



University of Southern Queensland  
Faculty of Engineering & Surveying

# **Flexural strength of three types of glass powder reinforced Vinyl Ester composites**

A dissertation submitted by

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In fulfilment of the requirements of  
**Courses ENG4111 and 4112 Research Project**

towards the degree of

**Bachelor of Engineering (Mechanical)**

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# **Abstract**

Composites can be obtained from the combination of various materials, as long as the materials are distinct at a macroscopic level. The significant benefit of using composites is their superior mechanical properties (such as strength, corrosion resistance, light weight, etc.) as well as low manufacturing cost during mass production.

When very fine (powder) spherical, hollow glass beads are mixed with Vinyl Ester (VE) resin and a catalyst such as MEKP (Methyl ethyl ketone peroxide) are mixed together, the chemical reaction that takes place between the VE and MEKP binds the glass beads in a strong bond and becomes a composite of stronger mechanical properties than the glass spheres.

Therefore, there is scope to form specimens of different types of glass powder sphere reinforced composites and analyse them for their flexural behaviour. From such analysis, it would be possible to come up with conclusions on the appropriateness of future research and commercialisation of the composite.

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I further certify that the work is original and not been previously submitted for assessment in any other course or institution, except where specifically stated.

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Signature

29 October 2009

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Date

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University of Southern Queensland

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# Nomenclature

MSDS = Material Safety Data Sheet

F = Force (in Newtons)

A = Cross section area

L = Original length

D = Deflection

b = Width

d = Thickness

$\sigma$  = Stress

$\varepsilon$  = Strain

MPa = Mega Pascals

VE = Vinyl Ester resin

MEKP = Methyl ethyl ketone peroxide

# Chapter 1 Introduction

## 1.1 Introduction

Composites are engineered materials that are comprised of two or more constituent materials. The chemical or physical behaviour of the constituent materials are significantly different from each other and they remain separate at a macroscopic level within the finished composite structure.

Composite materials (or ‘composites’) have been around for more than 5000 years. In modern times, there has been growing interest in composites due to the fact that they can offer desired combination of properties (e.g., light, strong, corrosion resistant materials). This concept of achieving a better combination of properties is called the *principle of combined action*.

### *Examples*

*New:* Advanced materials, engineered to specific applications, such as plywood, oriented strand board, wood plastic composite (recycled wood fibre in polyethylene matrix), perlite steel (combination of hard, brittle cementite with soft, ductile ferrite to get a superior material).

*Old:* brick-straw composites, paper.

*Natural composites:* Fibre-reinforced polymers or FRPs such as wood (consisting of cellulose fibres in a lignin and hemicellulose matrix), bones (polymer-ceramics).

## **1.2 Research Objectives**

The aim of this research project is to produce various vinyl ester composite specimens with different percentage by weight of filler (glass powder). After the initial casting, curing and post curing, flexural strength tests were conducted in the university laboratory to obtain flexural strength data of the composite materials.

The data was analysed to evaluate trends and formulae for the theoretical prediction of the composite material behaviour.

## **1.3 Conclusions**

This dissertation aims at providing a detailed overview of the composite specimen casting, curing, post curing, testing and result analysis on three (3) types of glass powders, namely, Spherical 60P18, QCel 5020 and QCel 6019.

A review of literature associated to this research would help determine the key concepts and important background research data and information. Also, it would provide a good foundation for future research work.

The outcome of this research may provide the starting point for future research, as composites have been attracting much attention from researchers. Also, the results obtained during this exercise may be used to design and develop newer, cost effective composites by using hollow glass spheres and vinyl ester resins in a similar manner, if not the same as used during this research.

# Chapter 2 Literature review

## 2.1 Formation of Composites

### 2.1.1 Phases

Solid materials are divided into four classes: polymers, metals, ceramics, and carbon. Generally, composites, alike other solid materials, are comprised of two materials or 'phases':

1. Matrix phase (continuous)
2. Reinforcing/dispersed phase (particulates, fibres)

As the basic formation method of composites, the 'reinforcing phase' is embedded in the other material called 'the matrix phase'. The reinforcing phase and the matrix phase can both be metal, ceramic, or polymer.

Normally, reinforcing phases are strong with low densities while the matrix phase is usually a ductile or tough material. If the composite is designed and fabricated correctly, it combines the strength of the reinforcement with the toughness of the matrix to achieve a combination of desirable properties not available in any single conventional material.

#### *(a) Matrix*

The matrix is the monolithic material into which the reinforcement is embedded, and is completely continuous. This means that there is a path through the matrix to any point in the material, unlike two materials sandwiched together. In structural applications, the matrix is usually a lighter metal

such as aluminium, magnesium, or titanium, and provides a compliant support for the reinforcement. In high temperature applications, cobalt and cobalt-nickel alloy matrices are common.

***(b) Reinforcement***

As mentioned earlier, the reinforcement material is embedded into the matrix. The reinforcement does not always serve a purely structural task (reinforcing the compound), but is also used to change physical properties such as wear resistance, friction coefficient, or thermal conductivity. The four key types of reinforcements used in composites are continuous fibres, discontinuous fibres, whiskers (elongated single crystals), and particles (refer to figure below).

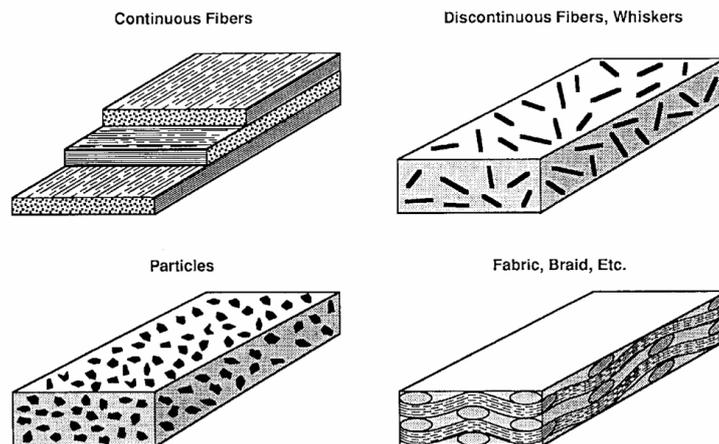


Figure 1: Types of reinforcements

**2.2 Types of composites**

***2.2.1 Classification based on constituents***

There are mainly 3 (three) basic types of composites with several sub-classifications:

1. Particle-reinforced

- Large-particle
- Dispersion-strengthened

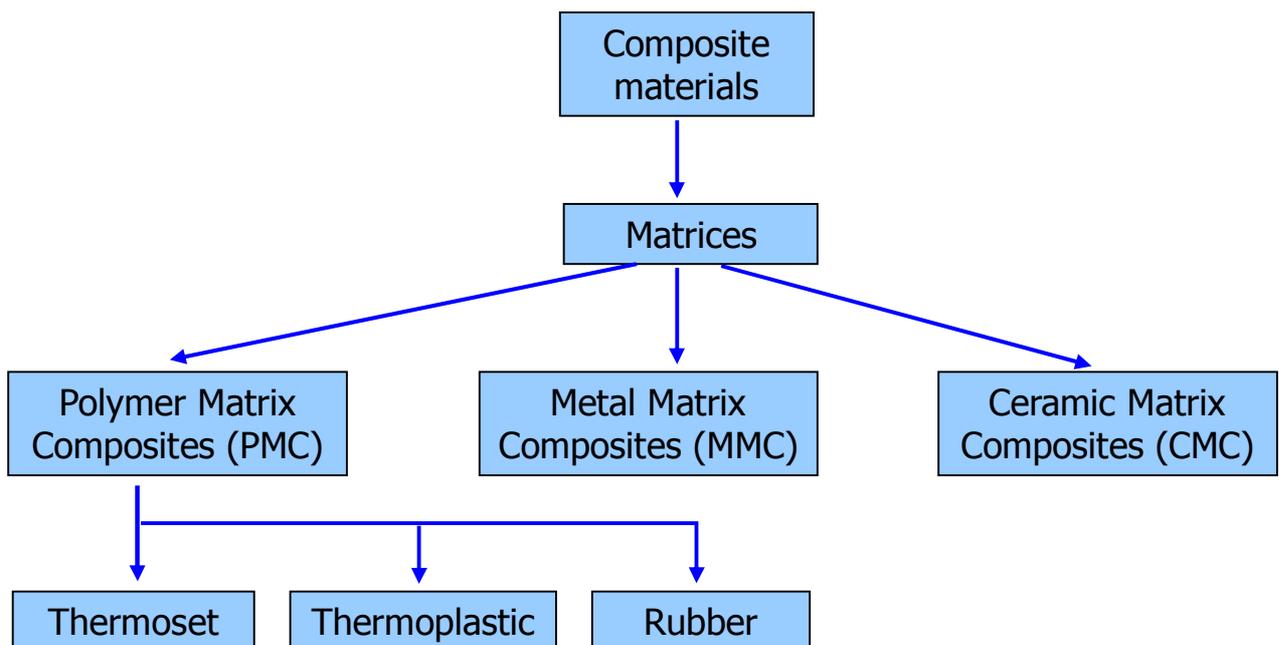
2. Fibre-reinforced

- Continuous (*aligned*)
- Discontinuous short fibres (*aligned or random*)

3. Structural

- Laminates
- Sandwich panels

2.2.2 *Classification based on matrices*



***(i) Polymer Matrix Composite (PMC)***

Polymer Matrix Composite (PMC) is the material consisting of a polymer (resin) matrix combined with a fibrous reinforcing dispersed phase. Polymer Matrix Composites are very popular due to their low cost and simple fabrication methods.

***(ii) Metal Matrix Composite (MMC)***

Metal Matrix Composites (MMC) is a composite where a metal or alloy forms a continuous network.

***(iii) Ceramic Matrix Composite (CMC)***

The key ceramics used as CMC matrices are silicon carbide, alumina, silicon nitride, mullite, and various cements. The properties of ceramics, especially strength, are even more process sensitive than those of metals.

## **2.3 Thermoset**

Thermosetting resins are used in moulded and laminated plastics. These resins are fluid at standard temperature and pressure. They are first polymerized into a low-molecular-weight linear or slightly branched polymer or oligomers, which are still soluble, fusible, and highly reactive during final processing. Thermoset resins are generally highly filled with mineral fillers and glass fibres. Thermosets are generally catalysed and/or heated to finish the polymerization reaction, cross linking them to almost infinite molecular weight. This step is often referred to as curing. Such cured polymers cannot be reprocessed or reshaped. The high filler loading and the high crosslink density of thermoset resins result in high densities and low ductility but high rigidity and good chemical resistance.

### **2.3.1 Vinyl Esters (VE)**

Vinyl esters are part of the unsaturated polyester family. They are prepared by the reaction of an epoxy resin with meth acrylic acid. Thus the epoxide group is converted into a meth-acrylate ester. Vinyl esters offer an enhancement in properties over unsaturated polyesters with greater toughness and better resistance to corrosion to a wide range of chemicals. This chemical resistance includes halogenated solvents, acids, and bases.

*Uses:* Applications for vinyl esters are similar to those for unsaturated polyesters but where added toughness and chemical resistance are required, i.e., electrical equipment, flooring, fans, adsorption towers, process vessels, and piping.

## **2.4 Flexural Strength**

### **2.4.1 Definition**

In brittle materials, flexural strength is a mechanical parameter that indicates a material's ability to resist deformation when it is placed under a load. Frequently, a transverse load is applied on a sample of rectangular cross-sectional area until the sample fractures. The highest stress the material experiences at the moment of failure is known to be the flexural strength of the material. Understandably, since it attempts to find the highest stress at the moment of failure, the measure of flexural strength is stress in MPa.

### **2.4.2 Testing Methods**

The International Organisation for Standards (ISO) specifies 2 methods for determining the flexural properties of fibre-reinforced plastic composites. They are:

1. Three point (3-point) flexural test
2. Four point (4-point) flexural test

For this project, testing has been carried out using the 3-point flexural test arrangement.

#### ***2.4.2.1 Three point (3-point) flexural test***

The three point (3-point) flexural test is a flexure test that 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.

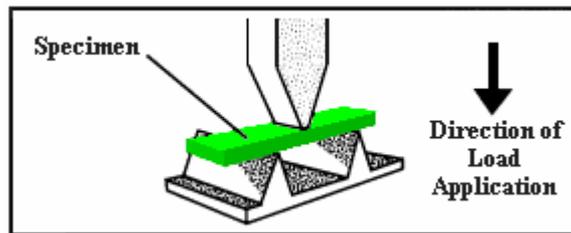


Figure 2: Three point (3-point) flexural test arrangement

The governing equation for calculating flexural strength of a rectangular sample in a 3-point test is:

$$\sigma = \frac{3FL}{2bd^2}$$

where,

$\sigma$  = Flexural strength [MPa]

$F$  = load (force) at the fracture point [N]

$L$  = length of the support span [mm]

$b$  = width of the support span [mm]

$d$  = thickness of the support span [mm]

## **2.5 Fillers, resins and catalysts**

### **2.5.1 Filler: Glass powder**

Fillers (reinforcement materials) help reduce shrinkage during moulding, lower the manufacturing cost and improve strength of the material. They are also used to improve electrical and thermal insulating properties and chemical resistance. The testing will be done by varying the amount of filler from 0 to 30 %.

Three (3) types of hollow glass spheres (powders) were used in this project to manufacture the composites. They are:

1. QCEL 5020
2. QCEL 6019
3. SPHERICEL 60P18

#### **QCEL 5020 & 6019:**

QCel Hollow Microspheres have sufficient pressure and shear resistance to withstand typical mixing processes. They dissolve easily into a wide range of liquid systems and because of their low density and spherical shape; they do not contribute significantly to viscosity. To ensure maximum effectiveness, density is commonly used as an indication of sphere content. QCel Hollow Spheres provide an economical way to reach the critical density required for the final product.

Typical Properties of QCel Hollow Glass Microspheres:

<b>Physical Form</b>	Free-Flowing Low Density Powder
<b>Color</b>	White
<b>Surface Treatment</b>	Oleophilic (having a strong affinity for oils rather than water)

Type	Density (g/cm <sup>3</sup> )		Particle Size $\mu\text{m}$ (Malvern)		Working pressure		
	Bulk	Effective	Mean	Range	psi	bar	MPa
<b>5020</b>	0.12	0.20	55	5 ~ 115	500	34	3.4
<b>6019</b>	0.13	0.21	75	5 ~ 175	500	34	3.4

### SPHERICEL 60P18:

Spherichel 60P18 hollow glass spheres are used to enhance performance and reduce viscosity in paints and coatings and as lightweight additives in plastic parts. They are chemically inert, non-porous, and have very low oil absorption.

Grade 60P18 is used in many high performance polymer systems.

Typical properties of the spheres are as following:

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

Spherichel 60P18 hollow spheres offer formulators flexibility in polymer composites. The addition of hollow spheres to fiberglass reinforced plastics (FRP), epoxy, compounds, and

urethane castings can provide weight reduction cost savings and improved impact resistance. Insulating features of hollow spheres also work to the chemists' advantage in thermal shock and heat transfer areas. Two densities available are 0.6 to 1.1 g/cc; it provides choices to best fit mixing and target weight requirements (Potters Industries, undated b). The density of the hollow glass powder used in this research is 0.6 g/cc because the other filler, ceramic hollow spheres or SLG used in similar study is 0.7 g/cc; this will give a better basis for comparison of results obtained in the future. When used in polymer concrete, hollow spheres provide a cost effective alternative without degrading physical properties. The material safety data sheet of Spherichel 60P18 hollow spheres was also carefully studied to avoid unnecessary accidents (Potters Industries, undated b; undated c).

The particle size of the white glass powder ranges from 6 to 32 microns with an average size of 20 microns. They are therefore micron fillers. These fused inorganic oxides are spherical and non-porous.

### **2.5.2 Resin: Vinyl Ester (VE)**

Vinyl esters (matrix phase) are a family of thermosetting resins that have many similarities to, and seem to fit between, both unsaturated polyesters and epoxies. These resins are slightly more expensive than unsaturated polyesters but are not as expensive as epoxies. It offers superior

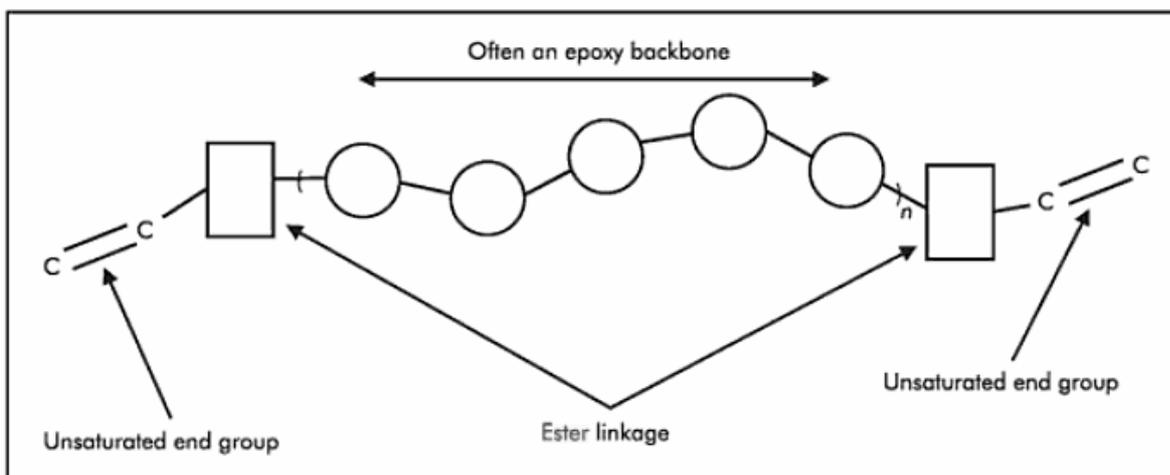
chemical resistance, maximum service temperature of 220<sup>0</sup>F (104<sup>0</sup>C), fast chemical set time and

low porosity. They have better toughness and corrosion resistance compared to polyesters and cure easier than epoxies. Therefore, vinyl esters are ideal candidates where cost is a major issue and chemical resistance and toughness over unsaturated polyesters are desired.

### *Advantages of vinyl ester resins*

Vinyl esters have found their use in bathroom fixtures, automotive body parts, chemical storage tanks, pipes and liners, furniture, boat hulls, fishing rods, light weight ladder rails, recreational vehicle parts and architectural components. Cost of vinyl ester resins is comparative with polyesters and are thus used in various applications, for its very low cost per unit volume. Vinyl ester resins have high chemical resistance as compared to other cheap resins. Also, they have good dimensional stability under temperature fluctuations and good adhesive properties. Due to these special properties, vinyl ester resins have been found to be attractive for aircraft, mass transit vehicles, and as interior construction materials. But due to styrene contents, this resin is not suitable for use in areas of food handling and preparation because of the risk of tainting.

### *Formation of Vinyl Ester resin*



Vinyl ester polymers are formed by reacting an epoxy resin with an acrylic acid. The acrylic acid opens the epoxy rings and makes way for multiple sites for cross linking (curing) reactions. The four main steps in this process are:

***Step 1:***

The cross linking reactions begin with a peroxide catalyst that splits into two parts, each containing a free radical. Those free radicals then react with the carbon-carbon double bond (as shown in the following figure).

***Step 2:***

In the presence of styrene molecules, the free radicals attack the polymer ends.

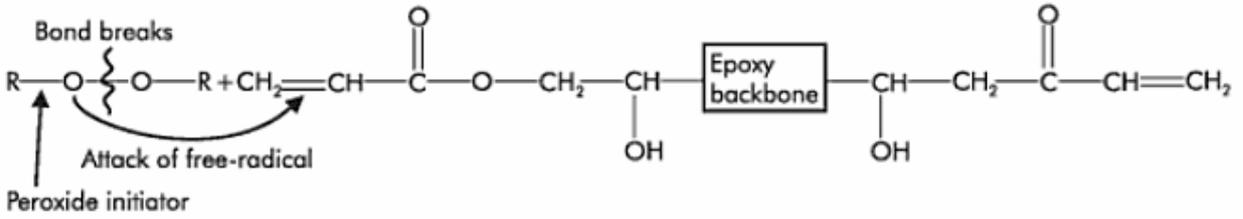
***Step 3:***

The styrene and the polymer bonds together. Depending on the relative concentrations of polymer and styrene, and reaction conditions, several styrene molecules can form the cross linking bridge.

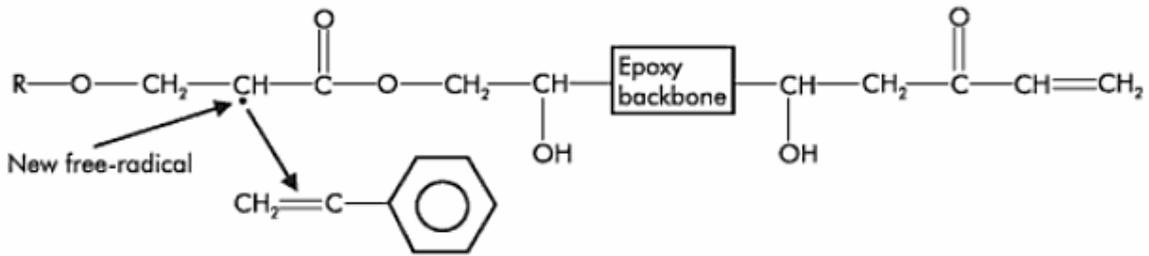
***Step 4:***

The first polymer attaches to a second vinyl ester polymer. The non-reacting ends are likely to join different molecules. That would result in a large cross linked network.

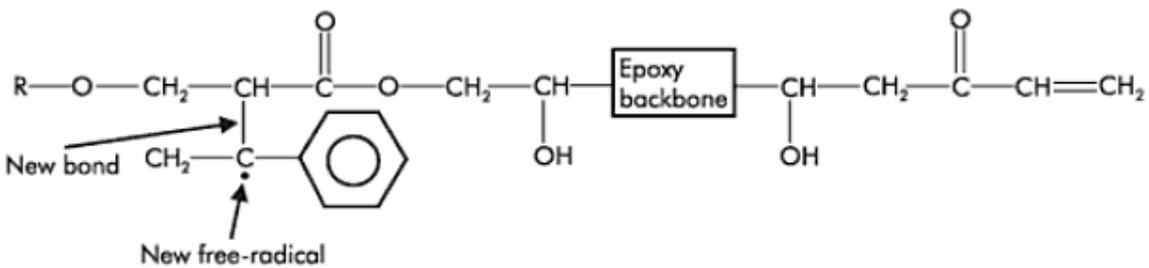
**Step 1: Activation of the free-radical and attack at the carbon-carbon double bond**



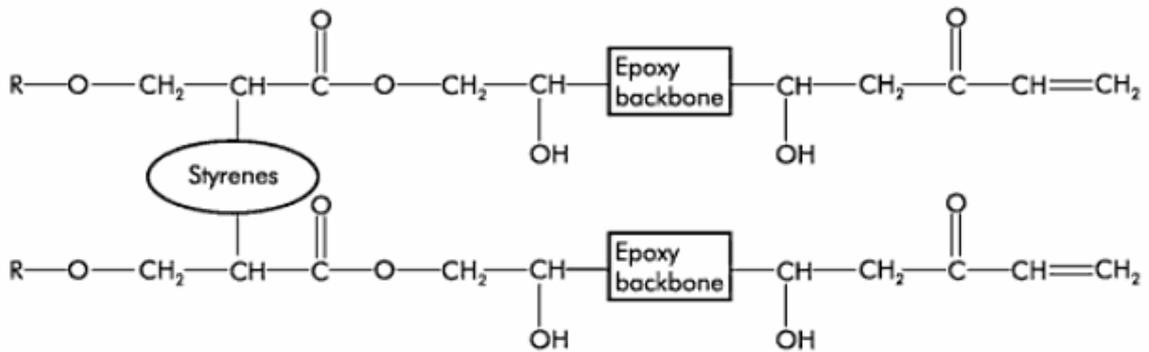
**Step 2: Reaction of the new free-radical with styrene**



**Step 3: Formation of a styrene bridge and creation of a new free-radical**



**Step 4: Linkage of the free-radical on styrene with a new vinyl ester polymer**



(Strong, A. Brent, *Fundamentals of Composites Manufacturing: Materials, Methods and Applications*, SME, 2008)

### 2.5.3 Choice of catalyst

For the purpose of cross linking reaction to initiate, a peroxide catalyst is required. As an optimal choice in the polyester system, the following catalysts for vinyl ester resin can be used:

- **Methyl ethyl ketone peroxide (MEKP)**

Methyl ethyl ketone peroxide (MEKP) is an organic, toxic peroxide. It is a colourless and less sensitive to shock and temperature, and more stable in storage.

- **AkzoNobel Butanox LA**

AkzoNobel Butanox LA is Methyl ethyl ketone peroxide (MEKP) in phthalate mixture. It is used for the curing/cross linking of unsaturated polyester resins (similar to MEKP).

Though MEKP was used as the primary catalyst, in the event of non-availability of MEKP, AkzoNobel Butanox LA at higher concentration is used at 1.5% by weight for similar effect.

#### 2.5.3.1 Methyl Ethyl Ketone Peroxide (MEKP)

MEKP is a colourless, oily liquid solution of methyl ethyl ketone peroxide at STP in dimethyl phthalate, with 9% active oxygen. It is used as a catalyst which initiates the polymerization of polyester resins used in glass-reinforced plastic, and casting.

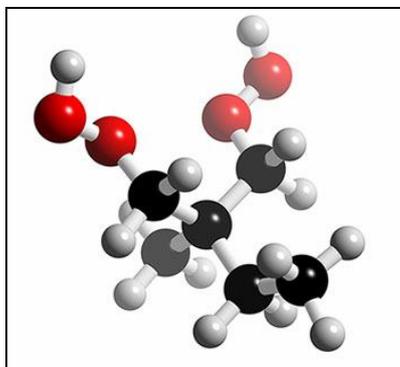


Figure 3: 3D model of the MEKP structure

<b>Molecular formula</b>	C <sub>4</sub> H <sub>10</sub> O <sub>4</sub>
<b>Appearance</b>	Colourless, high-viscosity liquid
<b>Density</b>	1.15 g/cm <sup>3</sup>
<b>Hazard classification</b>	Organic peroxide, Type D, class 5.2.

MEKP should be stored in a closed container in a cool, dry place away from all sources of heat, sparks, or flames, and out of direct sunlight. Explosive decomposition may take place if MEKP is exposed to high temperatures or contamination with foreign materials. This catalyst is not to be stored in unvented glass containers or stored close to cobalt naphthenate, dimethyl aniline, or other promoters, accelerators, acids, bases, or strong reducing agents. The contained used to store MEKP should not be reused for any other purpose. Maximum storage temperature for MEKP is 38°C (100°F) and decomposition temperature 68°C (155°F).

## 2.6 Microscopic analysis – Scanning electron microscopy (SEM)

The scanning electron microscope (SEM) is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. In raster scan, the beam sweeps horizontally left-to-right at a steady rate, then blanks and rapidly moves back to the left, where it turns back on and sweeps out the next line. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition and other properties such as electrical conductivity.

The SEM analysis provides magnification of a surface (in this case, the fracture surface) from 10 times to up to 500,000 times. Hence, important characteristics of composite materials can be revealed at matrix-reinforcement level, which is not possible under a normal light microscope, let alone by normal human vision.

Due to the construction and functional requirements of the scanning electron microscope, samples usually need some preparation before they can be successfully imaged or analysed.

Samples must be:

Dry (placed into high vacuum)

Clean (placed into high vacuum; imaging of sample surface)

Able to generate a signal (image (SEI or BSI), analysis (EDS etc.))

Conductive (dissipation of charge and heat)

### ***2.6.1 Sample preparation***

For SEM analysis, samples require specialised preparation through a number of steps in sequence, as they are required to withstand the vacuum inside the microscope:

#### *1. Surface cleaning:*

Scanning electron microscopy is a surface imaging and analysis technique. The surface of the sample must be exposed and it is very important that the surface is clean. The surface of the sample may be obscured by deposits eg. mucous, cell debris, blood cells, dust, wax, oil, silt, wear debris, etc. Such surface debris need to be removed during this stage of preparation.

#### *2. Coating:*

The sample surface must be electrically and thermally conductive to provide a good image in the scanning electron microscope. Heat build-up (from the electron beam) may damage the sample. Charge build-up (electrons from the beam have a negative charge) will repel the incident electron beam, resulting in loss of signal from the sample.

To improve conductivity, the sample is coated with a thin layer of metal or carbon. This layer usually is 10-25 nm thick; high resolution scanning electron microscopy may require a thinner coating.

For topographical imaging of the composite samples made during the project, a gold sputter coating was used. This method is used to gold coat samples for secondary electron imaging. It is a non-directional coating method, which means, all surfaces of the sample are coated.

In a sputter coater machine, an inert gas (argon) is introduced in a relatively low vacuum (10<sup>-3</sup> Torr/10<sup>-1</sup> Pa) into a high voltage (1-3 kV) field. The gas molecules are ionised and are accelerated into metal 'target'. For gold coating, the target is a gold foil. Metal atoms are dislodged from the target, and the dislodged atoms continue to interact with argon, producing a 'cloud'. Gold atoms preferentially deposit on the sample (due to the configuration of the sample chamber), and build up a metallic coating on the sample.

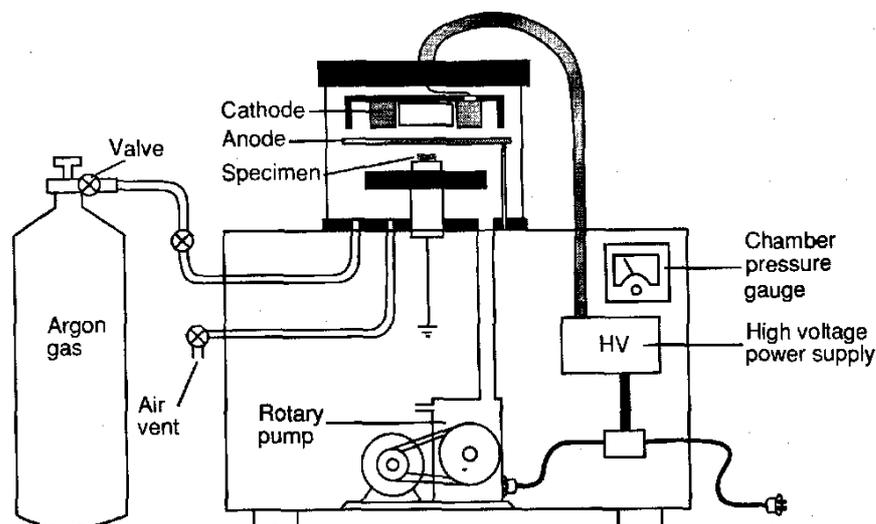


Figure 4: Basic construction of a sputter coating machine.



Figure 5: Actual sputter coating machine.

### 3. *Mounting:*

If there are possibilities that the sample would not stay stable inside the electron microscope, it would need to be mechanically stabilized using a ‘stub’. In the case of the samples prepared during this project, stubs were not required as they could be held on the stage of the SEM pretty firmly using double-sided tape.

#### 2.6.1 *Imaging*

During SEM imaging, an electron beam is thermionically emitted from an electron gun fitted with a tungsten filament cathode. Tungsten has the highest melting point and lowest vapour pressure of all metals, thereby allows to be heated for electron emission and also costs less. These are the reasons why it is often used as SEM filament.

As the scanning starts, the beam travels downward through a series of magnetic lenses designed to focus the electrons to a very fine spot. Near the bottom, a set of scanning coils moves the focused

beam back and forth across the specimen, row by row. As the electron beam hits each spot on the sample, secondary electrons are knocked loose from its surface. A detector counts these electrons and sends the signals to an amplifier. The final image is built up from the number of electrons emitted from each spot on the sample. The obtained image would appear similar to the one below.

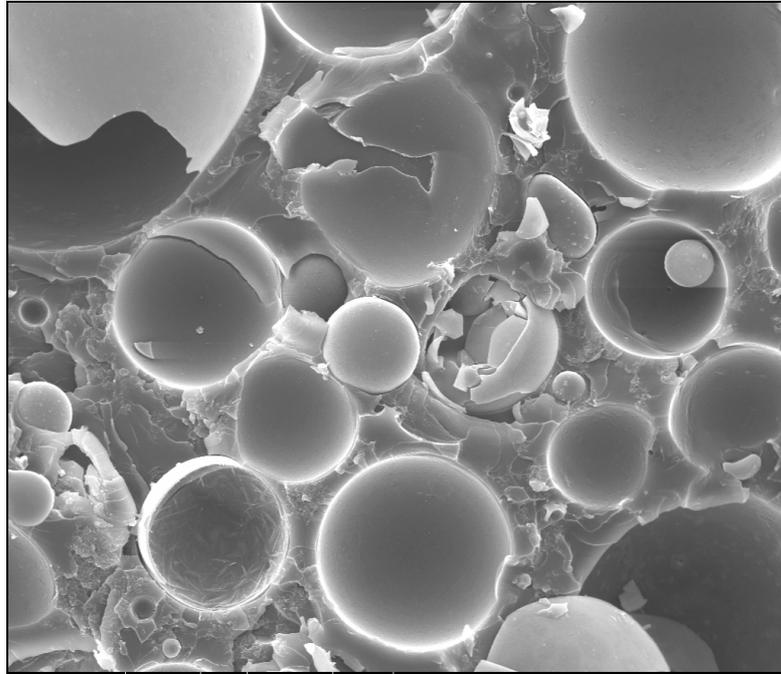


Figure 6: Example of SEM image at 1000 times magnification.

The SEM can magnify the surface image to 500,000 times to reveal detail and complexity that is otherwise not available for research.

## **2.7 Sustainability**

This project has negligible impact on the environment and therefore is environmentally sustainable. The resin, filler and catalysts used during sample preparation are mass produced and degradable. The only major use of energy during sample preparation and testing was the use of electricity, of course for which coal is a raw material. The carbon footprint of such a project can be reduced by opting for Green Energy, i.e. electricity generated from renewable energy sources. As the

technology, availability and incentive to use Green Energy are all available in Australia, the ecological impact of the project can be minimized to almost nil.

In the event of taking the research outcomes further by commercial ventures, it would be recommended that the possibility of using eco-friendly manufacturing practices are thoroughly carried out and chosen at every available opportunity.

## Chapter 3 Project Methodology

### 3.1 Sample preparation

#### 3.1.1 Composite formation

The composite formation takes place as:

$$\text{Composite (g)} = \text{Filler (g)} + \text{Catalyst (g)} + \text{Resin (g)}$$

For example,

**Composite:** 150 g

**Filler:** 15 g (10% of composite weight)

**Catalyst:** 2.7 g (2% by weight of 'Resin + Catalyst' weight, i.e. 135 g)

**Resin:** 132.3 g ('Resin + Catalyst' weight – Catalyst weight)

#### 3.1.2 Preparation of the open mould

Before mixing of resin, catalyst and glass powder, it is crucial to prepare the mould. Once the mix starts to cure, it starts losing its fluid nature and begins to solidify rapidly. Hence, a prepared mould allows the casting seamless and ensures the specimens to form according to specifications.

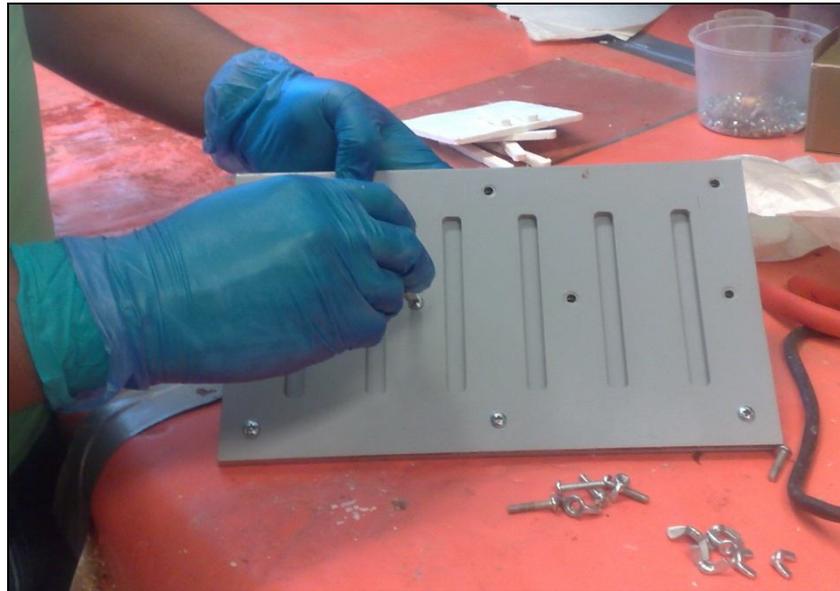


Figure 7: Mould cleaning.



Figure 8: Lubricant application.

Firstly the mould was scraped off thoroughly using a scrapper to make sure it was free from all external debris from previous use. This aids specimen extraction after the curing has taken place and ensures that the specimen does not contain foreign objects once it solidifies. After cleaning both the upper and lower moulds, they were clamped together using screws and wing nuts. The mould material is PVC and there were chances that cured specimens would get stuck to the surface. To prevent that, cooking oil was sprayed over the mould surface as a lubricant.

### 3.1.3 Weighing the constituents

It is very important that all the constituent materials of the composite are measured accurately before mixing so that the specimens conform to the standards. For example, the resin was measured using an electronic measuring scale in a plastic container. The scale was calibrated to zero after loading the container so that it measured the weight of the resin only. Similarly, the catalyst and glass powder was also measured and stored in separate containers ready to be mixed.



Figure 9: Weighing the glass powder.



Figure 10: Weighing the resin.

### 3.1.5 *Mixing the constituents*

As the mixing process of chemicals can emit toxic fumes, the process needs to be carried out under the ventilator chamber of the laboratory. The ventilator captures the toxic fumes produced during the reaction and safely disposes them into the external environment.

Firstly the glass powder was mixed with the resin gradually by slow stirring with a conventional whisk. It should be observed that the mix is not whisked rapidly as that would allow air to be trapped within the mixture and would reduce the structural integrity of the specimens. Next, the weighted catalyst is added to the resin-glass powder mixture and the overall mixture was further mixed until homogenous slurry was obtained.



Figure 11: Mixing the resin, glass powder & MEKP.

### ***3.1.6 Pouring the mixture in the mould***

Once the mixture was adequately homogenous, it was poured into the specimen slots of the mould using a spoon. In order to obtain the correct thickness of the specimens, extra care was exercised so that the slots were not over or under-filled. Under-filling the mould would mean the final specimens would shrink to smaller than required dimensions, whereas over-filling would cause difficulty while trying to extract the specimens out of the moulds.



Figure 12: Mixture ready to be poured.

### ***3.1.7 Natural curing***

After casting the specimens, the moulds were left on the laboratory shelf for natural curing at room temperature and standard pressure. Since the project aimed at preparing and testing 9 different sets of specimens, it was made sure that the moulds were clearly identifiable and therefore proper identifications were put on the moulds themselves. It was observed that the specimens took up to 48 hours to cure, whereas leaving them longer in the mould did not have significant impact in curing.

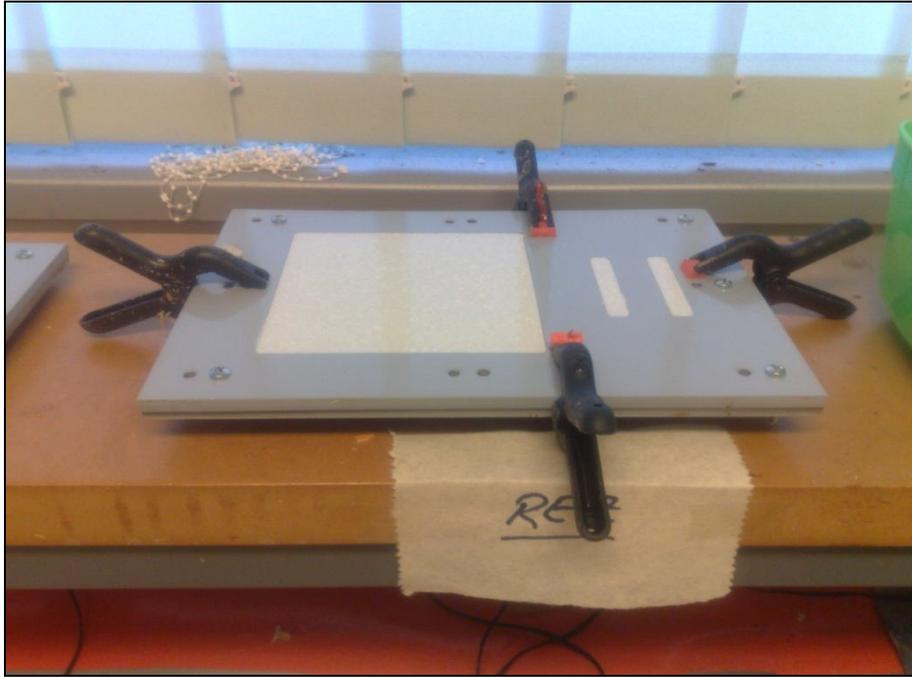


Figure 13: Natural curing at room temperature.

### **3.1.8 Post curing**

Post curing is a process where the specimens are baked in a conventional oven or microwave oven in order to further harden and set the composites and to increase its mechanical properties. The post curing was done as per the temperatures and durations as below:

- 4 hours at 50 °C;
- 4 hours at 80 °C;
- 4 hours at 100 °C.



Figure 14: Curing oven



Figure 15: Samples arranged inside the oven.

Conventional ovens are suited for post curing as per the scopes of this project goes, as they can

evenly cure the specimens. On the flip side, they consume considerable amount of electricity. Though post curing enhances mechanical characteristics of the specimens, heating them directly at high temperature can increase brittleness in the specimens. Hence the heating was done gradually in phases.

Due care was exercised during the heating process as it has been found in earlier experiments that the specimens tend to deform during heating. To prevent this from happening, the bunch of specimen is made and weights put at the ends during the heating. This kept the specimens straight and uniform in shape.

### ***3.1.9 Flexural property testing***

The specimens are tested for flexural strength. This is done in the University of Southern Queensland's engineering faculty laboratory on a Universal Testing Machine (UTM). The UTM that was available for use was an 810 Material Test System running TestStar 2S software.

The principle of the test is to test the specimen visualising it as a beam and deflecting it at a constant rate until the specimen fractures or the deformation reaches a pre-determined value. The force required to generate deflection is also measured.

The specimen can be three-point or four-point supported on the MTS. The MTS has a loading frame at the top and a supporting frame at the bottom of the test setup. The loading frame has hydraulic grips to hold the bending attachment that exerts force on the specimen during the test. Once the specimen is placed on the supporting frame, the bending attachment is lowered as close to the specimen surface as possible, so that testing time can be minimised without affecting the results.

At the beginning of the test, the width, height and thickness of the specimen need to be measured using digital slide callipers for dimensional accuracy. The specimens have to comply with the shape and dimensions chalked out in Table 3 (for three-point flexure) under section 6.1.3 of the ISO 14125:1998(E) reference manual.

It is very important to get these values correct, as otherwise future results would turn out to be erroneous. The width, height and thickness values are entered into the TestStar 2S software along with the span and speed values. Upon entering these input parameters, the machine is ready to carry out the test.

The test apparatus consists of a machine that complies with ISO 5893 and should be capable of maintaining test speeds specified in section 5.1.2 of the ISO reference manual. Also, the load and deflection indicators have to adhere to the section 5.1.4.

The results obtained by the tests are then used for analysis by software for calculation of flexural strengths of the specimens.

### **3.2 Assessment of Significant Effects**

The results obtained by this research may be used for analysis and may form the basis of future research work. Hence, the safety and other ethical issues related to the technical tasks performed/undertaken were also assessed.

The research has to encompass all facets of the Workplace Health and Safety Act, 1995 awareness as a responsibility of the researcher(s). The apparatus employed for testing is very expensive and

can be a source of injury as well. Codes of practice must be adhered to such that risks can be minimized to humans and machinery. Other risks are the loss of data from the experiments due to computer failures, etc. It is highly recommended to have periodical backups of all data obtained through testing.

Risk assessment comprises of:

- Risk identification
- Risk evaluation
- Risk control

The reference material can be found by browsing the internet and also from some course books from USQ, e.g. Engineering Management, Technology and Society, etc.

### **3.3 Safety issues and precautions**

#### **Resins**

The vinyl ester resin is not highly toxic from ingestion, but is capable of causing significant eye and skin effects. Because they contain relatively high amounts of styrene monomer, they also present a health problem due to inhalation. However, they may be handled safely if proper precautions are taken. These include, care to avoid inhalation of vapours and care to avoid skin and eye contact.

#### **MEKP**

**Ingestion:** MEKP is a strong irritant and highly toxic. Swallowing of MEKP can be fatal. In

the event of ingestion, large quantities of milk or water should be taken and medical help needs to be sought immediately.

**Contamination:** Contaminated clothing must be discarded. If skin comes in contact with MEKP, it must be washed with soap and water thoroughly.

**Contact with eye:** MEKP in the eye may result in permanent blindness, even if rinsed out with water or saline solution. Eyes must immediately be flushed with water for at least 30 minutes. It becomes a very time critical event and medical assistance need to be sought as soon as possible. Due to the potential dangers to human health that can be caused by this catalyst, it is always recommended to wear goggles, gloves, protective clothing, and a respirator.

### **Glass powders**

The glass powder fillers used during this research are not toxic, but are very fine spherical hollow beads. There is elevated risk of respiratory problems from inhalation of the powder and hence a respiratory mask is always recommended during handling them.

### **Cured samples**

Properly polymerized (cured) resins are considered to be toxicologically inert. Therefore, they do not present health problems from handling. The finished resins, however, may present a health hazard from inhalation of dust (such as during grinding) and also ingestion of surface contaminants.

### **Safe handling suggestions**

Suitable PPE should be worn. Impervious clothing can increase the hazard if it becomes contaminated on the inside. Suitable eye protection such as safety glasses or their equivalent should be worn to avoid eye contact. Contact with the fume should be particularly prevented. Ventilation sufficient to remove all vapour at the point of use should be maintained.

## Chapter 4

## Results, Discussion & Observations

### 4.1 Results & Discussion

#### 4.1.1 Flexural strength

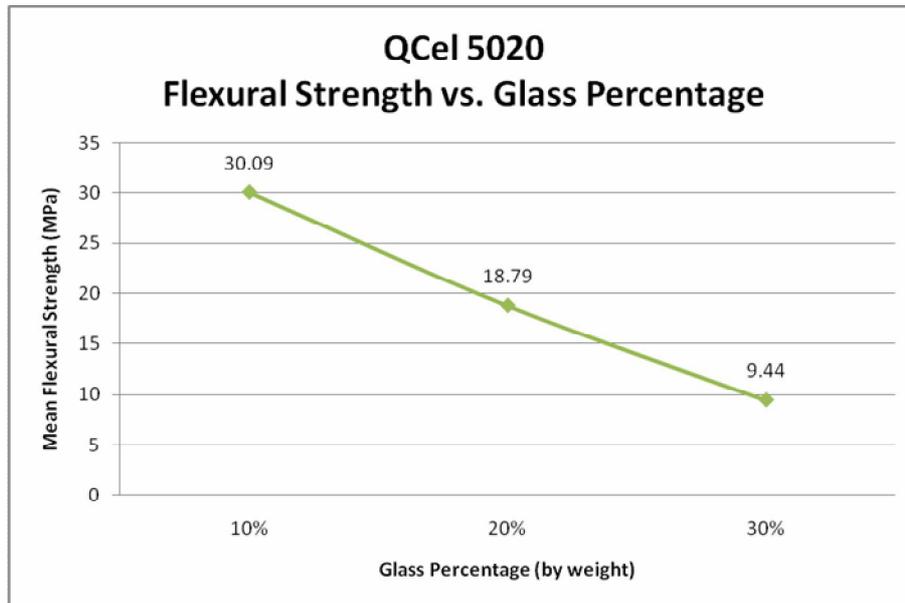
The flexural strength of individual specimens was calculated using the formula stated in section 2.4.2.1. In order to calculate the flexural strength at a particular glass percentage, the mean flexural strength of the specimens were calculated. In the same process, the mean flexural strength of all different percentages of QCel 5020 glass powder was calculated and tabulated in the following table:

	Glass Percentage		
	10%	20%	30%
Specimen 1	24.912	18.414	12.91
Specimen 2	27.684	13.385	7.97
Specimen 3	31.99	17.756	8.139
Specimen 4	30.254	23.324	8.755
Specimen 5	31.025	20.596	0
Specimen 6	34.672	19.236	0
Mean Flexural Strength (MPa)	30.089	18.785	9.443

Table 1: Flexural strengths for QCel 5020 specimens at different percentages.

From the data in the table above, the following graphical representation was obtained in order to

visually demonstrate the flexural strength of the QCel 5020 glass composite at different filler ratio.



Graph 1: Flexural Strength vs. Glass Percentage (QCel 5020)

It is to be noted that at 30% QCel 5020 filler ratio by weight, the calculations has been done using four (4) specimens only. At this ratio, the formations of specimens were very difficult as the amount of filler was too high for the resin to bind.

Nevertheless, it is evident from the graph that, QCel 5020 displays the highest flexural strength of 30.09 MPa at 10% filler ratio. The flexural strength almost linearly decreases as the filler ratio increases.

The flexural strength tables and graphs for QCel 6019 and Spherichel 60P18 are included in the Appendix, which are used for further discussion in the following chapter.

#### 4.1.2 Flexural strain

In order to calculate flexural strain of the specimens of a particular type of glass powder composite, the peak loads and maximum deflections during those loads need to be determined. The flexural strain of all three (3) types of glass powder composites were determined, and the exemplary process is as following:

Firstly, the peak loads experienced by the specimens were obtained from the UTS machine test reports. The table below demonstrates the peak loads in newtons (N) at all percentages of QCel 5020 specimens.

	Glass Percentage		
	10%	20%	30%
Specimen 1	77.212	87.283	36.927
Specimen 2	80.065	49.013	30.213
Specimen 3	110.782	70.498	26.185
Specimen 4	93.326	93.997	28.871
Specimen 5	91.983	57.07	0
Specimen 6	96.683	87.283	0
Mean Peak Load (N)	91.675	74.19	30.549

Table 2: Peak loads for QCel 5020 specimens at different percentages.

Next, the maximum deflection exhibited by all the specimens of QCel 5020 are tabulated in the following table from the data obtained from the UTS reports:

	Glass Percentage		
	10%	20%	30%
Specimen 1	13.53	9.61	9.76
Specimen 2	13.64	8.97	4.80
Specimen 3	14.29	10.92	4.91
Specimen 4	20.19	12.59	6.48
Specimen 5	18.78	14.47	0.00
Specimen 6	17.76	11.48	0.00
Avg. Max Deflection (mm)	16.35	11.24	6.41

Table 3: Max deflections for QCel 5020 specimens at different percentages.

Once the maximum deflection values are obtained, the following formula is used for calculating the flexural strain of the QCel 5020 specimens at 10%, 20% and 30%:

$$\varepsilon_f (\%) = \frac{6Dh}{L^2 \times 100}$$

where,

$\varepsilon_f$  = Flexural Strain (%)

$D$  = Maximum deflection (mm)

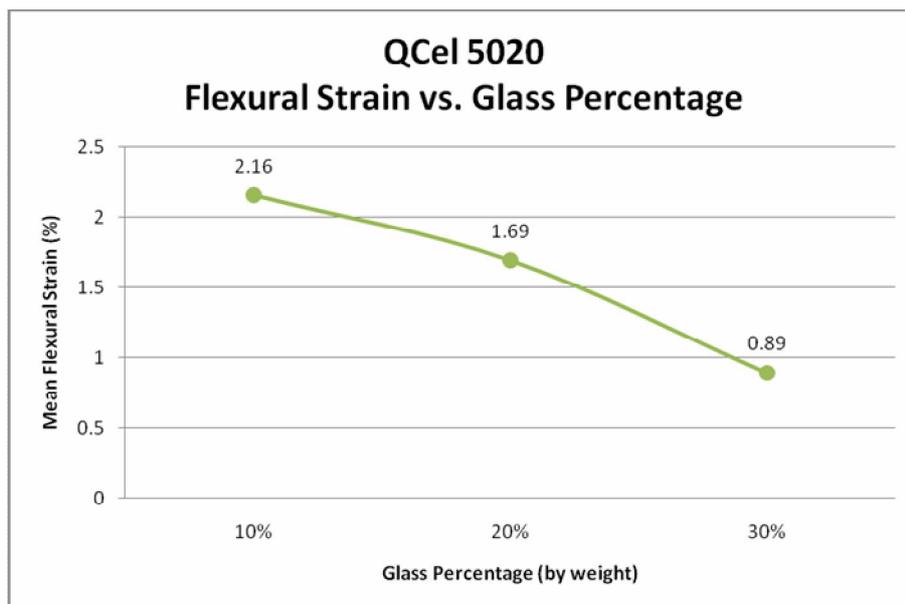
$h$  = Thickness of the specimen (mm)

$L$  = Span (mm)

	Glass Percentage		
	10%	20%	30%
Specimen 1	1.764	1.593	1.272
Specimen 2	1.771	1.309	0.715
Specimen 3	1.999	1.647	0.676
Specimen 4	2.687	1.93	0.904
Specimen 5	2.462	1.84	0.00
Specimen 6	2.255	1.839	0.00
Avg. Flexural Strain (%)	2.156	1.693	0.892

Table 4: Flexural strain for QCel 5020 specimens at different percentages.

The above mean flexural strain data was plotted on the graph below, which indicates that the QCel 5020 specimens with 10% glass powder had the highest strain, or in other words, had the highest load bearing capability.



Graph 2: Flexural Strain vs. Glass Percentage (QCel 5020)

Similar tables and graphs for QCel 6019 and Spherichel 60P18 are can be found in Appendix D.

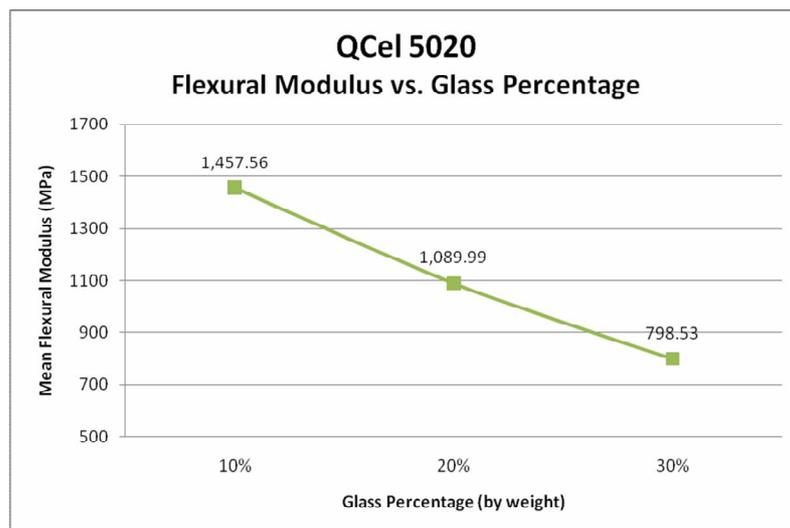
### 4.1.3 Flexural modulus

Another key prameter that was calculated from the bending test was the flexural modulus of the specimens. The UTS machine automatically generates this data, which has been populated in the following table:

	Glass Percentage		
	10%	20%	30%
Specimen 1	1482.134	1202.9	828.146
Specimen 2	1530.132	902.493	789.501
Specimen 3	1724.197	1022.68	775.753
Specimen 4	1197.545	1248.56	800.737
Specimen 5	1260.069	1018.54	0.00
Specimen 6	1551.291	1144.74	0.00
Mean Flexural Modulus (MPa)	1457.561	1089.99	798.534

Table 5: Flexural modulus for QCel 5020 specimens at different percentages.

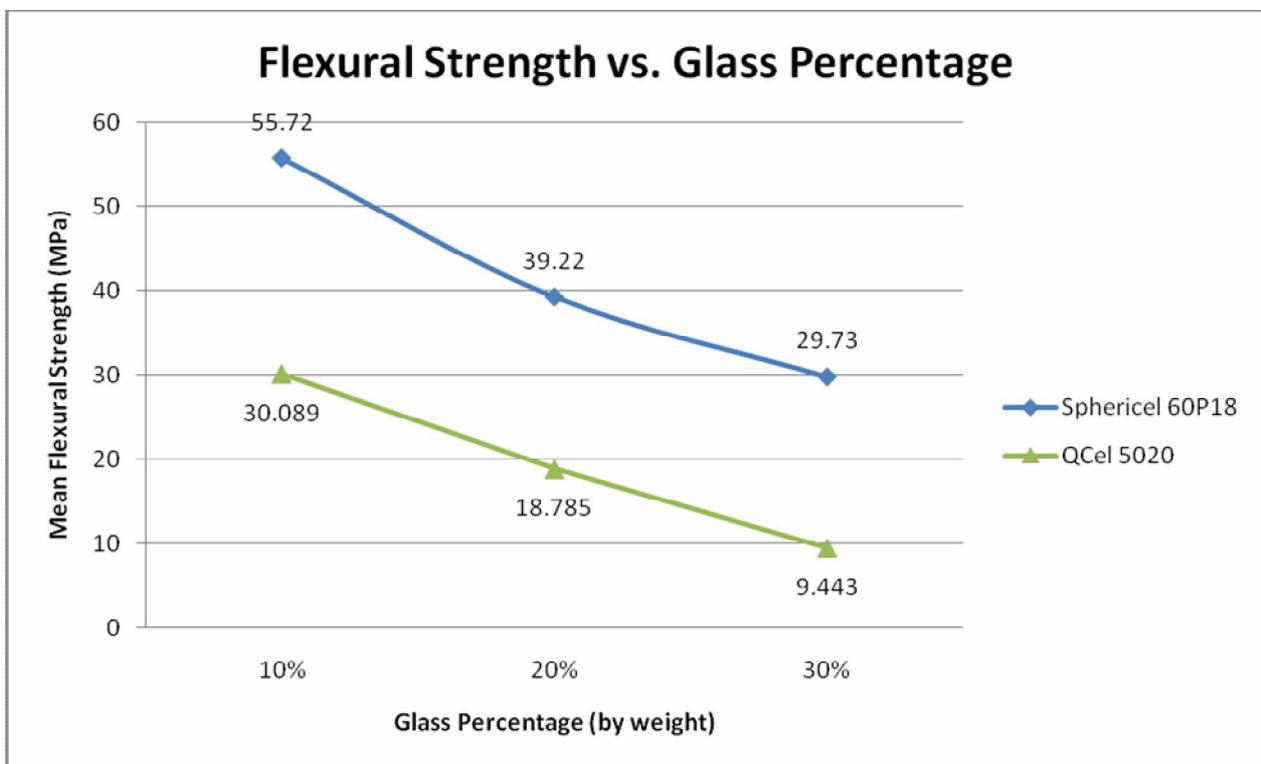
The above table has been visually represented in the following graph:



Graph 3: Flexural Modulus vs. Glass Percentage (QCel 5020)

## 4.2 Observations

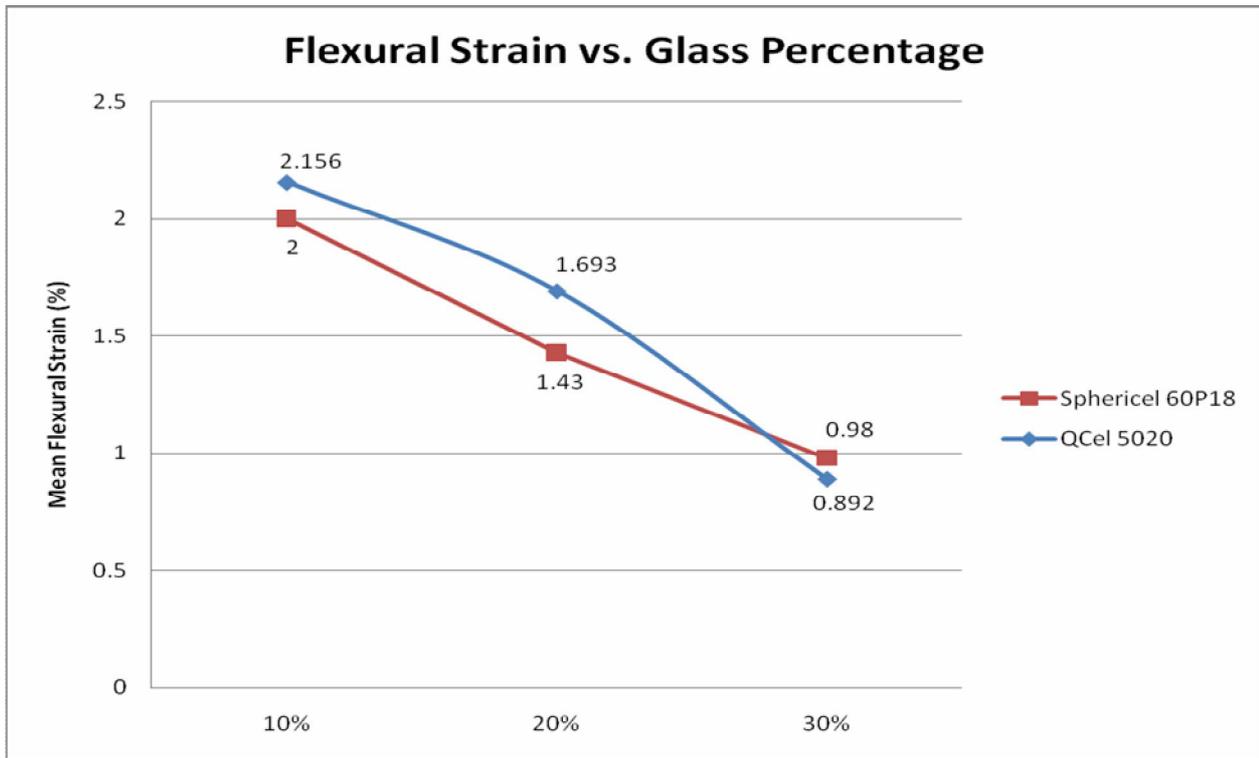
From analysing the data, it is evident that the data between Spherichel 60P18 and QCel 5020 can be compared for flexural strength, strain and modulus. QCel 6019 have been excluded from this comparison as specimens for that type of glass could not be formed at 30% reinforcement weight ratio. Also, all the aforementioned properties of QCel 5020 and QCel 6019 are quite close (Graph 7, 8 and 9), hence comparing Spherichel 60P18 with QCel 5020 should help reach a conclusion.



Graph 4: Flexural Strength comparison.

The analysis suggests that Spherichel 60P18 at 10% filler ratio by weight of the whole specimen possesses higher flexural strength (Graph 4), flexural strain (Graph 5) and flexural modulus (Graph 6) properties. For example, the flexural strength at 10% filler weight, Spherichel 60P18 displays an average flexural strength of 55.72 MPa, whereas QCel 5020 has average flexural strength of 30.089

MPa, which is almost half (54%) of that of Spherichel 60P18.



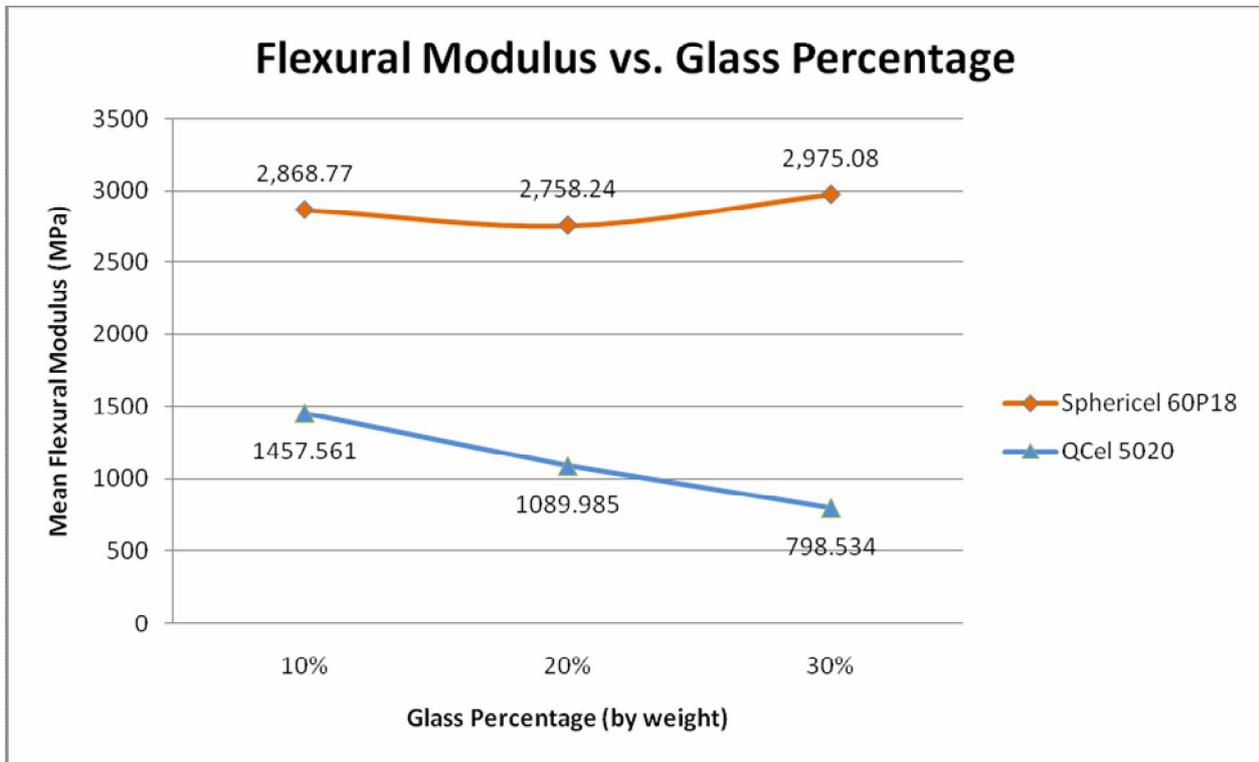
Graph 5: Flexural Strain comparison.

Similarly, the average flexural strain ratio for Spherichel 60P18 is also higher than that of QCel 5020 by 7.8%. This higher average flexural strain is observed upto 20% filler ratio, at which point the average flexural strain of Spherichel 60P18 is 18.4% higher than that of QCel 5020.

This trend starts changing after about 20% filler weight, at which the Spherichel 60P18's average flexural strain sharply declines and reduces by 9.8% when the filler weight becomes 30% of the composite weight. To be noted, the average flexural strain of QCel 5020 does not display any abrupt decline as Spherichel 60P18, but it also almost linearly reduces to 0.98%.

This interesting behaviour indicates that at higher than 25% filler weight, composites of Spherichel

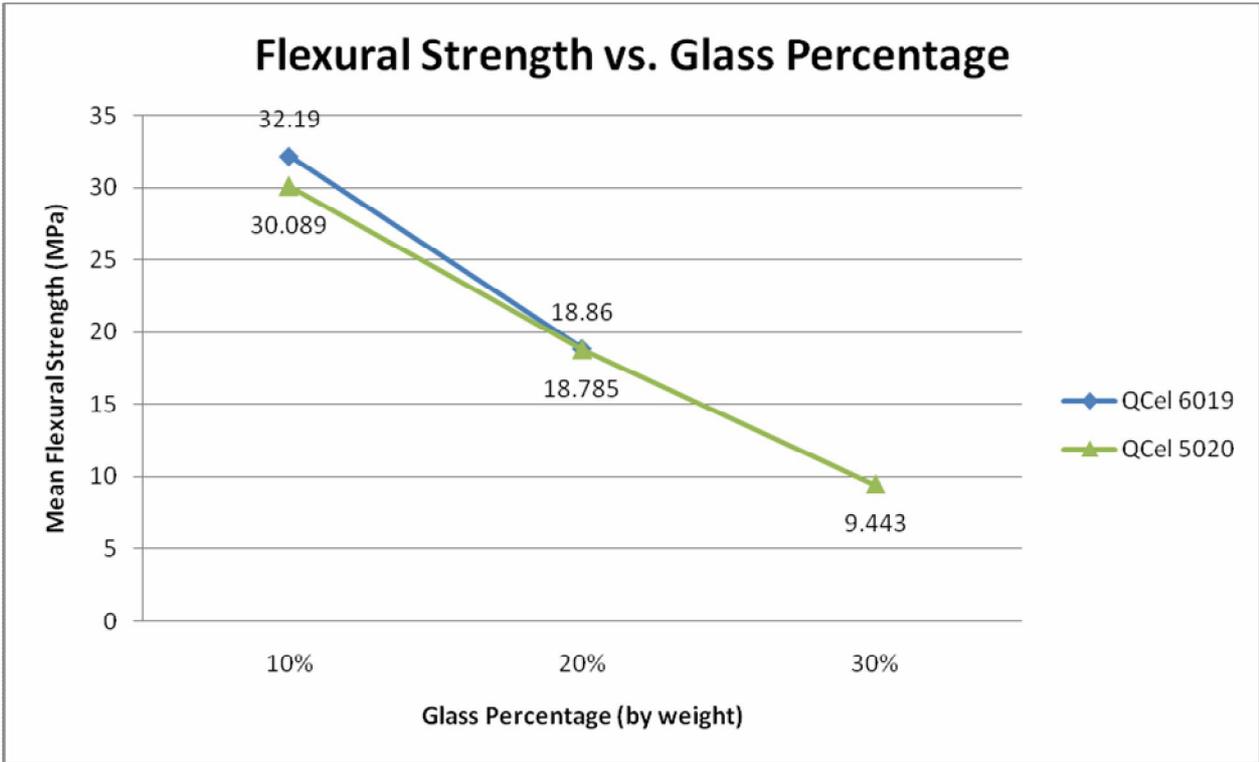
60P18 may start experiencing lower than expected strain, which would increase the flexural modulus at those filler weights.



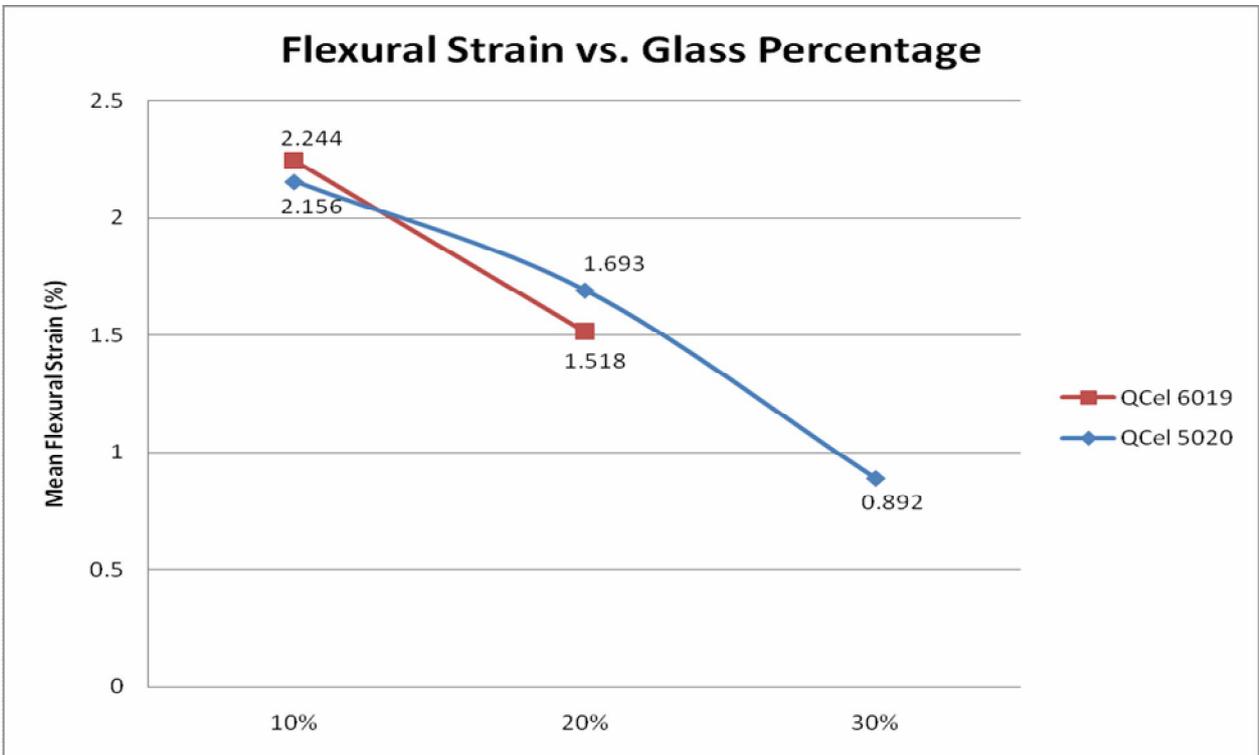
Graph 6: Flexural Modulus comparison.

The graph above supports the observation made in the preceding section, as the average flexural modulus for Spherichel 60P18 has increased by about 7.8%, whereas the average flexural modulus for QCel 5020 linearly decreased as expected.

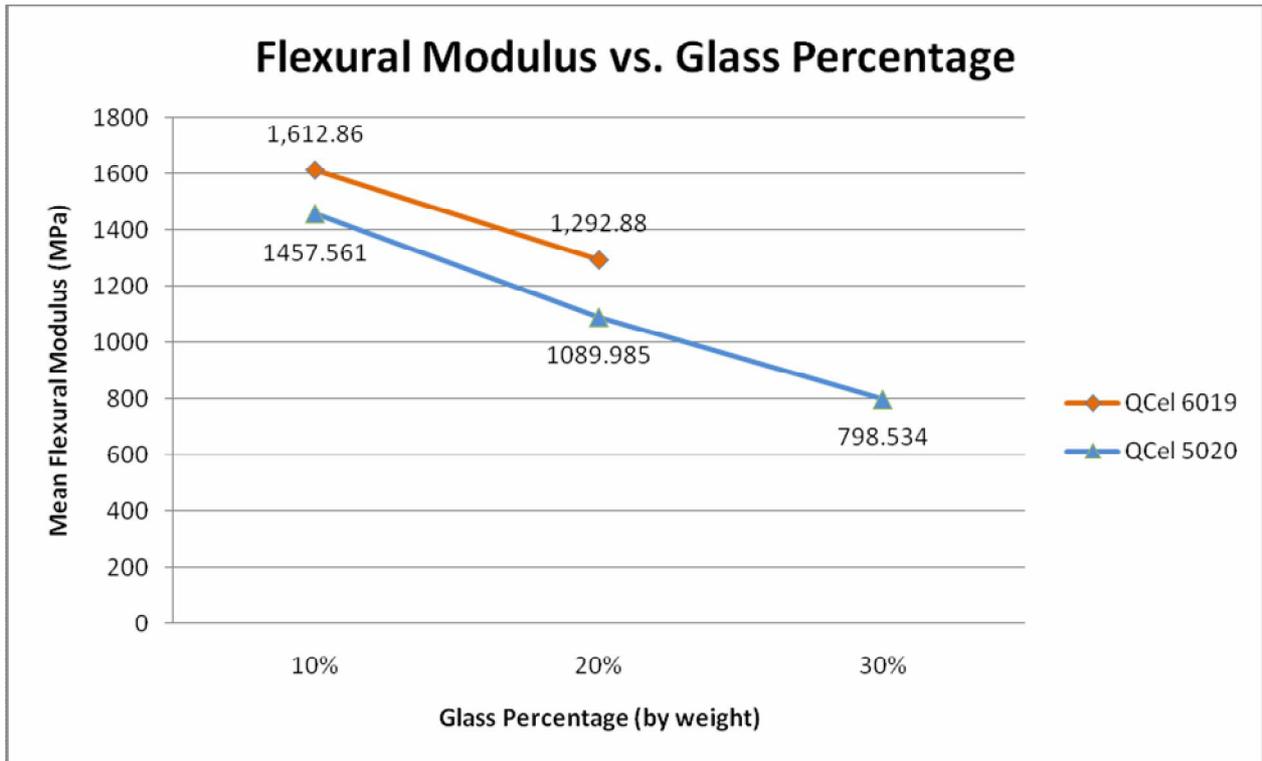
Also, it needs to be restated that the *QCel 6019 specimens could not be formed beyond the filler percentage of 25%*, and hence was not deemed appropriate for inclusion in the above analyses. Nevertheless, the following graphs provided indicated that QCel 5020 and QCel 6019 shared similar flexural properties and was the rational behind the exclusion of QCel 6019.



Graph 7: Flexural Strength comparison between QCel 6019 and QCel 5020.



Graph 8: Flexural Strain comparison between QCel 6019 and QCel 5020.



Graph 9: Flexural Modulus comparison between QCel 6019 and QCel 5020.

By analysing the aforementioned three graphs, it is quite justified to deduce that the flexural properties of QCel 5020 and QCel 6019 are similar, if not the same, but comparing Spherichel 60P18 to QCel 5020 would be a reasonable analysis for obtaining sound results.

#### **4.2.1 SEM observations**

The following SEM images for Spherichel 60P18 at 10% weight (which possesses the highest flexural strength) of the filler indicate that, the amount of porosity in the specimen was quite low due to the availability of VE resin matrix, which in turn suggests that the specimen cured properly and the obtained flexural properties are fairly accurate.

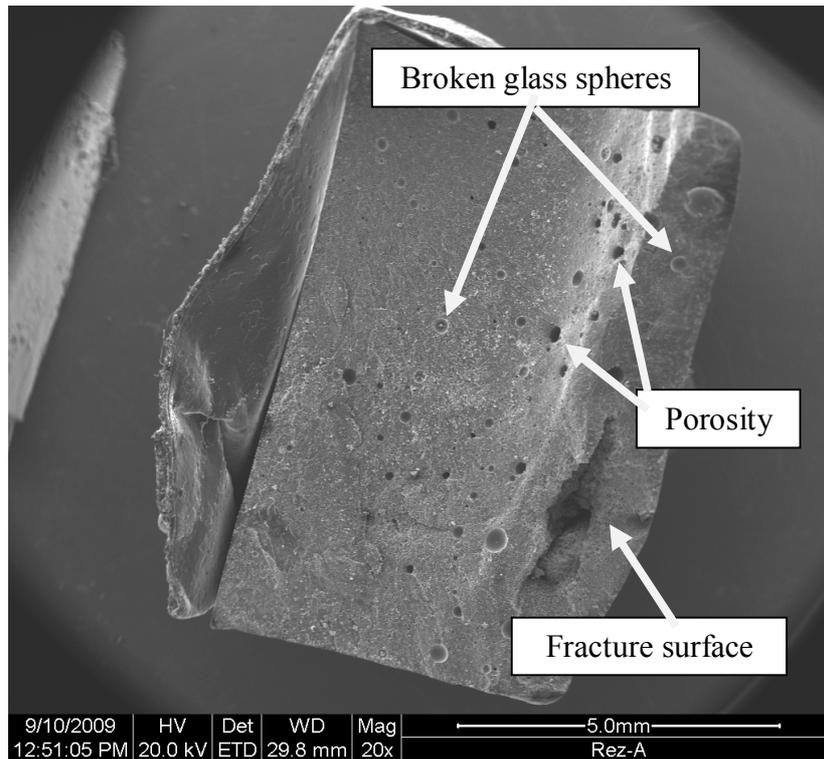


Figure 16: SEM image of composite for Sphericel 60P18 at 10% weight ratio (20 times magnification).

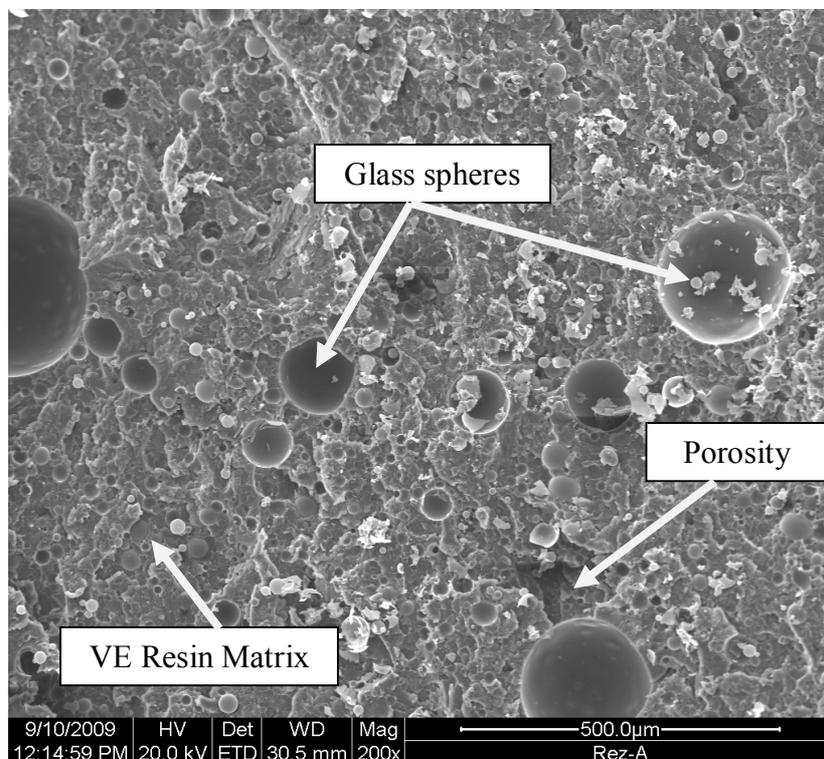


Figure 17: SEM image of composite for Sphericel 60P18 at 10% weight ratio (200 times magnification).

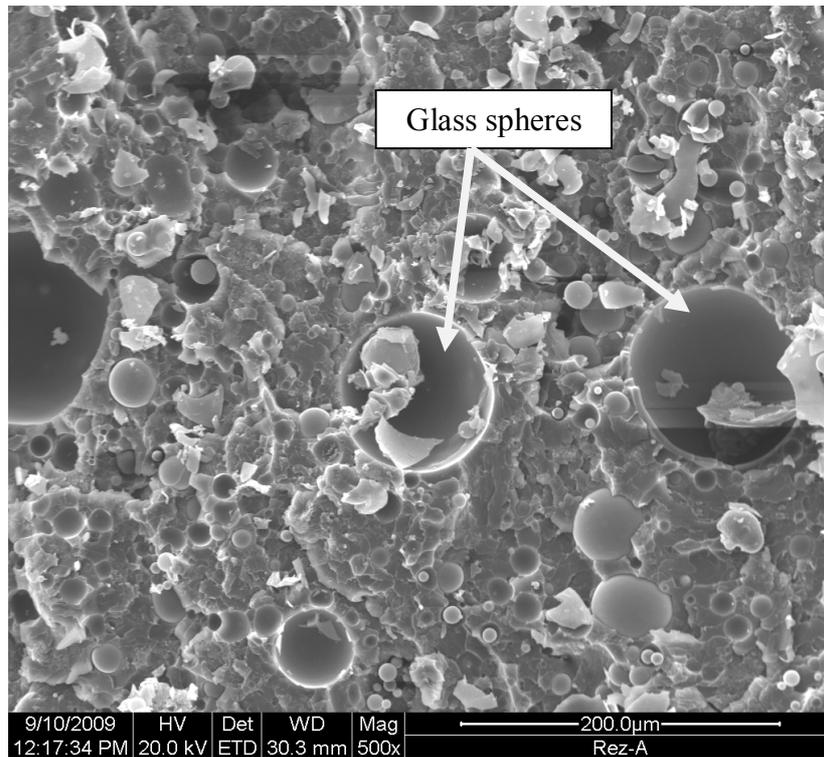


Figure 18: SEM image of composite for Sphericel 60P18 at 10% weight ratio (500 times magnification).

On the other hand, as the glass percentage increased, the flexural strength started to decline. A prime example of low flexural strength specimen is that of QCel 5020 at 30% weight, where the specimen did not have as much VE resin matrix available and was quite porous due to higher mean particle size.

From the figures 19, 20 and 21, it can be clearly observed that the proportion of glass spheres are very high and there is very little room for the VE matrix to wrap around the individual spheres. This would have caused the strength of the material to substantially decrease, as the flexural load would easily transfer to the surface of the hollow spheres and cause stress concentrations. As a result, the spheres would crack once their limit of stress is overcome by the flexural load. Also, at higher glass percentage, the chance of having porosities increase which makes the specimen more fragile.

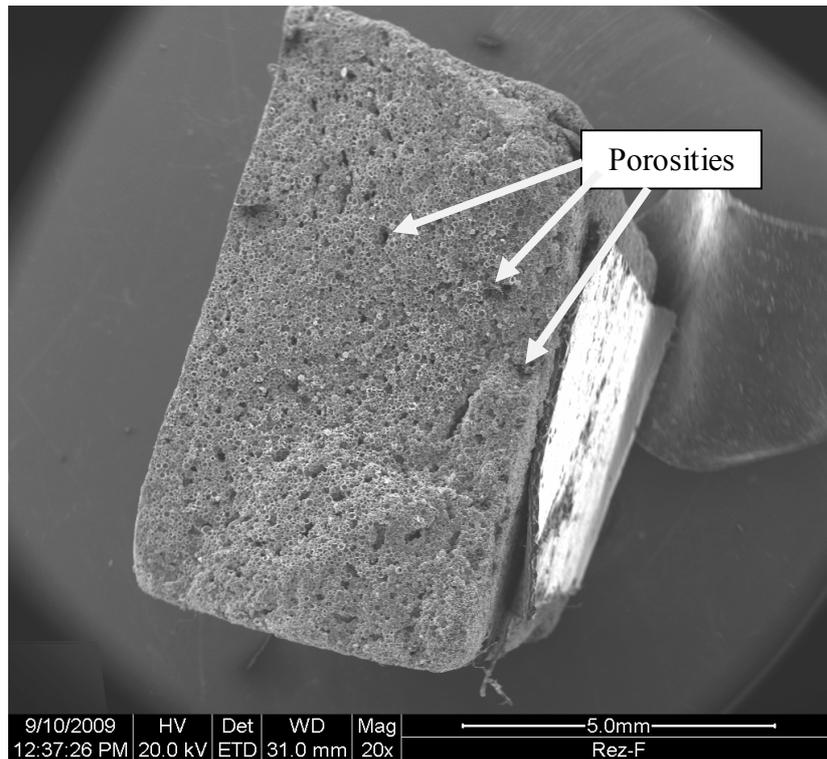


Figure 19: SEM image of composite for QCel 5020 at 10% weight ratio  
(20 times magnification).

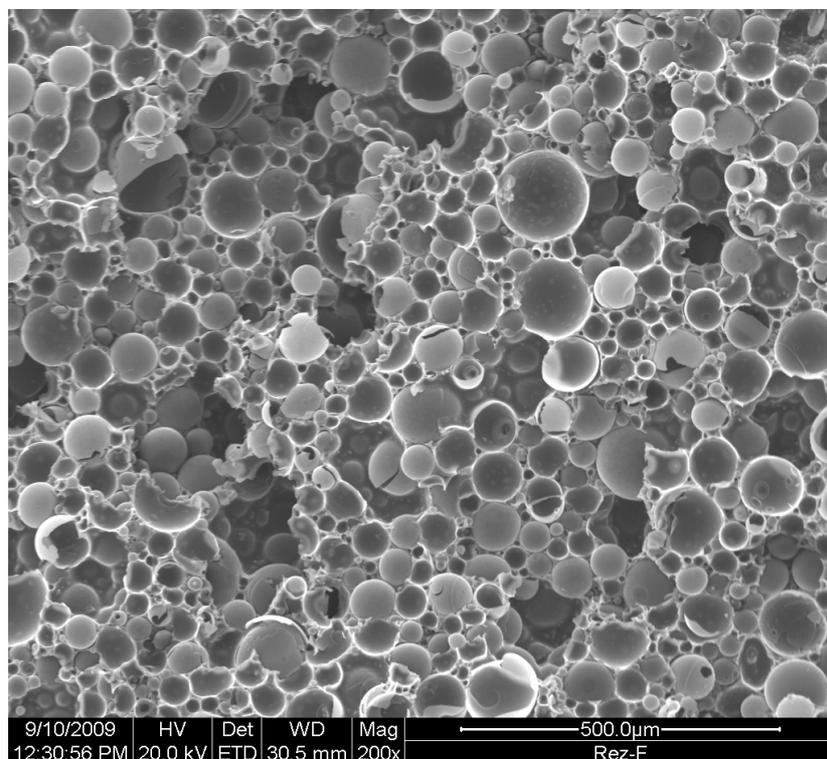


Figure 20: SEM image of composite for QCel 5020 at 10% weight ratio  
(200 times magnification).

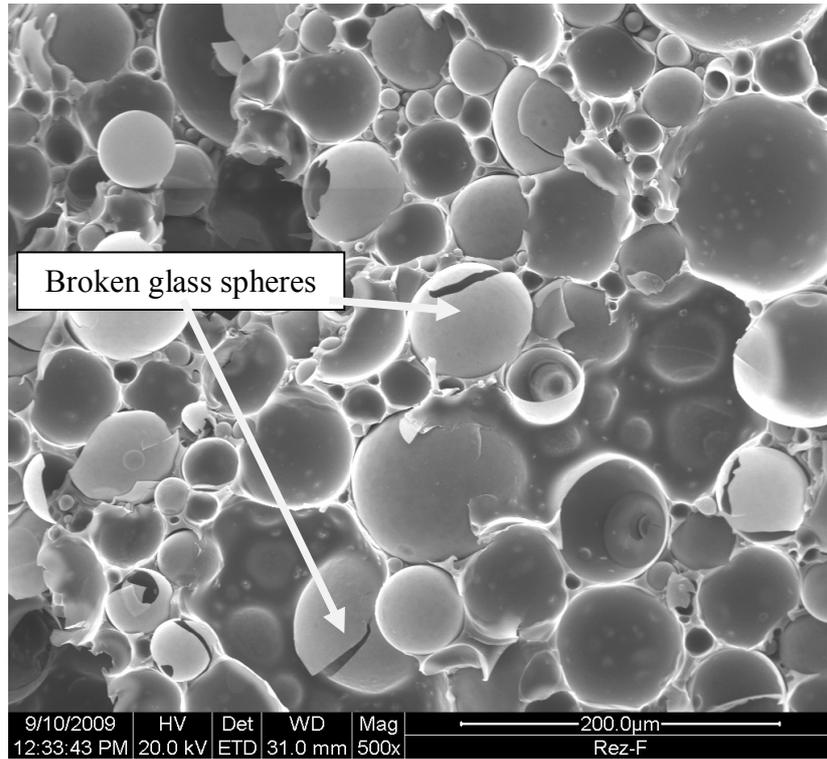


Figure 21: SEM image of composite for QCel 5020 at 10% weight ratio (500 times magnification).

## Chapter 5 Conclusion

This research was conducted to compare the flexural strength (and also flexural strain and modulus as related properties) for Spherichel 60P18, QCel 5020 and QCel 6019 glass powders, and for all types of glass, the composite slurry could not be obtained (i.e. QCel 6019 at 30% filler weight). Still, it was also demonstrated that QCel 5020 and QCel 6019 composites display similar flexural properties with QCel 6019 being slightly higher in mean particle size, flexural strength, strain and modulus values. The conclusion that Spherichel 60P18 at 10% filler weight would demonstrate superior flexural strength and other associated flexural properties among all three (3) types of glass powder would have to be confined to at most 25% filler weight of the composite specimen.

Much experimental information on these three types of glass powder reinforced composites on a comparative level was not available for cross comparison, and hence there is further scope for research on using other resins, catalysts and inclusion of other reinforcement particles along with these reinforcements and at other filler percentages.

The cost justification analysis can be carried out on these composites in order to measure the commercial viability of the whole exercise. Nonetheless, in the event of commercialisation, the research results indicate Spherichel 60P18 at lower filler weight ratio to be a more suitable candidate than the other two types of glass powder using VE as resin and MEKP as catalyst.

## References

3Dchem.com. (2007). *MEKP @ 3Dchem.com*. Available:

<http://www.3dchem.com/moremolecules.asp?ID=313&othername=MEKP>. Last accessed 2 October 2009.

Askeland, D R, *The Science and Engineering of Materials*, Third Edition, Stanley Thornes.

Callister, W D, *Material Science and Engineering: An Introduction*, 7<sup>th</sup> Edition, Wiley International, 2006, pg. 447-449.

Evans, Alexander, San Marchi, Christopher, Mortensen, Andreas, *Metal matrix composites in industry: an introduction and a survey*. 1st edition, Springer 2003, pg 392.

ISO, ISO 14125:1998(E), Fibre reinforced plastic composites— Determination of flexural properties, 1998, International Organization for Standardization, Geneva, Austria.

Jenkins, C H, Editor, *Manual on Experimental Methods for Mechanical Testing of Composites*, Second Edition, The Fairmont Press, 1998.

Kopeliovich, Dr. Dmitri. (2009). *Polymer Matrix Composites (introduction)*. Available: [http://www.substech.com/dokuwiki/doku.php?id=polymer\\_matrix\\_composites\\_introduction](http://www.substech.com/dokuwiki/doku.php?id=polymer_matrix_composites_introduction). Last accessed 14 September 2009.

Ku, H, Trada, N and Samchuk, M, *Flexural Strength of SLG Reinforced Phenol Formaldehyde Composites post-cured in Microwaves*, Key Engineering Materials, 2009.

*Metal matrix composite*. Available: [http://en.wikipedia.org/wiki/Metal\\_matrix\\_composite](http://en.wikipedia.org/wiki/Metal_matrix_composite). Last accessed 12 October 2009.

*Plastics: Information and Properties of Polymeric Materials, Mechanical Engineers' Handbook: Materials and Mechanical Design*, Volume 1, Third Edition. Edited by Myer Kutz Copyright 2006 by John Wiley & Sons, Inc.

Potters Industries, undated, <http://www.pottersbeads.com/markets/polySphericel.asp> <viewed on 27 August 2009>

Potters Industries, undated, <http://www.pottersbeads.com/markets/polycomposites.asp> <viewed on 27 August 2009>

Potters Industries, undated, Material Safety Data Sheet for SPHERICELO Hollow Spheres, pp. 1-4 <viewed on 27 August 2009>.

Shackelford, J F, *Introduction to Material Science for Engineers*, 3<sup>rd</sup> Edition, Macmillan, 1992.

Shekhar, Shashi, *Tensile strength of sawdust reinforced phenolic resin composite materials*, University of Southern Queensland, 2008.

Schaffer, J.P., Saxena, A., Antolovich, S.D., Sanders, Jr., T.H. and Warner, S.B., *The Science and Design of Engineering Materials*, IRWIN [now McGraw-Hill], Chicago. Retrieved from: <http://www.engr.sjsu.edu/WofMatE/Composites.htm>. <viewed on 2 October 2009>.

Strong, A. Brent, *Fundamentals of Composites Manufacturing: Materials, Methods and Applications*, SME, 2008.

Stenzel, Deb, *Lecture 6: The Scanning Electron Microscope*, Queensland University of Technology, Brisbane, 2009.

Swallowe, G M, *Mechanical Properties and Testing of Polymers*, Vol. 3, Kluwer, 1999.

Sweet Composites. (2008). *Methyl Ethyl Ketone Peroxide*. Available: <http://sweetcomposites.com/mekp.html>. Last accessed 1 September 2009.

# Appendix A: Project Specification

**University of Southern Queensland  
Faculty of Engineering and Surveying**

**ENG 4111/4112 Research Project  
PROJECT SPECIFICATION**

**Project title:** Flexural strength of three types of glass powder reinforced Vinyl Ester composites.

**Student:** Rezwanul Huq - 005 002 4547

**Supervisor:** Dr. Harry Ku

**Co-Supervisor:**

**Sponsorship:** Faculty of Engineering & Surveying  
University of Southern Queensland

**Project Synopsis:**

This project aims at analysing 3 (three) different types of glass powder (namely - Q cell 5020, Q cell 6091 and Spherichel 60P18) and their behaviour as filler substance of composites. Filler usage reduces the cost of composites and modifies their structural properties significantly, which is a critical factor in engineering applications. Hence, curing, post-curing and flexural testing would be carried out sequentially on a range of VE resin specimens to evaluate their flexural properties. Further in depth analysis of the findings would be carried out to determine behaviour trends and formulas suitable for theoretical prediction of the filled polymer behaviour.

**Timeline:**

1. Literature reviews with reinforced phenolic composites.  
Begin : 16<sup>th</sup> March 2009  
Completion : 10<sup>th</sup> April 2009  
Approx. Hours : 60 hours
  
2. Determine and design processes (parameters) for testing.  
Begin : 10<sup>th</sup> April 2009

- Completion : 16<sup>th</sup> April 2009  
Approx. Hours : 20 hours
3. Testing Methods and examination of specimens.  
Begin : 21<sup>st</sup> April 2009  
Completion : 30<sup>th</sup> May 2009  
Approx. Hours : 80 hours
4. Analysis of results.  
Begin : 2<sup>nd</sup> June 2009  
Completion : 27<sup>th</sup> June 2009  
Approx. Hours : 50 hours
5. Draw up conclusions.  
Begin : 21<sup>st</sup> July 2009  
Completion : 8<sup>th</sup> August 2009  
Approx. Hours : 40 hours
6. Discussion for the thesis outline with supervisors.  
Begin : 11<sup>th</sup> August 2009  
Completion : 22<sup>nd</sup> August 2009  
Approx. Hours : 10 hours
7. Thesis initial drafting – each chapter in draft form and shown to supervisors.  
Begin : 25<sup>th</sup> August 2009  
Completion : 26<sup>th</sup> September 2009  
Approx. Hours : 60 hours
8. Final draft of thesis, to incorporate modifications suggested by supervisor.  
Begin : 29<sup>th</sup> September 2009  
Completion : 10<sup>th</sup> October 2009  
Approx. Hours : 20 hours

9. Complete the thesis in requested format.

Begin : 13<sup>th</sup> October 2009

Completion : 28<sup>th</sup> October 2009

Approx. Hours : 20 hours

AGREED:

\_\_\_\_\_ (student)

\_\_\_\_\_ (Supervisor)

Date: \_\_\_/\_\_\_/\_\_\_

## Appendix B: Composite formation datasheet

Glass type: QCel 5020

Glass percentage: 10%

Composite (g)	Glass Powder (g)	Vinyl Ester Resin + MEKP (g)	Vinyl Ester Resin (g)	MEKP (g)
150	15	135	132.3	2.7

Glass percentage: 20%

Composite (g)	Glass Powder (g)	Vinyl Ester Resin + MEKP (g)	Vinyl Ester Resin (g)	MEKP (g)
150	30	120	117.6	2.4

Glass percentage: 30%

Composite (g)	Glass Powder (g)	Vinyl Ester Resin + LA (g)	Vinyl Ester Resin (g)	LA (g)
150	45	105	103.4	1.6

**Glass type: QCel 6019**

Glass percentage: 10%

Composite (g)	Glass Powder (g)	Vinyl Ester Resin + LA (g)	Vinyl Ester Resin (g)	LA (g)
150	15	135	132.97	2.03

Glass percentage: 20%

Composite (g)	Glass Powder (g)	Vinyl Ester Resin + LA (g)	Vinyl Ester Resin (g)	LA (g)
150	30	120	118.2	1.8

Glass percentage: 25%

Composite (g)	Glass Powder (g)	Vinyl Ester Resin + LA (g)	Vinyl Ester Resin (g)	LA (g)
150	37.5	112.5	110.8	1.7

**Glass type: Spherical 60P18**

Glass percentage: 10%

Composite (g)	Glass Powder (g)	Vinyl Ester Resin + MEKP (g)	Vinyl Ester Resin (g)	MEKP (g)
150	15	135	132.3	2.7

Glass percentage: 20%

Composite (g)	Glass Powder (g)	Vinyl Ester Resin + MEKP (g)	Vinyl Ester Resin (g)	MEKP (g)
150	30	120	117.6	2.4

Glass percentage: 30%

Composite (g)	Glass Powder (g)	Vinyl Ester Resin + MEKP (g)	Vinyl Ester Resin (g)	MEKP (g)
150	45	105	102.9	2.1

## Appendix C: UTS Data and Plots

3/09/2009

Sample ID: 60P18-10%-6.mss      Test Date:      3/09/2009

Method:      MMT - Flexural Test (ISO 14125).msm      Operator: Mohan Trada

### Specimen Results:

Specimen #	Width mm	Thickness mm	Modulus MPa	Peak Load N	UTS MPa	Load At Break N	Stress At Break MPa
1	9.69	5.14	2669.216	154.927	58.1	149.388	56.019
2	9.65	5.23	2492.492	101.886	37.1	100.711	36.628
3	9.64	5.57	2912.739	172.887	55.5	172.887	55.494
4	9.85	5.63	3079.13	190.847	58.7	184.973	56.876
5	9.95	5.67	2878.364	204.108	61.3	201.422	60.449
6	9.75	5.88	3180.674	243.049	69.2	241.707	68.834
Mean	9.755	5.520	2868.769	177.951	56.6	175.181	55.717
Std. Dev.	0.123	0.281	254.938	47.812	10.7	47.805	10.586

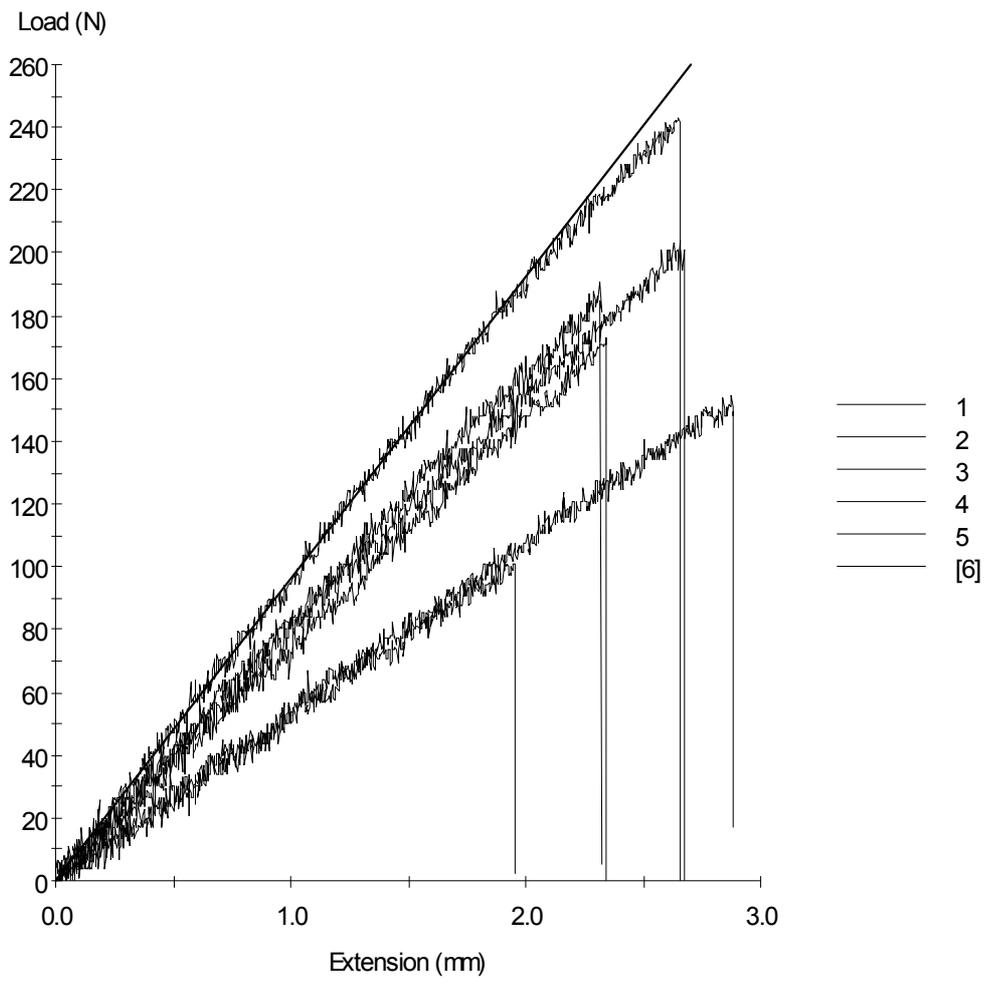
Specimen #	Strain at Break %
1	2.171
2	1.911
3	2.219
4	1.913
5	1.501
6	2.287
Mean	2.000
Std. Dev.	0.291

**Calculation Inputs:**

Name	Value	Units
Break Marker Drop	6.0	%
Break Marker Elongation	0.010	mm
Slack Pre-Load	4.448	N
Slope Segment Length	5.000	%
Span	64.000	mm
Strain Point 1	0.05	%
Strain Point 2	0.250	%
Strain Point 3	0.020	mm/mm
Yield Angle	0.000	rad
Yield Offset	0.002	mm/mm
Yield Segment Length	2.0	%

**Test Inputs:**

Name	Value	Units
Break Sensitivity	80	%
Break Threshold	40.000	N
DataAcqRate	5.0	Hz
Extension Endpoint	25.400	mm
Initial Speed	1.0	mm/min
Load Endpoint	4448	N
Outer Loop Rate	100	Hz
Secondary Speed	1.000	mm/min
Strain Endpoint	0.100	mm/mm



8/09/2009

Sample ID: 60P18-20%-6.mss

Test Date:

8/09/2009

Method: MMT - Flexural Test (ISO 14125).msm

Operator: Mohan Trada

**Specimen Results:**

Specimen #	Width mm	Thickness mm	Modulus MPa	Peak Load N	UTS MPa	Load At Break N	Stress At Break MPa
1	9.64	5.28	2948.798	127.567	45.6	126.225	45.089
2	9.7	5.44	2490.369	110.782	37	108.936	36.431
3	9.78	5.5	2869.701	102.725	33.3	102.725	33.334
4	9.72	5.61	2479.943	116.153	36.5	110.782	34.766
5	9.76	5.61	3041.571	124.042	38.8	120.853	37.771
6	9.69	6.02	2719.032	179.937	49.2	175.237	47.905
Mean	9.715	5.577	2758.236	126.868	40.1	124.126	39.216
Std. Dev.	0.050	0.250	236.539	27.500	6.0	26.436	5.903

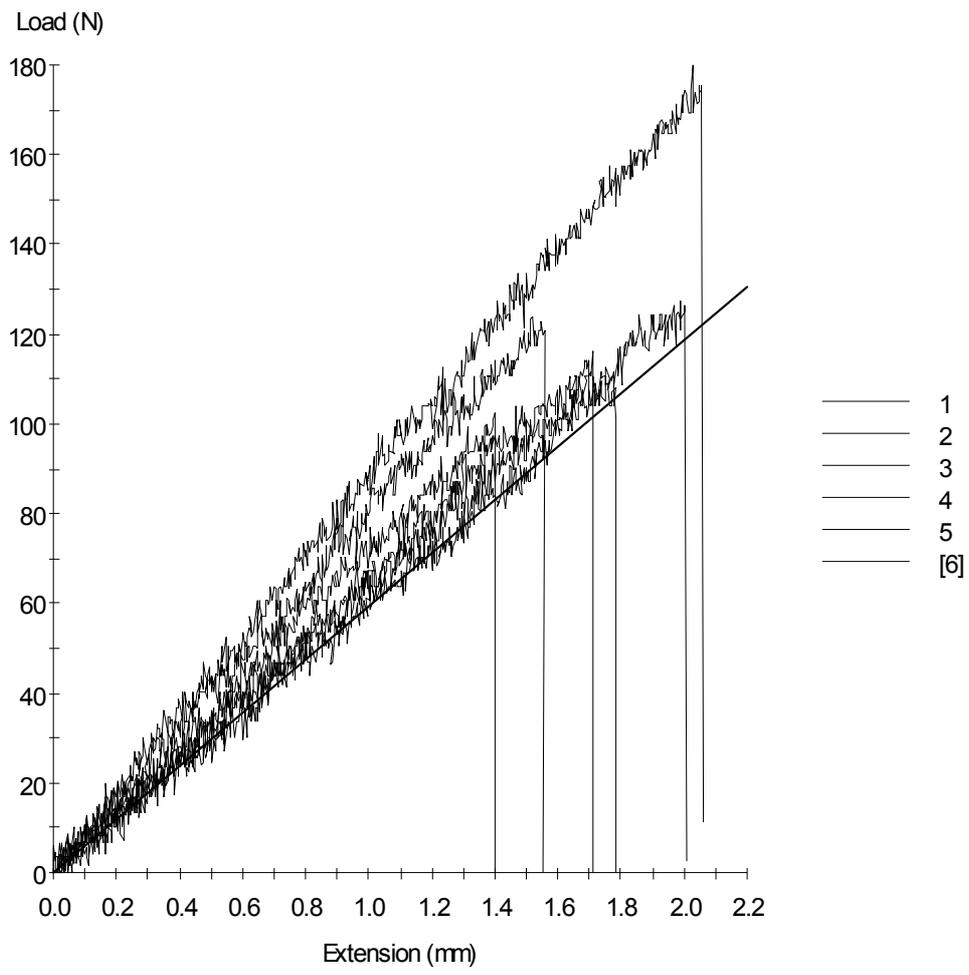
Specimen #	Strain at Break %
1	1.409
2	1.279
3	1.126
4	1.813
5	1.549
6	1.401
Mean	1.430
Std. Dev.	0.235

**Calculation Inputs:**

Name	Value	Units
Break Marker Drop	6.0	%
Break Marker Elongation	0.010	mm
Slack Pre-Load	4.448	N
Slope Segment Length	5.000	%
Span	64.000	mm
Strain Point 1	0.05	%
Strain Point 2	0.250	%
Strain Point 3	0.020	mm/mm
Yield Angle	0.000	rad
Yield Offset	0.002	mm/mm
Yield Segment Length	2.0	%

**Test Inputs:**

Name	Value	Units
Break Sensitivity	80	%
Break Threshold	40.000	N
DataAcqRate	5.0	Hz
Extension Endpoint	25.400	mm
Initial Speed	1.0	mm/min
Load Endpoint	4448	N
Outer Loop Rate	100	Hz
Secondary Speed	1.000	mm/min
Strain Endpoint	0.100	mm/mm



8/09/2009

Sample ID: 60P18-30%-6.mss

Test Date:

8/09/2009

Method: MMT - Flexural Test (ISO 14125).msm

Operator: Mohan Trada

**Specimen Results:**

Specimen #	Width mm	Thickness mm	Modulus MPa	Peak Load N	UTS MPa	Load At Break N	Stress At Break MPa
1	9.870	5.690	3212.111	117.496	35.3	114.139	34.290
2	9.860	5.560	2194.232	73.855	23.3	73.855	23.261
3	9.810	5.950	3530.041	122.196	33.8	120.853	33.406
4	10.050	5.690	2996.396	106.418	31.4	105.579	31.150
5	10.120	6.090	3125.777	104.068	26.6	104.068	26.618
6	10.060	5.500	2791.914	93.997	29.7	93.997	29.653
Mean	9.962	5.747	2975.079	103.005	30.0	102.082	29.730
Std. Dev.	0.130	0.229	454.005	17.447	4.5	16.589	4.192

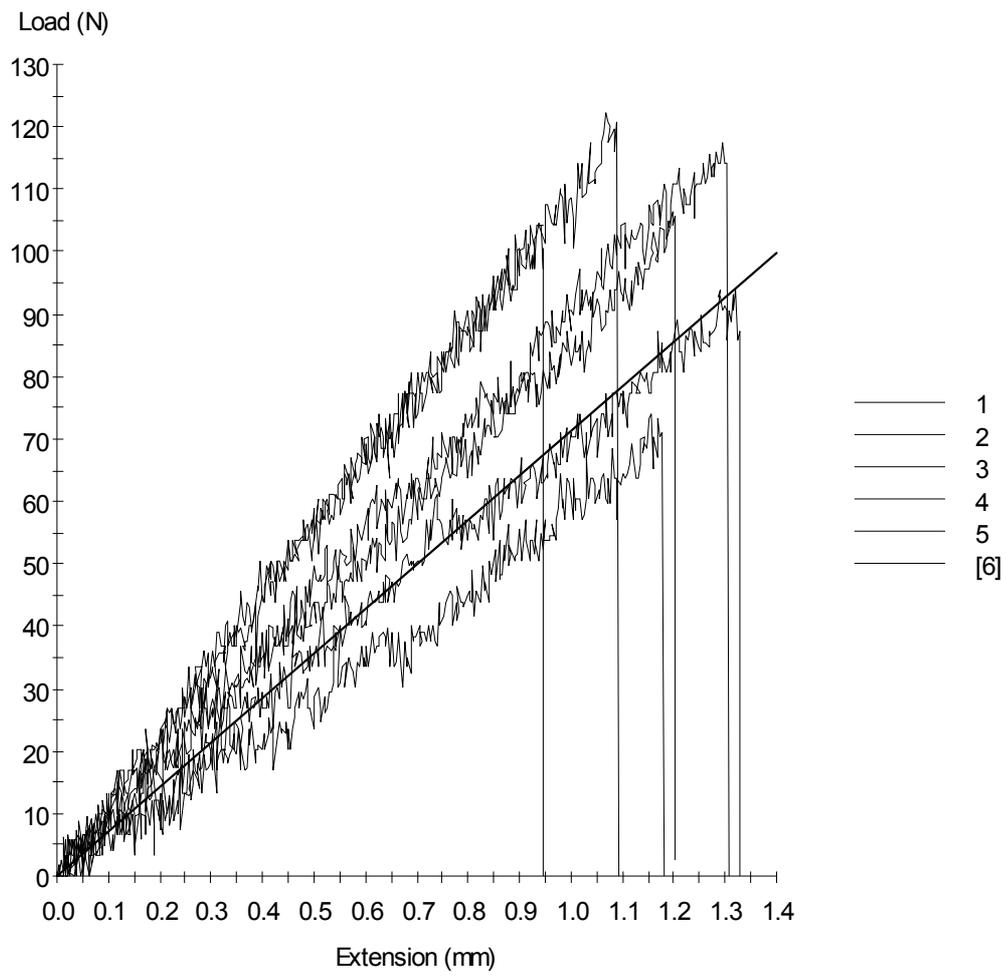
Specimen #	Strain at Break %
1	1.087
2	0.940
3	0.949
4	1.002
5	0.840
6	1.064
Mean	0.980
Std. Dev.	0.091

**Calculation Inputs:**

Name	Value	Units
Break Marker Drop	6.0	%
Break Marker Elongation	0.010	mm
Slack Pre-Load	4.448	N
Slope Segment Length	5.000	%
Span	64.000	mm
Strain Point 1	0.05	%
Strain Point 2	0.250	%
Strain Point 3	0.020	mm/mm
Yield Angle	0.000	rad
Yield Offset	0.002	mm/mm
Yield Segment Length	2.0	%

**Test Inputs:**

Name	Value	Units
Break Sensitivity	80	%
Break Threshold	40.000	N
DataAcqRate	5.0	Hz
Extension Endpoint	25.400	mm
Initial Speed	1.0	mm/min
Load Endpoint	4448	N
Outer Loop Rate	100	Hz
Secondary Speed	1.000	mm/min
Strain Endpoint	0.100	mm/mm



3/09/2009

Sample ID: 6019-10%-6.mss

Test Date:

3/09/2009

Method: MMT - Flexural Test (ISO 14125).msm

Operator: Mohan Trada

**Specimen Results:**

Specimen #	Width mm	Thickness mm	Modulus MPa	Peak Load N	UTS MPa	Load At Break N	Stress At Break MPa
1	9.950	5.470	1705.445	97.186	31.3	97.186	31.338
2	9.820	5.440	1536.702	97.354	32.2	93.997	31.051
3	9.700	5.470	1747.773	99.368	32.9	97.354	32.202
4	9.850	5.430	1840.880	110.111	36.4	104.068	34.400
5	9.830	5.480	1788.958	122.868	40.0	122.196	39.739
6	9.750	5.880	1057.421	85.604	24.4	85.604	24.379
Mean	9.817	5.528	1612.863	102.082	32.8	100.068	32.185
Std. Dev.	0.086	0.173	291.235	12.819	5.2	12.389	4.998

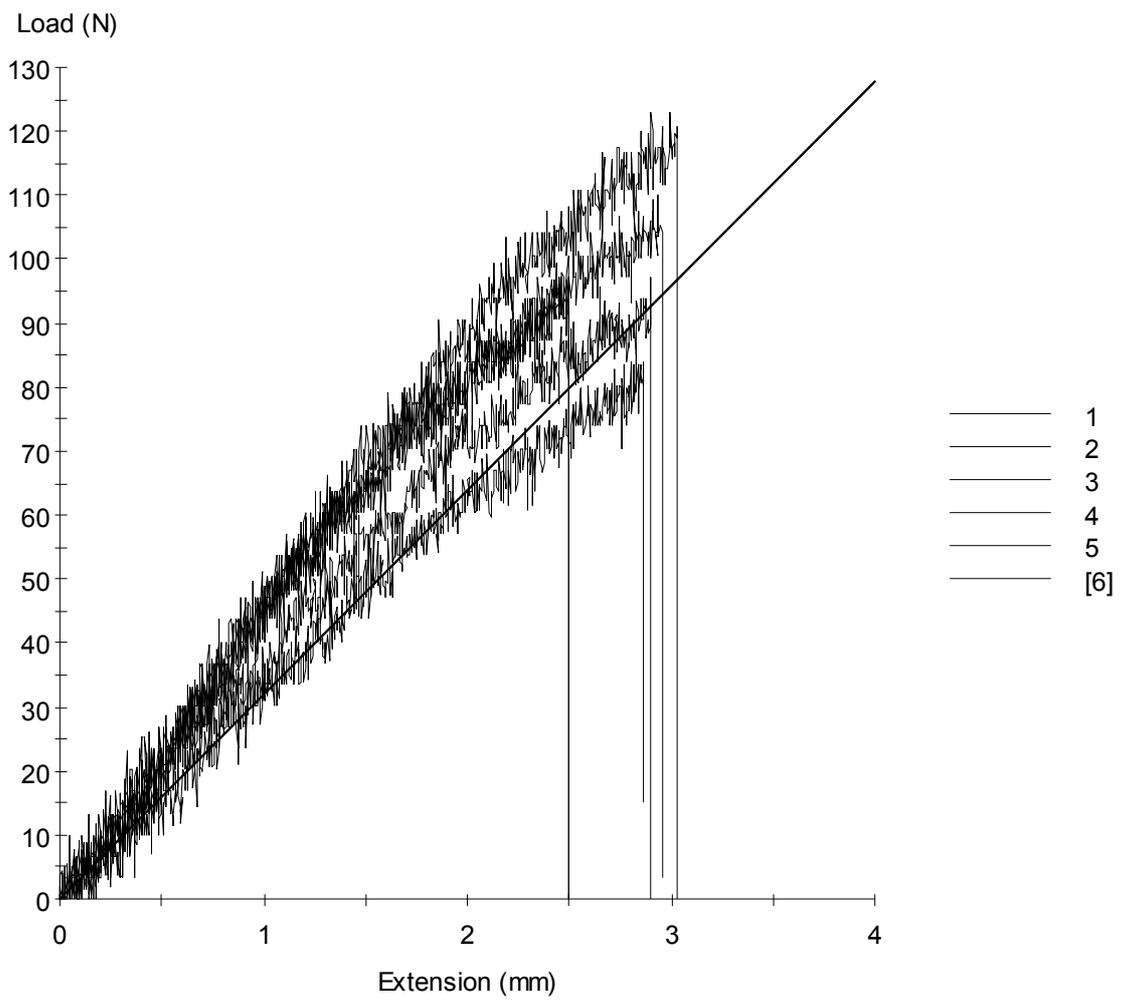
Specimen #	Strain at Break %
1	2.000
2	2.310
3	1.967
4	2.338
5	2.408
6	2.439
Mean	2.244
Std. Dev.	0.207

**Calculation Inputs:**

Name	Value	Units
Break Marker Drop	6.0	%
Break Marker Elongation	0.010	mm
Slack Pre-Load	4.448	N
Slope Segment Length	5.000	%
Span	64.000	mm
Strain Point 1	0.05	%
Strain Point 2	0.250	%
Strain Point 3	0.020	mm/mm
Yield Angle	0.000	rad
Yield Offset	0.002	mm/mm
Yield Segment Length	2.0	%

**Test Inputs:**

Name	Value	Units
Break Sensitivity	80	%
Break Threshold	40.000	N
DataAcqRate	5.0	Hz
Extension Endpoint	25.400	mm
Initial Speed	1.0	mm/min
Load Endpoint	4448	N
Outer Loop Rate	100	Hz
Secondary Speed	1.000	mm/min
Strain Endpoint	0.100	mm/mm



3/09/2009

Sample ID: 6019-20%-6.mss

Test Date:

3/09/2009

Method: MMT - Flexural Test (ISO 14125).msm

Operator: Mohan Trada

**Specimen Results:**

Specimen #	Width mm	Thickness mm	Modulus MPa	Peak Load N	UTS MPa	Load At Break N	Stress At Break MPa
1	9.860	5.400	1436.371	59.923	20.0	59.923	20.008
2	9.840	5.440	1226.992	55.727	18.4	53.713	17.707
3	9.750	5.160	1129.513	56.734	21.0	56.734	20.980
4	9.900	5.600	1542.389	67.141	20.8	64.119	19.827
5	10.010	6.390	1220.432	87.283	20.5	83.926	19.712
6	9.850	5.540	1201.600	47.670	15.1	46.999	14.924
Mean	9.868	5.588	1292.883	62.413	19.3	60.902	18.860
Std. Dev.	0.085	0.421	159.659	13.721	2.2	12.679	2.204

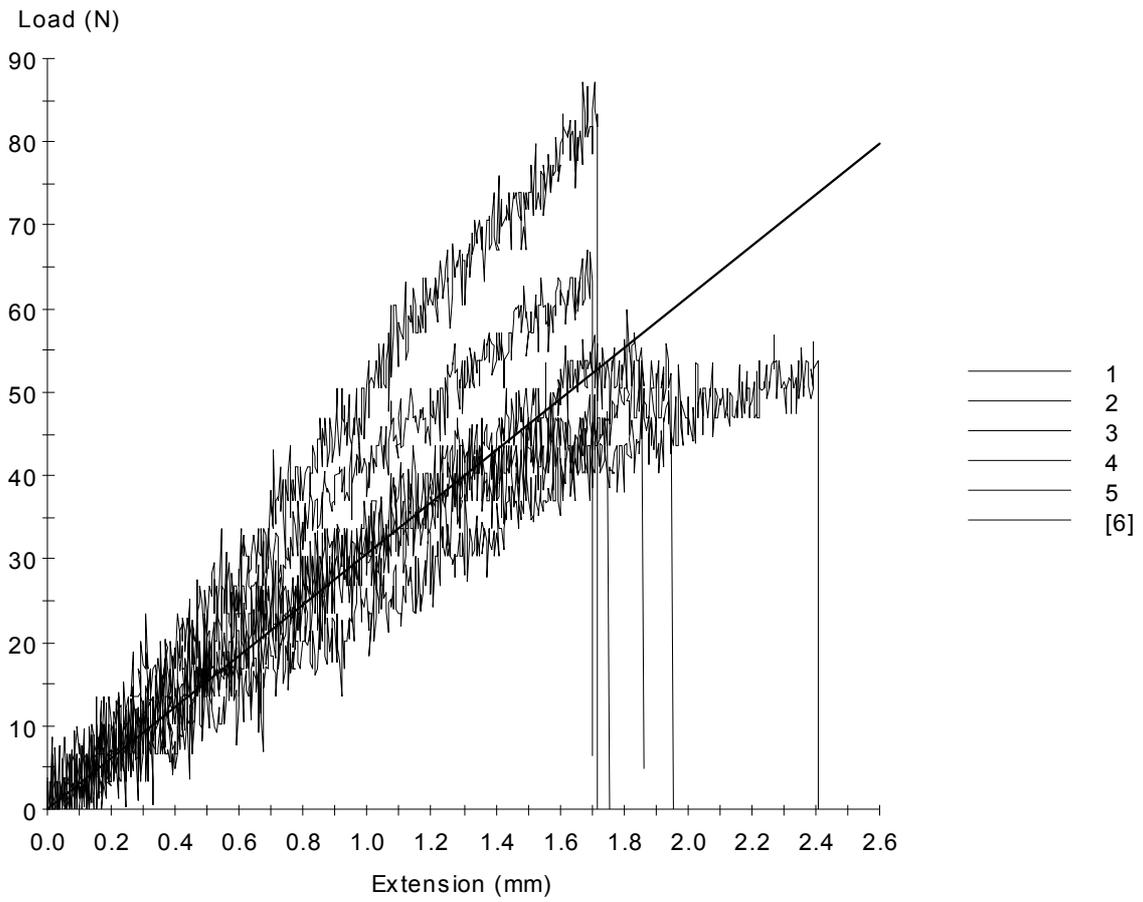
Specimen #	Strain at Break %
1	1.434
2	1.546
3	1.716
4	1.387
5	1.603
6	1.421
Mean	1.518
Std. Dev.	0.127

**Calculation Inputs:**

Name	Value	Units
Break Marker Drop	6.0	%
Break Marker Elongation	0.010	mm
Slack Pre-Load	4.448	N
Slope Segment Length	5.000	%
Span	64.000	mm
Strain Point 1	0.05	%
Strain Point 2	0.250	%
Strain Point 3	0.020	mm/mm
Yield Angle	0.000	rad
Yield Offset	0.002	mm/mm
Yield Segment Length	2.0	%

**Test Inputs:**

Name	Value	Units
Break Sensitivity	80	%
Break Threshold	40.000	N
DataAcqRate	5.0	Hz
Extension Endpoint	25.400	mm
Initial Speed	1.0	mm/min
Load Endpoint	4448	N
Outer Loop Rate	100	Hz
Secondary Speed	1.000	mm/min
Strain Endpoint	0.100	mm/mm



28/08/2009

Sample ID: Rez-6019-25%.mss

Test Date:

28/08/2009

Method: MMT - Flexural Test (ISO 14125).msm

Operator: Mohan Trada

**Specimen Results:**

Specimen #	Width mm	Thickness mm	Modulus MPa	Peak Load N	UTS MPa	Load At Break N	Stress At Break MPa
1	9.840	5.250	1281.867	49.013	17.3	49.013	17.349
2	9.780	5.290	1501.322	53.713	18.8	53.713	18.841
3	9.610	5.350	1103.555	36.927	12.9	36.927	12.888
4	9.520	5.320	1219.375	43.641	15.5	43.641	15.549
5	9.870	5.670	1095.549	44.313	13.4	42.970	13.000
6	9.890	5.450	1134.584	40.284	13.2	40.284	13.165
Mean	9.752	5.388	1222.709	44.649	15.2	44.425	15.132
Std. Dev.	0.152	0.154	154.447	6.016	2.5	6.056	2.541

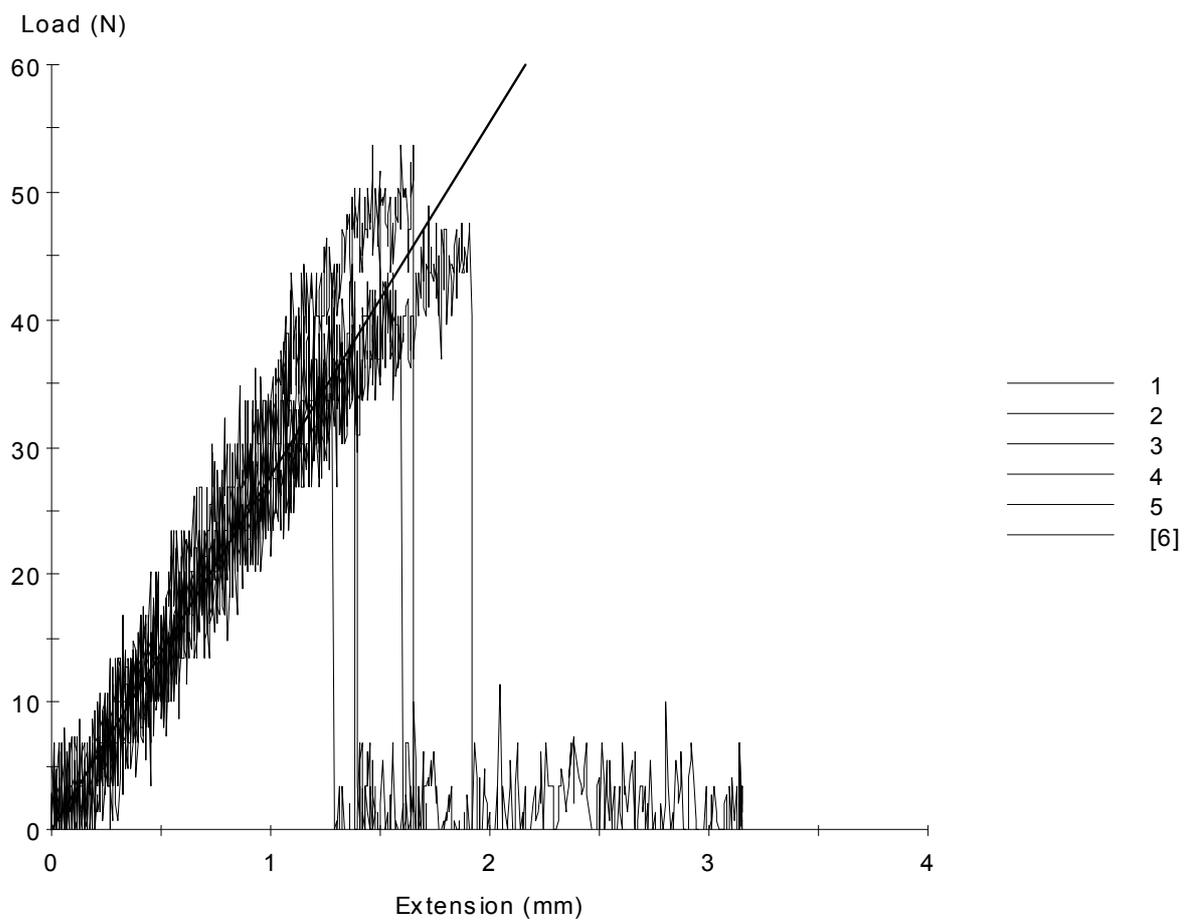
Specimen #	Strain at Break %
1	1.319
2	1.239
3	0.827
4	1.193
5	1.138
6	0.982
Mean	1.116
Std. Dev.	0.181

**Calculation Inputs:**

Name	Value	Units
Break Marker Drop	6.0	%
Break Marker Elongation	0.010	mm
Slack Pre-Load	4.448	N
Slope Segment Length	5.000	%
Span	64.000	mm
Strain Point 1	0.05	%
Strain Point 2	0.250	%
Strain Point 3	0.020	mm/mm
Yield Angle	0.000	rad
Yield Offset	0.002	mm/mm
Yield Segment Length	2.0	%

**Test Inputs:**

Name	Value	Units
Break Sensitivity	80	%
Break Threshold	40.000	N
DataAcqRate	5.0	Hz
Extension Endpoint	25.400	mm
Initial Speed	1.0	mm/min
Load Endpoint	4448	N
Outer Loop Rate	100	Hz
Secondary Speed	1.000	mm/min
Strain Endpoint	0.100	mm/mm



9/09/2009

Sample ID: QCel 5020-10%-6.mss Test Date: 9/09/2009

Method: MMT - Flexural Test (ISO 14125).msm Operator: Mohan Trada

**Specimen Results:**

Specimen #	Width mm	Thickness mm	Modulus MPa	Peak Load N	UTS MPa	Load At Break N	Stress At Break MPa
1	9.890	5.340	1482.134	77.212	26.3	73.183	24.912
2	9.810	5.320	1530.132	80.065	27.7	80.065	27.684
3	9.880	5.730	1724.197	110.782	32.8	108.097	31.990
4	9.970	5.450	1197.545	93.326	30.3	93.326	30.254
5	9.870	5.370	1260.069	91.983	31.0	91.983	31.025
6	9.900	5.200	1551.291	96.683	34.7	96.683	34.672
Mean	9.887	5.402	1457.561	91.675	30.5	90.556	30.089
Std. Dev.	0.052	0.180	196.144	12.140	3.1	12.384	3.409

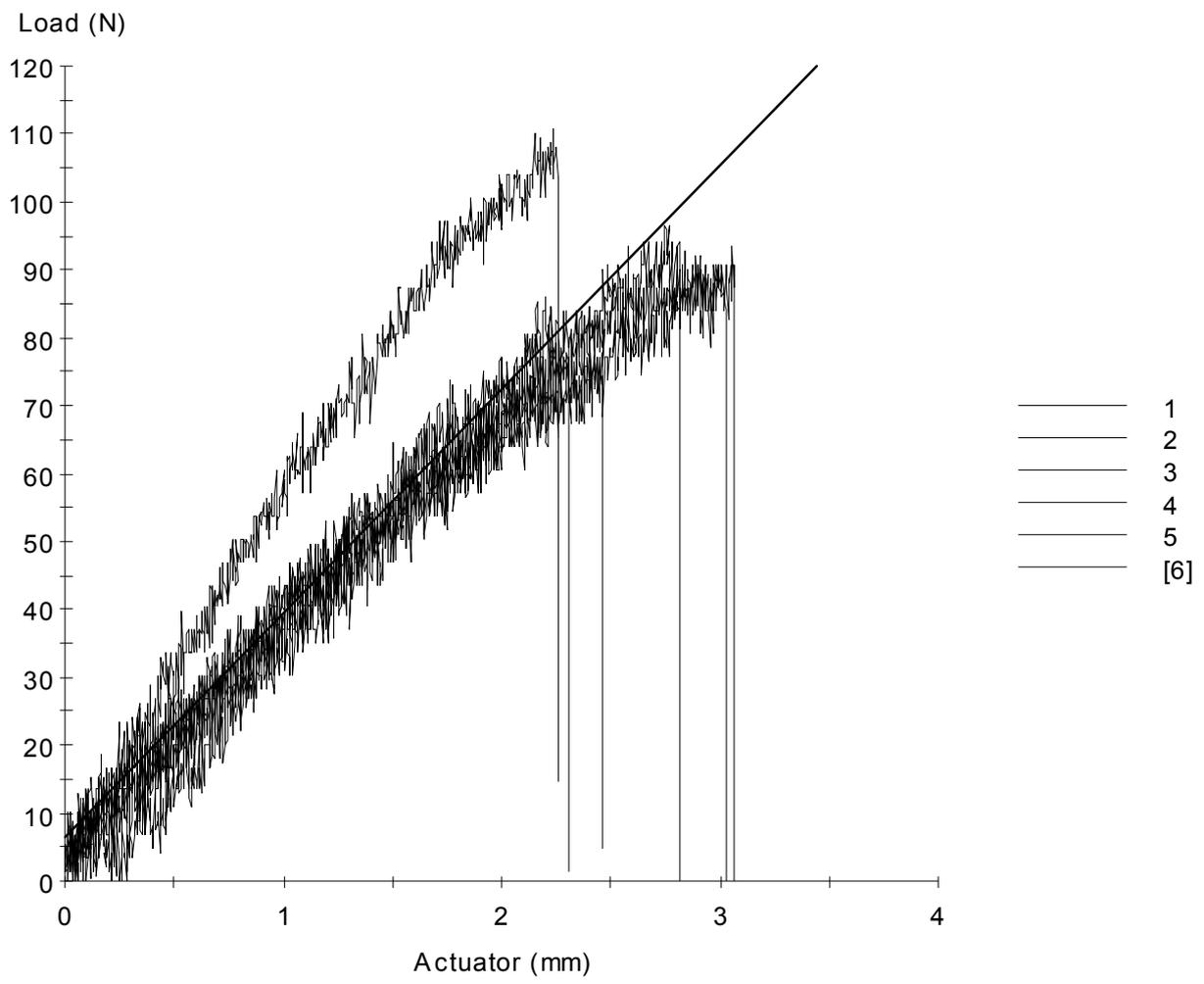
Specimen #	Strain at Break %
1	1.764
2	1.771
3	1.999
4	2.687
5	2.462
6	2.255
Mean	2.156
Std. Dev.	0.377

**Calculation Inputs:**

Name	Value	Units
Break Marker Drop	6.0	%
Break Marker Elongation	0.010	mm
Slack Pre-Load	4.448	N
Slope Segment Length	5.000	%
Span	64.000	mm
Strain Point 1	0.05	%
Strain Point 2	0.250	%
Strain Point 3	0.020	mm/mm
Yield Angle	0.000	rad
Yield Offset	0.002	mm/mm
Yield Segment Length	2.0	%

**Test Inputs:**

Name	Value	Units
Break Sensitivity	80	%
Break Threshold	40.000	N
DataAcqRate	5.0	Hz
Extension Endpoint	25.400	mm
Initial Speed	1.0	mm/min
Load Endpoint	4448	N
Outer Loop Rate	100	Hz
Secondary Speed	1.000	mm/min
Strain Endpoint	0.100	mm/mm



8/09/2009

Sample ID: QCel 5020-20%-6.mss Test Date: 8/09/2009

Method: MMT - Flexural Test (ISO 14125).msm Operator: Mohan Trada

**Specimen Results:**

Specimen #	Width mm	Thickness mm	Modulus MPa	Peak Load N	UTS MPa	Load At Break N	Stress At Break MPa
1	9.870	6.790	1202.898	87.283	18.4	87.283	18.414
2	9.830	5.980	902.493	49.013	13.4	49.013	13.385
3	9.980	6.180	1022.681	70.498	17.8	70.498	17.756
4	9.810	6.280	1248.560	93.997	23.3	93.997	23.324
5	9.800	5.210	1018.543	57.070	20.6	57.070	20.596
6	9.850	6.560	1144.739	87.283	19.8	84.933	19.236
Mean	9.857	6.167	1089.985	74.190	18.9	73.799	18.785
Std. Dev.	0.066	0.549	130.905	18.307	3.3	17.994	3.300

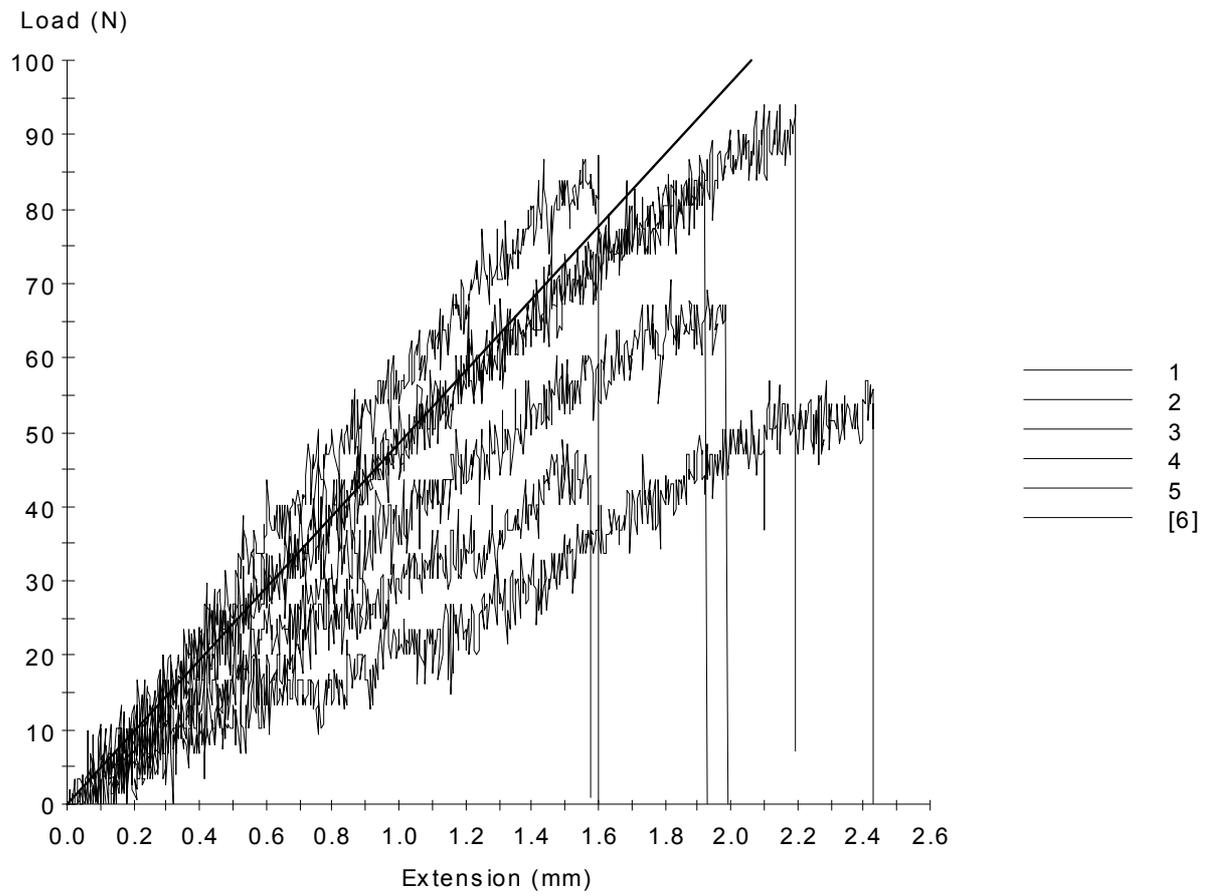
Specimen #	Strain at Break %
1	1.593
2	1.309
3	1.647
4	1.930
5	1.840
6	1.839
Mean	1.693
Std. Dev.	0.227

**Calculation Inputs:**

Name	Value	Units
Break Marker Drop	6.0	%
Break Marker Elongation	0.010	mm
Slack Pre-Load	4.448	N
Slope Segment Length	5.000	%
Span	64.000	mm
Strain Point 1	0.05	%
Strain Point 2	0.250	%
Strain Point 3	0.020	mm/mm
Yield Angle	0.000	rad
Yield Offset	0.002	mm/mm
Yield Segment Length	2.0	%

**Test Inputs:**

Name	Value	Units
Break Sensitivity	80	%
Break Threshold	40.000	N
DataAcqRate	5.0	Hz
Extension Endpoint	25.400	mm
Initial Speed	1.0	mm/min
Load Endpoint	4448	N
Outer Loop Rate	100	Hz
Secondary Speed	1.000	mm/min
Strain Endpoint	0.100	mm/mm



9/09/2009

Sample ID: QCel 5020-30%-4.mss      Test Date:                      9/09/2009

Method:      MMT - Flexural Test (ISO 14125).msm                      Operator: Mohan Trada

**Specimen Results:**

Specimen #	Width mm	Thickness mm	Modulus MPa	Peak Load N	UTS MPa	Load At Break N	Stress At Break MPa
1	9.630	5.340	828.146	36.927	12.9	36.927	12.910
2	9.780	6.100	789.501	30.213	8.0	30.213	7.970
3	9.710	5.640	775.753	26.185	8.1	26.185	8.139
4	9.710	5.710	800.737	28.871	8.8	28.871	8.755
Mean	9.708	5.698	798.534	30.549	9.4	30.549	9.443
Std. Dev.	0.061	0.313	22.228	4.570	2.3	4.570	2.335

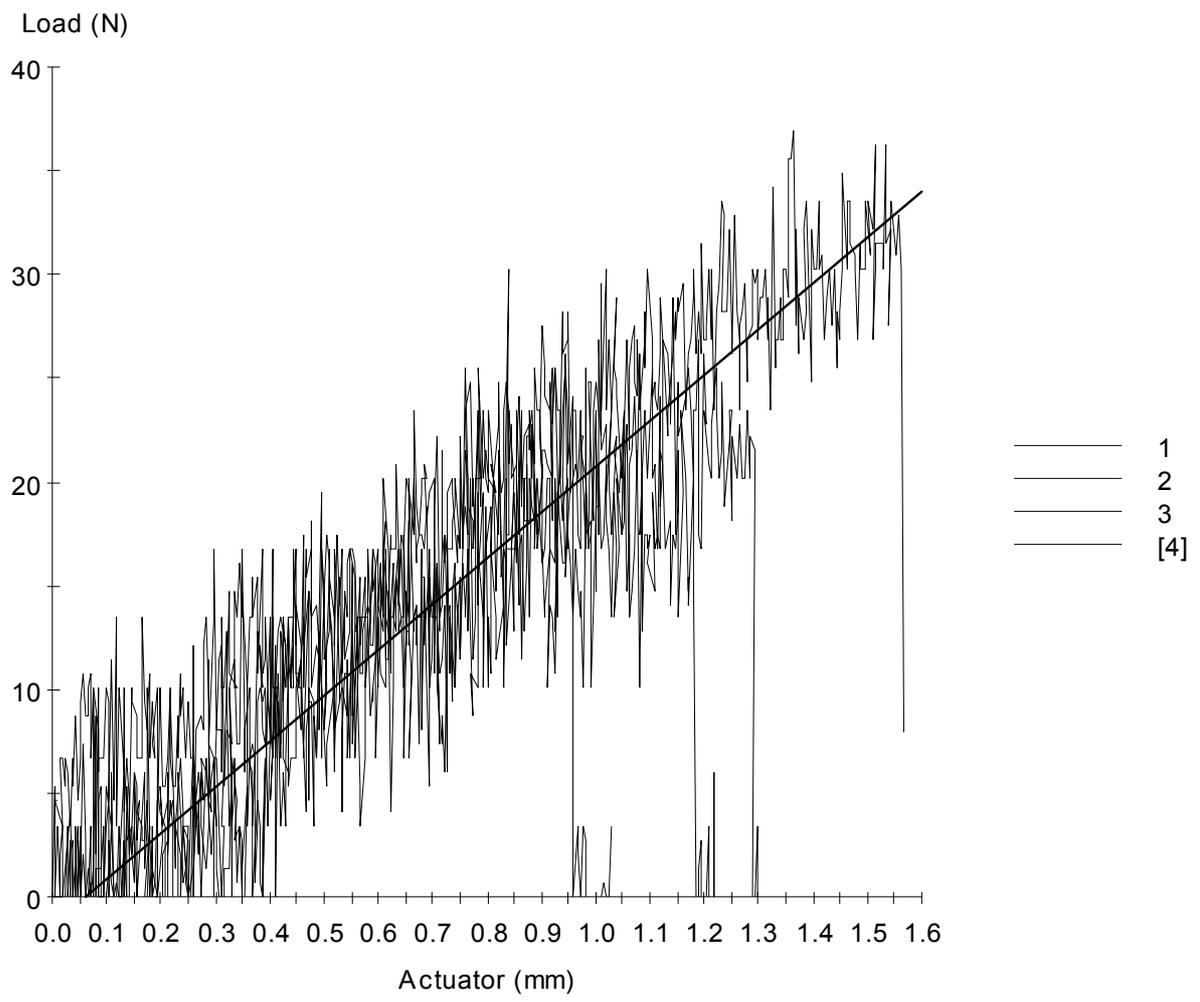
Specimen #	Strain at Break %
1	1.272
2	0.715
3	0.676
4	0.904
Mean	0.892
Std. Dev.	0.272

**Calculation Inputs:**

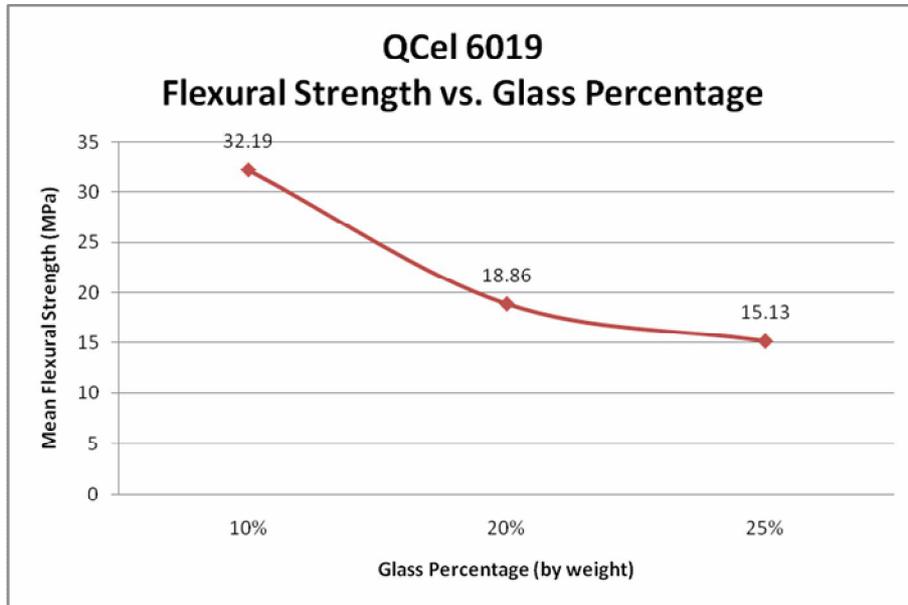
Name	Value	Units
Break Marker Drop	6.0	%
Break Marker Elongation	0.010	mm
Slack Pre-Load	4.448	N
Slope Segment Length	5.000	%
Span	64.000	mm
Strain Point 1	0.05	%
Strain Point 2	0.250	%
Strain Point 3	0.020	mm/mm
Yield Angle	0.000	rad
Yield Offset	0.002	mm/mm
Yield Segment Length	2.0	%

**Test Inputs:**

Name	Value	Units
Break Sensitivity	80	%
Break Threshold	40.000	N
DataAcqRate	5.0	Hz
Extension Endpoint	25.400	mm
Initial Speed	1.0	mm/min
Load Endpoint	4448	N
Outer Loop Rate	100	Hz
Secondary Speed	1.000	mm/min
Strain Endpoint	0.100	mm/mm



# Appendix D: Graphs and Tables



	Glass Percentage		
	10%	20%	30%
Specimen 1	56.019	45.089	29.653
Specimen 2	36.628	36.431	23.261
Specimen 3	55.494	33.334	34.29
Specimen 4	56.876	34.766	31.15
Specimen 5	60.449	37.771	33.406
Specimen 6	68.834	47.905	26.618
Mean Flexural Strength (MPa)	55.717	39.216	29.73

