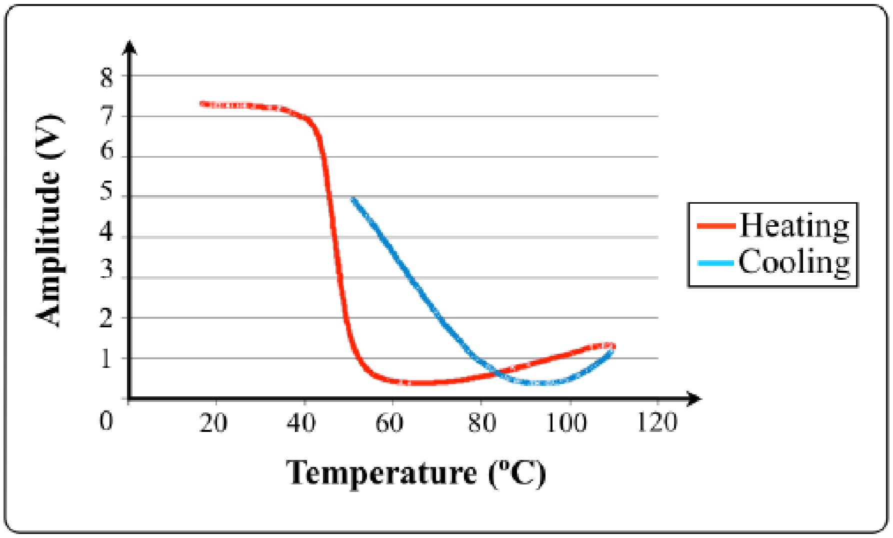


Figure 61 - Dynamic mechanical test results, Amplitude versus Temperature for the epoxy adhesive Araldite AV 138M measured with the previous system [18]

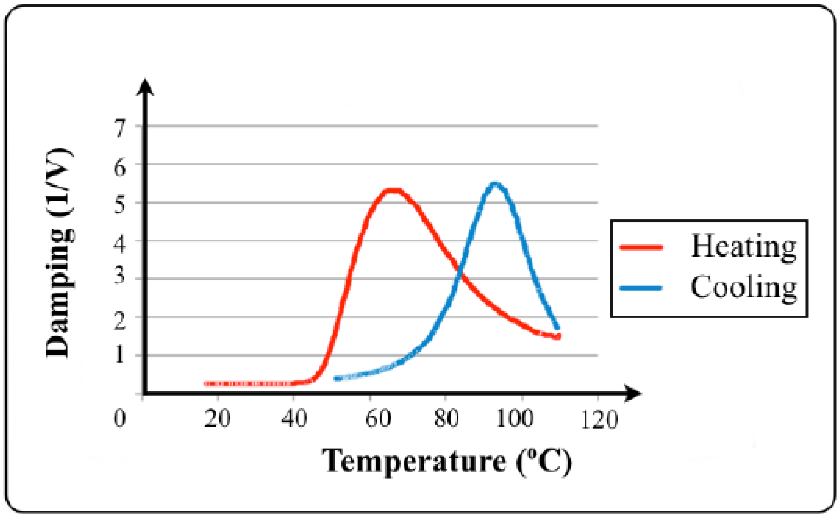


Figure 62 - Dynamic mechanical test results, Damping versus Temperature for the epoxy adhesive Araldite AV 138M measured with previous system [18]

Figure 61 and Figure 63 give curves of displacement amplitude (value inversely proportional to the damping of the adhesive) versus temperature for the heating and cooling cycles for the adhesive.

Figure 62 Figure 64 give curves of 1/(displacement amplitude) (value proportional to the damping of the adhesive) versus temperature for the heating and cooling cycles for the adhesive.

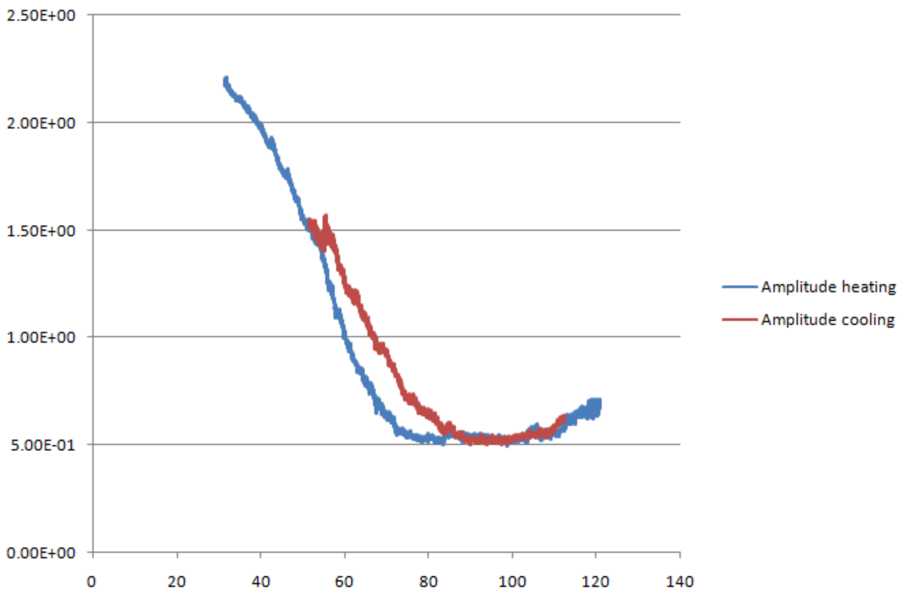


Figure 63 - Dynamic mechanical test results, Amplitude versus Temperature for the epoxy adhesive Araldite AV 138M measured with new system

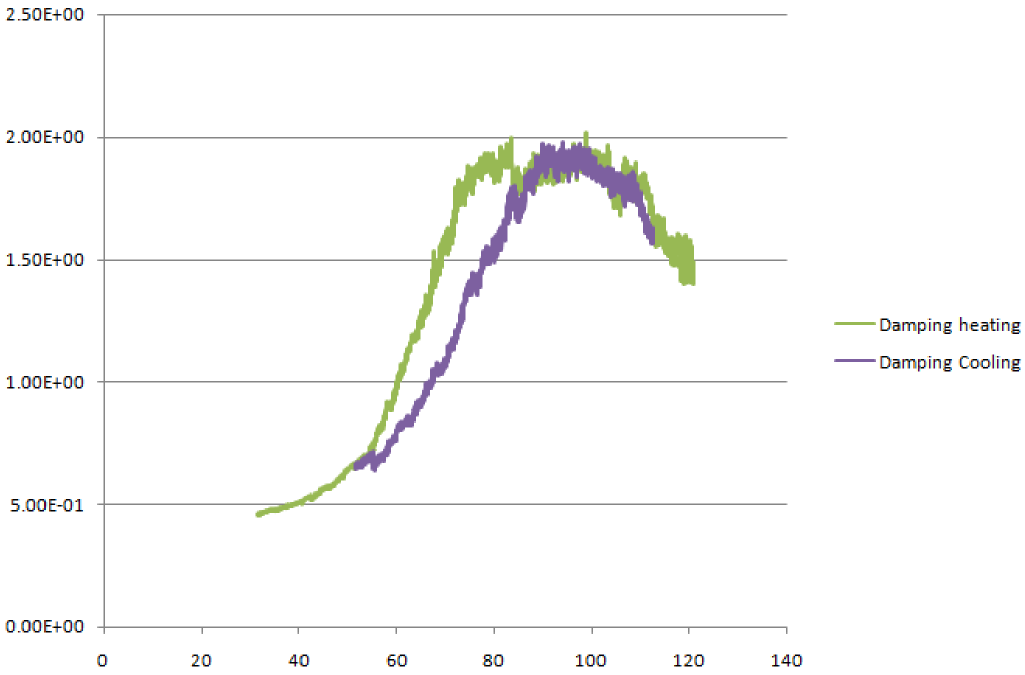


Figure 64 - Dynamic mechanical test results, Damping versus Temperature for the epoxy adhesive Araldite AV 138M measured with the new system

Table 2 presents the glass transition temperature from both measurements. As it can be seen, the values are almost similar and the average T_g is close to the expected T_g result.

Table 2 - Glass transition temperatures measured

Epoxy Adhesive	Expected result	Experimental result		
	T_g (ºC)	T_g (ºC) Heating	T_g (ºC) Cooling	T_g (ºC) Average
Araldite AV 138M (previous system)	85	74	91	83
Araldite AV 138M (new system)	85	83	90	87

5. Conclusions

This dissertation consisted in three main stages. The first stage consisted in the theoretical study of the glass transition phenomenon, its importance for engineering adhesive joints and the methods currently used for the determination of this property. Advantages and disadvantages of each method were compared, so that the possible points of improvement were identified. It was noted that current methods are expensive and time consuming, which make them unavailable for many laboratories. The development of a simpler and cheaper solution was therefore the logical aim of this work.

The next stage, was the development of simple electronic boards that replaced the more complex and bigger devices. Electronic boards were developed for power supply, function generator, amplifier, filter, feedback box, DC rectifier. The boards have only the functions needed for the T_g measurement. This work resulted in manufacturing an enclosure that contain all the key elements that replaced most of the devices used in the previous system. The new system is simpler to use, reliable, compact and it has the space and the design which allows future improvements to be made. It increased the reliability of the mechanical components of the apparatus and established a reliable and simple procedure to obtain data and validate the results.

In the last stage a small experimental procedure was made to validate the new built system made in this work. This procedure used a few adhesives with significantly different chemical compositions and mechanical properties, as a way to guarantee the accuracy of the method in different conditions. The results were very satisfactory and demonstrated the viability of the device and procedures presented in this work.

6. Future works

The system developed in this work has a very good functioning status, but there is always space for improvements. One of the improvements could be adding an electronic dial for frequency reading which could simplify the work of the operator who has to read the frequency from the oscilloscope.

Optimization of the system could be brought by adding a graphical user interface which will make the system easier to use. Making the measurements directly using a computer software will make the system more user friendly and operators will learn the experimental protocol faster.

Also the data acquisition board could easily be placed in the enclosure. By doing this the space occupied by the T_g measurement system will be reduced.

Further tests with different adhesives (or other polymeric materials) could be made to calibrate the device with more accuracy and improve the way of obtain results of data acquisition.

A portable low temperature chamber could be developed, to obtain negative Tg (instead of the portable oven, which allows to measure only positive Tg). The chamber must be compact (especially in width) to enable to place it where the oven is currently located without replacing the components of the machine.

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