

University of Southern Queensland
Faculty of Engineering and Surveying

**Fracture Toughness of Glass Powder
Reinforced Vinyl Ester Resin Post Cured
in a Conventional Oven using Flexural
Test**

A dissertation by

Marau Vuli Mautoga

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Courses ENG4111 and ENG4112 Research Project

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Abstract

The construction sector is one of the world's largest consumers of polymer composites. Unreinforced polymer composite materials have been used by the construction industry for many years in non-load bearing applications such as trimmings, kitchenware, vanities and cladding. In the last decade there has been a concerted effort to migrate reinforced polymer composites (RPCs) into the construction industry for use in primary load bearing applications. Potential advantages commonly expounded by proponents of RPC materials include high specific strength, high specific stiffness, tailor-able durability, good fatigue performance, versatile fabrication and lower maintenance costs. As a result reinforced polymer composites are being investigated in applications such as rehabilitation and retrofit, alternative reinforcement for concrete and, in rare cases, entire fibre composite structures.

However, to date the number of primary structural applications of RPCs in construction remains relatively low and there appears to be a number of issues contributing to their slow uptake by the construction industry. Issues such as cost, absence of design codes, lack of industry standardisation, poor understanding of construction issues by composites industry, lack of designers experienced with polymer composite materials and civil/building construction are commonly claimed to place these materials at a disadvantage when considered against traditional construction materials. However, this project proposes that as issues of sustainability become increasingly important to material choice, some fibre composite materials could be at an advantage over traditional materials.

The aim of this project was to determine which percentage of glass powder (by weight) would give the highest fracture toughness.

Specimens of vinyl ester resin reinforced with glass powder were made, at different percentages (by weight). The percentage composition of glass powder (by weight) was 0 % - 35 % in 5 % intervals. Six specimens of each percentage composition are to be made; therefore, forty (40) specimens should be made. The samples should be cured in ambient conditions. After curing, they should be post cured in a conventional oven over a period of ten hours, at different temperatures. This ensured the resin had fully cured throughout the specimen. Flexural tests were performed on the specimens. Using the data obtained, the fracture toughness was determined.

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CERTIFICATION

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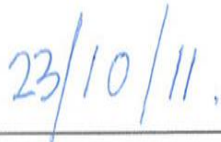
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Marau Vuli Mautoga

Student Number: 0050003387



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1. Introduction

1.1 Introduction

This chapter will describe the purpose, background and processes involved in the project. The aim of this project is to find the percentage (by weight) of glass powder to vinyl ester resin, which will give the best fracture toughness.

1.2 Project Topic

Fracture toughness of glass powder reinforced vinyl ester resins post-cured in a conventional oven using flexural tests.

1.3 The Composite Material

Composites have a long history in industry, and with advances in production techniques, it is found to be an important aspect in the materials engineering field. It is used in a wide range of applications such as civil engineering, transport, aerospace and marine. Civil engineering applications are influenced mainly by cost, while the transport, aerospace and marine applications are mainly influenced by performance (Ku et al., 2008). In all the applications, cost will always play an important role. The cost in producing composites can be reduced with the introduction of fillers. Fillers not only reduce costs but also influence the structural properties of the composites.

In this project, vinyl ester resin will be filled with glass powder at different percentages by weight to determine how much glass powder gives the best material properties. The samples will be cured in ambient conditions and then post cured in a conventional oven. The fracture toughness of the samples will be determined after the flexural testing and analysis of the test results.

1.4 Project Background

Composite materials are widely used in industry. Composites are used because they utilise a combination of materials which allows cost to be lowered, while at the same time, giving a new material with improved properties.

Vinyl ester resins have established and increasing uses in industry. They are regarded for their strong chemical, corrosion and heat resistant properties, as well as their mechanical properties namely fatigue performance and high elongation. The addition of fillers changes the structural properties and reduces costs. It can also minimise cracking and decomposition of thick parts of components. The most commonly used filler for vinyl ester resin is Type E fiberglass, however, other materials such as graphite, aramid, olefin, and ceramic fillers may also be used (Blankenship et al., 1989).

1.5 Project Objectives and Aims

Adding glass powder to the vinyl ester resin will improve the structural properties of the composite. The aim of this project is to find what percentage of glass powder will give the optimum fracture toughness. The percentage composition of glass powder (by weight) will be the same as previous studies; these are 0 % - 35 % in 5 % intervals. The samples will also include an accelerator to assist the curing. For an example, take the production of 100 grams of a 10% sample. 10% of the sample i.e. 10 grams, will be powder, and 90% i.e. 90 grams, will be resin with accelerator. The 90 grams will consist of 2% accelerator, i.e. 1.8 grams. The success of a test depends on the repeatability of the results; therefore, six specimens will be made for each percentage sample.

In this project, the resin used will be the vinyl ester resin, Hetron 922 produced by

Huntsman Composites, a division of Huntsman Chemical Company Australia Pty Ltd (Huntsman Composites, 2001). The accelerator used is methyl ethyl ketone peroxide (MEKP); this is an established and recommended accelerator (Blankenship et al., 1989).

Production of the samples will involve mixing the materials at room temperature.

The mixture will then be poured into a rectangular tray, and allowed to cure in room temperature. After curing, the samples will be taken out of the tray and cut into the required geometry before post cured in a conventional oven. They will be post cured for four hours at 50° Celsius, then four hours at 80° Celsius, and finally two hours at 100° Celsius.

To determine the fracture toughness, flexural tests using the Hounsfield Testing Machine will be used to test the samples.

The three point (3-point) flexural test produces tensile stress in the convex side of the specimen and compressive stress in the concave side as load is applied on a sample of rectangular cross-section. By calculating the highest stress experienced by the outermost fibre and the amount of deflection at failure, we can determine the flexural strength of that material.

2 Literature Review

2.1 Introduction

This chapter will describe in detail the relevant literature involved in the undertaking of this project. This chapter will provide details about the materials used, curing and post curing, fracture mechanics and the testing.

The majority of the information in this chapter comes from published sources such as texts, and journals. Other sources of information are from USQ study materials and previous reports by students. Also, the Material Safety Data Sheets (MSDS) are supplied by companies for use with their products.

2.2 Introduction to Vinyl Ester Resins

Vinyl esters are thermosetting resins that are successfully and continually being used in industrial applications. Its continued utilization is due to its thermal, mechanical, and chemical resistant properties, which prove to be good quality when compared with its relatively low cost. Vinyl ester resin is formed from the reaction of a multifunctional epoxy resin and ethylenically unsaturated monocarboxylic acid. The product of this reaction is dissolved in styrene and gives a thermosetting liquid with a low viscosity which can be cured by radical polymerization when peroxides (e.g. MEKP) are introduced. Copolymerization of the styrene with the unsaturated vinyl ester resin produces a three-dimensional structure which can elongate along the length of the epoxy chain. This allows high elongation under mechanical and thermal stress; it allows high elongation, fatigue resistance, and good thermal resistance (Blankenship et al., (1989).

Properties of vinyl ester resins can vary depending on various factors. These factors include (Blankenship et al., 1989):

1. Epoxy resin structure, which determines mechanical and thermal properties, as well as corrosion;
2. The unsaturated acid, which affects reactivity and chemical resistance; and
3. The diluting monomer, which affects viscosity, reactivity, and chemical resistance

Vinyl esters are more costly than polyesters, and because of this, they are more often used in applications that specifically require superior corrosion, thermal, and fatigue properties.

Different techniques are used to manufacture corrosion resistant tanks, piping, ducts, and a wide range of fittings. Aggregate and sand mixtures with vinyl ester resins form strong, chemically resistant polymer concrete used in waste handling applications. High volume fabrication techniques take advantage of vinyl esters low viscosity and adjustable curing time in the production of composites of automotive, industrial and military applications (Blankenship et al., 1989). It is evident from these applications that vinyl esters are a player in the composites field.

2.3 Vinyl Ester Resin Used

The vinyl ester resin used in this investigation is Hetron 922. It was first introduced into the United States in the mid 1960's as a Shell Chemical Co. product, and has since become a well-established resin. There are two variations of Hetron 922; these are Hetron 922PAW, used in winter and Hetron 922PAS, used in summer. The main difference between the two is the gel time variation with respect to temperature. Both Hetron 922 PAW and PAS have been developed for exceptional protection in corrosion as well as chemical resistance applications. Some of the features of Hetron 922 include (Sweet, 2002),

- Excellent corrosion and chemical resistance;

- Excellent impact strength;
- High tensile elongation; and
- FDA compliance for food contact (FDA regulation Title CFR 177.2420)

Some applications include corrosion resistant tanks, pipes, vats, vessels, pumps, and other equipment, as well as coatings and linings.

It is recommended that post curing is done for maximum chemical and heat resistance.

2.4 Vinyl Ester Resin and Catalyst

The curing of vinyl ester resin is attained by radical polymerizations with peroxide.

Methyl Ethyl Ketone Peroxide (MEKP) is organic peroxide that is commonly used with vinyl ester resin; this is the catalyst (or accelerator) used for the polymerizations of the vinyl ester resin. The ratio of resin to catalyst was selected to be 98% to 2%. This is recommended for boat layups at moderate temperatures, i.e. 20° to 25°C (Sweet, 2002).

2.5 Glass Powder

Glass powder is made of fused inorganic oxides, and is spherical and non-porous.

They are used to improve the performance and reduce viscosity in paints and coatings. Glass powder is also a common lightweight additive in plastic components.

Glass powder is chemically inert, meaning they do not react with chemicals, and also has very low oil absorption. Table 2.1 shows typical properties of glass powder.

Table 2.1: Typical properties of Glass Powder

Typical Properties	
Shape	Spherical
Colour	White
Composition	Proprietary Glass
Density	1.1 g/cc and 0.6g/cc
Particle Size Mean Diameter	11 and 18 microns
Hardness	6 (Moh's Scale)
Chemical Resistance	Low alkali leach/insoluble in water
Crush Strength	>10,000 psi

The addition of glass powder to epoxy, compounds, fiberglass reinforced plastics, and urethane castings lowers costs and also gives weight reduction. It also improves impact resistance. Glass powder hollow spheres have insulating properties and improve thermal shock and heat affected areas.

2.6 Glass Powder Used

Glass powder is the filler used in this project. The glass powder used is SPHERICAL® 60P18 Hollow Glass Spheres, manufactured by Potters Industries Inc. Table 2.2 gives properties of SPHERICAL® 60P18 Hollow Glass Spheres.

Table 2.2: Properties of SPHERICAL® 60P18 Hollow Glass Spheres

True Density (g/cc)		0.60
	Mean volume	16-20
Particle Size (µm)	D10	6-10
	D50	15-19
	D90	28-32
Working Pressure	10 Volume % Loss	8,000psi
Appearance		White powder
Composition		Fused Inorganic Oxides
Shape		Spherical, Non-Porous

2.7 Fracture Mechanics

Fracture mechanics may be defined as the field of solid mechanics that deals with the behaviour of cracked bodies subjected to stresses and strains. Many engineering failures have been caused by the pre-existing cracks or other defect. Fracture mechanics aims to determine the severity of a pre-existing defect in terms of its tendency to initiate fracture. It is also concerned with resistance to crack propagation and with crack growth rates in fatigue and stress corrosion cracking.

Fracture mechanics is a tool which relates the size of a flaw to the likelihood of it causing fracture in a given material under a given stress regime. The larger flaw will lower the stress at which failure will occur. Conversely, the lower the service stress the larger the flaw which may exist without endangering the structure (AWRA, 1980).

The loading mode is important with respect to the state of stress at a flaw and consequently the crack propagation. Cracks move through 3-D space, the crack path is dictated by the microstructure and the state of stress at any point ahead of the crack; hence the appreciation of the state of stress and modes of loading must be included in any fracture analysis. The three basic loading modes are (a) mode I, crack opening, (b) mode II, in-plane shear, and (c) mode III, anti-plane shear or tearing (Figure 2.1)

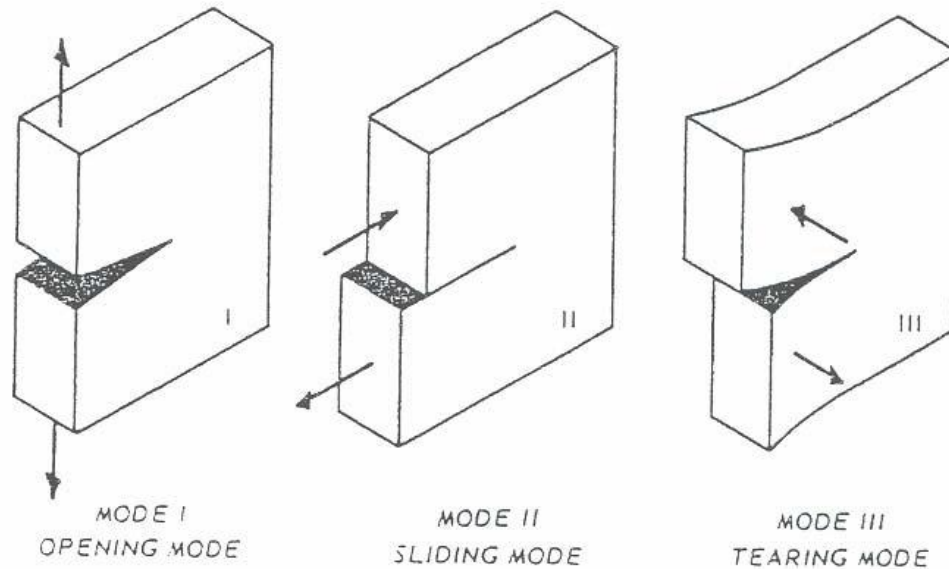


Figure 2.1: Three modes of loading.

2.8 Fracture Toughness

Toughness is the ability of a material to absorb energy when being deformed and thus resist deformation failure. If a material is tough, it is able to absorb a lot of energy before it fails under load. Fracture toughness can be measured and expressed in a number of ways such as Joules or stress intensity factor K depending on the nature and method of the test and end use of the results of these tests.

The concept of stress intensity factor K comes from Linear Elastic Fracture Mechanics (LEFM) and is strictly applicable only to materials in which cracks can grow with very little plastic deformation. That is cracks can grow within the linear elastic range of the material so that linear elastic analysis may be applicable. This implies that LEFM is particularly relevant to brittle materials since brittle materials undergo little or no plastic deformation before fracture or rupture.

A sharp crack-like defect of length $= 2a$ is assumed to be present in an infinite plate and the stress intensity factor, K , at the tip of the crack is calculated from

$$K = f \sigma \sqrt{\pi a}$$

Where σ is the elastic stress acting on the structure, and $f = 1$ for an infinite plate.

In the configuration shown in figure 2.2, where the crack is being opened by a tensile stress normal to it, the stress intensity factor is known as K_C .

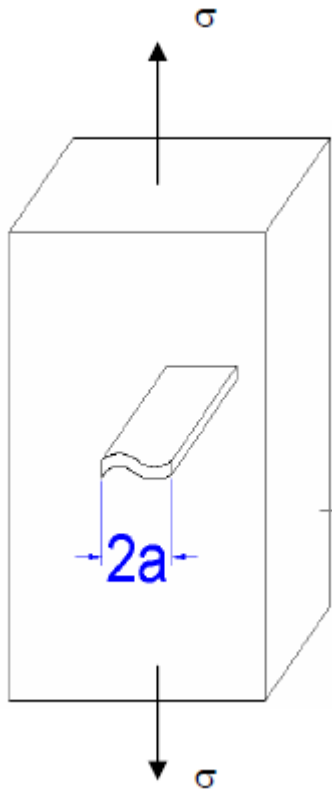


Figure 2.2: Rectangular through-thickness crack in finite plate.

Adopting a fracture mechanics approach to the design and selection of materials in engineering allows one to compensate for the inevitable presence of flaws. Three variables to consider in this approach are: The material property (K_C or K_{IC}), the stress σ that the material must withstand and the size of the flaw a . If we know two of these variables, the third can be determined. Chew (2003) summarized the importance of fracture mechanics in relation to the inevitable presence of flaws as follows:

- Selection of a material: If we know the maximum size a of flaws in the material and the magnitude of the applied stress, we can select a material that has a fracture toughness K_C or K_{IC} large enough to prevent the flaw from growing.

- Design of a component: if we know the maximum size of any flaw and the material (and therefore its (K_C or K_{IC})) has already been selected, we can calculate the maximum stress that the component can withstand. Then we can design the appropriate size of the part to ensure that the maximum stress is not exceeded.
- Design of a manufacturing or testing method: If the material has been selected, the applied stress is known, and the size of the component is fixed, we can calculate its fracture toughness.

The above concept of stress intensity factor, K has been provided for completeness. There are numerous issues involved in the use of the stress intensity factor:

- The stress intensity factor or toughness is obviously different for different materials.
- Toughness is a function of thickness of the specimen. Thicker more rigid materials have lower fracture toughness than thin materials. As thickness increases, fracture toughness K_C decreases to a constant value K_{IC} . This is the plane strain fracture toughness which is normally reported as the property of a material.
- Toughness is dependent upon temperature, increasing the temperature generally increases the fracture toughness of a material. In the case of metals this could mean raising the temperature above the brittle-ductile transition temperature changes the material behaviour from brittle to ductile.

- Toughness is sensitive to the rate of stress and strain applied to a specimen, this relates to state of stress on a specimen and how a material behaves under these stress conditions. An example is that of corn flour mixed with water which exhibits some degree of rigidity when subjected to a relatively high impact force. On the other hand when the same mixture is subjected to a low force, the mixture is easily deformed.
- Large flaws reduce the permitted stress. Manufacturing techniques can reduce flaw size and improve fracture toughness.
- The ability of a material to deform is critical. In ductile metals, the material near the tip of the flaw can deform, causing the tip of any crack to become blunt, changing the stress conditions in the material. Let's consider a blunt notch as depicted in Figure 2.3. Broek (1997) states that at a blunt tip, the plane stress condition changes into a plane strain condition very rapidly with respect to distance from the crack tip. The resultant tri-axial stress condition which is complimentary to a plane strain condition means that a higher axial force is required to initiate crack propagation, assuming the material fails under shear.

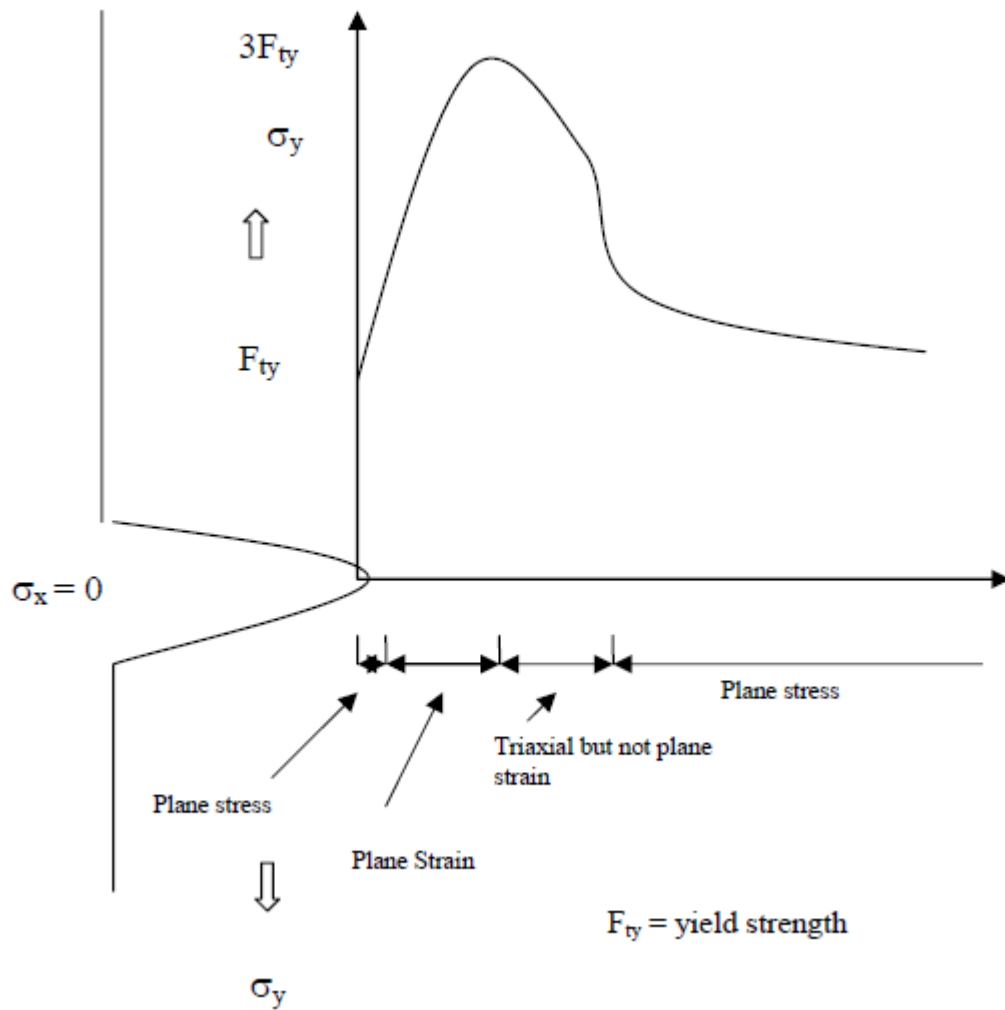


Figure 2.3: Progress of yield at a blunt notch.

The term toughness is the capacity of a material to absorb energy by deforming plastically before fracture. It is determined by the combined strength and ductility of a material and usually is measured by the amount of work absorbed during the propagation of a crack.

Toughness can be measured in a variety of ways, but this project focuses on the test method using the theory of a three point load acting on a simply support beam as shown in Fig. 2.4

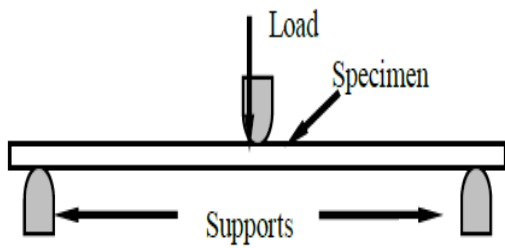


Fig. 2.4: Three-point loading system on a simply supported beam

Fractures are usually classed as brittle or ductile, depending on the amount of plastic deformation preceding failure. Brittle fractures occur suddenly with little or no prior deformation. The type of fracture in a material is often related to the temperature. With regards to steel, it has been observed that brittle fracture occurs at low temperatures whereas ductile fractures occur at higher temperatures.

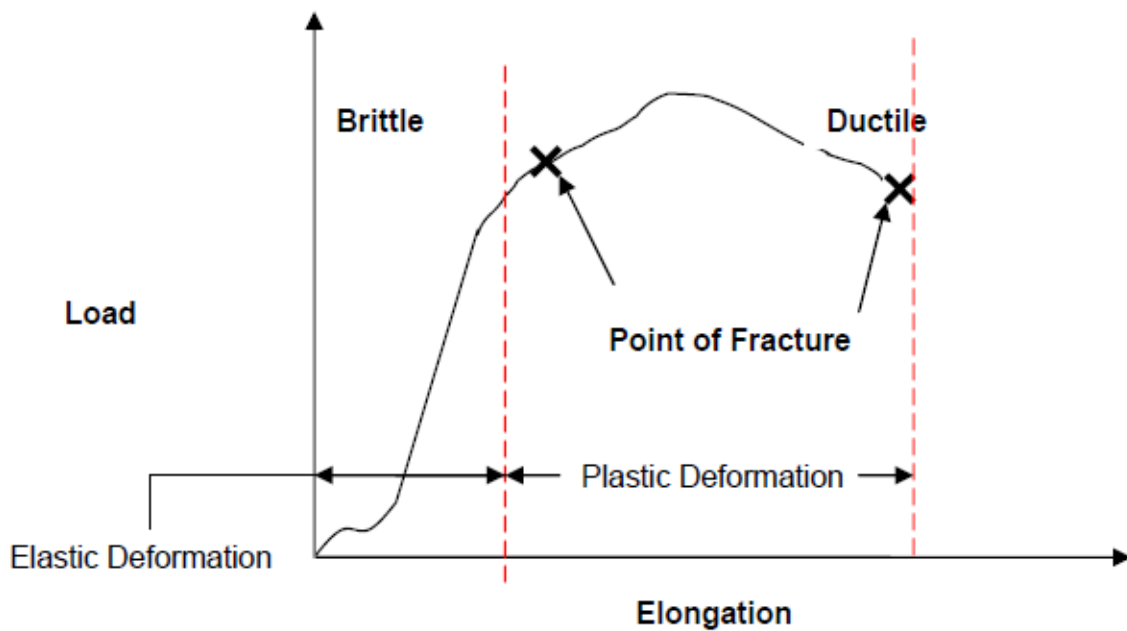


Figure 2.5: Typical stress-strain graph of a metal showing points at which brittle and ductile fracture occur.

2.9 Plane Strain Fracture Toughness

Plane strain exists when a specimen's thickness is large enough that the crack's size will not influence the specimen's fracture toughness. In plane strain, there will be no resulting strain perpendicular to the front and back faces of the sample. This means the load will be purely a tensile load, also known as mode I loading (Juvinall & Marshek, 2001). The fracture toughness will become the plane strain fracture toughness, i.e. K_{Ic} will be K_{Ic} .

$$K_{Ic} = f\sigma\sqrt{\pi a}$$

Brittle materials have low K_{Ic} , while ductile materials have high K_{Ic} values. Plane strain fracture toughness is an important property and can be affected by a number of factors including, temperature, microstructure, and strain rate. K_{Ic} decreases with increase strain rate, and decrease temperature (Askeland 1998).

2.10 Hounsfield Flexural Testing Machine

The Hounsfield flexural testing machine was used and it consists of the following apparatus: a fixed member, a moveable member and grips on both sides. The materials to be tested are held together by the grips on both the fixed and moveable member. With reference to the ASTM D638-00 standards, for test specimens of moulded plastics that are rigid or semi-rigid, the test specimen shall conform to certain dimensions as directed. The specimen thickness used in this study has a thickness of 3mm. The recommended number of specimens to be tested is at least six per sample. The speed of testing is the relative rate of motion of the grips during the test. In this case, the speed of testing for rigid or semi-rigid specimens of Type 1 is at $5 \pm 25\%$ mm/min. The controls were done by using a Windows based software that was connected to the Hounsfield machine. A graph of Force vs Extension was plotted on the screen and the force would be taken at the point where the sample fails. The Hounsfield

Flexural three point tester utilises the same Hounsfield machine as the Hounsfield Tensile test. This test determines the flexural properties of the unreinforced rigid or semi-rigid plastics that were moulded.



Figure2.6: Three-point loading system on a Hounsfield testing machine

2.11 Determining Fracture Toughness

Flexural measurements was carried out using the Hounsfield testing machine, according to ASTM D790 using a three-point bending configuration at 2.38 mm/min deformation rate. The single-edge notched samples were cut out and subjected to static tensile loading (SEN-T) samples. The sample notching is done by sawing and sharpening with a razor blade. SEN-T samples were tested at 1 mm/min at 238C. The geometry of the sample is shown in Figure

2.7. The fracture toughness was determined according to ISO 13586 with fracture toughness parameters, which are calculated by Equations (1) and (2):

$$K_c = \frac{F_{max}\sqrt{a}}{BW} f\left(\frac{a}{W}\right) \quad (1)$$

Where $f\left(\frac{a}{W}\right)$ is the geometry correction factor given by:

$$f\left(\frac{a}{W}\right) = 1.99 - 0.41\left(\frac{a}{W}\right) + 18.7\left(\frac{a}{W}\right)^2 - 38.48\left(\frac{a}{W}\right)^3 + 53.85\left(\frac{a}{W}\right)^4 \quad (2)$$

Where, F_{max} is the maximum force in the force–deflection trace, B is the thickness of the sample, W is the width of the sample, and a is the total notch length.

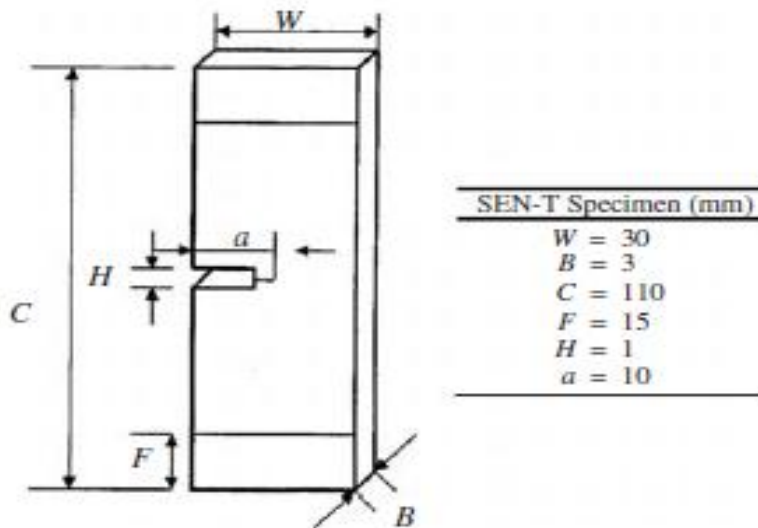


Figure2. 7. Geometry of SEN-T sample.

2.12 Flexural Stress

The flexural strength is the stress on the surface of the specimen at failure, which should be demonstrated as a shear in the middle of the sample vertically. The strength is calculated using the maximum bending moment, corresponding to the failure load. Flexural failure is encouraged by the use of a large span to specimen thickness ratio. The span of the beam has no influence on the shear stress but a large span may result in a high bending moment

producing longitudinal failure. When using a large span to thickness ratio it can produce large deflections under load that is why for this study a span of 64 mm is applied.

Flexural strength is measured in terms of stress, and is expressed in mega Pascals (N/mm²).

The value that is calculated is the highest stress before the moment the object breaks. The highest stress in a bending stress is normally found on the surface of the sample [Hodgkinson 2000, p.128/ Wikipedia, undated].

$$\sigma_f = \frac{3PL}{2bd^2}$$

- σ_f = Stress in outer fibres at midpoint, (MPa)
- P = load at a given point on the load deflection curve, (N)
- L = Support span, (mm)
- b = Width of test beam, (mm)
- d = Depth of tested beam, (mm)

2.13 Flexural Strain

Flexural strain is a dimensionless measure; it is defined as the ratio of elongation with respect to the original length. It is shown as the specimen is being tested as the bend in the sample.

Strain is important when internal stress considerations are needed [Wikipedia, undated].

$$\varepsilon_f = \frac{6Dd}{L^2}$$

- ε_f = Strain in the outer surface, (%)
- L = Support span, (mm)
- d = Depth of tested beam, (mm)
- D = maximum deflection of the centre of the beam, (mm)

2.14 Young's Modulus

The Modulus of Elasticity, also known as Young's modulus, is a measure of the stiffness of a material. It can be calculated in two of the following ways:

The Young's Modulus also called an elastic modulus, or modulus of elasticity, is the description of an object's tendency to bend elastically when a force is applied to it.

So it bends to a point of where if it bends further failure will occur. The elastic modulus of an object is defined as the slope of its stress-strain curve in the elastic deformation region. For comparisons of previous results this method of calculation will be utilized. The two formulas are listed below [Wikipedia, undated].

$$E = \frac{\text{stress}}{\text{strain}} = \frac{\sigma}{\epsilon}$$

$$E_B = \frac{L^3 m}{4bd^3}$$

- E_b = Modulus of elasticity in bending, (MPa)
- L = Support span, (mm)
- b = Width of test beam, (mm)
- d = Depth of tested beam, (mm)
- m = Slope of the tangent to the initial straight-line portion of the load deflection curve, (N/mm)

2.15 Curing and Post Curing

The samples will be cured at room temperature. For this, promoters (or accelerators) must be added to the resin to induce decomposition of the peroxides forming free radicals. This will ensure an adequate rate of curing. Certain metallic soaps and tertiary amines are effective accelerators; however, the most common is methyl ethyl ketone peroxide (MEKP). If a sufficient exothermic reaction is achieved, green strength develops rapidly. With this in mind, post-curing will give optimum properties. A strong exotherm can result in cracking and

possibly also decomposition. An alternative would be to use benzyl peroxide and dimethyl aniline; this also gives faster curing times and is less sensitive to moisture effects (Blankenship et al., 1989).

2.16 Microwave Curing

Microwaves are electromagnetic waves with wavelengths ranging from 1 mm to 1 m and frequency that ranges from 300 MHz to 30 GHz. According to international agreement, industrial microwaves operate at a frequency of 2.54 GHz, which is powered by a variable power generator up to 1.26kW.

The microwave oven uses a magnetron to create intense microwaves that are channelled to the microwave cavity using electromagnetic waves with a frequency of 2.45 GHz. However, if greater power penetration is required, a system with a frequency of 915 MHz can be used.

Important properties that are involved in the theory of microwave curing of materials include the wave propagation, microwave instrumentation (which includes the magnetron, impedance matching and tuning, waveguides used and the microwave cavity) and the dielectric properties of the material. The heating pattern of a sample that is heated by microwaves will depend on the dissipation factor which can be expressed by: $\tan \delta = \varepsilon''/\varepsilon'$ and the dielectric may be assumed to have a complex dielectric constant as: $\varepsilon = \varepsilon' - j\varepsilon''$

The energy that is absorbed by the sample as the microwave energy penetrates it is dependent on the sample's dissipation factor. Materials that are transparent to microwave energy, penetration is considered to be infinite. As in the case of reflective materials such as metals,

penetration is considered to be zero. However, the dissipation factors for absorptive materials are finite.

The permittivity, ϵ' , mostly determines how much of the incident energy is directed at the air-sample interface, and how much enters the sample. In microwave processing an important property is the loss tangent, $\tan \delta$, which predicts the ability of the material to convert the incoming energy into heat. For optimum microwave energy, a considerable value of ϵ' should be combined with high values of ϵ'' and $\tan \delta$, to convert microwave energy into thermal energy. Depending on the material the depth of penetration of energy varies, and so the amount of heat will vary. The depth of the energy is controlled by the dielectric properties. The depth is at which approximately $\frac{1}{e}$ (36.79%) of the energy has been absorbed. It is also approximately given by (Bows, 1999):

$$D_p = \left(\frac{4.8}{f}\right) \frac{\sqrt{\epsilon'}}{\epsilon''}$$

Where D_p is in cm, f is in GHz and ϵ' is the dielectric constant.

Microwave curing is a fairly new procedure because the microwave itself has only recently been introduced. Using a microwave rather than an oven gives a great potential for reduction in cycle time and cost. The benefits of using a microwave also include high heating rates, and the ability to heat the sample from the inside out. This gives the specimen a more uniform mechanical property, and should provide stronger more consistent results.

If this method is brought into a large scale manufacturing role it will see much improvements over the old oven method. First of all a reduction in the impact of materials processing; cost advantages in energy savings, space, and time; and an opportunity to produce new materials that cannot be achieved by other methods [Ku H S, 2003].

Although since the microwave can only be set with one temperature, or energy, rating a heating procedure must be developed to find the most accurate way to reach the desired 100°C. This is where the microwave is flawed, in comparison with the oven. With the oven the user simply sets the temperature and waits until it the oven has reached it.

The microwave used in this study is a Sanyo 800 watt compact microwave. At max energy preliminary tests where done and it was noted that temperatures exceeded the 100°C limited within a few minutes.

2.17 Oven Curing

In order to get a better understanding why the microwave is now being applied in these types of projects, a look into how the oven cures specimens is described. In any oven the temperature is set and the temperature rises in the entire oven until the desired temperature is acquired. That is a disadvantage to the microwave, since it uses waves to impregnate the sample with heat from the insight out and takes considerably less time. Also heating the entire cavity of the oven will cost a lot more.

In a large production scale this time difference will be expensive, because of the curing time difference the company will need a lot more, and a lot larger ovens then that of microwaves.

2.18 Permittivity

The permittivity of a dielectric material has both real and imaginary mathematical representations. The imaginary part of Permittivity is represented in mathematical equations as epsilon double prime (ϵ'') or sometimes kappa double prime (k''). This imaginary part of Permittivity describes the energy loss from an AC signal as it passes through the dielectric. The real part of permittivity, (ϵ'), epsilon prime or (k'), kappa prime, is also called dielectric

constant and relative permittivity. The permittivity of a material describes the relationship between an AC signal's transmission speed and the dielectric material's capacitance.

When the word "relative" is used in front of permittivity, the implication is that the number is reported relative to the dielectric properties of a vacuum. All measurements that you and I will ever use are relative permittivity numbers.

The relative permittivity number can then be used to calculate the impedance of a given circuit, helping the PWB designer optimize a circuit for impedance matching characteristics.

$$\text{Relative Permittivity } (\epsilon') = C_p / C_v$$

Where C_p = Capacitance of; dielectric between two parallel plates.

C_v = Capacitance of the same thickness of air (vacuum) between the same two parallel plates.

2.19 Dissipation factor

The simplest way to define dissipation factor (loss tangent) is the ratio of the, energy dissipated to the energy stored in the dielectric material. The more energy that is dissipated into the material, the less is going to make it to the final destination.

This dissipated energy typically turns into heat or is radiated as RF (Radio Frequencies) into the air. The optimal goal is to have 100% of the signal pass through the interconnection network, and not be absorbed in the dielectric. With "high power" signals, a material with a large dissipation factor could result in the development of a tremendous amount of heat, possibly culminating in a fire (advanced dielectric heating). When the signals are very weak a high loss material means that little or no signal is left at the end of the transmission path. In order to retain maximum signal power, a low loss material should be used.

Dissipation factor = ϵ''/ϵ' where: ϵ' is the real portion of permittivity, and ϵ'' is the imaginary portion of permittivity.

3 Project Methodologies

3.1 Introduction

This chapter will outline the process involved in the preparation, production, curing and post-curing, testing of the samples. The underlying method implemented in this project was intentionally kept similar to previous years methods for the purpose of obtaining repeatable results. These processes were demonstrated in reports previously done by students, which were provided by the supervisor as a guide. Elements such as the materials, mould, and post curing were among the aspects kept constant. Production techniques have improved to give the best possible samples with the least possible defects. Production technique is something that can change, but the main aim of the specimen production was kept in sight.

3.2 Mould and Mould Preparation

The mould used for preparing the samples for flexural test is shown in figure 3.1. A total of eight moulds made of aluminium sheets were used. They have strong bottom surfaces which are tough enough to support the weight of the specimens without any distortion. The moulds size is 280 mm long, 210mm wide and 15mm deep. These moulds were specifically chosen to make six (6) test samples. The moulds were then completely covered by wax paper before the mixtures of the specimens were poured into the moulds to prevent the specimens from sticking to the surface of the mould. This is a very important step; if strong force is used to remove the specimens it might cause visible and non- visible cracks in the samples, which would significantly alter the test results.



Figure 3.1: The mould for casting flexural test sample

Figure 3.2 shows the mould used to casting the samples for loss tangent test and dynamic mechanical thermal analysis. The mould was consisted of two PVC plates. A piece of wax paper was placed between the upper and lower plates to prevent the specimens from adhering to the surfaces of the plates. As illustrated in Figure 3.2, there were two figure size slots. It was very difficult to fold the wax paper to cover such a small area; hence a small amount of wax was smeared on the surface of the slots. The wax also was used to seal the edges of the slots to prevent the mixture seeping through the gaps between the two plates. Screws were also used to tightly hold the two plates together.



Figure 3.2: The mould used for casting the samples for loss tangent test and thermal analysis

3.3 Sample Production

During the casting process, a mould casts one specimen, with all the different percentage by weight of glass powder. Therefore, eight samples are made, with each sample to produce six specimens. The samples made were from the range of 0% glass powder to 35% glass powder, in increments of 5%.

3.4 Measuring materials

Before handling any of the materials, it was essential that their Material Safety Data Sheets (MSDS) were read and understood. Wearing the appropriate Personal Protective Equipment (PPE) was also required. Before bringing out the materials, their weights had to be determined. The samples varied from 0% glass powder to 35% glass powder, in increments of 5%. The accelerator or MEKP was to be 2% by weight to resin. Since the

density of MEKP is 1 cc (i.e. 1 gram equals 1 milliliter), it was quite easy to extract an accurate amount. For a mould of this size, 900 grams of material had to be used. This weight decreased however, as the percentage of glass powder increased. At 35% glass powder, 800 grams of material was being mixed.

Table 3.1 shows the different percentages of weight of glass powder to resin, as well as accelerator. Reading off this table, the materials are measured in separate containers. It is important to zero the scale before adding the materials into the containers. Once the measured amounts are obtained, they are ready to be mixed.

Table 3.1: Percentages by weight of Glass Powder/VE Resin

Percentage	Composite (g)	Resin (g) (VE)	VE: Cat Ratio	MEKP (ml) Cat	Glass Powder (g)
5 - 15	900	416.7	50 : 1	8.3	75
20 - 30	900	387.1	30 : 1	12.9	100
35	900	300	12 : 1	25	175

3.5 Mixing the Materials

Mixing of the materials is a very important process. This is the stage which can alter the structure of the specimens. The resin and glass powder were mixed together first, this had to be done slowly to minimize any formation of air bubbles in the mixture. If air bubbles formed in this stage, they would be in the specimen after curing, thus creating areas of localized stress concentration during testing. Mixing slowly in a figure eight motion around the container was found to be an adequate method; it allowed the glass powder to blend in with the resin, while the slow speed minimized the formation of air bubbles. After the resin and glass powder had been mixed together, the accelerator was ready to be mixed in. The accelerator had to be mixed in at a quicker rate than the resin and glass powder. This is

because the accelerator will actually start the curing process, making the mixture more viscous. When all three are mixed in together, the mixture can be poured into the mould.



Figure 3.3: Mixing the materials

The mixing was done in the ventilation chamber with the exhaust fan turned on. This allowed most of the fumes to exhaust out from the work environment. The windows were also opened to allow a flow of fresh air through the room.

3.6 Sample Cured in Room Temperature

The samples were left to cure in ambient conditions for three days. After this, they were removed from the mould and the sample notching is done by sawing, grinding and sharpening with a hack saw blade

3.7 Oven Post-Curing

The samples are to be post-cured in a conventional oven over a course of ten hours.

The oven was programmed using a Eurotherm 3200 Series Controller, to heat the specimens at 50 degrees Celsius for four hours, then 80 degrees for four hours, then 100 degrees for two hours. Using the controller allowed the oven to control itself, without interaction from anyone. It was observed however, to make sure the temperature did change after the prescribed time. All the specimens were able to fit into the oven at the one time, but care had to be taken to make sure they were evenly spaced. This meant the specimens could be evenly heated to the required temperature, without any uneven temperature regions.

Please note, care should always be taken when using the oven, as the temperature in the oven is high. The temperature on the controller was always checked before opening the oven door.

The oven and specimens were allowed to cool before retrieving the specimens from the oven.

The oven was made by Steridium, which are commonly installed with Eurotherm controllers.

The Eurotherm 3200 Series Controller user manual was used to program the controller to the desired requirements. The programming instructions may be seen in Appendix D.

ester composites. After the heated specimens cool down to room temperature, the specimens were then again placed into the oven and heated to 50°C. It required 20 minutes with selected power level of 160 W for the specimens to reach 50°C. The specimens then left in the oven to cool down to room temperature. The specimens were again heated to 60°C. This time, it took 25 minutes with selected power level of 160 W for the specimens to reach 60°C. One thing needs to be mentioned here, for safety and health reason; this microwave oven had been modified to remove the curing glass powder via an attached air duct. All the windows should keep open during the process to keep good air circulation in the room.



Figure 3.5: The microwave oven used for this project



Figure 3.6: The infra-red handheld thermometer

3.9 Flexural Tests

3.9.1 Preparation for Testing

Fig 3.7 shows the mould used to cast the test specimen and cured in room temperature.



Figure 3.7: The mould used to cast flexural test specimens.

The sample had to be sawn, grinded and sharpened with a hack saw blade to meet the required geometry needed for this test after been cured in room temperature as shown in Fig 3.8.

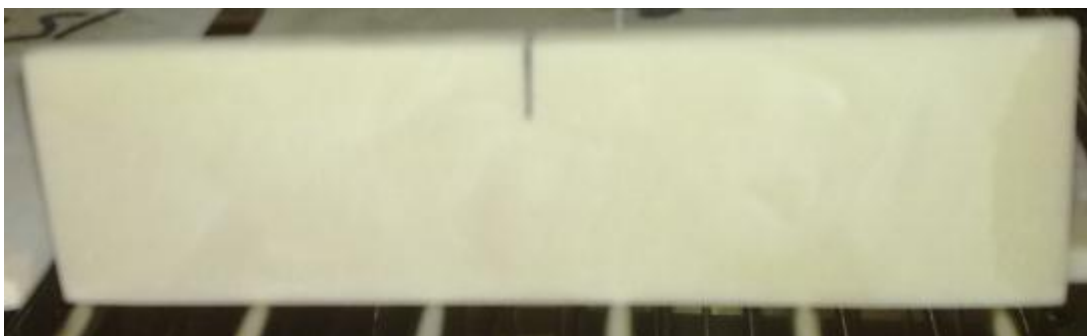


Figure 3.8: The finished product of flexural test specimens.

3.9.2 Flexural Testing

The Hounsfield flexural testing machine was used and it consists of the following apparatus: a fixed member, a moveable member and grips on both sides. Before placing the samples into the grips, the width and thickness were measured by an electronic vernier calliper and entered into the computer program to calculate the young's modulus and flexural strength. Figure 3.10 shows the calliper which is used for the dimension measurement of the entire project. The materials to be tested are held together by the grips on both the fixed and moveable member. With reference to the ASTM D638-00 standards, for test specimens of moulded plastics that are rigid or semi-rigid, the test specimen shall conform to certain dimensions as directed. The specimen thickness used in this study has a thickness of 3mm. The recommended number of specimens to be tested is at least six per sample. The speed of testing is the relative rate of motion of the grips during the test. In this case, the speed of testing for rigid or semi-rigid specimens of Type 1 is at $5 \pm 25\%$ mm/min. The controls were done by using a Windows based software that was connected to the Hounsfield machine. A graph of Force vs Extension was plotted on the screen and the force would be taken at the point where the sample fails. The Hounsfield Flexural three point tester utilises the same Hounsfield machine as the Hounsfield Tensile test. This test determines the flexural properties of the unreinforced rigid or semi-rigid plastics that were moulded. The result that was required from the testing was the maximum load; this would be used in the calculation of the specimen's fracture toughness.



Figure 3.9: Specimen loaded onto Hounsfield testing machine

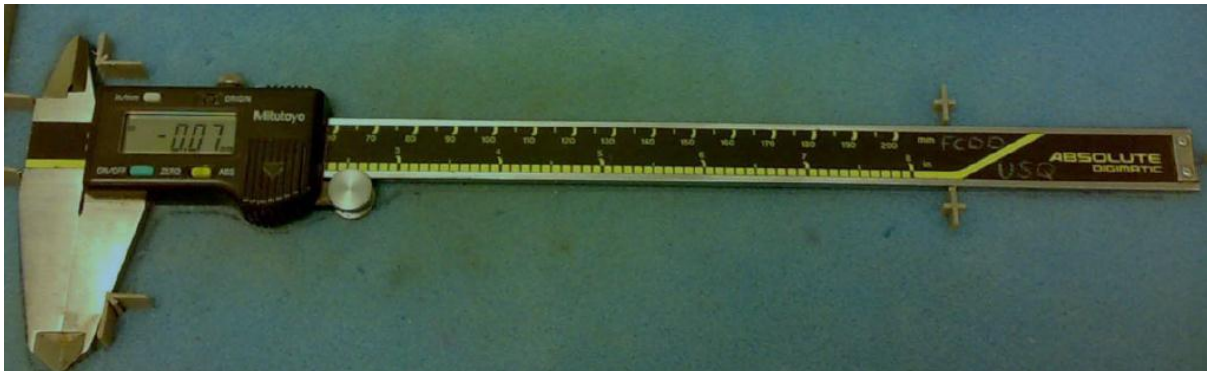


Figure 3.10: Electronic vernier calliper

3.10 Dielectric Constant and Loss Tangent Measurement

3.10.1 Preparation for Testing

Fig 3.11 shows the mould used to cast the test specimen and cured in room temperature.



Figure 3.11: The mould used to cast parallel plate test specimens.

3.10.2 Parallel Plate Testing

The method used for this project was called the parallel plate measurement method. Unlike the resonant cavity method, this method is established based on some assumptions; hence this method can only provide approximate values for the loss tangents and permittivity. This experiment was carried out in a specially designed room. The wall of the room and the roof were made of metal and earthed. The earthed metal wall provided a shielding to protect the room from the outside electromagnetic interference. The metal door has to be closed before the experiment starts. Figure 3.12 shows the equipment set-up for the loss tangent and dielectric measurement. First, the test sample was placed between two copper plates. The copper plates are 110mm x 110 mm which are slightly smaller than the sample which is 120mm x 120mm. Then copper plates and the sample were inserted into two wooden clampers. The copper plates and the sample have to be bolted tightly together to minimize the

air gap between the plates and the sample. The two measurement leads (black and red) of LCR meter were connected to the wires which were soldered to the middle surface of the two copper plates to allow the current to flow. After the LCR meter was turned on, the measuring parameters C_p and D which were parallel capacitance and dissipation factor respectively were selected for the measurement. The dissipation factor D is also known as loss tangent. The parallel capacitances and dissipation factors were measured at 100 Hz, 1 kHz, 10 kHz, 20 kHz and 100 kHz respectively. Ideally, the measurement should be conducted at higher frequencies since the frequency range of microwave is between 300 MHz to 1000 GHz. However, with the limitation of signal generating capability of the LCR meter, the maximum signal could be generated by the LCR meter is 100 kHz. Although the measurement was carried out at lower frequencies, the results could still give certain indications for the electrical properties of the test materials. The test results are read off the screen of the LCR meter and entered manually into a spread sheet for analysis.

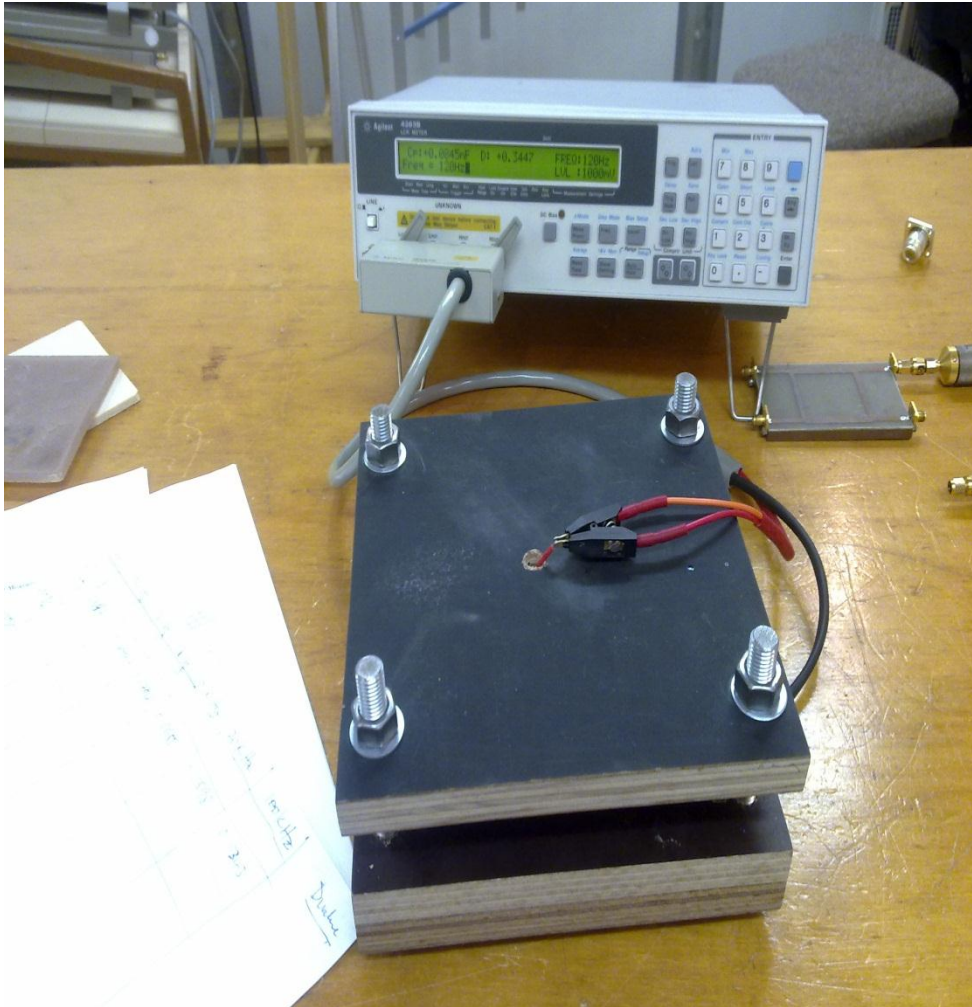


Figure 3.12 Parallel Plate measurement equipment set-ups.

Figure 3.13(a) shows the equivalent electrical circuit for the samples under test. The red lead of the LCR meter carried a small amount of current flow into the copper plate, and the black lead carried the current flow back to the LCR meter. The two copper plates with the test sample in the middle formed a parallel plate capacitor. C_p is the parallel capacitance of the samples and G is the shunt conductance of the sample. By applying the ac voltage across two copper plates, the ac current will flow through the equivalent circuit. Figure 3.13(b) shows the phasor diagram for the currents flow through the equivalent circuit. The Phase difference between I_c which is the current flows through the capacitance and I_g which is the current flows through the conductance is 90° out of phase. The angle between the conductance current I_g and the resultant current I is the loss angle δ . Hence the loss tangent can be

determined by measuring the phase angle of the resultant current respect to the capacitance current I_c . Therefore, the LCR meter is able to directly measure the loss tangent.

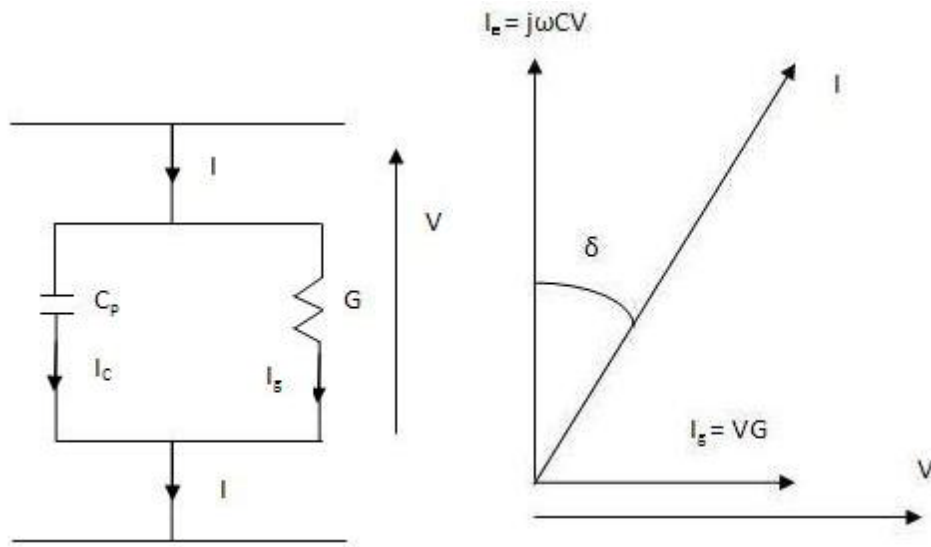


Figure 3.13: (a) equivalent circuit for the sample under test (b) phasor diagram

The LCR meter is also able to directly measure the capacitance and the conductance of the material. The permittivity can be calculated by rearranging

Eq (2.10):

$$\epsilon_r = \frac{S \times C_p}{A \times \epsilon_0} \quad (3.1)$$

Where S = the average thickness of the sample in mm^2

A = the surface area of the plate in mm^2

$$\epsilon_0 = 8.854187 \times 10^{-12} \text{ Fm}^{-1}$$

C_p = the measured parallel capacitance

Hence, once the area of surface of the plate, the thickness of the sample and the parallel capacitance are measured, the value of ϵ_r can be easily determined by Eq (3.1). Furthermore, the conductance of the samples is given by:

$$G = \frac{\sigma' \times A}{s} \text{ (S/m)} \quad (3.2)$$

Where σ' is the effective a.c. conductivity

$$\text{and } \sigma' = \sigma + \omega \epsilon_0 \epsilon''$$

Where σ is the DC conductivity of the material

ω is the frequency of the test signal

Divide Eq. (2.10) by Eq. (3.2), it would yield:

$$\frac{C}{G} = \frac{\epsilon_0 \times \epsilon'}{\sigma \times \omega \epsilon_0 \epsilon''} \quad (3.3)$$

Assuming the DC conductivity σ equal to zero for good dielectrics, Eq. (3.3) becomes:

$$\frac{C}{G} = \frac{\epsilon'}{\omega \epsilon''} \quad (3.4)$$

Substitute Eq. (2.7) into Eq. (3.4), the new equation becomes:

$$\tan \delta = \frac{G}{\omega C} \quad (3.5)$$

Hence, alternatively the loss tangent can be calculated by measuring the conductance and the capacitance of the material at different frequencies.

3.11 Dynamic Thermal Mechanical Analysis (DTMA) test

3.11.1 Preparation for Testing

Fig 3.14 shows the test specimen geometry.



Figure 3.14: The test specimens for DTMA.

3.11.2 DTMA Testing

DTMA test is a technique to measure the visco-elastic properties of the materials which included storage modulus, loss modulus and phase angle $\tan\delta$. Clarification needs to be noted that the phase angle $\tan\delta$ is different from the loss angle $\tan\delta$ which is an electrical property.

The main principle of the DMA test is an established fact based on the linearity between the stress and strain. The stress and the strain can be measured by applying a force to make the material oscillate.. There are two methods implemented to apply the force. One of the

methods is called free oscillation method. In this method, the force is only applied to the material for a very short period. Once the material starts oscillating, the external force is removed and allows the material to oscillate freely. Another method is called forced oscillation. In forced oscillation method, the oscillating force (usually sinusoidal oscillating force is used) is continuously applied to the material throughout the whole period of testing. Hence the material tested by the forced oscillation method would oscillate at the exactly same frequency as the applied force. Therefore, the forced oscillation can provide more reliable results than the free oscillation method when performing a temperature sweep. In this project the forced oscillation method is used and the test is conducted by applying a sinusoidal oscillating force to the material under test. Visco-elastic materials have two distinct physical states. Before reaching the glass transition temperature, the material is in an elastic solid state. The strain occurred to the material is proportional to the stress applied to the material and in phase, hence the material oscillates at the same frequency as the applied stress. After passing the glass transition temperature, the material starts turning into a viscous fluid state. In this state, the resulting strain gradually lags the stress as the temperature increases. When the material completely turns into the viscous fluid state, the strain lags the stress by 90 degree. The phase lag between the strain and the stress is called phase angle δ .

The storage modulus E' is the measure of the stiffness of the elastic material. It is proportional to the energy stored during the period of material elastic deformation occurs. Since the deformation is elastic, the process is reversible and the energy stored at this stage can be released back to the system. Thus the energy consumption at this stage is insignificant. However, after the testing temperature rises above the glass transition temperature, the material turns into the viscous fluid state. At this state, the resulting strain starts lagging behind the applied stress. As the strain and the stress are not in phase, the absorbed energy is converted to heat and cannot be recovered. The loss modulus E'' is the measure of this non-

reversible heat loss. The loss factor $\tan \delta$ is the ratio of loss modulus E'' to storage modulus E' . It is a measure of the energy lost, expressed in terms of the recoverable energy, and represents mechanical damping or internal friction in a viscoelastic system (Hanser 2006). Figure 3.14 shows the typical storage modulus and $\tan \delta$ curves. At the beginning of the test, the storage modulus is at its maximum value because the temperature is the lowest. As the material turns from elastic solid state to viscous fluids state after the testing temperature rise above the glass transition temperature, the storage modulus which representing elastic property drops dramatically to nearly zero MPa while the loss modulus reaches the maximum. Hence, maximum point on the curve of the loss modulus can be used to determine the glass transition temperature. There are two main test modes are used for DMA test. The mode was chosen for this project is temperature sweep. With this mode, the material under test is subject to a sinusoidal stress which is fixed at a low constant frequency while increasing the sample temperature. Another test mode is so called frequency sweep mode. In this mode, unlike the above mode, varying the temperature, the frequency of the sinusoidal stress is swept over a range of frequencies.

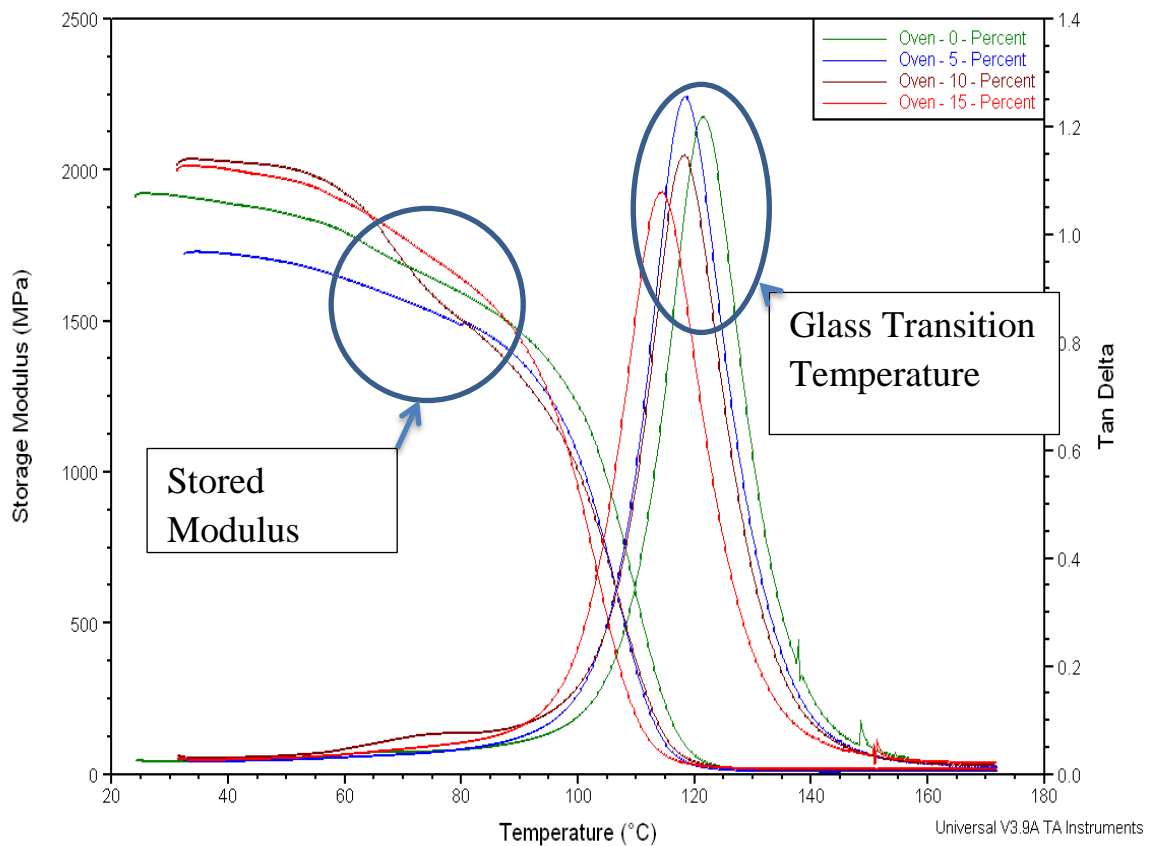


Figure 3.15: The DTMA test results for oven cured vinyl ester resins reinforced with 0% - 15% glass powder

Figure 3.16 shows the DMA instrument used for this project. The sample is firstly placed in the clamps, and held tightly by the clamps. After the sample been place in the clamps the cover would move down and close the test chamber. That gives better control for the testing temperature in an enclosed environment. The stepper motor underneath the clamps then drives the shaft to move the sample with it. The applied stress and resulting strain are sent to the computer for analyzing. The temperature ramp rate for this test was chosen at 3°C per minute. The maximum test temperature was set at 270°C. The sample dimension used in this test was 60mm long by 10mm wide by 6 mm thick.

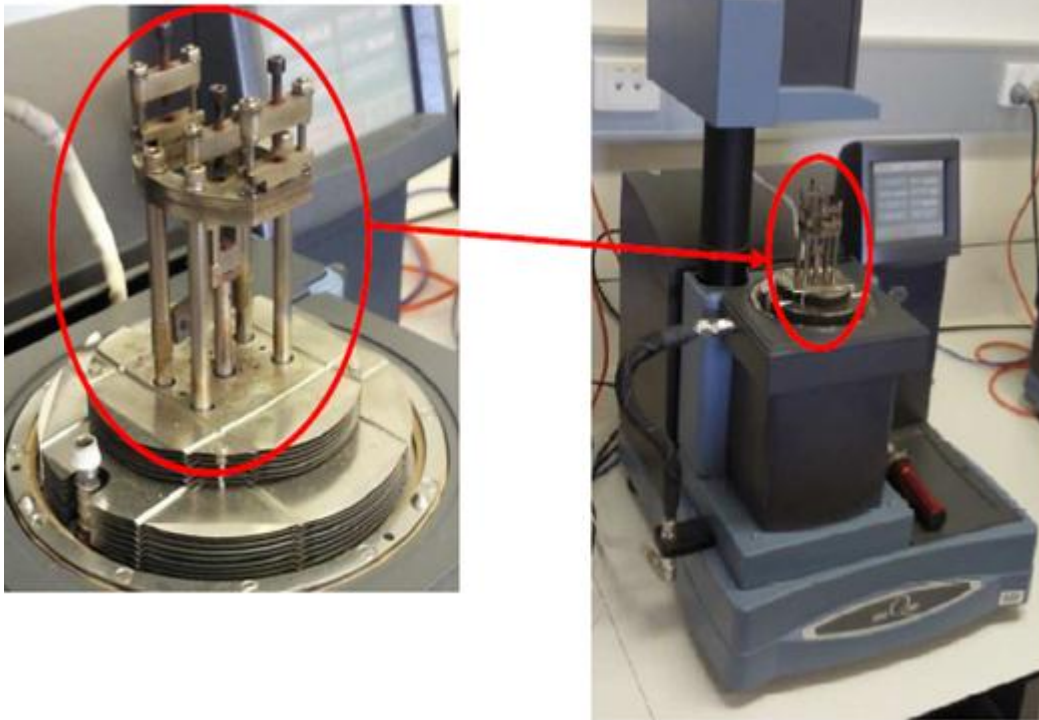


Figure 3.16: DTMA instrument

3.12 Data Retrieved

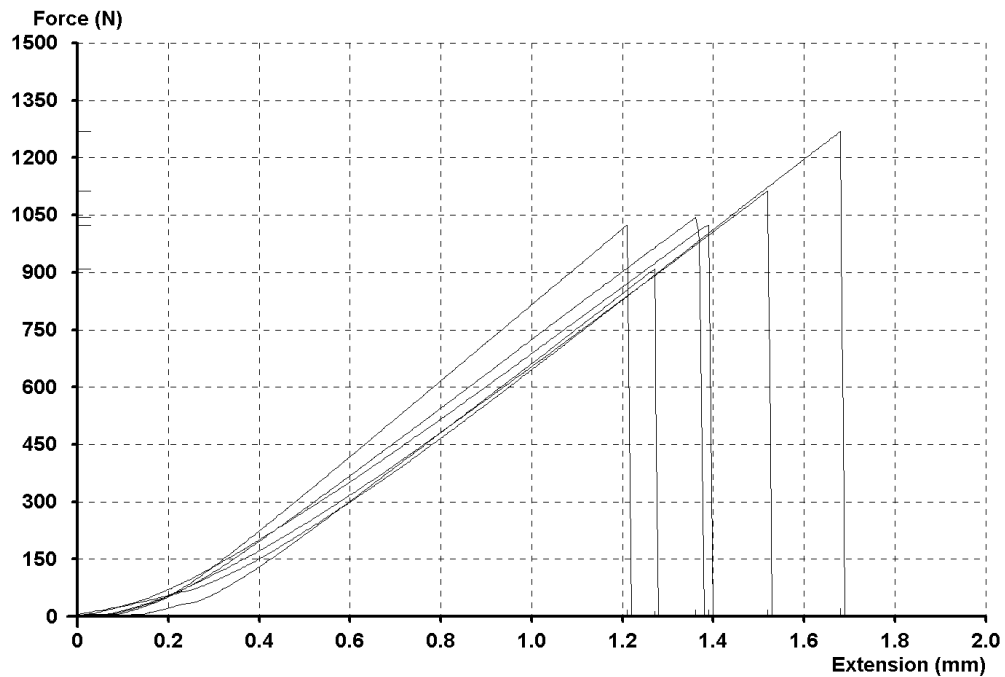
Figure 3.17 Shows the raw data obtained from the flexural testing. The average peak loads from the six specimens were used in the fracture toughness calculation while the flexural strength, flexural strain and young's modulus were obtained from Windows based software

that was connected to the Hounsfield machine.

3-pt Flexural - Rect.Beam/Centre load

Product Code	: phenolic	Load Range	: 2000 N
Batch Reference	:	Extension Range	: 10 mm
Product Description	:	Speed	: 1.0 mm/min
Date	: 7/09/2011	End Point	: 9.0 mm
Operator	:	Support Span Separation	: 80 mm
Temperature	:	Young's Modulus Point	: 1.0 mm
Relative Humidity	:	Preload	: 0.0 N
		Auto Return	: On

Specimen	Thickness mm	Width mm	Modulus @ 1 mm MPa	Flexural Strength MPa	Strain to Failure -
1	33.56	5.63	385.1	24.02	0.0529
2	33.72	5.16	524.4	20.94	0.0383
3	31.91	5.38	480.9	19.89	0.0380
4	33.39	5.21	428.2	23.01	0.0476
5	33.97	4.760	468.7	22.37	0.0443
6	32.70	5.33	494.2	21.98	0.0417
Mean			463.6	22.03	0.0438
Median			474.8	22.17	0.0430
Std. Dev.			49.73	1.469	0.0057
Coe. Var.			10.73	6.67	13.12
Maximum			524.4	24.02	0.0529
Minimum			385.1	19.89	0.0380



- Page 1 -

Figure 3.17: Raw data obtained from the Hounsfield Testing Machine.

Table 3.2 shows the raw data test results which were read off the screen of the LCR meter connected to the parallel plate testing.

Samples cured in oven				
Frequencies	0%	5%	10%	15%
100Hz	0.449	0398	0.3681	0.4518
120Hz	0.3574	0.3447	0.3084	0.3386
1kHz	0.2029	0.1704	0.1577	0.1866
10kHz	0.0759	0.0655	0.0588	0.0685
20kHz	0.067	0.0598	0.0541	0.0613
100kHz	0.0369	0.0343	0.0301	0.0347

Samples cured in microwave				
Frequencies	0%	5%	10%	15%
100Hz	0.3884	0.398	0.4127	0.4064
120Hz	0.2674	0.3447	0.3431	0.356
1kHz	0.1469	0.1704	0.1715	0.1783
10kHz	0.0581	0.0655	0.0673	0.0708
20kHz	0.0542	0.0598	0.0615	0.0641
100kHz	0.0315	0.0343	0.0343	0.0364

Table 3.2: Parallel plate test results

Figure 3.18 Shows the raw data obtained from the Dynamic Thermal Mechanical Analysis test.

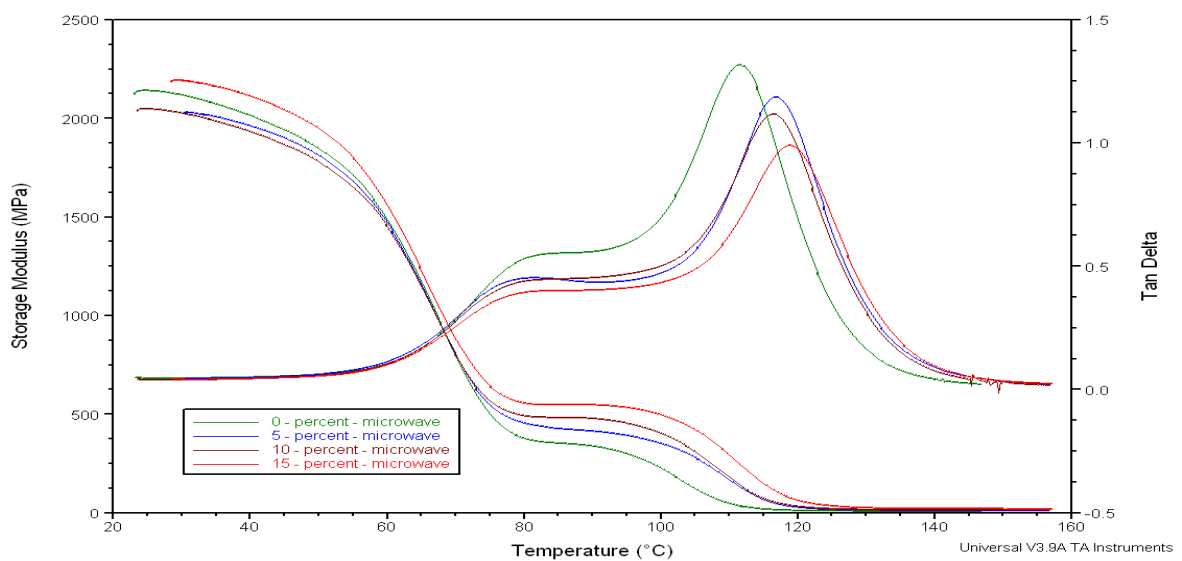


Figure 3.18: Raw data obtained from the Dynamic Thermal Mechanical Analysis Test.

3.13 Improvements in Methodology

There are a number of aspects that can be improved in the methodology. The main improvement one can see is that there is no special mould to take the geometric shape of the specimens that are to be tested for flexural toughness. This could be improved by making a mould more rectangular and to control the width and the height of the cast sample, therefore, no unnecessary disturbance would be done to the test specimen when we tried to cut out a sample from a mould much thicker from the width we want to achieve.

3.14 Conclusion

This chapter described the steps taken in the practical aspects of the project which included making the specimens, post curing and test preparation, testing. Improvements were also suggested at the end of the chapter.

4. Risk Management

4.1 Introduction

In this project, risks are present which have to be identified and minimised. If proper precautions are not taken, the consequences may include serious injury, damage to the environment, and damage to property. This chapter will analyse the potential dangers involved in the project, and steps taken to manage them.

4.2 Identification

In the production and testing of the samples, there are several risks that have to be identified in order to be eliminated or minimised. The materials used to create the samples themselves pose a danger. These materials can cause harm if not handled correctly. The samples require a chemical reaction to occur, hence heat may be involved. If the quantities used are incorrect, the reaction may prove violent or even explosive in an extreme case. The post curing process involves the use of an oven.

The temperature will reach 100° Celsius; this can cause serious injury if negligence occurs.

Finally, the testing of the specimens may cause injury. There are other dangers that are not so obvious that may cause injury or damage as well.

4.3 Preparation

Like any professional workplace, USQ takes measures to prevent injury occurring to people using its facilities; prevention of harm to people and damage to property is an important aspect. Before starting any practical work, a work permit must be granted.

This will outline the work area, equipment, procedures, and special precautions. It may be revoked at any time. As well as a work permit, a material safety data sheet (MSDS) must be read and understood by the student. These provide all precautions to be taken, e.g. personal

protective equipment (PPE), exposure limits, safe handling information, etc. As well, there is first aid information in case of an emergency.

Students are also shown how to proceed when making samples. Correct techniques are demonstrated to eliminate any confusion.

The engineering block is equipped with the necessary facilities to do the project. A ventilation chamber with an exhaust fan is at hand and its use is necessary for the mixing of the samples. The testing machine is fitted with a shield. This will protect from any flying chips resulting from the tensile testing.

4.4 Risks

Any activity that has risks involved has the potential to cause harm. After being identified, the appropriate action can be taken to minimise the likelihood of an accident occurring.

Resin

Hazards

- Hexion 922PAS and PAW will have adverse effects if in contact with eyes.
- Contact with skin will cause irritation and may also have adverse effects.
- Prolonged exposure to fumes will have adverse effects on respiratory system.

Recommendations

- Wear safety glasses.
- Wear rubber gloves.
- Limit exposure time, wear respirator, and open windows.

Accelerator

Hazards

- MEKP corrosive to eyes. Will cause blindness if not treated immediately.
- Corrosive to skin. Will cause burning if not treated immediately.
- Harmful if swallowed.

Recommendations

- Wear safety glasses.
- Wear rubber gloves.
- Do not swallow.

Glass Powder

Hazards

- Adverse effects on respiratory system if inhaled.

Recommendations

- Wear respirator when handling and filing.

Reaction of resin and accelerator

Hazard

- Reaction may be violent if wrong amounts of accelerator used.

Recommendation

- Consult MSDS for recommended amounts before mixing.

Testing

Hazard

- Chip may fly from specimen during testing.

Recommendation

- Close shield on testing machine when testing.

Laboratory Dangers

Hazards

- Risk of trip or slip in lab.
- Spills present on work areas.

Recommendations

- Keep laboratory working area clean and dry.

This information can be tabulated into a risk assessment sheet. It will make it easier to refer to certain aspects of the project to undertake the project safely. Table 4.1 shows the risk assessment sheet for this project.

Table 4.1: Risk Assessment

Description of Hazard	Risk Level	People at risk	Parts of Body	Control Measures
Inhalation of fumes	High	People in room	Respiratory system, Brain	Wear respirator, open windows, turn on exhaust fan, avoid long periods of exposure
Skin contact with resin and catalyst	Medium	Person mixing	Skin	Wear gloves, wear covered shoes, wear long sleeve shirt
Resin and catalyst touching eye	Medium	Person mixing	Eye	Wear safety glasses
Potentially violent chemical reaction	Low	People in room	Body parts exposed to reaction	Wear PPE, mix behind shield
Flying chip from test specimen	Low	People in room	Whole body	Close shield when testing
Trip or slip in lab	Low	People in room	All exposed parts	Keep lab tidy

5. Results and Discussions

5.1 Introduction

This chapter will analyse and discuss the results obtained from the flexural tests carried out. By using fracture mechanics with assumptions of linear elastic fracture mechanics, the fracture toughness was calculated. Comparison of the results to previous works will also be done. This will give an indication of whether the results are practical. Further to this, dielectric constant and loss tangent and DMA analysis were also carried out. This will assist in determining reasons for failure as well as factors that improve fracture toughness.

5.2 Flexural Test

Table 5.1 shows the raw data obtained from the flexural tests. Peak load was the only data obtained from the flexural test that was needed for the calculation of the fracture toughness. From the graphs and excel spread-sheet generated by the Hounsfield testing machine, the peak loads are determined to be the highest point reached during the duration of the testing before it fails. The results of all the testing is given in Appendix A.

Table 5.1: Peak Load

Percentage	0	5	10	15	20	25	30	35
Specimen 1 F_{\max} (N)	54.440	34.617	30.900	31.519	24.675	31.357	27.973	21.592
Specimen 2 F_{\max} (N)	54.440	34.617	30.900	31.519	24.675	31.357	27.973	21.592
Specimen 3 F_{\max} (N)	50.730	46.869	36.271	35.666	31.714	27.013	26.090	21.749
Specimen 4 F_{\max} (N)	54.440	34.617	30.900	31.519	24.675	31.357	27.973	21.592
Specimen 5 F_{\max} (N)	58.313	49.366	38.243	34.025	30.703	29.503	22.396	19.658
Specimen 6 F_{\max} (N)	56.508	45.298	36.001	31.441	30.663	28.652	23.908	22.764
Average	59.942	43.163	37.271	32.292	30.053	30.036	25.078	21.448

5.3 Fracture Toughness

The fracture toughness of the specimens was calculated using the formula,

$$K_c = \frac{F_{max}\sqrt{a}}{BW} f\left(\frac{a}{w}\right)$$

Where $f\left(\frac{a}{w}\right)$ is the geometry correction factor given by:

$$f\left(\frac{a}{w}\right) = 1.99 - 0.41\left(\frac{a}{w}\right) + 18.7\left(\frac{a}{w}\right)^2 - 38.48\left(\frac{a}{w}\right)^3 + 53.85\left(\frac{a}{w}\right)^4$$

Where, F_{max} is the maximum force obtained from the test, B is the thickness of the sample and it is determined by actual measurements of the specimens, W is the width of the sample also determined by actual measurements of the specimens, and a is the total notch length which is 10millimetres.

The fracture toughness of glass powder reinforced vinyl ester resin (VE/Glass Powder) at different percentages by weight was then calculated using the average peak load and is given in Table 5.2. Standard deviation is given in brackets.

Table 5.2: Table of Fracture Toughness results

Percentage	0	5	10	15	20	25	30	35
Fracture Toughness (MPa√m)	56.942	43.163	37.271	32.292	30.053	30.036	25.078	21.448
(Standard Deviation)	3.656	5.523	4.008	2.266	3.688	1.875	2.162	0.938

For visual representation, the results were plotted to provide a better comparison of the fracture toughness calculated. A five precents (5%) marker was included. This allowed unusually higher and lower measurements to be omitted from calculating fracture toughness.

The fracture toughness of varying percentages of VE/Glass Powder post cured in a conventional oven is given in Figure 5.1.

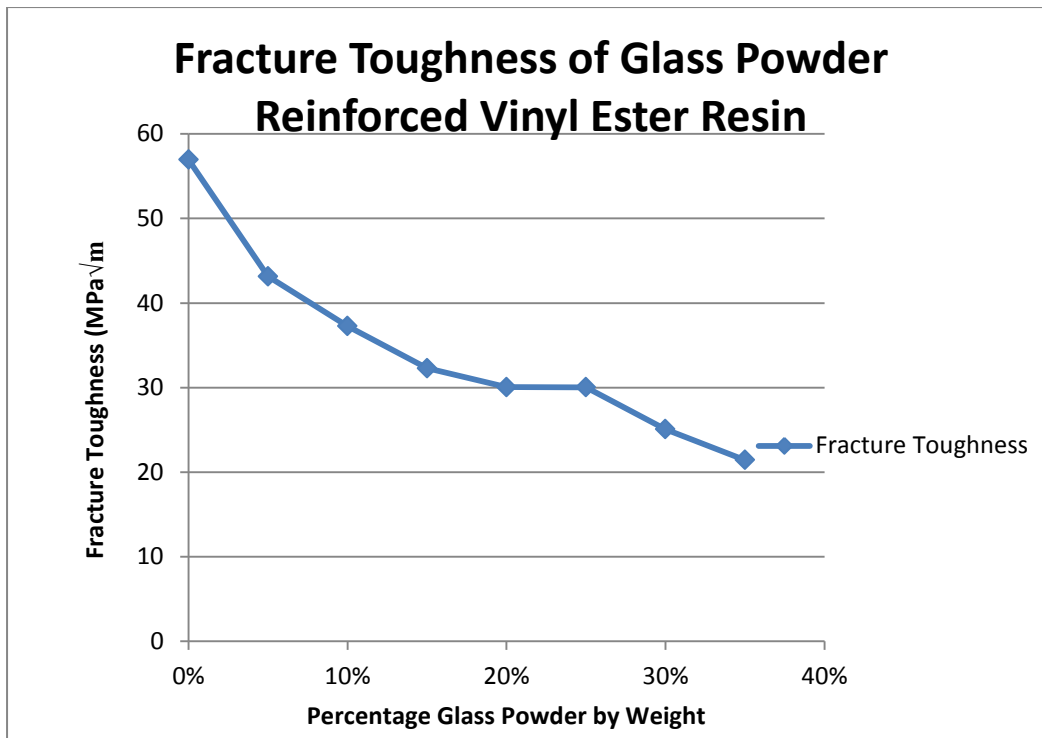


Figure 5.1: Fracture toughness of glass powder reinforced vinyl ester resin.

From Figure 5.1, it can be seen that the fracture toughness of VE/Glass Powder was highest at 0%. This is an unexpected result and would be discussed further in the next chapter. The fracture toughness for the different percentages of glass powder showed a gradual decrease in the fracture toughness. At neat resin (0% glass powder), in this test gave the highest fracture toughness, which was 56.942MPa√m and this may have been caused by the fact that when the specimens are been cut into the required geometry we may have somehow disrupt the bonding process of the composites. Irregularities of the specimen sizes may contribute immensely on the outcome of the result as it goes through the grinding process.

5.4 Comparison to Previous Works

Comparison of the results to previous work is a good indication of the viability of the fracture toughness measurements calculated. A previous study conducted investigated the fracture toughness of phenol formaldehyde composites. Geoffrey Korowa. (2009) used glass powder reinforced vinyl ester resin and also the fracture toughness was determined using a different method (i.e. the short bar method) but similar testing conditions for the studies were

maintained. Table 5.4 shows the fracture toughness of glass powder reinforced vinyl ester resin at varying percentages using short bar method and Table 4.4 shows the fracture toughness of glass powder reinforced vinyl ester resin at varying percentages using flexural test. Figure 5.3 shows a comparison of this and the previous studies results.

Table 5.3: Fracture toughness of glass powder reinforced vinyl ester resin using short bar method.

Percentage	0	5	10	15	20	25	30	35
Fracture Toughness (MPa√m)	31.12	31.71	31.63	38.62	33.49	33.23	31.53	31.61
(Standard Deviation)	3.92	1.06	0.66	0.76	0.88	0.46	0.77	0.85

Table 5.4: Fracture toughness of glass powder reinforced vinyl ester resin using flexural test.

Percentage	0	5	10	15	20	25	30	35
Fracture Toughness (MPa√m)	56.942	43.163	37.271	32.292	30.053	30.036	25.078	21.448
(Standard Deviation)	3.656	5.523	4.008	2.266	3.688	1.875	2.162	0.938

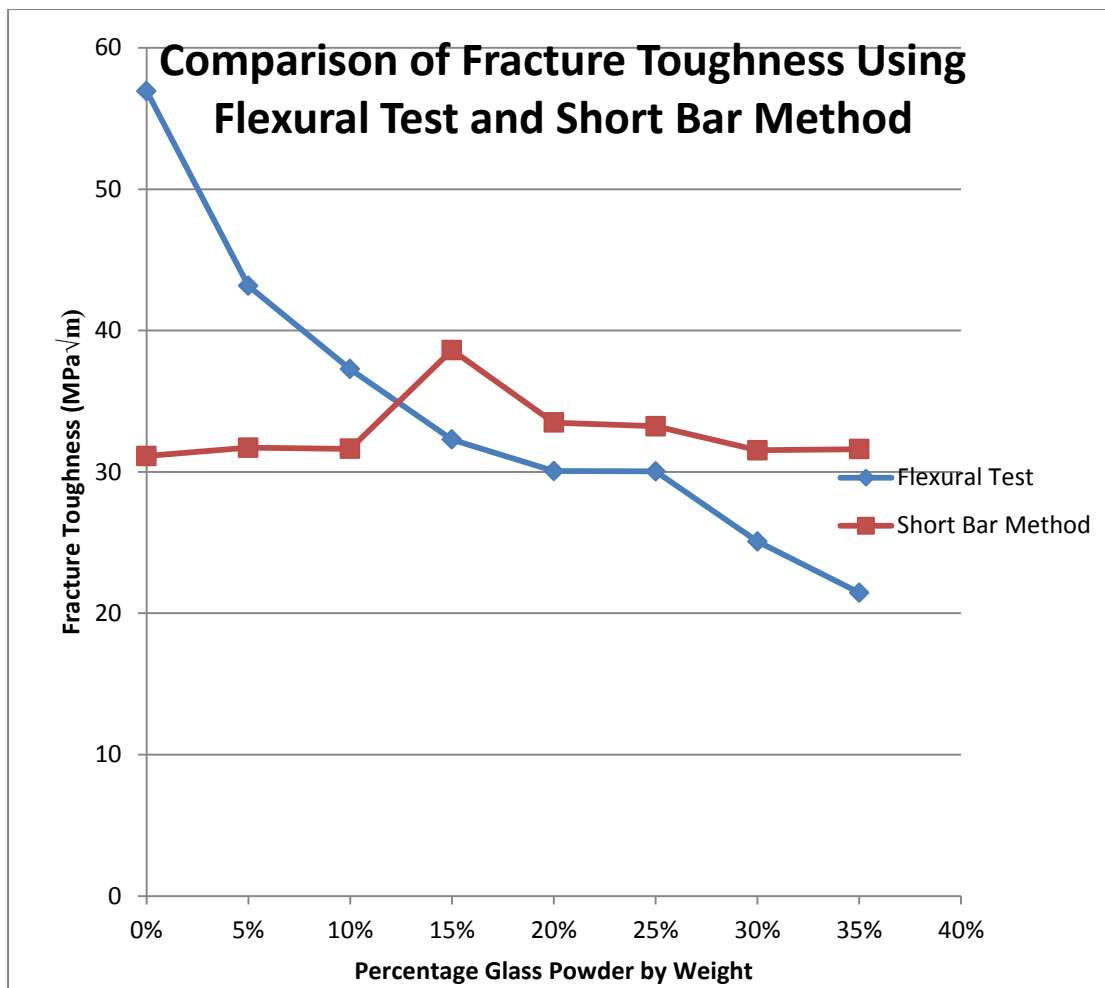


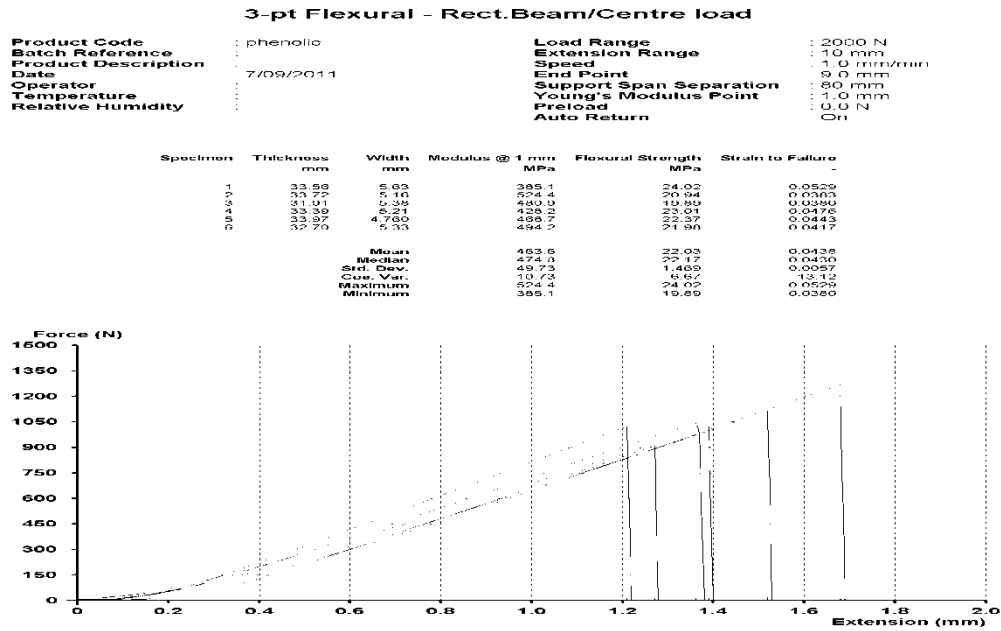
Figure 5.2: Fracture toughness of vinyl ester with varying percentage by weight of glass powder

It can be seen that there is a flaw in the results from this study and it is due to the gross dimensional error from the preparation of the test specimens. The expected result should be the fracture toughness to start low at neat resin before rising to the maximum at 15% by weight before it drops back down and this wasn't the case for this test and if I had the time I would definitely redo the test as a lot of lesson learnt after doing this first test. The main factor that must be taken into consideration is that the specimen's cross sectional area should be consistent throughout and on only than you can be guaranteed of a good result.

5.5 Flexural Strength

In this section flexural strength was obtained using a Windows based software that was connected to the Hounsfield machine which gives the average flexural strength of the six test

sample for each percentage. This test determines the flexural properties of the unreinforced rigid or semi-rigid plastics specimen. Fig 5.3 shows the result obtained from the Hounsfield machine.



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Figure 5.3: Test result obtained from the Hounsfield machine.

Percentage	0	5	10	15	20	25	30	35
Flexural Strength (MPa)	22.03	16.98	14.63	12.5	11.79	11.78	9.78	8.38
(Standard Deviation)	1.469	2.428	1.641	0.838	1.364	0.775	0.855	0.4145

Table 5.5: Table of Flexural Strength results

It is difficult to see the trend in flexural strength from the table; therefore a graph is made as seen in Figure 5.5.

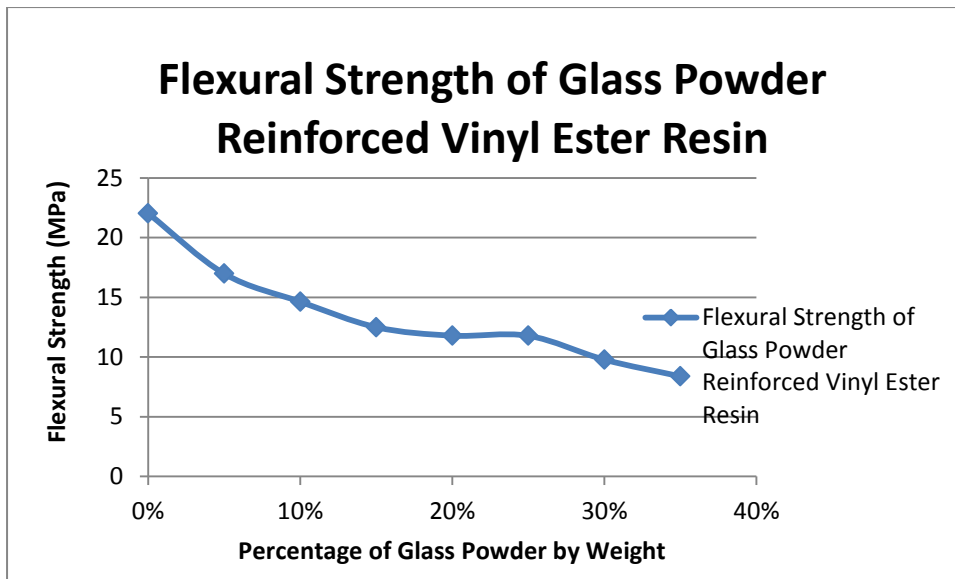


Figure 5.4: Flexural Strength of glass powder reinforced vinyl ester resin.

From Figure 5.4, it can be seen that the flexural strength of VE/Glass Powder was highest at 0%. The flexural strength for the different percentages of glass powder showed a gradual decrease in the fracture toughness. At neat resin (0% glass powder), in this test gave the highest fracture toughness, which was 22.03MPa and this may have been caused by the fact that when the specimens are been cut into the required geometry we may have somehow disrupt the bonding process of the composites. Irregularities of the specimen cross sectional dimension may contribute immensely on the outcome of the result. It is important to maintain a consistent cross sectional dimension throughout the whole dimension of the specimen.

5.6 Flexural Strain

In this section flexural strain was obtained using a Windows based software that was connected to the Hounsfield machine which gives the average flexural strain of the six test sample for each percentage. This test determines the flexural properties of the unreinforced rigid or semi-rigid plastics specimen. Fig 5.5 shows the result obtained from the Hounsfield machine.

Percentage	0	5	10	15	20	25	30	35
Flexural Strain (mm/mm)	0.044	0.0297	0.03	0.0274	0.0329	0.0288	0.0274	0.0192
(Standard Deviation)	0.0057	0.0034	0.0036	0.0026	0.0036	0.0033	0.0042	0.0015

Table 5.6: Table of Flexural Strain results

It is difficult to see the trend in flexural strain from the table; therefore a graph is made as seen in Figure 5.5.

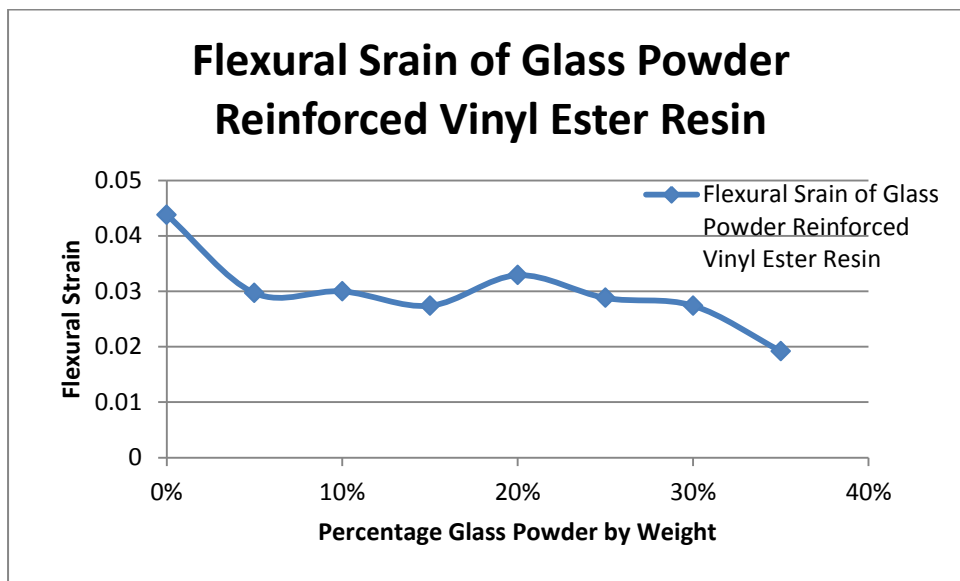


Figure 5.5: Flexural Strain of glass powder reinforced vinyl ester resin.

Figure 5.6 shows the flexural strain of varying percentage by weight of glass powder reinforced vinyl ester composite post-cured in conventional oven. At 0% the maximum flexural strain of 0.044 mm/mm was recorded. For other loadings, the flexural strains varied from 0.0297 to 0.0192 mm/mm, the variation was not too big. The value for the neat resin was 0.044 mm/mm, which was 44% higher than the minimum. It is shown by the graph that the strain value does not change too much but if an application required flexibility 0 % would be the best choice.

The results of my test seemed to be a lot different when compared to previous results. In that the optimum mixture for the strain occurred at a different percentage and for the 0% to record

the maximum strain. Although the strain values were close to previous results for the 5% - 35%, this leads me to believe that they are correct and possibly a different curing procedure would have caused the difference.

5.7 Young’s Modulus

In this section young’s modulus was obtained using a Windows based software that was connected to the Hounsfield machine which gives the average young’s modulus of the six test sample for each percentage. This test determines the young’s modulus of the unreinforced rigid or semi-rigid plastics specimen. Fig 5.3 shows the result obtained from the Hounsfield machine.

Young’s Modulus (MPa)	463.6	266.1	471.5	208.69	336.4	369.5	266.7	0
(Standard Deviation)	49.43	308.3	40.48	294.4	56.5	4093	4.688	0

Table 5.7: Table of Young’s modulus results

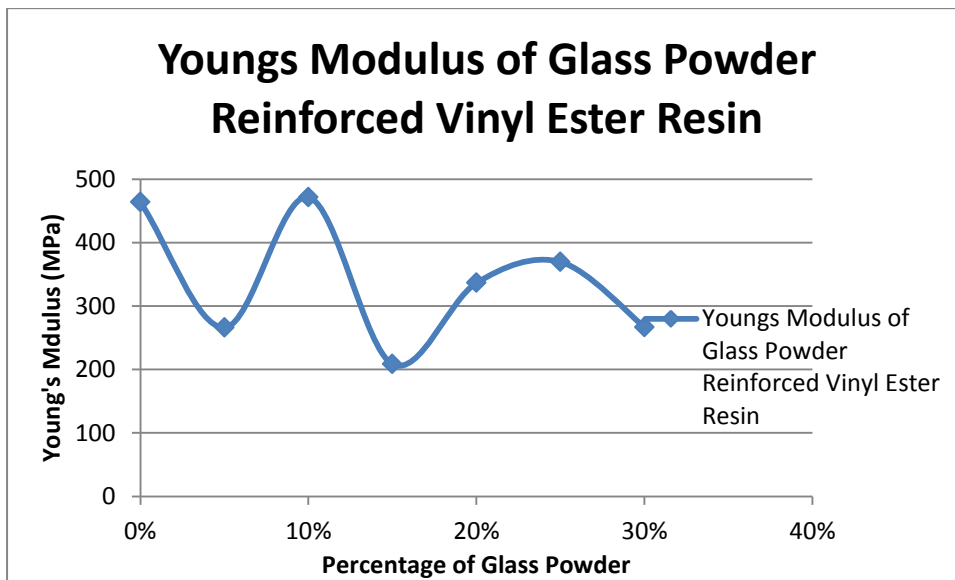


Figure 5.7: Young’s Modulus of glass powder reinforced vinyl ester resin.

Figure 5.7 shows the young’s modulus of varying percentage by weight of glass powder reinforced vinyl ester composite post-cured in conventional oven. At 10% the maximum young’s modulus of 471.5MPa was recorded. For other loadings, the young’s modulus fluctuates and was not consistent to any pattern from previous studies.

5.80 Loss Tangent and Dielectric Constant Test

So what does it mean when your material supplier says his material has a permittivity of 4.5 and a dissipation factor of 0.030? Well, first off, it has been standard practice in our industry to report permittivity and dissipation factor numbers at a test frequency of 1 MHz's. This may or may not help you with your impedance or signal loss calculations. Both permittivity and dissipation factor values are directly related to dielectric material capacitance, which in turn varies with signal frequency. It is very possible that this same material could have a permittivity of 4.3 and a dissipation factor of 0.070 when measured at 2 GHz.

Another factor affecting permittivity and dissipation factor has to do with the ratio within the dielectric material of resin to reinforcement. Standard di-functional epoxy resin has a 1 MHz permittivity of approximately 3.7, while E-glass reinforcement has a typical value of approximately 6.5. As you can see, each style of pre-prep will have slightly different dielectric properties due to the mixing of the resin value with the glass value.

The electrical classification of materials can be identified by measuring the loss tangent. If the loss tangent is greater than 100, the material is classified as conductor. On the other hand, if the loss tangent is less than 0.01, the material is classified as a dielectric which would stop the current to flow. The material fall in between the range is classified as a quasi-conductor.

Loss tangent is proportion to the dissipation in the dielectric. In order to suit for high-speed electronic applications and higher soldering temperature, it requires to decrease the dielectric constant ϵ' and loss tangent and increase the glass-transition temperature (T_g). High loss in transmission would result in reducing signal intensity and increasing thermal noise present (Morin, 2007). The dielectric constant of printed circuit boards (PCB) affects the signal speed of the circuit by the equation:

$$v = \frac{c}{\sqrt{\epsilon_r}} \text{m/s}$$

Where $C = 2.9979 \ 25 \times 10^8$ m/s which is the speed of light.

5.8.1 The loss tangent measurement

Figure 5.8 shows the oven post-cured pure vinyl ester resins have higher value of loss tangent than the microwave post-cured pure vinyl ester resins. Since loss tangent is proportion to the heat dissipation in the dielectric, hence the oven post-cured pure vinyl ester resins are more effective to absorb the microwave energy. At 100 Hz, it has much higher value of loss tangent than the values at other frequencies. This can be explained by considering that the signal frequency is too slow to align the dipoles of the molecules with the change of the signal. On the other hand, loss tangent is also inversely proportional to skin depth, therefore, the distance which microwave can penetrate into the oven cured pure vinyl ester resins is shorter than the distance which the microwave can penetrate into the microwave cured pure vinyl ester resins. Higher value of skip depth means the samples can be heated by the microwave more uniformly.

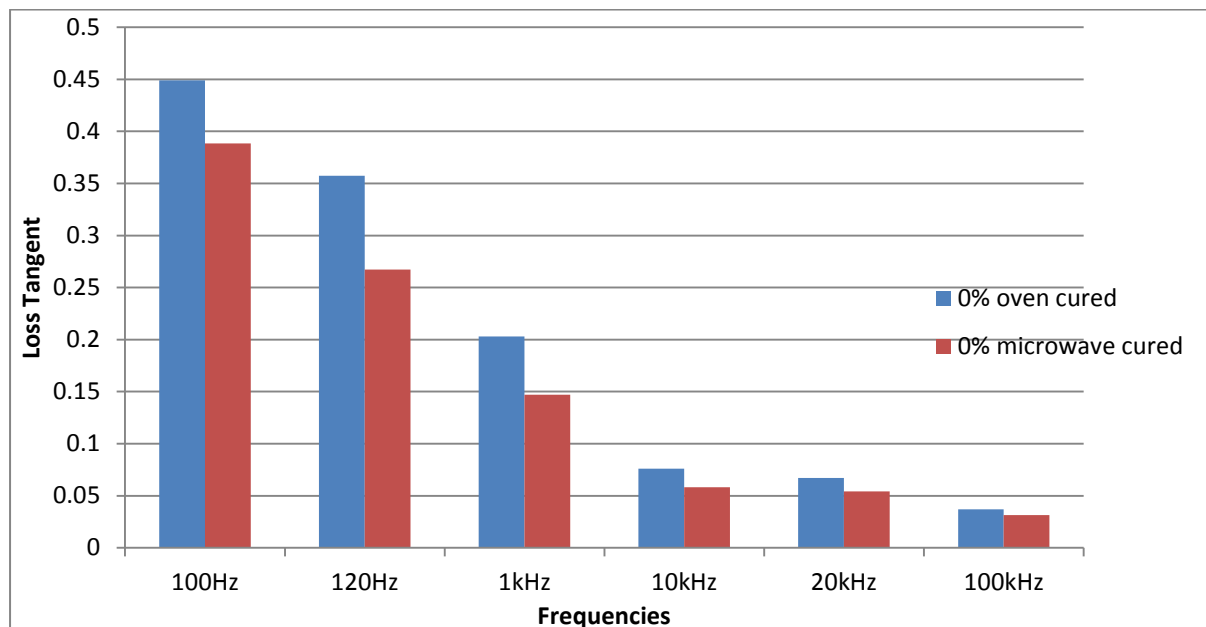


Figure 5.8: Comparison of loss tangent from different curing method of pure vinyl ester resins

Figures 5.9, 5.10 and 5.11 show the different percentage of glass powder reinforced vinyl ester resins exhibit different dielectric behaviours with the pure vinyl ester resin. The microwave cured glass powder reinforced vinyl ester resins have higher values of the loss

tangent than those oven cured samples especially with the 15% sample. The results obtain from glass powder reinforced vinyl ester resins are completely the opposite compared with the results obtained with pure vinyl ester resin samples. As mentioned before, loss tangent is proportional to the heat dissipation in the dielectric, therefore, for glass powder reinforced vinyl ester resins, microwave cured samples are more efficient to absorb the microwave energy. However, as mentioned before that loss tangent is also inversely proportional to skin depth, therefore, the distance which microwave can penetrate into microwave cured glass powder reinforced vinyl ester resin is shorter than the distance which microwave can penetrate into oven cured sawdust reinforced epoxy resin.

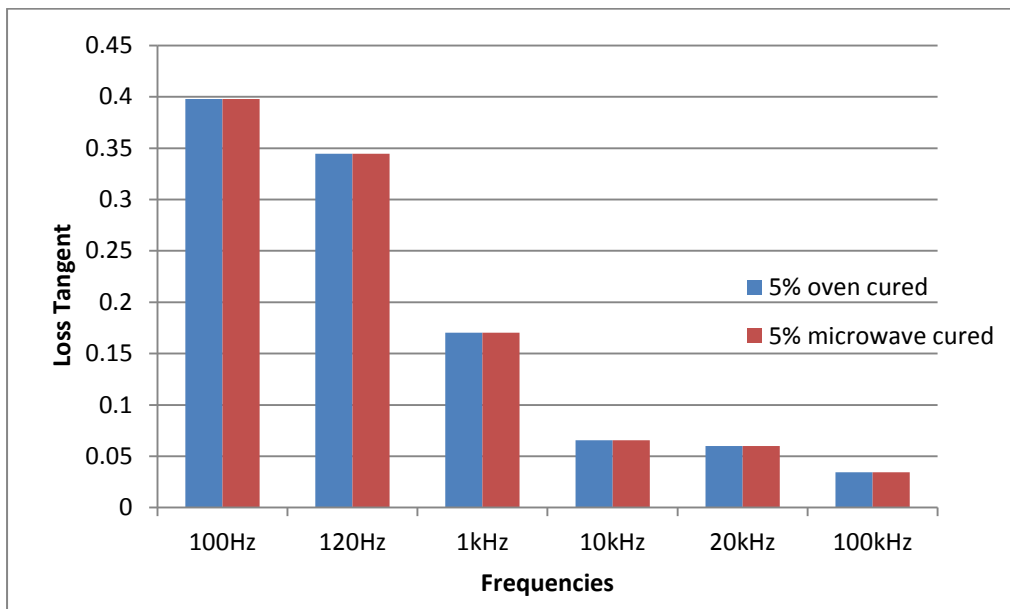


Figure 5.9: Comparison of loss tangent from different curing method of vinyl ester resins reinforced with 5% glass powder

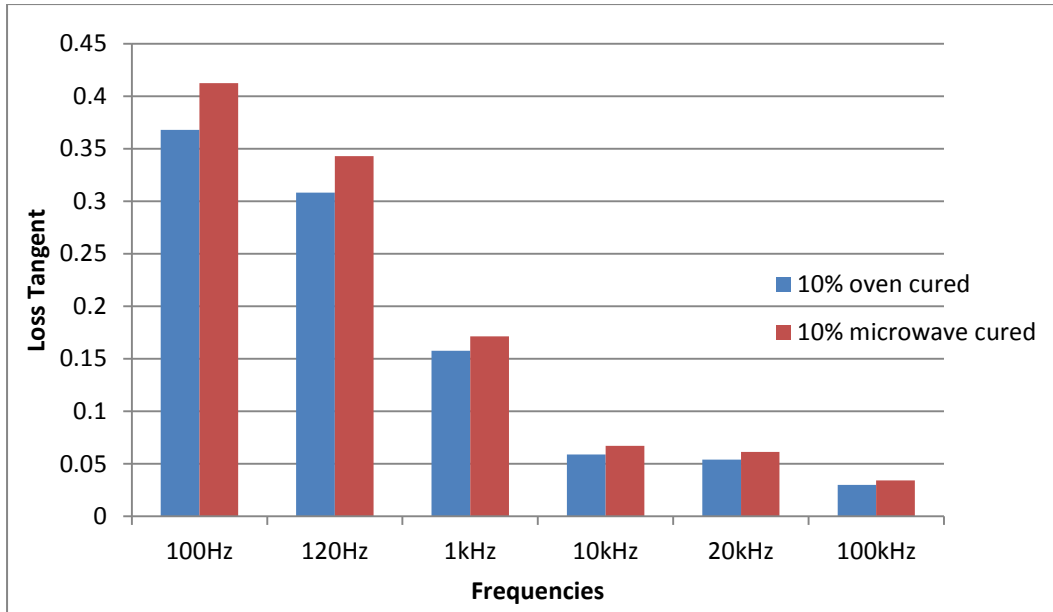


Figure 5.10: Comparison of loss tangent from different curing method of vinyl ester resins reinforced with 10% glass powder

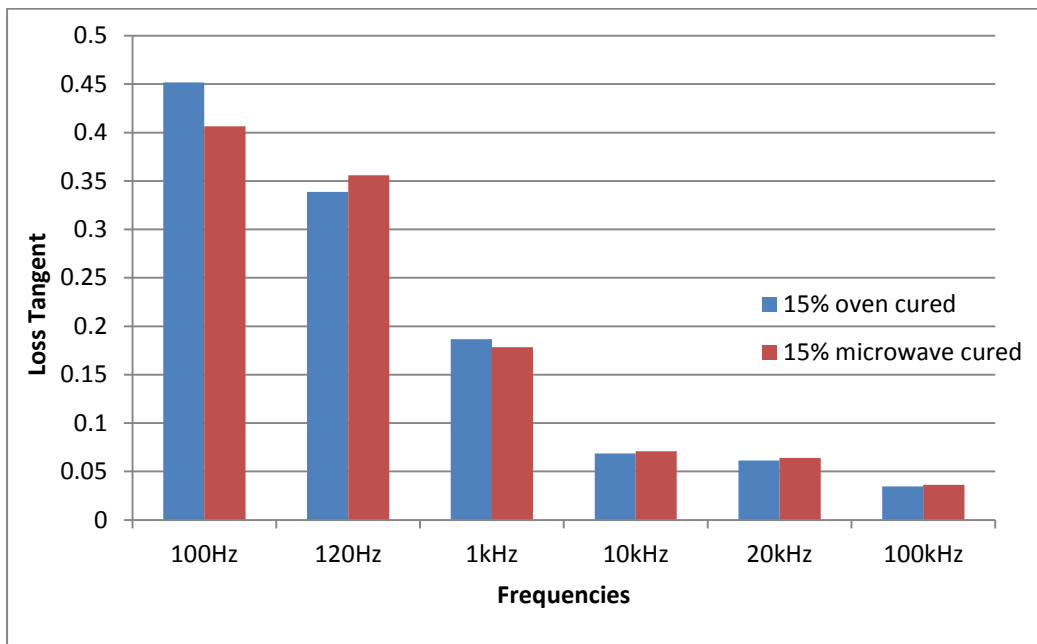


Figure 5.11: Comparison of loss tangent from different curing method of vinyl ester resins reinforced with 15% glass powder

Figure 5.12 shows the loss tangent measured over range of frequencies for the resins mixed with varying percentages of glass powder and cured with microwave. The pure vinyl ester resin is shown in Figure 4.5 to have the lowest value of loss tangent at all frequencies. These results match with the observation in the laboratory during the microwave heating process.

The samples with 15% and 10% sawdust were heated up faster than other samples; hence they needed to be removed from the microwave oven earlier. The results again verified the values of loss tangent were increased by adding glass powder into the vinyl ester resin.

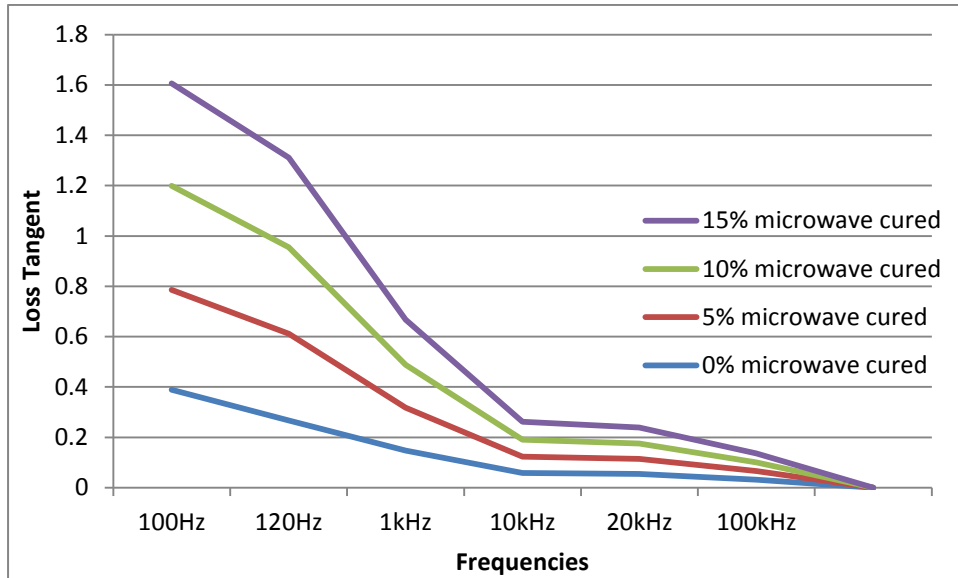


Figure 5.12: Comparison of loss tangent for varying percentage of glass powder cured in microwave

Figure 5.13 shows the loss tangent measured over range of frequencies for the resins mixed with varying percentages of glass powder and post-cured in a conventional oven. The samples post-cured in the microwave oven also gives the same result as for the microwave cured one, the loss tangent was the highest for the 15% sample with the 0% or pure vinyl ester resin recorded the lowest.

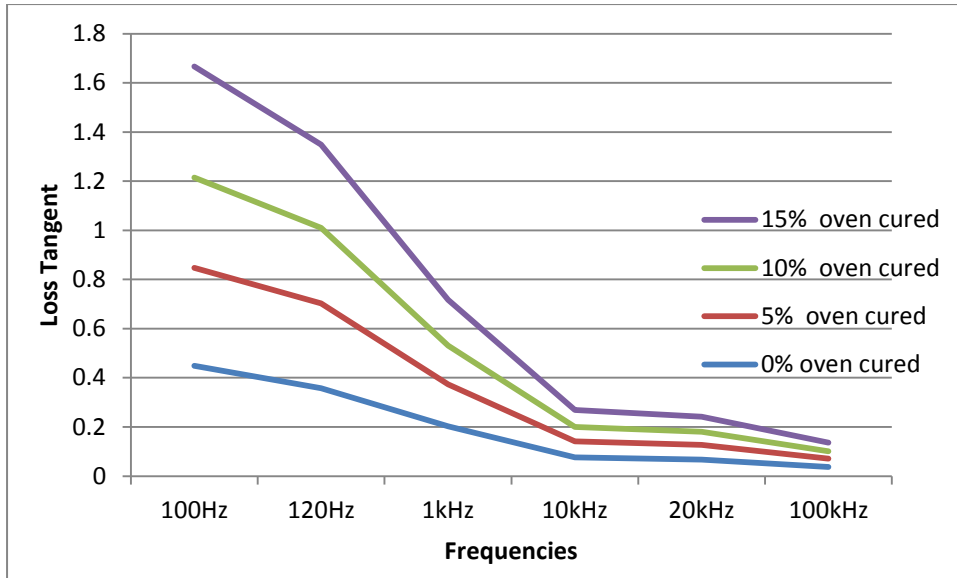


Figure 5.13: Comparison of loss tangent for varying percentage of glass powder cured in oven

5.9 Dynamic Thermal Mechanical Analysis Test Results

5.9.1 Glass Transition Temperature

The Glass Transition Temperature test was done to compare if samples cured by microwave improve the material's Glass Transition Temperature. The results show improvement in the Glass Transition temperature as compared to the specimen cured using the conventional oven.

Specimen Percentage	Conventional Oven	Microwave Oven
0	111.58	121.27
5	117.07	118.40
10	116.45	118.16
15	118.49	114.18

Table 5.8: Tabulated Results for Glass Transition Temperatures

Figures 5.14 and 5.15 shows the glass transition temperatures for microwave cured and oven cured samples were 117.07°C and 118.40°C respectively. These figures also illustrate that the storage moduli of them are 2032MPa and 1728MPa respectively. The loss factor $\tan \delta$ for

microwave cured and oven cured were 1.19 and 1.25 respectively. Since the loss factor $\tan \delta$ is the ratio of loss modulus E'' to storage modulus E' , loss modulus E'' can be easily calculated as E' and $\tan \delta$ are known. The calculated loss modulus E'' for microwave and oven cured samples were 2418.08MPa and 2160MPa respectively. Higher glass transition temperature and high storage modulus mean stiffer material. Higher loss modulus indicates the material is softer and has a higher water content and less degree of cure. The oven cured sample has the highest glass transition temperature and low storage modulus, it is the stiffest sample. Theoretically, for the sample that has a high storage modulus it should have low loss modulus, and this was achieved in this the case. The microwave cured sample has the lowest loss factor $\tan \delta$ which means the sample is stiffest and have highest degree of cure. That is consistent with the conclusion drawn from the glass transient temperature.

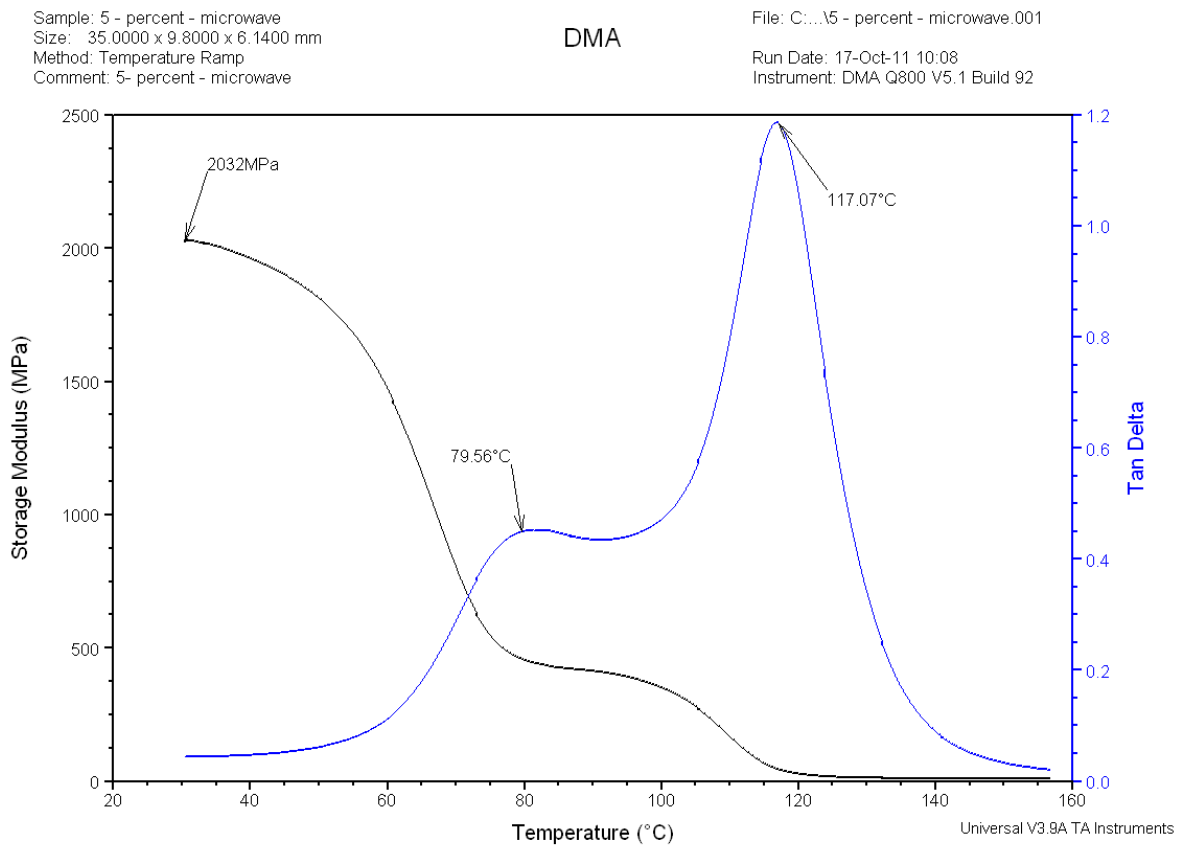


Figure 5.14: DMTA test results for microwave cured vinyl ester resin reinforced with 5% glass powder

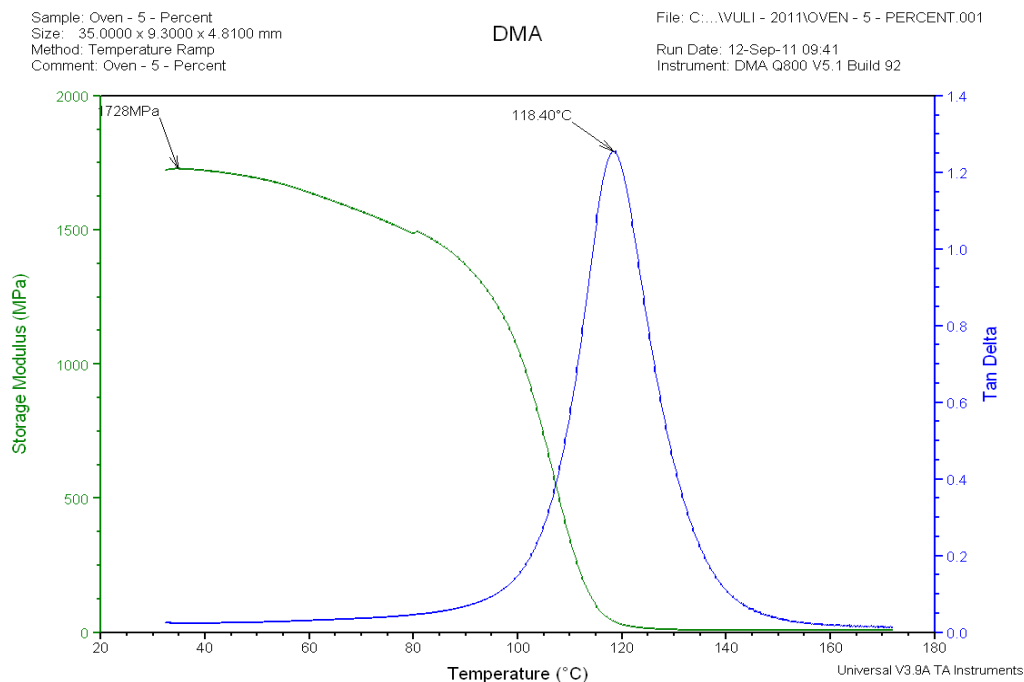


Figure 5.15: DMTA test results for oven cured vinyl ester resin reinforced with 5% glass powder

Figure 5.16 and 5.17 show the glass transition temperatures for microwave cured and oven cured samples were 116.45°C and 118.16°C respectively. These figures also illustrate that the storage moduli of them are 2048MPa and 2035MPa respectively. The loss factor $\tan \delta$ for naturally cured, microwave cured and oven cured were 1.125 and 1.15 respectively. Since the loss factor $\tan \delta$ is the ratio of loss modulus E'' to storage modulus E' , loss modulus E'' can be easily calculated once E' and $\tan \delta$ are known. The calculated loss modulus E'' for microwave cured and oven cured were 2304MPa and 2340.25MPa respectively. Higher glass transition temperature and high storage modulus mean stiffer material. Higher loss modulus indicates the material is softer and has a higher water content and less degree of cure. The oven cured sample has the highest glass transition temperature and low storage modulus, it is the stiffest sample. Theoretically, for the sample that has a high storage modulus it should have low loss modulus, and this was achieved in this the case. The microwave cured sample has the lowest loss factor $\tan \delta$ which means the sample is stiffest and have highest degree of cure. That is consistent with the conclusion drawn from the glass transient temperature.

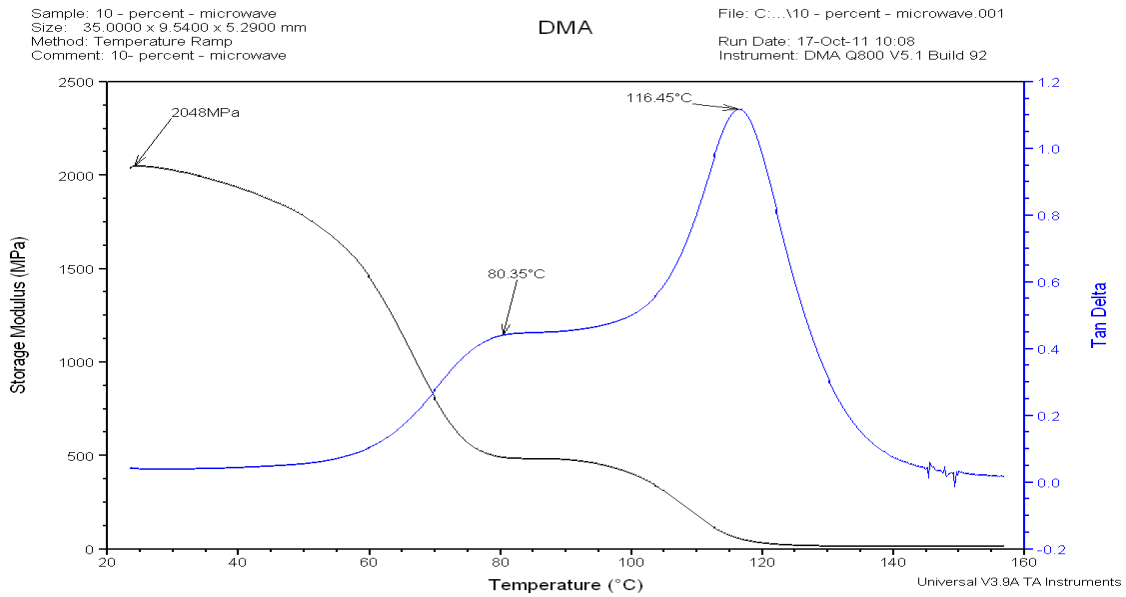


Figure 5.16: DMTA test results for microwave cured vinyl ester resin reinforced with 10% glass powder

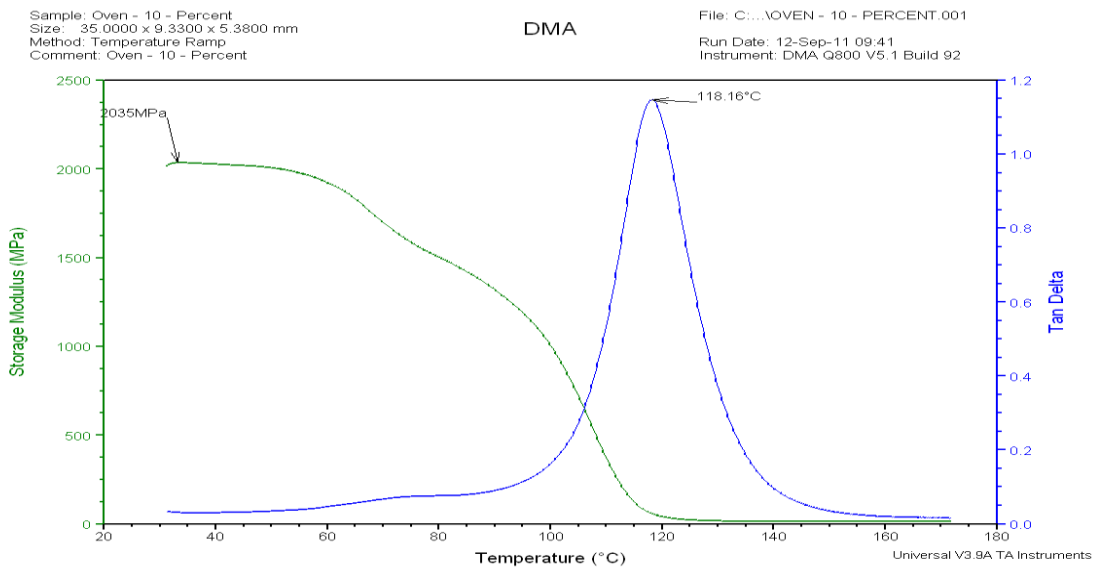


Figure 5.17: DMTA test results for oven cured vinyl ester resin reinforced with 10% glass powder

Figure 5.18 and 5.19 show the glass transition temperatures for microwave cured and oven cured samples were 118.49°C and 118.16°C respectively. These figures also illustrate that the storage moduli of them are 2193MPa and 2035MPa respectively. The loss factor $\tan \delta$ for microwave cured and oven cured were 0.99 and 1.15 respectively. Since the loss factor $\tan \delta$ is the ratio of loss modulus E'' to storage modulus E' , loss modulus E'' can be easily calculated once E' and $\tan \delta$ are known. The calculated loss modulus E'' for microwave

cured and oven cured were 2171.07MPa and 2340.25MPa respectively. Higher glass transition temperature and high storage modulus mean stiffer material. Higher loss modulus indicates the material is softer and has a higher water content and less degree of cure. The microwave cured sample has a slightly higher glass transition temperature and higher storage modulus, it is the stiffest sample. Theoretically, for the sample that has a high storage modulus it should have low loss modulus, and this was achieved in this the case. The microwave cured sample has the lowest loss factor $\tan \delta$ which means the sample is stiffest and have highest degree of cure. That is consistent with the conclusion drawn from the glass transition temperature.

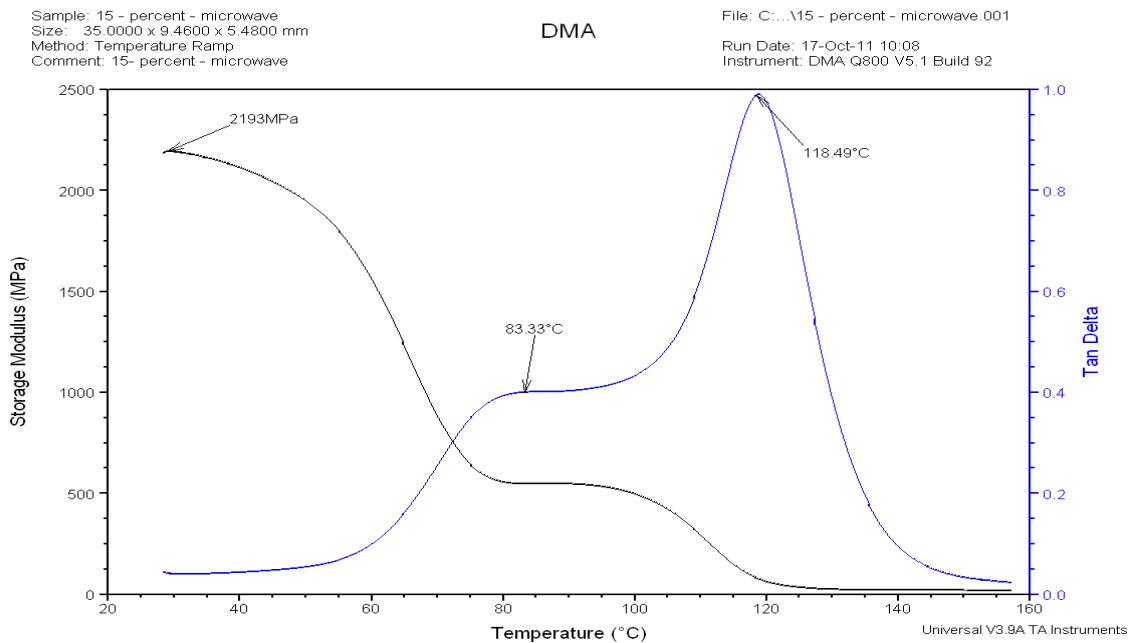


Figure 5.18: DMTA test results for microwave cured vinyl ester resin reinforced with 15% glass powder

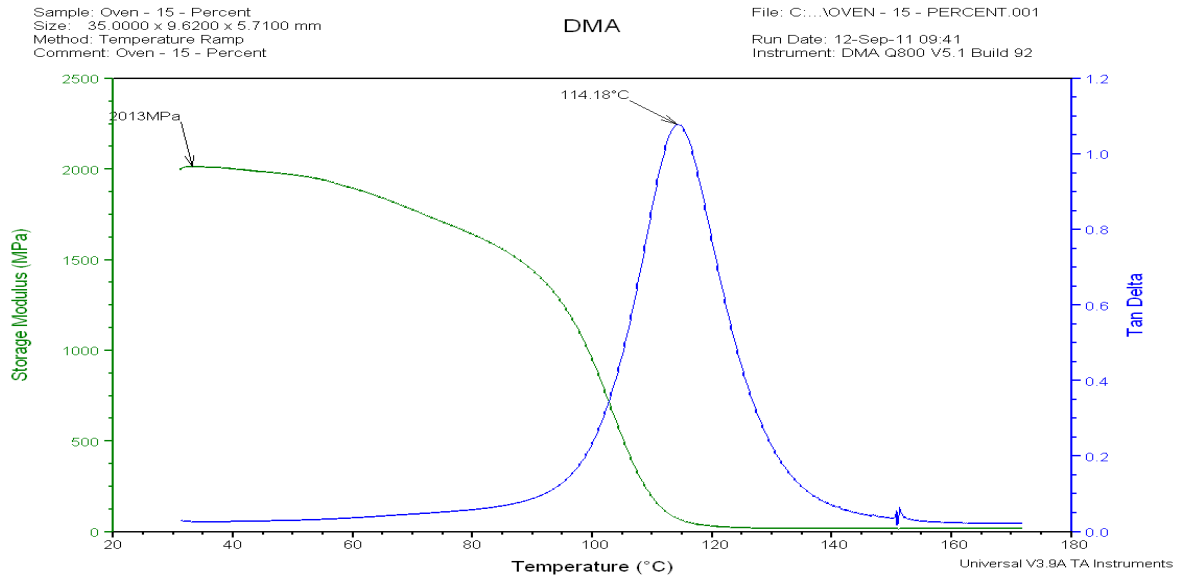


Figure 5.19 DMTA test results for oven cured vinyl resin reinforced with 15% glass powder

Figure 5.20 and 5.21 show the glass transition temperatures for microwave cured and oven cured samples were 111.58°C and 121.27°C respectively. These figures also illustrate that the storage modulus of them are 2141MPa and 1921MPa respectively. The loss factor $\tan \delta$ for naturally cured, microwave cured and oven cured were 1.325 and 1.225 respectively. Since the loss factor $\tan \delta$ is the ratio of loss modulus E'' to storage modulus E' , loss modulus E'' can be easily calculated once E' and $\tan \delta$ are known. The calculated loss modulus E'' for microwave cured and oven cured were 2836.83MPa and 2353.23MPa respectively. Higher glass transition temperature and high storage modulus mean stiffer material. Higher loss modulus indicates the material is softer and has a higher water content and less degree of cure. The microwave cured sample has a slightly higher glass transition temperature and higher storage modulus, it is the stiffest sample. Theoretically, for the sample that has a high storage modulus it should have low loss modulus, and this was achieved in this case. The microwave cured sample has the lowest loss factor $\tan \delta$ which means the sample is stiffest and have highest degree of cure. That is consistent with the conclusion drawn from the glass transition temperature.

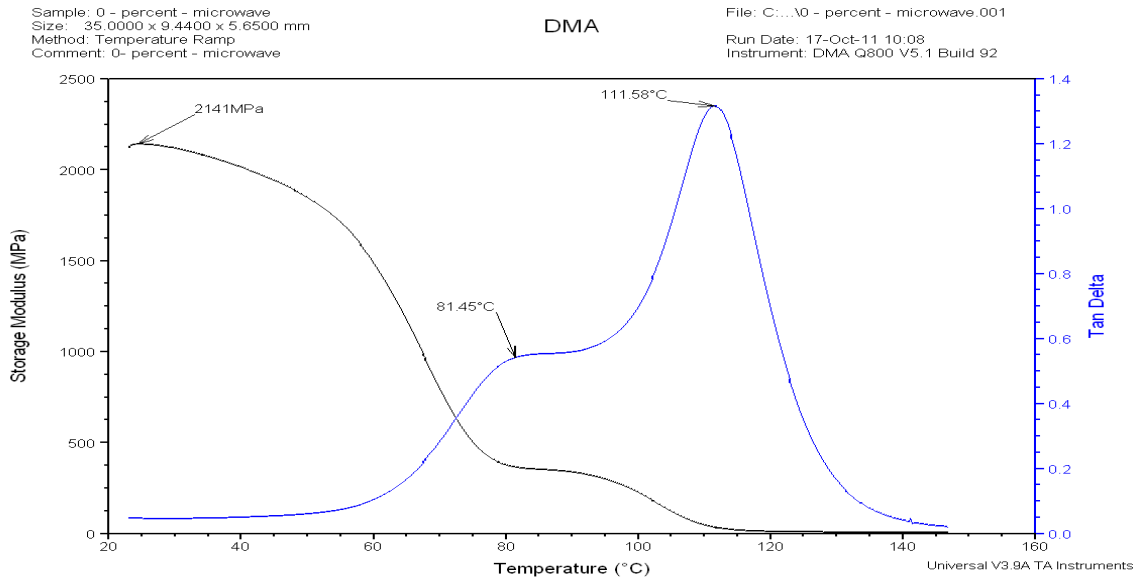


Figure 5.20: DMTA test results for microwave cured pure vinyl ester resin

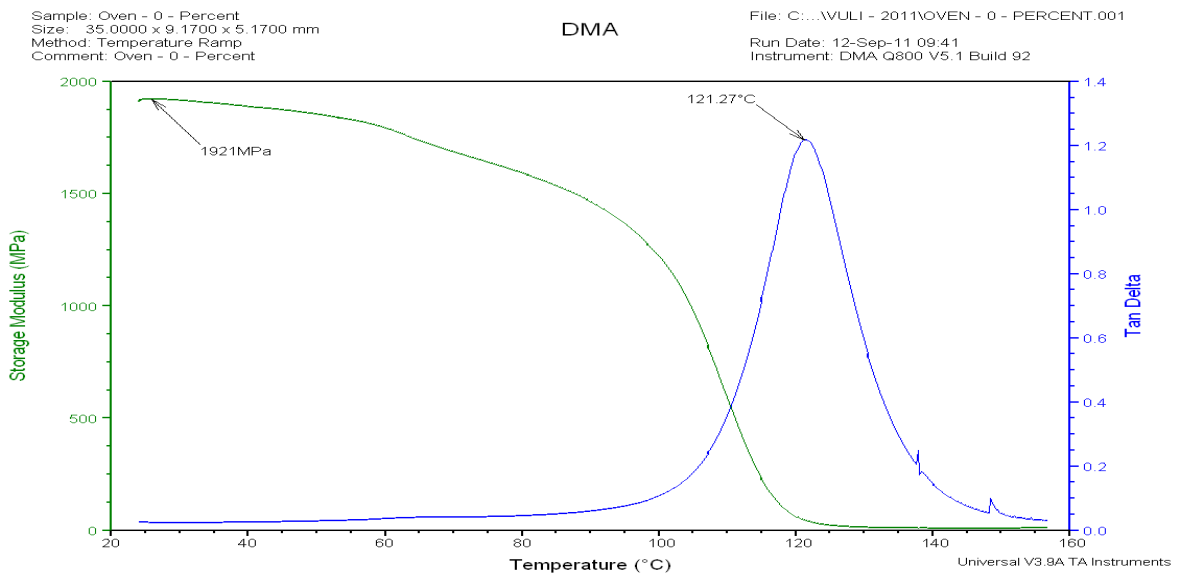


Figure 5.21: DMTA test results for oven cured pure vinyl ester resin

5.9.2 DTMA test summary

Table 5.9 shows the summary of DMA test results. It included the results for maximum loss modulus E'' , maximum storage modulus E' and maximum loss factor $\tan \delta$. It can be found that the oven cured samples generally have the highest glass transient temperature in all of the test samples.

Samples cured in oven				
Percentage of glass powder	Glass transient temperature Tg (°C)	Maximum Storage modulus E' (MPa)	Maximum loss factor tan δ	Maximum loss modulus
0%	121.27	1921	1.225	2353.23
5%	118.16	2035	1.15	2340.25
10%	118.16	2035	1.15	2340.25
15%	118.40	1728	1.25	2160.00
Samples cured in microwave				
Percentage of glass powder	Glass transient temperature Tg (°C)	Maximum Storage modulus E' (MPa)	Maximum loss factor tan δ	Maximum loss modulus
0%	111.58	2141	1.325	2836.83
5%	118.49	2193	0.99	2171.07
10%	116.45	2048	1.125	2304.00
15%	117.07	2032	1.19	2418.08

Table 5.9: DTMA test results

Figure 5.22 shows the glass transition temperature for oven cured and microwave cured vinyl ester resins reinforced with 5%, 10% and 15% glass powder. By comparing the results, it can be found that the oven cured samples in general have the highest glass transition temperature in the group of test samples. The microwave cured samples have the moderate glass transition temperature in the group of test samples. The results mean the oven cured samples can withstand higher temperature and still are capable of retaining reasonable mechanical strength compared to the microwave cured samples. Since all three curves in Figure 4.29 are flat, it can be concluded that the additions of glass powder do not have significant effect on the glass transition temperature of vinyl ester resin.

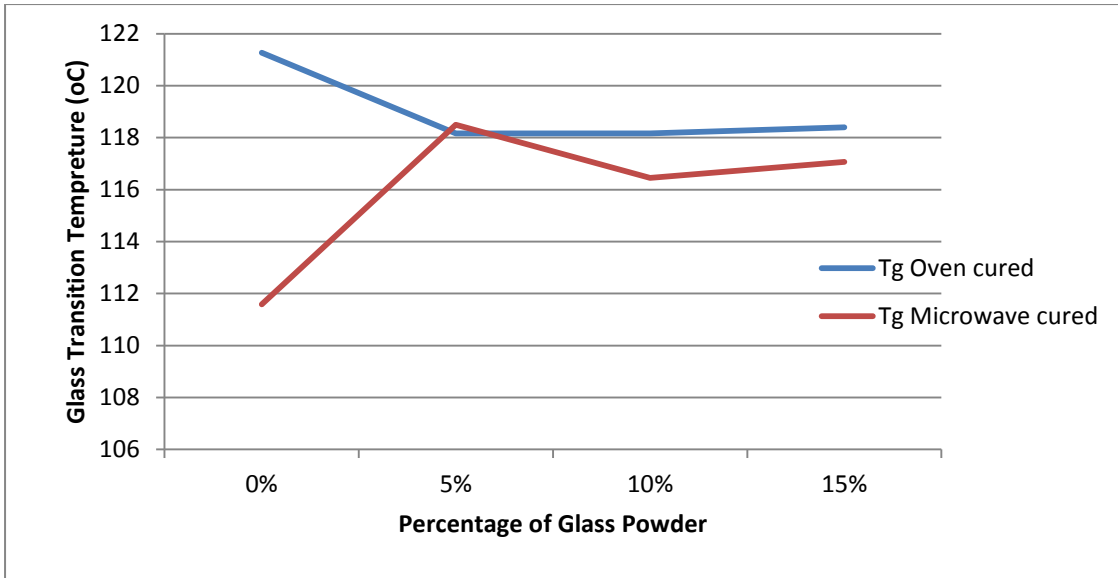


Figure 5.22: Glass transition temperature of vinyl ester resin

Figure 5.23 shows the storage modulus for oven cured and microwave cured vinyl ester resins reinforced with 5%, 10% and 15% of glass powder. As shown in figure 4.30, microwave cured samples have higher storage modulus than oven cured samples.

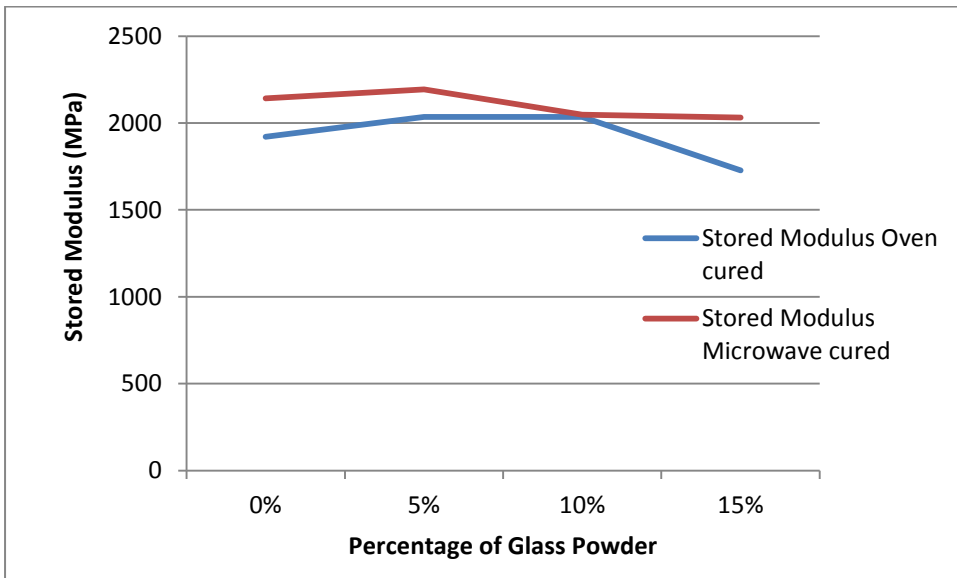


Figure 5.23: Storage modulus of vinyl ester resin

Figure 5.24 shows the loss modulus for oven cured and microwave cured vinyl ester resin reinforced with 5%, 10% and 15% of glass powder. As shown in figure 4.31, oven cured samples have higher loss modulus from 5% and then going down in the 15% while the microwave cured samples achieved the opposite. That means microwave cured samples have less degree of cure compared to oven cured samples.

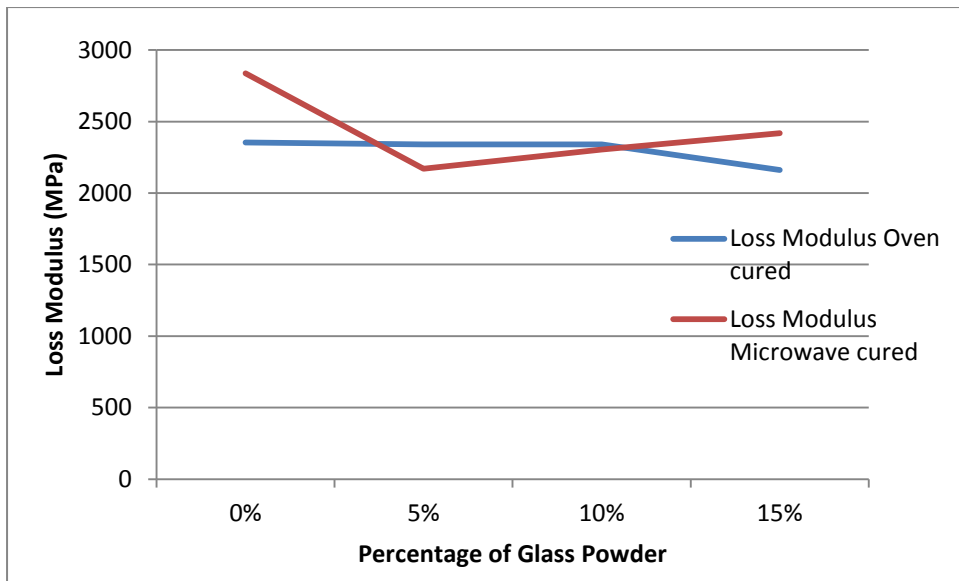


Figure 5.24: Loss modulus of vinyl ester resin

6. Conclusions and Further Work

6.1 Introduction

The results obtained in this research project were only a portion of the work that one originally wished to carry out. This was due to number of constraints, namely time and the difficulty experienced in producing good quality specimens, the need to work in within the schedules of other students and some unforeseen problems. The results, problems and conclusions arising from this project can be used as a basis for those who follow up this work. By studying the results and problems encountered in this project, the follow up should be made easier with better results in a short period of time. In this chapter, the achievements made over the course of this research project will be summarized. Recommendations will also be provided to aid those interested in following up work related to the study of the fracture toughness of VE/Glass Powder composites.

6.2 Achievements

6.2.1 Mould Implementation

Mould used in this research project had been implemented. The appropriate dimensions and how these moulds should be manufactured had been discussed.

6.2.2 Specimen production

The manufacture of specimens had been carried out successfully. The specimens produced were VE/Glass Powder (0 - 35%) for flexural tests and cured under room conditions and post cured in conventional oven. Another sets of specimen were also produced (0 – 15%) for Dielectric test and Dynamic Mechanical Thermal Analysis cured under room conditions and post cured in conventional oven and microwave.

6.2.3 Testing

Testing of the specimens was carried out successfully; but the anticipated result for the flexural test was not achieved due to the gross dimensional error in producing the specimen dimensions.

6.3 Recommendations for Further Work

6.3.1 Specimen Production

Specimen production is very important in any experimental exercise. The problems encountered in the production of specimens in this research project were with the fracture toughness test specimens:

- Time lapse between ambient and oven curing should be strictly been adhered to.
- There should be a special mould to at least to control the width of the specimen during pour to eliminate the gross dimensional error that would give inferior result.

In essence all specimens should be produced in the same way and under the same conditions. One is too wary of the effect on specimen characteristics of placing three specimens in the oven for curing and placing six specimens.

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Appendix A

PROJECT SPECIFICATION

University of Southern Queensland
Faculty of Engineering and Surveying

ENG 4111/4112 Research Project PROJECT SPECIFICATION

Topic: **Fracture toughness of glass powder reinforced epoxy composites using short bar tests post-cured in microwaves**

For: Marau Vuli Mautoga- 00050003387

Supervisor: Dr. Harry Ku
Co-Supervisor:

Sponsorship: Faculty of Engineering and Surveying

Enrolment: ENG 4111- S1, D, 2011
ENG 4112- S2, D, 2011

Project Synopsis:

Composites are being increasingly used in a wide range of structures such as aerospace, marine, transportation and civil engineering. Applications in aerospace, marine and transport are very much performance driven while civil engineering applications are largely cost driven. In order to reduce the cost of composites a wide range of fillers are being used. In this project, sawdust will be used as fillers. These fillers do not only reduce the cost of the composites but also have a significant influence on the final structural properties. This project involves the production of the resin specimens with different percentage by weight of fillers. After preliminary curing, the specimens will be post-cured in ovens.

Post-curing in ovens: 16 hours in 35°C;
16 hours in 50°C;

Fracture toughness tests will be used to evaluate its fracture toughness. The findings will have to be analysed in detail in order to establish behaviour trends and formulas that can be used for theoretical prediction of filled polymer behaviour.

Program: Issue A, 23/Mar/2011

- Review composites material (especially phenolic resins) uses, properties and synthesis

- Design the manufacture process of phenolic composites by different filler sizes and different percentage weights of fillers
- Casting specimens for tensile testing
- Doing tensile test and work out the tensile strength, yield strength and Young's modulus
- Compare and analysis the results and then draw a conclusion

Timelines:

1. Literature reviews

Begin : 14-Mar-2011
 Completion : 29-Mar-2011
 Approx. Hours : 50 hours

2. Familiarization of working environment and equipments.

Begin : 07-Mar-2011
 Completion : 11-Mar-2011
 Approx. Hours : 5 hours

3. Design of manufacture process of a cast/mould for tensile tests.

Begin : 23-Mar-2011
 Completion : 25-Mar-2011
 Approx. Hours : 20 hours

4. Casting Components.

Begin : 30-Mar-2011
 Completion : 26-Apr-2011
 Approx. Hours : 15 hours

5. Testing Methods and examination of specimens.

Begin : 27-Apr-2011
 Completion : 10-May-2011
 Approx. Hours : 80 hours

6. Analysis of results.

Begin : 11-June-2011
 Completion : 30-June-2011
 Approx. Hours : 50 hours

7. Draw up conclusions and discussion about results with supervisor.

Begin : 18-July-2011
Completion : 29-July-2011
Approx. Hours : 40 hours

8. Discussion for the thesis outline with supervisors.

Begin : 28-July-2011
Completion : 24-Aug-2011
Approx. Hours : 10 hours

9. Thesis initial drafting – each chapter in draft form and shown to supervisors.

Begin : 24-August 2011
Completion : 28-Sep-2011
Approx. Hours : 60 hours

10. Final draft of thesis, to incorporate modifications suggested by supervisor.

Begin : 29-Sep-2011
Completion : 07-Oct-2011
Approx. Hours : 20 hours

11. Complete the thesis in requested format.

Begin : 08-Oct-2011
Completion : 27-Oct-2011
Approx. Hours : 20 hours

AGREED:

_____ (Student)

_____ (Supervisor)

(Date) ___/___/___

(Date) ___/___/___

Appendix B

Operating Procedures for Hounsfield Testing Machine

Operating Procedure for Hounsfield testing machine Testing of materials flexural properties of phenolic resin

- A. Start-up procedure for Hounsfield Testing machine
1. Login the computer
 2. Press the ON button on the machine and make sure it is connected to the computer. (Match the COM port if necessary)
 3. Click the shortcut “QMAT” on the desktop > “QMatTestzone”
 4. “File” > “Open Test Method” > 3-PT Flexural – Rect. Beam-Centre Load
 5. Under “Vendor product” choose “phenolic”
- B. Start the test.
- Inside the software
 - >Sample Label: (enter your sample name)
 - >Thickness: (enter the value)
 - >Width: (enter the value)
 - Click > “OK”
- Do STEP C first
- Then, click “Test specimen x” to start the test
Click “Abort Test” after the specimen break
- C. Machine Set-up
1. Put the fixture in position.
 2. Press the button next to the “Test” button on the machine panel. (it will be flashing)
 3. Place the specimen on the fixture
 4. Adjust \uparrow \downarrow until the specimen is just fixed
 5. Press “F1” to set zero force ; Press “F2” to set zero extension
- D. Results saving (recommend to do it after every testing)
1. Click on the “Result Page” icon after your test has finished
 2. Click “Option” > “Export Data”
 3. Check all the six boxes in the export contents
Graphic format: Bitmap
Delimile : Tab
Destination: File
 4. Click “OK”
 5. The first file will be saving as “.raw” , it can be open with Microsoft Word
 6. The second file will be saving as “.bmp”
 7. Click “copy result” icon
Destination: Excel
Then save it to your folder
 8. Click “print results” icon to save all the data in report format.
 9. Finished

E. Finished one sample

1. Click the “Measure” icon to start testing another specimen.

OR

2. Start new batch

Appendix C

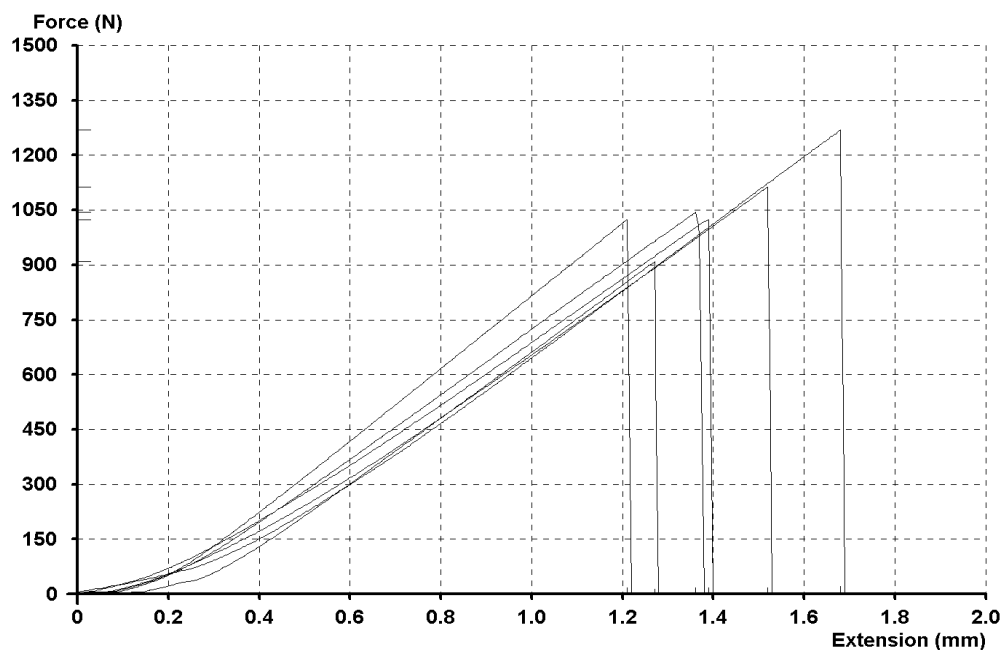
Flexural Testing Raw Data

0% by weight of vinyl ester resin

3-pt Flexural - Rect.Beam/Centre load

Product Code	: phenolic	Load Range	: 2000 N
Batch Reference	:	Extension Range	: 10 mm
Product Description	:	Speed	: 1.0 mm/min
Date	: 7/09/2011	End Point	: 9.0 mm
Operator	:	Support Span Separation	: 80 mm
Temperature	:	Young's Modulus Point	: 1.0 mm
Relative Humidity	:	Preload	: 0.0 N
		Auto Return	: On

Specimen	Thickness mm	Width mm	Modulus @ 1 mm MPa	Flexural Strength MPa	Strain to Failure -
1	33.56	5.63	385.1	24.02	0.0529
2	33.72	5.16	524.4	20.94	0.0383
3	31.91	5.38	480.9	19.89	0.0380
4	33.39	5.21	428.2	23.01	0.0476
5	33.97	4.760	468.7	22.37	0.0443
6	32.70	5.33	494.2	21.98	0.0417
	Mean		463.6	22.03	0.0438
	Median		474.8	22.17	0.0430
	Std. Dev.		49.73	1.469	0.0057
	Coe. Var.		10.73	6.67	13.12
	Maximum		524.4	24.02	0.0529
	Minimum		385.1	19.89	0.0380

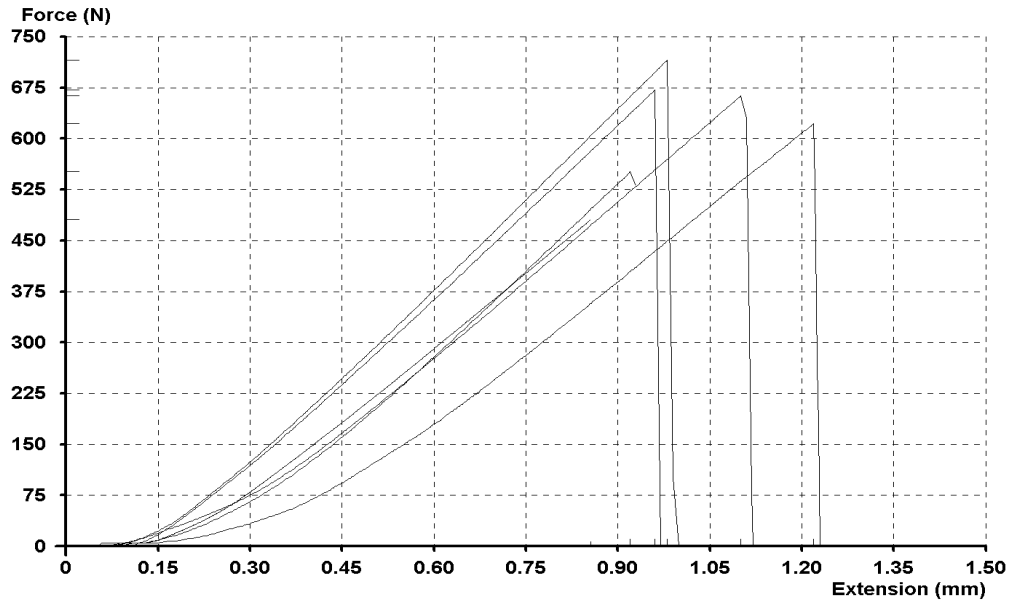


5% by weight of vinyl ester resin

3-pt Flexural - Rect.Beam/Centre load

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Batch Reference	:	Extension Range	: 10 mm
Product Description	:	Speed	: 1.0 mm/min
Date	: 7/09/2011	End Point	: 9.0 mm
Operator	:	Support Span Separation	: 80 mm
Temperature	:	Young's Modulus Point	: 1.0 mm
Relative Humidity	:	Preload	: 0.0 N
		Auto Return	: On

Specimen	Thickness mm	Width mm	Modulus @ 1 mm MPa	Flexural Strength MPa	Strain to Failure -
1	31.17	4.290	571.4	19.09	0.0321
2	33.39	4.440	-	13.36	0.0288
3	29.92	4.460	491.9	18.71	0.0342
4	32.60	4.370	0.6	18.49	0.0300
5	30.05	4.410	-	14.50	0.0241
6	32.10	4.410	0.6	17.73	0.0289
Mean			266.1	16.98	0.0297
Median			246.3	18.11	0.0294
Std. Dev.			308.3	2.428	0.0034
Coe. Var.			115.9	14.30	11.57
Maximum			571.4	19.09	0.0342
Minimum			0.6	13.36	0.0241

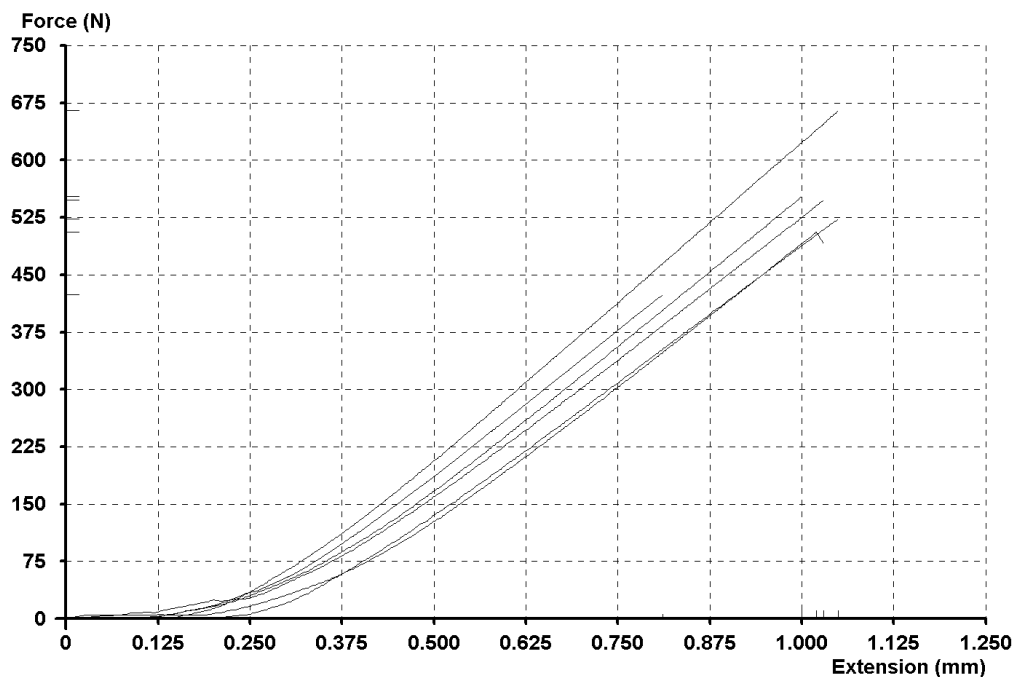


10% by weight of vinyl ester resin

3-pt Flexural - Rect.Beam/Centre load

Product Code	: phenolic	Load Range	: 2000 N
Batch Reference	:	Extension Range	: 10 mm
Product Description	:	Speed	: 1.0 mm/min
Date	: 7/09/2011	End Point	: 9.0 mm
Operator	:	Support Span Separation	: 80 mm
Temperature	:	Young's Modulus Point	: 1.0 mm
Relative Humidity	:	Preload	: 0.0 N
		Auto Return	: On

Specimen	Thickness mm	Width mm	Modulus @ 1 mm MPa	Flexural Strength MPa	Strain to Failure -
vuli-10%	33.41	3.860	430.5	14.56	0.0329
2	30.16	4.540	-	12.31	0.0229
3	32.25	4.500	464.8	14.17	0.0302
4	32.08	4.450	538.9	17.41	0.0316
5	32.33	4.210	468.7	14.93	0.0312
6	32.50	3.990	454.6	14.41	0.0311
	Mean		471.5	14.63	0.0300
	Median		464.8	14.49	0.0311
	Std. Dev.		40.48	1.641	0.0036
	Coe. Var.		8.59	11.22	11.88
	Maximum		538.9	17.41	0.0329
	Minimum		430.5	12.31	0.0229



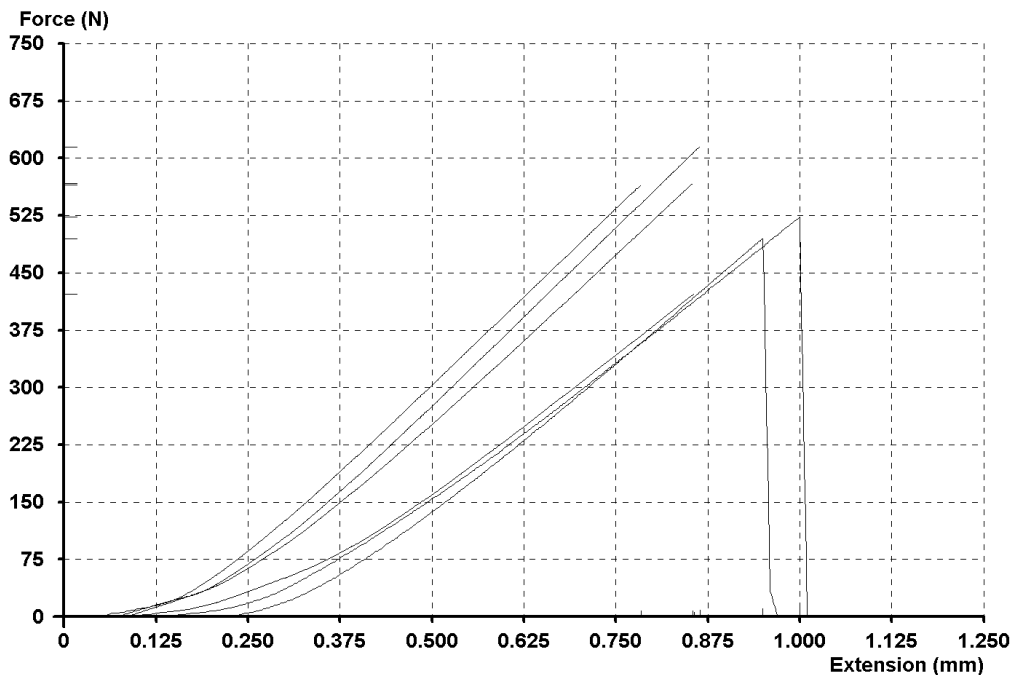
- Page 1 -

15% by weight of vinyl ester resin

3-pt Flexural - Rect.Beam/Centre load

Product Code	: phenolic	Load Range	: 2000 N
Batch Reference	:	Extension Range	: 10 mm
Product Description	:	Speed	: 1.0 mm/min
Date	: 7/09/2011	End Point	: 9.0 mm
Operator	:	Support Span Separation	: 80 mm
Temperature	:	Young's Modulus Point	: 1.0 mm
Relative Humidity	:	Preload	: 0.0 N
		Auto Return	: On

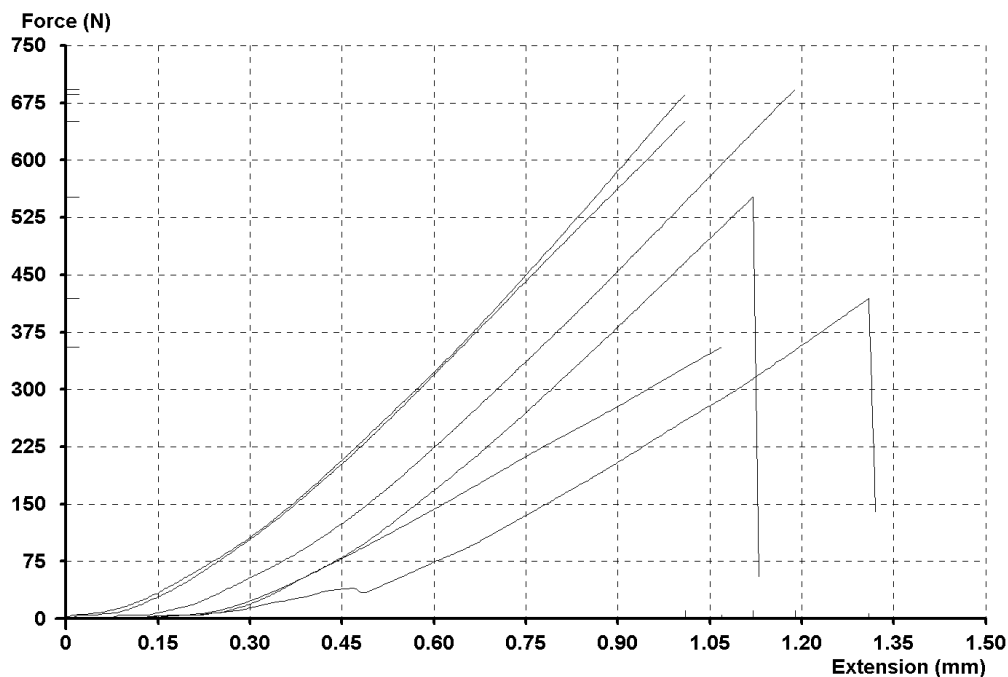
Specimen	Thickness mm	Width mm	Modulus @ 1 mm MPa	Flexural Strength MPa	Strain to Failure -
1	30.17	4.920	-	11.32	0.0242
2	34.30	4.800	-	12.04	0.0275
3	34.21	4.620	-	13.64	0.0277
4	32.57	4.400	0.4	12.73	0.0290
5	33.36	4.290	416.8	13.14	0.0313
6	33.64	4.950	-	12.10	0.0247
Mean			208.6	12.50	0.0274
Median			208.6	12.41	0.0276
Std. Dev.			294.4	0.838	0.0026
Coe. Var.			141.1	6.71	9.63
Maximum			416.8	13.64	0.0313
Minimum			0.4	11.32	0.0242



3-pt Flexural - Rect.Beam/Centre load

Product Code	: phenolic	Load Range	: 2000 N
Batch Reference	:	Extension Range	: 10 mm
Product Description	:	Speed	: 1.0 mm/min
Date	: 7/09/2011	End Point	: 9.0 mm
Operator	:	Support Span Separation	: 80 mm
Temperature	:	Young's Modulus Point	: 1.0 mm
Relative Humidity	:	Preload	: 0.0 N
		Auto Return	: On

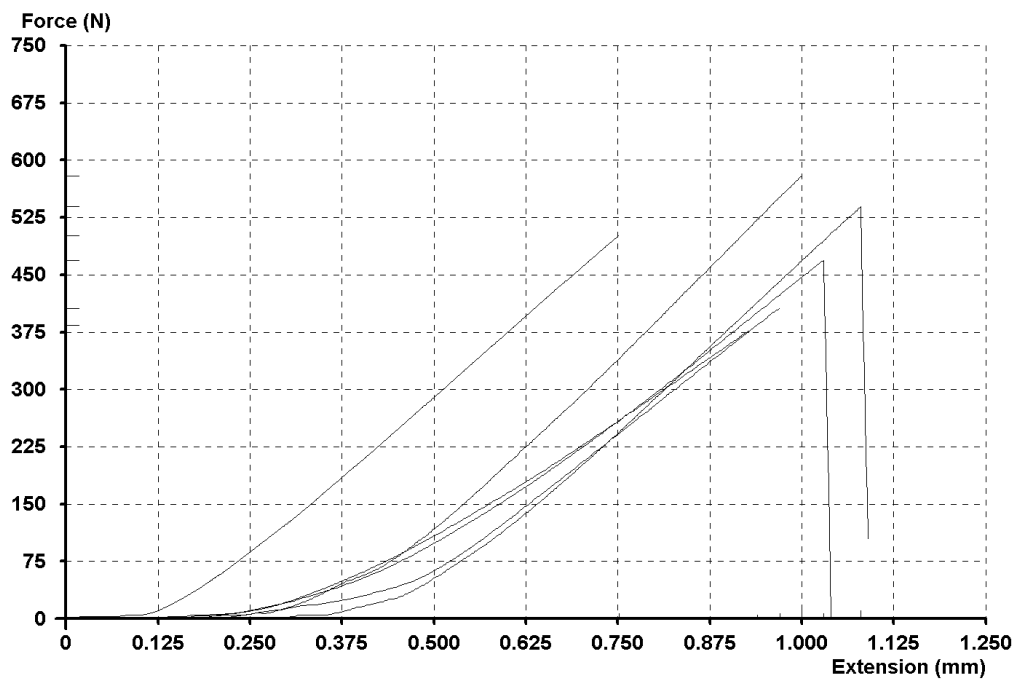
Specimen	Thickness mm	Width mm	Modulus @ 1 mm MPa	Flexural Strength MPa	Strain to Failure -
1	29.71	5.38	228.8	10.59	0.0365
2	28.11	5.41	342.5	9.97	0.0282
3	33.19	5.78	387.2	12.27	0.0314
4	34.04	5.19	333.0	13.83	0.0380
5	30.53	5.82	351.3	12.19	0.0321
6	33.10	6.33	375.4	11.87	0.0313
Mean			336.4	11.79	0.0329
Median			346.9	12.03	0.0317
Std. Dev.			56.5	1.364	0.0036
Coe. Var.			16.79	11.58	11.02
Maximum			387.2	13.83	0.0380
Minimum			228.8	9.97	0.0282



3-pt Flexural - Rect.Beam/Centre load

Product Code	: phenolic	Load Range	: 2000 N
Batch Reference	:	Extension Range	: 10 mm
Product Description	:	Speed	: 1.0 mm/min
Date	: 7/09/2011	End Point	: 9.0 mm
Operator	:	Support Span Separation	: 80 mm
Temperature	:	Young's Modulus Point	: 1.0 mm
Relative Humidity	:	Preload	: 0.0 N
		Auto Return	: On

Specimen	Thickness mm	Width mm	Modulus @ 1 mm MPa	Flexural Strength MPa	Strain to Failure -
1	32.06	5.58	400.0	12.13	0.0301
2	32.13	4.430	385.5	12.31	0.0310
3	31.55	4.360	-	10.63	0.0278
4	32.41	4.490	-	12.76	0.0228
5	31.49	4.230	-	11.62	0.0286
6	31.90	5.66	323.0	11.24	0.0323
Mean			369.5	11.78	0.0288
Median			385.5	11.87	0.0293
Std. Dev.			40.93	0.775	0.0033
Coe. Var.			11.08	6.58	11.55
Maximum			400.0	12.76	0.0323
Minimum			323.0	10.63	0.0228

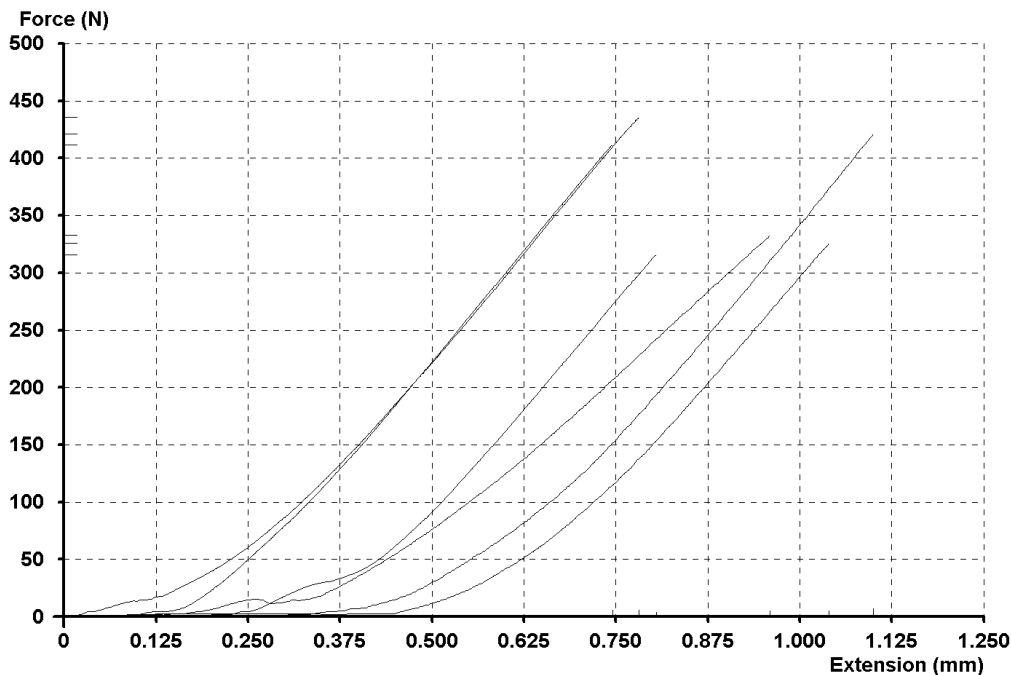


30% by weight of vinyl ester resin

3-pt Flexural - Rect.Beam/Centre load

Product Code	: phenolic	Load Range	: 2000 N
Batch Reference	:	Extension Range	: 10 mm
Product Description	:	Speed	: 1.0 mm/min
Date	: 7/09/2011	End Point	: 9.0 mm
Operator	:	Support Span Separation	: 80 mm
Temperature	:	Young's Modulus Point	: 1.0 mm
Relative Humidity	:	Preload	: 0.0 N
		Auto Return	: On

Specimen	Thickness mm	Width mm	Modulus @ 1 mm MPa	Flexural Strength MPa	Strain to Failure -
1	31.37	4.280	-	8.99	0.0237
2	32.28	4.590	-	10.93	0.0236
3	32.27	4.760	270.0	10.19	0.0333
4	34.43	4.000	-	10.41	0.0241
5	31.91	4.370	263.4	8.78	0.0311
6	31.66	4.240	-	9.40	0.0285
Mean			266.7	9.78	0.0274
Median			266.7	9.79	0.0263
Std. Dev.			4.688	0.855	0.0042
Coe. Var.			1.758	8.74	15.38
Maximum			270.0	10.93	0.0333
Minimum			263.4	8.78	0.0236

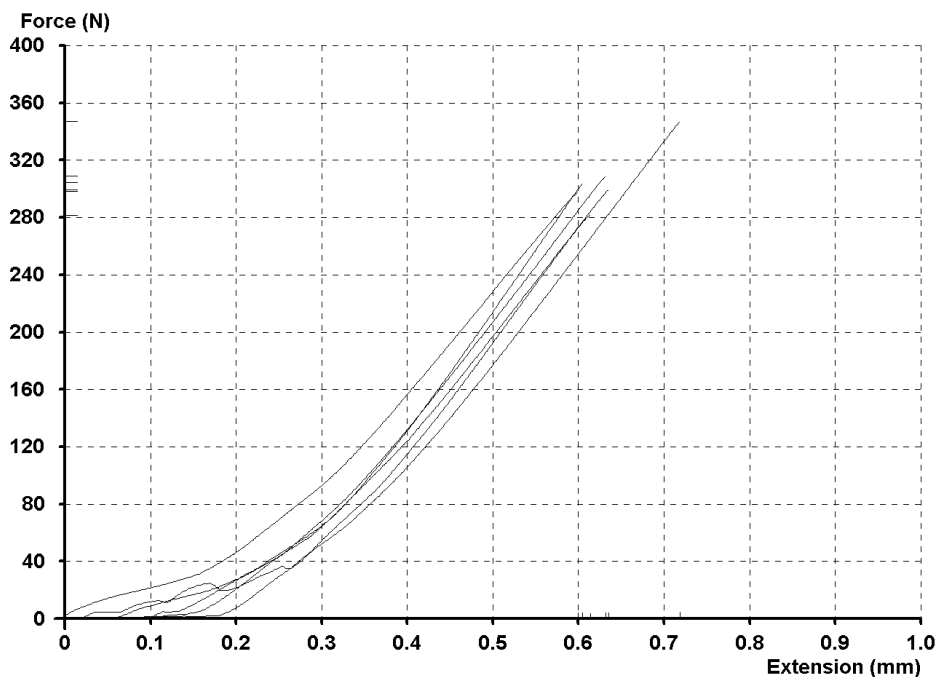


35% by weight of vinyl ester resin

3-pt Flexural - Rect.Beam/Centre load

Product Code	: phenolic	Load Range	: 2000 N
Batch Reference	:	Extension Range	: 10 mm
Product Description	:	Speed	: 1.0 mm/min
Date	: 7/09/2011	End Point	: 9.0 mm
Operator	:	Support Span Separation	: 80 mm
Temperature	:	Young's Modulus Point	: 1.0 mm
Relative Humidity	:	Preload	: 0.0 N
		Auto Return	: On

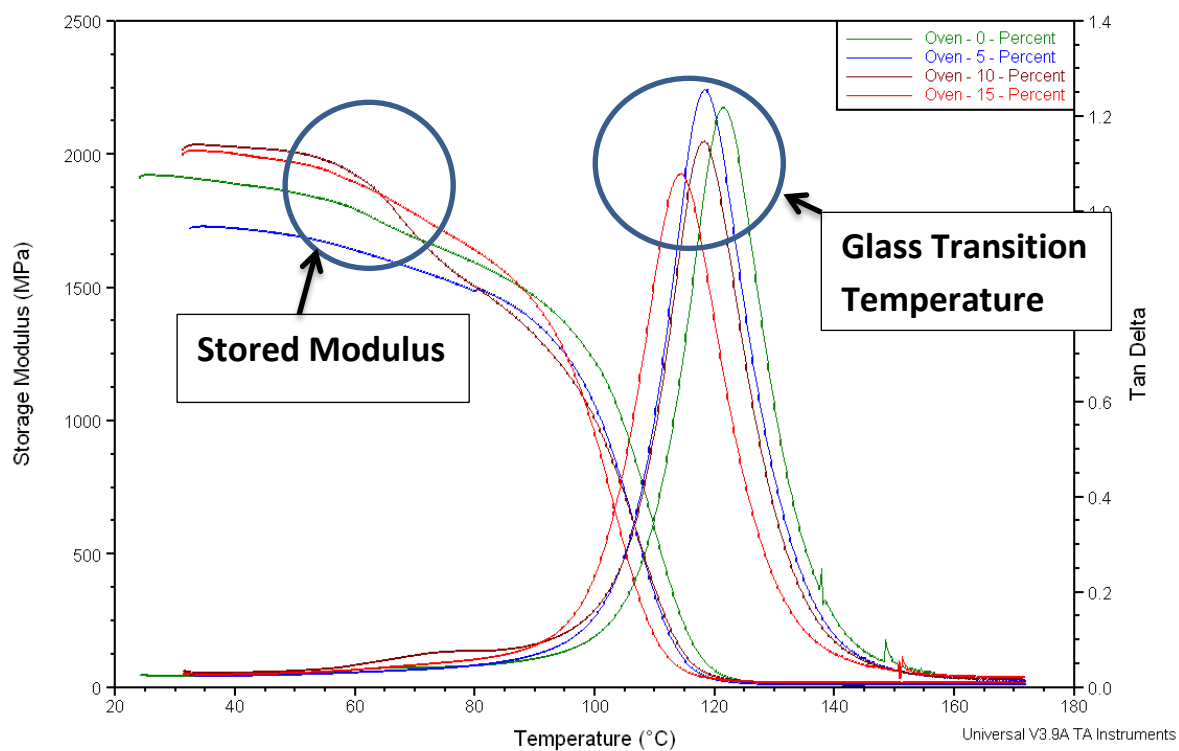
Specimen	Thickness mm	Width mm	Modulus @ 1 mm MPa	Flexural Strength MPa	Strain to Failure -
1	32.92	4.540	-	8.46	0.0222
2	31.55	4.380	-	8.50	0.0187
3	32.78	3.940	-	8.45	0.0184
4	32.32	4.240	-	8.25	0.0183
5	32.34	4.210	-	7.67	0.0186
6	31.69	4.000	-	8.94	0.0189
Mean			-	8.38	0.0192
Median			-	8.46	0.0187
Std. Dev.			-	0.4145	0.0015
Coe. Var.			-	4.947	7.72
Maximum			-	8.94	0.0222
Minimum			-	7.67	0.0183



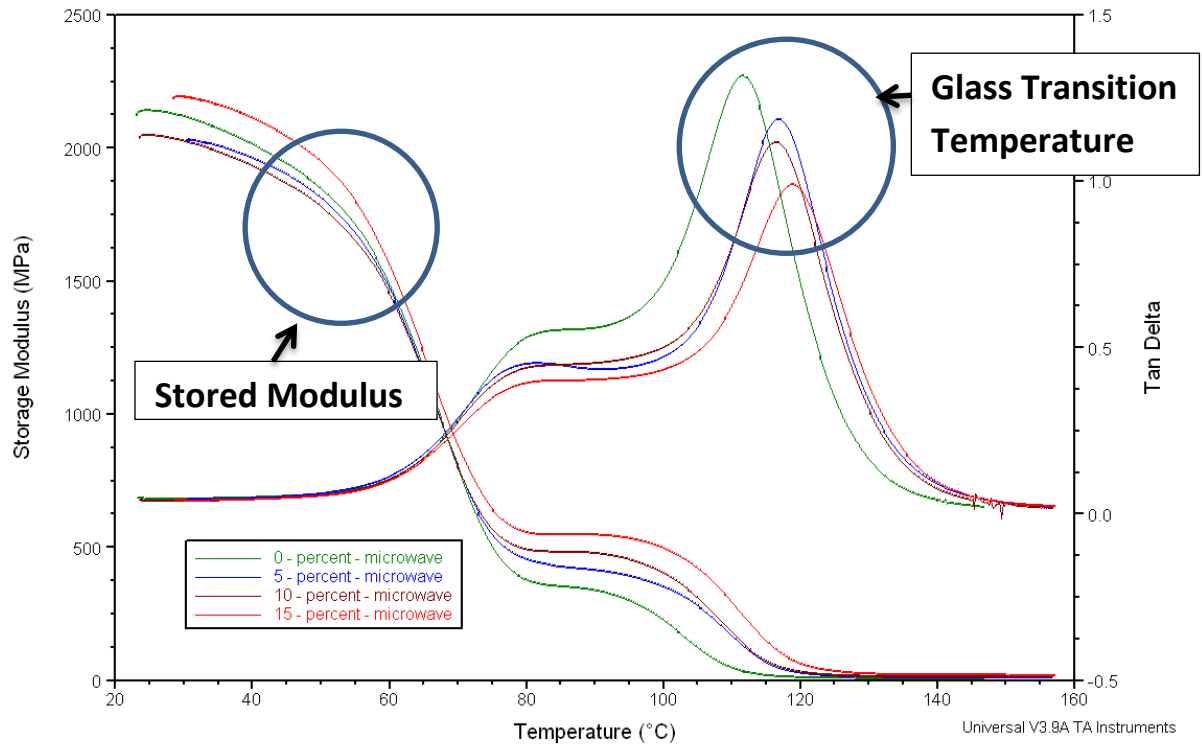
Appendix D

Dynamic Thermal Mechanical Analysis Test Data

Oven cured samples



Microwave cured samples



Appendix E

Eurotherm Controller Instruction









USER MANUAL

EUROTHERM 3200 SERIES CONTROLLERS

This manual covers the 3200 series of Eurotherm controllers. The images shown in the manual will vary and might not be the same as the controller installed on your Steridium product.

5.8.2 To Operate the Programmer

Operation of the programmer is the same as the timer.

Operation	Action	Indication
To Run a program	Press and quickly release  + 	Beacon -- RUN = On Scrolling display - TIMER RUNNING
To Hold a program	Press and quickly release  + 	Beacon -- RUN = Flashing Scrolling display - TIMER HOLD
To Reset a program	Press and hold  +  for more than 1 second	Beacon -- RUN = Off If End Type = Off then OFF will be displayed at the end of the program
	Program ended	Beacon -- RUN = Off SPX = On if End Type = SP2 Scrolling display - TIMER END

















Repeat the above to Run the programmer again (Note: it is not essential to reset it after the End state is reached)

Programs can also be operated from the 'T.STAT' parameter found in the Level 2 parameter list.

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5.8.3 To Configure the Programmer


Select Access Level 2 – see section 5.1.

Operation	Action	Indication	Notes
Configure the Timer as a Programmer	Press  to select 'TM.CFG' Press  or  to 'PrOf'		
Set the Resolution	Press  to select 'TM.RES' Press  or  to 'Hour' or 'm: n"		In this example Dwell is set in hours and Rate in units/hour
Set the Threshold	Press  to select 'THRES' Press  or  to adjust		In this example the dwell periods will not start until the PV is within 5 units of the setpoint
Set the action when the programmer times out	Press  to select 'END.T' Press  or  to 'OFF', or 'SP2', or 'dWElI'.		In this example the controller will continue to control indefinitely at the last setpoint OFF will turn the output power off and SP2 will control at setpoint 2


5. Operator Level 2

Level 2 provides access to additional parameters. It is protected by a security code.

5.1 To Enter Level 2

1. From any display press and hold .
2. After a few seconds the display will show:-



3. Release .



(If no button is pressed for 45 seconds the display returns to the HOME display)

4. Press  or  to choose LEU 2 (Level 2)



5. After 2 seconds the display will show:-



6. Press  or  to enter the pass code. Default = '2',




7. If an incorrect code is entered the controller reverts to Level 1.



5.2 To Return to Level 1



1. Press and hold .
2. Press  to select LEU 1

The controller will return to the level 1 HOME display. Note: A pass code is not required when going from a higher level to a lower level.

5.3 Level 2 Parameters

Press  to step through the list of parameters. The mnemonic of the parameter is shown in the lower display. After five seconds a scrolling text description of the parameter appears.

The value of the parameter is shown in the upper display. Press  or  to adjust this value. If no key is pressed for 30 seconds the controller returns to the HOME display

Backscroll is achieved when you are in this list by pressing  while holding down .

The following table shows a list of parameters available in Level 2.