

University of Southern Queensland
Faculty of Engineering and Surveying

Fracture Analysis of Vinyl Ester Composites Cured under Microwave Conditions

A dissertation submitted by

CHAN, Wooi Lieh

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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 vinyl ester 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%). The purpose of this project is to research, measure and compare the fracture toughness of vinyl ester composite cured under ambient and microwave conditions by using the short bar test.

The specimens will then fractured by using the MTS 810 Material Testing Systems and the value of fracture toughness was obtained through some calculations.

And finally the results will analyzed by using Latin Square, determine which treatments were most effective in maintaining the fracture toughness while reducing the shrinkage of vinyl ester composite, and by how much, and which are worthless, so we can weight the economic alternatives.

Declaration

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<p>ENG4111 Research Project Part 1 & ENG4112 Research Project Part 2</p>

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CHAN, Wooi Lieh

Student Number: 0050027434

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Chapter 1

Introduction

1.1 Project aim

The aim of this project was about the analysis of fracture toughness on particulate reinforced 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 33 % VE/FLYASH under ambient conditions and microwave conditions. These specimens are to investigate the fracture toughness of 33% VE/FLYASH cured under ambient condition and microwave conditions. During the curing process the vinyl ester suffers will occur shrinkage. Therefore microwave energy in multimode oven cavity is to be applied to samples the vinyl ester resins under controlled conditions to minimize its shrinkage.

1.2 Dissertation Overview

Here is the brief overview on the material presented on each chapter of the dissertation.

Chapter Two

Discuss 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

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

Explain the fracture mechanics which defined as a field of solids mechanics that deal with the behaviour of cracked bodies subjected to stresses and strains. The aims of the fracture mechanics are to determine the severity of a pre-existing defect in term of its tendency to initiate a fracture, which would cause failure.

Chapter Five

Introduce K_{IC} (fracture toughness) which define as material of a sharp crack that has the characteristics of its resistance to fracture under tensile conditions. It is extremely important property in many crucial design applications.

Chapter Six

In-depth understanding of short bar test and the geometry of specimens. Compare the disadvantages and advantages of both standard and non-standard testing.

Chapter Seven

Describe the preparation 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 Eight

Introduce the MTS 810 machine and the system which are use in the tensile testing of this project.

Chapter Nine

The results obtained from the experimental work will be present in this chapter. The chapter will be divided into sections; (1) related to e tensile testing results from the experimental work, and (2) fracture toughness determination and discussions.

Conclusion

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.

Chapter 2

Composite Material

2.1 Introduction

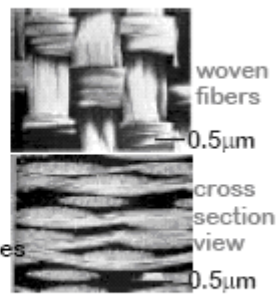
. In this project, particles will be dispersed in a polymer matrix. The background and description of polymer matrix composites (PMCs) as well as their classification will be introduced later. In the case of thermosets, the reader will be introduced to epoxies, polyesters and vinyl ester.

A composite is a complex material, in which two or more distinct substances, combine to produce functional and improved properties not present in any of the individual component. This project relates to a polymer-based composite, which is formed from the combination of fiber and resin, where the fibers are oriented to carry the loads. Because of these aligned fibers that are meant to carry the loads and their adaptive nature enabling the fibers to align in the direction to carry the load, these composites can be designed to the minimum weight without sacrificing the strength. Thus, generally, composites have stiffer, lighter and higher strength than the other usual materials. In the case of this project, particles will be dispersed in a polymer matrix. Introduction to the background, description and the classification of the polymer matrix composites (PMCs) will be done later as well as the introduction of epoxies, polyesters and vinyl ester in the case of thermosets.

2.2 Types of Composite Materials

Either thermoset or thermoplastic, composites as a polymer matrix provides a discernable reinforcing function in one or more directions when reinforced with a fiber or other material with adequate aspect ratio (length to thickness). Bear in mind that not all plastics are composites and that the majority of plastic materials available today are pure plastics. This is in cases where products such as household goods, toys and decorative products do not require very high strength for their functions and thus plastic resin would be sufficient to provide the strength needed. Whereas for those that are required to perform better, such as at increased heat distortion temperatures, “engineering-grade” thermoplastics are of a better quality than the normal plastic resins, but comes more costly. So, in fact, many types of plastics can be reinforced with structural materials when additional strength is needed to fulfill the requirements of higher performance. In most cases, reinforcing fibers are used. All these thermoplastic or thermoset plastic resins that is reinforced with other material is then considered 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

Several types of composites are available especially for industrial use and most of these composites are based on polymeric matrices, thermosets and thermoplastics. Usually, aligned ceramic fibers, for example glass or carbon, are used to reinforce the composites. Not long ago, interest in metal matrix composites (MMCs), such as aluminium reinforced with ceramic particles or short fibers, and titanium containing long, large-diameter fibers, has emerged. 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. The research and development area do come up with various industrial applications for MMCs but in comparison to that of polymer composites (PMCs), MMCs are still quite limited in terms of commercial usage. As for ceramic material composites (CMCs), this is also in the research stage. Figure 2.2 illustrates the properties of fiber composites.

Fiber Composite Properties

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

Elastic modulus in fiber direction:

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

\uparrow
 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

Since the strength and stiffness of the material are unlikely to be much affected, the main purpose is generally to bring about toughness to the matrix by the introduction of other components. But due to the difficulty in manufacturing the materials, most of these are still in the early stages of development. The popularity of composites among engineers, designers and manufacturers, due to their unique combinations of

performance benefits have caused the composites to arise, making jobs easier with increased effectiveness for those associated in turning design concepts into product realities.

It is crucial to look into the properties shown by the potential constituents when it comes to considering formulating a composite material for particular applications. The properties that are of particular interest are the stiffness (Young's modulus), toughness and strength of the material as well as density which is of great importance. Apart from that, thermal properties, such as expansivity and conductivity, must also be taken into account. Note that, because composite materials are subject to temperature changes during manufacture and/or in service, an internal residual stress would happen when there is a mismatch between the thermal expansivities of the constituents and thus leading to 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. Observing these data, it shows that some interesting property combinations (e.g. high stiffness/strength and low density) can be obtained through 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 K_{Ic} ($\text{MPa m}^{1/2}$)	Thermal conductivity K ($\text{W m}^{-1} \text{K}^{-1}$)	Thermal expansivity α (10^{-6}K^{-1})
Thermosetting resin (epoxy)	1.25	3.5	50	0.5	0.3	60
Engineering thermosetting (nylon)	1.1	2.5	80	4	0.2	80
Rubber (polyurethane)	1.2	0.01	20	0.1	0.2	200
Construction 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, there will usually be one or more of the following benefits (Figure 2.3 shows the benefits of 3 types of composites):

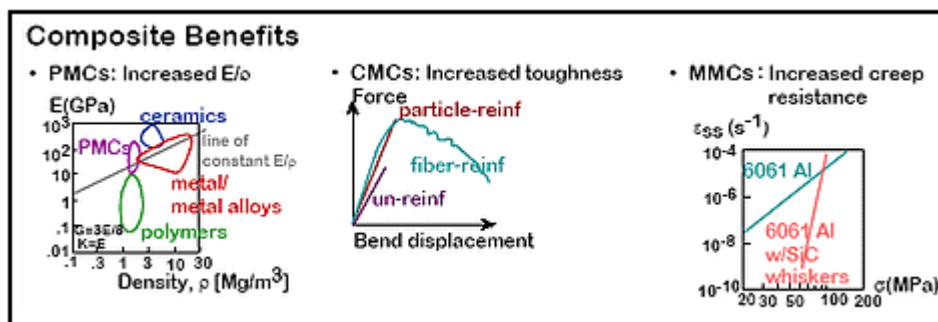


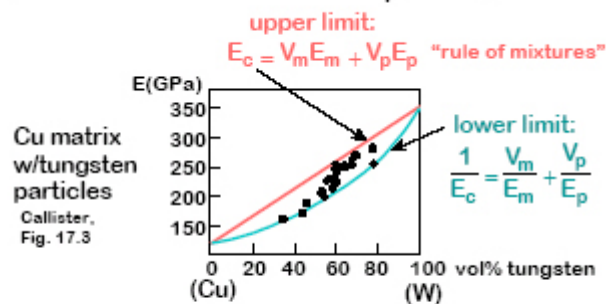
Figure 2.3: Composite Benefits

- 1) High strength – Composites are materials most effective in delivering high strength. Unlike the normal materials, it is able to have the strengths of composites oriented or made to meet specific design requirements of an

application. In general, composites are materials that could provide a wide range of mechanical properties (tensile, flexural, impact compressive strength).

- 2) Light weight – Comparing to the other plastics/metals which are not reinforced, composites deliver more strength per unit of weigh and thus this high strength/light weight combination is an additional benefit for the effective use of composites.
- 3) Design Flexibility – A designer may want to form a material into any shape and composites are flexible in this case to carry out this function, be it complex or simple small or large structurally, for decorative or functional purposes. With composites, many choices are available without having to make costly trade-offs. Besides that, composites give the designers freedom to try new approaches, from prototype to production of the product. 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 – Thermoset composites are capable of maintaining their shape and functionality under severe mechanical and environmental stresses. Unlike the not reinforced thermoplastics, composites do not exhibit

the viscoelastic or “cold-creep” characteristics. The coefficient of thermal expansion is reduced. So, basically, the yield point of a composite is its break point.

- 5) High Dielectric Strength – Another of composites’ excellent properties is electrical insulation, thus is a good choice for current carrying components. Through the use of suitable modifiers and additives, imparting electrical conductivity to composites is possible, which is required by the application.
- 6) Corrosion Resistance – Composites do not rust or corrode. For almost every chemical and temperature environment, there are a couple of resin systems which provide long-term resistance. Composite parts that are well designed typically have long service life as well as minimum maintenance.
- 7) Parts Consolidation – Often, the assemblies of many parts and fasteners required for normal materials, e.g. steel, are replaced by composite moldings. By doing so, manufacturing costs can be reduced and parts are generally more trouble-free.
- 8) Finishing – It is possible to mold colors into products in composite applications for appearance that are durable yet with minimum maintenance. Low profile and low-shrink resin systems are compatible with most metallic painting operations. To reduce trim waste, flash, sanding and other post-molding operations, proper design of molds and choice of materials are essential.

- 9) Low Tooling Cost – Tooling costs for materials such as steel, aluminum, alloys, etc, are generally higher than the tooling costs for composites, in spite of the processing methods selected.
- 10) Proven History of Successful Applications – In the last 45 years, there have been over 50,000 successful composite applications that has have contributed in proving the values of these materials. These days, engineers, designers and marketing professionals can point with confidence to an expansion of end uses and applications providing evidence of the cost and performance benefits of composite unlike the days where the pioneers of the industry once struggled to make this material accepted.

2.4 The Basics of Polymers

The term polymer comes from “poly” meaning many and “mer”, describing a unit. Polymers are generally monomers, a single building block, that are joined together. Composites usually are formed by polymers which come from sophisticated chemical processing. A simple introduction to the basics of polymers would help to understand the chemistry involved.

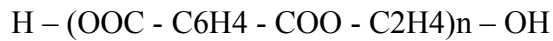
Basically, plastics are made up of the following significant chemical elements (in shorthand notation):

C = Carbon

H = Hydrogen

O = Oxygen

An example of a typical polyester resin in shorthand:



where the structure in 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.

Thus, styrene would be shown as:



2.5 Thermoset versus Thermoplastic

There are 2 major groups for resins or plastics: thermoset and thermoplastic. For thermoplastic resins, when heated, they soften and thus enabling the process of shaping or molding while in a heated semi-fluid state. As for thermoset resins, they are usually in liquid state or are low-melting-point solids in their initial form. When used to produce finished goods, these thermosetting resins are “cured” by using catalyst, heat or both. Once the solid thermoset resins are cured, conversion back to their original liquid form is no more possible. In other words, they will not melt and flow when heated and cannot be reshaped.

Due to the dissimilar requirements of the fabrication process for thermosets and thermoplastics, the composites industry has been divided into these two different camps with both types still benefit from reinforcement. Initially, the growth of composites was in thermosets; primarily glass fiber reinforced unsaturated polyester resins. These days, the growth has moved to reinforced thermoplastics and is expected to go on expanding due to the improved properties and effective cost of

thermoplastics. Thus, it is crucial that those who consider using the composites have sufficient knowledge about 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. In order to select the proper material for a specific application, understanding of the difference between these groups of resins is necessary.

2.7 Polyester Resins

Out of the total resins used, unsaturated polyester resins account for about 75% in the composites industry. For injection molding of both composite and non-composite parts, these resins are available in a different grade. Polyesters are produced by the condensation polymerization of dicarboxylic acids and diatomic alcohols (glycols). Apart from that, unsaturated materials, such as maleic anhydride or fumaric acid, could be found in unsaturated polyesters as part of the dicarboxylic acid component. The finished polymer is then dissolved in a reactive monomer, such as styrene, to give a low viscosity liquid. The monomer reacts with the unsaturated sites on the polymer once the resins are cured. Thus, this converts the polymer into a solid thermoset structure.

Many different acids and glycols are used in polyester resins. The following lists some of the common ones and the reasons as to why they are used:

Glycols

Propylene Glycols
Ethylene Glycols
Dipropylene Glycols
Diethylene Glycols
Neopentyl Glycols
Propoxylated Bisphenol A

Contributes

Water and chemical resistance
Low cost, rigidity
Flexibility, toughness
Flexibility, toughness
Ultraviolet, water and chemical resistance
Water and chemical resistance

Acids

Phthalic anhydride
Maleic anhydride
Adipic acid
Isophthalic acid

Terephthalic acid

Contributes

Low cost, styrene compatibility
Unsaturation
Flexibility, toughness
High heat deflection temperature, strong water and chemical resistance

High heat deflection temperature, strong water and chemical resistance

Due to the versatility of polyester, there are a range of raw materials and processing techniques that are available in achieving the desired properties in the formulated or processed polyester resins. Also, polyesters have been found to have almost absolute usefulness in all segments of the composites industry due to their capacity to be modified or tailored during the construction of the polymer chains. The principal advantage of these resins is a balance of properties (mechanical, chemical and electrical), dimensional stability, cost and ease of handling or processing.

Unsaturated polyesters are divided into classes depending on the structures of their basic building blocks. For example, Orthophthalic (“ortho”), Isophthalic (“iso”), Dicyclopentadiene (“DCPD”) and Bisphenol A. Fumarate resins. Besides that, polyester resins are also classified according to the end use application as either a general purpose (GP) or specialty polyester.

2.8 Specialty Polyesters

A number of specialty polyesters are available. This is due to the capability of polyesters in meeting the requirements of a range of applications when chemically tailored. 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

The chemical makeup of a polymer brings about the performance of specialty polyester. Properties such as fire resistance, fatigue performance and chemical resistance could be enhanced with the correct use of fillers or additives. When one property is improved, for example chemical resistance, other properties, such as temperature resistance, may also be improved. An example is Bisphenol A Fumarate. It has the ability to tolerate a range of chemical exposure and higher on-service temperatures, thus is used for fabrication purposes.

Liquid styrenated polyester resins can be easily shipped to fabricators who do the final shaping and curing of these materials into useful products. The reaction between the unsaturation in the polyester and the styrene monomer is the mechanism for curing resulting in the polyester chains being tied together by the styrene monomer. Unlike epoxies and urethanes or phenolics, the curing of polyester resins differs in the

sense than most epoxies and urethanes will begin to increase in viscosity the moment they are catalyzed until they are cured. As for polyester, there is minor viscosity increase or temperature change in a specific working time (gel time). Gelation takes place when less than 5% of the original unsaturation has reacted and full cure occurs very rapidly after this.

2.9 Epoxy Resin

Epoxy resin has a firm record where composite parts and structures are involved. Many different products with a diversity of levels of performance can be produced from engineered structure of epoxy resins. Also, they can be formulated with various materials or blended with other epoxy resins to obtain a particular performance characteristic. Hardeners and/or catalyst systems that are properly chosen enables cure rates to be controlled to match process requirements. Epoxies are basically cures by addition of an anhydride or an amine hardener and each of these hardener yields different properties in the finished composite.

The primary uses of epoxies are generally in the fabrication of high performance composites with superior mechanical properties, resistance to corrosive liquids and environments, superior electrical properties, great performance at elevated temperatures or any combination of these benefits. To provide a reasonable ground for the superior performance but higher cost resin systems, critical applications on the use of epoxies are usually required. An example of the use of epoxy resins are in the marine automotive electrical appliance, besides other composite parts and structures. However, provided that special performance is required, these are generally not cost

effective in these markets. In comparison to most polyester resins, epoxy resins have much higher viscosity and post-cure is required to achieve the ultimate mechanical properties, thus are more difficult to use.

Besides being used with a number of fibrous reinforcing materials, including glass, carbon and aramid, epoxy resins are also used as matrix resins for “whiskers” such as boron, tungsten, steel, boron carbide, silicon carbide, graphite and quartz. Nevertheless, the latter group is of small volume, comparatively high cost, and is usually only used to achieve high strength and/or high stiffness requirements. Among the benefits of epoxies are that they are readily usable with most composite manufacturing processes. The processes in particular are pressure-bag molding, vacuum-bag molding, compression molding, autoclave molding, hand lay-up and filament winding.

2.10 Thermoplastic Resins

When combined with reinforcing fibers to produce a composite material, thermoplastic resins provide unique and beneficial properties. Designers are drawing advantages from the properties of the thermoplastic composite material in order to improve the performance of the product as well as minimizing the manufacturing costs. This differs from thermoset resins that are described as hard and somewhat brittle. In other words, thermoplastic resins are characterized as tough and offers superior impact resistance. Apart from that, the curing process for thermoplastic resins to obtain their final properties also takes a shorter time, meaning, cycle times are shorter, productivity is increased and the costs of parts are lower. What is of

interest is that composites produced from thermoplastic resins especially in the automotive markets, can be readily recycled, making it meet the demands of environmental mandates. Besides, these resins are naturally impervious to attack from harsh chemicals, petroleum products, environment products and environmental elements. The group of thermoplastics resins is also large and wide-ranging, thus enabling the selection of a resin based on specific properties tailored to the end application.

2.11 Vinyl Ester

Vinyl ester was developed to merge the favorable qualities of epoxy resins with better handling/ faster curing process typical for unsaturated polyester resins. By reacting epoxy resins with acrylic or methacrylic acid, vinyl ester is produced providing an unsaturated site. This is basically similar to that produced in polyester resins when malefic anhydride is used. The material obtained it then dissolved in styrene, thus producing a liquid much like the polyester resin. For the curing, this is done so on vinyl esters with conventional organic peroxides (used with polyester resins).

In terms of mechanical toughness and corrosion resistance, vinyl ester shows these beneficial qualities and these qualities are obtained with no complex processing, handling or special shop fabricating practices involved unlike epoxy resins.

2.11.1 History and commercial of Vinyl Ester

Developed in the 1960s, vinyl ester resins are addition to the products of various epoxy resins and unsaturated monocarboxylic. These are the most common materials

to be mixed with acrylic acid. Combined with the best attributes of epoxies and unsaturated polyesters, vinyl ester resins are easy to handle in room temperature. The temperature and mechanical properties are alike to epoxies resins but generally have better chemical resistance than cheaper polyester resins.

Chemical-wise, vinyl ester (VE) is related to unsaturated polyesters and epoxies, or seems to be composed of these two. VE came about from an attempt in combining the fast and simple cross linking of unsaturated polyesters with the mechanical and thermal properties of epoxies.

This shrinkage is taken seriously especially when the components of the composites are large that could go up to a value of more than 10%, much higher than claimed by some researchers and resins' manufacturers (Clarke, 1996; Matthews, 1994). The primary problem is due to the stresses set up internally. Usually, these stresses are tensile in the core of the component and compressive on the surface (Osswald, 1995). When the stresses act together with the applied loads during service, it causes a premature failure in the components of the composites. The Fibre Composite Design and Development (FCDD) group has solved this shrinkage problem by breaking the large composite into smaller parts. The explanation to this was that the smaller composite parts would have less shrinkage. These smaller parts would then be combined into one to create the overall structure. All these are individual items of the components that are produced by casting in liquid form, 44% uncured (by volume) or 33% (by weight). The vinyl ester is reinforced by flyash particulates in the moulds. [VE/FLYASH (33%)].

Chapter 3

Interactions of Resins with Microwaves

3.1 Introduction

Unsaturated polyesters (UP), epoxies and vinyl esters were nowadays the most widespread used as composite matrices in industry. 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 °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 bisphenol A vinyl ester in Figure 3.1. 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 vinyl ester 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 (DGEBA) as shown in Figure 3.1 (Astrom, 1997).

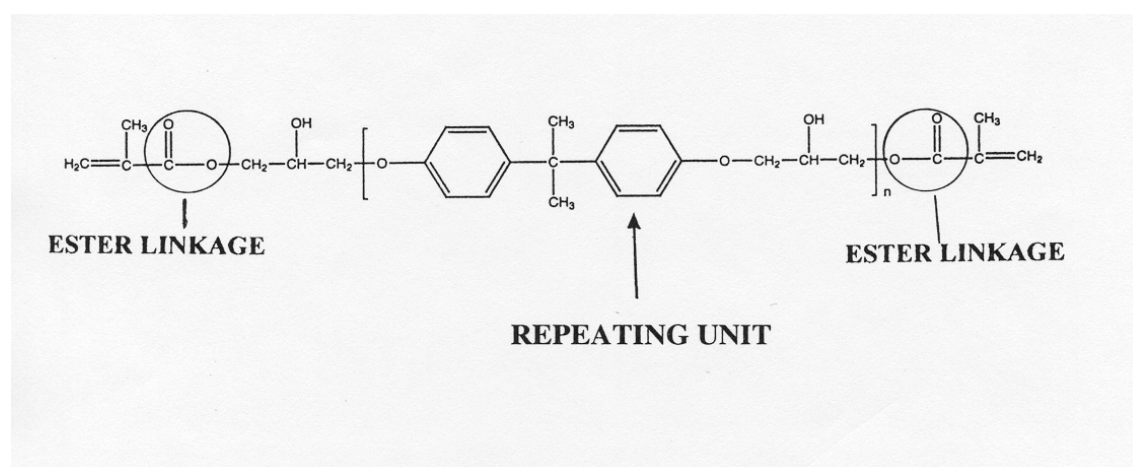


Figure 3.1: The structure of bisphenol A vinyl ester

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

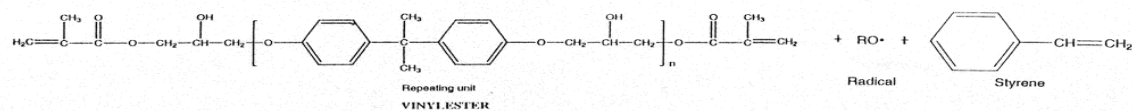
dissolved in a low molecular monomer, or reactive diluent, usually styrene, the result is a low viscosity liquid referred to as resin. The resin used in this research has 50% by weight of styrene. With the addition of a small amount of initiator to the resin the cross linking reaction, or curing, is 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 produces free radicals. The free radical 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

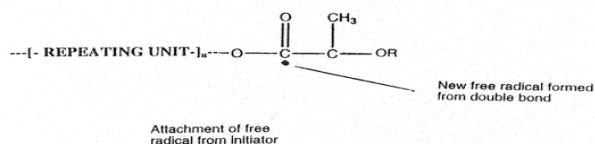
cross linked molecular morphology represents a lower energy state than the random molecular arrangement in the resin, the reaction is exothermal. Further the free radical production is stimulated by an increase in temperature also promotes molecular mobility. Until diffusion limitations reduce the reaction 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.

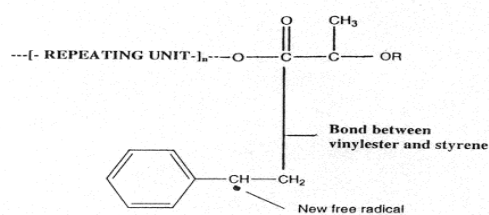
REACTANTS



INITIATION STEP



BRIDGING STEP



CROSSLINKED POLYMERS

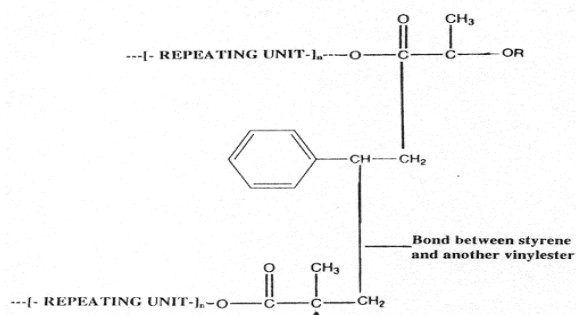


Figure 3.2: Schematic of addition or free radical cross linking of vinyl ester

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.

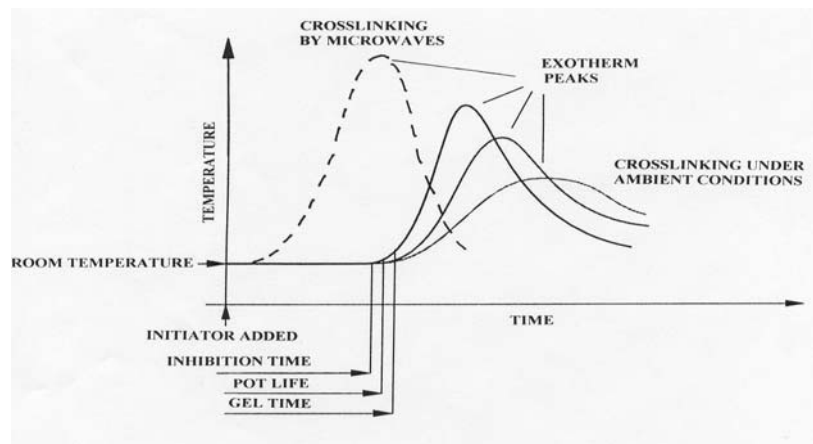


Figure 3.3: Temperature time relationships for cross linking of vinyl ester

3.4 Shrinkage in VE/FLYASH (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 (DGEBA). 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 3.1 has shown the volume of the composites after shrinkage and before shrinkage:

Table 3.1: 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 (°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 °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 $137 \times 1.1 = 151$ g of resin, where 1.1 is the relative density of the resin. Similarly, the mass of hardener required is $2.8 \times 1 = 2.8$ 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 = $110 \times 0.7 = 77$ g. 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):

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

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

The shrinkage is $\frac{200ml - 187.20ml}{200ml} \times 100\% = 6.4\%$.

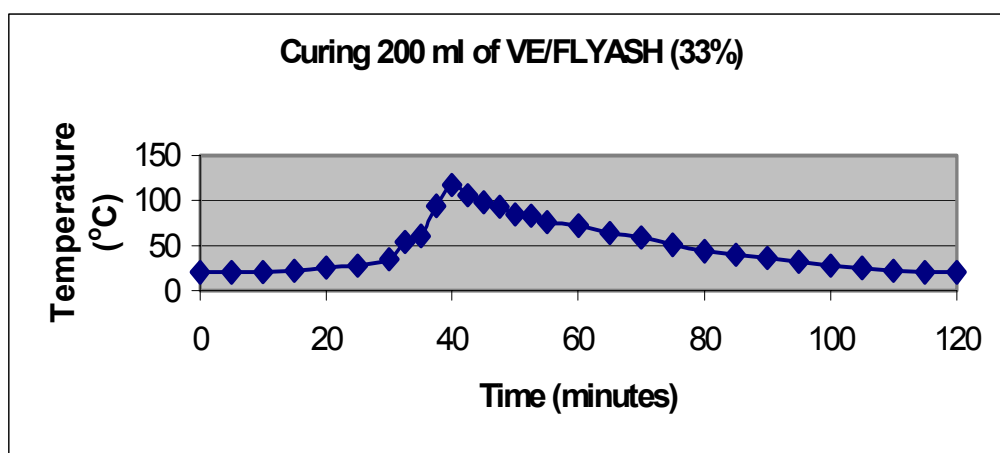


Figure 3.4: Relationship between temperature and time in curing 200 ml of vinyl ester composite, VE/FLYASH (33%) under ambient conditions

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.

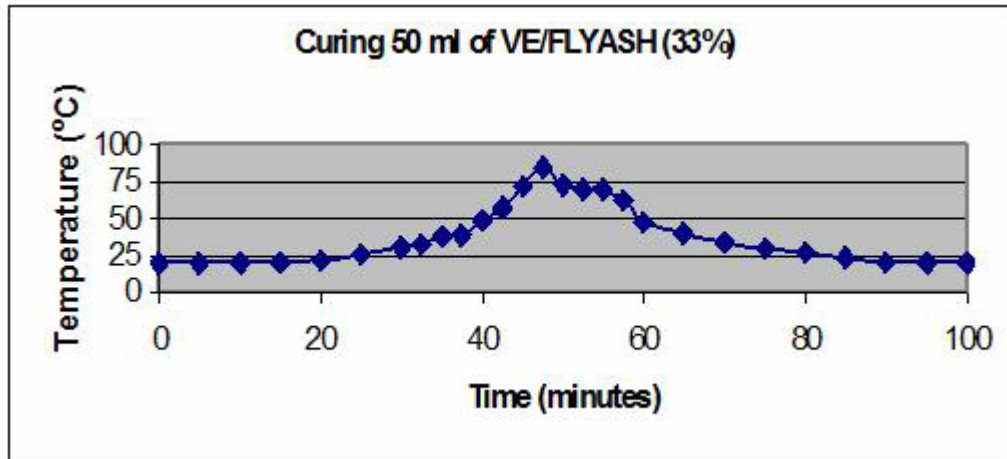


Figure 3.5: Relationship between temperature and time in curing 50 ml of vinyl ester composite, VE/FLYASH (33%)

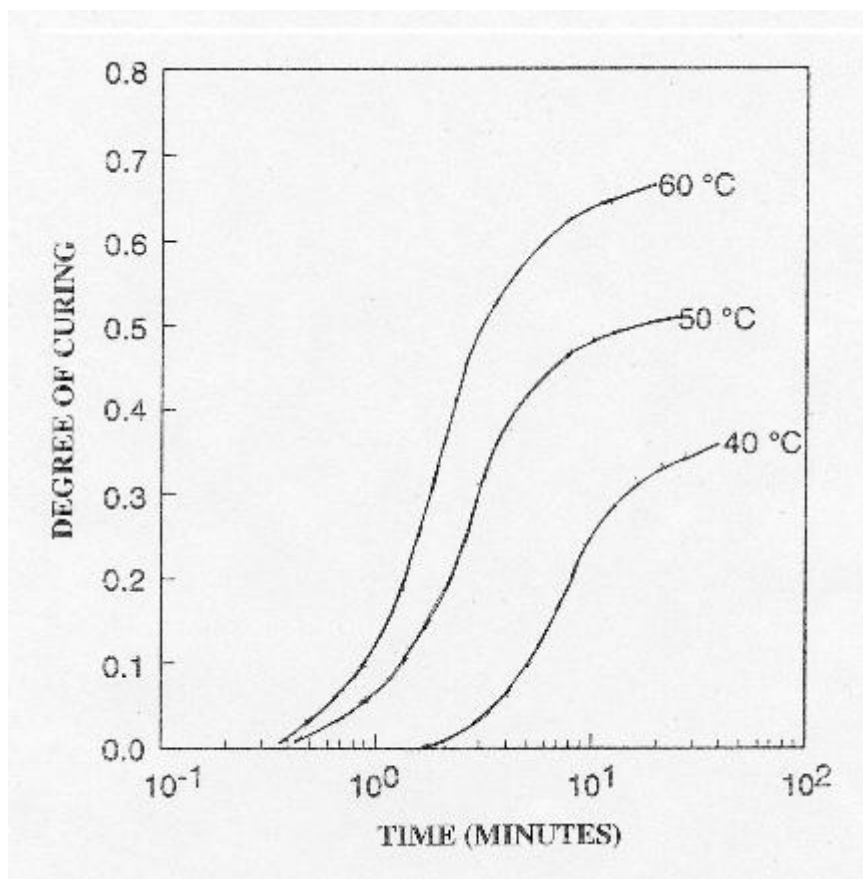


Figure 3.6: Degree of cure of vinyl ester at different curing temperatures

3.5 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 3.2 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).

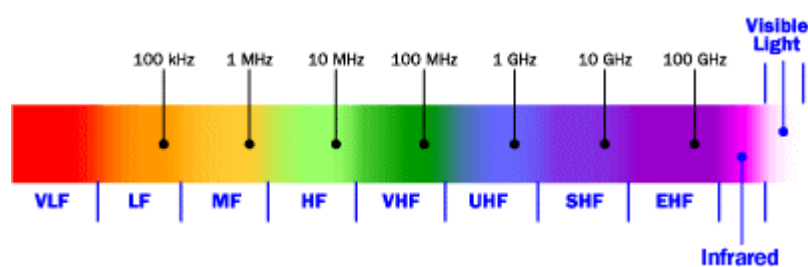


Figure 3.7: Frequency Bands for Radio Frequency Range

Table 3.2: 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.

3.6 Microwave and material interactions

The material properties of greatest importance in microwave processing of a dielectric are the complex relative permittivity $\epsilon = \epsilon' - j\epsilon''$ and the loss tangent, $\tan \delta = \epsilon'' / \epsilon'$ (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, $\tan \delta$ 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 $\tan \delta$, 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):

$$D_p = \left(\frac{4.8}{f} \right) \frac{\sqrt{\epsilon'}}{\epsilon''} \quad \text{(Eqn 3.2)}$$

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.

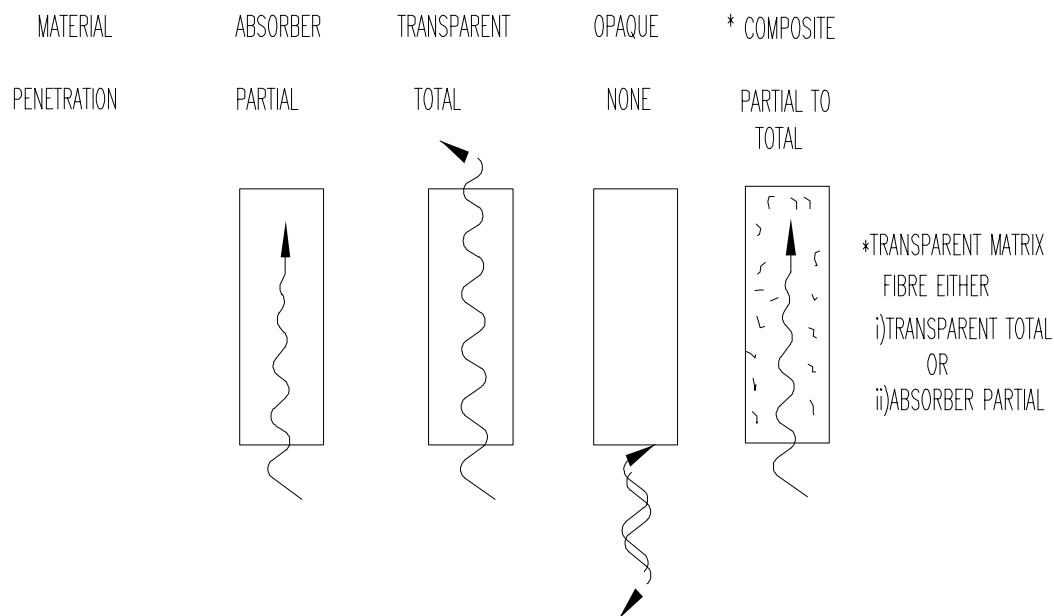


Figure 3.8: Interaction of Microwaves with Materials

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

Chapter 4

Fracture Mechanics

4.1 Description of Fracture Mechanics

Fracture mechanics can be defined as a field of solids mechanics that deal with the behaviour of cracked bodies subjected to stresses and strains. Determine the severity of a pre-existing defect in term of its tendency to initiate a fracture which would cause failure is the aims of fracture mechanics.

4.2 Fracture Toughness

Typical fracture toughness test can be carry out by applying a tensile stress to a specimen prepared with a flaw of known size and geometry which is shows in Figure 4.1. The stress applied to the material is increasing at the flaw, which it acts as a stress raiser. The stress intensity factor K for a simple test calculation is shown as below:

$$K=f \sigma \sqrt{\pi a} \quad (\text{Eqn 4.1})$$

Where f = geometry factor for the specimen and flaw

σ = the applied stress

a = the flaw size which defined in figure 4.1

If the specimen assumed to have an 'infinite' width then it calculate as $f \cong 1.0$.

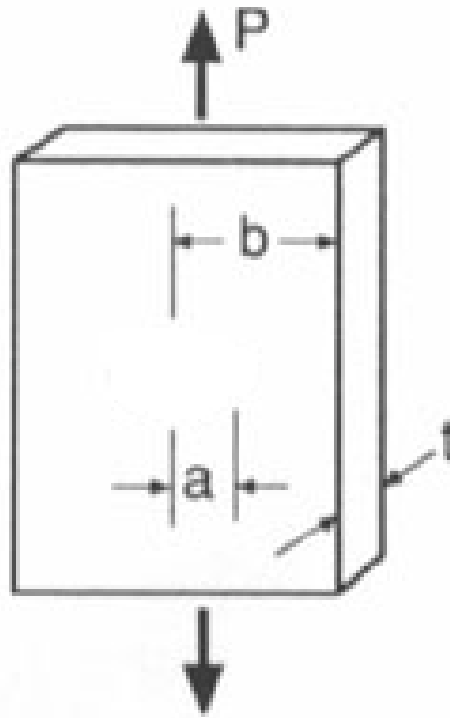


Figure 4.1: A specimen note that the entire crack length is equal to $2a$

By carry out a test on the specimen with known flaw size, the value of k that causes the flaw to grow and cause failure can be determined. The critical stress intensity factor was defined as fracture toughness K_c is the K , which required for a crack to propagate.

When specimen thickness is much greater than the crack dimension, fracture toughness will depend on the thickness of the sample. The fracture toughness K_c will

be then decreased to a constant value. *Plane strain fracture toughness* K_{IC} is what the constant named. It is K_{IC} that is normally cited for most situations as the property of a material. The critical fracture toughness value, K_{IC} can improve the reliability of a structure or component. The ability of a material to resist the growth of crack propagation based on several factors:

- **The ability of the large flaws to reduce its permitted stress**

It can be done by using the special manufacturing techniques, such as filtering impurities from liquid metals and hot pressing of particles to produce ceramic components. It can help to reduce the flaw size and improve the fracture toughness.

- **The ability of a material to distort is critical**

In ductile metals, the material close to the tip of the flaw can be distorted. It is causing the tip of any crack to become blunt, reducing the stress intensity factor, and preventing growth of the crack. The increasing strength of a given metal usually decreases ductility and gives lower fracture toughness. The fragile materials such as ceramics and polymers have lower fracture toughness than metals.

- **Thicker and rigid materials**

These two kinds of materials have lower fracture toughness than thin materials.

- **Increasing the rate of application of the load**

This application as in an impact test, typically reduces the fracture toughness of the material.

- **Increasing the temperature will normally increase its fracture toughness as in the impact test**
- **A small grain size will normally improve its fracture toughness**

This is where more points are defected and dislocations reduced its fracture toughness. Hence, a fine-grained ceramic material may provide improved resistance to crack growth.

4.3 The Role of Fracture Mechanics

Design and select material that deal with the behaviour of cracked bodies subjected to stress and strains was the role of fracture mechanics. Fracture mechanics was an important tool that let structural engineers to have a better understanding of concrete structure behaviour, a better design concrete structures and lighter concrete mixture.

The property of the material (K_C or K_{IC}), the stress σ that the material must withstand, and the size of the flaw a must be considered. We can get the third variable once we figure another two of these variables.

The roles of fracture mechanics been explained as in several steps as below (Donald R. Askeland, 1996):

- **Selection of a material:**

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

- **Design of a component:**

If we know the maximum size of any flaw and the material (and therefore its K_{IC} or K_{IC}) has already been selected, we can calculate the maximum stress that the component can withstand. Then we can design the appropriate size of the part to ensure that the maximum stress is not exceeded.

- **Design of a manufacturing or testing method:**

If the material has been selected, the applied stress is known, and the size of the component is fixed, we can calculate its fracture toughness

4.4 Theories of Mechanics and Fracture Toughness

The development of the field of fracture mechanics was lead by the modifications to Griffith's theory. Fracture mechanics deals with facture initiation and crack propagation, and provides quantitative methods for characterizing the behavior of an intact material as it fractures due to crack growth. The extension of fracture mechanics to rock is understandable since rock masses contain cracks and discontinuities. States of stress around these flaws cannot be predicted using macroscopic failure criteria. In order to deal with crack propagation, particularly in

terms of “intentional” fracturing as in size reduction processes, fracture mechanics must be used.

Although fracture mechanics has an undeniable place in mechanics applications, it was not developed for geomaterials. It should be recognized that differences exist between fracture mechanics for man-made materials (metals) and rock fracture mechanics, particularly in basic material response and engineering application. Whittaker (et al., 1992) gave a comprehensive list and explanation of these differences, which can be summarized as:

- i. ***Stress state*** – Many rocks structures are subjected to compressive stresses as opposed to tensile stresses. However, in comminution and crushing the induced stress state is tensile (from point-load compression) and thus tensile fracture is seen in rock.
- ii. ***Rock fracture*** – Rock materials usually fracture in a brittle or quasi-brittle manner and usually do not exhibit plastic flow.
- iii. ***Fracture process zone (FPZ)*** – Non-elastic behavior ahead of a crack tip in rock takes the form of micro-cracking as opposed to excessive shear stresses and the resultant plastic process zone seen in metals. If the size of the FPZ is small then linear elastic fracture mechanics applies.

- iv. **Crack surface** – Crack surfaces in rock can be non-planar with friction and inter-locking occurring, but linear elastic fracture mechanics assumes that no forces are transmitted across the surface of a smooth planar crack

- v. **Crack propagation** – In rocks there is a tendency for crack propagation to “wander” along grain boundaries or planes of weakness. The area of newly created surface is then larger than the assumed fracture area.

- vi. **Rock fracture mechanics applications** – In rock mechanics, as in (man-made) materials engineering, the prevention of failure by fracture growth is a concern. But the optimizing the generation and propagation of cracks is also a concern as in size reduction processes. Thus the application dictates how material parameters should be determined and used.

- vii. **Influence of scale** – Due to the complicated geologic nature of rock masses, the characterization of a rock mass is high. For the prevention of crack growth and failure, parameters measured experimentally are of secondary importance but for rock fragmentation applications, experimentally measured properties are of primary importance.

- viii. **Heterogeneity** – Changes in local structure and strength ahead of a crack tip affects the continuity of crack growth.

- ix. **Presence of discontinuities** – Pre-existing discontinuities affect the local stress states and crack propagation.

- x. **Anisotropy** – Rocks can be anisotropic affecting measured fracture parameters as a function of crack orientation.

There are more practical and developed concepts of fracture mechanics was lead by the recognition of these variations as it applies to behavior, with principles of linear elastic fracture mechanics being extended even to rocks that behave non-linearly and much of the focus centering on the measurement of fracture toughness.

The most fundamental aspect of rock fracture mechanics is the establishment of a relationship between rock fracture strength and the geometry of the flaws that result in fracture. Through this relationship an intrinsic material property that describes a materials' resistance to crack propagation can be measured. This property is called fracture toughness. The application of fracture toughness in size reduction processes is clear. Fracture toughness represents a critical level above which crack extension and fracture occurs. When individual rock particles are subjected to the applied forces of size reduction, it is most likely that the intrinsic tensile property measured as the fracture toughness will control breakage (Bearman, 1998). Since the amount of energy input into a size reduction process and the amount of size reduction achieved (i.e., the fractured size distribution) are related to the type of loading and the crack pattern in the material, there should be a relationship between these parameters and fracture toughness.

4.5 Transition Temperature Approach

The transition temperature approach assumes that every material below a certain temperature will become brittle. This is caused by the material not being able to

plastically yield so that the stress concentration at the crack tip can not be absorbed thus causing it to fracture a lower stress. Figure 4.2 shows the general effect of temperature of the fracture resistance of structural metal.

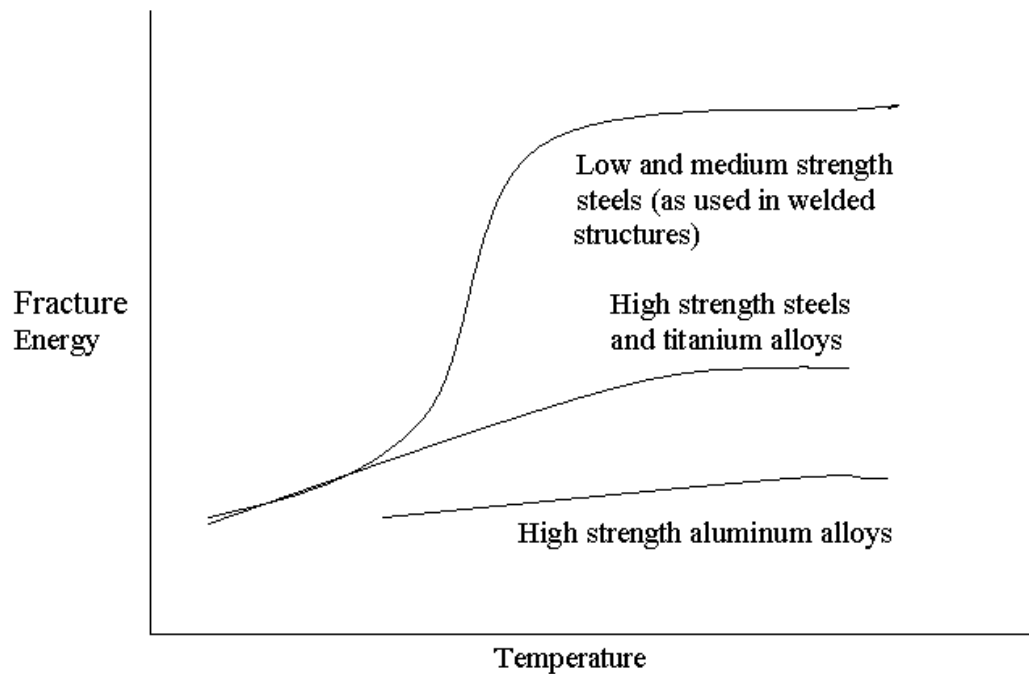


Figure 4.2: The general effect of temperature of the fracture resistance of structural metal

Various tests are used to determine the transition temperature of a material .The transition temperature is difficult to accurately find so a range of temperatures are used, these ranges of results are grouped into general categories (Osgood, 1971):

- ***Fracture-stress Transition temperature:***

At this point the fracture strength of sharp- notched specimen decreases rapidly well below the yield strength

- ***Fracture-mode Transition Temperature:***

At this point the crack propagates changes with decreasing temperature from full shear to flat fracture surface.

- ***Toughness-Fragile Transition Temperature:***

At this point the capability of the material to with stand gross plastic deformation reduced to near zero.

- ***Crack-Arrest Transition Temperature:***

Below this temperature, a running crack cannot be stopped.

The Charpy V-notch test was often used to determine the transition temperatures because several temperatures can be determined off the same which is shows in Figure 4.3.

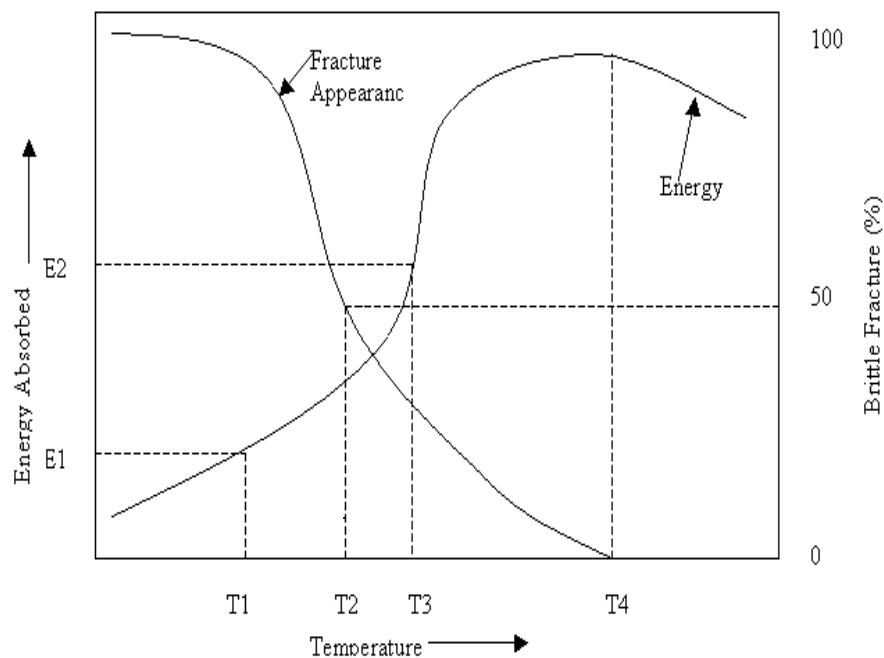


Figure 4.3: Results from Charpy V-notch impact test

T_1 – From fixed level of impact energy

T_2 - Fracture resistance

T_3 – Midpoint temperature

T_4 – Fracture entirely sheer

The transition temperatures are compared with fracture resistance of other materials, which are considered for the design. The material with the lowest transition temperature is considered to be the most fracture resistant.

This method is determining fracture resistance, the results cannot be expressed directly in terms of load-carrying term but with use of a fracture analysis diagram applied stress, defect size and temperature can be related. Figure 4.4 show the fracture analysis diagram

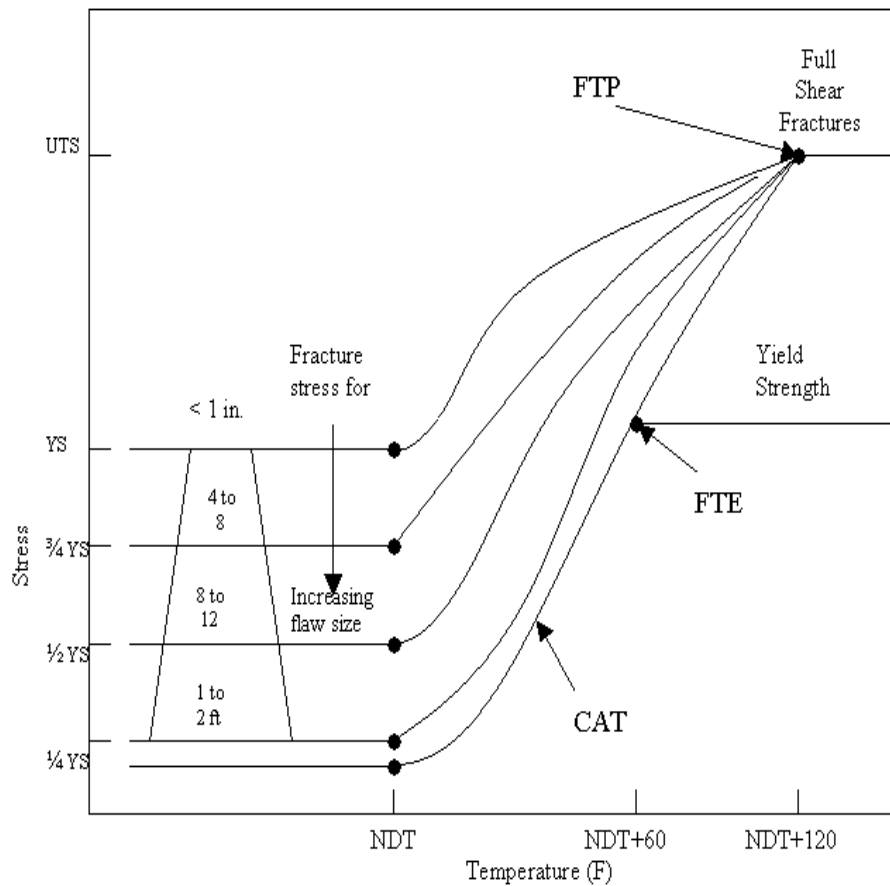


Figure 4.4: Fracture analysis diagram

4.6 Linear Elastic Fracture Mechanics

$$2\sigma_a E \pi a = \gamma \quad (\text{Eqn 4.2})$$

Above equation presented as a size independent expression characterizing the resistance of a material to fracture based on the stresses required for breakage and the presence of flaws. It showed that fracture initiation in a brittle solid is controlled by the product of a far-applied stress and the square root of the flaw length which reaches a critical value determined by the characteristic material properties E , ν , and γ_s . This critical value is called the critical stress intensity factor and is denoted K_c . Irwin (1957) used a stress intensity approach to relate the critical strain energy release rate G_c to the critical stress intensity factor K_c . Rather than follow Griffith's global approach, Irwin considered the crack tip region, which is small compared to the rest of the body but large enough with respect to atomic dimensions such that linear elastic theory applies (Knott, 1972). Irwin determined the work required to close up a small portion of a crack by superimposing tensile forces along the crack surfaces and hypothesized that this work is equal to the energy released when the crack extends. Thus the work required to close a unit length of the crack is the strain energy release rate and, based on the stresses and displacements occurring as a result of the tensile forces, is equal to:

$$G = \frac{(1 - \nu^2)K^2}{E} \quad (\text{Eqn 4.3})$$

Since crack propagation occurs when G reaches a critical value, the critical value of stress intensity can be defined as:

$$K_c = \sqrt{\frac{G_c E}{(1-\nu^2)}} \quad (\text{Eqn 4.4})$$

By demonstrating the equivalence of K and G , Irwin provided the basis for the development of Linear Elastic Fracture Mechanics (LEFM). In LEFM the crack tip stresses, strains, and displacements can be characterized by K as long as inelastic yielding ahead of the crack tip is small. The advantage of LEFM is that it provides a universal approach for determining a material's resistance to fracture, as defined by K_c . As long as an explicit function for the stress intensity near a crack tip is known for a given crack geometry and loading configuration, K_c can be measured experimentally.

4.7 Stress Intensity Factor

Severity of the crack condition as affected by crack dimension, stress, and geometry is mentioned by stress intensity factor K in previous section (Dowling, 1999). Determining K is based on a linear-elastic approach (hence LEFM), which assumes the material in which the crack is located is isotropic and behaves according to Hooke's Law.

Different loading configurations at a crack tip lead to different modes of crack tip displacement. The different types of crack deformation are generalized using three basic modes. Mode I is the opening mode due to tension, where the crack surfaces move directly apart; Mode II is the sliding mode due to shearing, where the crack surfaces move over one another in a direction perpendicular to the crack front; Mode III is the tearing mode also due to shearing, where the crack surfaces sliding over one another but in a direction parallel to the crack front. The three basic modes can also occur in combination as "mixed-mode" loading with the superposition of the modes

sufficient to describe most general three-dimensional cases of local crack tip stress and deformation fields (Tada et al., 2000). Mode I is the most commonly encountered mode in engineering applications and is also the easiest to analyze, produce experimentally on laboratory specimens, and apply (Schmidt and Rossmannith, 1983).

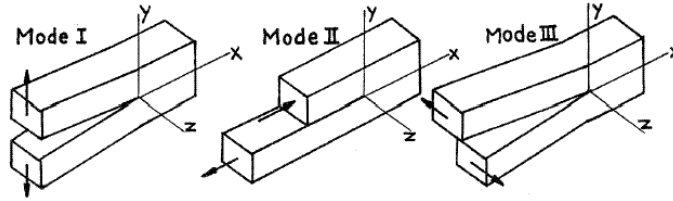


Figure 4.5: The three basic modes of crack surface displacement (After Tada et al., 2000)

Using theory of elasticity, namely the stress analysis methods of Muskhelishvili (1963) and Westergaard (1939), the crack tip stress and displacement fields (and hence K) for each mode of loading can be determined (for a complete derivation see Pook, 2000). Representing the coordinate system measured from the leading edge of a crack, the Mode I stress components are given according to the following equations 4.5:

$$\sigma_x = \frac{K_I}{\sqrt{2\pi r}} \cos \frac{\theta}{2} \left[1 - \sin \frac{\theta}{2} \sin \frac{3\theta}{2} \right]$$

$$\sigma_y = \frac{K_I}{\sqrt{2\pi r}} \cos \frac{\theta}{2} \left[1 + \sin \frac{\theta}{2} \sin \frac{3\theta}{2} \right]$$

$$\tau_x = \frac{K_I}{\sqrt{2\pi r}} \cos \frac{\theta}{2} \left[\sin \frac{\theta}{2} \cos \frac{3\theta}{2} \right]$$

$$\sigma_z = \nu(\sigma_x + \sigma_y), \text{ for plane strain}$$

$$\sigma_z = 0, \text{ for plain stress}$$

$$\tau_{xz} = \tau_{yz} = 0 \quad \text{(Eqn 4.5)}$$

where K_I is the stress intensity factor for Mode I. The displacements at the crack tip can be found by substituting above equation into Hooke's Law.

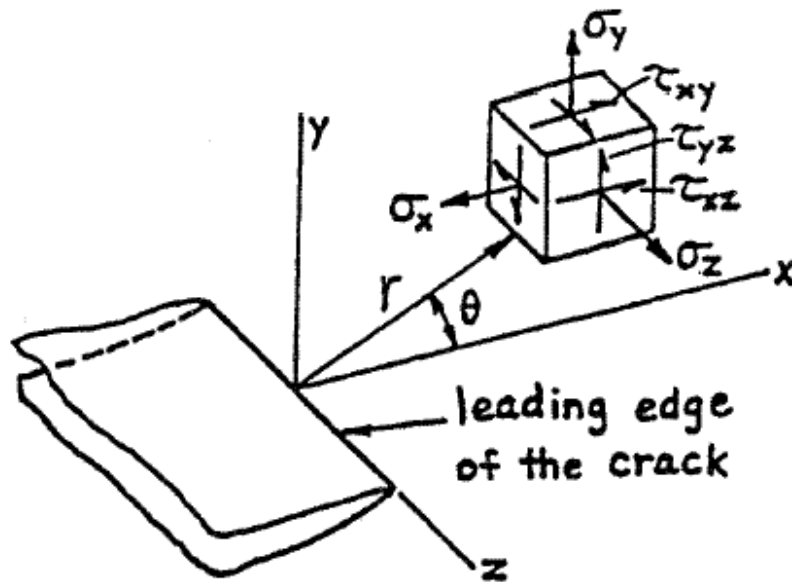


Figure 4.6: Coordinate system for a crack tip

It can be seen from Equations 4.5 that at the crack tip (as r approaches zero) the stresses approach infinity, as has already been indicated by Inglis' solution for stresses around an elliptical hole in a stressed plate. Since no value of stress at the crack tip can be given, and all non-zero stresses of Equation 4.5 are proportional to K_I , with the remaining factors varying only with r and θ , the stress field near the crack tip can be determined by giving the value of K_I , which has a formal definition of (Dowling, 1999; Pook, 2000):

$$K_I = \lim_{r, \theta \rightarrow 0} \sigma_y \sqrt{2\pi r} \quad (\text{Eqn 4.6})$$

It was noted earlier that K_I is affected by the crack size, stress, and geometry. In order to account for different geometries Equation 4.6 can be rewritten as:

$$K_I = F\sigma\sqrt{2\pi a} \quad (\text{Eqn 4.7})$$

where, F is a dimensionless constant dependent on the geometric configuration σ is the stress averaged over the gross area a is the half-crack length. F can generally be described as a function of loading geometry and a/w where w is defined as the maximum possible crack length. When F is determined for a given geometry the critical value of stress intensity, or fracture toughness, can be determined as long as inelastic yielding ahead of the crack tip is small and the conditions for LEFM are met. Equations and values of F for a wide range of crack, specimen, and loading geometries are determined using analytical, numerical, and experimental methods and have been compiled in various handbooks (see Tada et al., 2000; Murakami, 1987; Rooke and Cartwright, 1976; Sih, 1973).

Chapter 5

Tests of Fracture Toughness

5.1 Description of Fracture Toughness Tests

The K_{IC} (fracture toughness) can define as material of a sharp crack that has the characteristics of its resistance to fracture under tensile conditions. It is extremely important property in many crucial design applications. The crack will automatically become unstable and it will propagate quickly until fracture occurs when the stress intensity factor grows and becomes higher than K_{IC} .

Plane strain is the term when the stress state is characterized with thick or bulky parts for which the stress of the flaw is in triaxial tension. However, the material usually fails in a brittle manner when it is in plane strain conditions. In plane strain conditions, if the stress intensity factor exceeds its critical value, the flaw will propagate unexpectedly and run completely through the section.

Plane stress condition is related to all sections of two-dimensional parts which is the most complicated stress can occur and known as biaxial. Consequently, the flaw in plane stress will grow slowly until it is below the increased stress. It will then propagate unexpectedly and total fracture occurs.

The basic concepts of crack growth explained why the plane stress leads to slow fracture and why the plane strain leads to rapid fracture. Hence, it is the reason that

the plane strain fracture toughness K_{IC} is known as such an important property in fracture prevention.

There are several kinds of different testing methods that can be used to determine the fracture toughness, K_{IC} . The following sections will briefly explain some of the standard and non-standard testing procedures.

5.2 Standard Test Methods

United States of America (USA) and United Kingdom (UK) have adopted two similar testing procedures for determining K_{IC} . These tests are documented in The American Standard ASTM: E339 and the British Standard BS: 5447. All standard tests have their supposed dimensions for the different specimens recommend by their relevant standards. All the specimens have a single edge notches that are initiated by low stress fatigue cracking.

5.2.1 Compact Tensile Specimen

The compact tension is a flat plate with single notch, which is fatigue cracked (Figure 5.1). The load is applied throughout two pins in line with the crack tip. As the result, this causes the two-point load onto the specimen. Figure 5.1 show the compact tensile specimen.

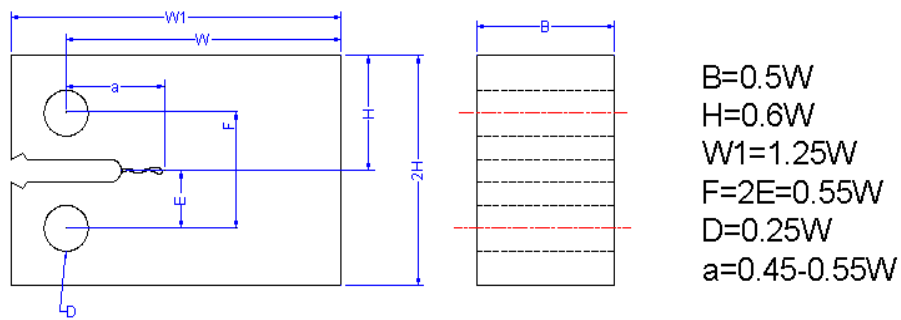


Figure 5.1: Compact tensile specimen

5.2.2 C-Shape Specimen

The purpose of C-shaped specimens is to check on portions of hollow cylinders. The C-shaped specimens have a single notch mid-center of arc which is fatigue cracked. The specimen is loaded throughout pins in two-point bending. Figure 5.2 shows the C-shape specimen.

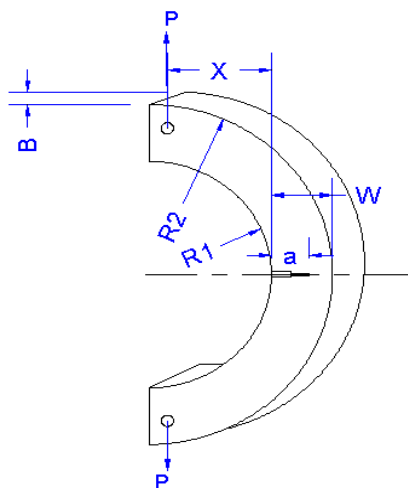


Figure 5.2: The C-shape specimen

5.3 Non-Standard Test Methods

Due to the cost and size of the sample required in the standard test, these type of tests were developed which is shown as Figure 5.3 for Charpy V-notch test rig and sample and Figure 5.4 for short rod specimen configuration and dimensions.

These types of tests are considered as non-standard tests, and the data from these tests which tell the mechanical properties of the material and there are related to the fracture toughness.

5.3.1 Charpy V-Notch Test

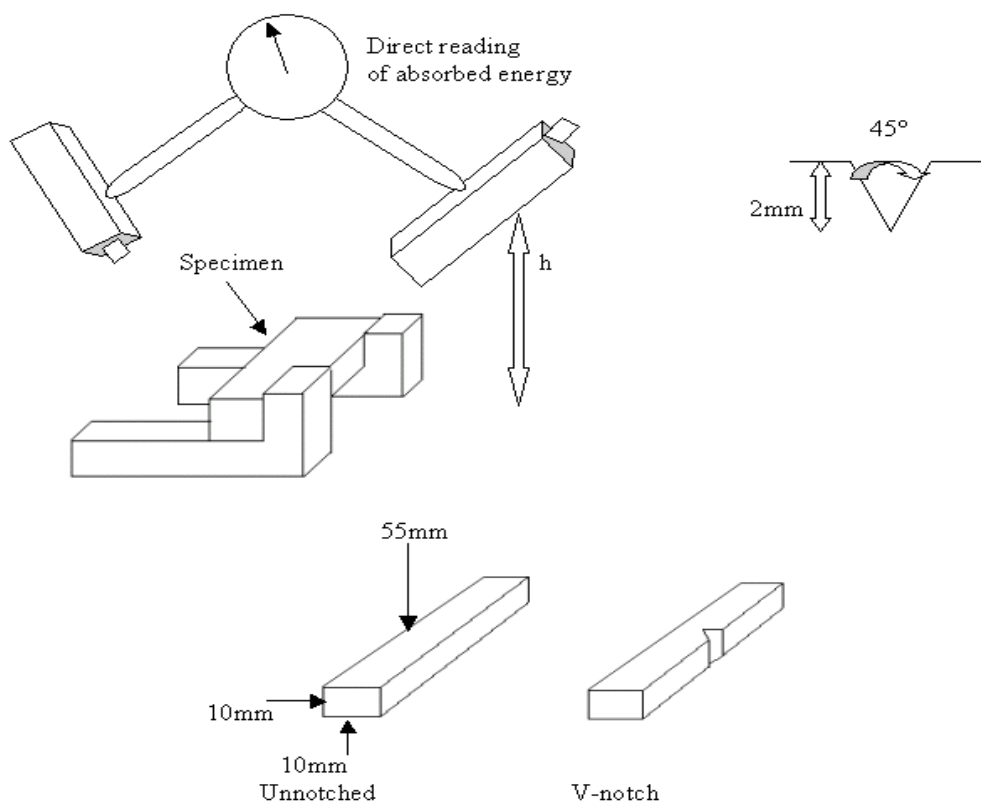


Figure 5.3: Charpy V-notch test rig and sample

The Charpy V-notch test is used to determine the resistance of the material to crack propagation. In this test, the impact energies are measured with reverence to the

temperature. The following two equations can be used to relate the impact test to fracture toughness in order to determine the K_{IC} value of a material:

$$K_{IC}^2 = 2 E (CVN)^{3/2} \quad (\text{Eqn 5.3.1.2})$$

$$K_{IC}^2 = 8 E (CVN) \quad (\text{Eqn 5.3.1.3})$$

The Charpy V-Notch (C_V -NDT) test is associated to the impact energy C_V , that is also associated to the nil-ductility transition temperature. The C_V energy does not correspond directly to the NDT level of fracture resistance. This makes the value of C_V unique for each type of steel; a correlation value should always be used. The V-notch impact test values are generally suitable for correlation with fracture toughness values.

5.3.2 Short Bar Test

Barker (1977) started to work on the concept of devising a simplified method to measure plane strain fracture toughness. The simplified method used small rod and bar shape specimens, that will be cracked through a crack mechanism or by other test rigs. The test is started with an opening load (F) applied to the mouth of the specimen. This causes a fracture to initiate at the point of the chevron. The constant widening of the crack front, as it evolved along the axis of the specimen causes stable crack growth, even in brittle materials. Hence, a "real" crack is created in the specimen before the toughness measurement is made.

For Linear Elastic Fracture Mechanics (LEFM) materials, the fracture toughness measurement is made at the time of maximum load. It is made when the crack is in the

central region (critical crack length) of the specimen. For additional ductile materials, specimen mouth opening displacement is recorded as a function of opening load and simple data analysis methods allocate the calculation of K_{IcSR} (Plane Strain Fracture Toughness, as measured by the Chevron-Notched Short Rod Method).

The load-displacement data allows correction for effects caused by residual stresses in the specimen or plasticity effects in the crack growth. Data analysis methods use to analyze the crack-jump as well as smooth-crack growth types of materials.

The advantages of using short bar test are that the samples size is smaller and it is cheaper to make and to test. The requirement of E 399 for fatigue pre-cracking is not required because of the chevron shaped cut and the short test will be discussed in details in chapter 6.

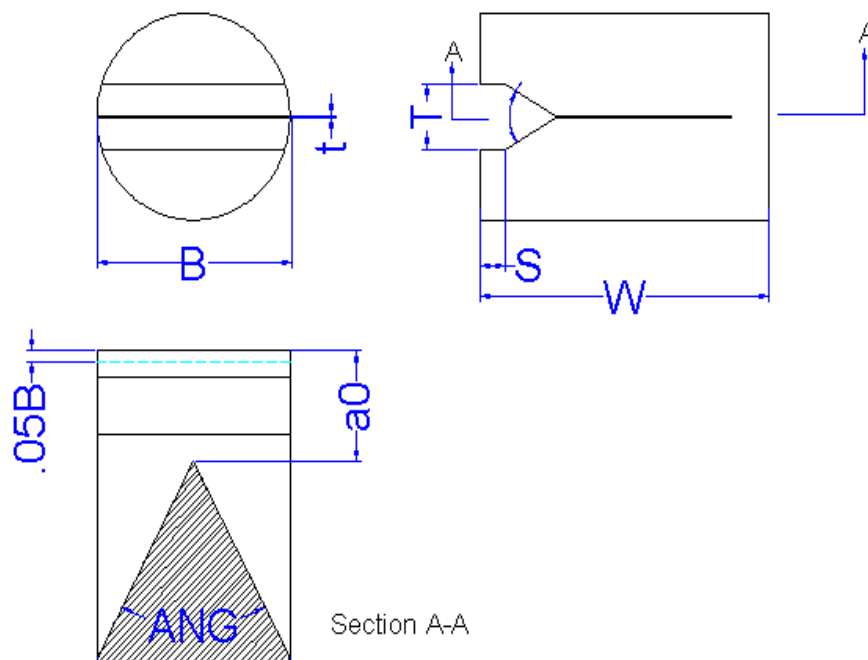


Figure 5.4: Short rod specimen configuration and dimensions

5.4 Analysis of Fracture

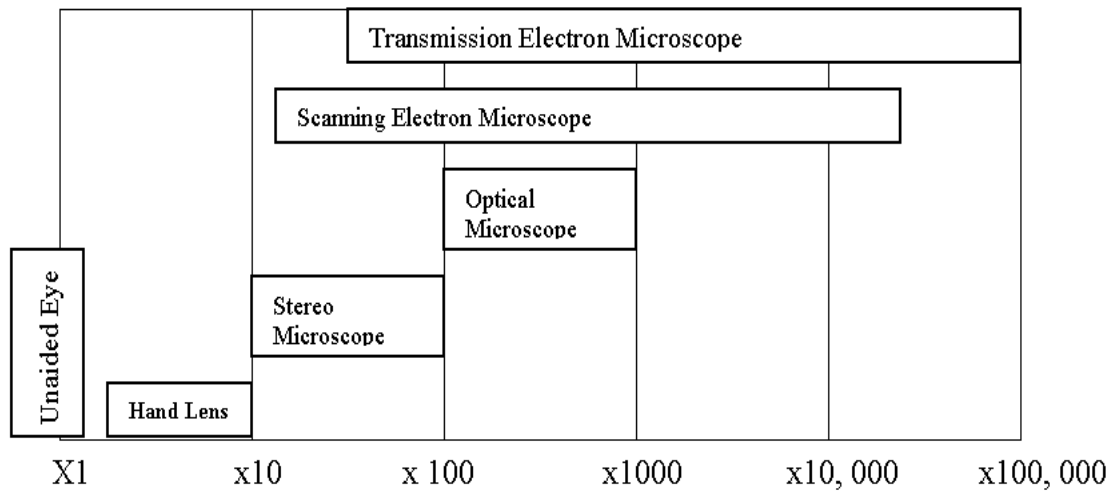


Figure 5.5: The instruments magnification ranges

Fractured surfaces show both macroscopic and microscopic features, where a wide range of instruments with varying magnifications are required. Figure 5.5 shows the magnification ranges, which the certain instruments are effective.

Macroscopic test of the fractured surface have to be completed first, as this can be done with the naked eye or a hand lens. This usually indicates the crack origins and the direction of crack growth. In recent failures, the mode of failure can sometimes be distinguished as the surface has not begun to corrode.

When the site of the crack nucleation is identified, a stereomicroscope is the most useful tool to examine the crack origin for notable features as this could assist in the determination of the mode of fracture.

The Scanning Electron Microscope (SEM) is a very useful tool to examine the fracture surfaces. The advantages of this SEM is because it has a large depth of field

and has a wide range of working magnification from low to high magnifications. The SEM has virtually replaced the optical microscope for direct examination of fractured surfaces.

The Transmission Electron Microscope (TEM) is very useful tool in the field of fractography due to its fracture surface details that can be studied at very high magnifications (up to x100, 000). There are two disadvantages of TEM; it is time consuming and it needs required skills to prepare samples that would be useful.

5.4.1 Brittle Fracture

Brittle fractures generally occur with slight plastic deformation. The strain rates within the material are regularly high because of the stress systems. Brittle fractures occur with little warning because the crack knows how to grow at the speed of sound. Excessive overloading or an impact force can cause a brittle fracture.

Macroscopic analysis of a brittle material failure shows that the materials fail differently which depends on whether it was in tension or compression. When the material fails in tension, the crack is perpendicular to the applied load. If the sample fails in compression the fracture will occur at 45 degrees to the applied load.

The crack is usually initiated from flaws of the material; these can be caused by either surface finish or impurities within the material. The cracks can be intergranular or transgranular depending on the material. A truly brittle fracture is caused by cleavage,

which means transgranular. Cleavage occurs when the material is under high constraint conditions (Baddeley, D T and Ballard J, 1991).

5.4.2 Ductile Fracture

A ductile fracture is the result of plastic deformation prior to failure. A ductile fracture usually occurs when the sample is in strain. Overloading usually causes ductile transgranular fractures. It can be sometimes recognized from macroscopic test of the failed specimen. Usually the specimen is thin size and there is contraction of the sample before failure occurs.

At a microscopic level, most of the structural materials fail by a process called microvoid coalescence. Microvoid coalescence caused the fractured surface to have a dimple appearance with both large and small dimples. The shape of the dimple is influenced by the type of loading which is applied to the sample. Failures caused by shear will produce extended shaped dimples that point in the opposite directions on the matching fracture surfaces. Tensile tearing produces extended dimples that point in the same direction on matching fracture surfaces (Baddeley, D T and Ballard J, 1991).

Chapter 6

Short Bar Test

6.1 Standard Tests

There are currently two similar testing procedures for determine K_{Ic} which are used by United States of America and the United Kingdom. These tests were documented in the ASTM (Americans Standard Test Method) and the BS (British Standards Institution Test Method). All standard tests have there supposed dimensions for the different specimens recommend by their relevant standards. All the specimens have a single edge notches that ate initiated by low stress fatigue cracking.

6.2 Non Standard Tests

Except both of Charpy V- notch test and Short rod/ bar test, there are several other tests that are considered as non-standard tests, the data from these tests which tell the mechanical properties of the material and there are related to the fracture toughness. These type of tests were developed due to the cost and size of the sample required in the standard test.

6.2.1 Short Bar Test

Since the fracture toughness of a material is an important property, which is vital in design applications. The valid measurement of K_{Ic} only can determine by using ASTM and BS standard tests; however, there are many disadvantages for these standards, such as:

- Example like a sufficiently large specimen, it will preclude the fracture toughness measurement, due to the size of specimen required by the standard is too large, it cannot be removed from the structure of interest.
- As the size of specimen required, the size of tensile machine is also very large which means that the availability of machine is limited and it will cost much in the testing as well.
- Some material properties such as brittleness and high fracture toughness combined with low yield strength sometimes makes it impossible to meet the specimen requirements of the standard tests.
- The fatigue pre-cracking which is required of the samples.
- The difficulties in measuring the crack length.
- Lastly, the overall cost of testing one sample is very expensive.

An alternative test method was considered, which was less complex and applicable to a wider range of materials. The short bar test was developed by Barker in 1977. And he considered that the test samples should exhibit some crack growth stability. His testing method used simple fracture toughness concepts to test sample configurations exhibiting crack growth even when loaded by a controlled force machine.

Once calibrated for specimen configurations, the only parameter required for fracture toughness values was the peak force required to completely fracture the sample. This new test method used samples of a circular or rectangular cross section, which were called short rod/bar samples accordingly (Barker, 1977). On the other hand, the advantages of short bar test were (DiJon Inc, Technical note 503):

- No fatigue pre-cracking was required therefore the method was applicable to brittle as well as ductile materials.
- No load versus deflection graph required.
- No crack position or crack length measurements required.
- The size of specimen for a valid test has been reduced.

Therefore, those advantages are able to bring a reduction in costs of testing equipment and samples. Therefore, ASTM committee is now considering this method as an additional specimen configuration for the determination of fracture toughness (K_{Ic}).

6.3 Selection of the Short Rod or Bar Geometry

The configuration of the short bar specimen was selected on the basis of large number of tests of specimens with different length-to-diameter ratios and various chevron slot geometries. The criteria on which the current geometry was selected was:

- The tendency for the crack to “pop” at the initiation should be minimized. The crack initiation should be as smooth as possible.
- The crack should tend to be well guided by the chevron slot.
- The width of the crack front should be considerable portion of the specimen diameter at the time of toughness measurement.
- The crack should be near the centre of the specimen (far from both ends) at the time of the toughness measurement.
- The load should be at or near its peak value at the time of toughness measurement.
- The specimen geometry should be as simple as possible for ease of fabrication.
- The specimen should be economical in its use of sample material.

Some of these criteria could not be achieved at the same time. The short rod and bar specimen configurations were selected as a reasonable compromise in an attempt for an optimum geometry. [Baker, 1981]

6.4 Geometry of Specimen

There are four main different types of basic geometries, which are illustrated in figure 6.1; therefore the mode of manufacture can be differed where required. The decision can be either use a short bar or rod specimens by depending on the available machinery equipments. However, the short rod is easier to manufactures when a lathe is available. The plan view (Section A-A) shows that the short bar and rod specimens are exactly the same. Therefore, the short bar and rod calibrations are the same and experimental studies have shown that the two samples can be considered equivalent (Barker, 1979).

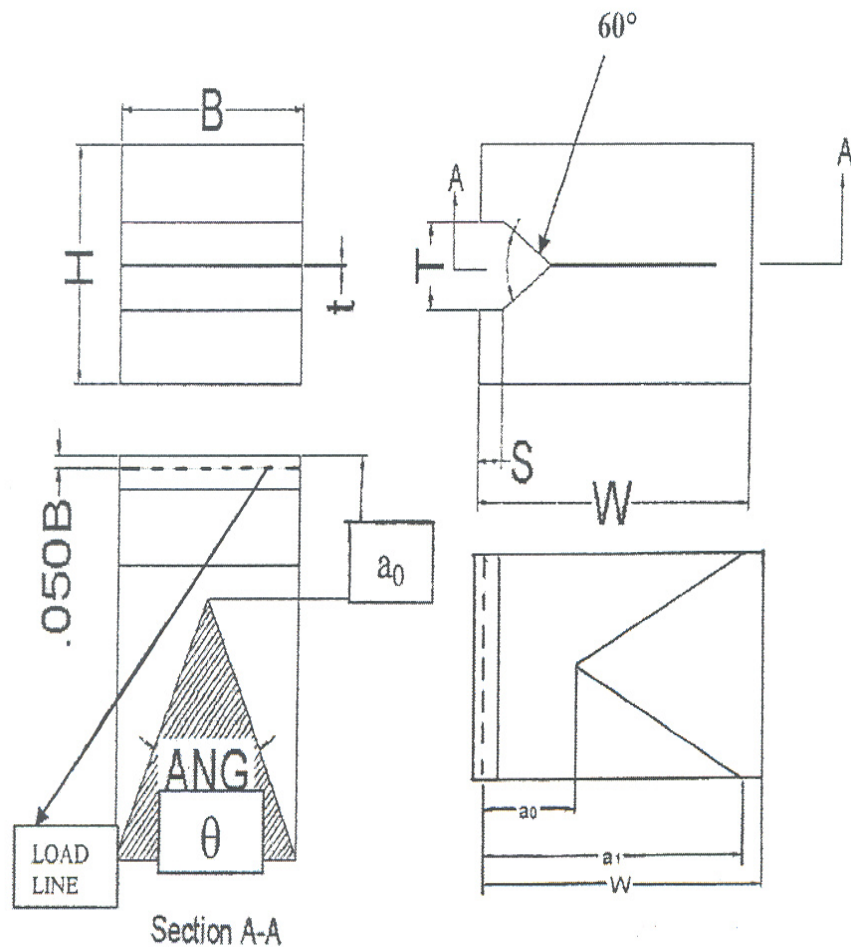


Figure 6.1: Short bar specimens with curved chevron slots

SYMBOL	DEFINITION	VALUE	TOLERANCE
B	BREADTH	B	
W	LENGTH	1.5B	$\pm .010B$
H	HEIGHT	.870B	$\pm .005B$
a_0	INITIAL CRACK LENGTH	.513B	$\pm .005B$
θ	SLOT ANGLE	55.2°	$\pm 1/2^\circ$
t	SLOT THICKNESS	SEE TABLE III (of Barker, 1981)	
S	GRIP GROOVE DEPTH	.130B	$\pm .010B$
T	GRIP GROOVE WIDTH	.313B	$\pm .005B$
R	RADIUS OF SLOT CUT	SEE FIG 4 (of Barker, 1981)	$\pm 2.5B$

However, the curved slots have different geometries because of the way in which the slots are cut. The straight-line slots are cut either by using a saw or cutter, which can move through the specimen. The curved slots are obtained using a cutter which has a plunge type feed, since the calibration of the straight slotted specimens and the curved-slotted specimens are not equivalent.

The geometries were modified by superimposing the both of the plan views and adjusting them until the slots configuration were a tangent to each others; therefore this will give a critical crack length, a_c . Figure 6.2 shows the diagram of critical crack length.

Since, it is easier to measure the curved slots in terms of a_0 , the distance from the edge of sample to the point of the slot, and chord angle θ . The critical crack length is where the peak load occurs which is the fracture toughness measurement is made which is shown in Figure 6.1.

A table of equivalent a_c for the versatile a_0 and θ are shown in Figure 6.3 diagram, this is because a constant specimen calibration can be used regardless of specimen size, when the crack is in the vicinity of the critical crack length, a_c .

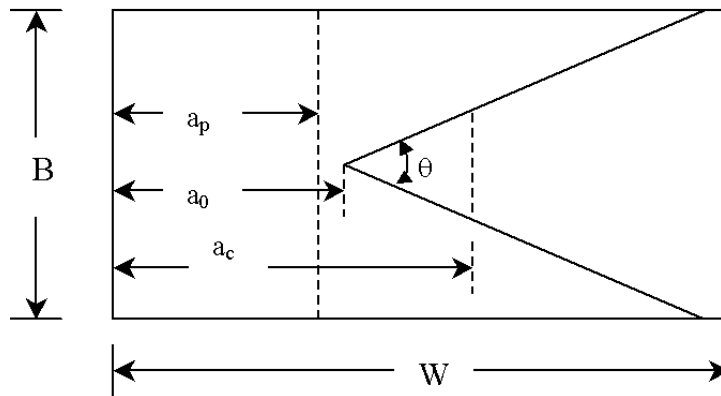


Figure 6.2: Diagram of critical crack length

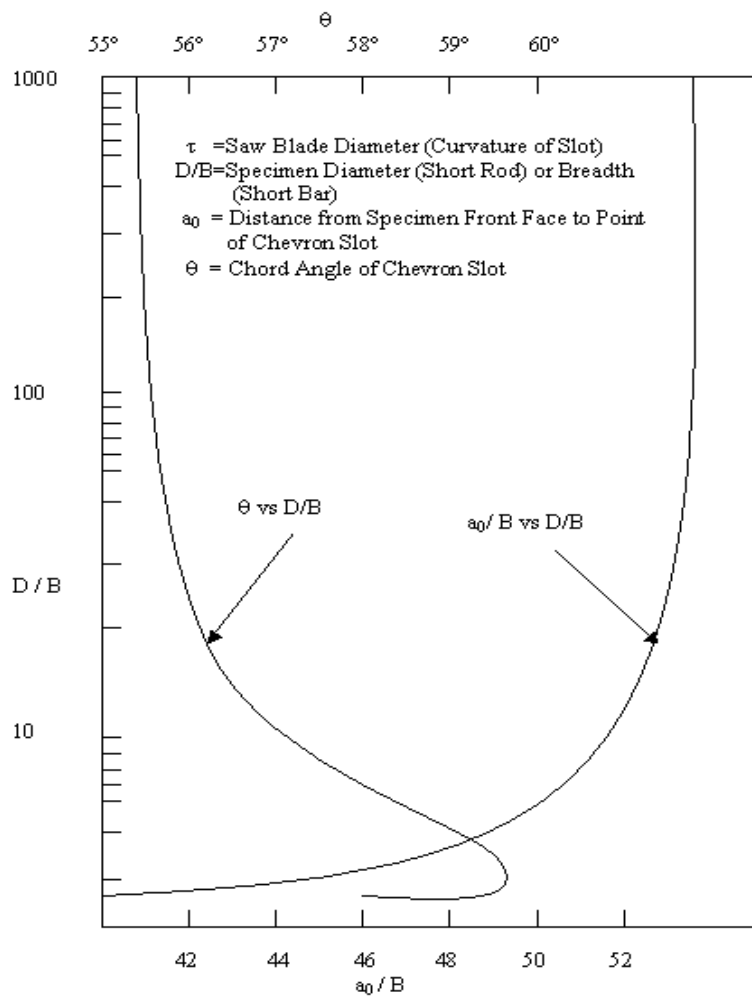


Figure 6.3: The equivalence for curved chevron slots

The configurations of correction factors were calculated because it is regarding how sensitive the test results were to variations in a_0 , 0 , W . The test results can be corrected by multiplying the results by C_c , this only works for specimens, which are out of tolerance by three times the tolerance limits.

6.5 Short Bar Test Description

With the short bar test, it is not only the specimen geometry and preparation important in fracture toughness results, but the testing procedure must also be controlled to obtain valid fracture toughness result.

The short bars tests involve an opening load being applied near the mouth of the specimen, causing a crack to initiate at the point of the chevron slot. Ideally, the opening load should be less than the load that will be required to further advance the crack.

A continually increasing load must be supplied until the crack length reaches the critical crack length, a_c , beyond a_c , the load should decrease, shown in Figure 6.4.

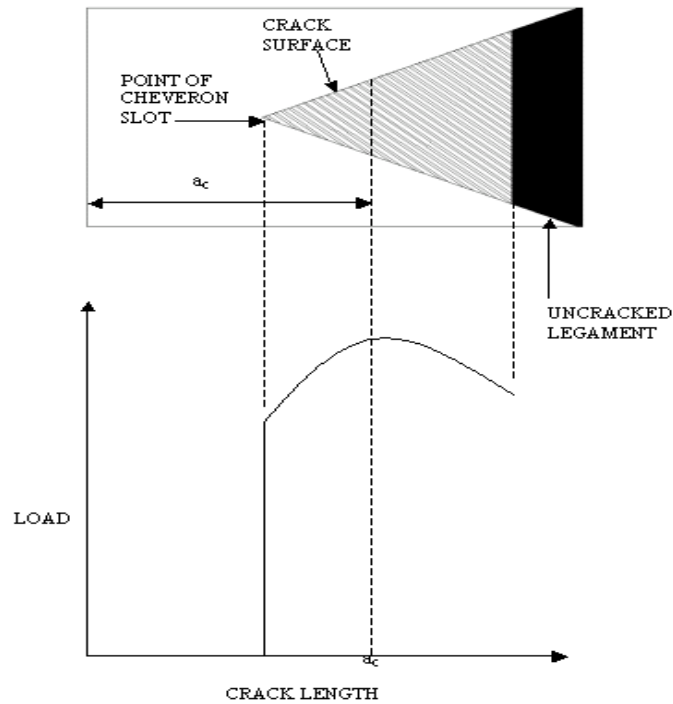


Figure 6.4 Variation of load versus crack length

The equation for fracture toughness in a short bar test can be derived from basic fracture mechanics using the assumptions of linear elastic fracture mechanics (LEFM). K_{IC} was determined with the compact specimens using 5% secant method and the K-calibration according to ASTM E-399-78. K was calculated from the maximum load applied and the crack length including the stable crack extension.

K_{ICSB} of the short bar and K_{ICSR} of the short rod specimens were calculated from the maximum load using compliance calibration:

$$K_{ICSB} = \frac{(F_{max} Y_m^*)}{B\sqrt{W}} \quad (\text{Eqn 6.1})$$

With

$$Y_m^* = \{- 0.36 + 5.48\omega + 0.08\omega^2 + (30.65 - 27.49\omega + 7.46\omega) \alpha_0$$

$$+ (65.90 + 18.44\omega - 9.76\omega)\alpha_0^2 \left\{ \frac{\alpha_1 - \alpha_0}{1 - \alpha_0} \right\}^{\frac{1}{2}} \quad (\text{Eqn 6.2})$$

$$K_{\text{ICSR}} = \frac{(F_{\text{max}} Y_m^*)}{B\sqrt{D}} \quad (\text{Eqn 6.3})$$

With

$$\begin{aligned} Y_m^* = & \left\{ 19.98 - 9.54 \frac{W}{D} + 6.8 \left(\frac{W}{D} \right)^2 \right. \\ & + \left[-118.7 + 125.1 \frac{W}{D} - 22.08 \left(\frac{W}{D} \right)^2 \right] \alpha_0 \\ & \left. + \left[379.4 - 363.3 \frac{W}{D} + 84.4 \left(\frac{W}{D} \right)^2 \right] \alpha_0^2 \right\} \left\{ \frac{\alpha_1 - \alpha_0}{1 - \alpha_0} \right\}^{\frac{1}{2}} \end{aligned} \quad (\text{Eqn 6.4})$$

Where, W = Width, H = Height, D = Diameter, B = Breadth

$$\omega = \frac{W}{H}, \alpha_0 = \frac{a_0}{W}, \alpha_1 = \frac{a_1}{W}$$

Chapter 7

Experiment Method

7.1 Specimen Preparation

Initially, the dimensions of specimen are accordingly modified to the geometrical requirements for the standard ISRM short rod or bar test, where the length to diameter (short rod) or breadth (short bar) ratio, $\frac{L}{D} = 1.45$; the range of diameters can only be varied from test to test in the range of 46 – 92 mm for the tensile test. The choice of scale is significant for the convenience of fabricating the features of the moulds. The dimensions of the short bar specimens used was attached in **Appendix**.

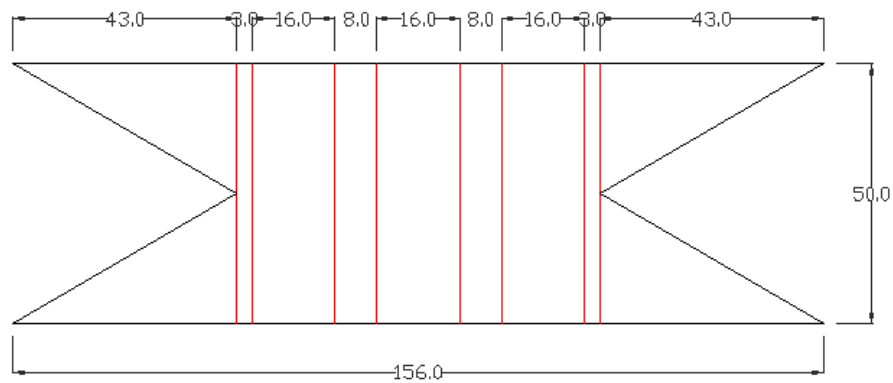
7.2 The Process of Building up the Mould

There are several criteria in preparing the moulds.

- How to recycle the moulds.
- How much will the vinyl ester specimen shrinkage be.

Therefore one has to improve the mould. The following procedures were carried out in fabricating the moulds:

1. Draft the part and base of the moulds.
2. Converted the draft into computer-aided drawing by using the Auto-CAD 2002 LT. as shown in the figure 7.1



Figures 7.1: The Auto-CAD drawing for the triangle part of the mould

3. The manila folders were cut according to the figure 7.1.
4. The manila folder being cut was folded and pasted around the plastic mould with cellophane tape which is shown in figure 7.2.
5. The manila folder part was pasted to the base.
6. The internal view of the complete mould is shown in the figure 7.3

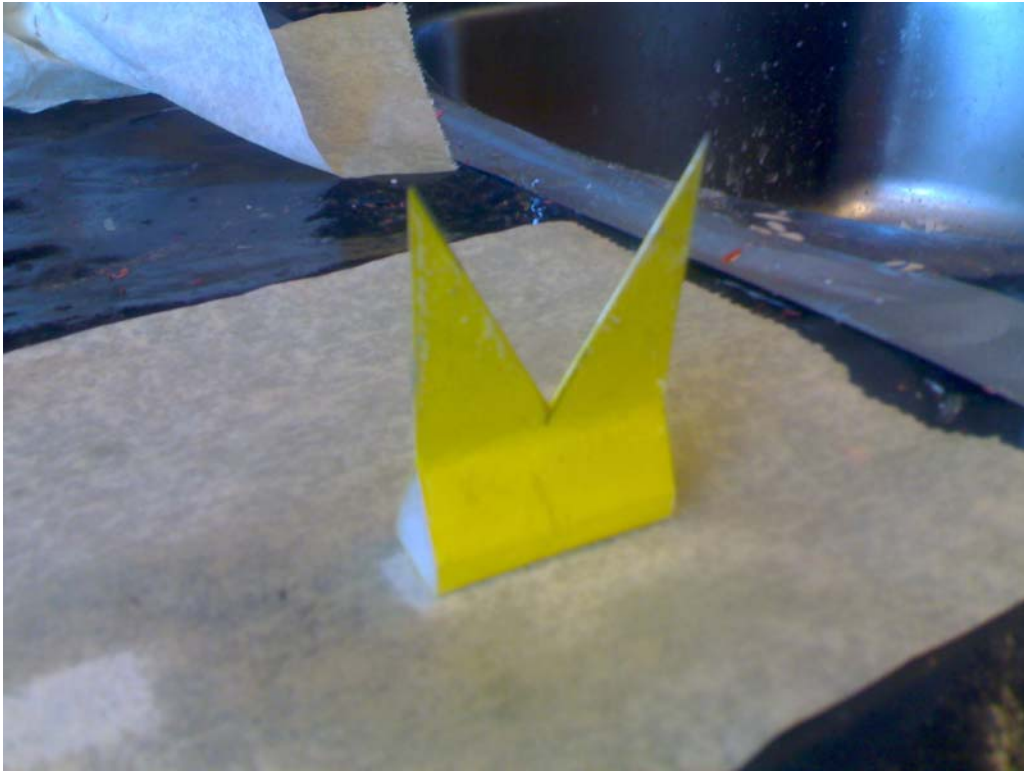


Figure 7.2: The triangle mould for making the slot and important features of short bar specimen

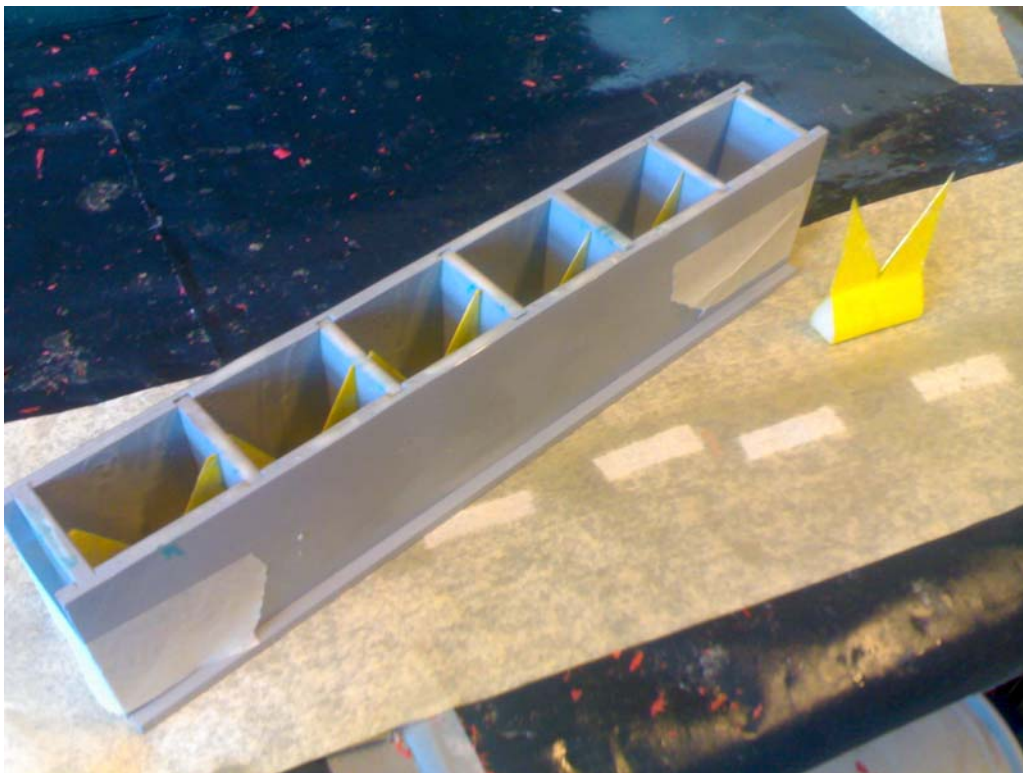


Figure 7.3: The internal view of short bar specimen mould

In this study, thanks have to be given to Mr. Robert Davey who designed the mould and shared it with me.

Steps in preparing the mould for casting:

1. First of all, clean up the surface of the mould nicely and fix it up as shown in figure 7.4.

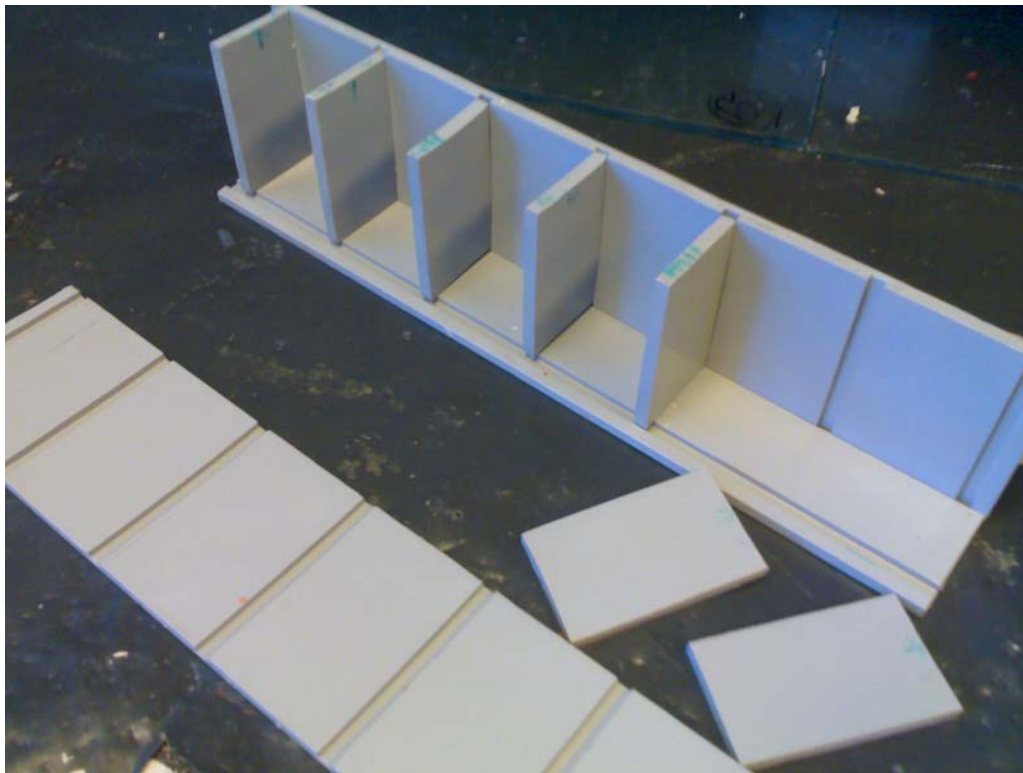


Figure 7.4: Explore view of mould

2. Spray canola oil (cooking use) on the inner surface of mould figure 7.5(a) & figure 7.5(b). For pasting the triangle part, some normal glue with low bonding strength was used.

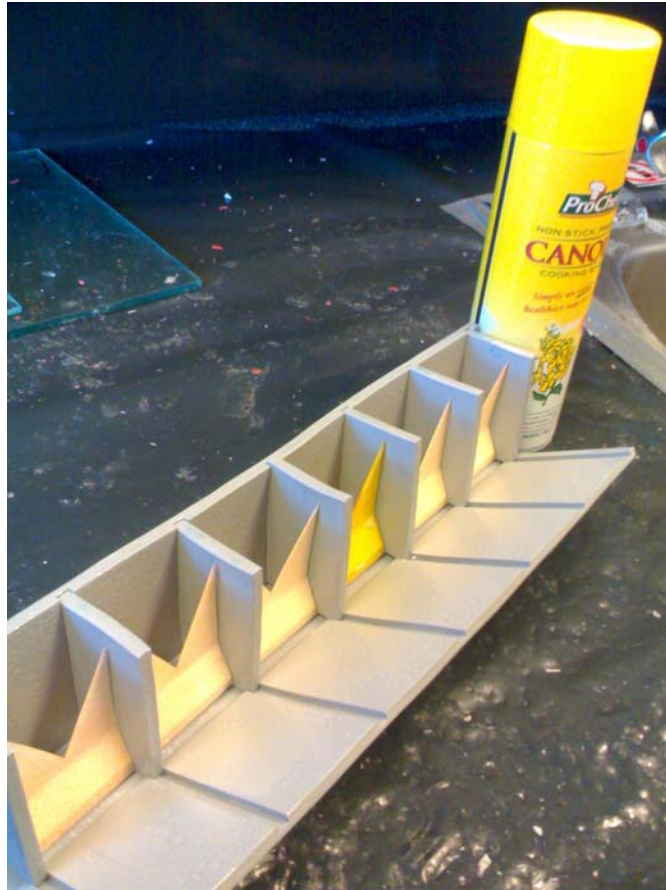


Figure 7.5(a)

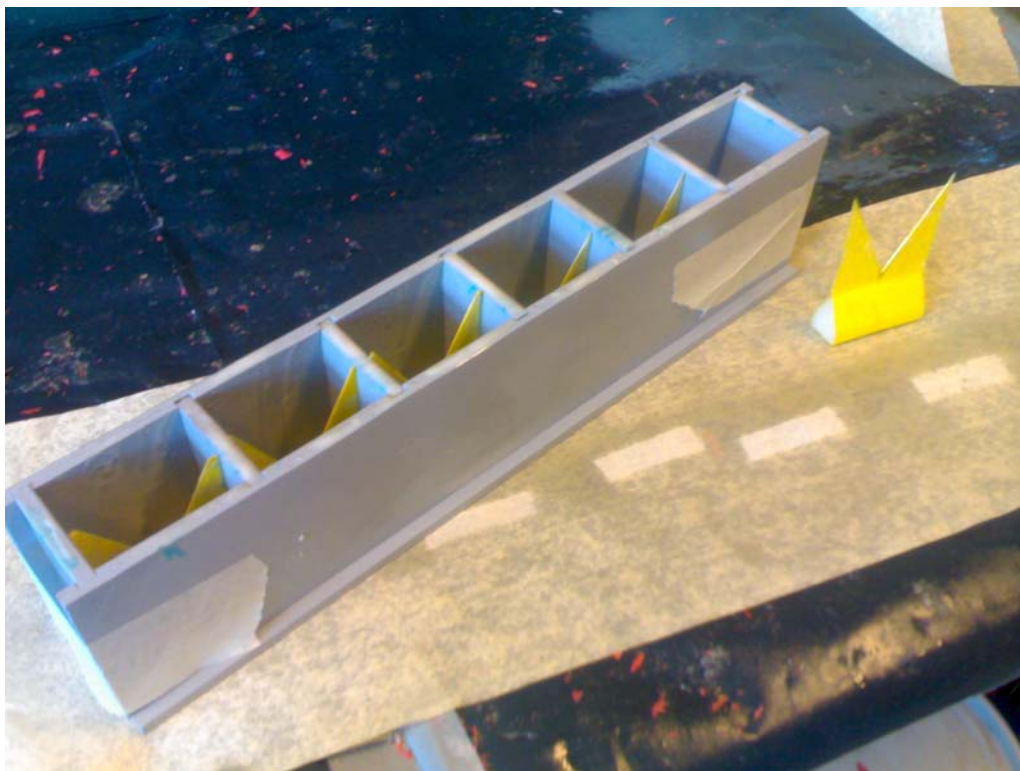


Figure 7.5(b)

7.3 Material Preparation

This is the method for prepare the specimens.

1. Determine the amount of vinyl ester needed and the percentage by weight of fly ash in vinyl ester.
2. Calculate the amount of fly ash, accelerator and resins needed by using the ratio shown in table 7.1.

Table 7.1: Weight of materials required to make 750ml of VE/FLYASH (33%)

Parameters	Resin	Accelerator(MEKP)	Fly ash	Composite
Relative density	1.05	1.0	0.7	---
Percentage by volume	56.5	1	42.5	100
Percentage by weight	67	---	33	100
Weight for 750g of composite	492.45(g)	10.05(g) or 8.55(ml)	247.5(g)	---

3. Pour the amount of each material according to the ratio into different containers.
The weight of fly ash and resins were weighted using a digital balance.
4. Pour the accelerator from its dispenser into the resins and mixed them thoroughly using a spoon.
5. Pour the fly ash into the mixture of resins and accelerator which is shown in step 4.
The styrene of the vinyl ester and the accelerator were inhalation hazardous, so that they have to be stirred under an exhaust fan

6. Pour the mixture into the mould slowly after stirring for a few minutes.
7. Took the material for microwave exposure and cured under the exhaust fan or leave the material to cure under exhaust fan (for ambient).

7.4 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.

7.5 Microwave Exposure of Composites

7.5.1 The Modified Microwave Oven

The microwave oven was slightly modified for use in this experiment. The main part is to direct the styrene gas evolved to exhaust rapidly.

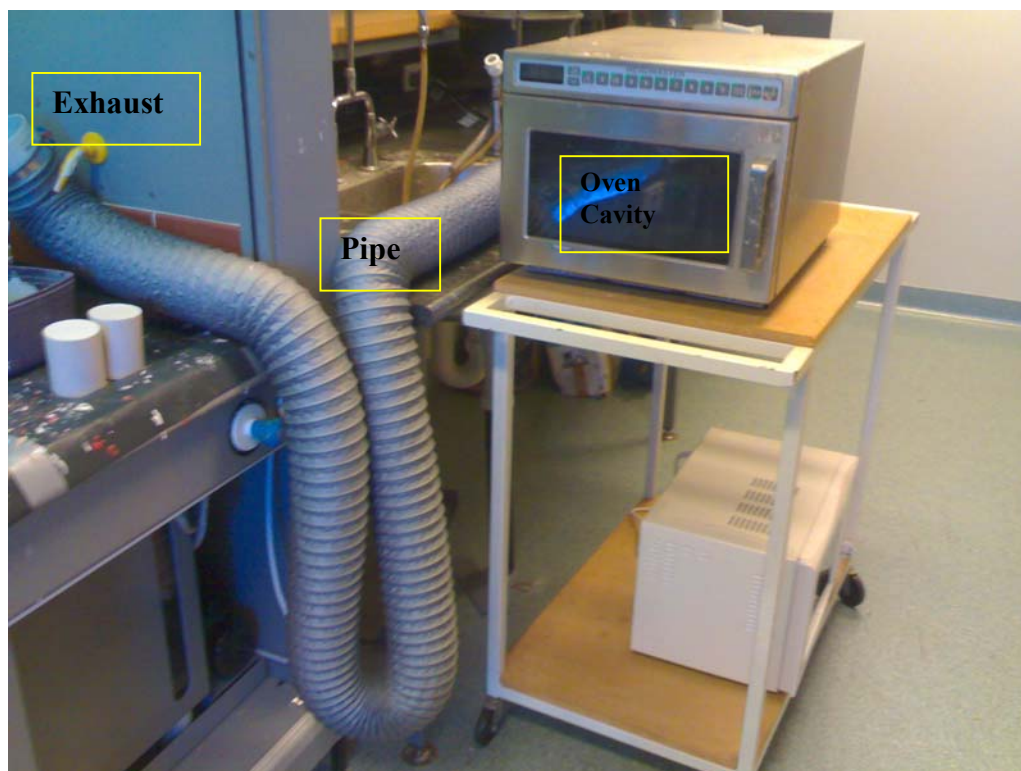


Figure 7.6: The modified oven and its peripherals (Ku, H S 2002b)

7.5.2 Type of the Microwave Exposure Time

There are 12 types of conditions for the microwave exposure of the specimens. They are 180-Watt power with exposure of 60seconds, 70 seconds and 80 seconds, and 360-Watt power with exposure of 60seconds, 70 seconds and 80 seconds, and 540-Watt power with exposure of 15 seconds, 20 seconds and 25 seconds, and 720-Watt power with exposure of 15 seconds, 20 seconds and 25 seconds.

Table 7.2 shows the changes in volume and other parameters of vinyl ester composites after exposure to the different power levels and exposure times.

Table 7.2: Volume shrinkage and other parameters for 400 ml of VE/FLYASH (33%) exposed to 180-W microwaves at different duration

Microwave exposure time (seconds)	0	55	60	65
Oven cavity Temperature (°C)	16	24	22	22
Relative humidity (%)	52	48	46	47
Temperature after microwave exposure	NA	30	32	34
Original volume (ml)	400	400	400	400
Final volume (ml)	363.64	387.69	389.26	390.81
Volume shrinkage (%)	9.09	3.00	2.70	2.30
Volume at maximum temperature (ml)	382.2	392.2	393.9	394.1
Time to reach gel time (minutes)	62.5	7.0	7.0	6.0
Maximum temperature	139	138	148	150
Time to reach maximum temperature (minutes)	67.5	10.0	10.0	9.0

Chapter 8

Test Rig and Apparatus

8.1 Test Rig Requirements

To obtain the accurate results, the test rig configuration for the MTS 810 Material Testing Systems was modified. The requirements are shown hereinafter:

- Sufficient tensile force provided by the loading mechanism.
- The grippers which are use will not deform during the testing.
- Accurate alignment of the grippers.
- The grippers are allowed for sufficient control the load line position.
- The plastic deformation of specimen apex caused by the grippers should be minimized.

8.2 Test Rig Available

For the short rod or bar test method, it has several test rig purposely designed for them. Some of the test rigs are combined with other advanced computer systems in order to produce the more accurately and consistently results. However, this test rig should be chosen by considering to its availability, functionality and suitability.

8.3 MTS 810 Material Testing Systems

The MTS 810 material testing systems (Figure 8.1), it especially design for small specimen, was used in this study for the short bar tensile testing. The fracture toughness of the short bar specimen was tested by an opening tensile load applied at the opening of the specimen by grippers as shown in Figure 8.2. And finally, the MTS 810 material testing systems will print out the detailed information results. This will show in **appendix**.



Figure 8.1: MTS 810 Material Testing Systems

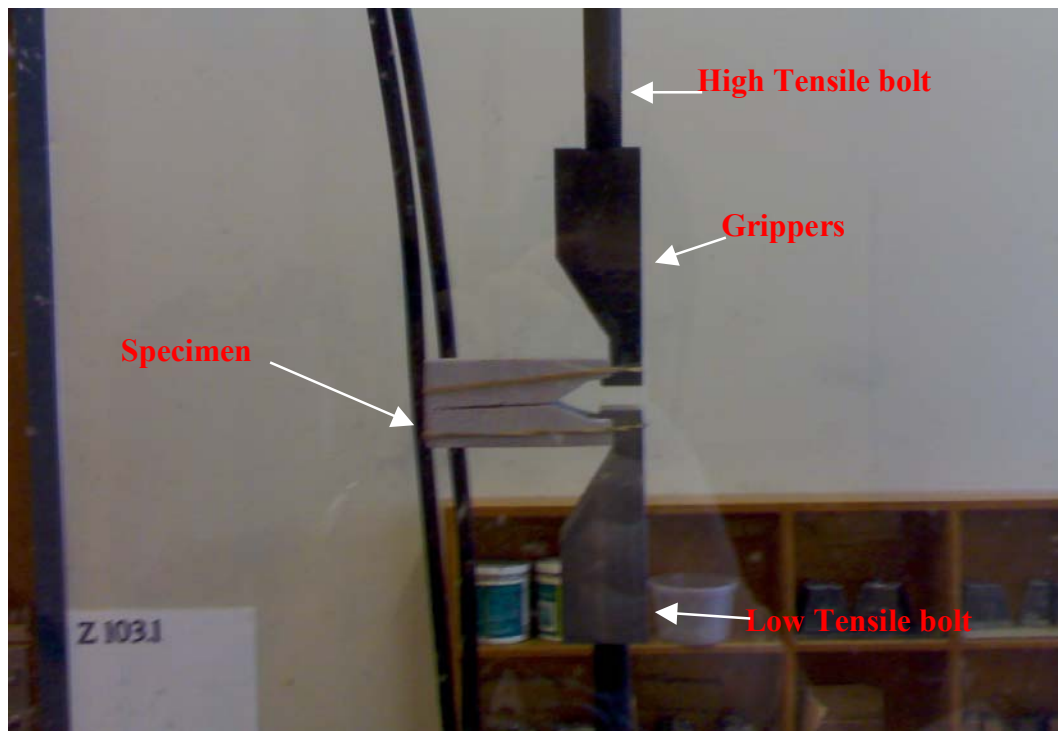


Figure 8.2: Test rig with specimen in position

8.4 The Advantages of MTS 810 Material Testing Systems

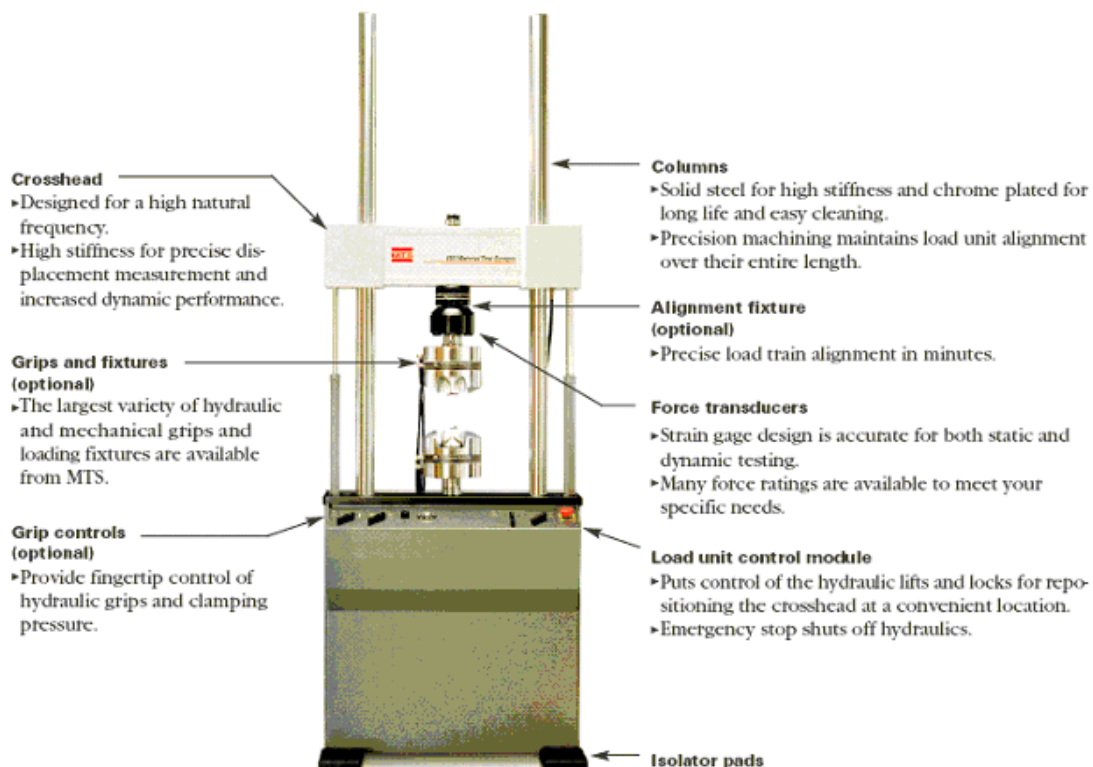


Figure 8.3: The systems of MTS 810 Material Testing Systems (**MTS 810 FlexTest™** Material Testing Systems)

Here are some of the advantages over then other test rig why MTS 810 machine was used; its advantages are hereinafter:

- **User-friendly:** This testing system is incorporated with the TestStart IIs control system. This system consisted of three major parts: (1) TestStart system software, (2) digital controller, and (3) remote station control panel as shown in Figure 8.6. The control system is able to produce the result in the form of graph or table with the ready programmed software and it also include some of the important statistical variable such as mean and standard deviations.

- **Accuracy:** Its superior axial and lateral stiffness are achieved through an integral actuator design, stiff, but low mass crosshead, and specially force transducer.
- **Flexibility:** By simply changing or adjusting its grips and fixtures. This testing machine can also be used for many types of testing such as tensile testing, fatigue life studies, Asphalt or soil testing and etc

8.5 Gripper Design

Due to MTS 810 machine is suitable for tensile testing involving smaller load and can provides more accurate results, the MTS 810 Material Testing System was used in this project. Small modification was made to the grippers for enable them to be placed to the machine. The grippers were hold by high tensile bolts as shown in Figure 8.2. There are some alternatives to design the grippers as illustrated in the Figure 8.4.

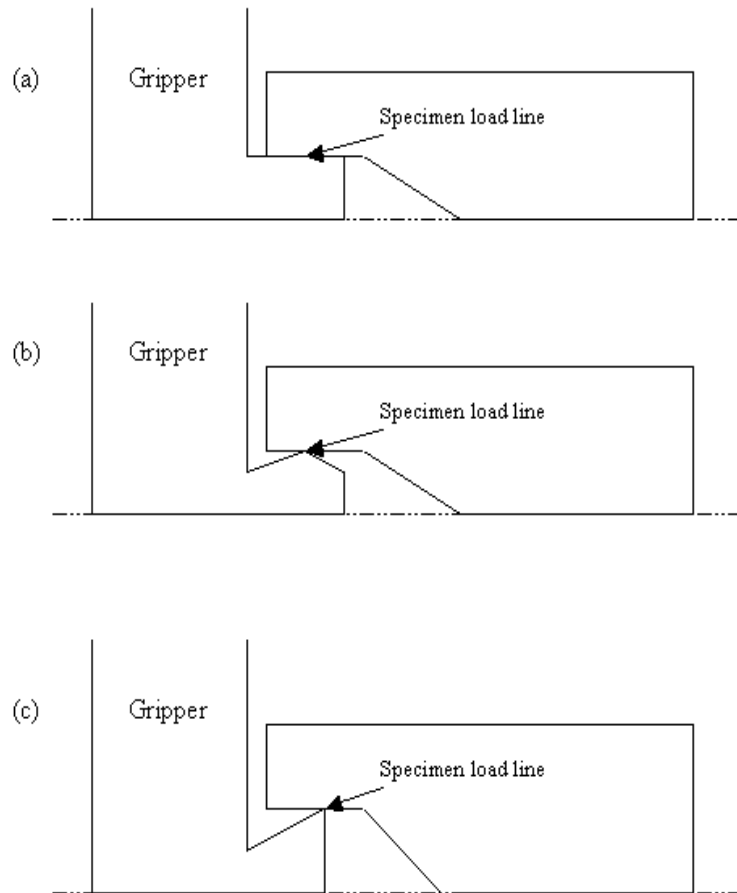


Figure 8.4 Grippers designs

The gripper shown in Figure 8.4(c) was used in this project because the load line positioning was good and it was easier to manufacture. However, the stress concentration occurred in the sharp corner so the grippers failed before the load reached 25 KN and the foreside of the specimen was opened. After modification, Phelan (1990) designed rounded profile grippers, and the profile from previous sample was ground out as illustrated in Figure 8.5.

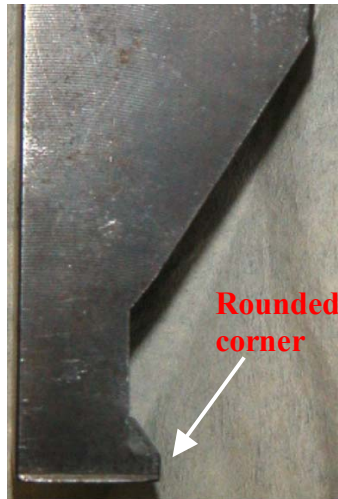


Figure 8.5: The rounded profile of the grippers



Figure 8.6: MTS 810 Material testing System

Chapter 9

Results and Discussions

9.1 Introduction

The results obtained from the experimental work will be present in this chapter. The chapter will be divided into sections; (1) related to e tensile testing results from the experimental work, and (2) discuss and determine the fracture toughness; then compare the results of the fracture toughness and other parameters for VE cured under different conditions. In the final section, specimens cured in ambient conditions will be compared with those cured with different microwave conditions having power levels of 180 Watts, 360 Watts, 540 Watts and 720 Watts.

9.2 MTS-810 tensile testing machine

This MTS 810 tensile testing was used to calculate the fracture toughness. During the test, a constantly increasing force will apply at the top edge of the specimen until the load reach maximum and crack occurred. A crack forms at the tip of the notch would proceed down the specimen along the notched path. By knowing the location of the crack at the maximum load under linear elastic fracture condition and the height of the crack that corresponds to the maximum constant load force, the fracture toughness of the composite can be calculated. Specimens after testing are illustrated in Figure 9.1.



Figure 9.1: The fractured specimens and show the part for making the chevron slot

The curing conditions in this project were divided into five major groups. First condition is cured under ambient conditions for 24 hours; second condition is cured under 180 Watts microwave conditions, third condition is cured under 360 Watts microwave conditions, fourth condition is cured under 540 Watts microwave conditions and fifth condition is cured under 720 Watts microwave conditions. For 180 Watts and 360 Watts, exposure times were 60 seconds, 70 seconds and 80 seconds; with a power of 540 Watts and 720 Watts exposure times were 15 seconds, 20 seconds and 25 seconds. The result obtained by MTS-810 for all curing conditions are shown in **Appendix C**. The tensile testing results generated by MTS-810 machine included the peak load, failure load, break load and the break load elongation of the specimen. The tensile test result obtained from MTS 810 is illustrated in Figure 9.2.

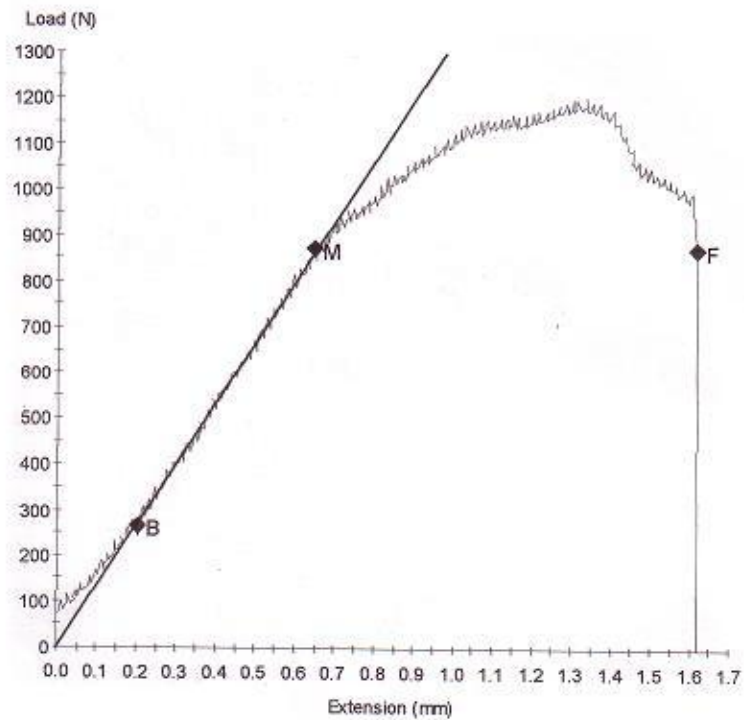
The three phases listed decide whether the tensile test specimen has been successful or failed.

- 1) The rate of extension was set at 1 mm per minute, which results in the increased load to initiate the specimen crack
- 2) The elongation behavior will be show when the specimen passes its peak load.
- 3) The crack will be elongated and will reach point 4 of the four critical points shown in Figure 9.3 and the specimen will show brittle behavior in this area.

In some of the samples, for example the 720 Watts power level with 25 seconds of exposure time (Figure 9.4), the specimen crack early. This happen, due to the cause of a bad quality casting when stick the chevron slot. Barker (1980) defined this effect as crack curve jumping as illustrated in Figure 9.4. Therefore this specimen is not fully fractured but accruing the crack at the side of it.

20-Sep-06

Sample ID: chan-720-15n-2.mss
Specimen Number: 2
Tagged: False

**Specimen Results:**

Name	Value	Units
Thickness	50.000	mm
Width	23.000	mm
Area	1150	mm ²
Peak Load	1205	N
Peak Stress	1.05	MPa
Break Load	871	N
Break Stress	0.76	MPa
Elongation At Break	1.614	mm
Stress At Offset Yield	0.891	MPa
Load At Offset Yield	1024.580	N

Figure9.2: The change of load versus crack length of a sample cured under microwave condition (720 Watt power level and 15-second exposure time)

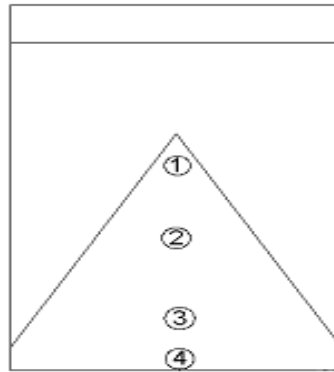


Figure 9.3: Five critical points for the fractures surface to be analyzed.

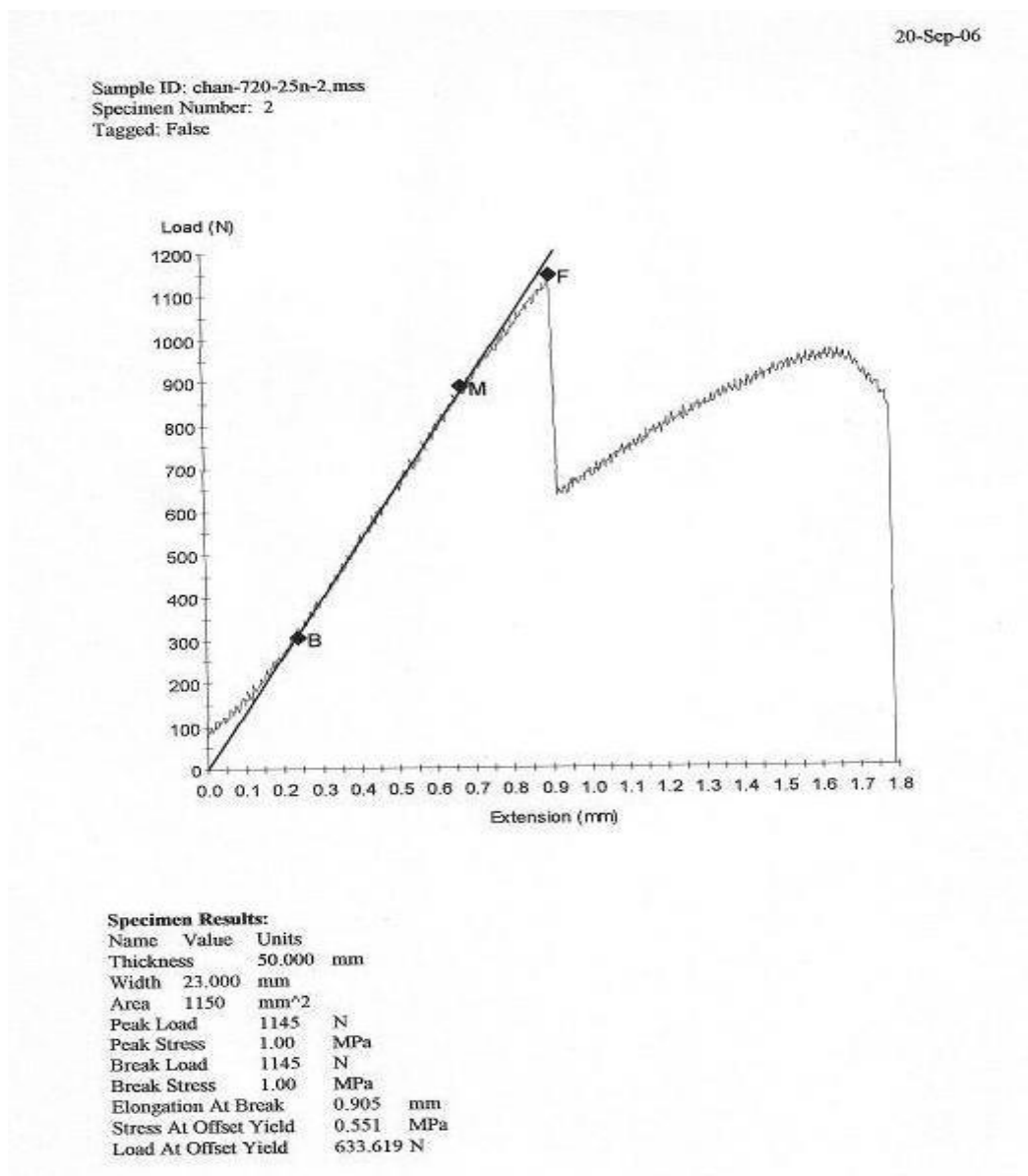


Figure 9.4: The change of load versus crack length of a sample cured under microwave condition (720 Watt power level and 25-second exposure time)

9.3 Fracture Toughness Determinations and Discussion

The purpose of the fracture toughness is to determine the severity of a pre-existing defect in term of its tendency to initiate a fracture. The critical fracture toughness value K_{IC} was determined with the compact specimens using 5 % secant method. To determine the fracture toughness (K_{IC}) of a material with the standard test methods, the K-calibration was made according to the ASTM E399-78.

For the evaluation of K-calibration, K was calculated from the maximum load applied from the tensile testing and the crack length which is shown as Figure 9.5.

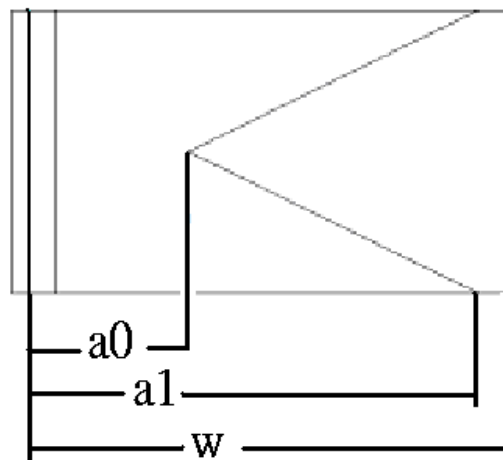


Figure 9.5: Cross-section dimension of short bar specimen

9.3.1 The formulas and methods for calculating the fracture toughness

Specimen cured in 720 Watts with 15 seconds exposure time was taken as an example.

The formulas and methods to obtain the fracture toughness were illustrated as follows:

The formulas used to calculate the fracture toughness were Equations 9.1 through 9.4.

$$K_{ICSB} = \frac{(F_{\max} Y_m^*)}{B\sqrt{W}} \quad (\text{Eqn. 9.1})$$

where F_{\max} = Peak load

Y_m^* is the compliance calibration according to ASTM E-399-78 standard.

$$\omega = \frac{W}{H} = \frac{70}{37.5} = 1.867 \quad (\text{Eqn. 9.2})$$

$$\alpha_0 = \frac{a_0}{W} = \frac{25}{70} = 0.357 \quad (\text{Eqn. 9.3})$$

$$\alpha_1 = \frac{a_1}{W} = \frac{65}{70} = 0.929 \quad (\text{Eqn. 9.4})$$

The calculations for it were made by Munz (1981).

$$Y_m^* = \{-0.36 + 5.48\omega + 0.08\omega^2 + (30.65 - 27.49\omega + 7.46\omega)\alpha_0$$

$$+ (65.90 + 18.44\omega - 9.76\omega)\alpha_0^2\} \left\{ \frac{\alpha_1 - \alpha_0}{1 - \alpha_0} \right\}^{\frac{1}{2}}$$

$$Y_m^* = 17.1718$$

The results of the 720-Watt power level and 15-second cured group (six specimens) were shown in Table 9.1. The mean ($\bar{\mu}$) of fracture toughness is $45.00 \text{ N.mm}^{-3/2}$.

<i>Specimens</i>	<i>Peak load</i> (N)	<i>Elongation at Break</i> (Mm)	<i>Break Load</i> (N)	<i>Fracture Toughness</i> (N.mm ^{-3/2})
1	1076	1.733	603	44.17
2	1205	1.614	871	49.46
3	1133	2.168	780	46.51
4	1023	1.693	746	41.99
5	1090	1.492	726	44.74
6	1050	1.43	810	43.10
Mean	1096	1.689	756	45.00
Standard Deviation	65	0.262	91	2.44

Table 9.1: Test results of 720 Watts and 15-second exposure

Using
$$K_{ICSB} = \frac{(F_{\max} Y_m^*)}{B\sqrt{W}} \quad (\text{Eqn. 9.5})$$

and let $B = 50$ (by design) and $W = 70$ (not $1.5B$);,

From the Table 9.1 mean of Peak Load = $F_{\max} = 1096 \text{ N}$

and $Y_m = 17.1718$

therefore
$$K_{ICSB} = \frac{(1096 \times 17.1718)}{50\sqrt{70}} = 45.00 \text{ (N.mm}^{-3/2}\text{)}$$

9.3.2 Summary Results of Specimens:

There are total thirteen groups of testing in this experiment, those were 180 Watts and 360 Watts with 60 seconds, 70 seconds and 80 seconds exposure time and 540 Watts and 720 Watts with 15 second, 20 second and 25 second exposure time and the specimen which cure in ambient condition. Each group has six specimens. For the mean fracture toughness, we will take the average of all six specimens in the same group. So we can examine that the mean of fracture toughness is become around 42 $N.mm^{-3/2}$ to 48 $N.mm^{-3/2}$. The results as shown as below from Table 9.2 – Table 9.14

1. Test results in ambient conditions

Table 9.2: Test results of ambient condition

<i>Specimens</i>	<i>Peak load</i> (N)	<i>Elongation at Break</i> (Mm)	<i>Break Load</i> (N)	<i>Fracture Toughness</i> ($N.mm^{-3/2}$)
1	1033	1.966	739	42.40
2	1036	2.348	908	42.53
3	1071	2.185	819	43.96
4	1075	1.766	657	44.13
5	1056	1.799	841	43.35
6	1012	1.806	622	41.54
<i>Mean</i>	1047	1.978	764	42.98
<i>Standard Deviation</i>	24	0.239	111	0.92

2. Test results of 180 Watts with 60 seconds

Table 9.3: Test results of 180 Watts with 60 seconds

<i>Specimens</i>	<i>Peak load</i> <i>(N)</i>	<i>Elongation at Break</i> <i>(Mm)</i>	<i>Break Load</i> <i>(N)</i>	<i>Fracture Toughness</i> <i>(N.mm^{-3/2})</i>
1	1074	1.483	952	44.09
2	1028	1.596	748	42.20
3	982	1.351	879	40.31
4	1044	1.847	779	42.85
5	1125	1.934	757	46.18
6	1071	1.902	795	43.96
<i>Mean</i>	1054	1.685	818	43.27
<i>Standard Deviation</i>	48	0.243	81	1.81

3. Test results of 180 Watts with 70 seconds

Table 9.4: Test results of 180 Watts with 70 seconds

<i>Specimens</i>	<i>Peak load</i> <i>(N)</i>	<i>Elongation at Break</i> <i>(Mm)</i>	<i>Break Load</i> <i>(N)</i>	<i>Fracture Toughness</i> <i>(N.mm^{-3/2})</i>
1	1126	1.57	662	46.22
2	1120	1.429	783	45.97
3	1159	1.956	1045	47.58
4	1124	1.624	1017	46.14
5	1124	1.822	773	46.14
6	1143	1.637	824	46.92
<i>Mean</i>	1133	1.673	851	46.49
<i>Standard Deviation</i>	15	0.188	150	0.57

4. Test results of 180 Watts with 80 seconds

Table 9.5: Test results of 180 Watts with 80 seconds

<i>Specimens</i>	<i>Peak load</i>	<i>Elongation at Break</i>	<i>Break Load</i>	<i>Fracture Toughness</i>
	<i>(N)</i>	<i>(Mm)</i>	<i>(N)</i>	<i>(N.mm^{-3/2})</i>
1	1196	1.536	1118	49.09
2	1080	1.749	953	44.33
3	1162	1.954	875	47.70
4	1040	1.791	764	42.69
5	1067	1.668	628	43.80
6	1069	2.073	551	43.88
<i>Mean</i>	1102	1.795	815	45.25
<i>Standard Deviation</i>	61	0.194	211	2.31

5. Test results of 360 Watt power with 60 seconds

Table 9.6: Test results of 360 Watts with 60 seconds

<i>Specimens</i>	<i>Peak load</i>	<i>Elongation at Break</i>	<i>Break Load</i>	<i>Fracture Toughness</i>
	<i>(N)</i>	<i>(Mm)</i>	<i>(N)</i>	<i>(N.mm^{-3/2})</i>
1	1191	1.627	868	48.89
2	1093	1.645	845	44.87
3	1293	1.966	1138	53.08
4	1126	1.92	986	46.22
5	1159	2.136	1068	47.58
6	1102	2.147	842	45.24
<i>Mean</i>	1161	1.907	958	47.64
<i>Standard Deviation</i>	74	0.228	126	2.79

6. Test results of 360 Watt power with 70 seconds

Table 9.7: Test results of 360 Watts with 70 seconds

<i>Specimens</i>	<i>Peak load</i> <i>(N)</i>	<i>Elongation at Break</i> <i>(Mm)</i>	<i>Break Load</i> <i>(N)</i>	<i>Fracture Toughness</i> <i>(N.mm^{-3/2})</i>
1	1111	1.977	992	45.60
2	1081	1.712	971	44.37
3	1098	1.747	626	45.07
4	1014	1.674	786	41.62
5	1088	2.234	747	44.66
6	1097	2.059	942	45.03
Mean	1082	1.9	844	44.39
Standard Deviation	35	0.225	147	1.30

7. Test results of 360 Watt power with 80 seconds

Table 9.8: Test results of 360 Watts with 80 seconds

<i>Specimens</i>	<i>Peak load</i> <i>(N)</i>	<i>Elongation at Break</i> <i>(Mm)</i>	<i>Break Load</i> <i>(N)</i>	<i>Fracture Toughness</i> <i>(N.mm^{-3/2})</i>
1	1079	1.985	928	44.29
2	1042	1.852	796	42.77
3	1020	2.126	787	41.87
4	1046	2.041	826	42.94
5	1141	1.949	963	46.84
6	1024	1.965	908	42.03
Mean	1059	1.986	868	43.46
Standard Deviation	45	0.092	74	1.70

8. Test results of 540 Watt power with 15 seconds

Table 9.9: Test results of 540 Watts with 15 seconds

<i>Specimens</i>	<i>Peak load</i> <i>(N)</i>	<i>Elongation at Break</i> <i>(Mm)</i>	<i>Break Load</i> <i>(N)</i>	<i>Fracture Toughness</i> <i>(N.mm^{-3/2})</i>
1	1215	1.4	1194	49.87
2	1080	1.515	771	44.33
3	990	1.498	601	40.64
4	1061	1.926	892	43.55
5	1097	1.544	874	45.03
<i>Mean</i>	1089	1.576	866	44.69
<i>Standard Deviation</i>	82	0.202	216	2.99

9. Test results of 540 Watt power with 20 seconds

Table 9.10: Test results of 540 Watts with 20 seconds

<i>Specimens</i>	<i>Peak load</i> <i>(N)</i>	<i>Elongation at Break</i> <i>(Mm)</i>	<i>Break Load</i> <i>(N)</i>	<i>Fracture Toughness</i> <i>(N.mm^{-3/2})</i>
1	1111	1.502	765	45.60
2	1053	1.783	801	43.22
3	1060	1.669	916	43.51
4	1212	1.843	830	49.75
5	1164	1.78	989	47.78
6	1182	1.962	898	48.52
<i>Mean</i>	1130	1.756	866	46.40
<i>Standard Deviation</i>	66	0.157	83	2.47

10. Test results of 540 Watt power with 25 seconds

Table 9.11: Test results of 540 Watts with 25 seconds

<i>Specimens</i>	<i>Peak load</i> <i>(N)</i>	<i>Elongation at Break</i> <i>(Mm)</i>	<i>Break Load</i> <i>(N)</i>	<i>Fracture Toughness</i> <i>(N.mm^{-3/2})</i>
1	1100	1.919	692	45.15
2	1023	1.506	566	41.99
3	1020	1.515	659	41.87
4	1039	1.5	727	42.65
5	1091	1.521	730	44.78
6	1121	1.767	1024	46.02
<i>Mean</i>	1066	1.621	733	43.74
<i>Standard Deviation</i>	43	0.178	155	1.63

11. Test results of 720 Watt power with 15 seconds

Table 9.12: Test results of 720 Watts with 15 seconds

<i>Specimens</i>	<i>Peak load</i> <i>(N)</i>	<i>Elongation at Break</i> <i>(Mm)</i>	<i>Break Load</i> <i>(N)</i>	<i>Fracture Toughness</i> <i>(N.mm^{-3/2})</i>
1	1076	1.733	603	44.17
2	1205	1.614	871	49.46
3	1133	2.168	780	46.51
4	1023	1.693	746	41.99
5	1090	1.492	726	44.74
6	1050	1.43	810	43.10
<i>Mean</i>	1096	1.689	756	45.00
<i>Standard Deviation</i>	65	0.262	91	2.44

12. Test results of 720 Watt power with 20 seconds

Table 9.13: Test results of 720 Watts with 20 seconds

<i>Specimens</i>	<i>Peak load</i>	<i>Elongation at Break</i>	<i>Break Load</i>	<i>Fracture Toughness</i>
	<i>(N)</i>	<i>(Mm)</i>	<i>(N)</i>	<i>(N.mm^{-3/2})</i>
1	1111	1.833	989	45.60
2	1100	1.76	1005	45.15
3	1173	1.926	1044	48.15
4	1104	1.94	329	45.32
5	1128	1.98	1003	46.30
6	1124	1.793	1077	46.14
<i>Mean</i>	1123	1.872	908	46.11
<i>Standard Deviation</i>	27	0.089	285	1.00

13. Test results of 720 Watt power with 25 seconds

Table 9.14: Test results of 720 Watts with 25 seconds

<i>Specimens</i>	<i>Peak load</i>	<i>Elongation at Break</i>	<i>Break Load</i>	<i>Fracture Toughness</i>
	<i>(N)</i>	<i>(Mm)</i>	<i>(N)</i>	<i>(N.mm^{-3/2})</i>
1	1125	2.014	1052	46.18
2	1145	0.905	1145	47.00
3	1043	1.669	617	42.81
4	1077	1.699	763	44.21
5	1103	1.588	671	45.28
6	1153	1.589	927	47.33
<i>Mean</i>	1108	1.577	863	45.47
<i>Standard Deviation</i>	42	0.365	213	1.58

The summary of fracture toughness values of the composites cured under different conditions are summarized in Table 9.15. The values of fracture toughness of samples cured under ambient conditions, $42.9846 \text{ N.mm}^{-3/2}$ were converted to 100 % for ease of comparison; other values were also similarly converted to percentage using $42.9846 \text{ N.mm}^{-3/2}$ as base.

Table 9.15: Result of the fracture toughness and other parameters for VE/FLYASH (33%) cured under different condition

Condition	Ambient			180 Watt			360 Watt			540 Watt			720 Watt		
	60s	70s	80s	60s	70s	80s	60s	70s	80s	15s	20s	25s	15s	20s	25s
Time															
Elongation At Break (mm)	1.978	1.685	1.673	1.795	1.907	1.9	1.986	1.576	1.756	1.621	1.689	1.872	1.577		
Peak Load (N)	1047	1054	1133	1102	1161	1082	1059	1089	1130	1066	1096	1123	1108		
Break Load (N)	764	818	851	815	958	844	868	866	866	733	756	908	863		
Fracture Toughness (N.mm ^{-3/2})	42.9846	43.2651	46.4943	45.2491	47.6436	44.3939	43.4567	44.6854	46.3985	43.7440	44.9960	46.1111	45.4681		
Relative fracture toughness	100%	101%	108%	105%	111%	103%	101%	104%	108%	102%	105%	107%	106%		
Standard Deviation	0.92	1.81	0.57	2.31	2.79	1.30	1.70	2.99	2.47	1.63	2.44	1.00	1.58		

From Table 9.15, we can find that the fracture toughness of the sample treated with 180 Watts with exposures time of 60, 70 and 80 seconds is higher than that of the ambient cured samples from 1- 8 %; With microwave parameters of 360 Watts with exposures time of 60 – 80 seconds, specimens cured under microwave are higher than specimen which cured under ambient condition from 1 - 11%. With microwave parameters of 540 Watts and shorter duration of exposures of 15, 20 and 25 seconds, the fracture toughness is higher than those cured under ambient conditions from 2 – 8 %; for 720 Watts and same exposures time of 15, 20 and 25 seconds, the fracture toughness is higher than those cured under ambient conditions from 5 – 7 %.

All values of fracture toughness of VE/FLYASH (33%) cured under microwave conditions are within or above 5 percent upper marker of those cured under ambient conditions. This means microwave irradiation reduces the shrinkage of the composite but at the same time improves its fracture toughness. The results are shown in Figure 9.6 and Figure 9.7.

The results have been published by Dr. Harry Ku be entitled “An Evaluation of Fracture Toughness of Vinyl Ester Composites Cured Under Microwave Condition” in Journal of Materials Engineering and Performance.

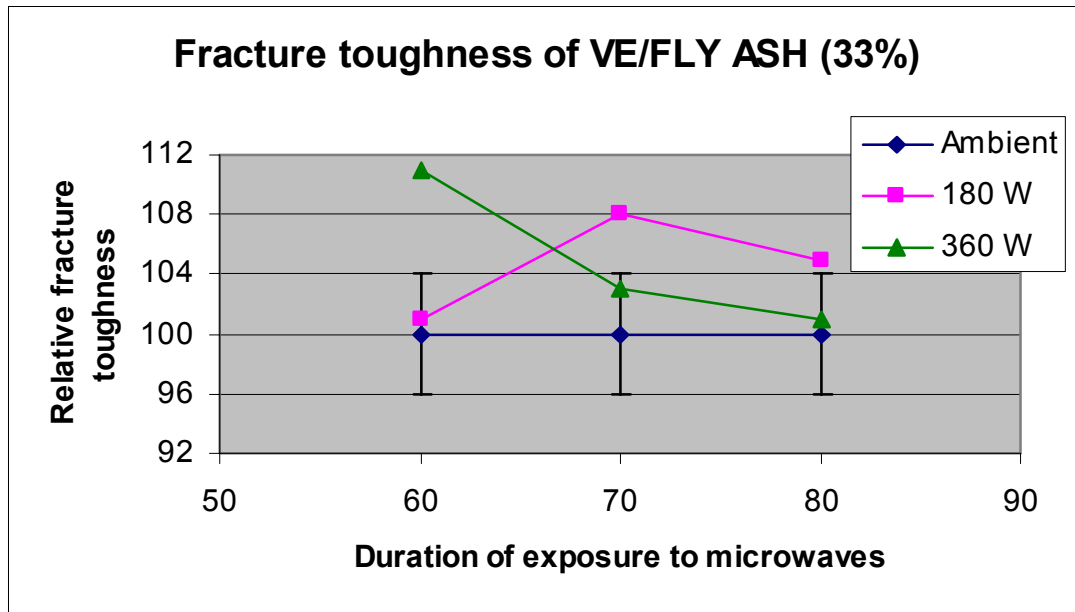


Figure 9.6: Fracture toughness of VE/FLYASH (33%) cured under 180 Watts and 360 Watts of microwave power

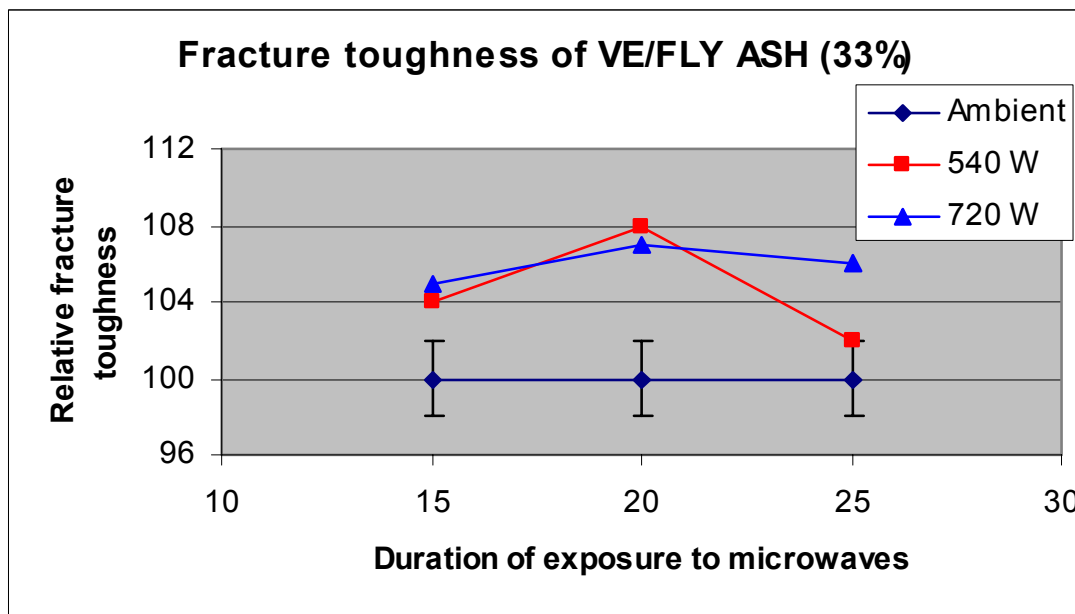


Figure 9.7: Fracture toughness of VE/FLYASH (33%) cured under 540 Watts and 720 Watts of microwave power

Chapter 10

Conclusion and Recommendations

10.1 Conclusion

In this study, the fracture toughness values obtained are higher than those obtained by previous study. In general, the higher fracture toughness values obtained in this study is might because of the better material used to make the mould and improvement in design. In previous study, cardboard was used to make the moulds and this allows the resin to penetrate into the cardboard and seep through the gaps between the adjoining areas of the cardboard; the resulting composite would then have a higher percentage by weight of fly ash and hence they are less tough. When the samples were cured under microwave conditions, the situation became worse as the initial expansion of the composite due to microwave irradiation forced more resin to penetrate into the cardboard and seep through the adjoining gaps. However in this study, this did not happen as the moulds were made of plastic, which may deform after several microwave processing and a new mould has to be machined.

However, all the study prove that the exposure of the samples by microwave irradiation did reduce the shrinkage of the vinyl ester composite but at the same time the toughness of the material was retained. Provided a suitable combination of power level and duration of exposure were selected in the treatment. For those higher power levels like 540 Watts and 720 Watts, much shorter duration of exposure is required.

10.2 Recommendations

The results can be improved throughout the following efforts:

- Changing a new mould after several testing to prevent the deformation of mould after several microwave processing. Maintain the specimen size to get more accurate results.
- Improve material properties of the triangle mould which and recyclable use and structure of specimen: By using a better material especially the chevron slot part should be made of harder material so that the part would not be bend aside.
- Use Latin Square to analyse the result and fractured surface analysis using SEM (Scanning Electron Microscope), in which five critical points of the fractured surface to be analyzed. The surface of chevron cut may deteriorated by scratching or compressing it with the other materials. These actions might damage the important features of the crack on the chevron surface, thus the specimens are suggested to keep in a solid container and they should be taken for SEM testing as soon as possible after the tensile testing.
- Simulation by analysing software: More information can be obtained if computer software is used to analyse the experimental results, example like the area of maximum fracture toughness and etc. Suggest using the software

package called ANSYS, because it is user-friendly and it can be incorporated with other software such as PRO-ENGINEER and so on.

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Appendix A

Project Specification

University of Southern Queensland
Faculty of Engineering and Surveying

ENG 4111 Research Project Part 2

PROJECT SPECIFICATION

Project Title : Fracture Analysis of Vinyl Ester Composite Cured under
Microwave Condition
Student Name : Mr. Wooi Lieh, CHAN
Student Number : 0050027343
Supervisor : Dr. Harry Ku

Project Synopsis:

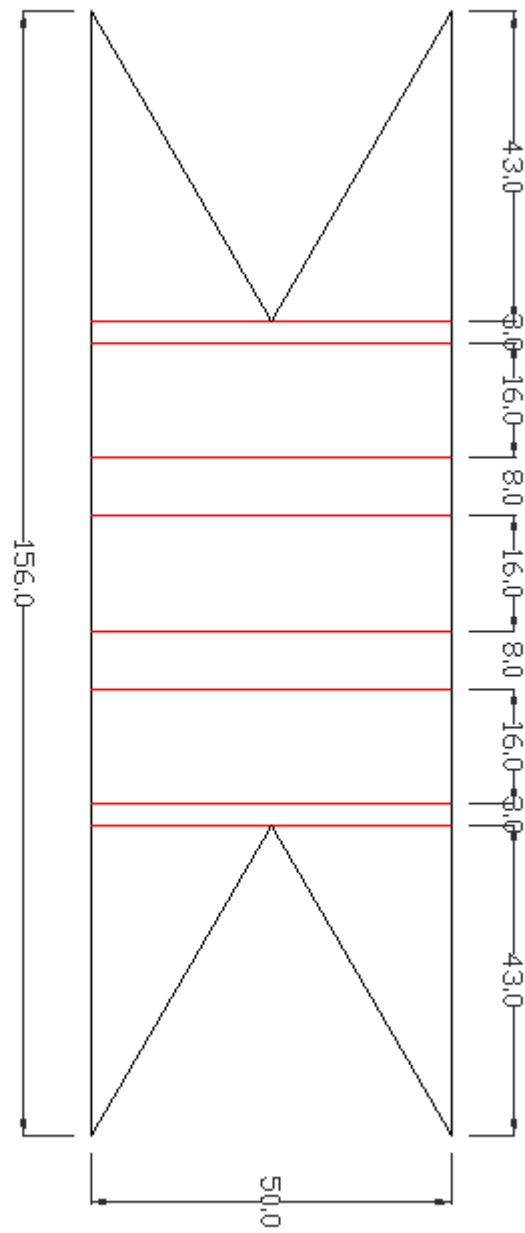
This project is to find out the fracture toughness of particulate reinforced vinyl ester resins cured microwave conditions. Short bar method will be used. SEM will also be used to analyze the fracture mechanics of the composites cured under ambient conditions. The percentages by weight of the fly ash (reinforcement) are 25% and 33%. The data collected may be useful in supporting the development of FCDD group in future.

Timelines of Specific Activities:

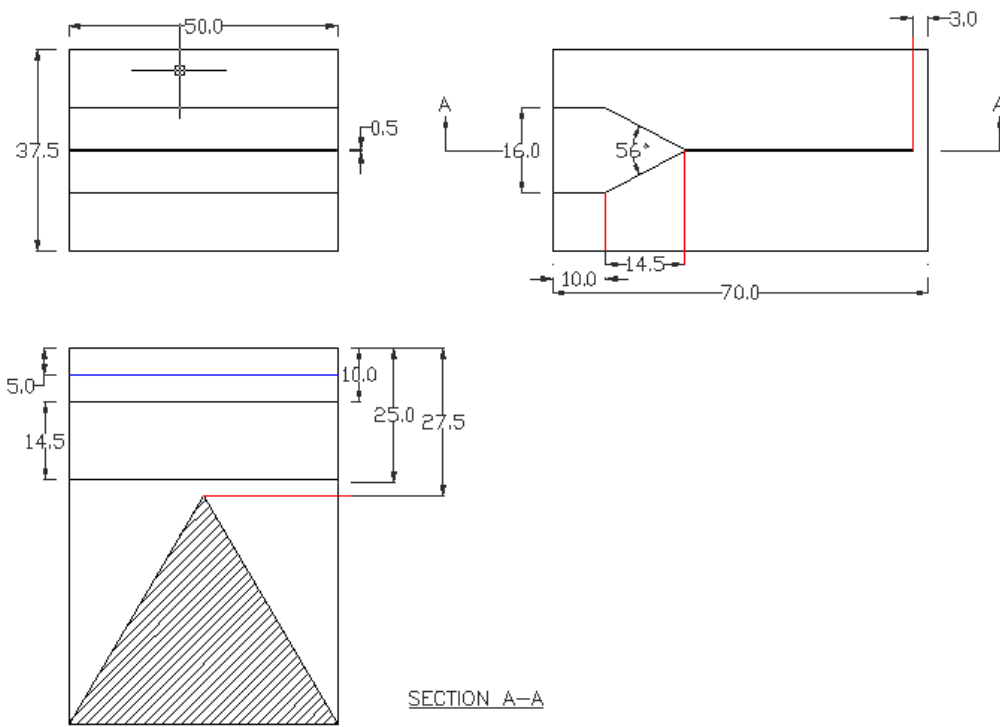
1. Familiarization of equipment and literature reviews.
Begin : 6th March 2006
Completion : 16th April 2006
Approx. Hours : 55 hours
2. Make samples of Vinyl Ester for testing using microwave and at ambient condition.
Begin : 17th April 2006
Completion : 26th May 2006
Approx. Hours : 60 hours
3. Analysis of results and compare other variables.
Begin : 29th May 2006
Completion : 31st July 2006

Appendix B

Auto-CAD drawing



The Auto-CAD drawing for the triangle part of the mould



Dimension of Short Bar using in Testing

Appendix C

Testing Results Obtained from MTS-810 of all Conditions

Appendix

Chan-ambient

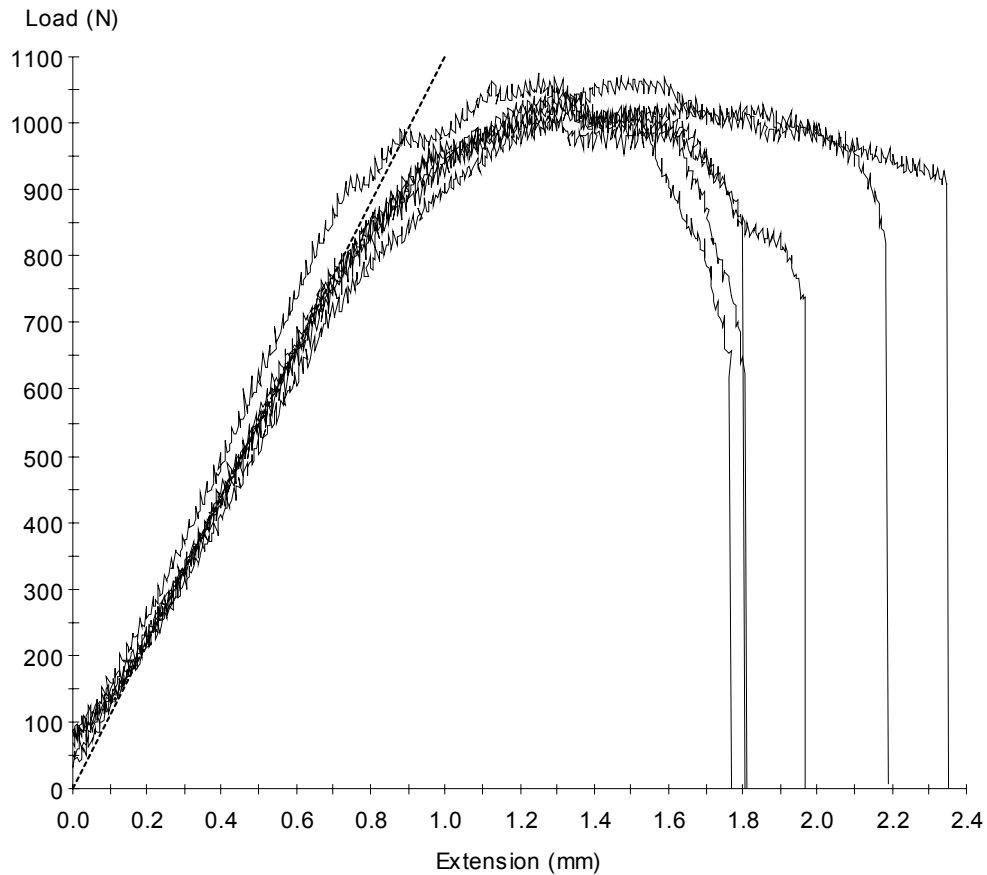
Test Date : 20-Sep-06

Method : MMT fracture toughness Test .msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm ²	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	50.000	23.000	1150	1033	0.90	739	0.64
2	50.000	23.000	1150	1036	0.90	908	0.79
3	50.000	23.000	1150	1071	0.93	819	0.71
4	50.000	23.000	1150	1075	0.94	657	0.57
5	50.000	23.000	1150	1056	0.92	841	0.73
6	50.000	23.000	1150	1012	0.88	622	0.54
Mean	50.000	23.000	1150	1047	0.91	764	0.66
Std Dev	0.000	0.000	0	24	0.02	111	0.10

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N
1	1.966	0.744	856.158
2	2.348	0.818	941.296
3	2.185	0.794	912.636
4	1.766	0.841	966.754
5	1.799	0.786	903.364
6	1.806	0.742	852.786
Mean	1.978	0.787	905.499
Std Dev	0.239	0.039	45.392



Appendix

Chan-180-60n

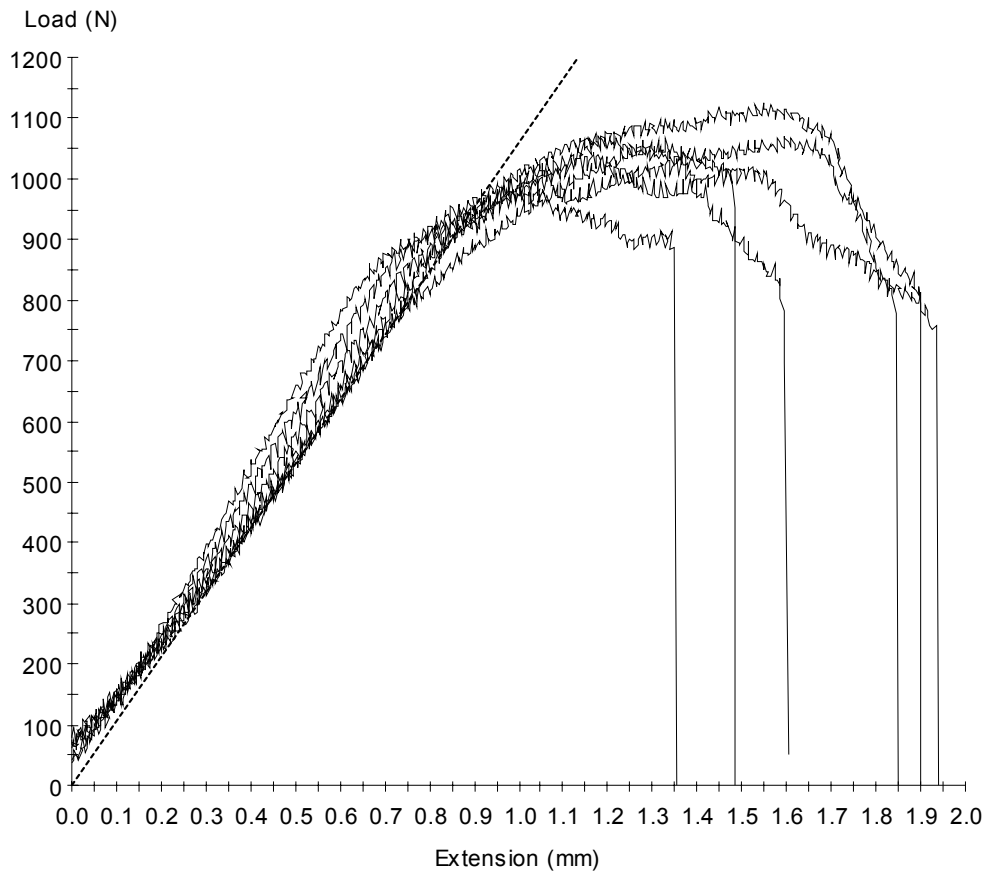
Test Date : 20-Sep-06

Method : MMT fracture toughness Test .msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm ²	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	50.000	23.000	1150	1074	0.93	952	0.83
2	50.000	23.000	1150	1028	0.89	748	0.65
3	50.000	23.000	1150	982	0.85	879	0.76
4	50.000	23.000	1150	1044	0.91	779	0.68
5	50.000	23.000	1150	1125	0.98	757	0.66
6	50.000	23.000	1150	1071	0.93	795	0.69
Mean	50.000	23.000	1150	1054	0.92	818	0.71
Std Dev	0.000	0.000	0	48	0.04	81	0.07

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N
1	1.483	0.780	897.294
2	1.596	0.874	1005.192
3	1.351	0.788	906.735
4	1.847	0.791	910.107
5	1.934	0.843	969.957
6	1.902	0.798	918.200
Mean	1.685	0.813	934.581
Std Dev	0.243	0.037	43.059



Appendix

Chan-180-70n

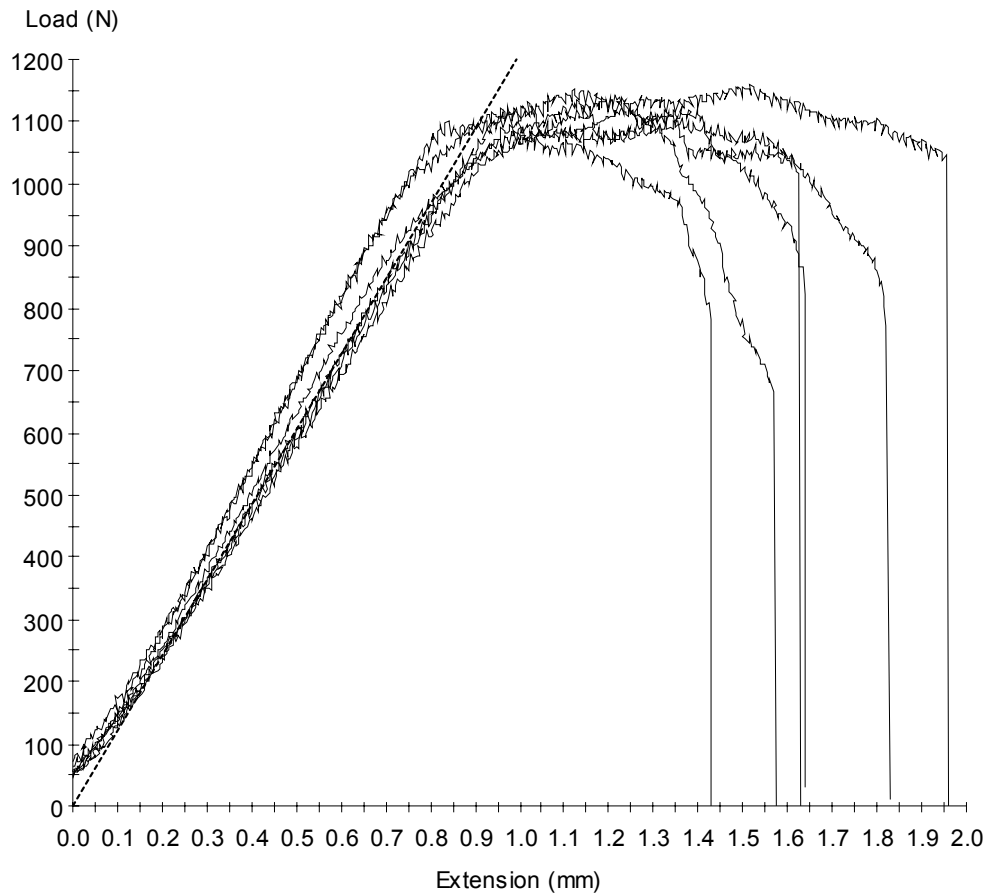
Test Date : 31-Aug-06

Method : MMT fracture toughness Test .msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm ²	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	23.000	50.000	1150	1126	0.98	662	0.58
2	23.000	50.000	1150	1120	0.97	783	0.68
3	23.000	50.000	1150	1159	1.01	1045	0.91
4	23.000	50.000	1150	1124	0.98	1017	0.88
5	23.000	50.000	1150	1124	0.98	773	0.67
6	23.000	50.000	1150	1143	0.99	824	0.72
Mean	23.000	50.000	1150	1133	0.98	851	0.74
Std Dev	0.000	0.000	0	15	0.01	150	0.13

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N
1	1.570	0.853	981.456
2	1.429	0.888	1021.244
3	1.956	0.938	1078.565
4	1.624	0.969	1114.643
5	1.822	0.907	1042.824
6	1.637	0.906	1041.475
Mean	1.673	0.910	1046.701
Std Dev	0.188	0.040	45.997



Appendix

Chan-180-80n

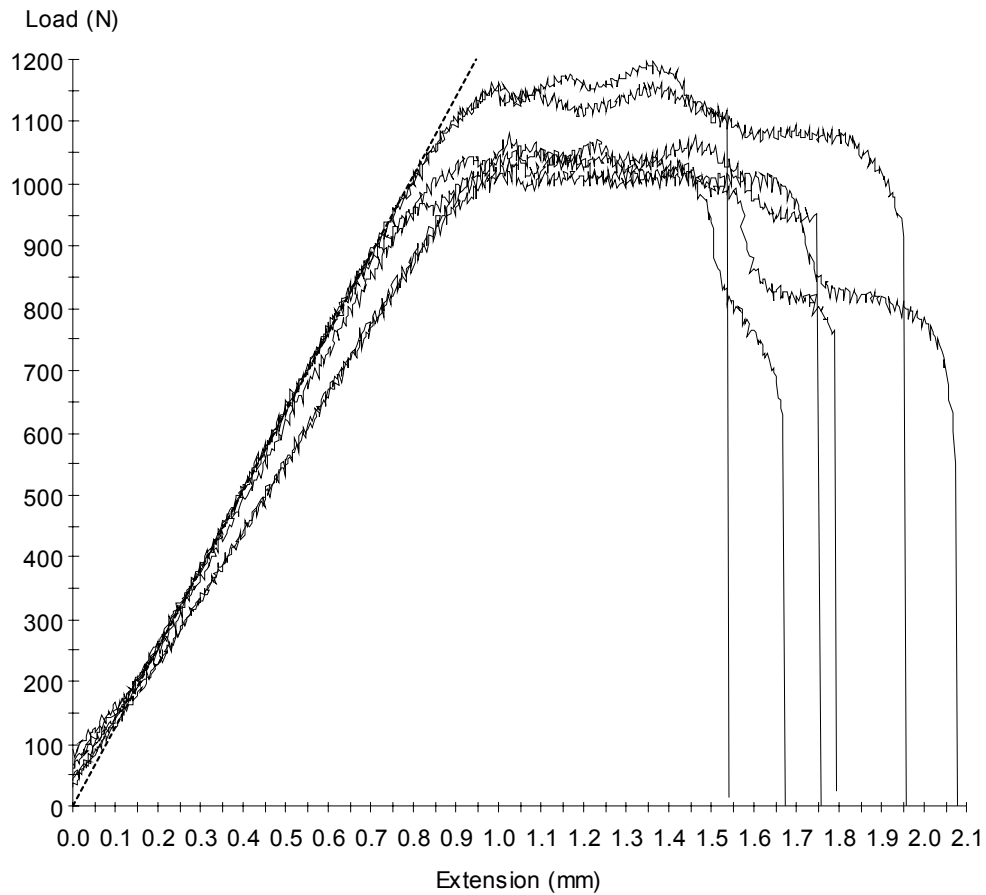
Test Date : 20-Sep-06

Method : MMT fracture toughness Test .msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm ²	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	50.000	23.000	1150	1196	1.04	1118	0.97
2	50.000	23.000	1150	1080	0.94	953	0.83
3	50.000	23.000	1150	1162	1.01	875	0.76
4	50.000	23.000	1150	1040	0.90	764	0.66
5	50.000	23.000	1150	1067	0.93	628	0.54
6	50.000	23.000	1150	1069	0.93	551	0.48
Mean	50.000	23.000	1150	1102	0.96	815	0.71
Std Dev	0.000	0.000	0	61	0.05	211	0.18

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N
1	1.536	0.998	1147.577
2	1.749	0.906	1042.208
3	1.954	0.980	1126.503
4	1.791	0.888	1021.303
5	1.668	0.895	1028.795
6	2.073	0.831	955.627
Mean	1.795	0.916	1053.669
Std Dev	0.194	0.062	71.461



Appendix

Chan-360-60n

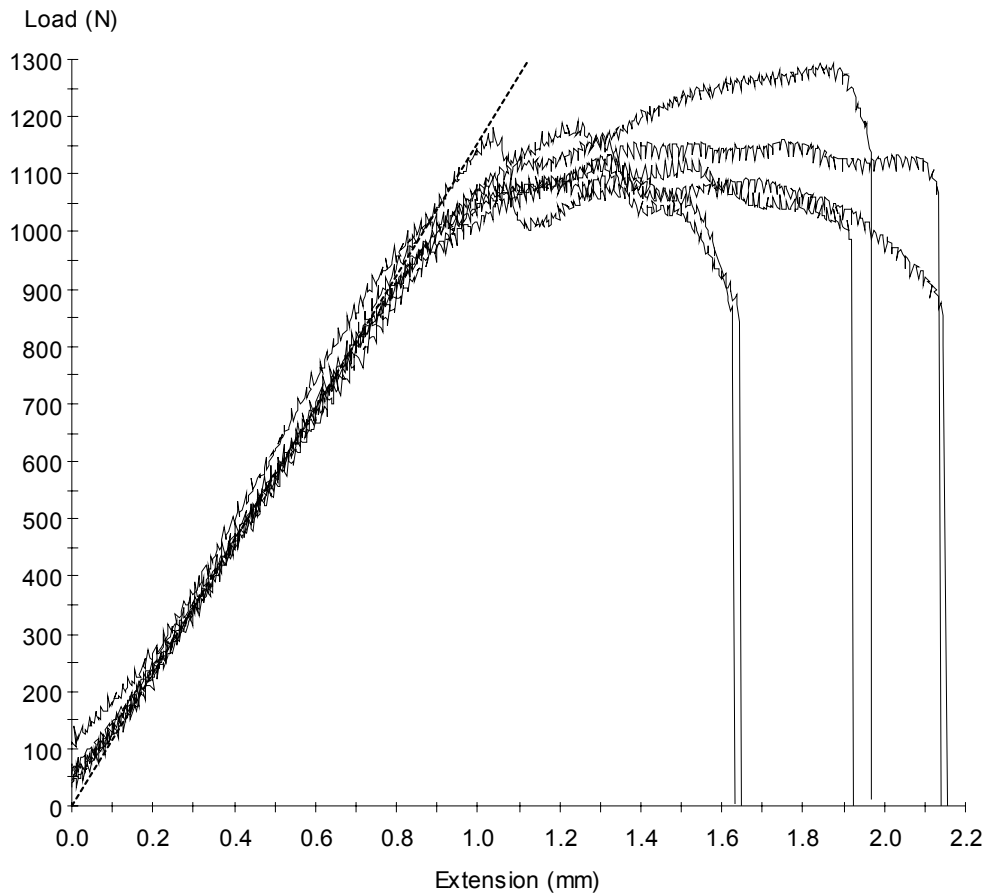
Test Date : 20-Sep-06

Method : MMT fracture toughness Test .msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm ²	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	50.000	23.000	1150	1191	1.04	868	0.75
2	50.000	23.000	1150	1093	0.95	845	0.73
3	50.000	23.000	1150	1293	1.12	1138	0.99
4	50.000	23.000	1150	1126	0.98	986	0.86
5	50.000	23.000	1150	1159	1.01	1068	0.93
6	50.000	23.000	1150	1102	0.96	842	0.73
Mean	50.000	23.000	1150	1161	1.01	958	0.83
Std Dev	0.000	0.000	0	74	0.06	126	0.11

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N
1	1.627	0.972	1118.316
2	1.645	0.932	1071.954
3	1.966	0.948	1089.993
4	1.920	0.878	1009.913
5	2.136	0.907	1043.462
6	2.147	0.849	976.195
Mean	1.907	0.914	1051.639
Std Dev	0.228	0.046	52.592



Appendix

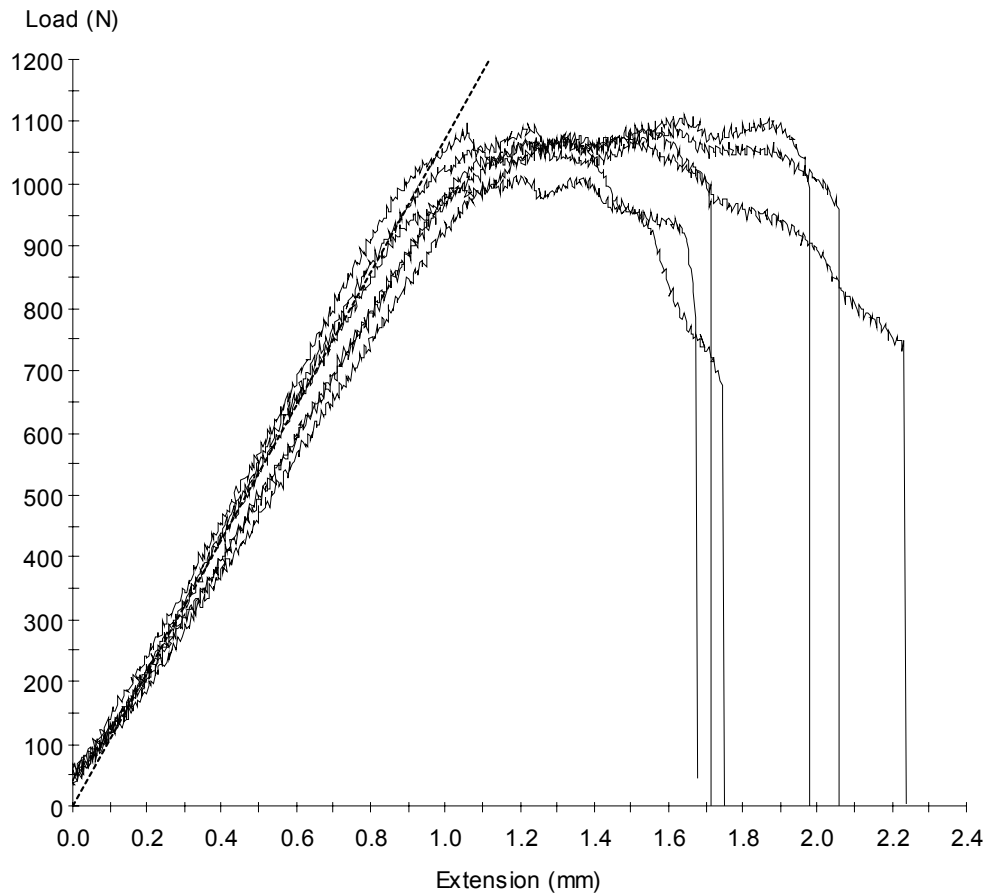
Chan-360-70n

Test Date : 31-Aug-06

Method : MMT fracture toughness Test .msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm ²	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	23.000	50.000	1150	1111	0.97	992	0.86
2	23.000	50.000	1150	1081	0.94	971	0.84
3	23.000	50.000	1150	1098	0.95	626	0.54
4	23.000	50.000	1150	1014	0.88	786	0.68
5	23.000	50.000	1150	1088	0.95	747	0.65
6	23.000	50.000	1150	1097	0.95	942	0.82
Mean	23.000	50.000	1150	1082	0.94	844	0.73
Std Dev	0.000	0.000	0	35	0.03	147	0.13
Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N				
1	1.977	0.859	987.534				
2	1.712	0.892	1025.973				
3	1.747	0.894	1028.091				
4	1.674	0.849	976.671				
5	2.234	0.860	988.809				
6	2.059	0.813	934.692				
Mean	1.900	0.861	990.295				
Std Dev	0.225	0.030	34.608				



Appendix

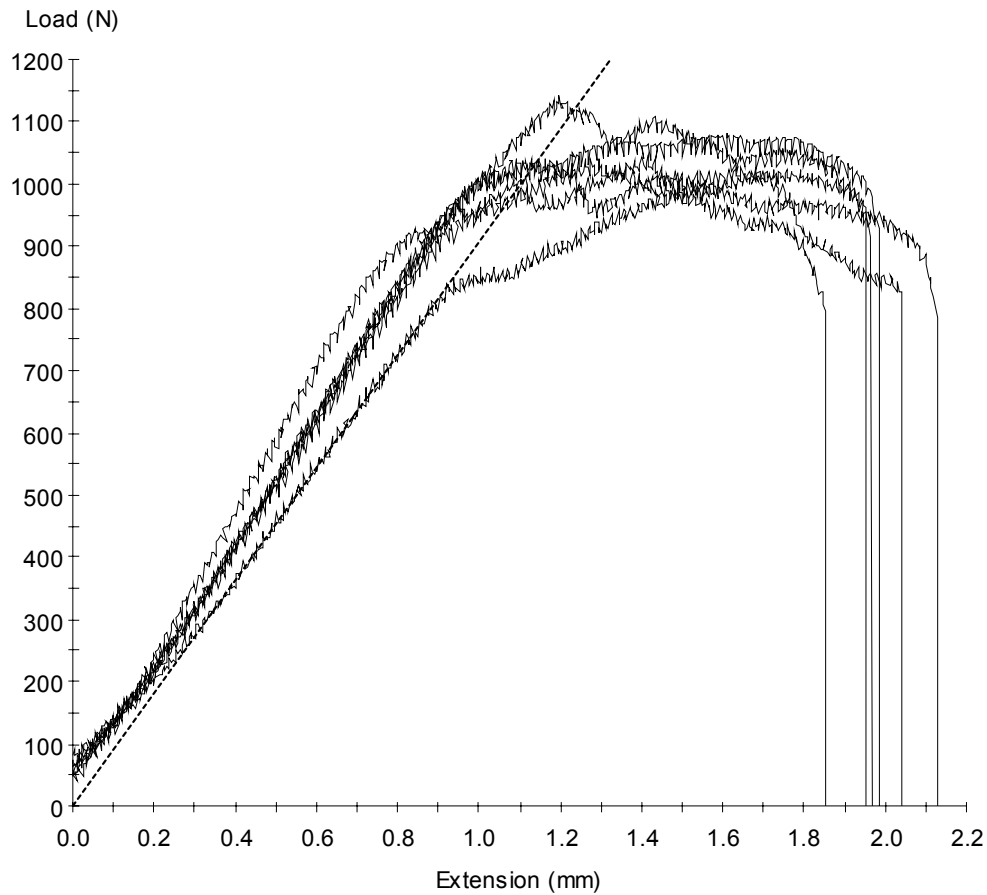
Chan-360-80n

Test Date : 20-Sep-06

Method : MMT fracture toughness Test .msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm ²	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	50.000	23.000	1150	1079	0.94	928	0.81
2	50.000	23.000	1150	1042	0.91	796	0.69
3	50.000	23.000	1150	1020	0.89	787	0.68
4	50.000	23.000	1150	1046	0.91	826	0.72
5	50.000	23.000	1150	1141	0.99	963	0.84
6	50.000	23.000	1150	1024	0.89	908	0.79
Mean	50.000	23.000	1150	1059	0.92	868	0.75
Std Dev	0.000	0.000	0	45	0.04	74	0.06
Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N				
1	1.985	0.788	906.735				
2	1.852	0.873	1003.843				
3	2.126	0.825	948.883				
4	2.041	0.843	969.114				
5	1.949	0.936	1076.337				
6	1.965	0.733	843.514				
Mean	1.986	0.833	958.071				
Std Dev	0.092	0.070	80.059				



Appendix

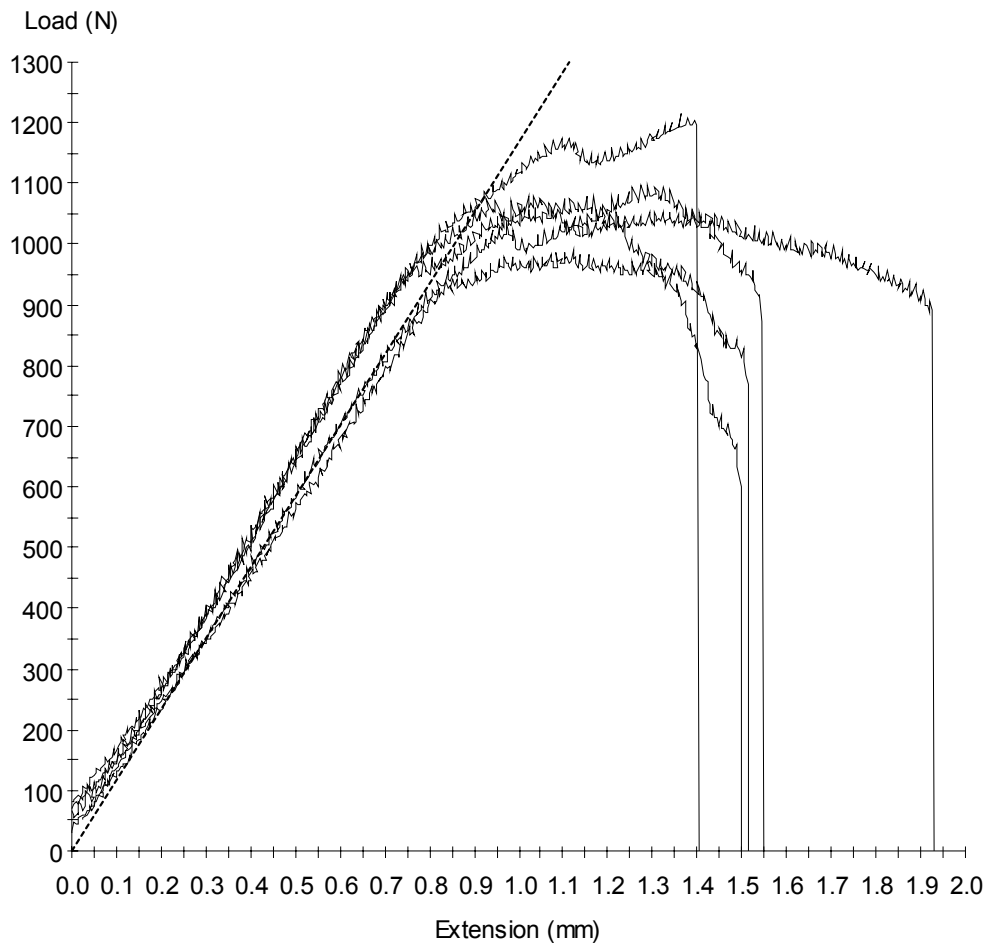
Chan-520-15n

Test Date : 20-Sep-06

Method : MMT fracture toughness Test .msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm ²	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	50.000	23.000	1150	1215	1.06	1194	1.04
2	50.000	23.000	1150	1080	0.94	771	0.67
3	50.000	23.000	1150	990	0.86	601	0.52
4	50.000	23.000	1150	1061	0.92	892	0.78
5	50.000	23.000	1150	1097	0.95	874	0.76
Mean	50.000	23.000	1150	1089	0.95	866	0.75
Std Dev	0.000	0.000	0	82	0.07	216	0.19
Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N				
1	1.400	0.943	1084.864				
2	1.515	0.910	1046.931				
3	1.498	0.830	954.881				
4	1.926	0.842	968.031				
5	1.544	0.878	1009.673				
Mean	1.576	0.881	1012.876				
Std Dev	0.202	0.047	54.145				



Appendix

Chan-540-20n

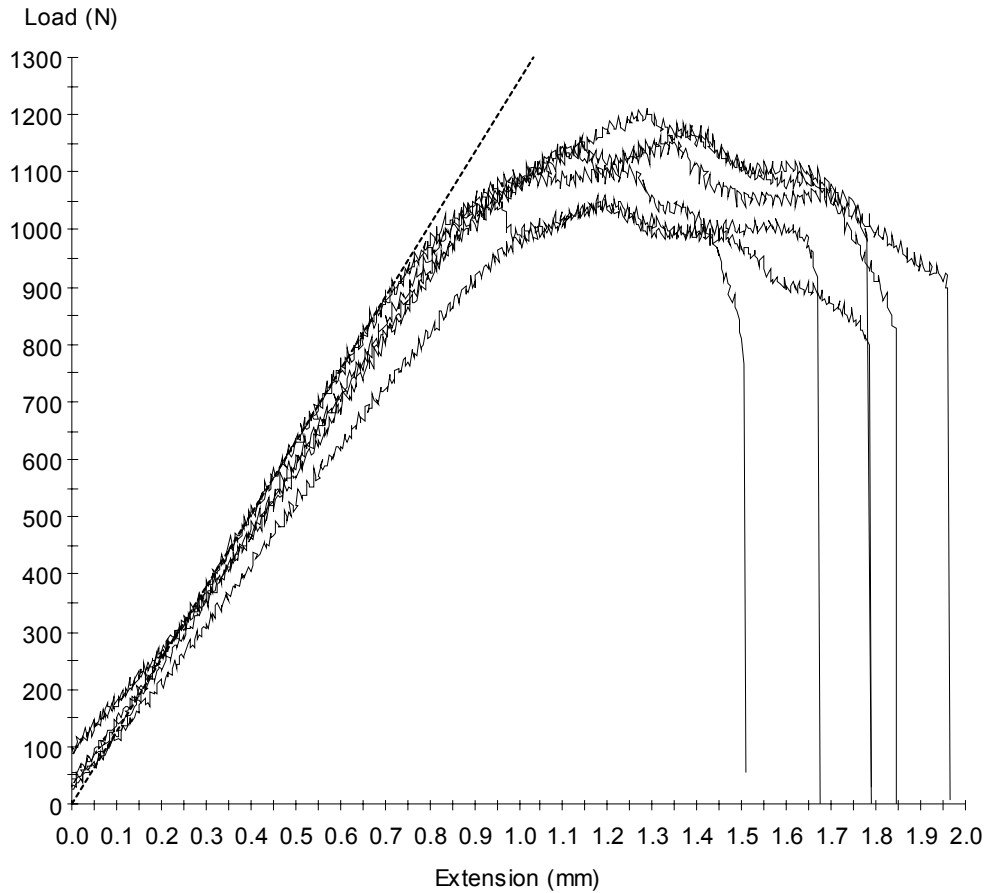
Test Date : 20-Sep-06

Method : MMT fracture toughness Test .msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm ²	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	50.000	23.000	1150	1111	0.97	765	0.66
2	50.000	23.000	1150	1053	0.92	801	0.70
3	50.000	23.000	1150	1060	0.92	916	0.80
4	50.000	23.000	1150	1212	1.05	830	0.72
5	50.000	23.000	1150	1164	1.01	989	0.86
6	50.000	23.000	1150	1182	1.03	898	0.78
Mean	50.000	23.000	1150	1130	0.98	866	0.75
Std Dev	0.000	0.000	0	66	0.06	83	0.07

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N
1	1.502	0.910	1046.931
2	1.783	0.865	994.668
3	1.669	0.857	985.396
4	1.843	0.945	1087.056
5	1.780	0.941	1082.335
6	1.962	0.891	1025.014
Mean	1.756	0.902	1036.900
Std Dev	0.157	0.037	43.030



Appendix

Chan-540-25n

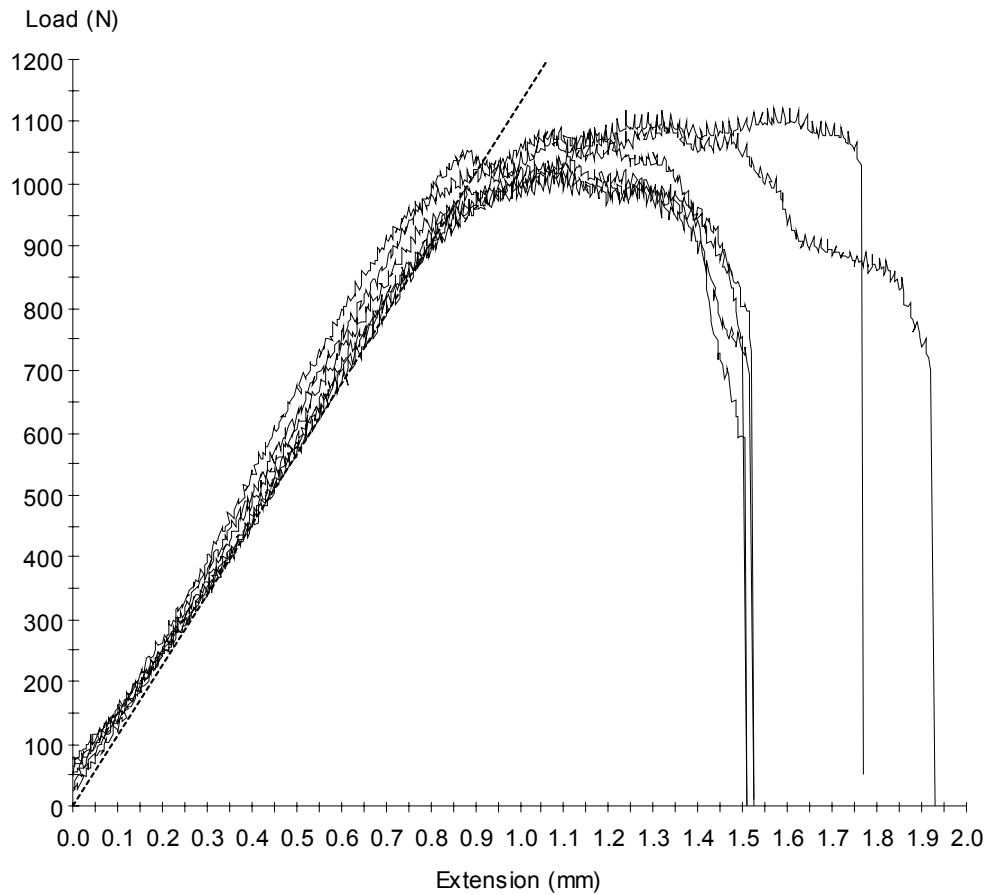
Test Date : 20-Sep-06

Method : MMT fracture toughness Test .msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm ²	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	50.000	23.000	1150	1100	0.96	692	0.60
2	50.000	23.000	1150	1023	0.89	566	0.49
3	50.000	23.000	1150	1020	0.89	659	0.57
4	50.000	23.000	1150	1039	0.90	727	0.63
5	50.000	23.000	1150	1091	0.95	730	0.63
6	50.000	23.000	1150	1121	0.97	1024	0.89
Mean	50.000	23.000	1150	1066	0.93	733	0.64
Std Dev	0.000	0.000	0	43	0.04	155	0.13

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N
1	1.919	0.834	959.264
2	1.506	0.844	971.066
3	1.515	0.825	948.812
4	1.500	0.857	985.902
5	1.521	0.891	1024.677
6	1.767	0.895	1028.892
Mean	1.621	0.858	986.435
Std Dev	0.178	0.029	33.631



Appendix

Chan-720-15n

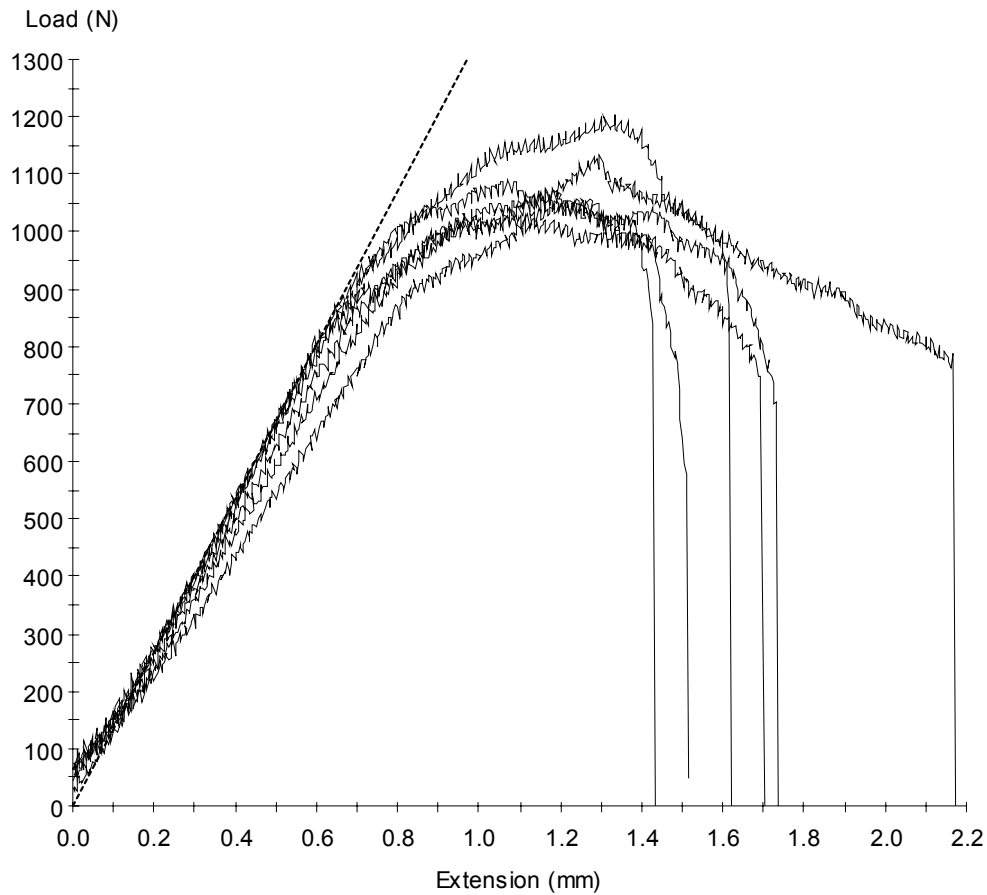
Test Date : 20-Sep-06

Method : MMT fracture toughness Test .msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm ²	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	50.000	23.000	1150	1076	0.94	603	0.52
2	50.000	23.000	1150	1205	1.05	871	0.76
3	50.000	23.000	1150	1133	0.98	780	0.68
4	50.000	23.000	1150	1023	0.89	746	0.65
5	50.000	23.000	1150	1090	0.95	726	0.63
6	50.000	23.000	1150	1050	0.91	810	0.70
Mean	50.000	23.000	1150	1096	0.95	756	0.66
Std Dev	0.000	0.000	0	65	0.06	91	0.08

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N
1	1.733	0.849	976.195
2	1.614	0.891	1024.580
3	2.168	0.875	1005.866
4	1.693	0.817	939.273
5	1.492	0.890	1023.231
6	1.430	0.799	918.537
Mean	1.689	0.853	981.280
Std Dev	0.262	0.039	44.646



Appendix

Chan-720-20n

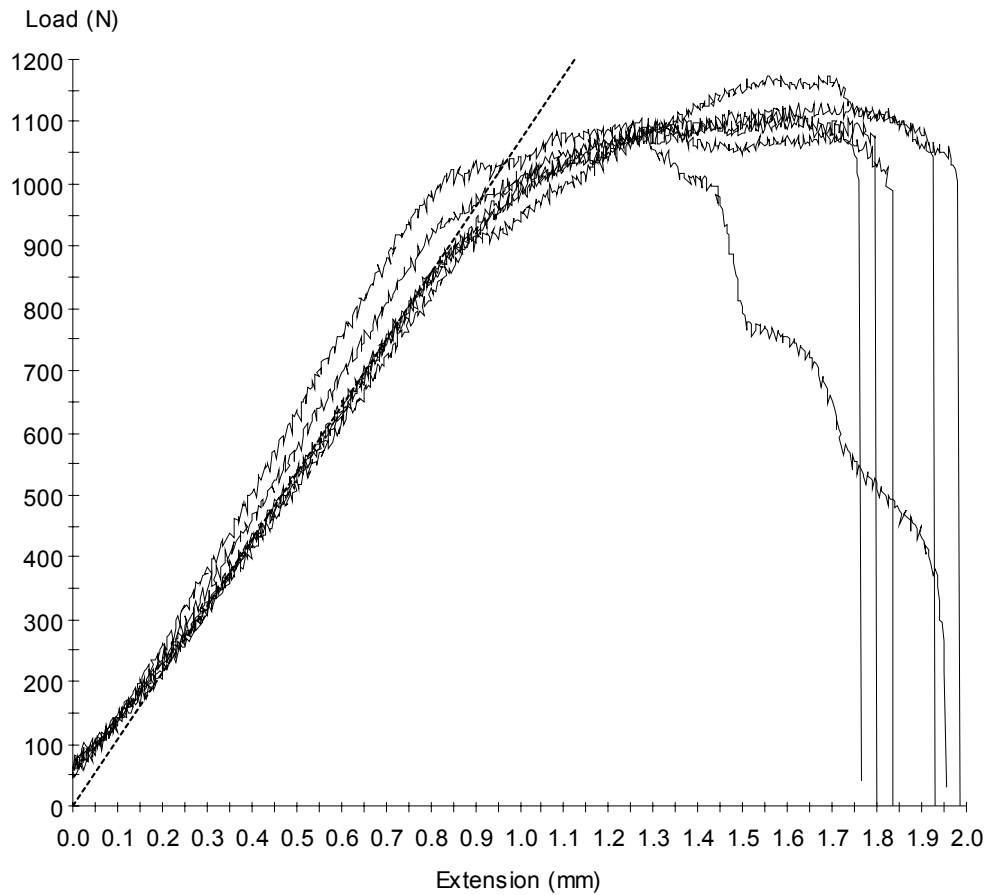
Test Date : 31-Aug-06

Method : MMT fracture toughness Test .msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm ²	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	23.000	50.000	1150	1111	0.96	989	0.86
2	23.000	50.000	1150	1100	0.96	1005	0.87
3	23.000	50.000	1150	1173	1.02	1044	0.91
4	23.000	50.000	1150	1104	0.96	329	0.29
5	23.000	50.000	1150	1128	0.98	1003	0.87
6	23.000	50.000	1150	1124	0.98	1077	0.94
Mean	23.000	50.000	1150	1123	0.98	908	0.79
Std Dev	0.000	0.000	0	27	0.02	285	0.25

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N
1	1.833	0.807	928.454
2	1.760	0.852	980.043
3	1.926	0.869	999.430
4	1.940	0.869	999.430
5	1.980	0.829	953.068
6	1.793	0.774	889.847
Mean	1.872	0.833	958.379
Std Dev	0.089	0.038	43.497



Appendix

Chan-720-25n

Test Date : 20-Sep-06

Method : MMT fracture toughness Test .msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm ²	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	50.000	23.000	1150	1125	0.98	1052	0.91
2	50.000	23.000	1150	1145	1.00	1145	1.00
3	50.000	23.000	1150	1043	0.91	617	0.54
4	50.000	23.000	1150	1077	0.94	763	0.66
5	50.000	23.000	1150	1103	0.96	671	0.58
6	50.000	23.000	1150	1153	1.00	927	0.81
Mean	50.000	23.000	1150	1108	0.96	863	0.75
Std Dev	0.000	0.000	0	42	0.04	213	0.18
Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N				
1	2.014	0.851	978.892				
2	0.905	0.551	633.619				
3	1.669	0.882	1014.633				
4	1.699	0.898	1032.504				
5	1.588	0.870	1000.134				
6	1.589	0.938	1078.698				
Mean	1.577	0.832	956.413				
Std Dev	0.365	0.141	161.707				

