University of Southern Queensland

Faculty of Engineering and Surveying

Evaluation Impact Strength on Particulate Reinforced (25% and 33%) Vinyl Ester Composite Cured Under Microwave Conditions

A dissertation submitted by

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Abstract

The use of composites plays an important role in the fields of Mechanical and Civil Engineering. The idea of using composite materials in the two afore mentioned fields are no longer new. In the Fibre Composite Design and Development (FCDD), University of Southern Queensland (USQ) many researches and experiments on new lightweight materials and structures have been carried out. In the research, FCDD found that the composites made from vinylester resins suffer considerable shrinkage during hardening. With this issue in mind, research on the methods to decrease the shrinkage of the composites had been carried out. Ku (2002) claimed that by having vinyl ester composites cured under microwave conditions, such shrinkage can be reduced. The material used was thirty three percent by weight flyash particulate reinforced vinyl ester composite VE/FLYASH (33%). Unfortunately, the impact strength of the composites cured under microwave conditions with power level 540 Watts as compared to the composites cured under microwave conditions with power level 180 Watts and 360 Watts is still in doubt.

With the above doubt in mind, this project was carried out to determine whether impact strength of the composites cured under microwave with power level 540 Watts, 360 Watts and also 180 Watts. Drop weight impact test was used to carry out the impact strength test. It was found that having the specimens cured with power level of 540 Watt, the impact strength had on these specimens were alike the one cured with power level 360 Watts and 180 Watts.

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Certification

I certify that the ideas, designs and experimental work, results, analyses and conclusions se out in this dissertation are entirely my own effort, except where otherwise indicated and acknowledged.

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SOH FONG, ANG

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Signature

Date

Acknowledgments

To my parents Khoon Seng, Ang and Lean Tee, Ooi who are currently in Malaysia for their supports and sacrifices to enable me to have the opportunity and privilege to study in Australia to attain my degree. This would not be possible without them.

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Nomenclature

E	energy (joules)
g	gravitational acceleration constant (m/s^2)
m	mass (kilograms)
h	height (m)
pC	pico coulombs
DC	direct currect
F	force (newtons)
mV	milivolts
k	kilo

Chapter 1

Introduction

1.1 Project Aim

The aim of this project was about the evaluation of impact strength on particulate reinforced (25% and 33%) Vinyl Ester composite cured under microwave conditions. The shrinkage of the composites will be measured under ambient conditions and microwave conditions.

1.1.1 Specific Objectives

This project will be produced specimens of 25% and 33 % VE/FLYASH under ambient conditions and microwave conditions. These specimens are to investigate the impact strength of 25% and 33% VE/FLYASH cured under ambient condition and microwave conditions. By the way, the shrinkage of the specimens also will be measured under microwave conditions in various exposure times.

1.2 Dissertation Overview

The reader will be introduced in this section to a very brief overview on the material presented on each chapter of the dissertation.

Chapter Two

Chapter two discusses on the background information of vinyl ester composites. This will be followed by introducing to the reader a more detailed overview of the family of vinyl ester material. The background overview on the materials used and produced will also be introduced to the reader in the later part of this chapter.

Chapter Three

Chapter three discusses on the interaction between microwaves and materials used in this project. The ways of microwaves can be used to reduce the shrinkage of the composite will then be introduced to the reader. Various risks involved in curing using microwave irradiation and the safety measures that needed to be undertaken will be discussed in the later part of the chapter.

Chapter Four

Chapter four discusses on the impact test method that are widely used. The impact test method used in this research project will then be introduced to reader. Instrumentation that incorporated into this test method and test rig setup will be discussed in more detail in later part of this chapter.

Chapter Five

Chapter five discusses on the experimental work in this research project. Discussion on production of specimens will be made first follow by preliminary testing on these specimens. Discussion on data preparation and failure analysis will then be introduced to reader.

Chapter Six

Chapter six discusses on the results obtained from the experimental work and testing. This will include the impact strength had on every specimens cast. Comparison of the results will also be made to determine whether specimens cured with microwave tended to have less impact strength or not. The chapter will then be ended with discussion on overall results obtained.

Chapter 7

Chapter seven is the conclusions and recommendations for this research project. This will include the recommendations given by the author as a guide for further work in this project area.

1.3 Publication

One paper, Impact Strength of Vinyl Ester Composites Cured by Microwave Irradiation: Preliminary Results, submitted for publication to the Proceedings of the IMechE, Part L, Journal of Materials: Design and Applications.

Chapter 2

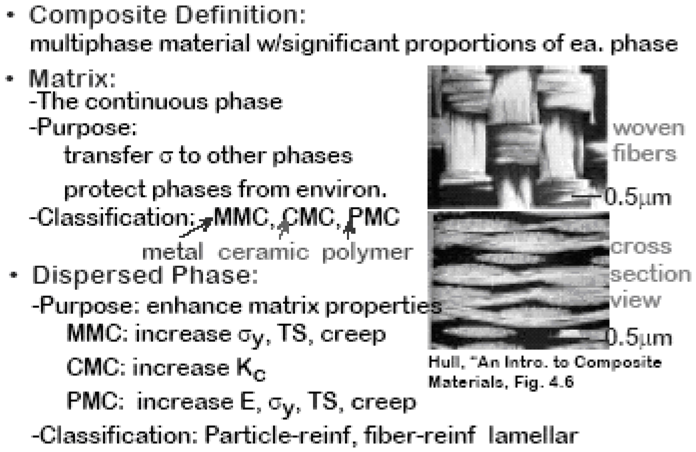
Composite Material

2.1 Introduction

Composites comprise of two or more types of materials those, when combined, lead to improved properties over the individual components. In this case, fiber and resin are combined to form polymer-based composites, in which the fibers are oriented to carry the loads. Composites have lighter, higher strength and stiffer than conventional materials. This all was due to aligned fibers carrying the loads; their adaptive nature can align fibers in the direction to carry the load. They can be designed to minimum weight without sacrificing strength. In this project, particles will be dispersed in a polymer matrix. The background and description of polymer matrix composites (PMCs) as well as their classification will be introduced later. In the case of thermosets, the reader will be introduced to epoxies, polyesters and vinyl ester.

2.2 Types of Composite Materials

Composites are polymer matrix, either thermoset or thermoplastic, reinforced with a fiber or other material with a sufficient aspect ratio (length to thickness) to provide a discernible reinforcing function in one or more directions. Not all plastics are composites. In fact, the majority of plastic materials today are pure plastic and not some form of composite. Many products such as toys, decorative products, household goods and similar applications require only the strength of the plastic resin to perform their functions. “Engineering-grade” thermoplastics can offer improved performance characteristics, such as increased heat distortion temperatures, but usually at higher cost than general-purpose plastic resins. When additional strength is needed, many types of plastics can be reinforced with structural materials- usually reinforcing fibers to meet the demands for higher performance. Any thermoplastic or thermoset plastic resin that is reinforced is considered as a composite. Figure 2.1 shows the classification of composites.

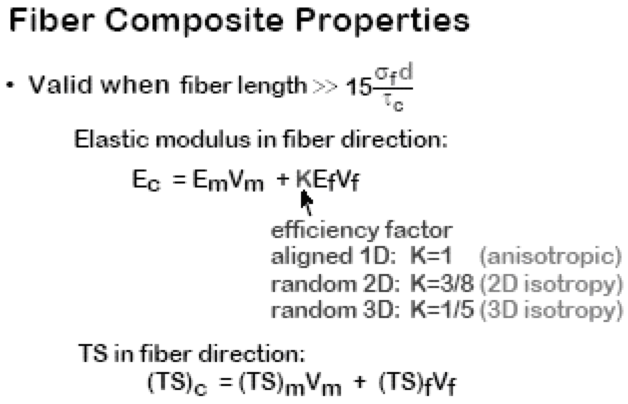


- Composite Definition:
  multiphase material w/significant proportions of ea. phase
- Matrix:
  -The continuous phase
  -Purpose:
    transfer σ to other phases
    protect phases from environ.
  -Classification: MMC, CMC, PMC
    metal ceramic polymer
- Dispersed Phase:
  -Purpose: enhance matrix properties
    MMC: increase σ_y, TS, creep
    CMC: increase K_c
    PMC: increase E, σ_y, TS, creep
  -Classification: Particle-reinf, fiber-reinf lamellar

Hull, "An Intro. to Composite Materials, Fig. 4.6

Figure 2.1: The classification of composites.

There are several types of composites. Most composites in industrial use are based on polymeric matrices: thermosets and thermoplastics. These are usually reinforced with aligned ceramic fibres, such as glass or carbon. Most recently, there has been considerable interesting in metal matrix composites (MMCs), such as aluminium reinforced with ceramic particles or short fibres, and titanium containing long, large-diameter fibres. The property enhancements being sought by the introduction of reinforcement are often less pronounced than for polymers, with improvements in high-temperature performance or tribological properties. While various industrial applications have been developed or are being explored for MMCs, their commercial usage is still quite limited when compared to that of polymer composite (PMCs). Finally, composites based on ceramic materials (CMCs) are also being studied. Figure 2.2 illustrates the properties of fiber composites.



Fiber Composite Properties

- Valid when fiber length >> $15\frac{\sigma_f d}{\tau_c}$

Elastic modulus in fiber direction:

$$E_c = E_m V_m + K E_f V_f$$

efficiency factor
aligned 1D: K=1 (anisotropic)
random 2D: K=3/8 (2D isotropy)
random 3D: K=1/5 (3D isotropy)

TS in fiber direction:

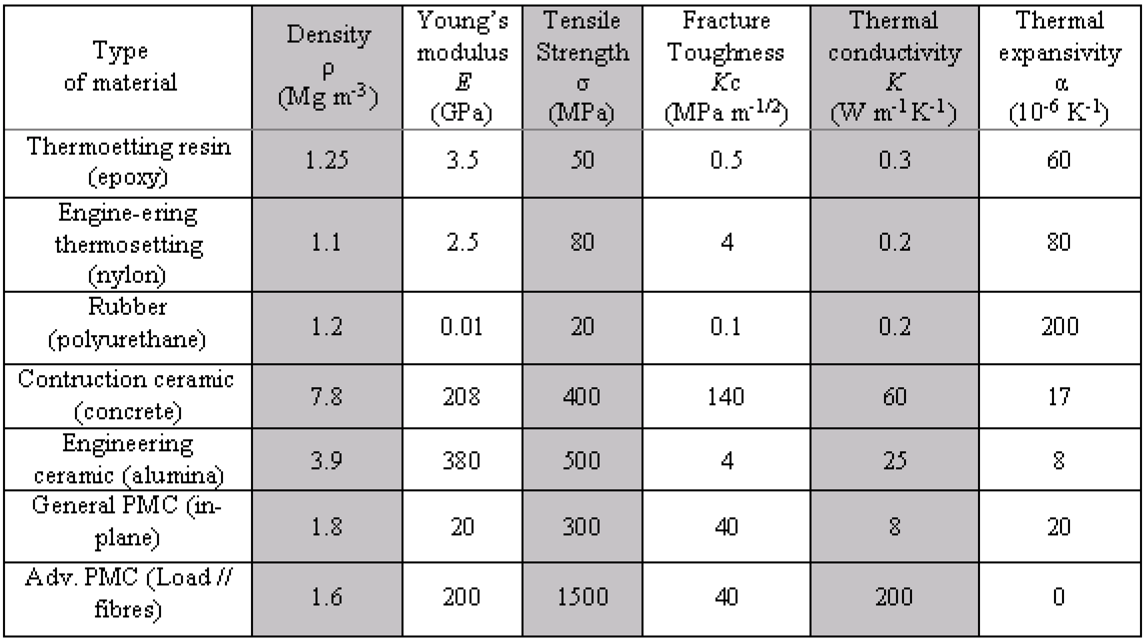
$$(TS)_c = (TS)_m V_m + (TS)_f V_f$$

Figure 2.2: The properties of fiber composites.

The objective is usually to impart toughness to the matrix by the introduction of other constituents, since the stiffness and strength are unlikely to be much affected. Such materials are still, for the most part, in the early stages of development, partly because they are rather difficult to manufacture. Composites have come of age due to the widespread recognition and acceptance among engineers, designers, manufacturers and management for their unique combinations of performance benefits which these materials offer. Composite features translate into multiple benefits; designers, engineers, and others associated with turning design concepts into product realities can make their jobs easier and more effective.

In considering the formulation of a composite material for a particular type of application, it is important to consider the properties exhibites by the potential constituents. The properties of particular interesting are the stiffness (Young's modulus), strength and toughness. Density is of great significance. Thermal properties, such ad expansivity and conductivity, must also be taken into account. In particular, because composite materials are subject to temperature changes (during manufacture and/ or in service), a mismatch between the thermal expansivities of the constituents leads to internal residual stresses. These can have a strong effect on the mechanical behavior. Some representative property data are shown in the Table 2.1 for various types of matrix and reinforcement, as well as for some typical engineering materials and a few representative composites. Inspection of these data shows that some attractive property combination (for example, high stiffness/strength and low density) can be obtained with composites.

Table 2.1: Overview of properties exhibited different classes of material



Type of material	Density ρ (Mg m^{-3})	Young's modulus E (GPa)	Tensile Strength σ (MPa)	Fracture Toughness Kc (MPa m$^{-1/2}$)	Thermal conductivity K (W m^{-1}K^{-1})	Thermal expansivity α (10^{-6} K^{-1})
Thermoetting resin (epoxy)	1.25	3.5	50	0.5	0.3	60
Engine-ering thermosetting (nylon)	1.1	2.5	80	4	0.2	80
Rubber (polyurethane)	1.2	0.01	20	0.1	0.2	200
Contruction ceramic (concrete)	7.8	208	400	140	60	17
Engineering ceramic (alumina)	3.9	380	500	4	25	8
General PMC (in-plane)	1.8	20	300	40	8	20
Adv. PMC (Load // fibres)	1.6	200	1500	40	200	0

2.3 Composite Benefits

In any successful composites application, one or more of the following benefits will normally be at work (Figure 2.3 shows the benefits of 3 types of composites):

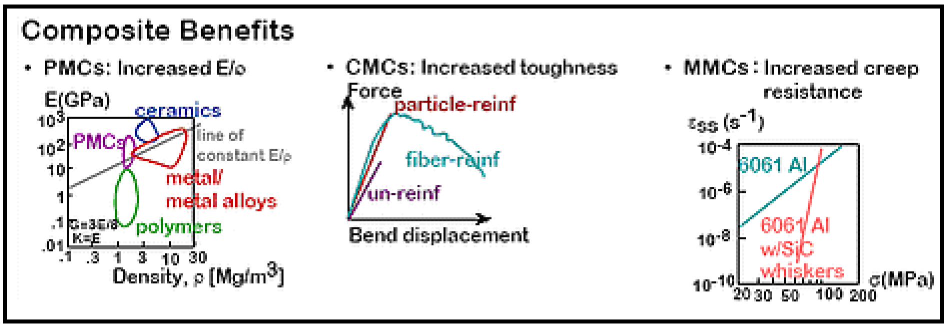


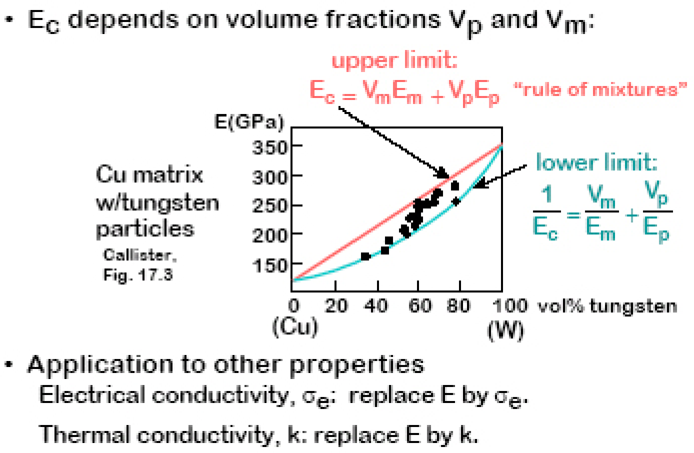
Figure 2.3: Composite Benefits

1. High strength – Composites are among the most effective materials in delivering high strength. These materials can be designed to provide a wide range of mechanical properties including tensile, flexural, impact compressive

strength. Unlike traditional materials, composites can have their strengths oriented or tailored to meet specific design requirements of an application.

2. Light weight –Composites deliver more strength per unit of weight than unreinforced plastics, as well as most metals. This combination of high strength/light weight is powerful incentive for the effective use composites.

3. Design Flexibility -Composites can be formed into virtually any shape a designer may have in mind: complex or simple, large or small structural or appearance, decorative or functional. With composites, many choices are available without having to make costly trade-offs. Addition, composites free designers to try new approaches, from prototype to production. From the Figure 2.4, it illustrates the particle-reinforced of elastics modulus.



- E_c depends on volume fractions V_p and V_m:

- Application to other properties
  Electrical conductivity, σ_e: replace E by σ_e.
  Thermal conductivity, k: replace E by k.

Figure 2.4: Particle-reinforced of elastics modulus

4. Dimensional Stability- Under severe mechanical and environmental stresses, thermoset composites maintain their shape and functionality. Typically, composites do not exhibit the viscoelastic or "cold-creep" characteristics of

unreinforced thermoplastics. The coefficient of thermal expansion is reduced. Generally speaking, the yield point of a composite is its break point.

5. High Dielectric Strength – Composites have outstanding electrical insulating properties, making them obvious choices for current carrying components. It is also possible to impart electrical conductivity to composites through the use of appropriate modifiers and additives, this is required by the application.

6. Corrosion Resistance – Composites do not rust or corrode. There are a number of resin systems available which provide long-term resistance to nearly every chemical and temperature environment. Properly designed composite parts have long service life and minimum maintenance as well.
7. Parts Consolidation – Composite moldings often replace assemblies of many parts and fasteners required for traditional materials such as steel. This can reduce manufacturing cost and frequently results in better, more trouble-free part.

8. Finishing- In many composites applications color can be molded into the product for long lasting, minimum maintenance appearance. Low profile and low-shrink resin systems are compatible with most metallic painting operations. Proper design molds and choice of materials can reduce trim waste, flash, sanding and other post-molding operations.

9. Low Tooling Cost – As a general rule, regardless of the processing methods selected, tooling costs for composites can be lower than tooling costs and materials such as steel, aluminum, alloys and other materials.

10. Proven History of Successful Application – In the last 45 years, over 50,000 successful composite applications have helped to prove the value these materials. Where once the pioneers of the industry struggled to blaze a trail for composite materials acceptance, today's engineers, designers and marketing professionals can point with confidence to a growing array of end uses and applications testify to the cost and performance benefits of composites.

2.4 The Basics of Polymers

The term polymer comes from "poly" meaning many and "mer", describing a unit. Monomers are single building blocks that when joined together form polymers. All polymers commonly used in composites are the products of sophisticated chemical processing. Before entering the world of polymers, it is helpful to have an understanding of the chemistry involved. You don't have to be a chemist to understand these materials.

Chemists use a shorthand notation for various chemical elements. The significant elements which make up most of the plastics we will discuss here are:

C = carbon

H = hydrogen

O = oxygen

For example, using this shorthand, a typical polyester resin might be something like:

$$H – (OOC - C_6H_4 - COO - C_2H_4)n – OH$$

In this case the structure inside the brackets repeats itself many times, as designated by the number n. For a polymer, this would be a long chain and the value for n could be greater than 100.

Using the same shorthand, styrene would be shown as:

$C_6H_5 – CH = CH_2$

2.5 Thermoset versus Thermoplastic

Resins or plastics are divided into two major groups known as thermoset and thermoplastic. Thermoplastic resins become soft when heated, and may be shaped or molded while in a heated semifluid state. Thermoset resins, on the other hand, are usually liquids or low melting point solids in their initial form. When used to produce finished goods, these thermosetting resins are “cured” by the use of a catalyst, heat or a combination of the two. One cured solid thermoset resins cannot be converted back to their original liquid form. Unlike thermoplastic resins, cured thermosets will not melt and flow when heated and once formed they cannot be reshaped.

The composites industry had been divided into thermoset and thermoplastic camps primarily because of differing requirements of their fabrication processes. Both types of plastics can benefit from reinforcement. The initial growth of composites was in thermosets – primarily glass fiber reinforced unsaturated polyester resins. Recently, however, rapid growth has been occurring in the use of reinforced thermoplastics. This trend is expected to continue as thermoplastics improve in properties and cost effectiveness. Therefore, it is increasingly important that persons contemplating the use of the composites be well versed in both thermosetting and thermoplastic polymers.

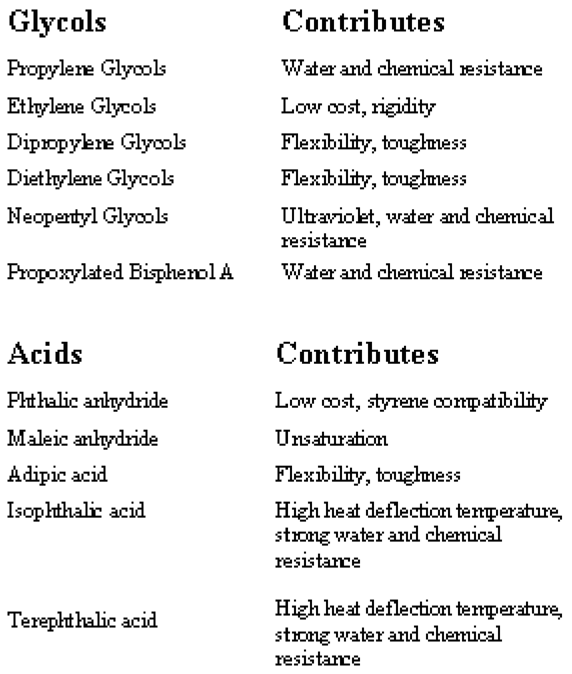
2.6 Thermosetting Resins

The most common thermosetting resins used in the composites industry are unsaturated polyester, epoxies, vinyl ester, polyurethanes and phenolics. There are differences between these groups that must be understood to choose the proper material for a specific application.

2.7 Polyester Resins

Unsaturated polyester resins are the workhorse of the composites industry and they represent approximately 75% of the total resins used. These resins are also available, in a different grade, for injection molding of both composite and non-composite parts. Polyesters are produced by the condensation polymerization of dicarboxylic acids and diayoric alcohols (glycols). In addition, unsaturated polyester polyesters contain an unsaturated material, such as maleic anhydride or fumaric acid, as part of the dicarboxylic acid component. The finished polymer is dissolved in a reactive monomer such as styrene to give a low viscosity liquid. When this resins cured, the monomer reacts with the unsatured sites on the polymer converting it to a solid thermoset structure.

There are many different acids and glycols used in polyester resins. Some of the common ones and their reasons for use are listed in the following.



Glycols	Contributes
Propylene Glycols	Water and chemical resistance
Ethylene Glycols	Low cost, rigidity
Dipropylene Glycols	Flexibility, toughness
Diethylene Glycols	Flexibility, toughness
Neopentyl Glycols	Ultraviolet, water and chemical resistance
Propoxylated Bisphenol A	Water and chemical resistance

Acids	Contributes
Phthalic anhydride	Low cost, styrene compatibility
Maleic anhydride	Unsaturation
Adipic acid	Flexibility, toughness
Isophthalic acid	High heat deflection temperature, strong water and chemical resistance
Terephthalic acid	High heat deflection temperature, strong water and chemical resistance

A range of raw materials and processing techniques are available to achieve the desired properties in the formulated or processed polyester resins because polyester are so versatile and because of their capacity to be modified or tailored during the building of the polymer chains, they have been found to have almost unlimited usefulness in all segments of the composites industry.

The principal advantage of these resins is a balance of properties (including) mechanical, chemical, electrical), dimensional stability, cost and ease of handling or processing.

Unsaturated polyesters are divided into classes depending upon structures of their basic building blocks. Some common examples would be orthophthalic (“ortho”), isophthalic (“iso”), dicyclopentadiene (“DCPD”) and bisphenol A fumarate resins. In addition, polyester resins are classified according to end use application as either general purpose (GP) or specialty polyester.

2.8 Specialty Polyesters

Because polyesters can be chemically tailored to meet the requirements of a wide range of application, a number of specialty polyesters are available. The specialty polyesters include:

- Flexibilized polyesters
- Electrical grade polyesters
- Corrosion-resistant polyesters
- Heat resistant polyesters
- Fire retardant polyesters
- Translucent polyesters
- Low shrink/low profile polyesters

Specialty polyester typically derives their performance from the chemical makeup of the polymer. The proper use of fillers or additives can also enhance properties like fire resistance, fatigue performance or chemical resistance. Improvements in one property such as chemical resistance, may also improve other properties, such as temperature resistance. Bisphenol A fumarate, for example, is used in fabrication because of its ability to tolerate a range of chemical exposure and higher on –service temperatures.

Liquid styrenated polyester resins can be easily shipped to fabricators who do the final shaping and curing into useful products. The mechanism for curing is a reaction between the unsaturation in the polyester and the styrene monomer. This results in the polyester chains being tied together by the styrene monomer. The curing of polyester resins is much different than for epoxies and urethanes or phenolics. Most epoxies and urethanes begin to increase in viscosity as soon as they are catalyzed and continue to increase until they are cured.

Polyester provide a specific working time (gel time) with very little viscosity increase or temperature change. Gelation takes place when less than 5% of the original unsaturation has reactioned and full cure occurs very quickly after this.

2.9 Epoxy Resin

Epoxy resins have a well-established record in a wide range of composite parts and structures. The structure of the resin can be engineered to yield a number of different products with varying levels of performance. Epoxy resins can also be formulated with different materials or blended with other epoxy resins to achieve specific performance features. Cure rates can be controlled to match process requirements through the proper selection of hardeners and/or catalyst systems. Generally, epoxies are cures by addition of an anhydride or an amine hardener. Each hardener produces different properties to the finished composite.

Epoxies are used primarily for fabricating high performance composites with superior mechanical properties, resistance to corrosive liquids and environments, superior electrical properties, good performance at elevated temperatures or a combination of these benefits, Use of epoxies usually requires a critical application that can justify the use of superior performing but higher cost, resin systems. Epoxy resins are used in marine automotive electrical appliance and other composite parts and structures, although they are generally not cost effective in these markets unless special performance is required. Because their viscosity is much higher than most polyester resins and requires post cure to obtain ultimate mechanical properties, epoxies are more difficult to use.

Epoxy resins are used with a number of fibrous reinforcing materials, including glass, carbon and aramid. They are also used as a matrix resins for “whiskers” such as boron, tungsten, steel, boron carbide, silicon carbide, graphite and quartz. This latter group is of small volume, comparatively high cost and is usually used to meet high strength and/or high stiffness requirements. Epoxies are readily usable with most composite manufacturing processes, particularly vacuum-bag molding, autoclave molding, pressure-bag molding, compression molding, filament winding and hand lay-up.

2.10 Vinyl Ester

Vinyl Ester was developed to combine the advantages of epoxy resins with the better handling/faster cure which are typical for unsatured polyester resins. They are produced by reacting epoxy resins with acrylic or methacrylic acid. This provides an unsatured site, much like that produced in polyester resins when malefic anhydride is used. The resulting material is dissolved in styrene to yield a liquid that is similar to polyester resin. Vinyl esters are also cured with the conventional organic peroxides used with polyester resins.

Vinyl ester offer mechanical toughness and excellent corrosion resistance. These enhanced properties are obtained without complex processing, handling or special shop fabricating practices that are typical with epoxy resins.

2.11 Thermoplastic Resins

Thermoplastics resins offer unique and advantageous properties, particularly when combined wit reinforcing fibers to make a composite material. Increasingly, designers are capitalizing on the properties of thermoplastic composite material. Increasingly, designer is capitalizing on the properties of thermoplastic composites to improve product performance and reduce manufacturing costs. Whereas thermoset resins are typically characterized as hard and somewhat brittle, thermoplastic resins are inherently tough and provide superior impact resistance. Also important is the fact that thermoplastic resins do not undergo a time-consuming curing process to achieve final properties. This quality translates to shorter cycle times, increased productivity and lower part costs. Of growing importance, particularly in automotive markets, is that composites made from thermoplastic resins can be readily recycled, meeting the demands of environmental mandates. In addition, these are naturally impervious to attack from harsh chemicals, petroleum products and environment products and environmental elements. Finally, since the group of thermoplastics resins is large and varied, one can select a resin with specific properties tailored to the end application.

2.12 Stiffness of Composites

The fibers may be oriented randomly within the material, but it is also possible to arrange for them to be oriented preferentially in the direction expected to have the highest stresses. Such a material is said to be *anisotropic* (different properties in different directions) and control of the anisotropy is an important means of optimizing the material for specific applications. At a microscopic level, the properties of these composites are determined by the orientation and distribution of the fibers, as well as by the properties of the fiber and matrix materials. The topic known as composite micromechanics is concerned with developing estimates of the overall material properties from these parameters.

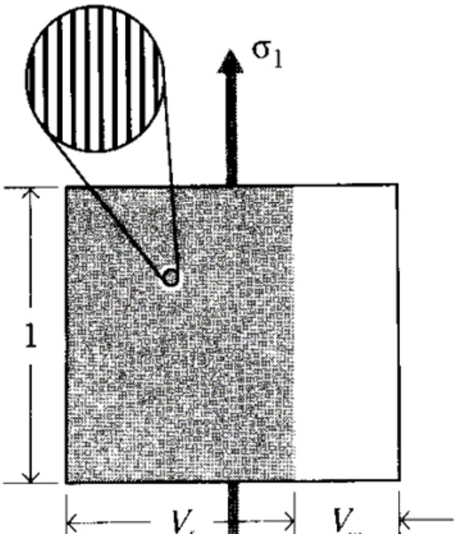


Figure 2.5: Loading parallel to the fibers.

Consider a typical region of material of unit dimensions, containing a volume fraction Vf of fibers all oriented in a single direction. The matrix volume fraction is then Vm = 1−Vf. This region can be idealized as shown in Figure 2.5 by gathering all the fibers together, leaving the matrix to occupy the remaining volume this is sometimes called the “slab model”. If a stress σ_1 is applied along the fiber direction, the fiber and matrix phases act in parallel to support the load. In these parallel connections the strains in each phase must be the same, so the strain ϵ_1 in the fiber direction can be written as:

$$\epsilon_f = \epsilon_m = \epsilon_1 \tag{2.1}$$

The forces in each phase must add to balance the total load on the material. Since the forces in each phase are the phase stresses times the area (here numerically equal to the volume fraction), we have

$$\sigma_1 = \sigma_f V_f + \sigma_m V_m = E_f \epsilon_1 V_f + E_m \epsilon_1 V_m \tag{2.2}$$

The stiffness in the fiber direction is found by dividing by the strain:

$$E_1 = \frac{\sigma_1}{\epsilon_1} = V_f\, E_f + V_m\, E_m \tag{2.3}$$

This relation is known as a *rule* of *mixtures* prediction of the overall modulus in terms of the moduli of the constituent phases and their volume fractions.

If the stress is applied in the direction transverse to the fibers as depicted in Fig. 2.6, the slab model can be applied with the fiber and matrix materials acting in series. In this case the stress in the fiber and matrix are equal (an idealization), but the deflections add to give the overall transverse deflection.

$$\frac{1}{E_2} = \frac{V_f}{E_f} + \frac{V_m}{E_m} \tag{2.4}$$

Figure 4 shows the functional form of the parallel (Eqn. 1) and series (Eqn. 2) predictions for the fiber- and transverse-direction moduli. The prediction of transverse modulus given by the series slab model (Eqn. 2) is considered unreliable, in spite of its occasional agreement with experiment. Among other decencies the assumption of uniform matrix strain being untenable; both analytical and experimental studies have shown substantial nonuniformity in the matrix strain. Figure 2.6 shows the photoelastic fringes in the matrix caused by the perturbing effect of the stiffer fibers. (A more complete description of this photo elasticity can be found in the Module on Experimental Strain Analysis, but this figure can be interpreted simply by noting that closely-spaced photoelastic fringes are indicative of large strain gradients.

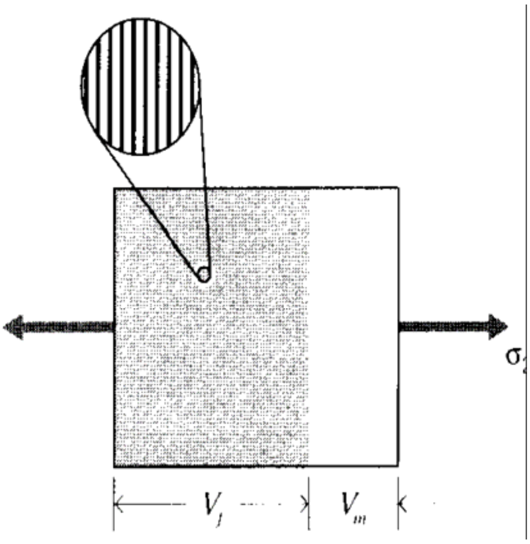


Figure 2.6: Loading perpendicular to the fibers.

In more complicated composites, for instance those with fibers in more than one direction or those having particulate or other nonfibrous reinforcements, Eqn. 2.1 provides an upper bound to the composite modulus, while Eqn. 2.2 is a lower bound (see Fig. 2.7). Most practical cases will be somewhere between these two values, and the search for reasonable models for these intermediate cases has occupied considerable attention in the composites research community. Perhaps the most popular model is an empirical one known as the Halpin-Tsai equation 2.2, which can be written in the form:

$$E = \frac{E_m[E_f + \xi(V_f E_f + V_m E_m)]}{V_f E_m + V_m E_f + \xi E_m} \tag{2.5}$$

Here ξ is an adjustable parameter that results in series coupling for $\xi = 0$ and parallel averaging for very large ξ.

2.11 Strength of Composites

Rule of mixtures estimates for strength proceed along lines similar to those for stiffness. For instance, consider a unidirectional reinforced composite that is strained up to the value at which the fibers begin to break. Denoting this value ϵ_{fb}, the stress transmitted by the composite is given by multiplying the stiffness (Eqn. 2.1):

$$\sigma_b = \epsilon_{fb} E_1 = V_f \sigma_{fb} + (1 - V_f)\sigma^* \tag{2.6}$$

The stress σ^* is the stress in the matrix, which is given by $\epsilon_{fb}E_m$. This relation is linear in V_f, rising from σ^* to the fiber breaking strength $\sigma_{fb} = E_f \epsilon_{fb}$. However, this relation is not realistic at low fiber concentration, since the breaking strain of the matrix ϵ_{mb} is usually substantially greater than ϵ_{fb}. If the matrix had no fibers in it, it would fail at a stress $\sigma_{mb} = E_m\epsilon_{mb}$. If the fibers were considered to carry no load at all, having broken at $\epsilon = \epsilon_{fb}$ and leaving the matrix to carry the remaining load to

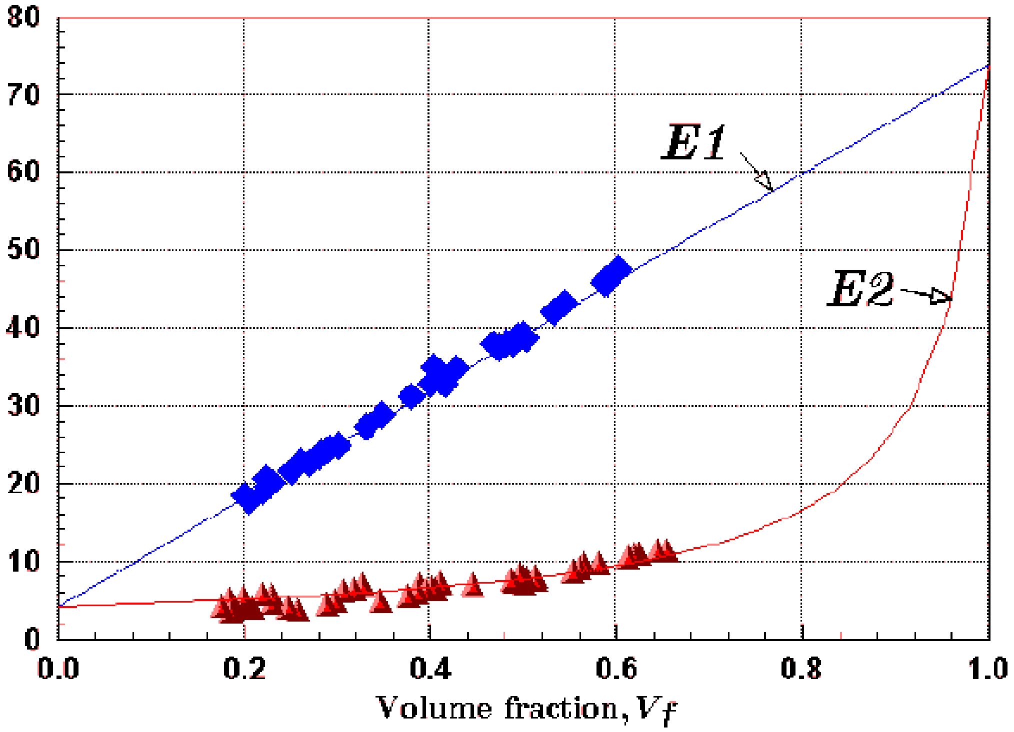


Figure 2.7: Rule-of-mixtures predictions for longitudinal (E1) and transverse (E2) modulus, for glass-polyester composite (Ef = 73:7 MPa, Em = 4 GPa).

carry the remaining load, the strength of the composite would fall off with fiber fraction according to

$$\sigma_b = (1 - V_f)\sigma_{mb} \tag{2.7}$$

Since the breaking strength actually observed in the composite is the greater of these two expressions, there will be a range of fiber fraction in which the composite is weakened by the addition of fibers. These relations are depicted in Fig. 2.9.

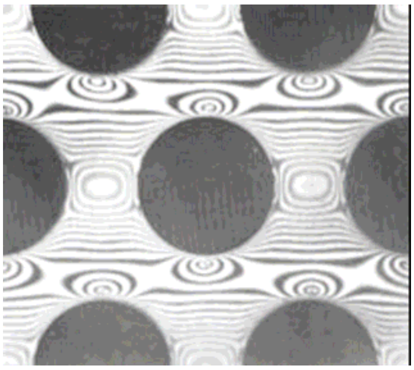


Figure 2.8: Photoelastic (isochromatic) fringes in a composite model subjected to transverse tension

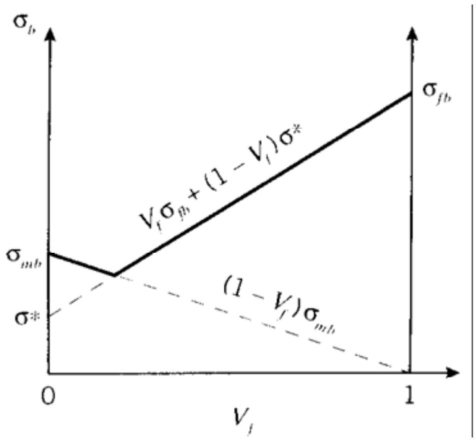


Figure 2.9: Strength of unidirectional composite in fiber direction.

Chapter 3

Resins and Their Interactions with Microwaves

3.1 Introduction

The most common thermosets used as composite matrices are unsaturated polyesters (UP), epoxies and vinyl esters. Unsaturated polyesters dominate the market, whereas epoxies are preferred in high-performance applications. Unsaturated polyester offers an attractive combination of low price, reasonably good properties, and simple processing. However, basic unsaturated polyester formulations have drawbacks in terms of poor temperature and ultra-violet tolerance.

Additives may significantly reduce these advantages to suit most applications. Where mechanical properties and temperature tolerance of unsaturated polyesters no longer suffice, epoxies (EP) are often used due to their significant superiority in these respects. Of course, these improved properties come at a higher price and epoxies are used most commonly in areas where cost tolerance is highest (Astrom, 1997). When epoxy resins are used to make composite structures, there are three main drawbacks (Pritchard, 1999):

i) Because of their two-step hardening process, they are slow to cure, and they require a minimum post cure of 2 to 4 hours at 120 ^{o}C to achieve 70-80% of optimal properties.

ii) Their viscosity makes it difficult to wet the glass fibres efficiently.

iii) The use of amine hardeners renders the cured resins susceptible to acid attack.

With this issue in mind, the so-called epoxy vinyl ester range of resins (vinyl ester resins) was developed in the 1960s (Pritchard, 1999). Vinyl esters (VE), as they are usually called, are chemically closely related to both unsaturated polyesters and epoxies and in most respects represent a compromise between the two. They were developed in an attempt to combine the fast and simple cross linking of unsaturated polyesters with the mechanical and thermal properties of epoxies (Astrom, 1997).

To achieve the project objectives, i.e. to reduce the shrinkage of vinyl esters, it will be necessary to apply microwave energy in a multimode oven cavity to samples of vinyl ester resins under controlled conditions. A commercial 1.8 kW microwave oven will be used. The 1.8 kW power is actually achieved by launching microwaves from two 0.9 kW magnetrons. The power inputs can be varied from 10% (180 W) to 100% (1800 W) in steps of 180 W.

3.2 Vinyl Ester Resins

Unsaturated resins such as polyesters and vinyl esters have ester groups in their structures. Esters are susceptible to hydrolysis and this process is accelerated and catalyzed by the presence of acids or bases. Vinyl esters contain substantially less ester molecules than polyesters. They contain only one at each end of the resin molecule. This is illustrated by the structure of bishophenol A vinyl ester in Figure 1. This means that vinyl esters, just like epoxies, have few possible crosslink sites per molecule. Vinyl esters of high molecular weight will therefore have relatively low crosslink density and thus lower modulus than if the starting point is a lower molecular-weight polymer. Vinyl esters crosslink in time frames and under conditions similar to those of unsaturated polyesters, i.e. fairly quickly and often at room temperature (Astrom, 1997). Methacrylic acid is used to manufacture the vinyl esters. This means that next to each ester linkage is a large methyl group. This group occupies a lot of space and sterically hinders any molecule approaching the ester group by impeding their access. These two aspects of the design of the vinlyester molecule combine to make them more chemically resistant than polyesters (Pritchard, 1999). There are three families of vinyl esters. The most commonly used family is based on the reaction between methacrylic acid and diglycidylether of bisphenol A (DGEBPA) as shown in Figure 3.1 (Astrom, 1997). This family of vinyl ester is used in this research and the weight of materials has show in Table 3.1:

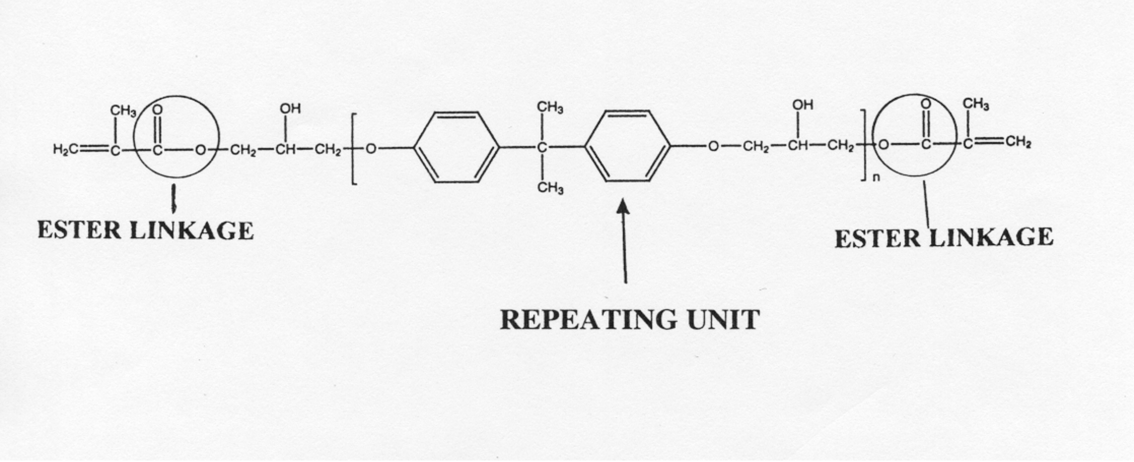


Figure 3.1: The structure of bishophenol A vinyl ester

Table 3.1: Weight of materials required to make 500 ml of VE/FLYASH (33%)

Materials Parameters	Resin	MEKP	Flyash	Composite
Relative density	1.1	1.0	0.7	---
Percentage by volume	56	---	44	100
Percentage by weight	67	---	33	100
Weight for 500 ml of composite	301.8 (g)	5.6 (g)	154 (g)	---

3.3 Cross Linking of Vinyl Esters Resin

The polymerisation product between methacrylic acid and bisphenol A is vinyl ester, which can be a highly viscous liquid at room temperature or a low melting point solid, depending on the acid and bisphenol A used. For further processing, the polymer is dissolved in a low molecular monomer, or reactive dilutent, usually styrene, the result is a low viscosity liquid referred to as resin. The resin used in this research has 50% by weight of styrene. With the addition of a small amount of initiator to the resin the cross linking reaction, or curing, is initiated. The initiator used is organic peroxide, e.g. methyl ethyl ketone peroxides (MEKP). The added amount is usually 1 to 2 percent by weight. The initiator is a molecule that producers free radicals. The free radicle attacks one of the double bond of the ends of the polymer and bonds to one of the carbon atoms, thus producing a new free radical at the other carbon atom, see the initiation step of Figure 3.2, which illustrates the whole cross linking process. This newly created free radical is then free to react with another double bond. Since the small monomer molecules, the styrene molecules, move much more freely within the resin than the high molecular weight polymer molecules, this double bond very likely belongs to a styrene molecule, as illustrated in the bridging step of Figure 3.2. The bridging step creates a new free radical on the styrene, which is then free to react with another double bond and so on. Obviously the styrene is not only used as solvent, but actively takes part in the chemical reaction. Monomers are consequently called curing agents and initiators are called catalysts. As the molecular weight of the cross linking polymer increases it gradually starts to impair the diffusion mobility of the growing molecules and the reaction rate slows down. When the prevented from finding new double bonds to continue the movement of the free radicals is also impaired they are reaction which then stops.

The result of the cross linking reaction is gigantic, 3D molecules that form a macroscopic point of view leads to the transformation of the liquid resin into a rigid solid. The cross linking reaction is intimately linked to temperature. Since the cross linked molecular morphology represents a lower energy state than the random molecular arrangement in the resin, the reaction is exothermal. Further the free radical production is stimulated by an increase in temperature also promotes molecular mobility. Until diffusion limitations reduce the reaction rate, the cross linking rate therefore increases; heat is released by the formation of new bonds, which promotes an increase in rate of bond formation (Astrom, 1997).

Figure 3.2 shows typical temperature time relations for cross linking of a vinyl ester following addition of initiator. The three solid curves on the right hand side of the figure represent room temperature cross linking of vinyl esters.

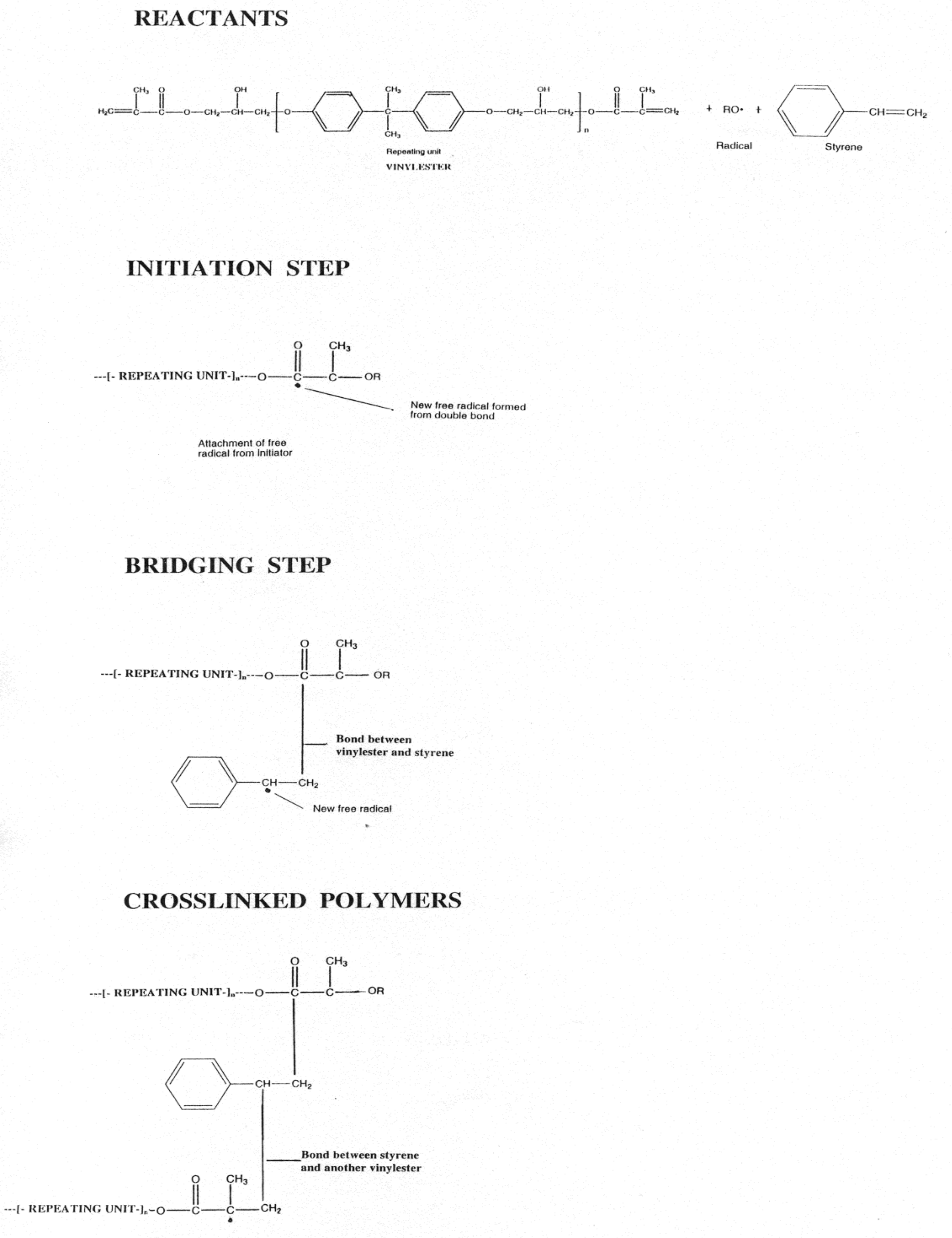


Figure 3.2: Schematic of addition or free radical cross linking of vinyl ester

The different curves illustrate different amount of initiator, inhibitor, accelerator or volume of resin. A reduced amount of initiator and accelerator, as well as an increased amount of inhibitor, leads to later cross linking at lower temperature, and vice versa. The larger the volume of the resin, the faster the reaction will be. The temperature does not immediately increase after addition of an initiator despite free radicals being produced. The cross linking reaction does not start and the temperature does not increase until all inhibitor molecules have reacted with free radicals, which corresponds to inhibition time. As cross linking commences, the pot life is over. The resin becomes a rubbery solid quickly and the gel time is reached. The cross linking activity now accelerates very rapidly until the increasing molecular weight of the cross linking polymer starts restricting molecular movement, which occurs around the maximum temperature, and the cross linking gradually tapers off. On the other hand, the dashed line curve on the left hand side of the figure illustrates the hypothetical cross linking as a result of the application of microwave to the resin. In this case, the inhibition time is short and maximum temperature is reached quickly. It is anticipated that the result of such a curing will reduce the shrinkage of vinyl ester.

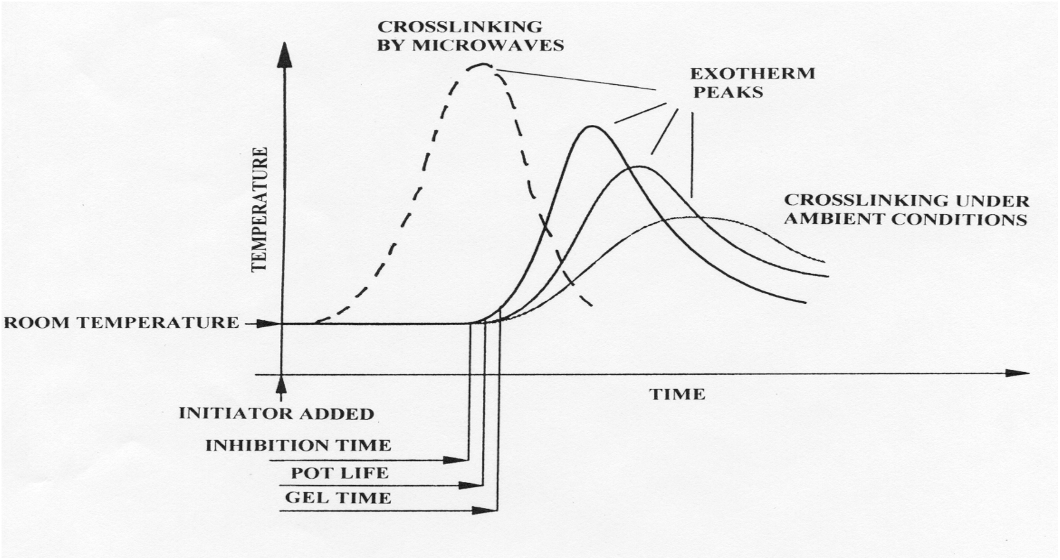


Figure 3.3: Temperature time relationships for cross linking of vinyl ester

3.4 Shrinkage in VE/Fly-ash (33%)

In the workshop of the Fibre Composite Design and Development (FCDD) group, University of Southern Queensland (USQ), the vinyl ester resin used is Hetron 922 PAS in summer and Hetron 922 PAW in winter. The vinyl ester is dissolved in 50% by weight of styrene. The curing rate for Hetron 922 PAS will be slower in winter and hence Hetron 922 PAW has to be used for this study. They are both based on the reaction between methacrylic acid and diglycidylether of bisphenol A (DGEBPA). Suppliers of the raw vinyl ester resins claim that shrinkage in cured vinyl esters is around 5 to 6 %. However, the engineers in the Fibre Composite Design and Development (FCDD) group, University of Southern Queensland (USQ) found that the shrinkage varied from 10 to 12 % for their large components. Lubin (1982) also claimed the same amount of shrinkage for the resin with 50% by weight of styrene. In order to estimate the real shrinkage percentage, one experiment was carried out. Two beakers of 50 milliliters (internal diameter is 44.10 mm) and 200 milliliters (internal diameter is 74.95 mm) were employed for the experiment. To start with polyvinyl acetate (PVA) release agent has to be smeared on the inner surfaces of the beakers to enable the release of the cured vinyl ester at a later stage. From the Table 3.2 has shown the volume of the composites after shrinkage and before shrinkage:

Table 3.2: Comparison of original and final volumes of VE/FLYASH (33%)

Original volume (ml)	600	400	200	50
Final volume (ml)	535.8	363.94	187.2	47.44
Ambient temperature	16	16	20	20
Relative humidity	52	52	19	19
Peak temperature (^{o}C)	143	139	106	85
Gel time (minutes)	60	65	32.5	35
Percentage of shrinkage	10.7	9.02	6.40	5.13

The resin hardener ratio used in the experiment was 98% resin by volume and 2% hardener (MEKP) by volume. The reinforce was fly ash (ceramic hollow spheres) particulate and was made 33 % by weight in the cured vinyl ester composite. Thirty three percent by weight of fly ash in the composite is considered optimum by FCDD group because the composite will have a reasonable fluidity for casting combined with a good tensile strength in service. The curing rate of the mixture of resin, hardener and fly ash will be faster with higher percentage by volume of hardener, higher humidity and higher temperature. The ambient temperature when the experiment was carried out was 20 ^{o}C and the relative humidity was 19%. The resin is a colorless liquid and is first mixed with the red hardener. After that the fly ash is added to the mixture and they are then mixed to give the uncured composite. To make a volume of 250 milliliters of uncured composite (of 44% by volume of fly ash or of 33% by weight), the total volume of resin plus hardener = 250 milliliters x 0.56 = 140 milliliters. For a composite with 98% resin and 2% hardener by volume, the volume of resin required = 140 milliliters x 0.98 = 137.2 milliliters and that of hardener required is 2.8 milliliters. It is easier to measure mass rather than volume so 137.2 milliliters of resin is converted to 137 x 1.1 = 151 g of resin, where 1.1 is the relative density of the resin. Similarly, the mass of hardener required is 2.8 x 1 = 2.8 g, where 1 is the relative density of the hardener. Since the relative density of the fly ash is 0.7, the mass of the fly ash required = 110 x 0.7 = 77g. After mixing, 200 milliliters of the composite was poured into the beaker with a volume of 200 milliliters and the rest was poured into another beaker. Data of temperature against time for the beakers were collected. Temperature measurements were carried out from the top of the beakers at three points around the centre of the beaker and an average value was used. Figure 3.4 shows the relationship between temperatures against time in curing 200 ml of vinyl ester. The curve is similar to that found in one of the three, on the right hand side of Figure 3.3. The inhibition time was 10 minutes. At time equaled to 30 minutes, the temperature was 54 °C and a crest was formed on the surface. At time equaled to 40 minutes, the temperature peaked and was 106 °C. The temperature then began to drop. Room temperature was regained at time equaled to 115 minutes. To determine the initial and final volumes of the composite in the beaker, the height of the level of VE/FLYASH (33%) was measured by a digital height gauge. The initial height was 48.24 mm, which represents a volume of 200 ml. Twenty four hours later, the height was re-measured and was found to be 47.19 mm. The linear shrinkage of the composite after curing was:

$$\frac{48.24mm - 47.19mm}{48.24mm} = \frac{1.05}{48.24} = 0.0218$$

The volumetric shrinkage of the composite can be expressed as (Kalpakjian, 1991):

$$V_{cured} = V_{uncured} \text{ x} \left(1 - \frac{\Delta L}{L_0}\right)^3 \qquad (3.1)$$

Therefore, $V_{cured} = 200 \text{ ml } (1-0.0218)^3 = 187.20$ ml.

The shrinkage is $\frac{200ml - 187.20ml}{200ml} x100\% = 6.4\%$.

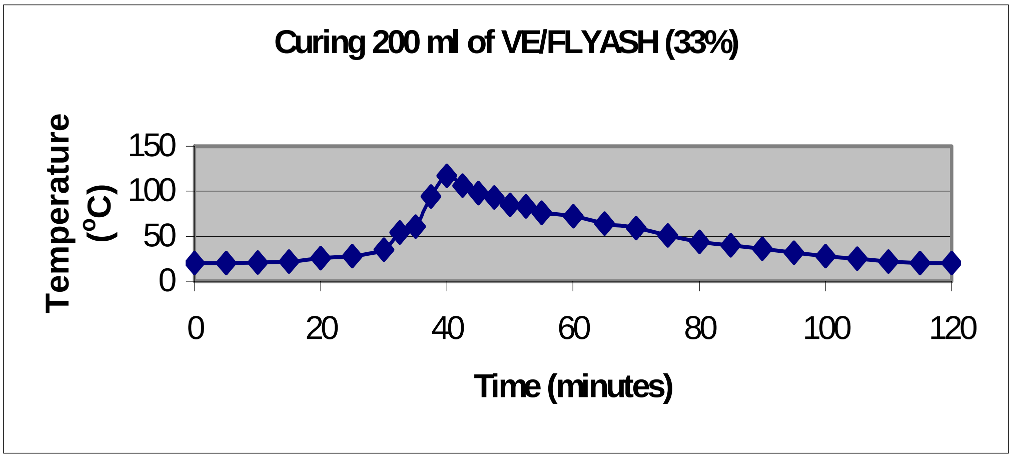


Figure 3.4: Relationship between temperature and time in curing 200 ml of vinyl ester composite, VE/FLYASH (33%) under ambient conditions

Figure 3.5 illustrates the relationship between temperatures against time in curing 50 ml of vinyl ester. The inhibition time was 10 minutes. A crest was formed at time equaled to 35 minutes. The temperature peaked at time equaled to 45 minutes and the temperature was 85°C. Temperature returned to 20°C at time equaled to 95 minutes. The volume was measured by the same method described above after twenty-four hours (1440 minutes) and it was found that the volume was 47.36 ml. The shrinkage was 5.28%. From the results of the experiment, it was found that the larger the volume of vinyl ester, the larger the shrinkage and the higher the peak temperature would be during curing. This is in line with the historical data kept by the FCDD group.

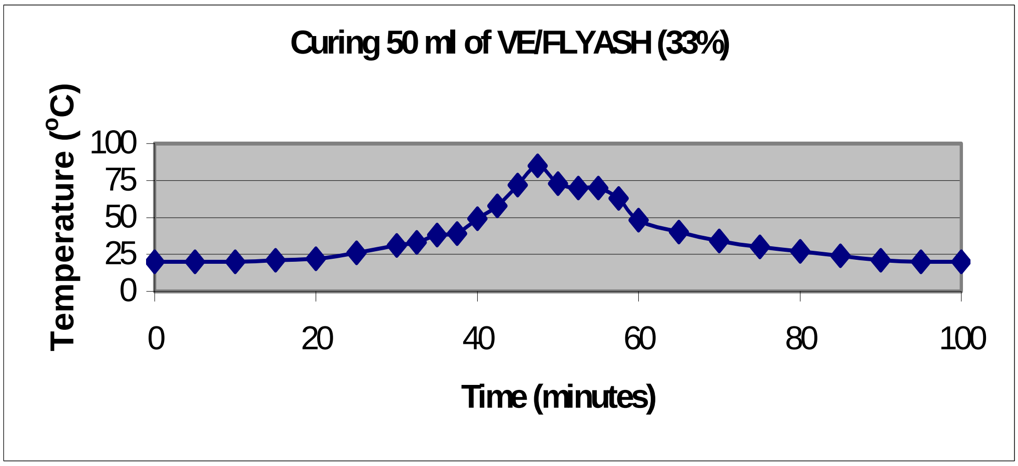


Figure 3.5: Relationship between temperature and time in curing 50 ml of vinyl ester composite, VE/FLYASH (33%)

3.5 Rheology of curing Vinyl Esters

Figure 3.6 depicts the degree of cure for vinyl ester against time at various isothermal cure temperatures (Osswald and Menges, 1995). It is found that if the cure temperature is 60^{o}C, the curing will complete 11 minutes after the inhibition time and the degree of cure is 0.69. Similarly, if the cure temperature is 40^{o}C, the curing will complete 13 minutes after the inhibition time and the degree of cure is 0.36. Referring to the previous experiment, in the curing of 200 ml of vinyl ester, it was found that the curing started 20 minutes after the inhibition time and the peak temperature was 106 ^{o}C. It can be argued that the degree of curing in the experiment is higher than 0.69 and is possibly approaching 0.85. In order to prove this, the cured composite (200 ml initially) was stripped off the beaker and studied. It was found that the degree of cure was near complete. The other cured composite (initially 50 ml) was found to be fully cured as well.

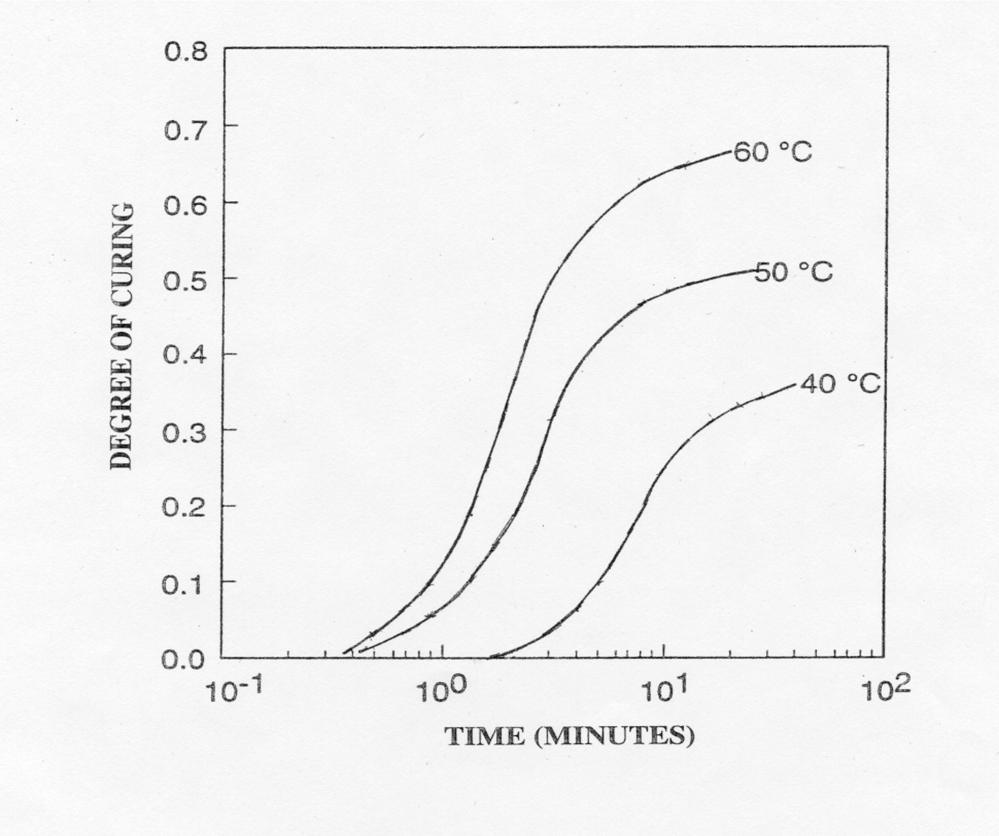


Figure 3.6: Degree of cure of vinyl ester at different curing temperatures

3.6 Fundamentals of Microwaves

Microwaves form part of a continuous electromagnetic spectrum that extends from low-frequency alternating currents to cosmic rays. In this continuum, the radio-frequency range is divided into bands as depicted in Table 3.3 and figure 3.7. Bands 9, 10 and 11 constitute the microwave range that is limited on the low-frequency side by very high-frequency (VHF) and on the high-frequency side by the far infrared (Thuery, 1992). These microwaves propagate through empty space at the velocity of light and their frequency range from 300 MHz to 300 GHz. The HF and ultra high frequency bands constitute a natural resource managed by three international organizations. These organizations delegate their power to national organizations for allocating frequencies to different users. Industrial microwaves are generated by a variety of devices such as magnetrons, power grid tubes, klystrons, klystrodes, crossed-field amplifiers, traveling wave tubes, and gyrotrons (NRC, 1994).

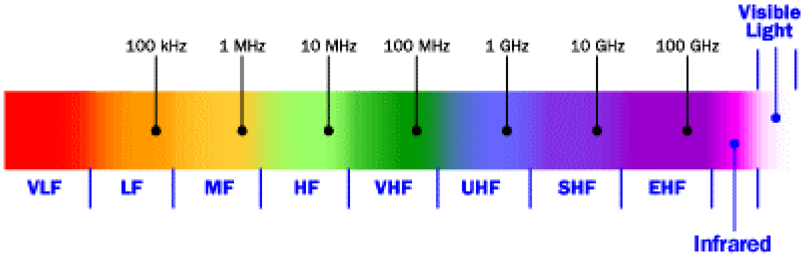


Figure 3.7: Frequency Bands for Radio Frequency Range

Table 3.3: Frequency Bands for Radio Frequency Range

Band	Designation	Frequency limits
4	VLF very low frequency	3 kHz - 30 kHz
5	LF low frequency	30 kHz - 300 kHz
6	MF medium frequency	300 kHz - 3 MHz
7	HF high frequency	3 MHz - 30 MHz
8	VHF very high frequency	30 MHz - 300 MHz
9	UHF ultra high frequency	300 MHz - 3 GHz
10	SHF super high frequency	3 GHz - 30 GHz
11	EHF extremely high frequency	30 GHz - 300 GHz

Frequency bands reserved for industrial applications are 915 MHz, 2.45 GHz, 5.8 GHz and 24.124 GHz. At the customary domestic microwave frequency of 2.45 GHz, the magnetrons are the workhorse. Material processing falls into this category (NRC, 1994). Magnetrons are the tubes used in conventional microwave ovens found almost in every kitchen with a power of the order of a kilowatt. Industrial ovens with output to a megawatt are not uncommon. Huge sums of money and effort have been spent in developing microwave-processing systems for a wide range of product applications. Most applicators are multimode, where different field patterns are excited simultaneously.

3.7 Microwave and material interactions

The material properties of greatest importance in microwave processing of a dielectric are the complex relative permittivity $\varepsilon = \varepsilon' - j\varepsilon''$ and the loss tangent, $\tan\delta = \varepsilon''/\varepsilon'$ (Metaxas and Meredith, 1983). The real part of the permittivity, ε', sometimes called the dielectric constant, mostly determines how much of the incident energy is reflected at the air-sample interface, and how much enters the sample. The most important property in microwave processing is the loss tangent, tan δ or dielectric loss, which predicts the ability of the material to convert the incoming energy into heat. For optimum microwave energy coupling, a moderate value of ε', to enable adequate penetration, should be combined with high values of ε'' and tan δ, to convert microwave energy into thermal energy. Microwaves heat materials internally and the depth of penetration of the energy varies in different materials. The depth is controlled by the dielectric properties. Penetration depth is defined as the depth at which approximately $\frac{1}{e}$ (36.79%) of the energy has been absorbed. It is also approximately given by (Bows, 1994):

$$D_p = \left(\frac{4.8}{f}\right)\frac{\sqrt{\varepsilon'}}{\varepsilon''} \tag{3.2}$$

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

Note that ε' and ε'' can be dependent on both temperature and frequency, the extent of which depends on the materials. The results of microwaves/materials interactions are shown in Figure 3.8.

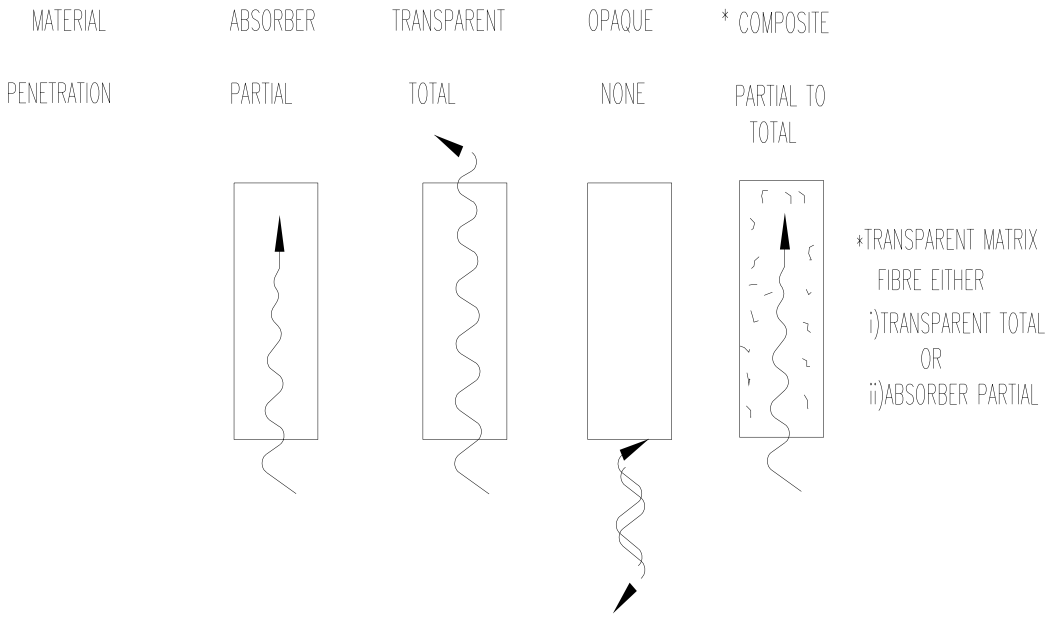


Figure 3.8: Interaction of Microwaves with Materials

During microwave processing, microwave energy penetrates through the material. Some of the energy is absorbed by the material and converted into heat, which in turn raises the temperature of the material such that the interior parts of the material are hotter than its surface, since the surface loses more heat to the surroundings. This characteristic has the potential to heat large sections of the material uniformly. The reverse thermal effect in microwave heating does provide some advantages. These include:

- Rapid heating of materials without overheating the surface
- A reduction in surface degradation when drying wet materials because of lower surface temperature
- Removal of gases from porous materials without cracking
- Improvement in product quality and yield
- Synthesis of new materials and composites

3.8 Interaction of Microwaves and VE/Flyash (33%)

Whether a material will absorb microwave energy and convert it into heat depends on its relative complex permittivity and loss tangent. Ku et al. (2001) showed that liquid rapid Araldite (epoxy resin) has a dielectric constant of 2.81 and a loss tangent of 0.244 at 2.45 GHz at room temperature. The loss tangent is quite high and it is expected that Araldite will absorb microwaves readily and convert it into heat. Vinyl ester resin is produced from modified epoxy resin and methacrylic acid (Peters, 1998); it is therefore expected that it will also absorb microwaves readily. A possible risk in applying microwave energy to the vinyl ester composite is the interaction of styrene in the resin with the high voltage (HV) transformer of the oven cavity is spot welded together and is not necessarily water/air/steam proof. Styrene is a flammable vapour and will be given off during the curing process of the composite. The gas may spark if it is affected by the heat of the HV components. The oven does not have an exhaust fan. A blower motor inside sucks air through the air filter at the front and cools the HV transformer as the air passes. The air from the fan is blown into a duct and cools the magnetrons. Some air is forced into the cavity at the back and then out of the steam exhaust outlet at the back. As a precaution, a beaker of 10 ml of the composite was exposed to microwave irradiation for 30 seconds for trial. The power used was 180 W. There was no arcing at all. Longer exposures (1 to 2 minutes) of microwaves were tried for the same composite, again there was no arching. It seems either the forced air can blow out some of the styrene out of the cavity or the cavity is shielded from the HV transformer. A beaker of 50 ml vinyl ester composite VE/FLYASH (33%) was then located in the cavity of the oven and a power level of 180 W was selected to cure the composite.

The ambient temperature was 19 °C and relative humidity was 25%. Temperature was recorded every 1 minute of microwave irradiation.

After one minute of exposing the composite to microwaves, the beaker was taken out and the temperature was measured. The temperature was 33 °C and there was no noticeable change in the composite. The composite was then re-located into the oven cavity and exposed to microwaves for another minute. It was found that the volume of the composite expanded dramatically (by about 15%) and the temperature was 80 °C. A drop (projection on top surface of a casting) with a hard crust floating on the uncured composite beneath was found. The surface finish was rough. It appeared that the increase in volume had been at the expense of the formation of voids in the composite. The crust was forced into the uncured composite, by a piece of small stick, which then covered most of it. The composite was then exposed to microwave irradiation for another 30 seconds. Temperature was found to be 119 °C and the whole composite was hard. It seemed that curing had completed. The volume increased a little bit further to 20% more than the original (50 ml). The surface was rough and irregular. After cutting the composite block through, it was found that voids appeared in the middle part of the block. It can be argued that the heat generated by microwave sped up the evolution of gases from the cast component and the volume increased.

In another occasion, a power level of 180 W and exposure duration of 75 seconds, were selected to cure 50 milliliters of VE/FLYASH (33%) in a beaker. The ambient temperature was 24 °C and the relative humidity was 26%. The oven cavity temperature was 28 °C because the oven has been exposed to open space and under sunshine for 5 minutes. An ambient temperature of 28 ^{o}C should be used in carrying out analysis. After taking out the composite, its temperature was found to be 90 ^{o}C, which was 5 ^{o}C higher than the maximum temperature reached in curing 50 milliliters of the composite under an ambient condition of 19 ^{o}C and relative humidity of 25%. It was found that a drop with partially cured crust was formed. The volume of the drop was about 4 milliliters. The other part of the composites was not cured and was a rubbery solid. Forty five seconds later, the temperature was 135 ^{o}C and the composite became cured. At time equaled to 35 minutes, the temperature dropped back to 24 ^{o}C. After 24 hours, the height of the level of the composite was measured and was found to be 31.22 mm. The original height was 29.09 mm. This results in an increase in volume by 10.2ml. Adding the volume of the drop formed during curing, the total increase in volume was 14.2 ml. In this case, there was no shrinkage but an expansion in volume. The increase in volume was 28.4%. The resulting structure had a lot of spores and the strength of the composite was expected to be inferior. From Table 3.4, the volume shrinkage will be decrease by following the microwave exposure time increase.

Table 3.4: Volume shrinkage and other parameters for 200 ml of VE/FLYASH (33%) exposed to 180-W microwaves at different duration

Microwave exposure time (seconds)	0	30	35	40
Oven cavity Temperature (^{o}C)	20	28	25	25
Temperature after microwave exposure	NA	41	45	52
Original volume (ml)	200	200	200	200
Final volume (ml)	187.2	202.32	199.36	200.06
Volume shrinkage (%)	6.4	1.16	0.32	-0.03
Volume at maximum temperature (ml)	187.22	204.64	201.28	201.00
Time to reach gel time (minutes)	32.5	3	1	1
Maximum temperature	117	143	144	145
Time to reach maximum temperature (minutes)	37.5	9	6	6

The decision to microwave the composite for 75 seconds in the second trial has been made in accordance with the results obtained in the first trial. In the first trial, the temperature was 33 ^{o}C after the composite was exposed to microwaves for 60 seconds. It seemed that by exposing the composite to an extra 15 seconds to microwaves, the temperature would be raised to around 10-20 degrees above the ambient temperature. This was wrong, the temperature after exposing the composite to microwaves for 75 seconds was 90 ^{o}C, which was 5 ^{o}C above the peak temperature for curing the composite in ambient conditions. This is due to the difference in ambient temperature and relative humidity in two occasions. They are 9 ^{o}C (oven cavity temperature) and 7 % higher in temperature and relative humidity in the microwave cured conditions than in ambient conditions respectively. With the ambient conditions of the second trial, the microwave exposure time should be 40 seconds or less. This may result in an increase in temperature of 10 –20 degrees Celsius. A drop formation could also be avoided. The composite was then cured under ambient conditions. The final volume is expected to increase or decrease slightly in accordance with temperature reached by the application of microwave irradiation.

In the third trial, the ambient conditions were the same as in the second trial. After taking out the composite, its temperature was found to be 44 ^{o}C, which was still far away from the maximum temperature in the second case. At time equaled to 9 minutes, a peak temperature of 137 ^{o}C was attained and the volume was 54.74 ml, an increase of 2.32 % from 53.5 ml. The composite was also cured. At time equaled to 40 minutes, the temperature returned to 24 ^{o}C. After 24 hours, the volume of the composite was measured and was 52.27 ml. The percentage of shrinkage was 2.30 %.

Chapter 4

Impact Strength and Testing

4.1 Introduction

Impact tests are performed to measure the response of a material to dynamic loading. There are three impact tests. One is known as the Izod impact test, Charpy impact test and Drop-weight test. Impact testing studies the dependence of notch toughness or impact energy on the temperature. The impact strength is one of the most commonly tested and reported properties of composites. It means the amount of energy consumed to break a test specimen divided by the unit-width of the specimen, when the specimen is broken by the stroke with the pendulum-type or drop-type hammer having specific energy and linear velocity. For analyzing the impact strength of the impact test, a charger amplifier is used. Most of the test machines can be provided with strain gauge instrumentation. Instrumented test systems enable measurement of the force applied to the test specimen during an impact event. Then**,** the instrumented test data can be used to calculate the energy absorbed by the test specimen. In addition, the crack initiation and arrest loads can be used in fracture mechanics models.

4.2 Impact Properties

4.2.1 Impact Testing

Impact testing measures the energy required to break a specimen by dynamically applying a load. Impact strength is one of the most commonly tested and reported properties of composites. As the composites industry grows so do the number of different methods for measuring impact strength.

4.2.2 Charpy Impact Test

(a) Definition

- This test is actually the modification of the Izod test. Charpy modified the test by orientating the specimen in a horizontal position instead of the vertical position as in the Izod test.

(b) Test method

- The two tests is worked by the apparatus is illustrated in Figure 4.1. The weighted end of the pendulum is raised to a fixed height, h, and is then released.
- The knife-edge mounted on the pendulum will strike and fracture the notch.
- The notch is the stress raiser, where the stress of this high velocity impact blow is concentrated.

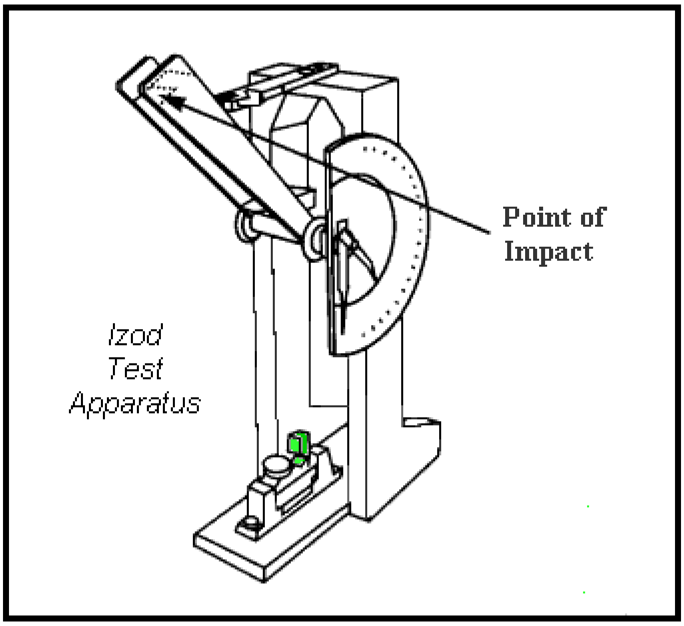


Figure 4.1: Pendulum Machine

- Due to torque, the pendulum arm would continue swinging after fracturing the notch, but the amplitude of its swing will not be as high as when it was first released from the height, h, due to the lost of its energy when fracturing the notch. (The notch would absorb some energy before it fractures so the energy is lost here).
- This maximum displacement is noted down as h_2. The difference in height ($h - h_2$) is actually the energy absorbed which is also known as the impact energy. Figure 4.2 was show a Charpy test specimen.

Figure 4.2: A Charpy test specimen

- The specimen is broken by the impact of a heavy pendulum hammer, falling through a fixed distance (constant potential energy) to strike the specimen at a fixed velocity (constant kinetic energy).
- Tough materials absorb a lot of energy when fractured and brittle materials absorb very little energy.

(c) Note

- This approach is often still used to specify minimum impact energy for material selection, though the criteria are also based on correlations with fracture mechanics measurements and calculations
- To understand how the Charpy impact energy is affected by the properties of the material, we need to understand the different contributions which make up the measured energy.

4.2.3 Izod Impact Test

(a) Definition

- Izod invented this test for determining a suitable metal to be used as cutting tools. His test involves a pendulum with a weight that is known at the end of its swinging arm.
- The pendulum would strike the specimen, as it stands clamped in a vertical position. The specimen is in the shape of a bar with a square cross section. The bar is machined into a V-notched.

(b) Test method

- The shape and the size of the test specimen follow ASTM D-256.
- A notch with the angle of incidence of 45 degree and the bottom radius of 0.25 with diameter 0.05mm is cut in each of the injection molded specimens.
- After the notches are cut, the specimens are annealed at 23℃±2℃ and 50%RH±5%RH for more than 48hrs before the test.
- Figure 4.3 shows the measuring method for Izod impact strength.

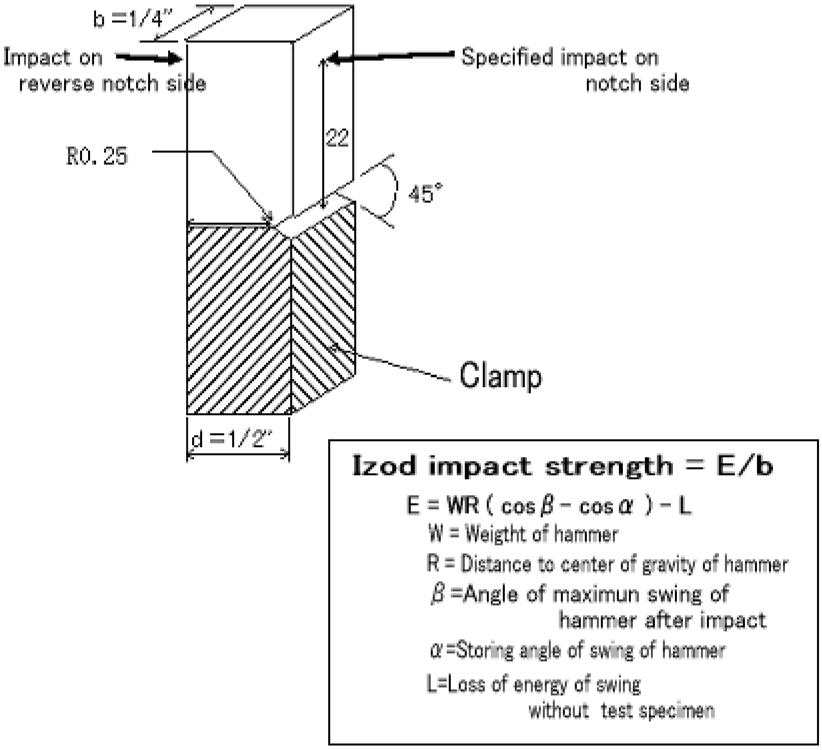


Figure 4.3: Measuring method for Izod impact strength

(c) Note

- These data are obtained with the simple test specimens. Therefore, the properties of practical moldings do not always conform to these data. These data should be taken for references.

4.2.4 Measuring impact toughness

The important factors which affect the toughness of a structure include low test temperatures, extra loading and high strain rates due to wind or impacts and the effect of stress concentrations such as notches and cracks. These all tend to encourage fracture. To some extent, the complex interaction of these factors can be included in the design process by using fracture mechanics theory.

In circumstances where safety is extremely critical, full scale engineering components may be tested in their worst possible service condition. For example, flasks for the transportation of nuclear fuel were tested in a full scale crash with a train to demonstrate that they retained their structural integrity (a 140 tonne locomotive and three 35 tonne coaches at 100 mph crashed into a spent fuel flask laid across the track with its lid facing the train. The train was demolished but the flask remained sealed. The peak impact force of the test train was greater than that of an Inter-City 125 traveling at 125 mph. Inter-City 125 was a diesel train and it could often significantly reduce journey times not only because of its high speed but also because of the much more superior acceleration/deceleration than other diesels. A theoretical fracture mechanics structural integrity assessment of this situation would have been very difficult.

Such full scale tests are extremely expensive and are very rarely conducted. Fracture mechanics is also a fairly recent development in engineering design, and measurement of the fracture toughness parameters that are required to perform a structural integrity assessment during the design process (such as K_{1c}) is quite time-consuming and expensive.

Tests for the impact toughness, such as the Charpy Impact test were developed before fracture mechanics theory was available. The impact test is a method for evaluating the relative toughness of engineering materials. The Charpy impact test continues to be used nowadays as an economical quality control method to assess the notch sensitivity and impact toughness of engineering materials. It is usually used to test the toughness of metals. Similar tests can be used for polymers, ceramics and composites.

4.2.5 The Impact Energy

The impact energy measured by the Charpy test is the work done to fracture the specimen. On impact, the specimen deforms elastically until yielding takes place (plastic deformation), and a plastic zone develops at the notch. As the test specimen continues to be deformed by the impact, the plastic zone work hardens. This increases the stress and strain in the plastic zone until the specimen fractures. The Charpy impact energy therefore includes the elastic strain energy, the plastic work done during yielding and the work done to create the fracture surface. The elastic energy is usually not a significant fraction of the total energy, which is dominated by the plastic work. The total impact energy depends on the size of the test specimen, and a standard specimen size is used to allow comparison between different materials.

The impact energy is affected by a number of factors, such as:

a) Yield Strength and Ductility
b) Notches
c) Temperature and Strain Rate

d) Fracture Mechanism

4.2.5.1 Yield Strength and Ductility

Increasing the yield strength of a metal by processes such as cold working, precipitation strengthening and substitution or interstitial solution strengthening generally decreases the ductility. This plastic strain to failure is shown in Figure 4.4.

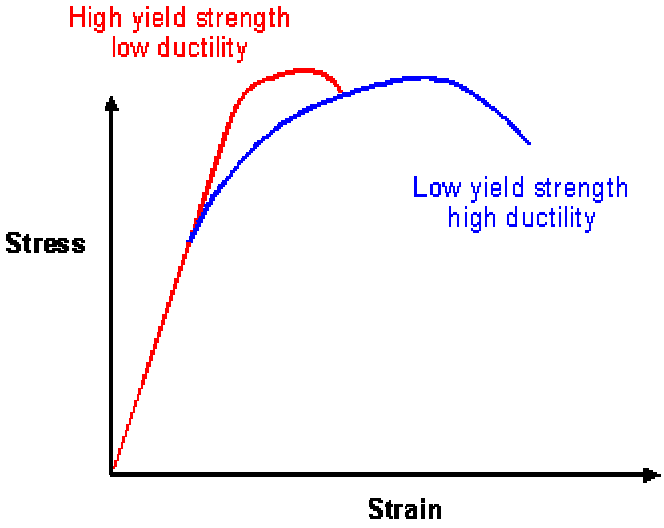


Figure 4.4: The yield strength, tensile strength and ductility

Increasing the yield strength by these mechanisms therefore decreases the Charpy impact energy since less plastic work can be done before the strain in the plastic zone is sufficient to fracture the test specimen. An increase in yield strength can also affect the impact energy by causing a change in the Fracture Mechanism.

4.2.5.2 Notches

The notch in the test specimen has two effects. Both can decrease the impact energy. First, the stress concentration of the notch causes yielding or plastic deformation to occur at the notch. A plastic hinge can develop at the notch, which reduces the total amount of plastic deformation in the test specimen. This reduces the work done by plastic deformation before fracture. Secondly, the constraint of deformation at the notch increases the tensile stress in the plastic zone. The degree of constraint depends on the severity of the notch (depth and sharpness). The increased tensile stress encourages fracture and reduces the work done by plastic deformation before fracture occurs.

Some materials are more sensitive to notches than others and a standard notch tip radius and notch depth are therefore used to enable comparison between different materials. The Charpy impact test therefore indicates the notch sensitivity of a material.

4.2.5.3 Temperature and Strain Rate

Since the Charpy impact energy comprises mostly of the plastic work of yielding of the specimen, it is affected by factors which change the yield behavior of the material, such as temperature and strain rate, through their effect on the behavior of dislocations.

Increasing the yield strength by low temperatures or high strain rates decreases the ductility, and therefore decreases the Charpy impact energy. The yield strength of body centred cubic (bcc) metals is more sensitive to strain rate and temperature than that of face-centred cubic (fcc) metals. The Charpy impact energy of bcc metals such as ferritic carbon steel therefore has a stronger dependance on strain rate and temperature than that of fcc metals such as aluminum, copper and austenitic stainless steel.

4.2.5.4 **Fracture Mechanism.**

The Charpy impact energy is affected by changes in the fracture mechanism. For metals, there are two types of fracture surfaces. Where the temperature at which fracture occurs is high, the surfaces are dull or fibrous and show that shearing has occurred. Where the temperature is low, the surfaces have a granular or shiny texture, showing that cleavage has occurred. There is a range of temperatures at which the surfaces have both characteristics. The rough and fibrous surfaces are caused by the ductile fracture mechanism. At high temperatures the Charpy V-notch impact energy is high and ductile fracture occurs by plastic flow. At the point of fracture, the cross-sectional area is reduced. However, the smooth or granular surfaces are caused by the brittle fracture mode which occurs by cleavage. There is a clean break and not much deformation occurs. There is a little reduction in cross sectional area at the point of fracture.

Polymers also exhibit brittle and ductile fracture according to temperature. At low temperatures, the surfaces are smooth, glassy and fractured with some splintering. The clean fracture break shows that the material is brittle. At high temperatures, there is no significant appearance to the fracture. However the cross sectional area is reduced considerably. Ductile fracture occurs and there is tearing at the notch.

- **Brittle and Ductile Fractures**

  There are two kinds of fractures, which is ductile fracture and brittle fracture. With ductile fracture, there is a large plastic region where plastic deformation occurs during crack propagation. The fracture process occurs slowly as the crack propagates. This crack is considered stable as it resists further propagation with an increase in applied stress. Conversely, cracks propagate rapidly in brittle fracture with very small plastic region. The crack also propagates continuously and spontaneously without an increase in applied stress.

4.2.6 Drop Weight Test

(a) Definition

- In this research, drop weight test had been used for testing of specimens under impact loading.
- Drop weight test method is a falling drop weight impact test.
- This drop weight test is better than a number of the conventional tests like Charpy and Izod tests in its ability to produce the conditions under which a ‘real life’ component would be when subjected to impact loading.
- Using the instrumented drop weight test method in impact testing, it can provide more information about the impact.

- Accelerometer, charge amplifier and data acquisition system will be required in this testing.

(c) Test method and equipments

- The drop weight test method uses the impact tup (hammer) for dropping test.
- In Figure 4.5, the pointer area is cable protected. This cable protection is the way to minimize the cable noise as well as to protect the connector between the micro-cable and accelerometer from breaking due to the force acting on it were considered seriously.

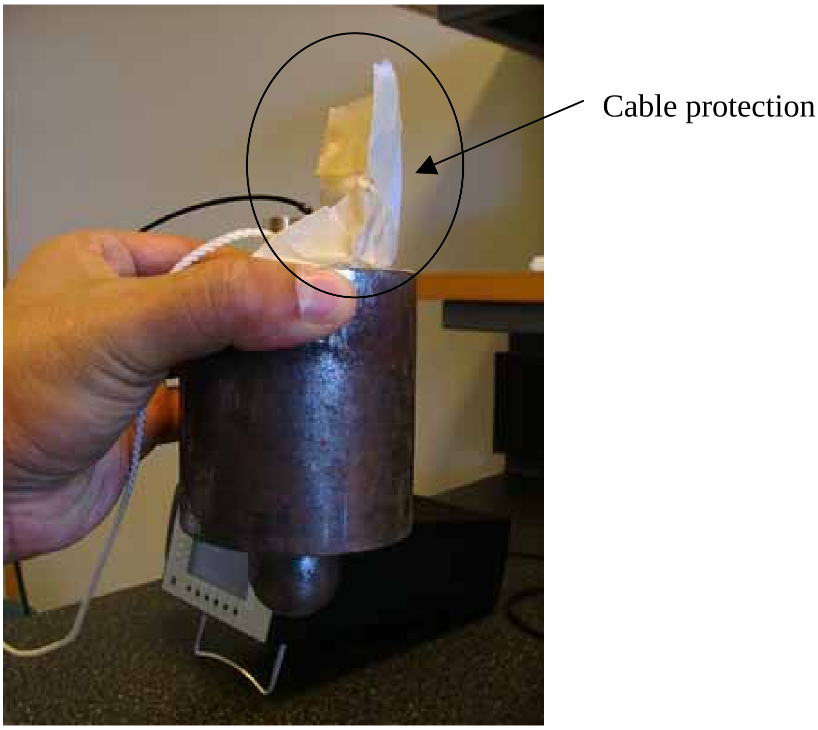


Figure 4.5: The protection of micro cable

- The accelerator was using in this research is “Bruel and Kjaer” piezoelectric delta shear type (Type: 4371).
- The advantages of using piezoelectric accelerometers over other types of accelerometers are (Serridge and Licht, 1986) broad frequency range, good linearity in dynamic applications and performance in a wider range of environmental conditions and the data can be integrated.
- The accelerometer is connected with micro cable to the amplifier. It place on the top of the impact tup. This micro-cable mainly used for sending signal from the accelerometer to the charge amplifier. The connection of the impact tup has shown in Figure 4.6.

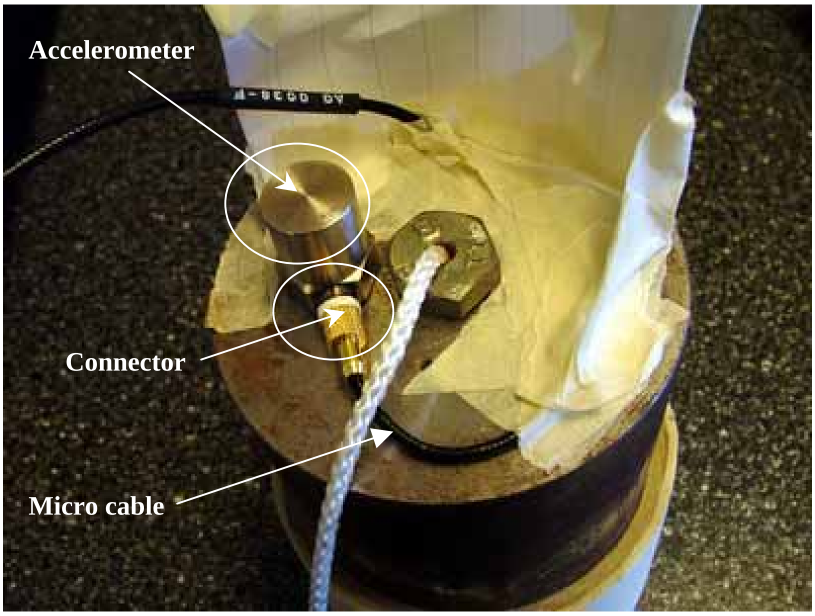
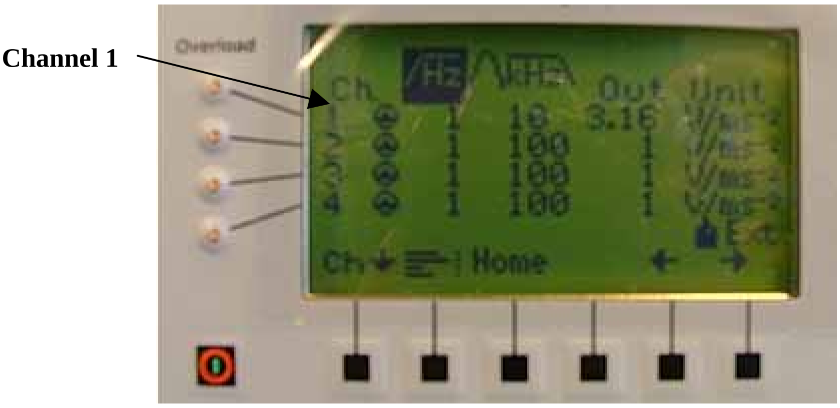


Figure 4.6: The connection of the impact tup

- Charger amplifier was used to amplify the signal from the accelerometer to an appropriate level so that it can be displayed. The mainly reasons for using charger amplifier is that it can provide more accurate result and more information in the test like force distribution, peak force, duration, energy required to initiate the crack and propagating them. Figure 4.7 is showing the charger amplifier that was using in this research.

Figure 4.7: Charger Amplifier (Left) and impact tup (Right)

- The setting of the band pass filter is 1Hz – 10 kHz with an output of is 3.16 V/ms^{-2}, this is an important signal that produced by accelerometer. From the figure 4.8, it is pointing the micro cable is connecting to the channel 1.



Ch	Hz	kHz	Out	Unit
1	1	10	3.16	V/ms-2
2	1	100	1	V/ms-2
3	1	100	1	V/ms-2
4	1	100	1	V/ms-2

Figure 4.8: The setting of the amplifier

- Charger amplifier and the PC cable connection have shown in the figure 4.9.

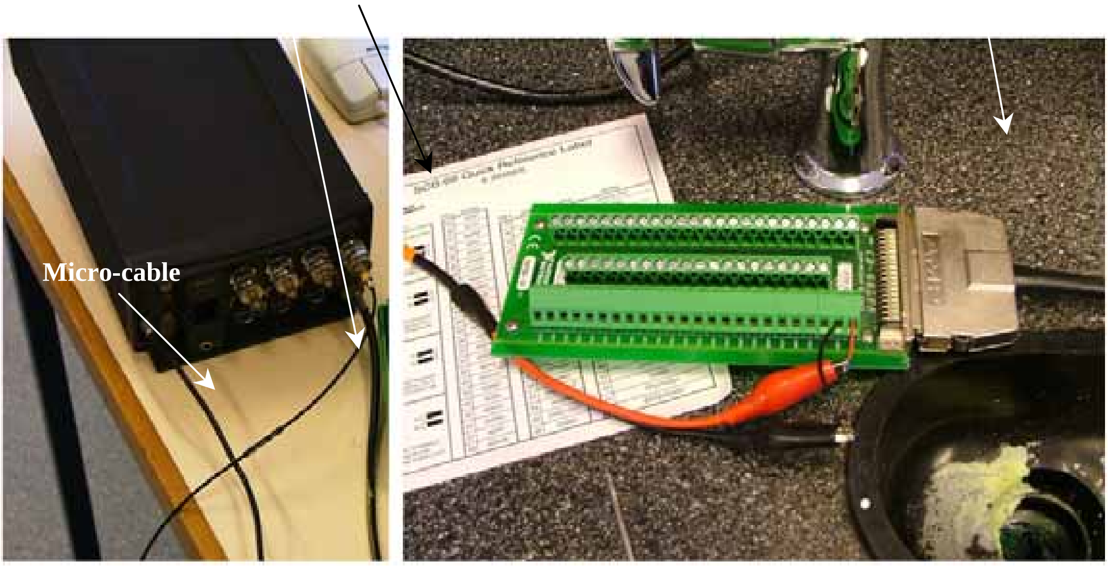
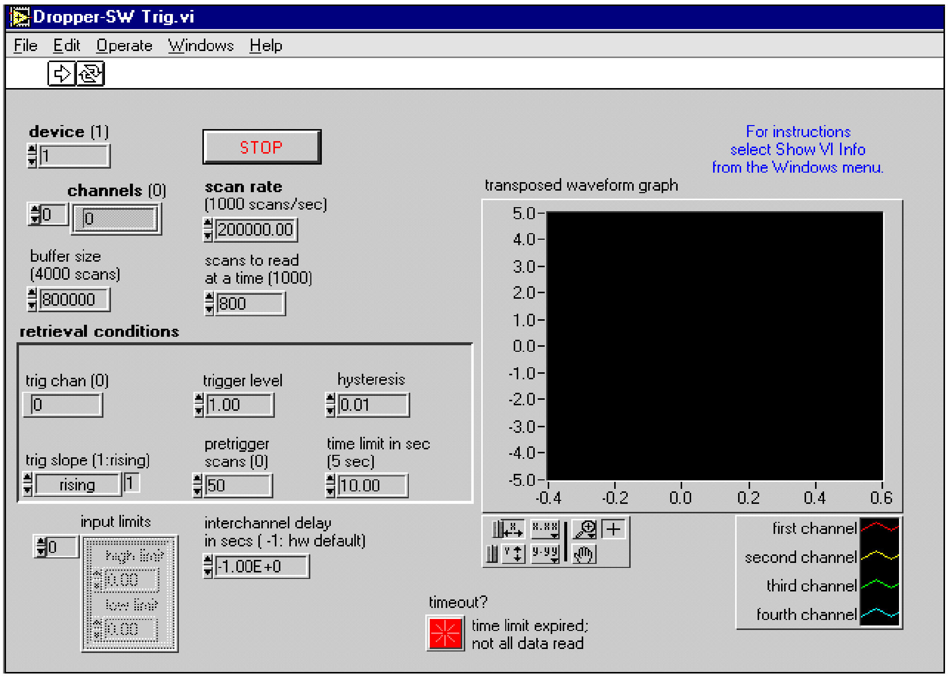


Figure 4.9: The connection between the amplifier and PC

- The sampling interval was set to 5 microseconds that gave a total recording time of approximately 4 milliseconds. The specimens of [VE/FLYASH (33%)] cured under ambient condition were tested first, followed by the specimens cured under microwave conditions as well as [VE/FLYASH (25%)] cured under ambient condition.

- The data acquisition hardware is used for data processing. It was attached to the PCI slot on the motherboard of computer. This can be used to acquire signal with a sampling rate of 200k samples/second from the charge amplifier. All the setting is shown in the Figure 4.10.



For instructions
select Show VI Info
from the Windows menu.

device (1): 1

channels (0): 0 | 0

buffer size (4000 scans): 800000

scan rate (1000 scans/sec): 200000.00

scans to read at a time (1000): 800

retrieval conditions

trig chan (0): 0

trigger level: 1.00

hysteresis: 0.01

trig slope (1:rising): rising | 1

pretrigger scans (0): 50

time limit in sec (5 sec): 10.00

input limits: 0

high limit: 0.00

low limit: 0.00

interchannel delay in secs (-1: hw default): -1.00E+0

timeout? time limit expired; not all data read

Figure 4.10: The setting of sampling rate from charger amplifier

- The data collected can be directly stored into the computer after data processing.
- The result will be taken by using different height level of impact. Figure 4.11 shows the height level of the impact test and the position of the impact point. All the marking level is in millimeter (mm).

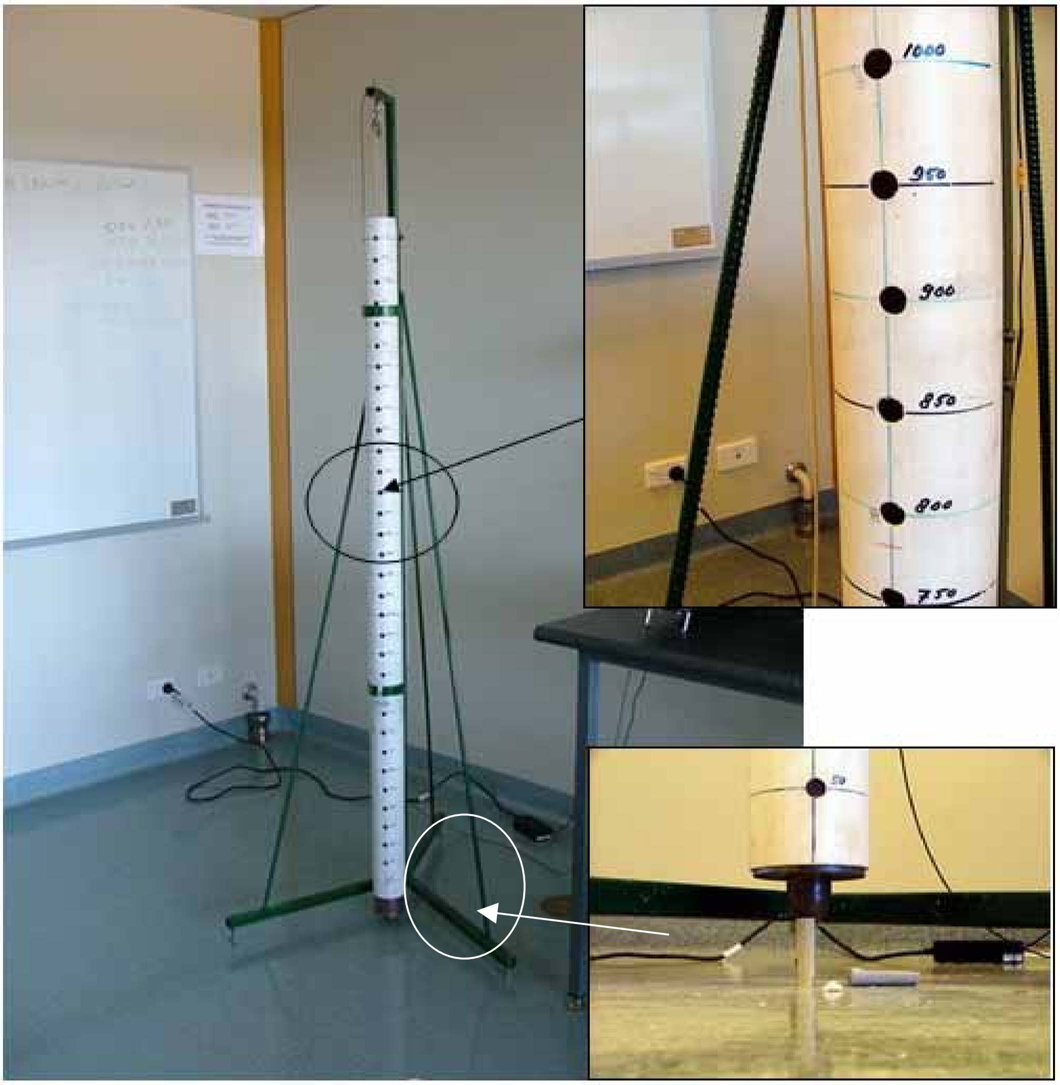


Figure 4.11: The height level and the position of impact test

- After data processing, the data collected from the impact testing was processed with Microsoft Excel or Matlab.

(c) Note

- These data were obtained with the simple test specimens. Therefore, the properties of practical moldings do not always conform to these data. These data should be taken for references only.

Chapter 5

Experimental Method

5.1 Introduction

This chapter will discuss about the experimental design of this research. The first is the processes of making the specimens. This section mainly discusses the casting moulds and the calculations of material quantity for VE/FLYASH 25% and 33%. Safety measures and steps taken in casting specimens will be discussed in the second section. The next section is discussing the correct and safety ways for using microwaves to cure the specimens. This part mainly discusses the height of the drop hammer as well as the setup of the test rig guide tube. Besides that, some of the results of the drop weight test will also be discussed in this section. This is to compare different conditions of curing the specimens. The final section of this research is fracture analysis; it is to investigation the fractured surface on these specimens.

5.2 The Specimens

5.2.1 Introduction

In this research, there were two combinations by weight of resins and the fly ash. They were 25% and 33% by weight flyash particulate reinforced vinyl esters VE/FLYASH (25%) and VE/FLYASH (33%). Both of them will be cured under different conditions, including microwave condition.

For ambient condition, it is cured under room temperature without any microwave treatment. Microwave conditions have 9 different curing ways; it is curing under different power levels of microwaves and in different exposure times as well. The suitable power level and time of exposure of microwave will make the specimens stronger as well as having low shrinkage.

Figure 5.1 shows the curing conditions employed for the specimens; they were broken down into two main conditions, ambient condition and microwave conditions. In the group of curing under microwave conditions, it was broken down into three more groups by having the specimens cured under three power levels of 180 Watts, 360 Watts and 540 Watts respectively. For 180W and 360W power levels, the exposure times are 30 seconds, 35 seconds and 40 seconds, and for 540W power level, the exposure times are 10 seconds, 15 seconds and 20 seconds.

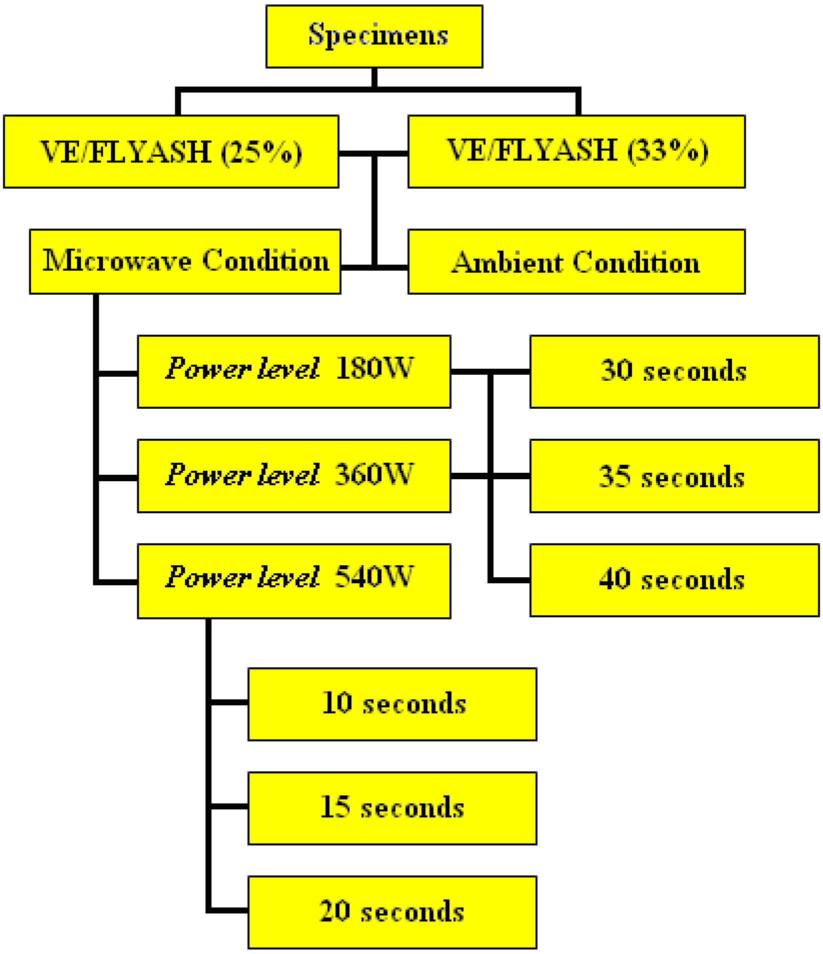


Figure 5.1: Grouping of production of specimens

5.2.2 Casting Moulds

The specimens were to be exposed to microwave so the moulds must be non-metallic. It was decided to use PVC pipe because it is most appropriate as it can be exposed to microwaves. Another reason to use of PVC pipe is that a smaller cast of material with a smoother surface finish could be made. Figure 5.2 shows moulds of the specimens.

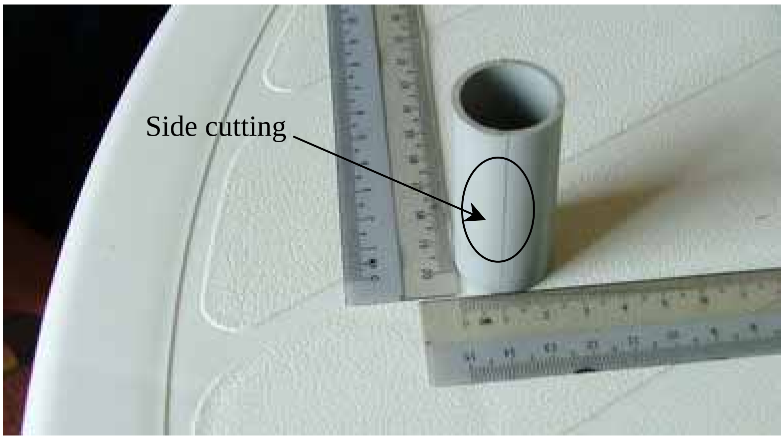


Figure 5.2: The casting mould

The specimens is 50mm long and the height of the moulds is therefore 60 mm. Figure 5.3 shows the dimensions of the PVC pipe, the outer and inner diameters are 25mm and 21mm respectively. Figure 5.2 illustrates that a cut made on the side of PVC pipe so that the specimen cast can be easily taken out.

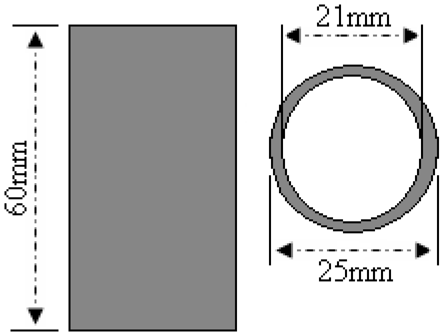


Figure 5.3: The dimension of mould casting

The inner surface of the mould must be clean-up because it will affect the surface of specimen. The bottom section has to be sealed with paper tape to restrict the leaking of the uncured composite during the pouring process. Figure 5.4 shows the dirt on the inner of the moulds.

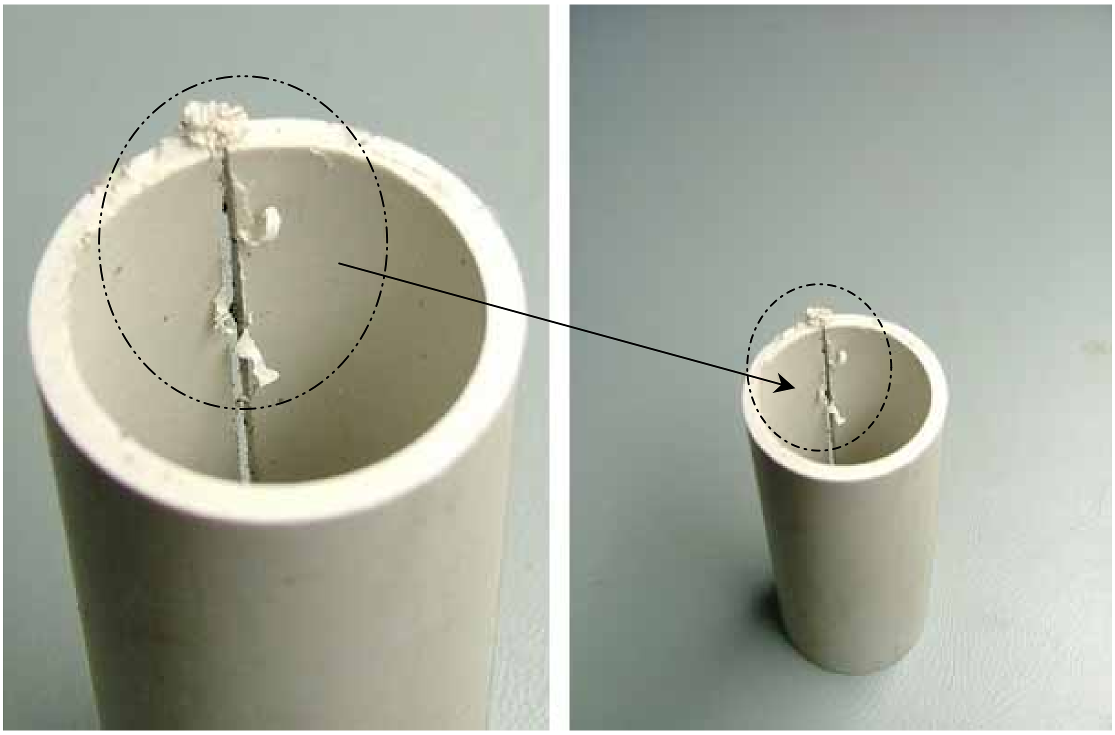


Figure 5.4: The unclean surface of the specimens

5.2.3 Weights of Materials

This section will discuss about the mixture and the mass calculations of the materials.

The materials used were:

- Vinyl Ester Resin (Hetron 922 PAW)
- Methyl Ethyl Ketone Peroxide (MEKP)
- Flyash (Ceramic Hollow Sphere)

5.2.3.1 VE/FLYASH (33%)

Table 5.1 shows the weight of materials required to make 500ml of VE/FLYASH (33%) (Ku, 2003). VE/FLYASH (33%) means resin + MEKP 67% by weight and fly ash 33% be weight.

Table 5.1: Weight of materials required to make 500 ml of VE/FLYASH (33%)

Materials	**Resin**	**MEKP**	**Flyash**	**Composite**
Parameters				
Relative density	1.05	1.18	0.7	---
Percentage by volume	56.5	1	42.6	100
Percentage by weight	67	---	33	100
Weight for 600 g of composite	**394 (g)**	8 (g) or **7 (ml)**	**198 (g)**	---

Other volumes of the composites can be easily calculated by ratio from Table 5.1. For small volume of the specimens, 15 to 20 specimens can be cast at the same time. The mass calculations required to fracture these specimens are attached as Appendix E.

5.2.3.2 VE/FLYASH 25%

Table 5.2 shows the weight of materials required to make 500ml of VE/FLYASH (25%) (Ku, 2003)

Table 5.2: Weight of materials required to make 500 ml of VE/FLYASH (25%)

Materials	**Resin**	**MEKP**	**Flyash**	**Composite**
Parameters				
Relative density	1.1	1.0	0.7	---
Percentage by volume	65.4	1.2	33.4	100
Percentage by weight	75	---	25	100
Weight for 600 g of composite	**441(g)**	9 (g) or **8ml**	**150(g)**	---

5.2.4 Safety Measures

The safety measures when processing the specimens included risks of styrene, risks of MEKP and risks on microwaves and vinyl ester resins interaction. Due to these high risks involved in specimen making, various safety measures must be considered seriously.

The testing and specimen making were carried out in the Faculty of Engineering and Surveying, USQ. Before mixing the material, two different kinds of safety gloves were required to be worn, one after the other. The 1st layer is mainly for protecting skin and it is thicker and less porous than the 2nd layer. The 2nd layer glove is just for extra protection.

Goggles were worn for eye protection and it must be worn while mixing the mixture because the styrene emitted will be harmful to eyes. Besides that, mask was also used to avoid any inhalation of styrene vapour into the body. The exhaust fan must also be on during mixing the material. If not, the styrene will always be in the room. The gloves must be thrown away after use and hands have to be washed before leaving the room.

While curing the specimens with microwave, the microwave should be located outdoor as the styrene go to the open air. Figure 5.5 shows the microwave oven that is used in this research project. During microwave processing, the door of the microwave oven should be facing onto a wall to reduce damage in case of explosion.

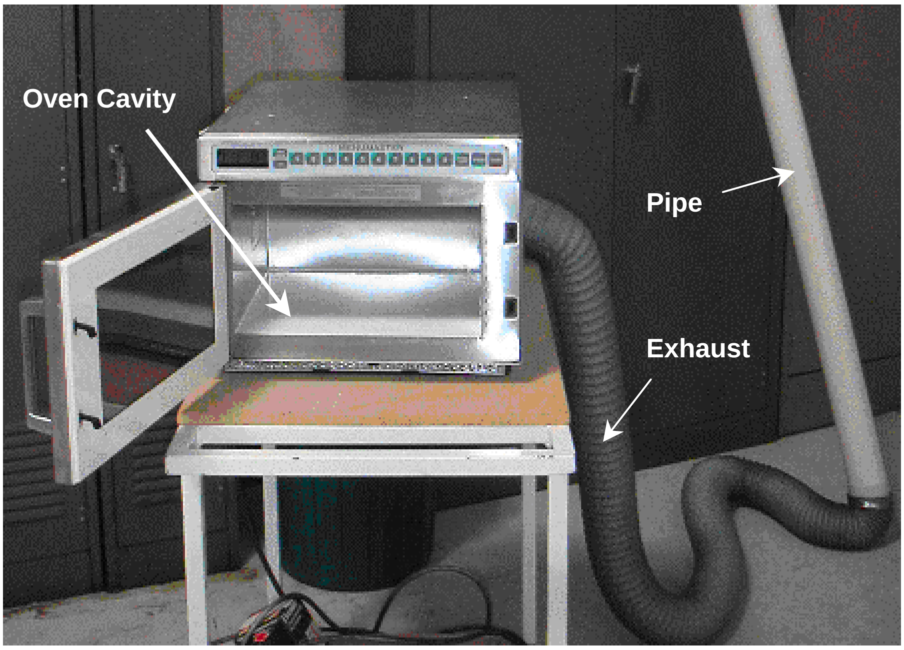


Figure 5.5: Microwave Oven

5.2.5 The Processes of Producing Specimens

The processes of producing specimens are listed below:

Step 1: Clean or remove any particles on the inner surface of the mould. Seal the side and bottom part of the casting moulds to stop leaking because when the composite is poured into the moulds.

Step 2: Mark the height of specimens on inner surface of the mould with pen. This gives the height to which the composite should be poured. Spray some canola oil on the inner surface of the mould for ease of stripping when the composite is cured.

Step 3: Prepare the amount of different materials in different containers as tabulated in Table 5.1. All weights were measured using an electronic weight scale as illustrated in Figure 5.6

Figure 5.6: The electronic weight scale (right) and Canola Oil (left)

Step 4: Flyash was the first weighed by using an ice-cream plastic box that sat on an electronic weight scale. The vinyl ester resin was then poured and weighed with the use of another ice-cream plastic box. The volume of hardener required was dispensed out from the special chemical dispenser so that it was ready for use (See Figure 5.7). The resin was then mixed with the fly ash. After this, pour the hardener into the resin/flyash mixture and mix it together until the harder has been totally mixed in resin and flyash.

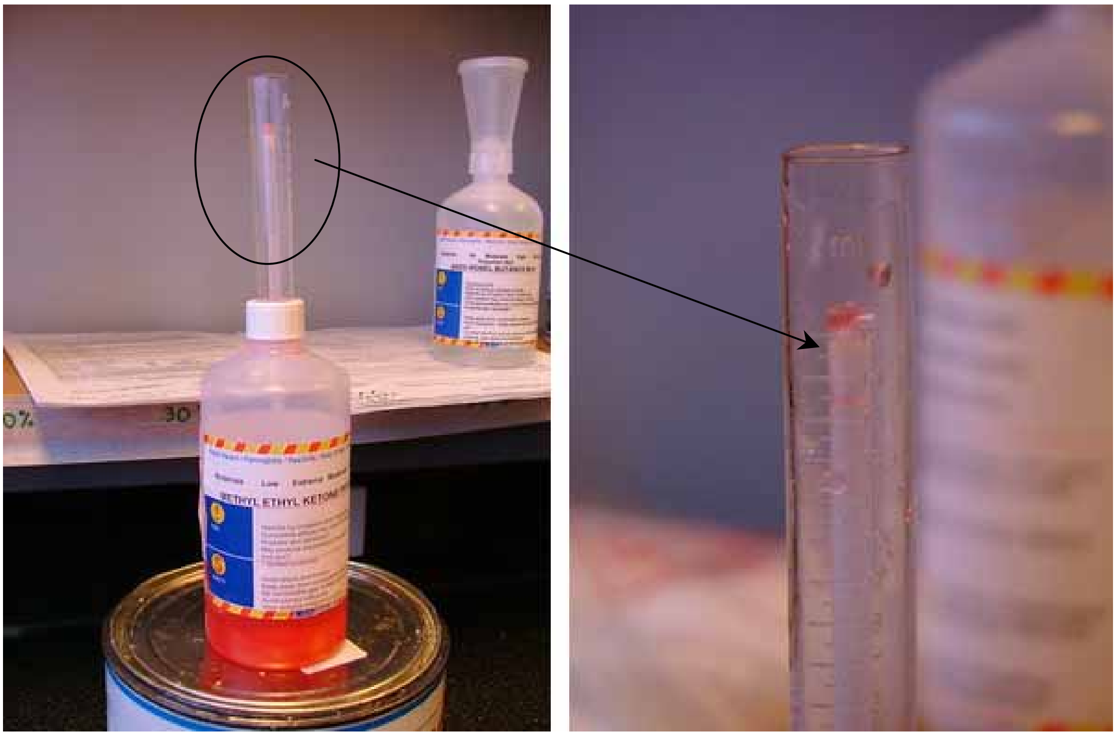


Figure 5.7: MEKP dispenser

Step 5: After mixing these materials, they were poured into the moulds. Use the infrared thermometer to measure and record the temperature on each casting. Figure 5.8 shows mixtures had been poured into the moulds.

Figure 5.8: The mixture were pour into the mould

Step 6: For microwave treated specimens, it will be cured in microwave with different power levels and different times of exposures. Figure 5.9 shows the microwave menu and the arrows point to power level selection and time entry button.

Figure 5.9: microwave menu

Step 7: After microwave treatment, the temperature of the composite was measured again.

Step 8: The mixtures will be completely cured after 24 hours and the composite was stripped out from the bottom part of the mould.

Step 9: Make sure the surfaces of the specimens are flattened before impact test, otherwise the result will be inaccurate.

5.3 Preliminary Testing

5.3.1 Introduction

The drop weight impact test was used in this research. It is used to find out which type of curing conditions has higher impact strength. Dropper will be dropped at different height to find out the potential energy required to initiate crack in the sample. The results of impact test are important as it will form a better guide for the composite industry in producing their items.

5.3.2 Drop Weight Tower

The drop weight tower is one of the equipment used in the drop weight test. The drop weight tower and its dimension is shown in Figure 5.10. The maximum height of this drop weight tower is 1.5 meter but the height used in this research ranges from 250mm to 500mm.

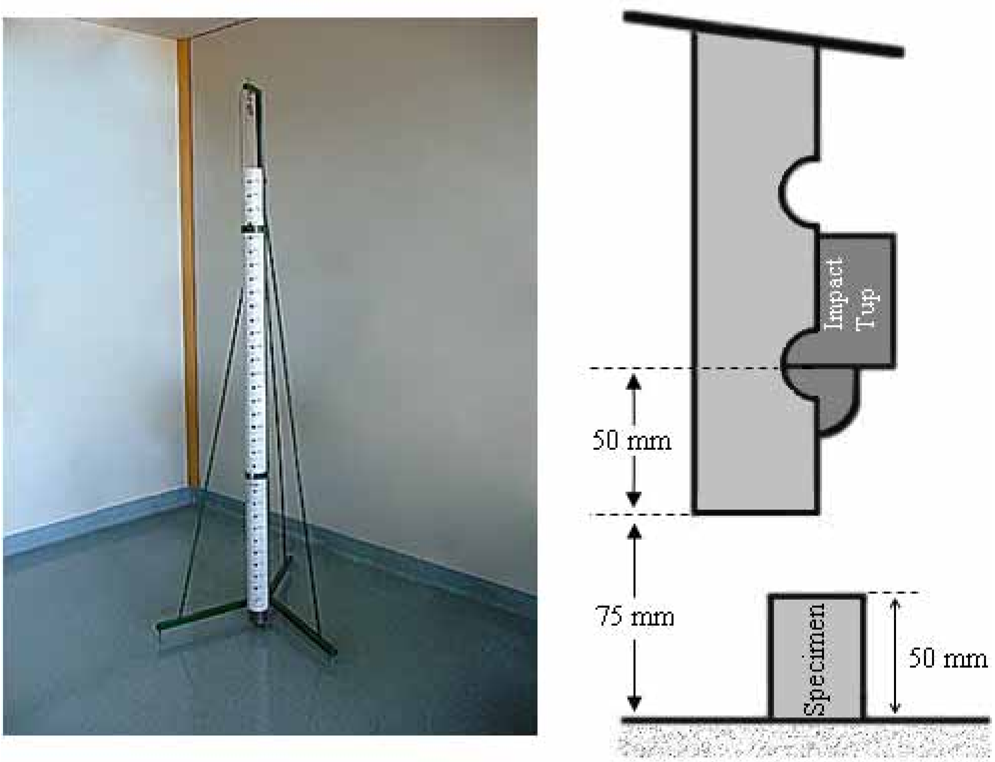
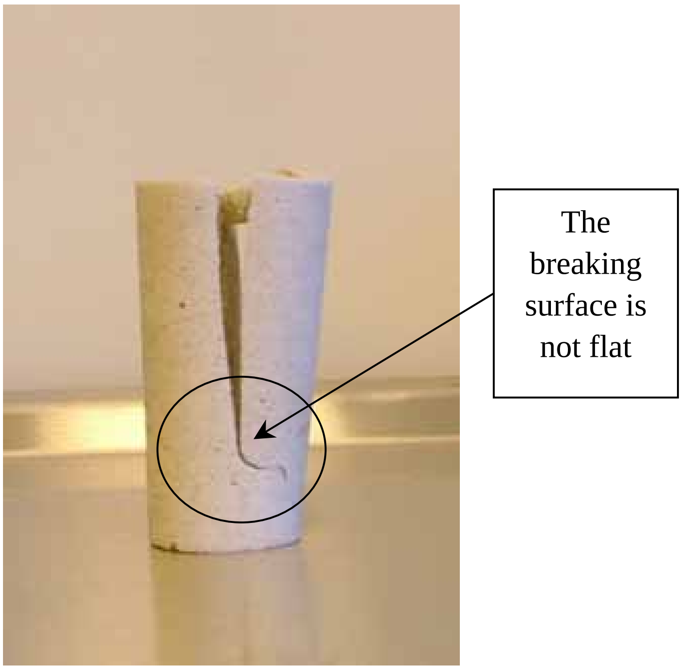


Figure 5.10: Drop Weight Tower

5.3.3 Drop Weight Testing

The specimens used for impact were VE/FLYASH (33%). The dropper will be dropped from the range of 250mm to 500mm height; each increment will be 50mm. The stair-case method was used to determine the height of the tup for the short rod test. After impact, the specimens were analyzed and separated into fractured or non-fractured. It has to be ensured that the impact tup must be hitting on the middle of the specimen; otherwise the result will be inaccurate. A fractured specimen is shown in Figure 5.11.



The breaking surface is not flat

Figure 5.11: Failure impact specimens

Table 5.3 shows the impact test results of VE/FLYASH (33%) cured under microwave conditions of 540W and 10 seconds.

Table 5.3: Impact test results of VE/FLYASH (33%) cured under microwave conditions of 540W and 10 seconds

Specimens	VE/FLYASH (33%); 540W, 10 seconds					
Dimensions of Specimens (mm)	Diameter: 21mm Length: 50mm					
Symbol Representation	NF = normal fracture SF = seriously fracture O = non-fracture					
Specimen No.	1	2	3	4	5	6
Drop Height(mm)						
500						SF
450					NF	
400				NF		
350			O			
300		O				
250	O					

Personal computer data acquisition system was used in this research but it can only received voltage in the range of -10V to +10V and therefore oscilloscope was incorporated to increase the range of signal received.

5.4 Data Preparation

The instrumentation of the drop-weight test gave a lot of convenience to this research. It was easy to analyze the graphs of force versus displacement, acceleration versus velocity or force versus velocity.

By integrating the acceleration data (Equation 5.1), velocity distribution can be obtained. The displacement distribution (Equation 5.2) over time interval of the impact can then be obtained simply by integrating the velocity distribution. The t_0 and t_1 represent the times of first and last data points respectively.

$$v(t) = \int_{t0}^{t1} a(t)dt \tag{5.1}$$

$$s(t) = \int_{t0}^{t1} v(t)dt \tag{5.2}$$

The smoothing of the graph was made possible with the aid of Savitzky-Golay filter of the Signal Processing Toolbox in MATLAB ®. After smoothing the graph of acceleration versus time, the smoothed graph can then be converted into graph of velocity versus time followed by graph of displacement versus time and force versus displacement. Figure 5.12 and 5.13 show before and after using Savitzky-Golay filter respectively.

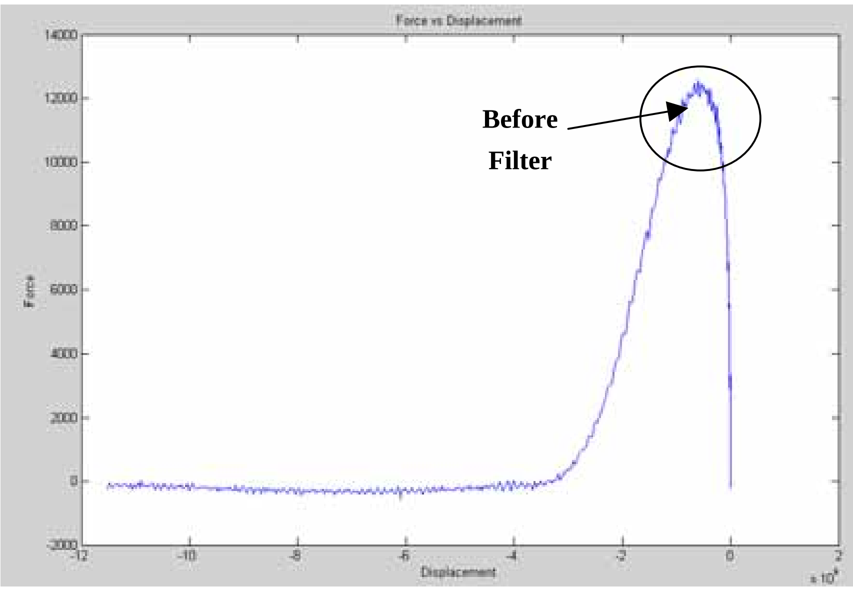


Figure 5.12: Noise along with the data acquired of curve force versus displacement

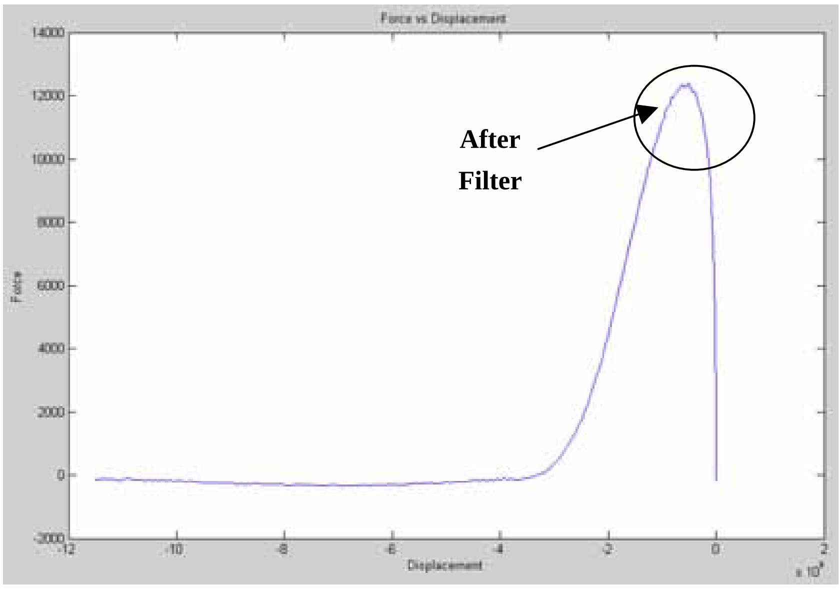


Figure 5.13: The curve of force versus displacement looks smoothly

The potential energy of the tub when the impact occurs can also be calculated by using of the formula of potential energy (Equation 5.3).

$$PE = mgh \tag{5.3}$$

Assuming negligible resistance from the rope as well as the guide tube, the velocity can be calculated by using formulae of conservation of energy and kinetic energy (Equations 5.4 through 5.6).

$$PE = KE \tag{5.4}$$

$$mgh = \frac{1}{2}mv^2 \tag{5.5}$$

$$v = \sqrt{2gh} \tag{5.6}$$

The proportion of energy absorbed up to the point where complete failure occurs can now be estimated by knowing the initial conditions of the impact using the above formula. Figure 5.14 shows force versus displacement, the curve after the point where peak force occurs is the energy required to propagate the crack.

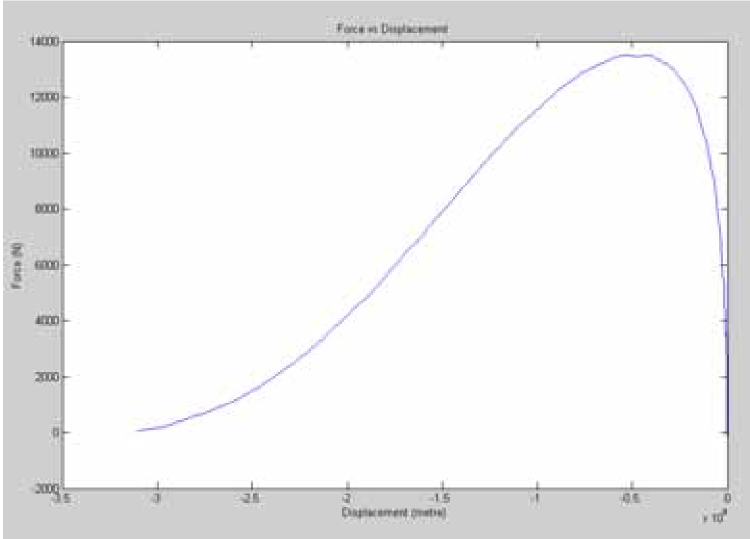


Figure 5.14: The curve of Force versus Displacement

5.5 Fracture Analysis

After the impact test, the mostly fractured specimens were taken to Queensland University of Technology (QUT) for investigation of the fractured surface using Scanning Electron Microscopy (SEM). These two mostly fractured specimens chosen were:

- VE/FLYASH (33%) cured under microwave condition with a power level of 180 Watts and exposure time of 40 seconds. The height of impact was 500mm.

- VE/FLYASH (33%) cured under microwave condition with a power level 540 Watts and exposure time of 10 seconds. The height of impact was 450mm.

The reason for choosing these two specimens was to compare the difference in fractured surfaces.

5.5.1 Scanning Electron Microscope (SEM)

The Scanning Electron Microscope (SEM) is a microscope that uses electrons rather than light to form an image. There are many advantages in using the SEM instead of a light microscope.

The SEM has a large depth of field, which allows a large amount of the sample to be in focus at one time. The SEM also produces images of high resolution, which means that closely spaced features can be examined at a high magnification. SEM requires conductive samples and Figure 5.15 shows the Scanning Electron Microscopy (SEM).

Figure 5.15: Scanning Electron Microscopy (SEM)

The combination of higher magnification, larger depth of focus, greater resolution, and ease of sample observation makes the SEM one of the most heavily used instruments in research areas today.

5.5.2 Mounting the specimen

Any specimen, whether it has been sputter coating or is naturally conductive, must be firmly attached to the specimen support before being viewed in the SEM. Attention to detail in the mounting procedure is very important if a researcher desires a quality result.

Make sure the support (specimen stub) is clean before use and also check to make sure the stub you are using is compatible with the stage of the SEM you will be using. Place the specimen on the stub before the sputter coating procedure. This will increase the conductivity and therefore the quality of your results. Figure 5.16 shows the sputter coaster used in this research.

Figure 5.16: Sputter Coater

For some samples, it will be necessary and perhaps more convenient to place the specimen on a substrate that will be mounted on the stub. Substrate materials range from glass and plastic cover slips to metal and crystalline disks, plastics, waxes and many membrane filters. Since it is frequently necessary to attach the specimen or substrate to the stub, a variety of adhesives is available for this purpose. You should make sure to choose an adhesive that will not decrease the quality of your results.

5.5.3 Sputter coater

The sputter coater is used to coat non-metallic samples with a thin layer of gold and it is shown in the Figure 5.17. The surface will look like Figure 5.18 after coating a thin layer of gold. This makes them conductive, and ready to be viewed under the SEM. If the samples are metallic, they can simply be mounted and placed in the SEM.

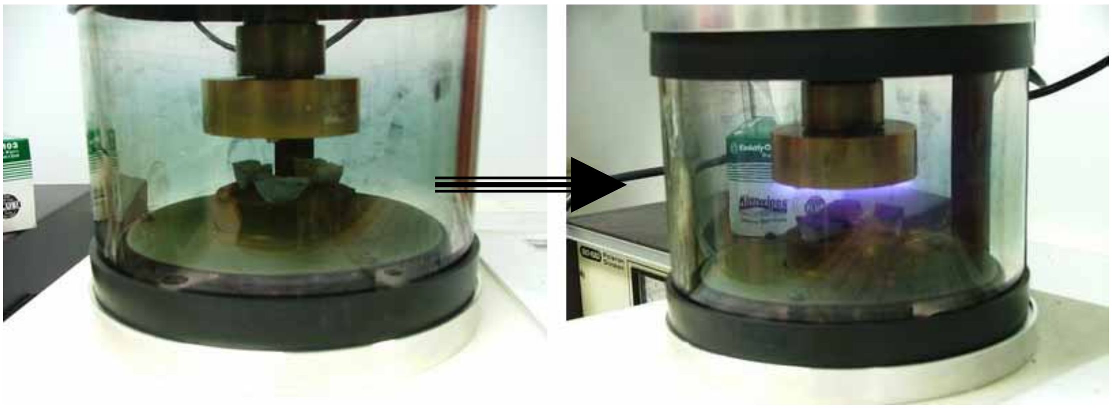


Figure 5.17: The sputter coaster coating a thin layer of gold on the specimen's surface

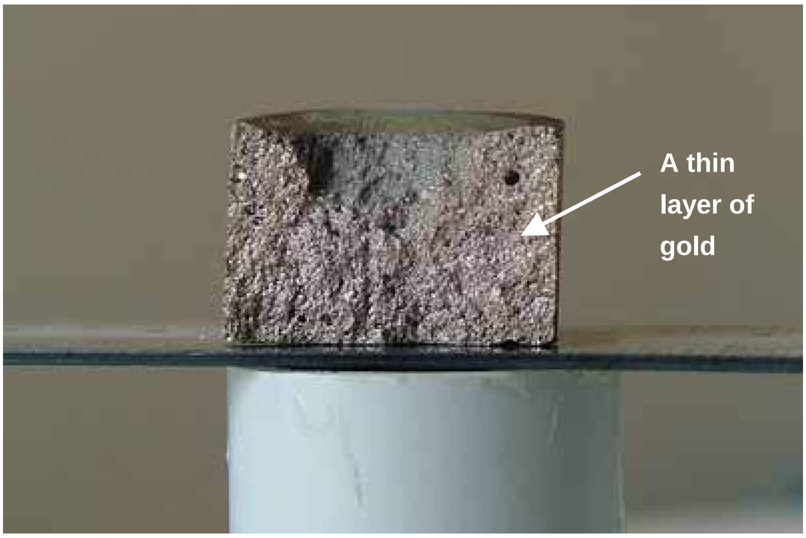


Figure 5.18: The specimens after coating a thin layer of gold look like

Make a mark on the bottom of the specimen with a maker pen like that shown in Figure 5.19; it is for convenience of searching the specimen in a large group of samples, especially after scanning.

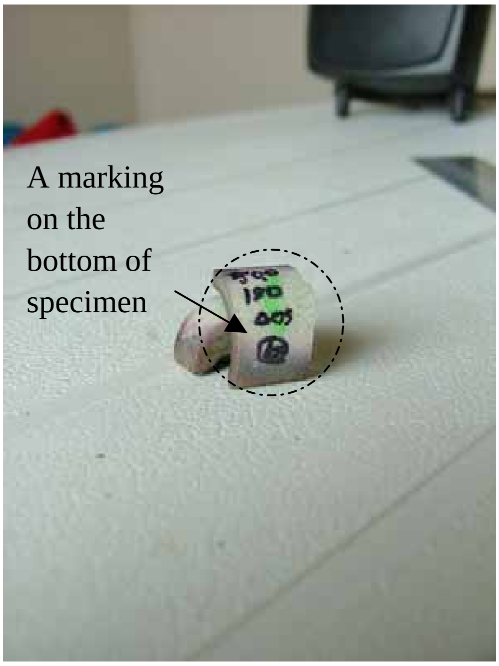


Figure 5.19: A marking on the bottom of specimen

5.5.4 The Setup of SEM

When the electron beam strikes the sample, both proton and electron signals are emitted. Figure 5.20 shows the electron and specimens interaction. While all these signals are present in the SEM, not all of them are detected and used for information. The signals most commonly used are the Secondary Electrons, the Backscattered Electrons and X-rays. Figure 5.21 shows the lens and detectors located inside the sample chamber.

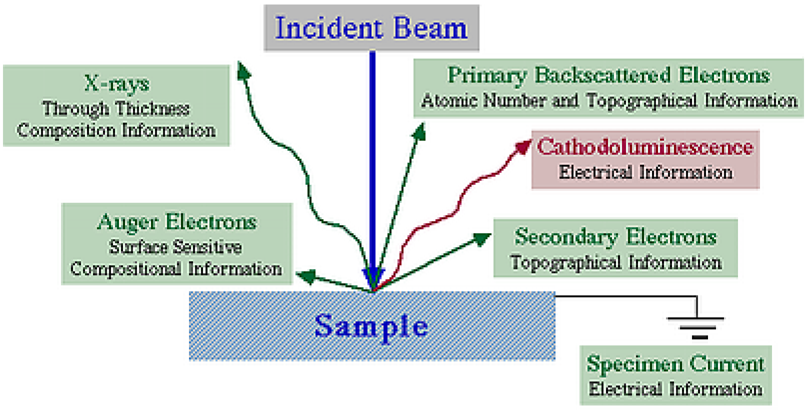


Figure 5.20: Electron and Specimens Interaction

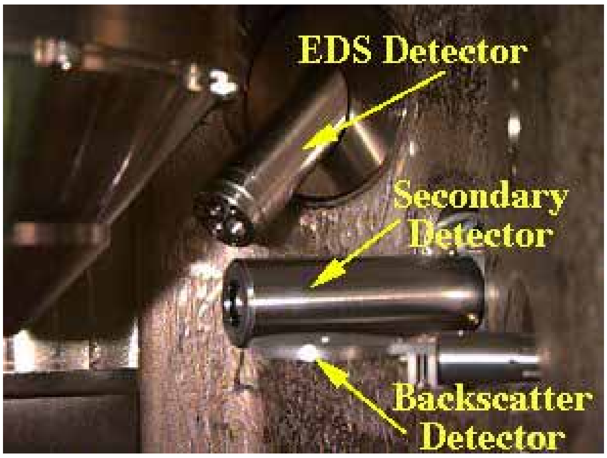


Figure 5.21: The lens and detectors located inside the sample chamber

5.5.5 Methods of using SEM

When a SEM is used, the column must always be at a vacuum. There are many reasons for this. If the sample is in a gas filled environment, an electron beam cannot be generated or maintained because of a high instability in the beam. Gases could react with the electron source, causing it to burn out, or cause electrons in the beam to ionize, which produces random discharges and leads to instability in the beam.

The transmission of the beam through the electron optic column would also be hindered by the presence of other molecules. Those other molecules, which could come from the sample or the microscope itself, could form compounds and condense on the sample. This would lower the contrast and obscure detail in the image. A vacuum environment is also necessary in part of the sample preparation. One such example is the sputter coater. If the chamber is not at vacuum before the sample is coated, gas molecules would get in the way of the argon and gold. This could lead to uneven coating, or no coating at all. Figure 5.22 shows a prepared sample mounted on a specimen stub and placed on the stage.

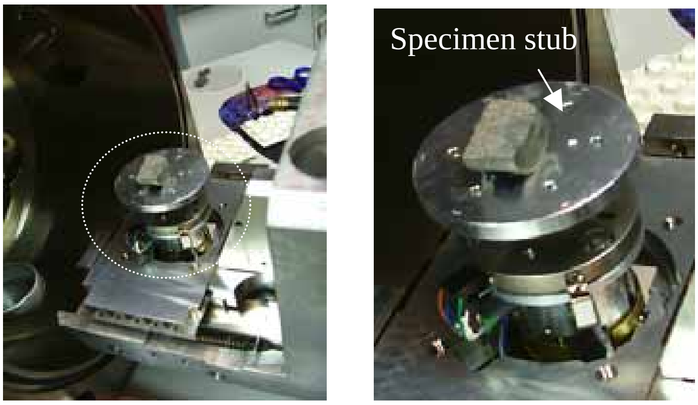


Figure 5.22: Specimen stub

In each of the specimen’s investigation, 5 points had been chosen for investigation. Figure 5.23 shows the 5 points chosen to be investigated on specimens cured under microwave conditions with a power level 180 Watts, 40 seconds exposure time and height of impact of 500mm.

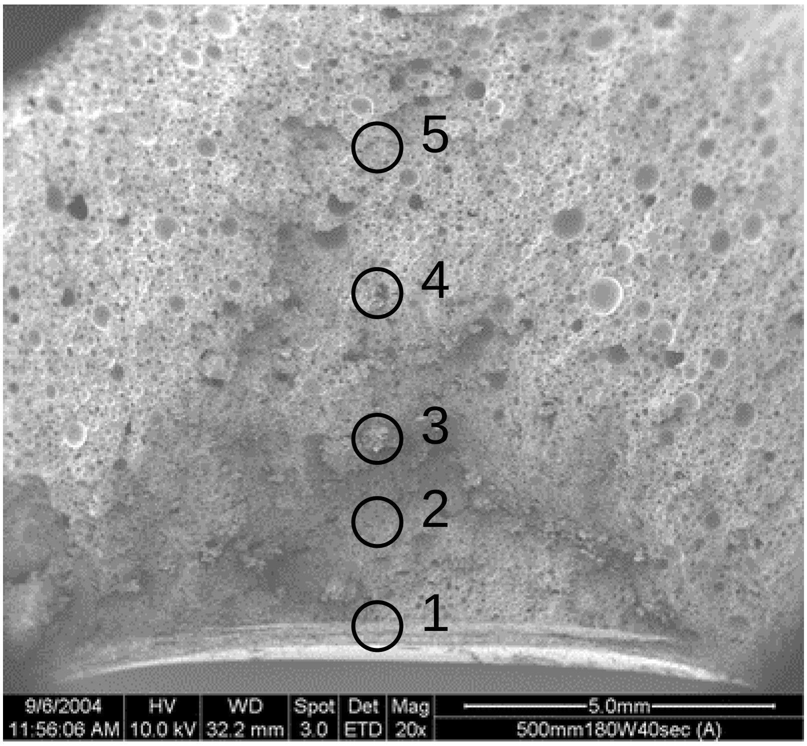


Figure 5.23: Microwave condition with 180W and 40 sec expose time

Figure 5.24 shows the 5 points chosen to be investigated on specimens cured under microwave condition with power level 540 Watts, 10 seconds exposure time and height of impact of 450mm. The reason for choosing these 5 points was because these points showed the crushed zone and fracture propagation zone of these two specimens.

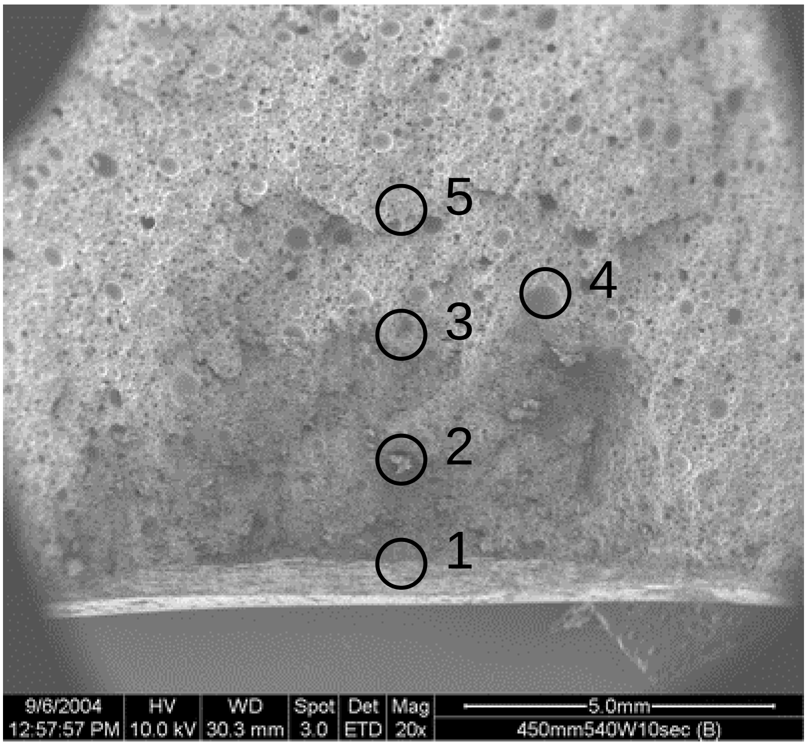


Figure 5.24: Microwave condition with 540W and 10 sec expose time

The magnifications used in this investigation were 80X and 300X. At some point the magnifications will be increased to 600X, 1200X and 5000X respectively; this is used to investigate some small fracture surface.

Chapter 6

Results and Discussion

6.1 Introduction

The results obtained from the test undertaken in this investigation will be discussed here. Comparison of results will be made after the discussion on the results from the experimental work. The comparisons of results and discussions include comparison of specimens cured under microwave conditions with differences power levels and exposure times.

6.2 Initial Test

These initial tests were conducted to obtain energy absorption data and investigate the failure height of the cylindrical specimens. The impact energy required to fracture these specimens are attached as Appendix C.

6.2.1 Power Level of 180 Watts

Exposure time of 30 seconds

To determine the height at which failure would begin to occur, a similar method of testing to the staircase method was used. The first specimen was initially impacted from a height 250mm and this specimen was cured under microwave with a power level of 180 Watts and exposed to microwaves for 30 seconds. When failure did not occur, the tup was raised another 50mm and it was impacted again. This testing also provides information on damage tolerance and variations. The first specimens was found that most of the specimens non-fractured at a drop height of 350mm or below. The specimens failed while the impact tup raised another 50mm to 400mm and that 72.8% of the total energy was used to initiate the crack and the remaining 27.2% was used to propagate the crack.

Expose time of 35 seconds

The second specimens were tested in the same manner and these specimens were cured under microwave with an exposure time of 35 seconds. It was found that the drop height used to fracture most of these specimens was 450mm. For each impact that did not result in failure a certain amount of the energy was released back into the specimen to cause the bounce. Observable damage on the impact surface was visible after 450mm impact test height. The damage consisted of an indentation into the material. The indentation at this stage was not dramatic but it became more severe after subsequent impacts. The force versus displacement curves show evidence of plastic deformation in that the unloading path does not coincide with the loading path and they do not start and finish at the same point. However, observations found that no cracks were visible until complete failure had occurred. Further investigation into the indentation is required to determine the extent of the effect it had on the failure energy.

Expose time of 40 seconds

The next impact test was for specimens cured under microwaves with an exposure time of 40 seconds. At this initial height (300 mm) the specimen did not fail, so the height was increased to 400mm. The force versus displacement curves for these tests can be seen in Figure 6.1 and it showed a failure specimen during the test.

The drop-height was again increased and specimen went on to fail at a drop-height 400mm. This energy was found to vary 73.2% to 86.1% of the energy absorbed by the specimen.

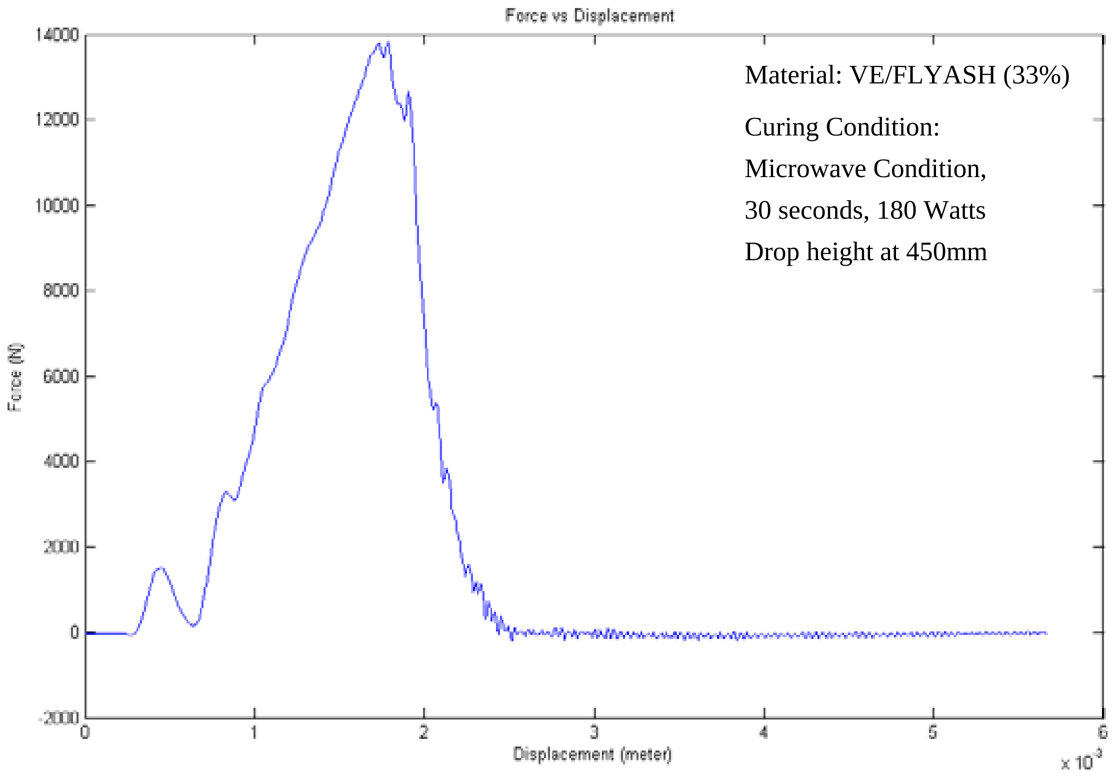


Figure 6.1: Plot showing the force versus displacement tested at a drop-height of 500mm

Table 6.1 shows the average energy on the specimens cured with a power level of 180 Watts tended to fracture at a drop height of 400mm.

Table 6.1: Average energy required to fracture specimens cured with a power level 180 Watts

Curing Condition	Power Level of 180 Watts		
Exposure time	30 seconds	35 seconds	40 seconds
Energy used to initiate the crack	8.36 joules	10.87 joules	9.42 joules
Energy used to propagate the crack	2.68 joules	-1.46 joules	1.82 joules
Total Energy Dissipated	11.04 joules	10.87 joules	11.24 joules
Displacement at Peak Force (m = meter)	0.0017 m	0.0019 m	0.0018 m

The drop-height was again increased and specimens went on to fail at a drop-height of 500mm. The force-displacement curves for these tests can be found in Figure 6.2. Figure 6.2 it showed the comparison of three specimens of different exposure times.

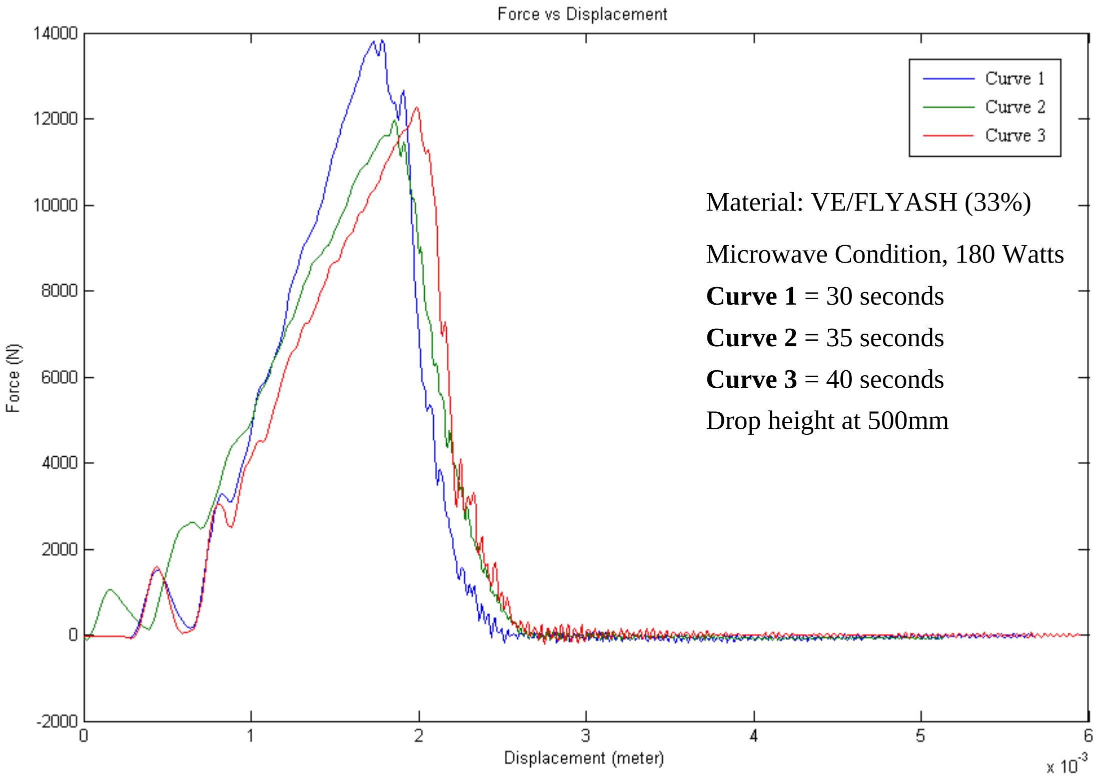


Figure 6.2: Resulting force-displacement curve of tested at a drop-height of 500mm

Due to the good failure surface for specimen exposed to microwaves for 40 seconds, the exact drop height to fracture of these specimens can be a source of investigation.

6.2.2 Power Level of 360 Watts

Expose time of 30 seconds

The fourth impact testing was the specimens cured under microwave with power level 360 Watts exposed to 30 seconds. It was found that most of the specimens did not fracture at a drop height of 350mm or below. The specimens failed while the impact tup was raised to 400mm and that 90.17% of the total energy was used to initiate the crack and the remaining 9.83% was used to propagate the crack.

Expose time of 35 seconds

The fifth impact testing was for specimens cured under microwaves with an exposure time of 35 seconds. It was found that the drop height used to fracture these specimens was 350mm. The investigation found that up to 80.03% of the energy dissipated was required to initiate the crack. The remaining energy was used in propagating the crack though the specimen.

Expose time of 40 seconds

The sixth impact testing was for specimens cured under microwaves with an exposure time of 40 seconds. It was found that the drop height used to fracture some of these specimens was 350mm. A total of 88.73% of the average total energy was found to contribute to initiate the crack and remaining 11.27% was found used to propagate the crack and displacement when peak force was 0.0017 meter. Some of the non-failed specimens were found to have a drop height below 350mm.

Table 6.2 shows the average energy of specimens cured with microwaves with a power level of 360 Watts; their fracture happened at a drop height of 400mm.

Table 6.2: Average energy on specimens cured with power level 360 Watts

Curing Condition	Power Level of 360 Watts		
Exposure time	30 seconds	35 seconds	40 seconds
Energy used to initiate the crack	9.45 joules	9.02 joules	8.50 joules
Energy used to propagate the crack	1.03 joules	2.12 joules	2.53 joules
Total Energy Dissipated	10.48 joules	11.14 joules	11.03 joules
Displacement at Peak Force (m = meter)	0.0016 m	0.0018 m	0.0018 m

The drop-height was again increased and specimens went on to fail at a drop-height of 500mm. The force-displacement curves for these tests can be seen in Figure 6.3. It was shown that curve 2 was had the highest force for fracture.

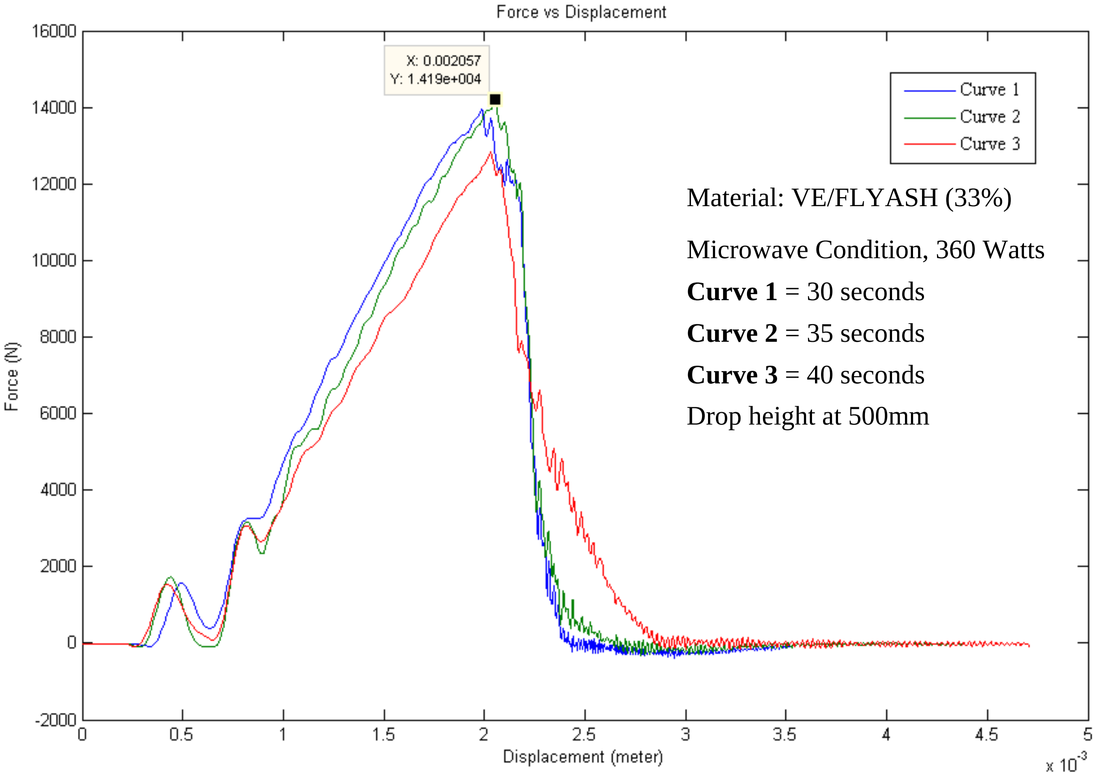


Figure 6.3: Resulting force-displacement curve of tested at a drop-height of 500mm

The specimens that were exposed to microwaves for 35 seconds and 40 seconds were found to fail and not to fail at the same drop height of 350mm. It was possible the impact tup was not running smooth during dropping.

6.2.3 Power Level of 540 Watts

Expose time of 10 seconds

The first impact test on these specimens was performed at a drop-height of 250mm. Non-failure occurred on the first impact and the resulting force versus displacement curve was shown in Figure 6.4. It was found that the specimens failed while the impact tup was raised to 400mm; 81.8% of the total energy was used to initiate the crack and the remaining 18.2% was used to propagate the crack.

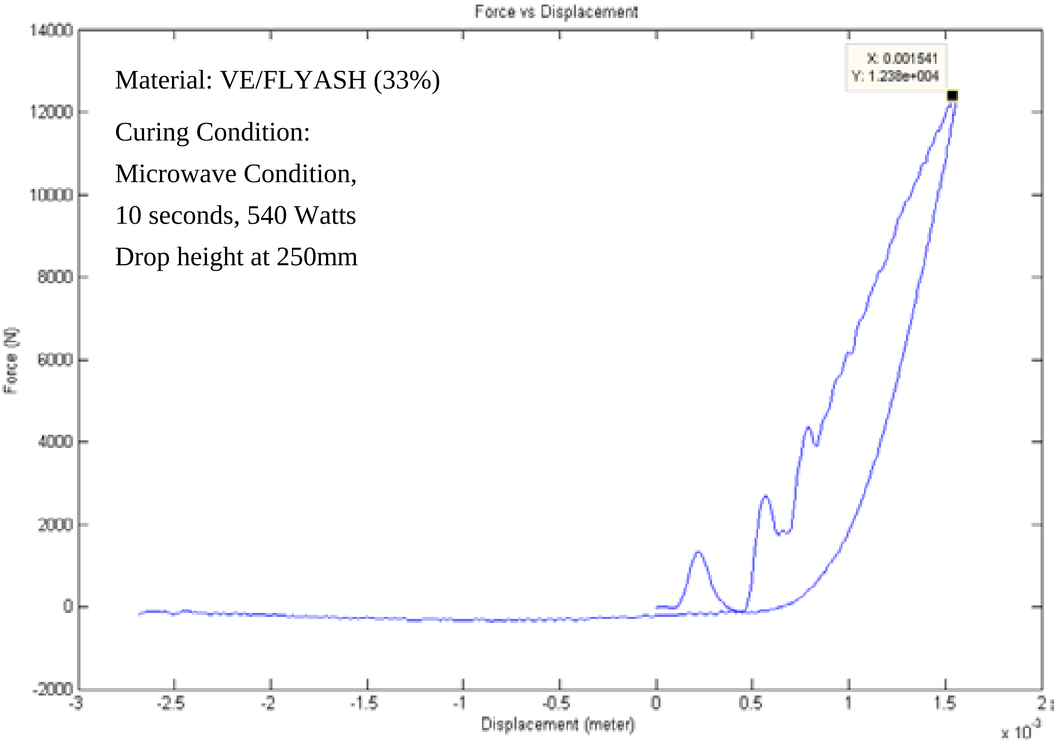


Figure 6.4: Force-displacement curve of a specimen tested at a drop-height of 250mm

Expose time of 15 seconds

The next impact testing was for specimens cured under microwaves with exposure time of 15 seconds. The results proved that there were no fractured specimens in the group. Non-failure occurred on the fourth impact (drop height 400 mm) and the resulting force versus displacement curve was shown in Figure 6.5.

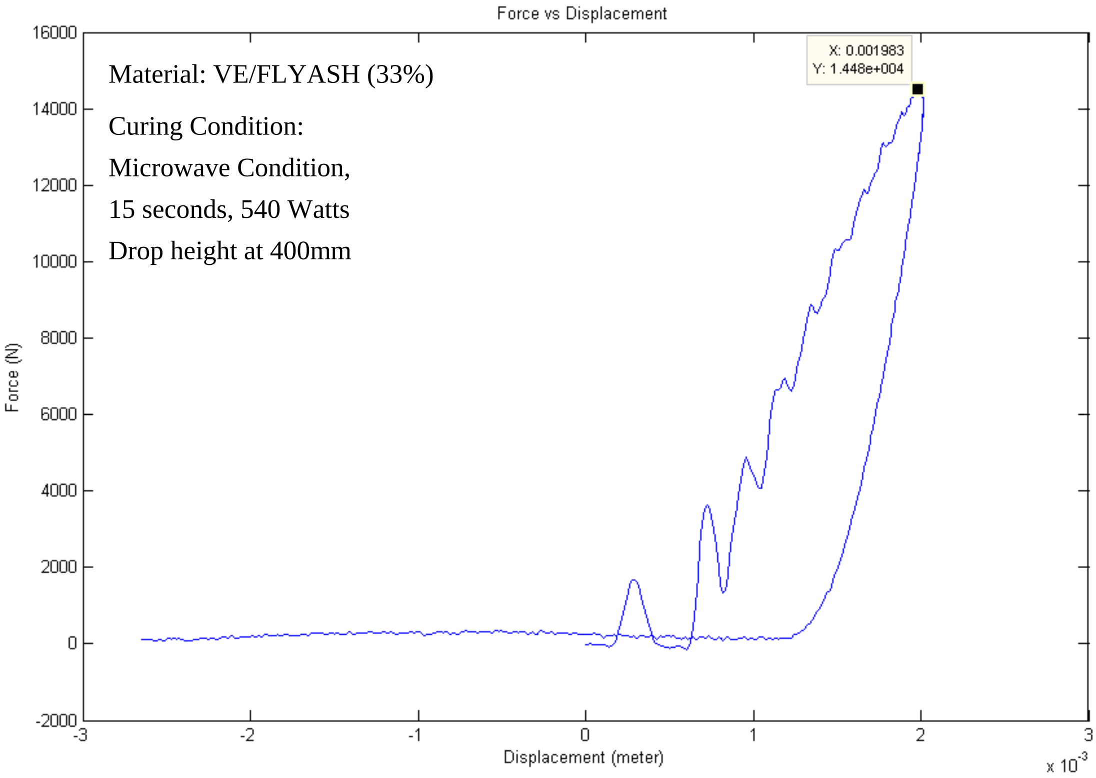


Figure 6.5: The Force vs. Displacement curve for non-failure specimen (drop height of 400mm)

This is means that an impact from a height of 400mm will not guarantee a failure. In this circumstance the total amount of energy released on the specimen is 100% and 0% of this amount was released back into the tup producing the bounce phenomena.

20 seconds of Expose Time

The last impact testing was the specimens cured under microwave with exposed to 20 seconds. It was found that the specimens were over curing under microwave and not enough harder if compared with previous condition. These specimens failed while the drop height was increasing to 450mm and it was show in figure 6.6. A total of 87.9% of the average total energy was found to contribute to initiate the crack and remaining 12.1% was found used to propagate the crack.

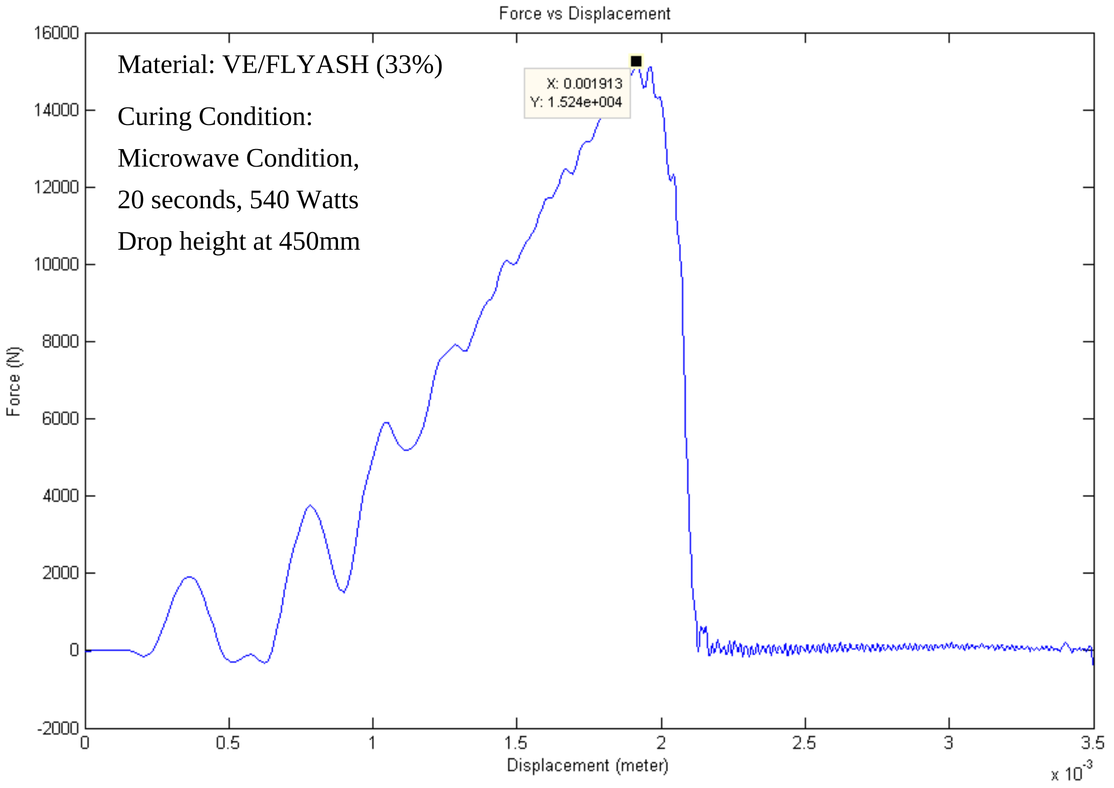


Figure 6.6: The Force vs. Displacement curve for failure from a height of 450mm

Table 6.3 shows the average energy of specimens cured with microwaves with a power level of 540 Watts to fracture at a drop height of 450mm.

Table 6.3: Average energy on specimens cured with power level 540 Watts

Curing Condition	Power Level of 540 Watts		
Exposure time	10 seconds	15 seconds	20 seconds
Energy used to initiate the crack	8.27 joules	7.91 joules	3.71 joules
Energy used to propagate the crack	1.83 joules	-2.77 joules	0.507 joules
Total Energy Dissipated	10.1 joules	7.91 joules	4.217 joules
Displacement when Peak Force (m = meter)	0.0018 m	0.0017 m	0.0015 m

It was found that most of the specimens were not fractured at a drop height of 400mm or below. These specimens failed while the impact tup was raised another 50mm to 450mm. The force-displacement curves for these tests can be seen in Figure 6.7.

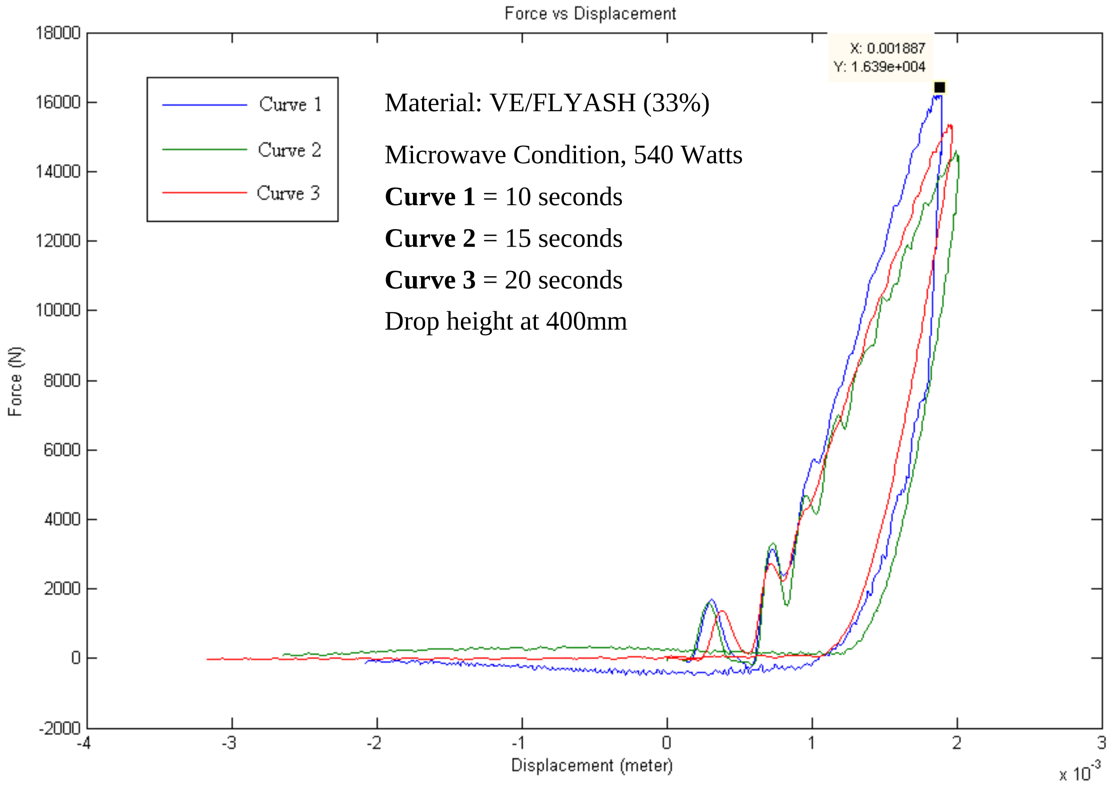


Figure 6.7: Force-displacement curves of tested specimens at a drop-height of 400mm

6.3 Comparisons

The strength of the polymeric material, the main constituent of the tested material, is dependent on the rate at which the load is applied (strain rate). One indication of this is that the energy released back into the tup was decreased as the drop-height was increased from 250mm to 500mm. Overall, the data gained from this experimentation had not shown a significant effect of strain rate. A greater variation in strain rates may be required before observable change was observed.

There appear to be two different slopes in regard to specimen failures. Each of the slopes showed a certain degree of linearity. From Figure 6.8, the fifth slope (approx.17, 000 kNm) occurs in specimens that underwent a previous impact and it failed. The fourth slope (approx.16, 000 kNm) was exhibited in the specimens that no failure on all impact. This is the strongest evidence to show accumulative damage of the material. The slope of this curve is thought to be affected by the stiffness of the specimen.

The depth of penetration when failure was complete and approximated to be less than 2mm. In the repeated impact specimens (From Data 1 and Data 2) it appeared slight less than the fifth-impact failure specimens (From Data 5). This is also an indication that accumulative damage may exist with this material.

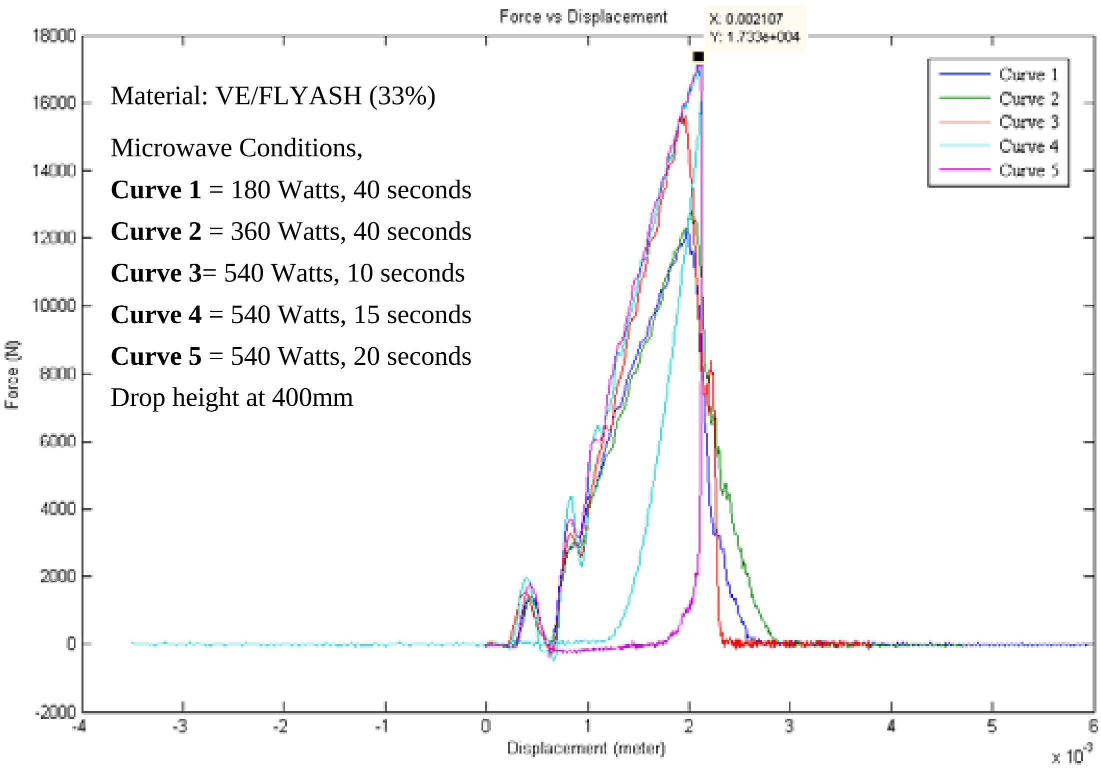


Figure 6.8: A similarity in the slope of the loading curves had been exhibited

The peak force on all failure curves ranged from 12 kN to 17 kN. No significant relationship between peak force and drop-height could be attained with the small number of specimens tested. From these results one can say that a peak force in this range is likely to result in failure.

However when observing Figure 6.8 it can be seen that the irregularity does not diminish with subsequent impacts. To determine the cause of the feature of the loading curve, the use of more specialized equipment is required (e.g. high speed camera)

After the impact test, the best fracture specimens were taken to Queensland University of Technology (QUT) for investigation for fractured surface using the Scanning Electron Microscopy (SEM). One of the specimens was cured with an exposure time of 40 seconds and a power level of 180 Watts and the other one was cured under a power level 540 Watts and exposed to microwaves for 10 seconds.

The reason of the choice was to compare the difference fractured surface cured under different power levels and exposure times. Tables 6.4 showed the results of the investigation into these two specimens using magnifications of 80 and 300 times respectively. The area named 1, 2, 3, 4 and 5 for investigation had been discussed in section §5.5. At some point, the magnifications were increased to 600X, 1200X and 5000X respectively; this was used to investigate some small fractured surface. The pictures taken at these five areas with magnifications of 80 and 300 times can be in Appendix E.

Table 6.4: Viewing results of magnifications of 80 times to 300 times

Investigation Fracture Surface of Five Areas		
Area	Microwave Condition	
	Specimens A1 (180Watts;40seconds;500mm)	Specimen A2 (540Watts;10seconds;450mm)
1	A big crushed on the top surface (curving area) of the specimens was shown in Figure 6.9.	Most of the phenomenon was same with specimen A1 and nothing special was found in this area 1. .
2	Figure 6.10 showed the fracture surface started in this area 2. This area 2 showed a curving fractured surface. A small empty hole was found when the magnification increase to 600X.	Same case with specimen A1 but the fracture surface has a little bit longer.
3	50% of crushed zone was found and 50% of fractured propagation zone was found. Besides that, a secondary cracking was found. The force was pulled to different directions from area 3.	70% of crushed zone was found and 30% of fractured propagation zone was found. Some small pieces of debris were found on the cracked surface when the zooming was increased to 2500X.
4	Another secondary cracking was in this area 4. 90% of fractured propagation zone was found and 10% of crushed zone was found.	Less debris appeared as compared with specimen A1. Brittle fracture was also found in this area. Crack was also found propagating through fly ash particle.
5	Figure 6.9 showed some small pieces of debris were on the crack surface. A secondary crack was found in 600X zooming and a direction of crack growth to left hand side.	Figure 6.11 showed much of brittle fracture area appeared in the crack propagation zone. There was a lot of small bubble in fractured propagation zone.

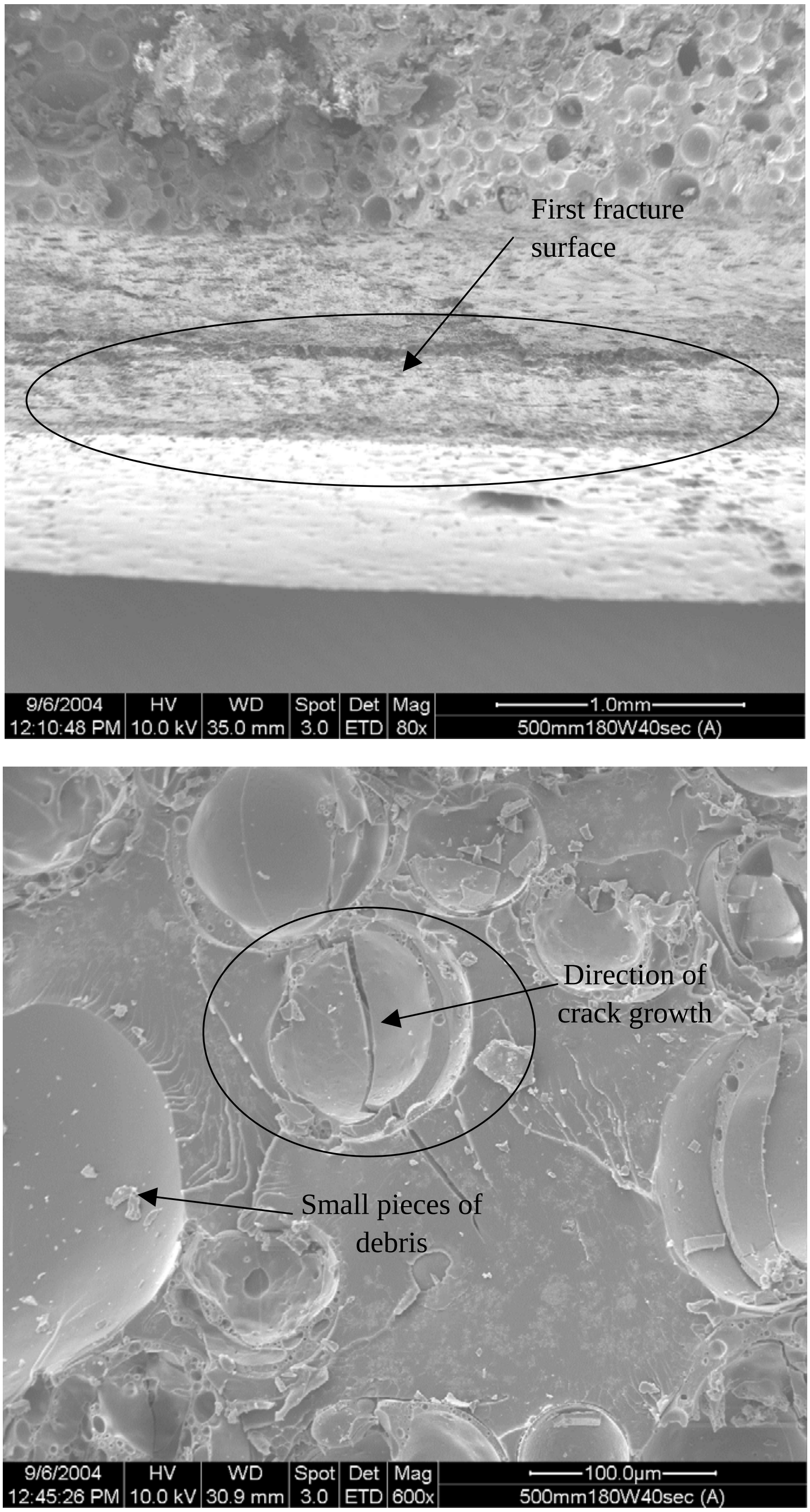


Figure 6.9: Different zooming fracture surface in between 80X to 600X

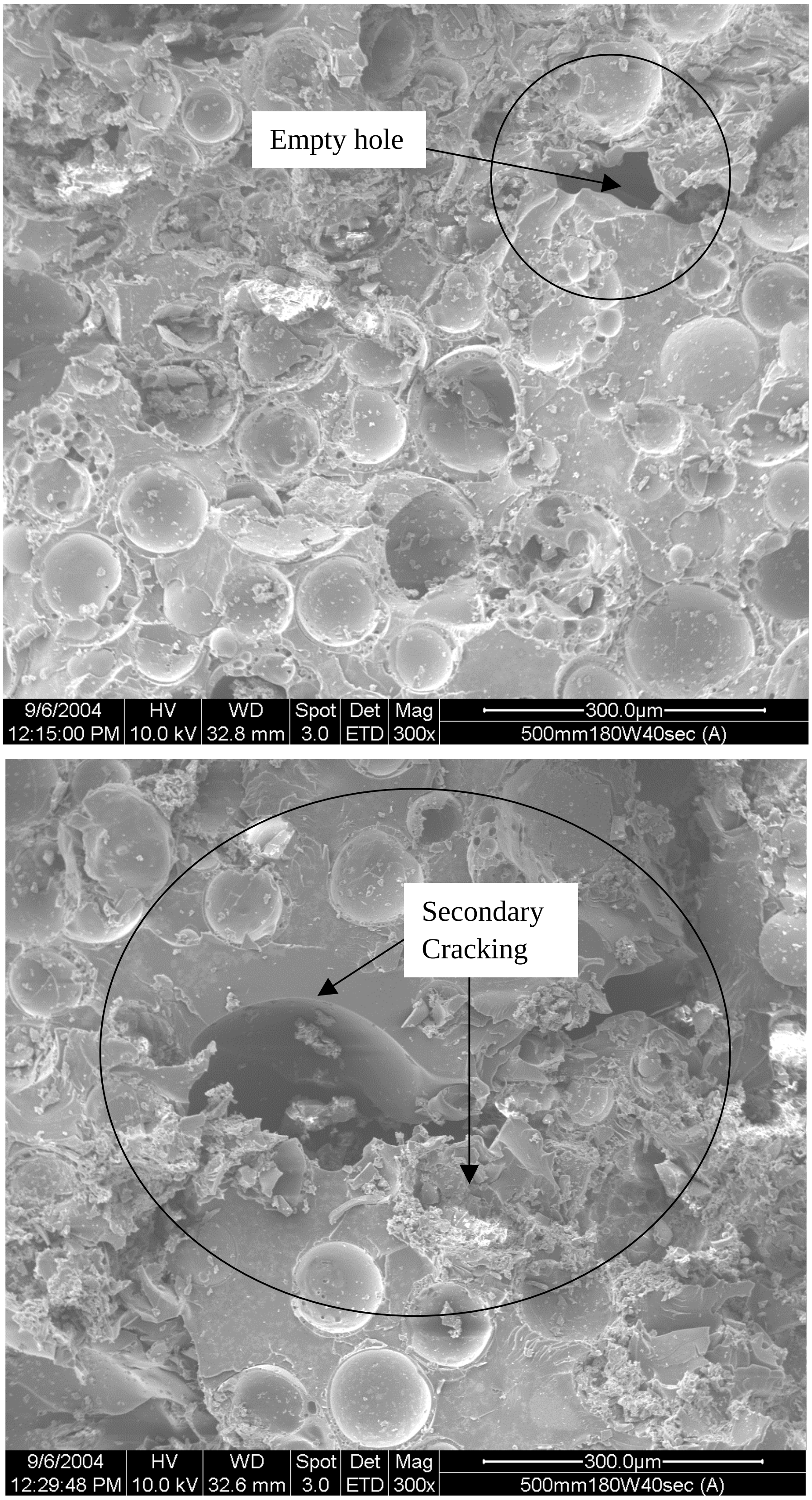


Figure 6.10: Fracture surface in 300X zoom

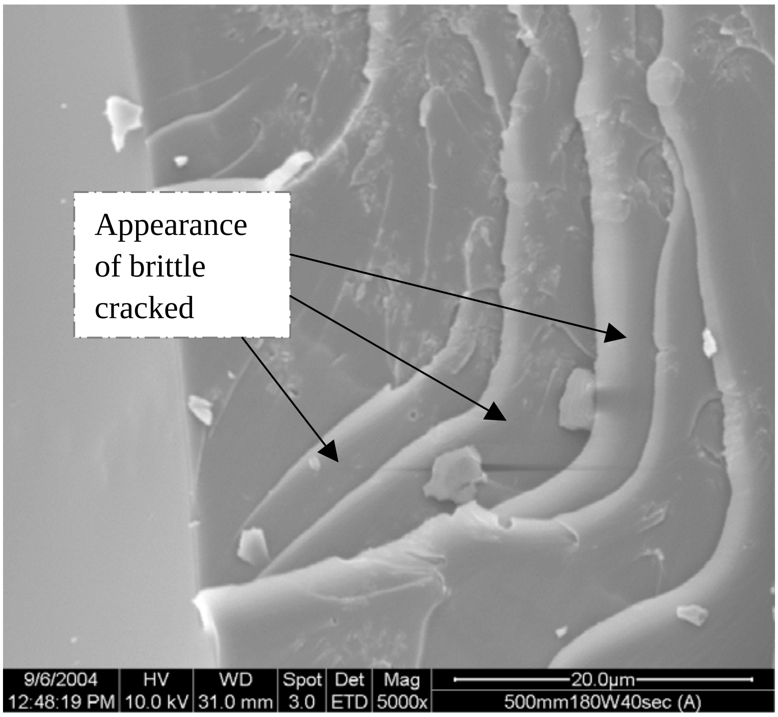


Figure 6.11: Appearance of brittle cracked

In general, the results obtained for the groups of specimens cured under microwave conditions with a power level of 180 Watts showed not much difference with the one cured under microwave conditions with a power level of 540 Watts. The differences in average energy required to complete fracture between these two specimens were found to be very small.

6.4 Summary

Important points from this chapter are:-

1. The results proved that there were no fractured specimens in the group of 540 Watts and 15seconds.

2. The specimens cured with microwaves using a power level of 540 Watts and 20 seconds tended to fracture at a drop-height of 450mm.
3. Peak forces of the failure curves ranged between 12kN to 17kN.
4. The impact resistance of VE/FLYASH greatly depended on the amounts of fly ash used.
5. An irregularity at the beginning of the loading curve was found to be common to all specimens. The displacement range over which the irregularity existed was 1.3mm to 1.8mm.

Chapter 7

Conclusions and Further Work

7.1 Introduction

These research findings can be used as a foundation for those who continue this project. The achievements made over the course of this investigation will be summarized in this chapter. The problems solving and recommendations will also be provided to aid the future workers.

7.2 Achievements

The achievements made over the course of this investigation are summarized in the following subsections:

7.2.1 Specimens Production

The specimens had been successfully produced under microwave conditions. In the group of curing under microwave conditions, it was broken down into two more groups by having the specimens cured under microwave conditions with power levels of 180 Watts and 360 Watts, each with different exposure time of 30 seconds, 35 seconds and 40 seconds respectively. Besides that, 540 Watts with exposure time of 10 seconds, 15 seconds and 20 seconds were the extra group cured under microwave conditions this research.

7.2.2 Data Processing

Matlab software makes the data processing more convenient. Once the noise made the data obtained difficult to analyze. The Savitzky-Golay filter used the Signal Processing Toolbox in MATLAB made the graph looked smooth, more accurate; It also captured the heights and widths of narrow peaks.

7.2.3 Impact Strength

The impact strength on specimens cured with microwaves had been found and evaluated. From the results, the specimen cured under microwave with a power level of 540 Watts and 15 seconds of exposure time was the strongest specimen. The best result was to drop the tub from a height of 500mm.

7.2.4 Fracture Analysis

Fracture analysis had been carried out with the use the Scanning Electron Microscopy (SEM), the analysis had shown that there was a difference between the specimens cured under microwave conditions with power levels of 180 Watts and 540 Watts. In 180 Watts group, the fracture surface of the specimen is bigger when compared with that of the 540 Watts one. This was because the group cured by a power level of 540 W was stronger.

7.3 Further Work

7.3.1 Mould Casting

The specimens cannot be cast at 100% identical. Some of the specimens cast were found to have uneven height and surface flatness as well. This problem arose when pouring the mixtures into the moulds. This problem will affect the final result of the impact test. It was suggested the cast the specimens as 55mm height and cut it around 5mm height after curing. It was recommended to cut them with diamond saw to produce a flat and same height on every specimen

7.3.2 Drop-weight Tower

The drop-weight tower was used in this research had a few problems. The problem was the reading had to be increased by a step of 50mm. The impact tup was not running smooth during dropping. Some vibration occurred in the drop-weight tower because the support it was not stable enough. It was suggested to modify or change a new drop-weight tower because this could improve results.

7.3.3 Matlab Programming

The MATLAB programming used was still in the developing stage. There were some inconvenient in running the program such as the data could not be loaded directly from MATLAB program. The program had to be restored every single time in every testing.

7.3.4 Simulation

Nowadays, ANSYS software is popular in the market and user-friendly for simulation. ANSYS can be incorporated with other software such as PRO-ENGINEER and so on. More information can be obtained if computer software is used to analyze the experimental results, such as the area of maximum fracture toughness etc. By using this software some process will be simplified. Hence, I suggest this software has to be used on next attempt.

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