EFFECTS OF ENVIRONMENT ON FRACTURE TOUGHNESS OF GLASS FIBER/POLYESTER COMPOSITE

By

A.K.M. Kais Bin Zaman

A THESIS PRESENTED TO THE INDUSTRIAL AND PRODUCTION
ENGINEERING DEPARTMENT IN PARTIAL FULFILLMENT OF THE
REQUIREMENTS FOR THE DEGREE OF
MASTER OF SCIENCE
BANGLADESH UNIVERSITY OF ENGINEERING AND TECHNOLOGY
DECEMBER 2005

The thesis titled **"Effects of Environments on Fracture Toughness of Glass Fiber/Polyester Composite"** submitted my A.K.M. Kais Bin Zaman, Student No. 040308018, Session. April 2003, has been accepted as satisfactory in partial fulfillment of the requirement for the degree of Master of Science in Industrial & Production Engineering on December 18, 2005.

BOARD OF EXAMINERS

1.	Dr. A.K.M. Masud Assistant Professor Dept. of Industrial & Production Engineering, BUET, Dhaka, Bangladesh	Chairman
2.	Dr. Nikhil Ranjan Dhar Professor & Head Dept. of Industrial & Production Engineering, BUET, Dhaka, Bangladesh	Member (ex-officio)
3.	Dr. Mahiuddin Ahmed Professor Dept. of Industrial & Production Engineering, BUET, Dhaka, Bangladesh	Member
4.	Dr. Abdullahil Azeem Assistant Professor Dept. of Industrial & Production Engineering, BUET, Dhaka, Bangladesh	Member
5.	Dr. Md. Afsar Ali Assistant Professor Dept. of Mechanical Engineering, BUET, Dhaka, Bangladesh	Member - External

DECLARATION

It is hereby declared that this thesis or any part of this has not been submitted elsewhere for the award of any degree or diploma.

Kzaman

A.K.M. Kais Bin Zaman

This dissertation is dedicated to my parents,

Late A.K.M. Assaduzzaman

and

Dil Roushan Ara

Contents

List of Figures

List of Tables

Nomenclature

1PB	One-point bending
LVDT	Linear variable displacement transducer
3PB	Three-point bending
4ENF	Four-point bend end-notched flexure
4PB	Four-point bending
MCC	Modified compliance calibration
MCCI	Modified crack closure integral
MMB	Mixed-mode bending
MMF	Mixed-mode flexure
MVCCT	Modified virtual-crack-closure-technique
CLS	Cracked-lap shear
RB	Reaction-bonded
CT	Compact tensile
RT	Room temperature
DCB	Double-cantilever beam
SC	Surface-cracked
DEN	Double-edge-notched
SCB	Single cantilever beam
DENF	Double-end-notched flexure
SEM	Scanning electron microscopy
SEN	Single-edge notched
SENB	Single-edge-notched beam
ELS	End-loaded split
SENT	Single-edge notched tensile
ENF	End-notced flexure
SEVNB	Single-edge V-notched bend
FEA	Finite element analysis
TM	Travelling microscope
UD	Unidirectional
VIC	Vickers indentation crack
ILSS	Interlaminar shear strength

IPC	Interpenetrating phase composite
IS	Indentation strength
WOF	Work of fracture
LEFM	Linear elastic fracture mechanics
C	Crack length
K_I	Stress intensity factor
K_c	Critical stress intensity factor
M_i	Initial weight of the specimen
M_f	Final weight of the specimen after environmental exposure
N	Normality of the solution
W	Width of the test specimen
Y	Geometric constant
σ	Normal stress
σ_c	Critical stress
σ_f	Failure stress
σ_n	Stress at notch

Acknowledgements

I am very grateful to Dr. A.K.M. Masud for providing me the opportunity to complete my M.Sc. studies under his exceptional guidance. He is not only my thesis supervisor but also a great influence in my life. Throughout this research, I have greatly appreciated his consistent encouragement, patience and positive attitude.

Also, I would like to thank Dr. Nikhil Ranjan Dhar, Head of the Department of IPE, for his permission to enable start work on the project.

Many thanks should go to my friend, Kazi Bayazid Kabir, for his expert help in preparing the thesis.

I would like to thank Prof. Quamrul Islam, Head of the Department of ME, for his kind permission to use the Solid Mechanics Lab.

I would like to thank Mr. Gilbert Adhikery of MIT Bangladesh Ltd. for his continuous support in preparing the specimens.

I would like to thank Mr. Hanif and all other staffs of Solid Mechanics Lab of ME Department for their kind cooperation.

I would like to thank all my friends and colleagues, especially Abdullah Al Khaled and Nouroz Islam, who have always encouraged me and helped me in various ways.

I would like to express my deepest appreciation to my mother for continuous support and love. I am also thankful to God for giving me the opportunity to extend my education.

And I like to thank all the people who are not listed here but have contributed in any way, being my side or in any other senses.

Abstract

The experimental determination of the resistance to delamination is very important, since composite materials have superior properties only in the fiber direction. The resistance to delamination is known as the fracture toughness or fracture resistance. Fracture toughness of a material has immense importance in the determination of the resistance of the material to crack propagation. The fracture and damage process can be influenced by many parameters. In this thesis, an experimental investigation has been carried out on the fracture toughness of chopped strand mat glass fibre reinforced composite after exposing to various adverse environments, like, water, saline water, acidic water, organic fuel, ice temperature and hot air of 60°C, for different durations using single edge notched (SEN) specimens. The specimens were prepared from the fabricated glass fiber reinforced composite plate. The sharp notch of different length sizes, namely, 2mm, 4 mm, 6mm, were cut in different specimens. Microscopic examination has been carried out to verify uniform length of the notches. The required number of specimens with single edge sharp notch was made in the same way.

It has been drawn a number of important inferences from this work. Fracture toughness is independent of crack size. There is a sizeable effect of environments on mechanical properties and on fracture toughness. The environmental effects depend on the type of environment, hostility, corrosivity and constituent ingredients. After exposure to various environments, the highest amount of degradation in fracture toughness has been recorded in acidic water and then in low temperature followed by water, saline water, high temperature and organic fuel.

A relationship between fracture toughness and duration of exposure under these adverse environments has been established. The fracture toughness has been found to decrease continuously with increased duration of environmental exposure, whereas the fracture toughness has been found to be independent of pre-crack length.

Chapter 1

Introduction

1.1 Background

Fracture in all materials, brittle or ductile, homogeneous or composites. is governed more or less by microscopic discontinuities and imperfections, such as, cracks, inclusions or dispersed phases. The strength of a material may be correlated with its fracture energy, elastic modulus, and the size of the crack initiating the fracture. In this domain, the fracture toughness of the material has a major role in the determination of the fracture behaviour and resistance of the material to crack propagation under the influence of an external load. The material cannot be used without analyzing the fracture behaviour or ultimate strength of structure. In assessing the strength of a structural element, fracture mechanics provides a greater insight when a crack-like defect is explicitly taken into account. Central to the process is the determination of stress intensity factor, which is a function of the geometry of the structure and the character of the load. The advent of fracture mechanics has accelerated the methods for determination of crack growth rate and maximum allowable damage at limit load conditions [1, 2]. In that line, the degradation of strength of composite materials in presence of micro cracks must be understood precisely under any situation for their wider application. The glass fibre reinforced composites are finding wide applications in primary structures of the aircraft, space craft, ocean vehicles, automotive industries as well as other cladding situations. These applications sometimes face a wide range of environmental exposure like water, saline water, low temperature, high temperature, organic fuel, acidic water, etc. Thus the understanding of the fracture behaviour and prediction of fracture toughness of composite materials after different environmental exposure have assumed greater importance.

The importance of estimating the fracture load of notched plates has more or less been established and the influence of related parameters like width and thickness of specimen, notch geometry, notch angle, notch root radius, and notch depth have widely been studied [3-5]. Thus, the determination of stress intensity factor has become mandatory for solving the problems like residual strength analysis, fatigue crack growth rate and stress corrosion mechanism. However, the fracture strength of the composites afrer exposure to adverse environmental condition is yet to be substantially established. In this context, an extensive experimental investigation of fracture toughness after different environmental exposure has been carried out in the present study.

1.2 Goals and Significance

The goal of this research was to study the fracture toughness of fiber glass reinforced plastic under different environmental conditions. A series of model experiments has been conducted. Fracture toughness of a material has immense importance in the determination of the resistance of the material to crack propagation. In this thesis, an experimental investigation has been carried out on the fracture toughness of 2D randomly oriented glass fiber chopped strand mat composite after exposing to various adverse environments, like, water, saline water, acidic water, organic fuel, ice temperature and hot air of 60°C, for different durations using single edge notched (SEN) specimens. A relationship between fracture toughness and duration of exposure under these adverse environments has been investigated.

1.3 Thesis Organization

This report will begin with a review of relevant literature to give background and support to the methods and ideas used in this testing. Following the literature review, materials, specimen preparation, and test methods/equipment will be discussed. Finally analysis methods, test results, and conclusions will be presented. The chapter layout is as follows:

Chapter 1 gives a brief introduction to the topic of this research as well as background information.

Chapter 2 reviews literature relevant to the topics of this research including: test method standardization, general fracture mechanics, and data analysis methods for fracture testing.

Chapter 3 focuses on the materials and test methods employed to gather the data presented in this thesis.

Chapter 4 covers the data analysis methods utilized to analyze test data collected using the methods described in Chapter 3.

Chapter 5 presents the results of this testing as well as relevant insight into trends and phenomena found during the testing and analysis process as well as relevant conclusions.

Finally, Appendix A represents the figures that reveal that fracture toughness is independent of crack size

Chapter 2

Literature Review

2.1 Introduction

Ships, aircraft and rockets are extremely complex engineering systems with many thousands of components In the construction of such systems it is impossible to complete avoid the presence of flaws such as cracks. Understanding the strength of materials in the presence of cracks is thus key to developing reliable aerospace and ocean engineering hardware. The fracture toughness can be used to characterize the fracture behavior of composite materials under varied conditions. Several methods were developed by designers. Beam theory can be applied to obtain closed from equations for the fracture toughness. These equations can be used only in UD beam shaped specimens (ENF, SCB, etc.), and generally have to be completed by various factors to approach the experiments. The compliance of specimens with different shape (CT, SEN, VIC, etc.) and different structure (e.g : woven-fabric) can be determined by the area method or by fitting the experimentally measured compliance. The fracture toughness can be determined numerically by many ways (MVCCT, MCCI, etc). The fracture toughness can be affected by many parameters Tests can be conducted by using wide range of specimen types, e.g. specimens with woven fabric structure, IPCs, varied particle or fiber-volume fraction, different fiber orientation, and different specimen configuration. The composites combined with metal laminates can be used to improve the fracture toughness. The specimens always contain a crack, notch or hole, which acts as an artificial starting defect Crack propagation can be expected in the starting defect. The shape, length and width of the crack can be different. There are a lot of facilities to vary the extrinsic test conditions. The effect of displacement rate varies in wide range, and different behavior can be observed for different materials The tests conducted at cryogenic and elevated temperature or elevated pressure facilitates to investigate the composites under practical conditions. During the test several type of failure prior to delamination fracture can occur. These effects can decrease or increase the fracture toughness. This work is designed to illustrate how strength in the presence of cracks -termed *Fracture Toughness* - is characterized and measured after exposing to different adverse environments.

2.2 Stress around a crack

Consider the idealized situation shown in Figure 2.1. This shows a uniform material of infinite extent that contains a semi-infinite horizontal crack coincident with the negative x axis. The crack is being pulled apart by a stress acting in the y direction σ_y that far away from the crack is uniform throughout the material. The stress concentration in the vicinity of the crack may be determined analytically if the crack tip is assumed to be sharp and the material is allowed to deform only in a linear elastic fashion. Such an analysis shows that, along the positive x axis, the stress increases to infinity at the crack tip. Note that the overall magnitude of the stress field around the crack tip is controlled by the parameter K, called the *stress intensity factor*. In this idealized situation, K is proportional to the uniform tension being applied to the material. This type of analysis is an example of *Linear Elastic Fracture Mechanics*, LEFM.

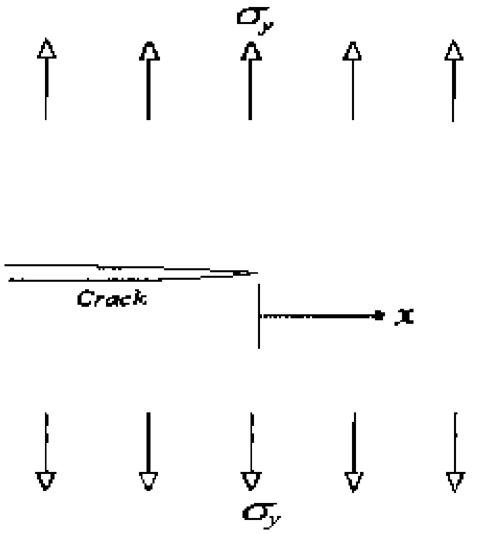

Figure 2.1 Crack in a perfectly elastic material under load

The real situation is of course more complicated. Consider the cracked specimen in Figure 2.2. Immediately surrounding the crack tip the large stresses predicted by LEFM is not realized because the material does not behave in a linear elastic fashion here. In a metal, plastic yeilding occurs to relieve and redistribute the stresses. In other materials, such as polymers or ceramics, different types of deformation, such as cracking or micro-cracking, may occur. The above condition is also unrealistic far from the crack tip where the shape of the specimen and the loading conditions determine the stress field. In between these regions, however, is a region where the crack dominates the stress field and the material deforms elastically. This is called the *region of K dominance*.

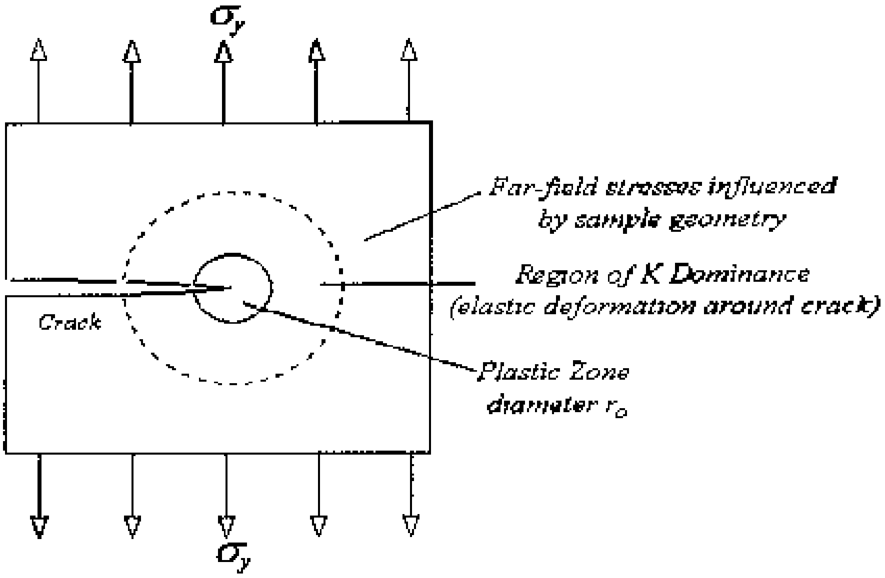

Figure 2.2 Crack in a real specimen under load.

Fortunately, as long as the plastic zone remains small compared to the specimen size, the region of K dominance controls the behavior of the crack. This means, for example, that we can use the stress intensity factor K to characterize the strength of the stress field surrounding the crack tip [6]

2.3 Fracture and fracture toughness

Suppose the load on the specimen in Figure 2.2 is increased until it fractures, i.e. the crack grows. The resistance to fracture may be characterized by the stress intensity at fracture K_c, called the *fracture toughness*. The fracture toughness and the manner in which the crack grows are heavily dependent upon the material thickness.

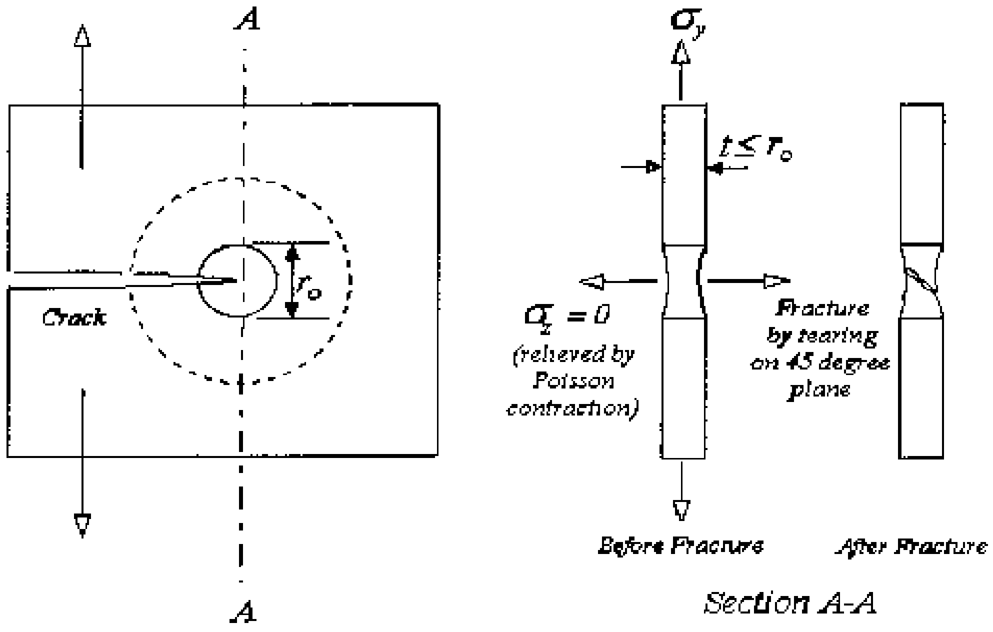

Figure 2.3 Plane stress fracture. Plastic zone diameter r_o comparable to or greater than sample thickness.

Consider a specimen having a thickness t that is small compared to the diameter of the plastic zone r_o, as shown in Figure 2.3. As the crack is pulled apart the plastic zone will undergo Poisson contraction, relieving stresses σ_z acting through the sample in the z direction. We call this situation *plane stress* because stresses are only acting in the x-y plane With σ_y large and σ_z near zero the shear stress on the 45° plane between the y and z axes is at a maximum The crack therefore tends to orient itself along this plane as it grows. This type of crack growth is usually stable and gradual and is characterized as *tearing*.

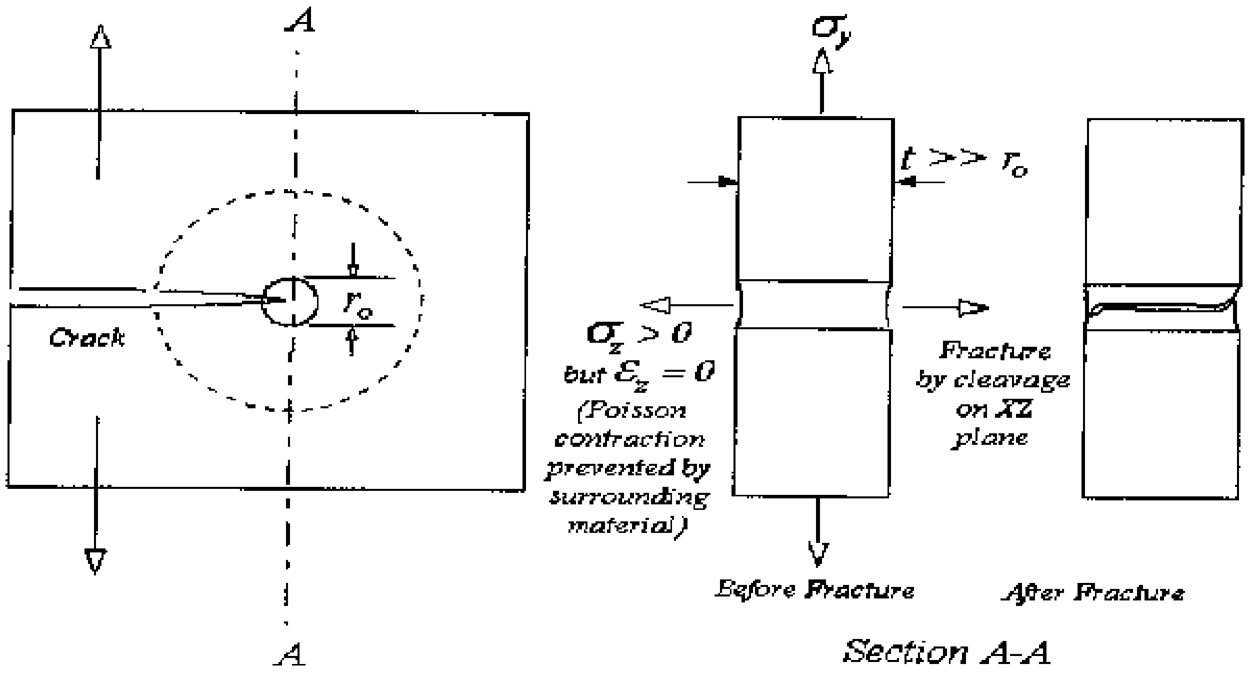

Figure 2.4 Plane strain fracture. Plastic zone diameter r_o much less than sample thickness.

Now consider a specimen having a thickness t that is large compared to r_o, as shown in Figure 2.4. As the crack is pulled apart the material above and below the plastic zone prevents Poisson contraction from occurring througout most of the sample This sets up large σ_z stresses in the plastic zone We call this situation *plane strain* because material is straining only in the x-y plane. With σ_z comparable to σ_y, the shear stresses are small so the crack tends to orient itself in a plane perpendicular to σ_y as it grows. This type of growth is usually unstable and is characterized as *cleavage*. Note that even with a thick sample there will be a thin regions close to its surfaces where Poisson contraction will take place and failure on 45° planes will occur.

Thicknesses between those that result in plane strain or plane stress are termed *mixed*, with plane strain occurring in the interior and plane stress some significant distance from the surface.

Because the Poisson contraction in a thin specimen relieves some of the stress, the fracture toughness of such a sample is relatively high. As the sample thickness increases and the form of the stress distribution changes the fracture toughness falls, asymptoting to a constant value for plane strain. Because of its independence of sample thickness this asymptote, termed the *plane strain fracture toughness* K_{Ic}, is considered a material property [6]

2.4 Interlaminar Fracture Toughness

It became a common practice to characterize the resistance to delamination using fracture mechanics. There is competing terminology in literature, such as fracture toughness, average fracture energy, J integral, WOF and critical strain energy release rate. The critical fracture toughness or critical strain energy release rate is the value at the onset of crack propagation. Crack propagation under pure mode-I (opening mode), pure mode-II (shearing or sliding mode) loading has been extensively studied in the literature, but more attention must be paid to mixed-mode I+II loading because it relates to most realistic situations. In fact, composite structures are generally subjected to combinations of mode I and mode II [7]. The contribution of mode III (tearing) fracture to delamination is small in comparison with modes I and II [8]. The critical strain energy release rate is also a common fracture mechanics terminology for the work rate with respect to crack growth [9]. Static and dynamic fracture behaviour of composites has been extensively studied by many authors. Static or dynamic toughness depends on the displacement rate of the crosshead, which loads the specimen. Quasi-static, dynamic and impact tests are intensively applied. Measuring mode I fracture toughness the stability of the crack growth depends on the test applied. Crack growth is stable when the specimen with a crack is loaded perpendicularly the fibers. By this method the crack initiation fracture toughness can be measured. In the DCB test the crack growth is stable, the first point is the initiation and the others are the crack growth fracture toughness. The characteristics obtained from different tests correspond to each other only if the fracture toughness is independent from the crack length. An increase in fracture toughness is caused by inaccuracy of the DCB test due to the effects of shear stresses and fiber bridging in the opposite sides of a crack [10] The DCB, WIF [11] and CT test [12, 13] for mode I are the accepted methods for measuring the interlaminar fracture thoughness (GIC). The WIF specimen, which is the compression version of the DCB specimen is more suitable to investigate the dynamic fracture properties of composites [11]. The CT specimen is suitable only to investigate the initiation damage due to the insufficient ligament length for further testing [9]. The DEN, SEN and SC specimen under uniaxial tension measures the mode I fracture toughness. The SEN and VIC specimen can be used to characterize also mode I fracture properties in 3PB or 4PB setup. The VIC specimen is mainly used in ceramic composites through the IS method [14, 15]. The stress intensity around the cracks is used to characterize the fracture properties. The SENB specimen is consistent with the SEN specimen in 3PB setup, while the SENT is the tensile mode SEN specimen. Both specimens measure the mode I fracture properties [16, 17] The SEVNB [18] is similar to the SEN specimen but it has V-shaped notch. The mode II fracture toughness is measured by ENF (3PB), 4ENF (4PB) and ELS (1PB) specimens. In the ENF specimen the crack growth is unstable and only one data per specimen can be obtained, initiation fracture toughness can be measured. Applying 4ENF test, crack growth is stable under displacement control and the complete R-curve can be determined from the data of just one test [19]. In contrast, splitting of UD specimens with central hole under longitudinal tensile is stable and the entire R-curve may be determined [12]. The R-curve is the relationship between the fracture toughness and crack length. In many work the R-curve behavior, R-curve resistance or J-resistance [13] were observed, which means that the fracture toughness increases with the crack size, this can be attributed to the fiber- bridging and fiber pullout during the fracture [20, 14]. These fibers increase the resistance to delamination. The UD tensile specimen with central hole also determines the mode II dominated fracture and damage properties of composite laminates. Notches and holes occur in many applications of composites and are likely sites of crack initiation and stress concentration, understanding the response in the presence of notches and holes is important in establishing a design methodology for a particular composite [9]. In literature the SCB, DENF, MMB, MMF and CLS specimens are presented [21]. The ELS test can be modified by moving the crack from the mid-plane, this will cause also mixed mode loading [8] However, the fracture strength of the composites after exposure to adverse environmental condition is yet to be substantially established.

2.5 General Experimental Procedure

Specimens can be manufactured by different methods and materials or the combination of composites with other materials. The mechanical properties of composite materials can be previously determined. In the fracture toughness tests rectangular shape panel has to be cut from the laminated prepregs. An articial starter crack or initial delamination can be generated in the specimen, using a non-adhesive Teflon or Al film to reduce the frictional force. The load can be applied through pins or Al blocks bonded to the specimen. Instron machine, servo-hydraulic or screw-driven machines [18] can be used to conduct static and quasi-static test with constant cross-head displacement. Dynamic and impact test can be presented by increasing the cross-head displacement rate, or using falling weight impact tower [20], Charpy test fixture [22], SHPB system [11] or dropweight impact tester [23]. Test conditions and the tested materials can be varied according to the aim of the investigation During the loading the specimen fractures, during the test the crack length and load/displacement curves can be measured. The crack length can be measured by visually or using TM [18, 24], to measure the surface damage path [9]. The fracture process can be followed by video camera and recorder to analyze the crack initiation and propagation [25]. The compliance/deflection curve is generated defining compliance as the ratio of maximum deflection and maximum load for each crack length. The crack length is defined as the mean values of measured on the left and right sides of the specimen during the test. The crack length can be measured by periodically stopping the load. The SEM is widespread to examine the microdamages in the specimens [26, 16]. The desired part of the specimen has to be polished and to facilitate the micro failure it is useful to coat the crack surface with gold or palladium [26, 23]

2.6 The effects of Temperature on Fracture Toughness

CELEMIN and LLORCA presented a study in which, the mechanical properties of *Al_2O_3/Nicalon SiC* composite were measured at ambient, intermediate and elevated temperatures [27]. The mode I fracture test were carried out by 3PB of notched bi-directional [0°/90°] prismatic bars, placed in a furnace to test at elevated temperature. The crosshead displacements were monitored through an LVDT. Significant decrease in the tensile strength and modulus were observed increasing the temperature. The nominal fracture toughness and the fracture energy decreased significantly from ambient to 800° C, and remained constant above this temperature. According to the SEM the average fiber pullout length in the specimens tested at 1000 and 1200° C was shorter than in those tested at 25° C. The fraction of fibers broken in the matrix crack plane was maximal at 800° C. The fiber strength and interfacial sliding resistance were estimated as a function of temperature through quantitative microscopy techniques. Both parameters remained constant up to 800° C, but decreased rapidly above this temperature. The composite strength was computed based on the assumption of global load redistribution takes place when fiber fracture occurs. The theoretical results are in good agreement with the experiments. The fracture energy were calculated based on the results of microscopy techniques. At 25° C the experiments agreed well with the model predictions, the damage zone propagated containing multiple cracks. At high temperature failure was localized around one single crack.

The thermal and mode I fracture behavior of three $Al_2O_3 - SiC_w$ composites and Al_2O_3 have been investigated in reference [28]. Composites, designated as *A30R, A30T and A30B* were manufactured. The"*R*" and"*T*" *SiC* grades were well-defined whiskers with mean diameter of 0.3 − 0.7μm, while the"*B*" grade material was coarser and more variable with mean diameter of 1−2μm. The thermal conductivity decreases increasing the temperature. The mode I fracture toughness of the composites was evaluated from the stress intensity of the *VICs*. The indentation load was varied. The crack was assumed to stop when the stress intensity at the crack tip equals to the fracture toughness of the material All composites exhibited higher fracture toughness values than the Al_2O_3 .The *A30R* composite exhibited R-curve behavior. The thermal shock resistance was also measured. Pre cracks were made with a Vickers indenter. The crack length-ΔT curve can be divided into three regimes. At low ΔT no significant crack growth can be observed. In medium ΔT interval stable crack growth occurred. At certain values of ΔT, some of the cracks grow unstably. The ranking of the composites and the Al_2O_3 according to thermal shock resistance agrees well with the ranking according to fracture toughness, but disagrees with the ranking according to thermal conductivity. These experiments indicate that the fracture toughness is very important in defining the thermal shock resistance.

An experimental and analytical investigation in cryogenic mode I interlaminar fracture behavior and toughness of *SL-E* woven glass-epoxy laminates was conducted in reference [29]. The DCB tests were performed with varied specimen lengths at *RT*, *4K* and *77K*. Low temperature environments were achieved by immersing the loading fixture and specimen in liquid *N* or *He*. Several G_I values for each specimen were obtained. The fracture toughness was determined using the MCC and the area method. At RT and 77K the G_I/crack length curves show, the G_{IC} are approximately constant in the crack length. The area method gives lower values than the *MCC* method. The load-displacement curve of *4K* is similar to that of *77K*, but it provides lower values. Stable crack growth occurred at *RT*, at *77* and *4K* the crack growth was unstable. No significant differences were observed between the values obtained by the *MCC* and the area method at *RT*. A 3D FEA using the quarter of the DCB was carried out. The energy release rate was evaluated by the global energy method. The FEA result averages about 25% lower than the area method result at *RT*. At cryogenic temperature the MCC overestimates the G_{IC}, but there is a good agreement between the results of the area and the MCC method. According to SEM, the crack generally progresses at the fiber-matrix interface. The dominant failure mode at cryogenic temperatures is interfacial failure.

The processing and mechanical properties of Al_2O_3/Ni_3Al *IPCs* were presented by SKIRL et al [30]. Composites with Ni_3Al contents of 15% to 30% were examined. Al_2O_3/Ni_3Al and Al_2O_3/Al composites were investigated experimentally. Strength measurements were made using rectangular specimens in 4PB for each microstructure at RT. High temperature mechanical testing were performed between 600° C and 1000° C. At RT and 800° C the strength was approximately constant against the content, but above the content value of 30% strength is dramatically increased. The fracture strength of composite with 20% content was constant against the temperature. Fracture toughness (K_{IC}) was measured with the same equipment using SEVNB specimen. At RT the fracture toughness increased almost linearly with the content of metal. At elevated temperature the composite exhibits lowest values of fracture toughness The fracture toughness of composite with content of 20% decreases with temperature. SEM shows debonding between the ceramic and Ni_3Al. The Young moduli were also measured, calculated with the rule of mixtures and another special theory. Experimental values clearly differ from the formers, the moduli of composites only with content of 30% corresponds to the theoretically estimated value.

The mechanical properties of $SiC\!-\!Mo_5(Si, Al)_3C$ composites with infiltrated phase were investigated and compared with *RB-SiC* and $SiC/MoSi_2$ composites by ZHU and SHOBU [15]. Fracture strength and toughness were measured experimentally up to

1600° C. The fracture strength was determined applying 3PB test. The average RT flexural strength and the standard deviation are similar and in the same interval in all the three types of composites. A significant increase in the strength can be observed between 1200 and 1600° C. The behavior is linear up to 1200° C and plastic deformation is observed at ≥1400° C. The $SiC-Mo_5(Si, Al)_3C$ composite has superior strength over the *RB-SiC* composite in the range of 1400 – 1600° C. The fracture toughness were determined by the *IS* method using the same equipment. The indentation was made by a Vickers-indenter. The fracture was initiated from the indent. The fracture toughness significantly increases at elevated temperatures; it was mainly attributed to the plastic deformation of the infiltrated phase of the composites. The influence of annealing in air was also studied and dramatic increase was observed in the fracture toughness

2.7 The Effects of Acidic Environment and Elevated Pressure

The effects of acidic stress environment on the stress intensity factor for GRP laminates were investigated by KAWADA and SRIVASTADA [24]. Specimens were used with the stacking sequence of [0°/90] and were exposed in *HCl* acid of various concentration and temperature. Constant tensile loading was applied; the crack length was measured by a TM. The rate of crack propagation was determined. The trend in acidic behavior is divided into two regions according to the crack propagation rate/K_I relation. First the crack propagation is converged where crack growth proceeds on a single plane, then stable crack propagation region is observed, the stress corrosion leads to fiber pullout and roughening of the fracture surface. The rate of crack propagation increased with temperature and concentration of acid.

The effects of pressure on the fracture behavior of rubbery particulate composite were investigated by MILLER and LIU [31]. SENT and SC specimens were used. The tests were conducted in a pressure chamber and crack growth in both the SENT and SC specimens, growth rate only in the SENT specimens were examined. The processes were followed by a video camera. In the SC specimen only crack width was measured. For the SENT specimens FEA was conducted to obtain stress intensities in each data point. The K_I was determined based on the J-integral. The initiation fracture toughness was defined as the stress intensity at the given point in the time, when crack growth first occurs. The overall initiation fracture toughness of the SENT specimen was determined using a regression plot at ambient and elevated pressure. No thickness effect on the fracture toughness was observed, so plane strain condition is apparent. The effect of pressure is to increase the K_C value by a factor of about 2. The crack speeds were obtained by fitting a polynomial curve into the points of each data set and using the derivatives. The constants of the power law then were obtained from these curves. The pressure increases the initiation fracture toughness and slows the void nucleation and growth in the composite.

Chapter 3

Specimen Preparation and Test Methods

3.1 Materials used

Resin Types

The resins that are used in fiber-reinforced composites are sometimes referred to as 'polymers'. All polymers exhibit an important common property in that they are composed of long chain-like molecules consisting of many simple repeating units. Manmade polymers are generally called 'synthetic resins' or simply 'resins'. Polymers can be classified under two types, 'thermoplastic' and 'thermosetting', according to the effect of heat on their properties.

Thermoplastics, like metals, soften with heating and eventually melt, hardening again with cooling. This process of crossing the softening or melting point on the temperature scale can be repeated as often as desired without any appreciable effect on the material properties in either state. Typical thermoplastics include nylon, polypropylene and ABS, and these can be reinforced, although usually only with short, chopped fibers such as glass.

Thermosetting materials, or 'thermosets', are formed from a chemical reaction in situ, where the resin and hardener or resin and catalyst are mixed and then undergo a non-reversible chemical reaction to form a hard, infusible product. In some thermosets, such as phenolic resins, volatile substances are produced as by-products (a 'condensation' reaction). Other thermosetting resins such as polyester and epoxy cure by mechanisms that do not produce any volatile by products and thus are much easier to process ('addition' reactions). Once cured, thermosets will not become liquid again if heated, although above a certain temperature their mechanical properties will change significantly. This temperature is known as the Glass Transition Temperature (Tg), and varies widely according to the particular resin system used, its degree of cure and whether it was mixed correctly. Above the Tg, the molecular structure of the thermoset changes from that of a rigid crystalline polymer to a more flexible, amorphous polymer. This change is reversible on cooling back below the Tg. Above the Tg properties such as resin modulus (stiffness) drop sharply, and as a result the compressive and shear strength of the composite does too. Other properties such as water resistance and color stability also reduce markedly above the resin's Tg [32].

Although there are many different types of resin in use in the composite industry, the majorities of structural parts are made with three main types, namely polyester, vinyl ester and epoxy [33].

Polyester Resins

Polyester resins are the most widely used resin systems, particularly in the marine industry. By far the majority of dinghies, yachts and workboats built in composites make use of this resin system.

Polyester resins such as these are of the 'unsaturated' type. Unsaturated polyester resin is a thermoset. capable of being cured from a liquid or solid state when subject to the right conditions. Unsaturated polyester differs from saturated polyester such as Terylene™, which cannot be cured in this way [34]. It is usual, however, to refer to unsaturated polyester resins as 'polyester resins', or simply as 'polyesters'. In chemistry the reaction of a base with an acid produces a salt. Similarly, in organic chemistry the reaction of an alcohol with an organic acid produces an ester and water.

By using special alcohols, such as a glycol, in a reaction with di-basic acids, a polyester and water will be produced. This reaction, together with the addition of compounds such as saturated di-basic acids and cross-linking monomers, forms the basic process of polyester manufacture. As a result there is a whole range of polyesters made from different acids, glycols and monomers, all having varying properties.

There are two principle types of polyester resin used as standard laminating systems in the composites industry. Orthophthalic polyester resin is the standard economic resin used by many people. Isophthalic polyester resin is now becoming the preferred material in industries such as the marine industry where its superior water resistance is desirable. Figure 3.1 shows the idealized chemical structure of a typical polyester. Note the positions of the ester groups (CO - O - C) and the reactive sites (C* = C*) within the molecular chain.

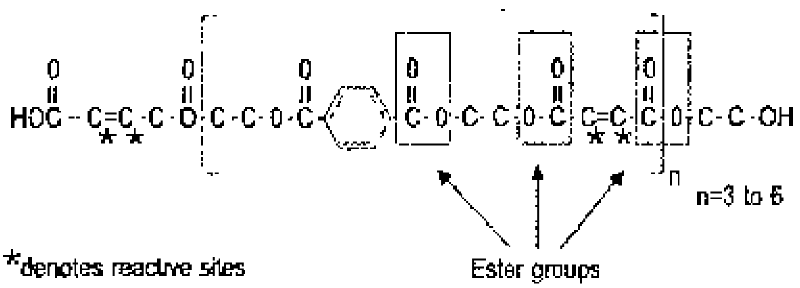

Figure 3.1 Idealized chemical structure of typical isophthalic polyester.

Most polyester resins are viscous, pale colored liquids consisting of a solution of polyester in a monomer, which is usually styrene. The addition of styrene in amounts of up to 50% helps to make the resin easier to handle by reducing its viscosity. The styrene also performs the vital function of enabling the resin to cure from a liquid to a solid by 'cross-linking' the molecular chains of the polyester, without the evolution of any by-products. These resins can therefore be moulded without the use of pressure and are called 'contact' or 'low pressure' resins. Polyester resins have a limited storage life as they will set or 'gel' on their own over a long period of time. Often small quantities of inhibitor are added during the resin manufacture to slow this gelling action. For use in molding, a polyester resin requires the addition of several ancillary products. These products are generally Catalyst, Accelerator and Additives.

A manufacturer may supply the resin in its basic form or with any of the above additives already included. Resins can be formulated to the molders requirements ready simply for the addition of the catalyst prior to moulding. As has been mentioned, given enough time an unsaturated polyester resin will set by itself. This rate of polymerization is too slow for practical purposes and therefore catalysts and accelerators are used to achieve the polymerization of the resin within a practical time period. Catalysts are added to the resin system shortly before use to initiate the polymerization reaction. The catalyst does not take part in the chemical reaction but simply activates the process. An accelerator is added to the catalyzed resin to enable the reaction to proceed at workshop temperature and/or at a greater rate. Since accelerators have little influence on the resin in the absence of a catalyst they are sometimes added to the resin by the polyester manufacturer to create a 'pre-accelerated' resin. The molecular chains of the polyester can be represented as follows (Figure 3.2), where 'B' indicates the reactive sites in the molecule.

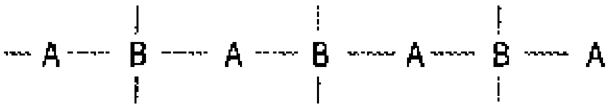

Figure 3.2 Schematic representation of polyester resin (uncured).

With the addition of styrene 'S', and in the presence of a catalyst, the styrene cross links the polymer chains at each of the reactive sites to form a highly complex three-dimensional network as represented in Figure 3.3.

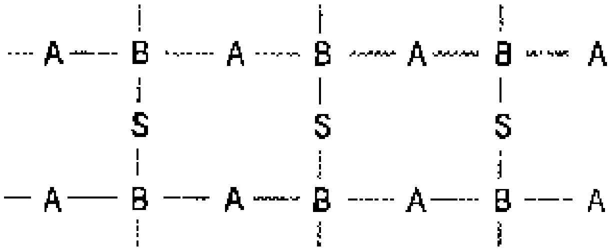

Figure 3.3 Schematic representation of polyester resin (cured).

The polyester resin is then said to be 'cured'. It is now a chemically resistant (and usually) hard solid. The cross-linking or curing process is called 'polymerization'. It is a non-reversible chemical reaction. The 'side-by-side' nature of this cross-linking of the molecular chains tends to mean that the polyester laminates suffer from brittleness when shock loadings are applied.

Great care is needed in the preparation of the resin mix prior to moulding. The resin and any additives must be carefully stirred to disperse all the components evenly before the catalyst is added. This stirring must be thorough and careful as any air introduced into the resin mix affects the quality of the final moulding. This is especially so when laminating with layers of reinforcing materials as air bubbles can be formed within the resultant laminate, which can weaken the structure. It is also important to add the accelerator and catalyst in carefully measured amounts to control the polymerization reaction to give the best material properties. Too much catalyst will cause too rapid a gelation time, whereas too little catalyst will result in under-cure. Coloring of the resin mix can be carried out with pigments. The choice of a suitable pigment material, even though only added at about 3% resin weight, must be carefully considered, as it is easy to affect the curing reaction and degrade the final laminate by use of unsuitable pigments.

Filler materials are used extensively with polyester resins for a variety of reasons including:

- To reduce the cost of the moulding
- To facilitate the moulding process
- To impart specific properties to the moulding

Fillers are often added in quantities up to 50% of the resin weight although such addition levels will affect the flexural and tensile strength of the laminate. The use of fillers can be beneficial in the laminating or casting of thick components where otherwise considerable exothermic heating can occur. Addition of certain fillers can also contribute to increasing the fire-resistance of the laminate.

Resin Comparison Summary

The polyesters, vinyl esters and epoxies discussed here probably account for some 90% of all thermosetting resin systems used in structural composites. In summary the main advantages and disadvantages of each of these types are summarized in Table 1[35].

Table 3.1 Advantages and Disadvantages of Common Thermosetting Resins Used in Structural Composites

POLYESTER: Advantages	**Disadvantages**
• Easy to use • Lowest cost of resins available	• Only moderate mechanical properties • High styrene emissions in open moulds • High cure shrinkage • Limited range of working times
VINYL ESTER: Advantages	**Disadvantages**
• Very high chemical/environmental resistance • Higher mechanical properties than polyesters	• Post cure generally required for high properties • High styrene content • Higher cost than polyesters • High cure shrinkage
EPOXIES: Advantages	**Disadvantages**
• High mechanical and thermal properties • High water resistance • Long working times available • Temperature resistance can be up to 140°C wet and 220°C dry • Low cure shrinkage	• More expensive than vinyl esters • Critical mixing • Corrosive handling

Fibers

Fibers are the principal constituent in a fiber-reinforced composite material. They occupy the largest volume fraction in a composite laminate and share the major portion of the load acting on a composite structure. Proper selection of the type, amount, and orientation of fibers is very important. since it influences the following characteristics of a composite laminate:

- Specific gravity
- Tensile strength and modulus
- Compressive strength and modulus
- Fatigue strength as well as fatigue failure mechanisms
- Electrical and thermal conductivities
- Cost

Glass Fiber

Glass fibers are the most common of all reinforcing polymeric matrix composites. The principal advantages of glass fibers are low cost, high tensile strength, high chemical resistance, and excellent insulating properties. The disadvantages are low tensile modulus, relatively high specific gravity (among the commercial fibers), sensitivity to abrasion with handling, relatively low fatigue resistance, and high hardness.

The two types of glass fibers commonly used in the fiber-reinforced plastics are E-glass and S-glass. Another type known as C-glass, is used in chemical applications requiring greater corrosion resistance to acids than is provided by E-glass. E-glass has the lowest cost of all commercially available reinforcing fibers, which is the reason for its widespread use in the FRP industry. S-glass, originally developed for aircraft components and missile casings, has the highest tensile strength among all fibers in use. However, the compositional difference and higher manufacturing cost make it more expensive than E-glass [33].

Chopped Strand Mat

Chopped Strand Mat is made by chopping strands into 50mm length, randomly distributing, and binding in mat form with powder binder. Chopped Strand mat has good impregnation and is easy to remove bubble; it is widely used as reinforced for FRP.

Table 3.2 Typical properties of E-glass chopped strand Mat/Unsaturated polyester resin at various glass contents.

Resin: Glass Ratio (by wt)	1.855	2	2.25	2.5	2.75	3
Wt Frac. (%)	35.03	33.33	30.77	28.57	26.67	25.00
Vol Frac Fibre (%)	20.24	19.05	17.30	15.84	14.61	13.56
Laminate thickness (mm)	1.94	2.06	2.27	2.48	2.68	2.89
Tensile Str (N/mm^2)	103	97	88	81	75	69
Tensile Mod. (N/mm^2)	7224	6800	6175	5655	5216	4641

Table 3.3 Typical properties of E-glass woven roving/unsaturated polyester resin at various glass contents.

Resin: Glass Ratio (by wt)	0.85	1	1.15	1.3	1.5	1.65
Wt Frac. (%)	54.1	50.0	46.5	43.5	40.0	37.7
Vol Frac Fibre (%)	35.6	32.0	29.0	26.6	23.9	22.2
Laminate thickness (mm)	1.10	1.23	1.35	1.48	1.64	1.77
Tensile Str (N/mm^2)	227	204	185	169	152	141
Tensile Mod. (N/mm^2)	14539	13056	11848	10844	9743	9054

3.2 Composite Fabrication

This work has been carried out on a Polyester resin (268BQTN: a product of SHCP) and a hardener (Methyl Ethyl Ketone Per Oxide, commercially known as Butanox-50) reinforced by Chopped strand mat Glass fiber (CSM 501-300: a product of KCC). The composite specimens with two layers of embedded ply were fabricated in the laboratory at room temperature in the shape of a rectangular plate by Hand Lay UP (HLU) Technique. Ample precaution was taken to minimize voids in the material and maintain homogeneity. The plates were post-cured at 70°C for 4-h after 24-h curing at room temperature. The GFRC plate was found to have a thickness of 2.53mm and fiber volume fraction of 35% (±0.5%).

The fabrication process of GFRC by HLU is as follows:

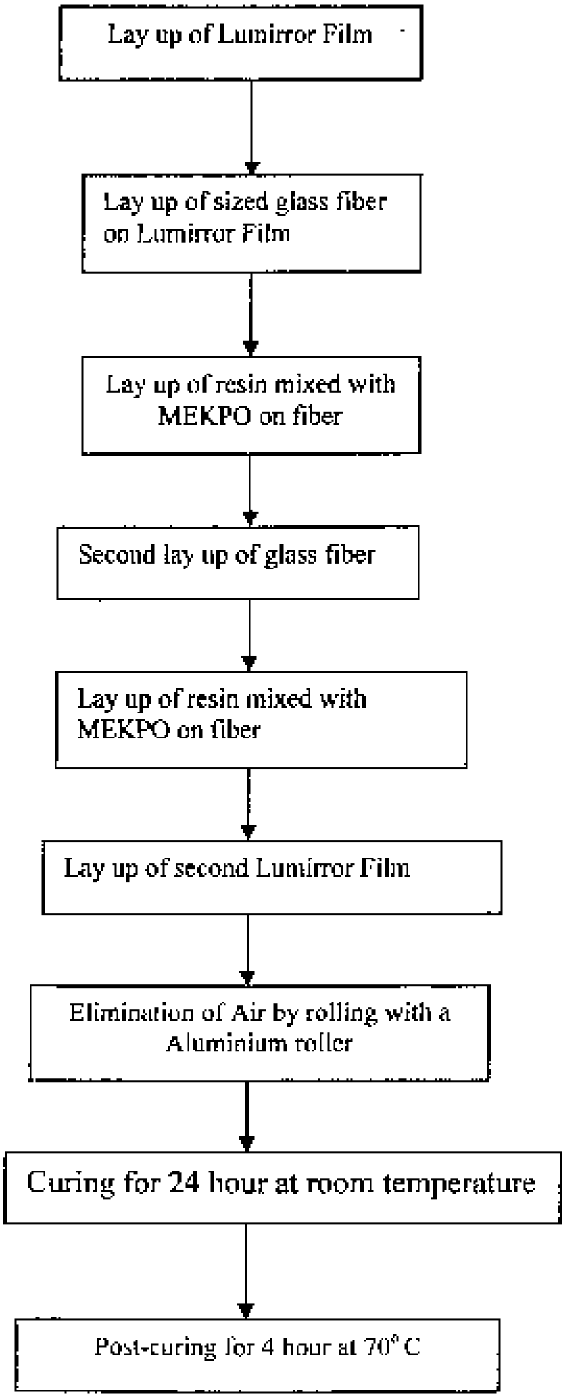

Figure 3.4 Fabrication Process of GFRC

3.3 Specimen Preparation

The specimens with ASTM Standard (D-3039) dimensions were prepared from the fabricated Glass fiber reinforced composite plate (Figure 3.5). The specimens were finally cut according to the sizes by a powered disc cutter. The sharp notch of different length sizes, namely, 2mm, 4mm, 6mm were cut in different specimens by the help of Surgical Blades (Size 22, Huaiyin Medical Instruments Co., Ltd., China), which provide a sufficiently sharp notch for valid fracture toughness testing for this class of material. Microscopic examination has been carried out to verify uniform length of the notches and the notch lengths have been readjusted as 2.25mm, 4mm and 5.725 mm. The required number of specimens with single edge sharp notch was made in the same way.

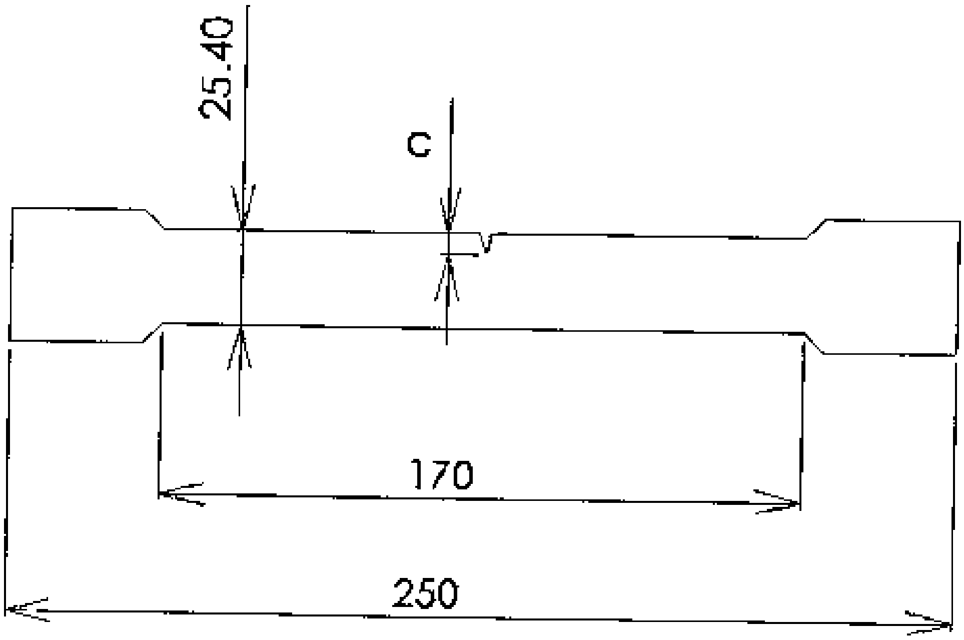

Figure 3.5 Dimension of the Specimen

(Dimensions in mm)

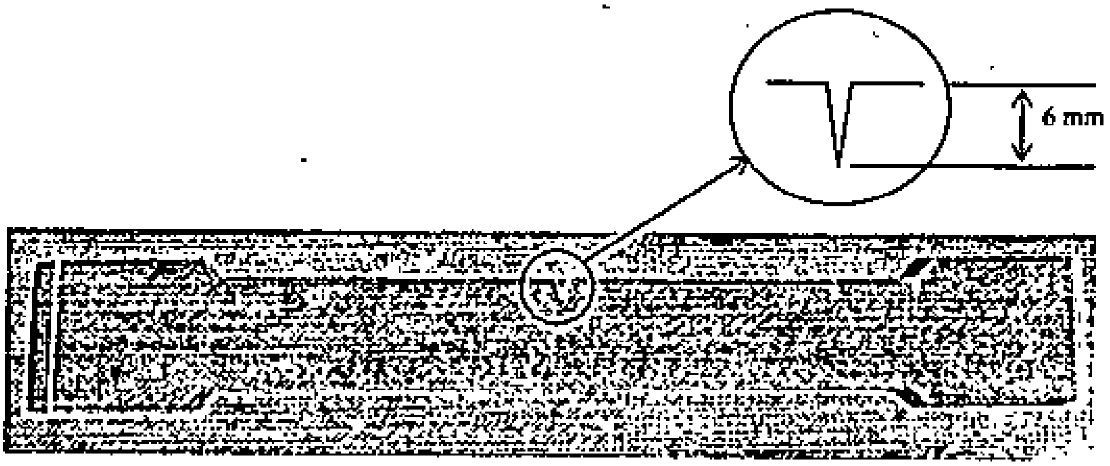

Figure 3.6 Specimen with notch length of 6 mm

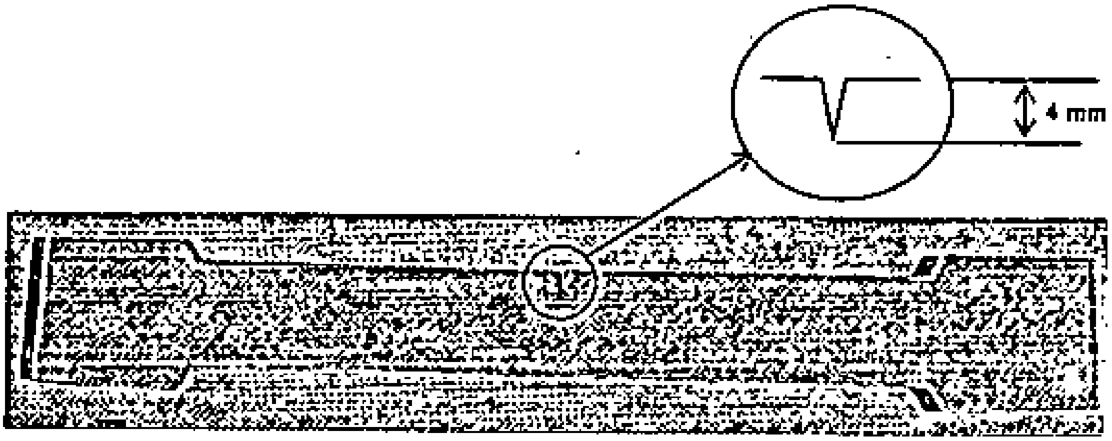

Figure 3.7 Specimen with notch length of 4 mm

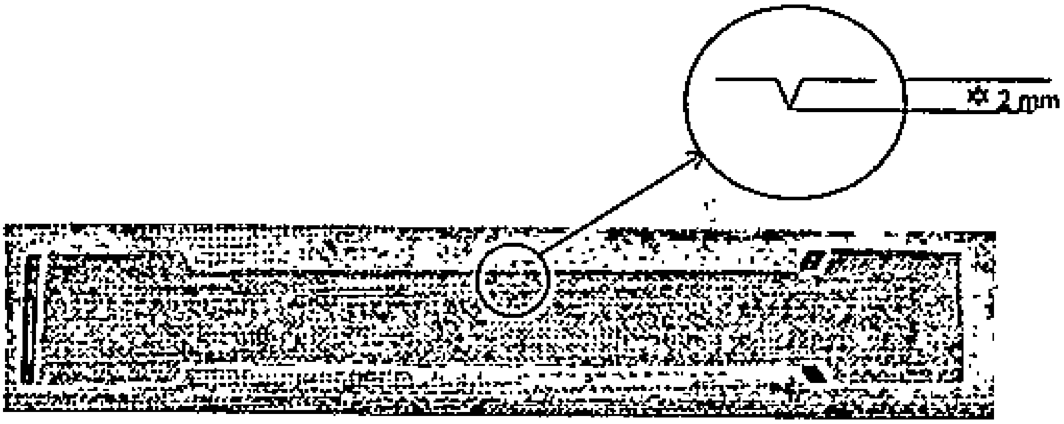

Figure 3.8 Specimen with notch length of 2 mm

3.4 Test Setup

Environmental Selection

In accordance to exposure of aerospace and ocean vehicles structures, the following six types of environment have been selected for the present investigation:

- Water; Pure distilled water has been used.
- Saline water : A mass of 200 mg of NaCl has been mixed in 1000ml of distilled water to prepare the saline water.
- Acidic water : Sulphuric acid of strength N/100 has been taken as acidic water. Here, distilled water has been used to prepare N/100 strength from concentrated H_2SO_4(80%).
- Organic fuel : Commercially available kerosene oil has been used.
- Low temperature : Ice temperature has been considered as the low temperature environment.
- High temperature: Specimens have been kept in a closed oven and the temperature has been monitored by a thermo-couple. The temperature was recorded as 60 ± 5^0C.

Fracture Toughness Test

The fracture toughness test on the specimens exposed to various environments has been carried out by applying tensile loading on a servo-hydraulic tensile testing machine (Universal Testing Machine, MIDDLE SENSTAR, Capacity: 5000N) for the specimen having a single edge sharp notch. The specimens were clamped in pin-loaded grips and subjected to monotonic uniaxial tension at a displacement rate of 1.50mm/min. The tests were closely monitored with rate of crack displacement. The load, at which the complete fracture of the specimens occurred, has been accepted as the critical load.

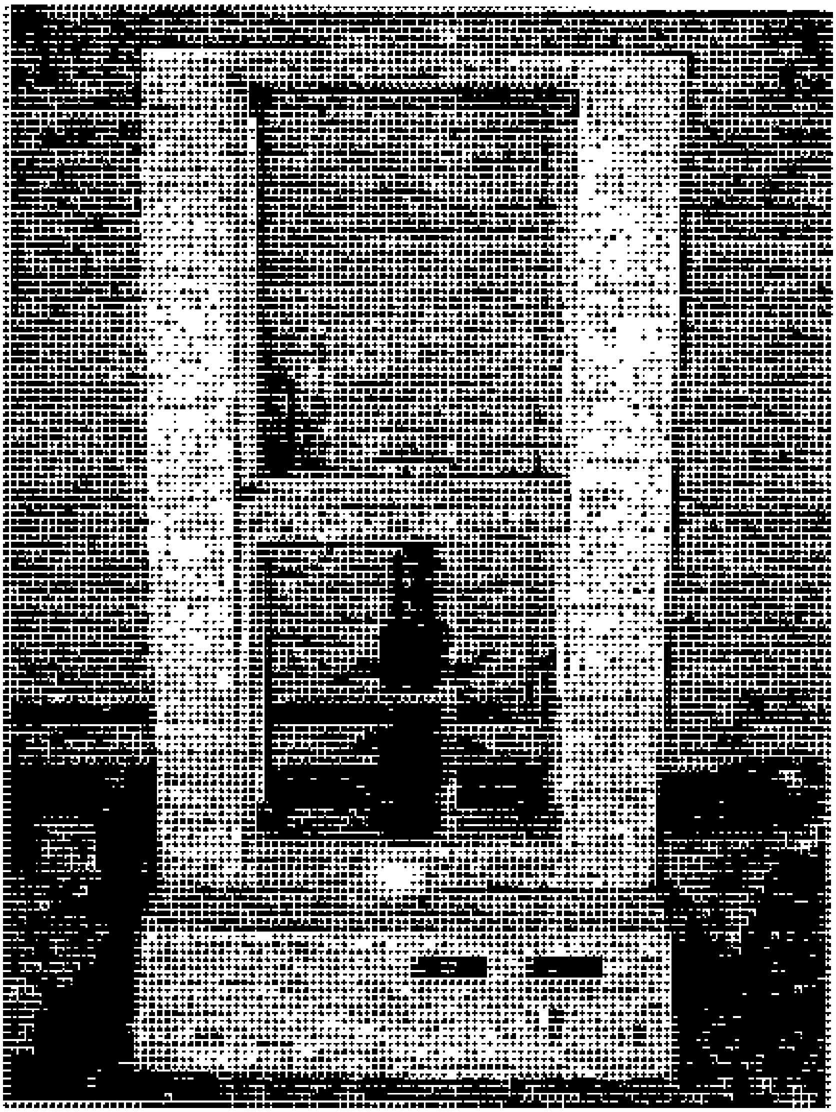

Figure 3.9 Universal Testing Machine

Chapter 4

Fracture Mechanism Analysis

4.1 Crack Growth Resistance

Many researchers [36-40] have used the linear elastic fracture mechanics (*LEFM*) approach for studying the crack growth resistance of fiber-reinforced composite materials. The *LEFM* approach [41], originally developed for metallic materials, is valid for crack growth with little or no plastic deformation at the crack tip. It utilizes the concept of stress intensity factor K_I, which is defined as

$$K_I = \sigma Y \sqrt{\pi C} \qquad (4.1)$$

where

K_I= mode *I* stress intensity factor

σ= applied stress

C= crack length

Y= geometric function that depends on the crack length, crack location, and mode of loading

Here, mode *I* refers to the opening mode of crack propagation due to an applied tensile stress normal to the crack plane.

Equation 4.1 shows that the stress intensity factor increases with both applied stress and crack length. An existing crack in a material may propagate rapidly in an unstable manner (i.e., with little or no plastic deformation) when the K value reaches a critical level. The critical stress intensity factor, also called the fracture toughness, indicates the resistance of the material to unstable crack growth.

4.2 Fracture Toughness Analysis

The stress field at the tip of a crack is characterized by a singularity of stress, and it decreases as a function of inverse square root of the distance from the crack tip. In this region, the stress field may, therefore, be regarded as the sum of three independent stress patterns depending upon the loads, and the dimensions and shape factors of the structure. Accordingly, the crack surface displacement may occur in three modes, namely, Mode I (opening mode) [where the crack surface displacements are normal to the crack plane], Mode II (forward mode) [where the crack surface displacements are in the crack plane normal to the crack border] and Mode III (parallel or anti-plane shear mode) [where the crack surface displacement are in the crack plane and parallel to the crack border] The determination of stress intensity factor has acquired considerable importance in the analysis of residual strength, fatigue crack growth rate and stress corrosion of composite materials, thereby in determination of strength of the material. The stress intensity factor may be defined in terms of amplitude or strength of the stress singularity and is dependent on state of stress at crack tip. The stress intensity factor is, therefore, obviously found to be dependent of radius of crack-tip, angle of crack and the distribution of the stresses for each mode, mentioned earlier. Thus, the applied load and size of the crack determines the stress intensity factor on the specimen. In the case of flat tensile fracture, in the forward mode (Mode II), as well as the parallel mode (Mode III), crack surface displacements are found to be generally absent and, therefore, the stress intensity factors, K_{II} and K_{III} may be assnmed to be zero. Thus, the stress intensity in the opening mode (Mode I) may be expressed by equation (4.1).

From equation (4.1), K_I provides the severity of the crack tip environment, and it is logical to characterize resistance to fracture by a critical value, that is, K_C Generally, K_C is determined by laboratory tests of the material.

By simplifying the stress field in the local direction, normal to the axis of the main crack, the stress on the ith ligament from the original crack tip with the main crack located at the edge of the jth ligament can be assumed as σ_{ij}. For the case of precut notch with no prior crack extension, the stress at the notch tip is σ_{11} and it can be determined by using the failure stress (σ_f), the stress intensity factor K_I and the critical stress intensity factor (K_C) can be expressed as[42]

$$\sigma_{11} = \sigma_f\left(\frac{K_1}{K_c}\right) \qquad (4.2)$$

The crack tip extends gradually under increasing load before main crack extension occurs and its extension diminishes the local stress σ_{11}. Equation (4.2) is equivalent to the assumption of a stress concentration at the crack tip and can be written as

$$\frac{\sigma_{11}}{\sigma} = \frac{\sigma_f Y \sqrt{C}}{K_c} \qquad (4.3)$$

From classical fracture mechanics relationship, equation (4.3) in the form of stress intensity factor can be expressed as[42]

$$K_I = \sigma Y \sqrt{C} \qquad (4.4)$$

The K_I calculated through equation (4.4) is also called as the fracture toughness of the material. For a pre-crack length 'C' and maximum applied stress σ, the parameter 'Y' is a geometrical factor, which accounts for proximity effects of boundaries or other cracks, orientation of the crack, shape of the crack and the restraints on the structure containing the crack, and is usually determined by a simple relationship [43] as explained below

$$Y = 1.99 - 0.41\left(\frac{C}{W}\right) + 18.70\left(\frac{C}{W}\right)^2 - 38.48\left(\frac{C}{W}\right)^3 + 53.85\left(\frac{C}{W}\right)^4 \qquad (4.5)$$

4.3 Environmental Exposure

The pre-notched specimens were divided in seven groups for exposure to the above mentioned six different environments and for without environmental effect study. Specimens of 72 in a group were exposed in each type of environment for a fixed duration, which was kept as 100-h, 200-h, 300-h and 400-h. The rate of environmental effect has been calculated for each exposed specimen in the form of percentage of weight gain (or loss) by using the following equation

$$\text{Weight gain (\%)} = \frac{M_f - M_i}{M_i} \times 100 \qquad (4.6)$$

The average of percentage weight change of the specimen in a given environment for different duration has been calculated using six specimens for each case.

Chapter 5

Results and Discussions

5.1 Fracture Toughness after Environmental Exposure

The mechanical properties of chopped strand mat glass fiber reinforced composite have been measured by conventional methods and given in Table 5.1 for ready reference.

Table 5.1 Mechanical properties of chopped strand mat Glass fiber/Polyester composite

Type of Fiber	Tensile Modulus, GPa	Tensile Strength, MPa
CSM 501-300	3.721	68.31

Table 5.2 Test results of average fracture toughness after various environmental exposures for different durations of exposure

Duration of Exposure	Environments						
	Virgin, MPa $\sqrt{mm}$	Water, MPa $\sqrt{mm}$	Saline Water, MPa $\sqrt{mm}$	Low Temperature, MPa $\sqrt{mm}$	High Temperature, MPa $\sqrt{mm}$	Acidic Water, MPa $\sqrt{mm}$	Organic Fuel, MPa $\sqrt{mm}$
100 hours	223.3	213.78	213.89	210.93	215.1	204.84	216.56
200 hours	223.3	204.98	207.56	202.32	208.8	197.11	208.9
300 hours	223.3	199.56	201.67	192.98	202.74	189.21	205.13
400 hours	223.3	194.97	197.94	185.13	199.47	181.26	202.12

Table 5.3 Percentage variation of Fracture Toughness after exposure into various environments

Types of Environments	Fracture Toughness, %			
	100 hours	200 hours	300 hours	400 hours
Virgin	100	100	100	100
Water	95.74	91.80	89.37	87.31
Saline Water	95.79	92.95	90.31	88.64
Low Temperature	94.46	90.60	86.42	82.91
High Temperature	96.33	93.51	90.79	89.33
Organic Fuel	96.98	93.55	91.86	90.52
Acidic Water	91.73	88.27	84.73	81.17

Table 5.4 Percentage of Weight Gain after various environmental exposures for different durations of exposure

Duration of Exposure	Environments					
	Water, %	Saline Water, %	Low Temperature, %	High Temperature, %	Acidic Water, %	Organic Fuel, %
100 hours	1.66	0.43	1.18	-0.02	0.35	0.05
200 hours	1.95	0.68	1.87	-0.05	0.62	0.09
300 hours	2.22	0.73	2.48	-0.16	0.84	0.13
400 hours	2.36	0.74	2.94	-0.29	1.05	0.17

In the present investigation, extensive studies have been carried out to find the fracture toughness of chopped strand mat glass fiber reinforced composite and the extent of its degradation by exposure to different environments up to 400-h. Fracture toughness obtained after exposing to various environments is given in Table 5.2. Percentage variation of fracture toughness after exposing to various environments has been shown in Table 5.3. Percentage of weight gain after various environmental exposures for different durations has been shown in Table 5.4. Each value is an average of six specimens.

5.2 Effects of Notch Size on Fracture Toughness

The fracture toughness of sharply, pre-cut, and single edge notched specimen of chopped strand mat glass fiber/ polyester composite has been shown in Figure 5.1 for various notch sizes. It is observed that the fracture toughness remains constant, irrespective of notch size. Physical examination of the root of the notch during the loading was also made. It was found that a yielded zone grew with loading, which was followed by a fine crack nucleating at the tip of this zone.

The failure of specimen can be attributed to the generation and propagation of the crack. While the size of the notch would generate tensile stress of varying magnitude, the specimens were found to exhibit almost equal fracture toughness. The geometric factor, *Y*, also varies with the size of the notch, and this is responsible for the observed result.

The specimens were found to fail under tension by separating into two halves at the point of notch in the form of controlled fracture. The necessary study for fracture is supplied either by the movement of point of stress concentration on tensile loading or by elastic relaxation of the material in the form of crack, such that the interface at the notch absorbs a large amount of energy.

The failure strain of the fibers must be higher than that of the brittle matrix, causing the bridging of matrix crack, where the fiber fractured at some distance on the crack plane and pulled out of the matrix at the interface as the crack separated. In the process, the load on crack bridging fibers was increased resulting into debonding of the interface between the fibers and the matrix. Since higher stress is borne by the crack bridging, the fibers would also absorb strain energy. The strain energy at the fracture point would be dissipated into the composite, thus is not available to assist the propagation of the primary crack. Therefore, the initiation of composite failure is considered as a cumulative failure of the fibers within the composite with increasing values of strain.

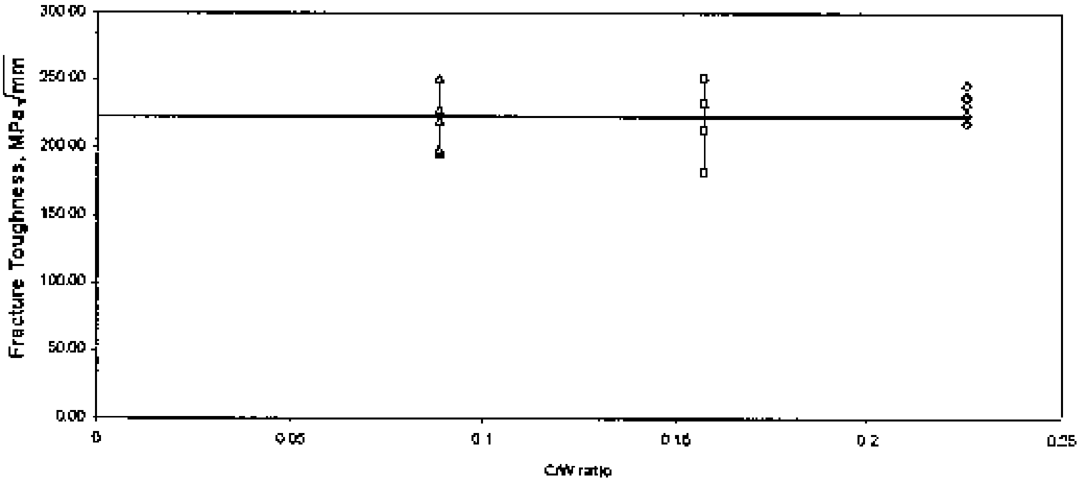

Figure 5.1 Fracture toughness of Chopped strand mat Glass Fiber/Polyester composite with various notch sizes

In addition, the maximum interfacial shear strain developed at the surface due to high interfacial shear stress may be taken to initiate the failure on interfacial bond, propagating the debonded region on the line of fiber as an interfacial crack. Frictional contact of debonded interface would influence the residual stress and produce differential thermal contraction of fiber and matrix (both longitudinal and lateral) depending on Poisson's ratio effect. Thus, the lateral expansion of the matrix is restrained by the presence of adjacent fibers, where the fiber and matrix are still being held in contact.

Some other factors like, the fiber surface roughness, weaving structure and micro mechanical properties of composite determines the fiber fracture stress for a particular embedded length, and is influenced by the fracture toughness of the composite. These effects are more aggravated due to various environmental exposures The fracture toughness test results of chopped strand mat Glass Fiber/Polyester composite with various notch sizes and different duration of environmental exposure have been shown in Figures 5.2-5.8.

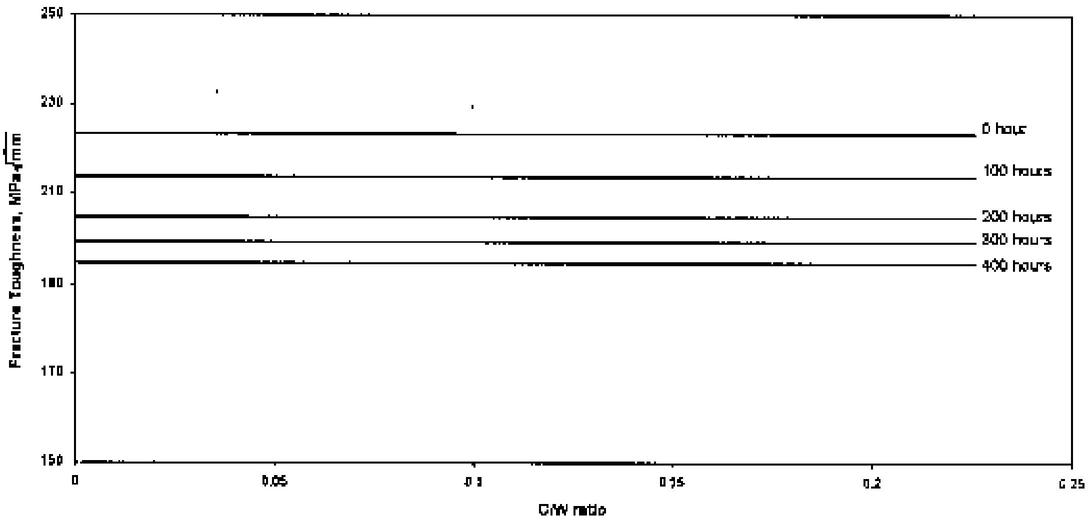

Figure 5.2 Fracture toughness of Chopped strand mat Glass Fiber/Polyester composite after exposure into water

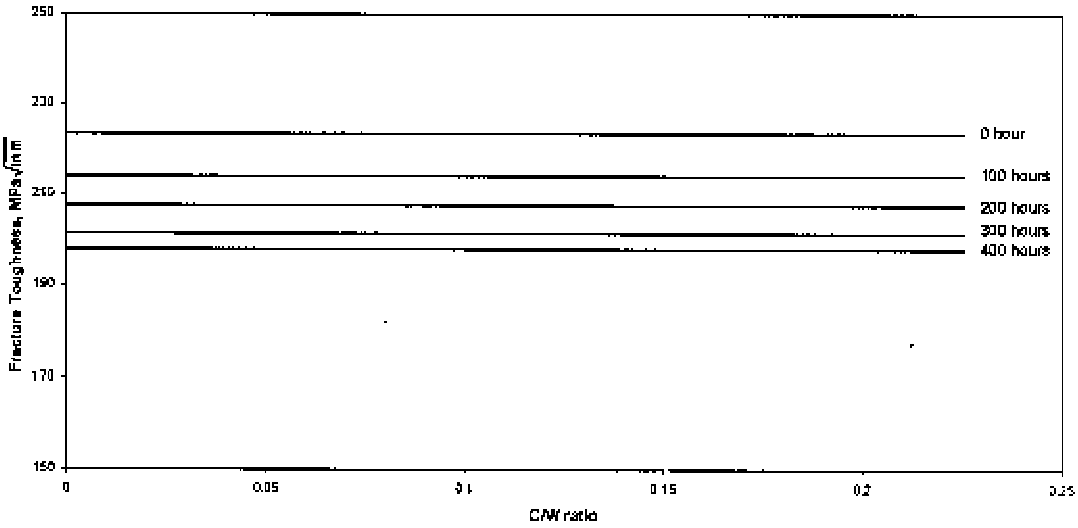

Figure 5.3 Fracture toughness of Chopped strand mat Glass Fiber/Polyester composite after exposure into saline water

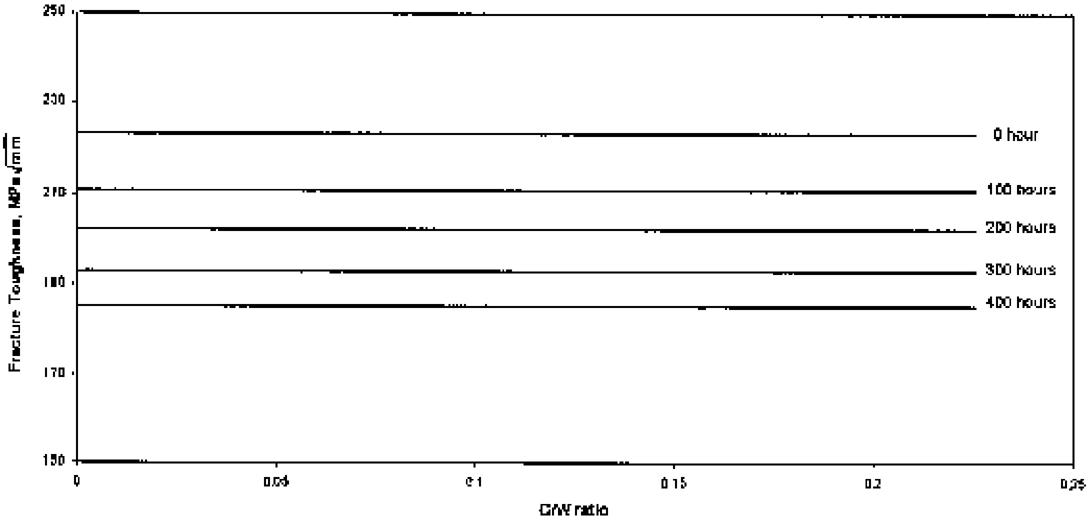

Figure 5.4 Fracture toughness of Chopped strand mat Glass Fiber/Polyester composite after exposure into low temperature(ice)

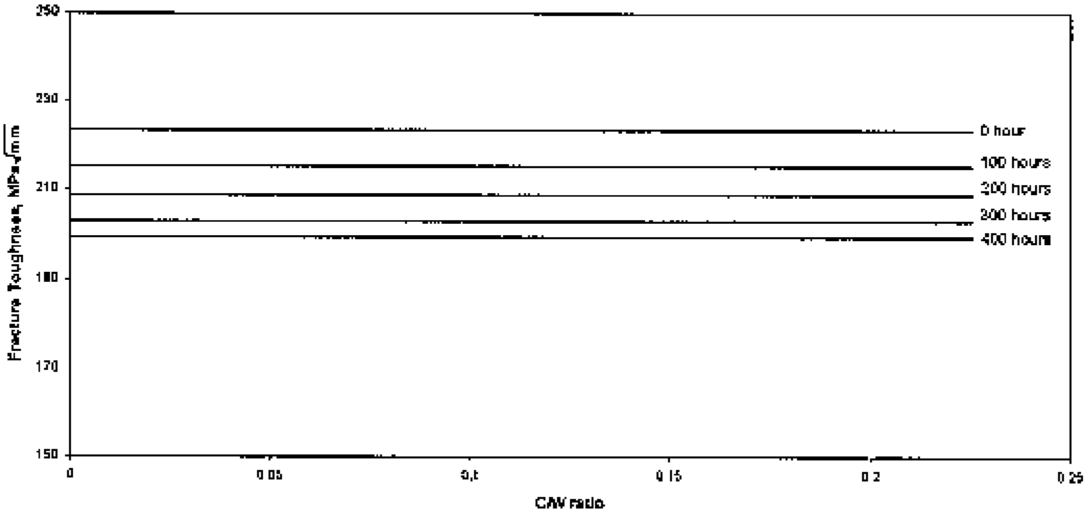

Figure 5.5 Fracture toughness of Chopped strand mat Glass Fiber/Polyester composite after exposure into High temperature(60° C)

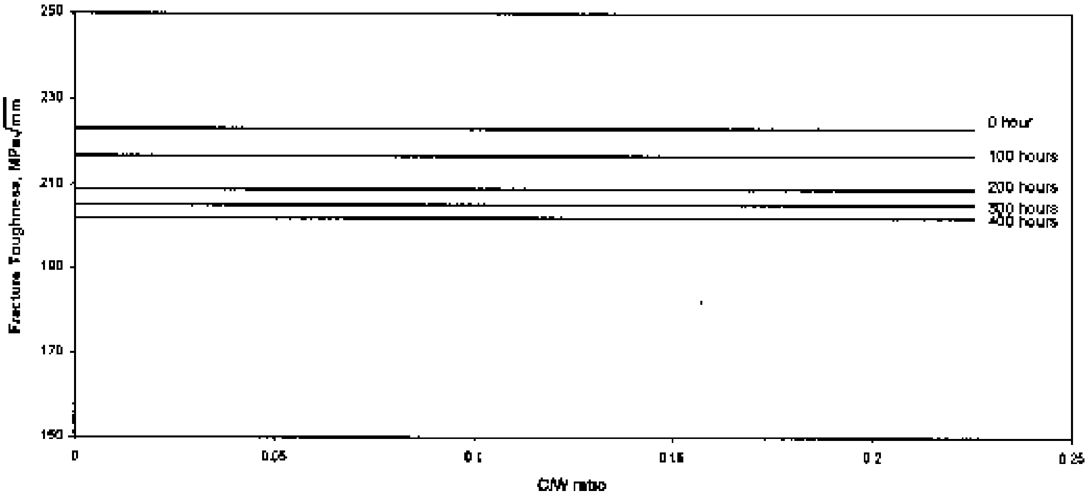

Figure 5.6 Fracture toughness of Chopped strand mat Glass Fiber/Polyester composite after exposure into organic fuel

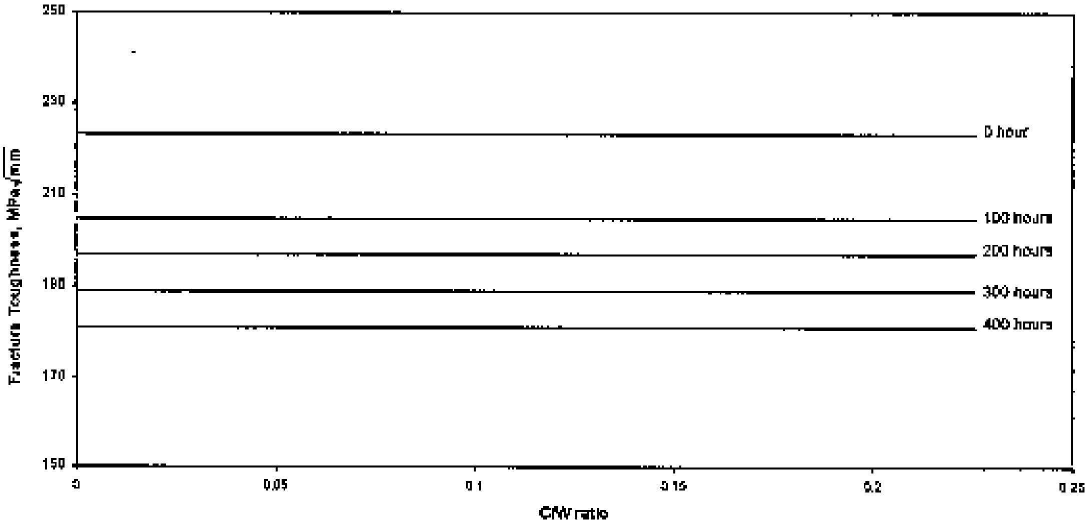

Figure 5.7 Fracture toughness of chopped strand mat Glass Fiber/Polyester composite after exposure into acidic water

5.3 Effects of Environment on Fracture Toughness

The results of fracture toughness after different durations of environmental exposure and percentage weight gain in same period by the composite in different environments are shown in Figures 5.8-5.15. The highest amount of degradation in fracture toughness has been recorded in acidic water (18.83% after 400 hours) and then in low temperature (17.09% after 400 hours) followed by water, saline water, high temperature and organic fuel (9.48% after 400 hours).

The highest amount of weight gain has been recorded in low temperature (2.94% after 400 hours) and then in water (2.36% after 400 hours) followed by acidic water, saline water and organic fuel. Weight loss of 0.29% has been recorded after exposing into high temperature for 400 hours. For first two hundred hours of exposure, water exposed specimens gained more weight (1.95%) than specimens exposed into low temperature (1.87%) and specimens exposed into saline water gained more weight (0.68%) than specimens exposed into acidic water (0.62%). For the next two hundred hours of exposure, specimens exposed into low temperature gained more weight (2.94%) than water exposed specimens (2.36%).

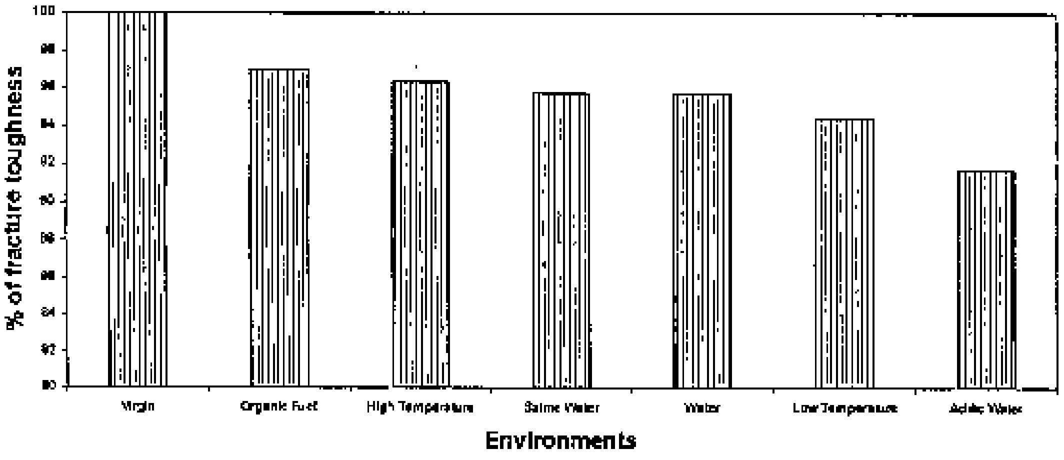

Figure 5.8 Percentage of fracture toughness after exposure into various environments for 100 hr

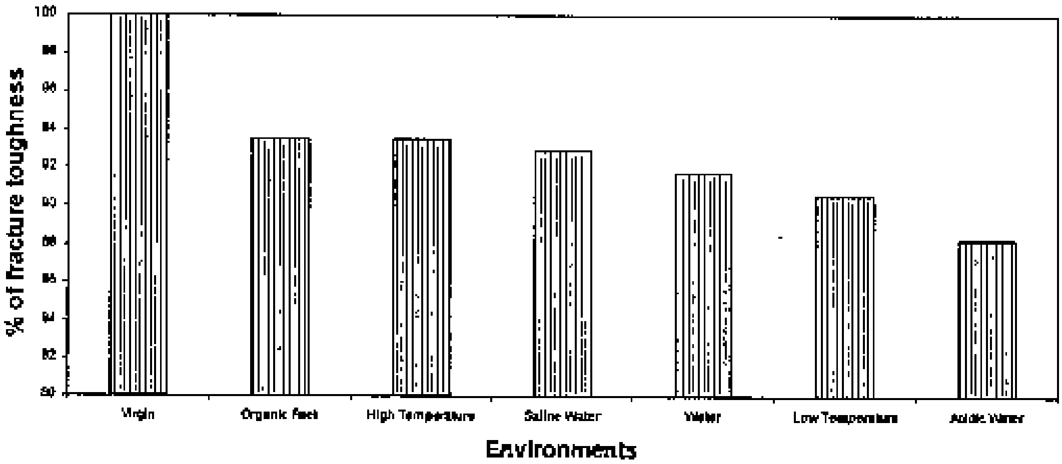

Figure 5.9 Percentage of fracture toughness after exposure into various environments for 200 hr

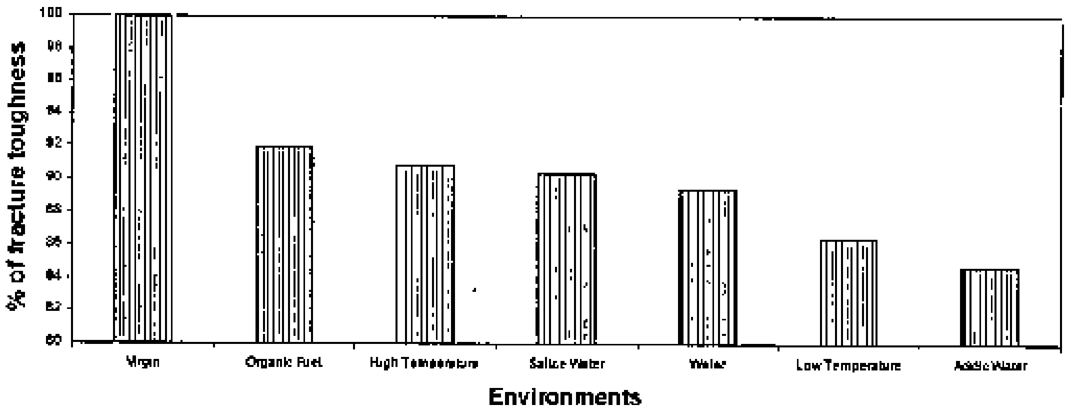

Figure 5.10 Percentage of fracture toughness after exposure into various environments for 300 hr

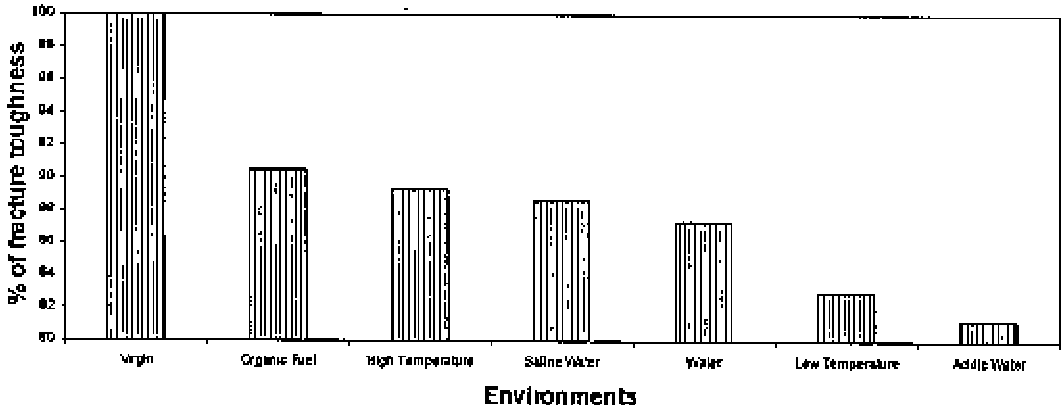

Figure 5.11 Percentage of fracture toughness after exposure into various environments for 400 hr

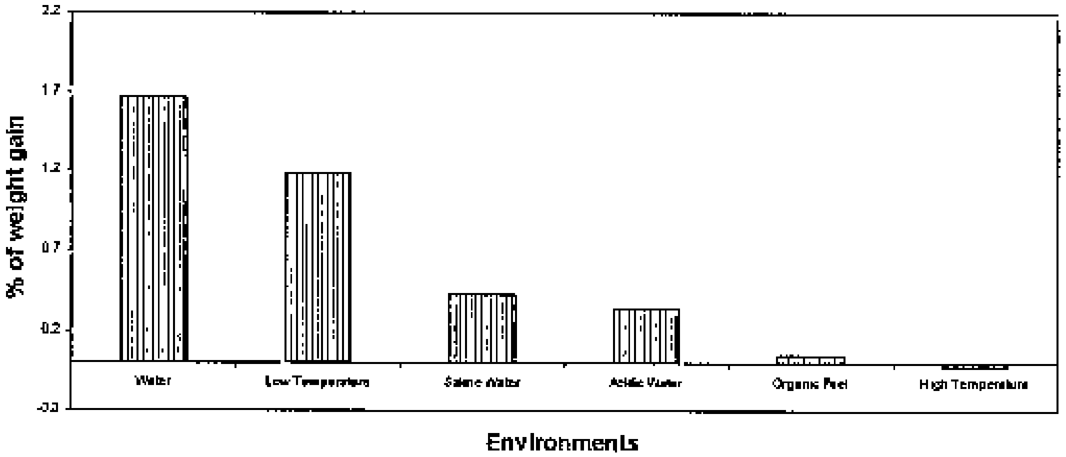

Figure 5.12 Percentage of weight gain after exposure into various environments for 100 hr

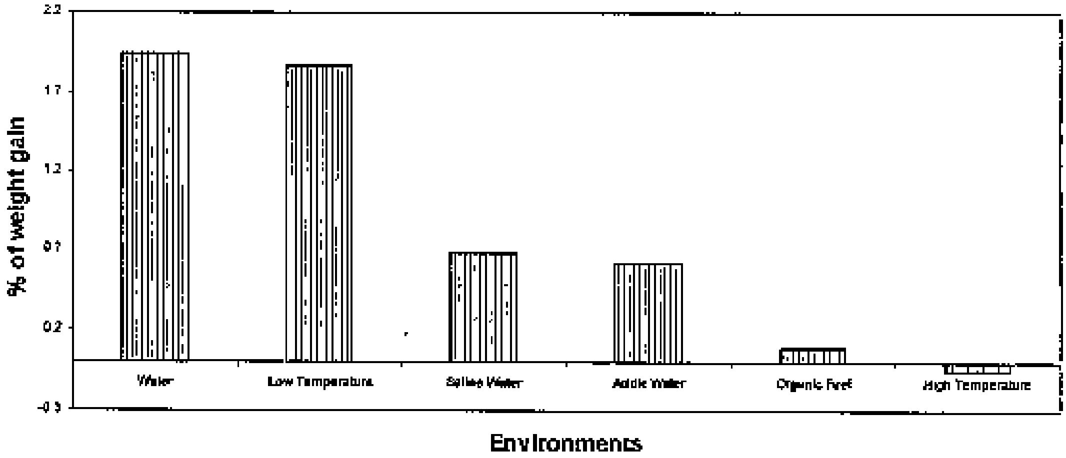

Figure 5.13 Percentage of weight gain after exposure into various environments for 200 hr

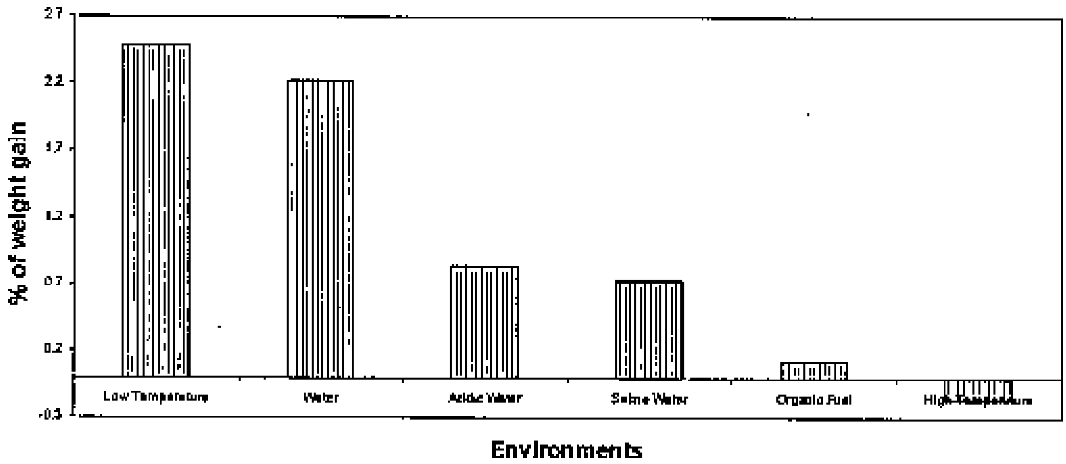

Figure 5.14 Percentage of weight gain after exposure into various environments for 300 hr

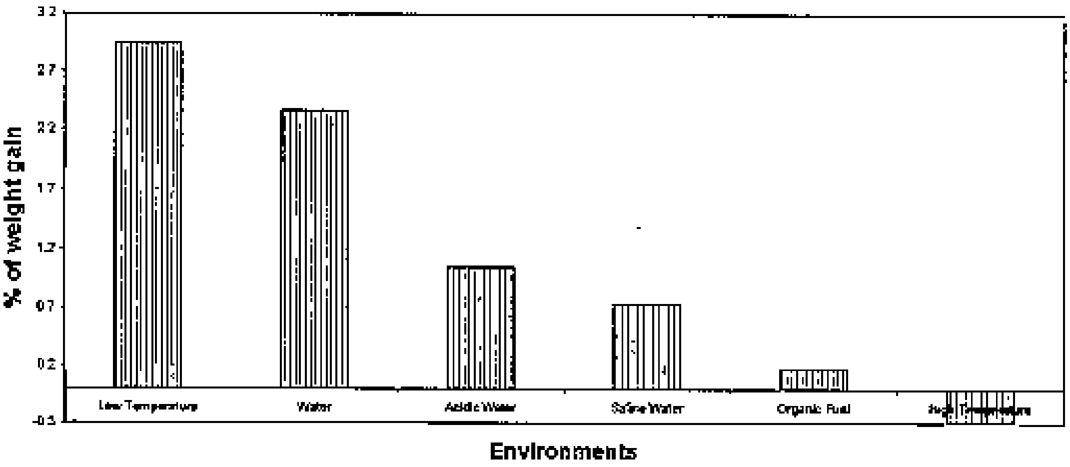

Figure 5.15 Percentage of weight gain after exposure into various environments for 400 hr

The results of fracture toughness after different durations of environmental exposure and percentage weight gain in same period by the composite in similar environments are shown in Figures 5.16-5.21. An attempt has also been made to explore relationship between percentage weight gain and fracture toughness strength.

For the water-exposed specimens for exposure of 400-h (Figure 5.16), it has been observed that there is 12.69% degradation in fracture toughness, while a weight gain of 2.36% has been recorded in the same duration. The penetration of water molecules in the interface region through the voids of the composite may be the reason of weight gain. The osmotic pressure of the water molecule might have facilitated the entrance of water molecules into matrix phase, and may have expanded its boundary in due course. As a result, the bond strength of the fiber and matrix has been weakened and load-bearing strength of the composite has been reduced, and this caused the reduction in fracture toughness.

When the specimens are exposed to saline water, only 0.74% weight gain has been recorded after 400-h duration of exposure (Figure 5.17). The higher density of the liquid may be held responsible for lower penetration of the saline water as compared to water. The reduction in fracture toughness has been found to be at a lower value of 11.36% as compared to 12.67% for the case of water in the same duration, although the liquid penetration is almost less than one third. The reduction in fracture toughness should be taken as a simultaneous effect of corrosivity of saline water and liquid penetration into the material in this case.

The investigation has been carried out in two groups of temperatures–one is lower value of environmental temperature and the other at a higher value. At the lower value of temperature, that is, ice temperature, a notable rednction in the fracture toughness has been observed (Figure 5.18) and it is about 17.09% after 400-h duration of exposure, while 2.94% of weight gain has been recorded in same duration. In ice, the water is in state of transition phase and temperature is about 0°C. The low temperature of the environment has caused the shrinkage of the constituent phases of the chopped strand mat glass fiber/polyester composite and transition pressure has caused a higher order of water absorption. This is mainly due to dissimilar contraction of matrix and fiber in low temperature. In consequence, detachment has been observed at the fiber and matrix interface and development of microcrack, debonding and delamination in the interface region of fiber and matrix. The process is further aggravated by formation of more crack and more detachment at the interface when there is a prolonged exposure. Therefore, the fracture toughness has been reduced significantly.

When the specimens are exposed to hot temperature of 60°C, only 0.29% weight loss has been recorded after 400-h duration of exposure. High temperature may be held responsible for dissociation of matrix and weight loss of the specimens. The reduction in fracture toughness has been found to be at lower value of 10.67% as compared to

17.09% for case of low temperature in the same duration. The matrix behavior has been changed due to prolong exposure to hot temperature (60°C), and has ablated a fraction of matrix from the specimen. In addition, under high temperature the dissimilarity in the thermal coefficient behavior of matrix and fiber causes different expansion and contraction characteristics, resulting microcracks, debonding and delamination at the interface region similar to low temperature environment The ablation of matrix may be increased as temperature rises, or time increases and thermal stress produced over the specimen, which results the micro buckling of fiber. It is resulted to the lower value of fracture toughness, as shown in Figure 5.19. In the present case, the environmental temperature was kept quite below than the melting temperature of matrix. So less reduction in fracture toughness has been anticipated in comparison to quite high temperature (above than ablation temperature of matrix).

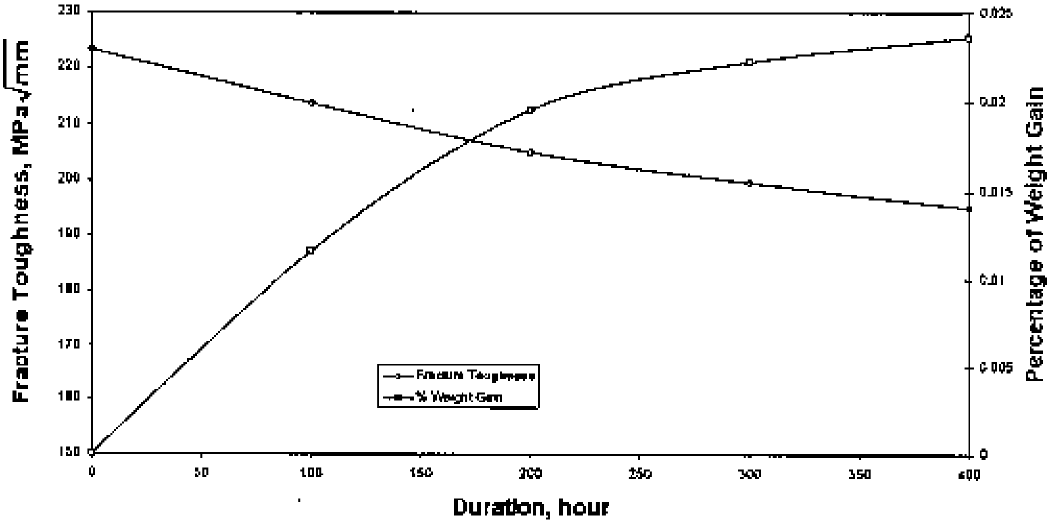

Figure 5.16 Degradation of fracture toughness after exposure in water in comparison to percentage weight gain

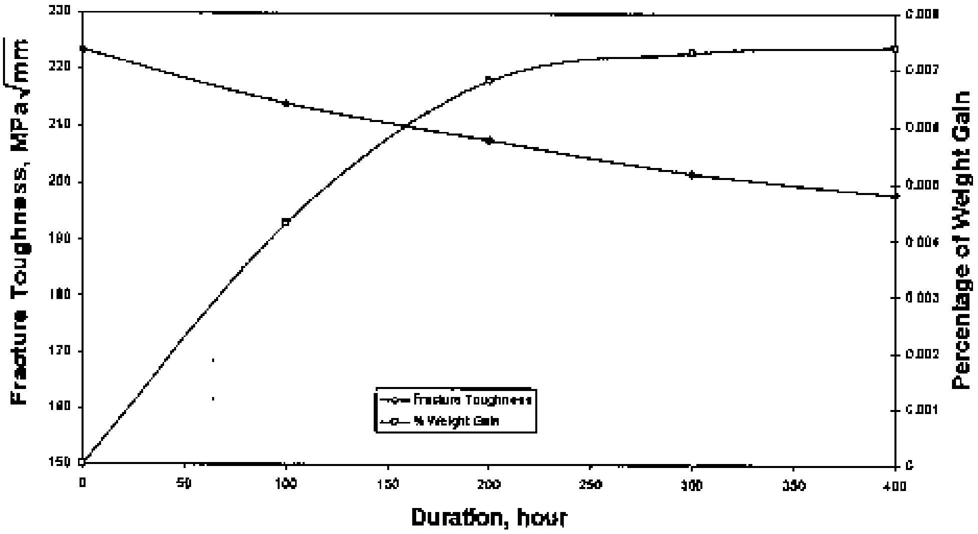

Figure 5.17 Degradation of fracture toughness after exposure in saline water in comparison to percentage weight gain

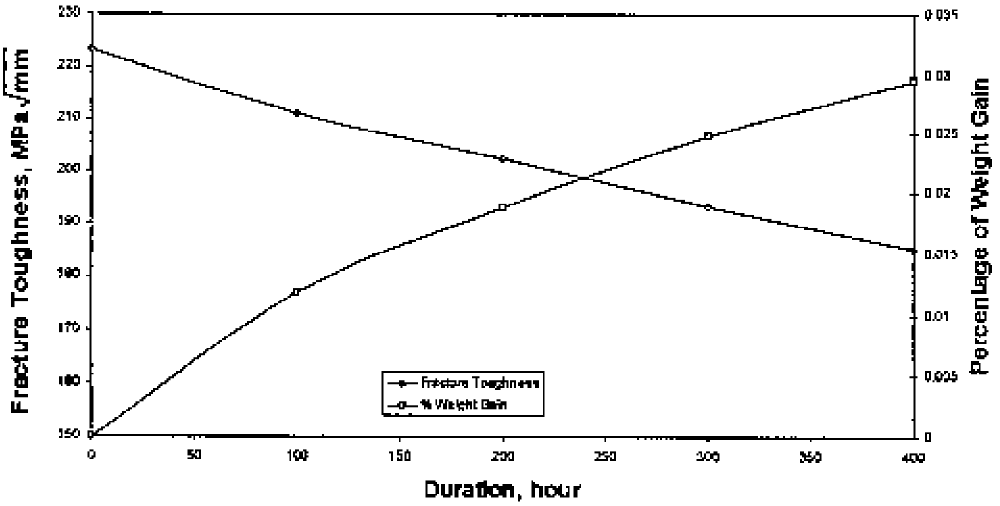

Figure 5.18 Degradation of fracture toughness after exposure in low temperature (ice) in comparison to percentage weight gain

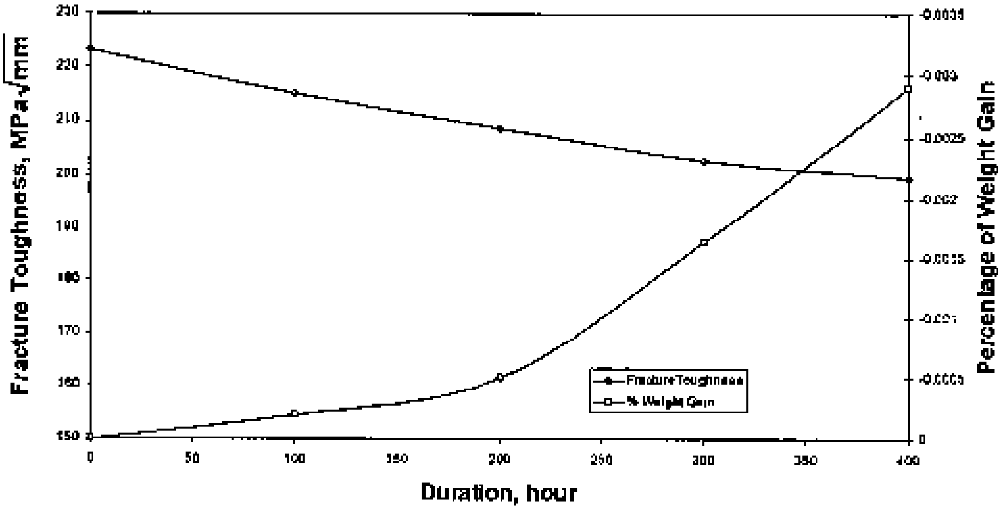

Figure 5.19 Degradation of fracture toughness after exposure in high temperature (60°C) in comparison to percentage weight gain

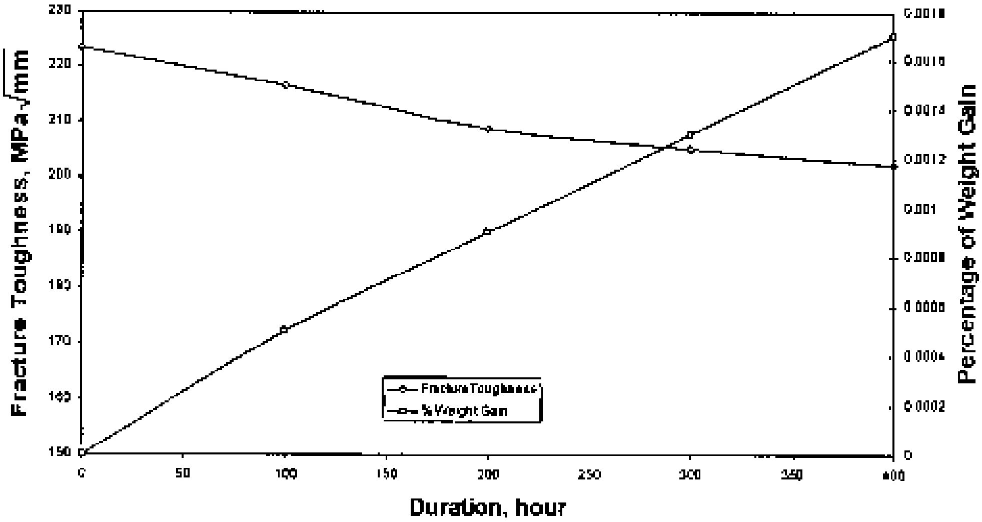

Figure 5.20 Degradation of fracture toughness after exposure in organic fuel in comparison to percentage weight gain

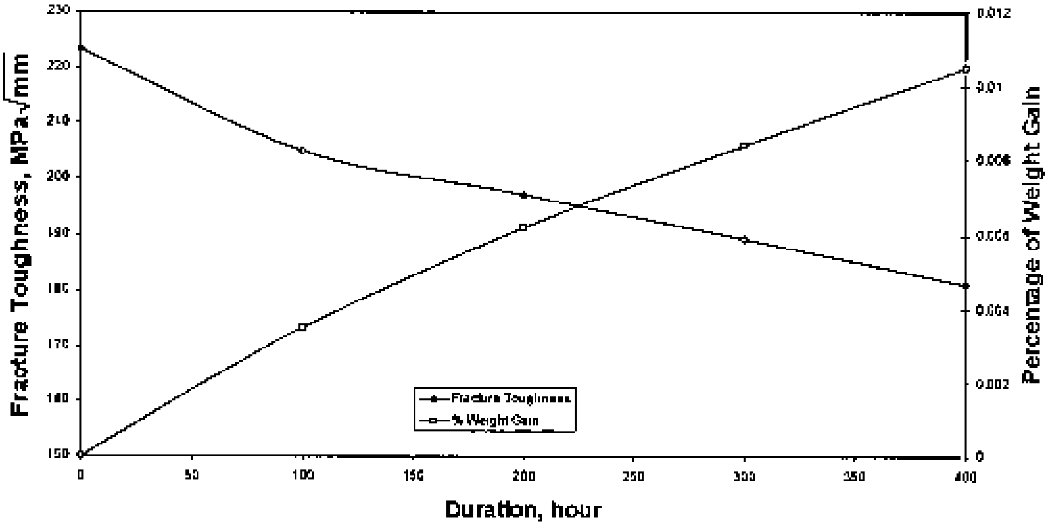

Figure 5.21 Degradation of fracture toughness after exposure in acidic water in comparison to percentage weight gain

When the specimens are exposed to organic fuel, only 0.17% weight gain has been recorded after 400-h duration of exposure (Figure 5.20). The reduction in fracture toughness has been found to be at a lower value of 9.48% as compared to previous four cases, that is, water, saline water, lower temperature and higher temperature in same duration, although the liquid penetration is quite less than water, saline water and ice. The absorption of aromatic constituents of liquid fuel may be taken to cause a marginal weight gain. The apparent cause of fracture toughness degradation may be due to small amount of fuel absorption through the voids, which corrode the fiber exposed to it. Loosely bonded carbon particles may be detached from fiber by chemical reaction resulting in a rough surface Thus, there is a marginal reduction in fiber strength and in fracture toughness

When the specimens are exposed to acidic-water, only 1.05% weight gain has been recorded after 400-h duration of exposure (Figure 5.21). The reduction in fracture toughness has been found to be at a higher value of 18.83% as compared to all the previous cases in the same duration, although the liquid penetration is less than water, ice and more than saline water and organic fuel. This high value of degradation on fracture toughness is mainly attributed to the reaction of acid on composite Concentrated H_2SO_4 acid would digest the binder material and debond the fibers from the matrix. When the concentration decreases to N/100, these effects would substantially decrease. However, prolonged exposure of composite may be taken to cause a slow reaction of acid and matrix, and developed debonding, delamination and micro-cracking in composite phases Thus, there is a high amount of fracture toughness degradation.

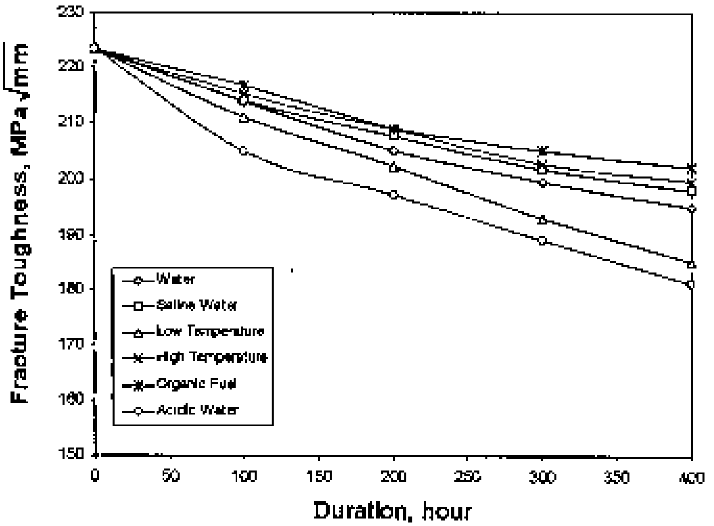

Figure 5.22 Comparision of average fracture toughness after exposure to various environments

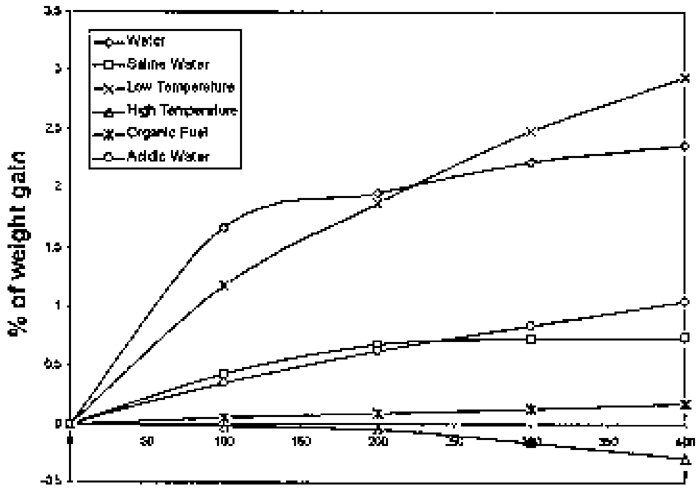

Figure 5.23 Comparison of Percentage weight gain after exposure into various environments

The overall results obtained from various adverse environments (Figures 5.22-5.23) have shown the rate of reduction of fracture toughness, which depend on reaction of constituent phases towards the exposed environments, duration of exposure and type of environment. Highest amount of fracture toughness strength reduction is noticed for acidic water and followed by low temperature and water.

5.4 Conclusion

The fracture toughness can be used to characterize the fracture behavior of composite materials under varied conditions. The test and analysis methods presented in this thesis provide the tool to collect data necessary for design with above mentioned environmental effects. A significant amount of data was collected to assess the impact of environment on this composite. As this study does not show any R-curve behavior, attention should be given on environmental effects for this range of crack length. This study will help select a particular duration of environmental exposure as well as particular environment for this class of composite materials according to the design limits.

Specimen preparation method was improved considerably, reducing the amount of error in test data due to inconsistencies in test specimens. Along with the data collected for use in design, the methods developed to collect and analyze fracture data under environmental impact may lead the way for future work in this field.

Based on the experimental results found for various environmental exposed specimens with different sizes of pre notches, and with diverse duration of environmental exposure, the following important conclusions can be drawn.

- Fracture toughness is independent of crack size.
- The highest amount of degradation in fracture toughness was found after exposure into acidic water and then in low temperature followed by water, saline water, high temperature and organic fuel for the same duration of exposures.
- Among six environments specimens exposed in organic fuel, high temperature of 60^0, saline water showed lower values of degradation of fracture toughness. So, the fabricated glass fiber/polyester composite may be suitable for use in these environments.
- There is a sizeable effect of environments on mechanical properties and on fracture toughness. The environmental effects depend on the type of environment, hostility, corrosivity and constituent ingredients.

- After exposure to various environments, it has been found that debonding and delamination occurred in initial phase of fracture toughness testing. This phenomenon has been predominant in all the cases prior to complete failure of the specimens under tensile loading.

- It has been observed that the temperature has significant influence over matrix debonding and reduced the original strength in greater margin.

- In case of liquid environment, the density of the medium has significant influence over the rate of diffusion of moisture into material. It depends on the constituent particles of the liquid; its osmotic pressure and number of voids in material.

References:

[1] D Broke. 'Elementary Engineering Fracture Mechanics (Fourth Edition)'. *Martinus Nijhoff Publishers*, 1987.

[2] G G Chell (ed). 'Developments in Fracture Mechanics–1'. *Applied Science Publication Ltd*, 1979.

[3] J Awerbach and M S Madhukar. 'Notched Strength of Composite Laminates · Predictions and Experiments–a Review'. *Journal of Reinforced Plastics and Composites*, vol 4, 1985, p 105.

[4] R B Pipes, R C Wetherhold and J W Gillespie (Jr). 'Notched Strength of Composite Materials'. *Journal of Composite Materials*, vol 12, 1979 p 148.

[5] Y Weixing. 'On the Notched Strength of Composite Laminates'. *Journal of Composite Science and Technology*, vol 45, 1992, p 105.

[6] William J. Devenport, 'Fracture Toughness Testing'. Course notes in Department of Aerospace & Ocean Engineering, Virginia Polytechnic Institute & State University, Blacksburg, VA 24061.

[7] Kenane M., Benzeggagh M.L. Mixed-mode delamination fracture toughness of unidirectional glass/epoxy composites under fatigue loading. Composites Science and Technology 1997; 57; 597-605

[8] Dahlen C., Springer G S. Delamination in composites under cyclic loads. Journal of Composite Materials 1994;28;732-781

[9] Filatows G.J., Sadler R.L., El-shiekh A.H.M. Fracture behavior of 3-D braid graphite/epoxy composite Journal of Composite Materials 1994;28;526-542

[10] Bazhenov S.L. Longitudinal splitting in unidirectional fibre-reinforced composites with can open hole. Composites Science and Technology 1998;58;83-89

[11] Kusaka T., Hojo M., Mai Y.-W , Kurokawa T., Nojima T., Ochiai S. Rate dependence of mode I fracture behaviour in carbon-fibre/epoxy composite laminates. Composites Science and Technology 1998;58;591-602

[12] S. Jose, R Ramesh Kumar, M. K. Jana, G. VenkateswAra Rao Intralaminar fracture thoughness of a cross-ply laminate and its constituent sub-laminates. Composites Science and Technology 2001; 61; 1115-1122

[13] Tohgo K., Fukuhara D., Hadano A. The influence of debonding damage on fracture thoughness and crack-tip field in glass- particle-reinforced Nylon 66 composites. Composites Science and Technology 2001;61;1005-1016

[14] Wang C., Huang Y., Xie Z. Improved Resistance to damage of silicon carbide-whisker-reinforced silicon nitride-matrix composites by whisker-oriented alignment. Journal of the American Ceramic Society 2001;84;161-164

[15] Zhu Q., Shobu K. High-temperature mechanical properties of SiC-Mo5(Si,Al)3C composites. Journal of the American Ceramic Society 2001;84;413 419

[16] Mcnulty J.C., Begley M.R., Zok F.W. In-plane fracture resistance of a crossply fibrous monolith. Journal of the American Ceramic Society 2001; 84; 367-375

[17] Wegner L D., Gibson L.J. The fracture toughness behaviour of interpenetrating phase composites. International Journal of Mechanical Sciences 2001;43;1771- 1791

[18] Goto K., Hatta H. Effect of shear damage on the fracture behavior of carbon-carbon composites. Journal of the American Ceramic Society 2001; 84;1327-1333

[19] Schueker C., Davidson B.D. Evaluation of the accuracy of the four-point bend end-notched flexure test for mode II delamination thoughness determination. Composites Science and Technology 2000; 60;2137-2146

[20] Reyes G., Cantwell W.J. The mechanical properties of fibre-metal laminates based on glass fibre reinforced polypropylene. Composites Science and Technology 2000;60,1085-1094

[21] Allix O., Leveque D., Perret L. Identification and forecast of delamination in composite laminates by an interlaminar interface model Composites Science and Technology 1998;58;671-678

[22] Li T.Q., Li R.K.Y. A fracture mechanics study of polypropylene-wood flours blends. Polymer-Plastics Technology Engineering 2001; 40;1-21

[23] Compston P., Jar P.-Y.-B., Burchill P.J., Takahashi K The effect of matrix toughness and loading rate on the mode-II interlaminar fracture toughness of glass-fibre/vinyl-ester composites. Composites Science and Technology 2001;61;321-333

[24] Kawada H., Srivastava V.K. The effect of an acidic stress environment on the stress-intensity factor for GRP laminates. Composites Science and Technology 2001;61;1109-1114

[25] Lawcock G., Ye L., Mai Y.-W., Sun C.-T. The effect of adhesive bonding between aluminium and composite prepreg on the mechanical properties of carbon-fiber-reinforced metal lamiantes. Composites Science and Technology 1997;57;35-45

[26] Collin M.I K., Rowcliffe D.J. Influence of thermal conductivity and fracture toughness on the thermal shock resistance of alumina- silicon-carbide whisker composites. Journal of the American Ceramic Society 2001; 84; 1334-1340

[27] Celemin J.A., Llorca J. The embrittlement of Nicalon/alumina composites at intermediate and elevated temperatures. Composites Science and Technology 2000;60;1067-1076

[28] Collin M.I.K., Rowcliffe D.J. Influence of thermal conductivity and fracture toughness on the thermal shock resistance of alumina- silicon-carbide-whisker composites. Journal of the American Ceramic Society 2001; 84;1334-1340

[29] Shindo Y., Horiguchi K., Wang R., Kudo H. Double cantilever beam measurement and finite element analysis of cryogenic mode I interlaminar fracture toughness of glass-cloth/epoxy laminates Journal of Engineering Materials and Technology 2001; 123;191-197

[30] Skirl S., Krause R., Wiederhorn S.M., Rodel J. Processing and mechanical properties of Al2O3/Ni3Al composites with interpenetrating network microstructure. Journal of the American Ceramic Society 2001; 84; 2034-2040

[31] Yang Z., Sun C.T. Interlaminar fracture toughness of a graphite/epoxy multidirectional composite. Journal of Engineering Materials and Technology 2000,122,428-433

[32] M.M. Schwartz, 'Composite Materials Handbook'. McGraw-Hill, Inc., New York, 1984.

[33] P K Mallik.'Fiber Reinforced Composites (2nd edition)' Marcel Dekker, Inc., New York, 1993, p15-23, 40.

[34] T.J. Reinhart, 'Polymer Matrix Composites'. Advanced Materials and Processes, January, 1990.

[35] T.J. Reinhart et al, 'Engineered Materials Handbook Volume 1 Composites, ASM International, Materials Park, OH, 1987.

[36] H J Konish and T A Cruse. 'Determination of Fracture Strength in Orthotropic Graphite/Epoxy Laminates'. *Composite Reliability, ASTM STP* 580, 1975 p 490

[37] H J Konish, J L Swedlow and T A Cruse. 'Experimental Investigation of Fracture in an Advance Composite'. *Journal of Composite Materials*, vol 6, no 1, January 1972, p 114.

[38] F A McClintock and G R Irwin. 'Plasticity Aspects of Fracture Mechanics in Fracture Toughness Testing and It's Applications'. *ASTM STP* 381, 1984, p 84

[39] T K O'Brien, N J Johnson, D H Morris and R A Simonds. 'A Simple Test for the Interlaminar Fracture Toughness of Composites'. *SAMPE Journal*, July/August 1982.

[40] P Stanleyo (ed). 'Fracture Mechanics in Engineering Practice'. 'The Fracture Toughness of Glass Fabric Reinforced Polyester Resins' by G Smith, A K Green and W H Bowyer. *Applied Science Publishers*, p 271.

[41] H Hyakutake, H Nisitani and T Hagio. 'Fracture Criterion of Notched Plates of FRP'. *Journal of Japan Society of Mechanical Engineers, Series* I, vol 32, no 2, 1989, p 300.

[42] Ronald F. Gibson, 'Principles of Composite Material Mechanics'. McGraw-Hill, Inc., 1994, P 341.

[43] W F Brown and J E Srawly. 'Plane Strain Crack Toughness Testing of High Strength Metallic Materials'. *ASTM STP* 410, 1966, p 292

Appendix A: Effects of Notch Size

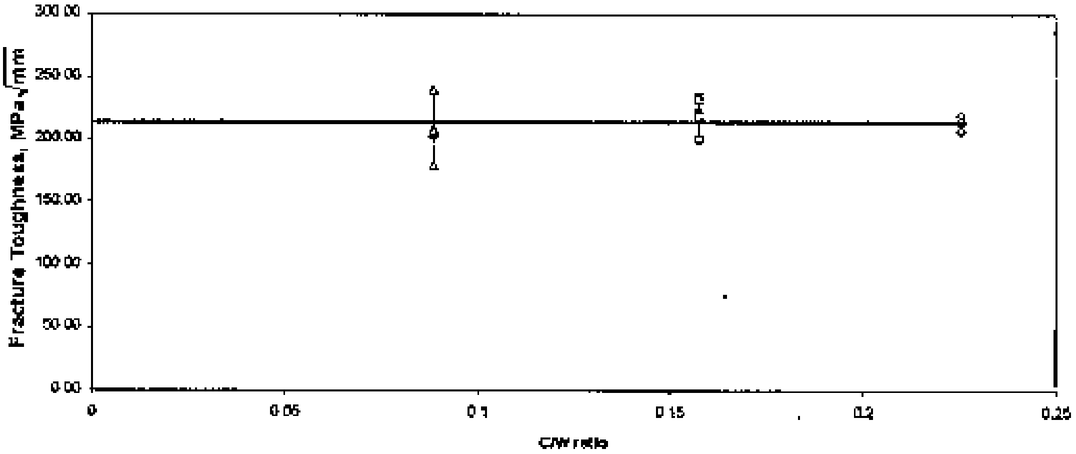

Figure A.1 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into water after 100 hours

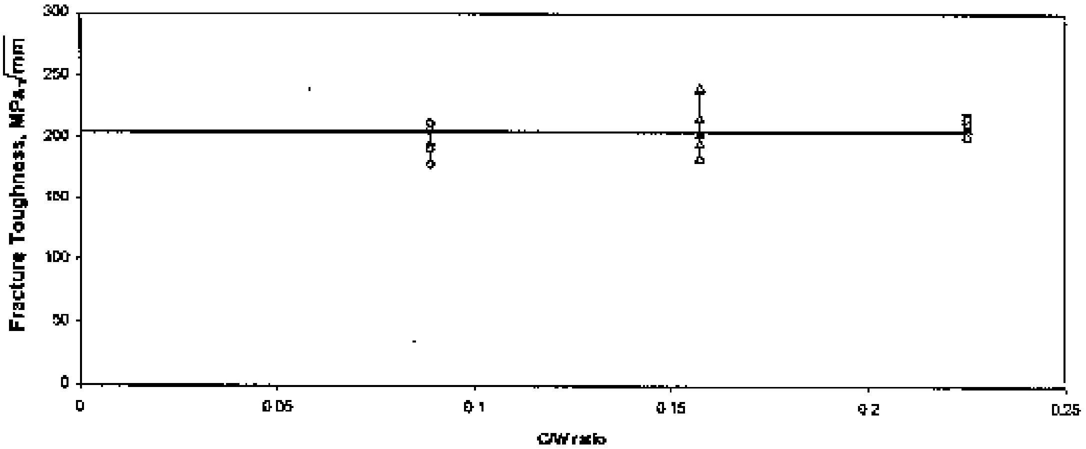

Figure A.2 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into water after 200 hours

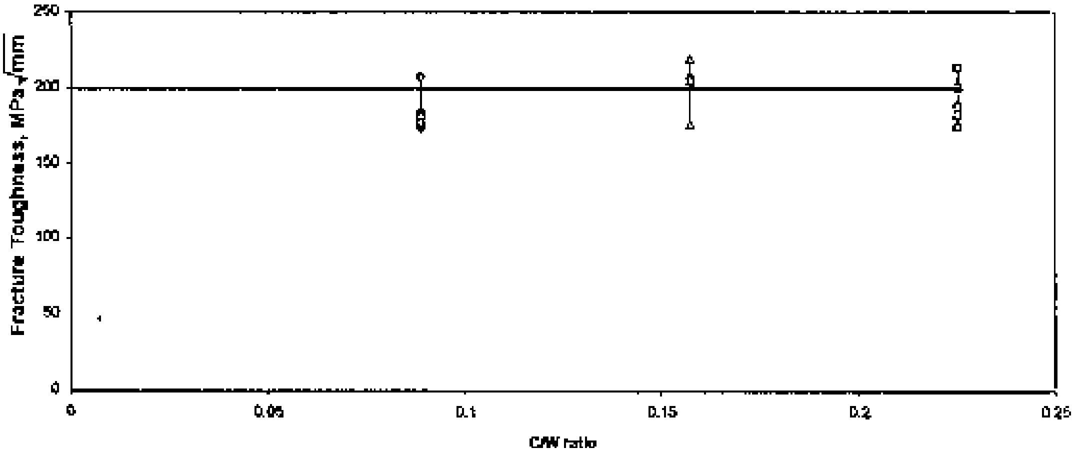

Figure A.3 Fracture toughness strength of Chopped strand matGlass Fiber/Polyester composite after exposure into water after 300 hours

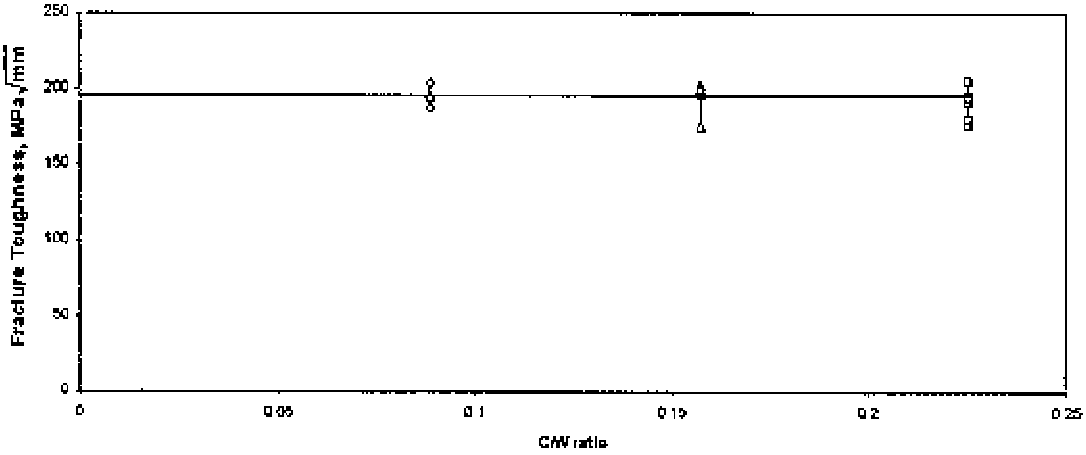

Figure A.4 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into water after 400 hours

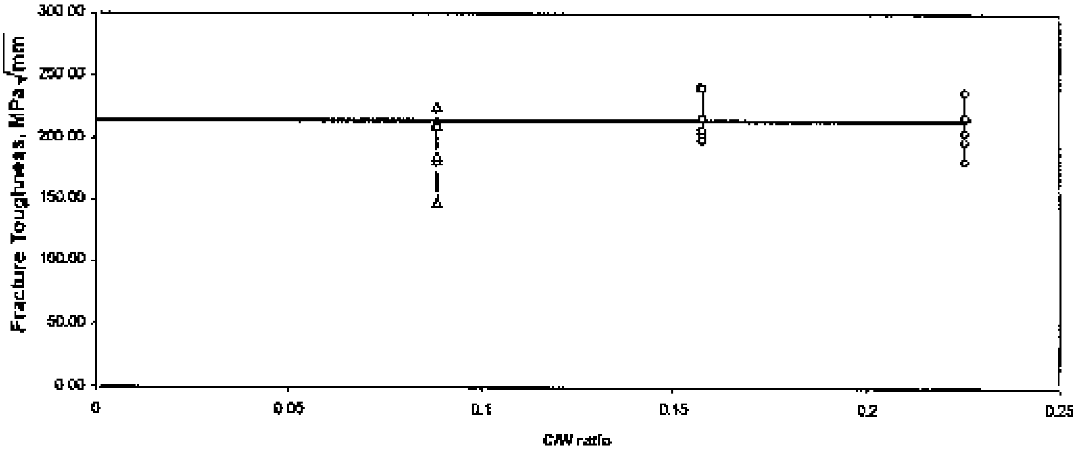

Figure A.5 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into saline water after 100 hours

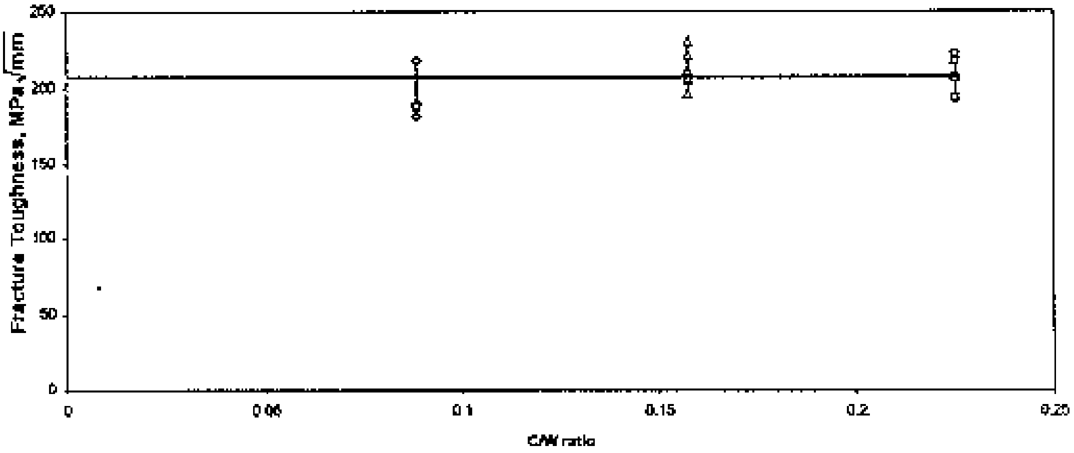

Figure A.6 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into saline water after 200 hours

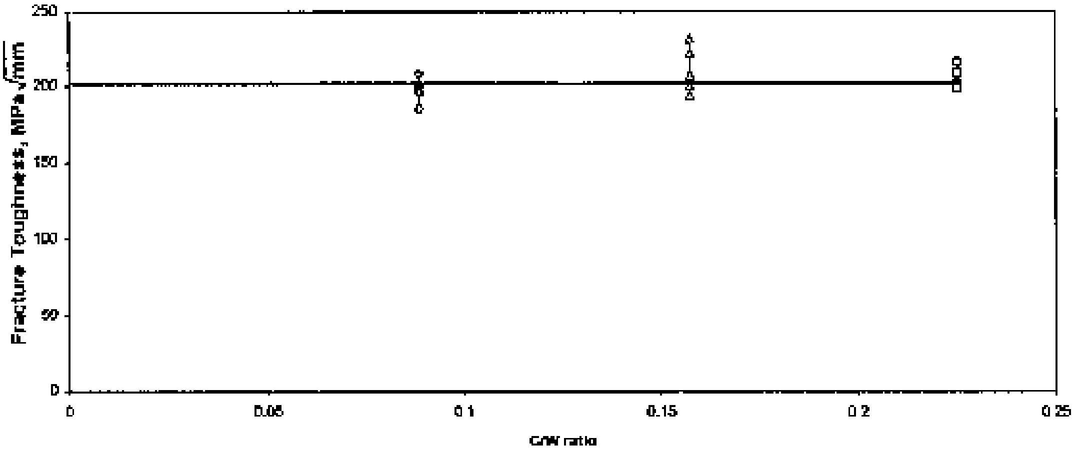

Figure A.7 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into saline water after 300 hours

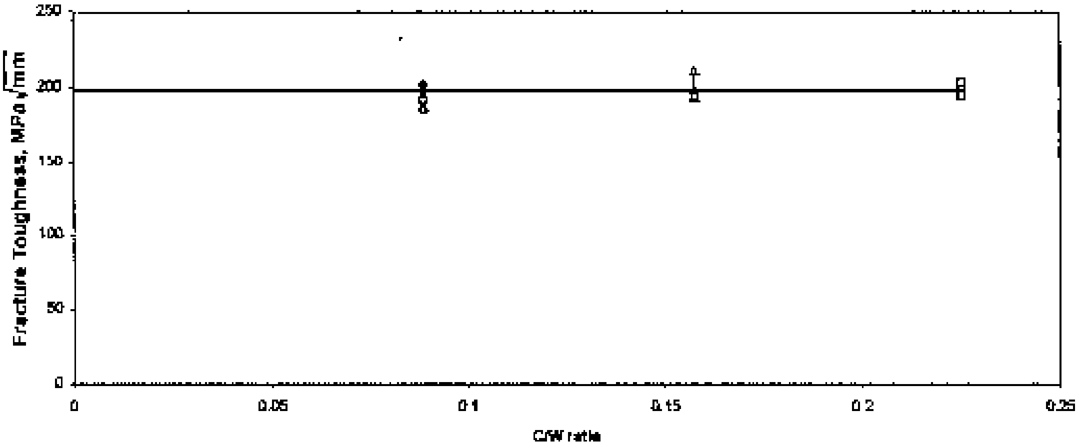

Figure A.8 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into saline water after 400 hours

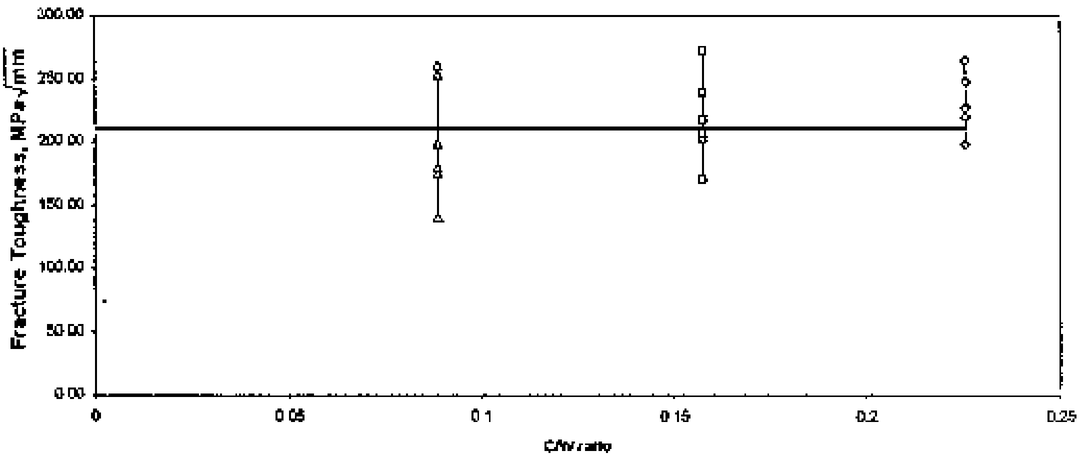

Figure A.9 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into low temperature(ice) after 100 hours

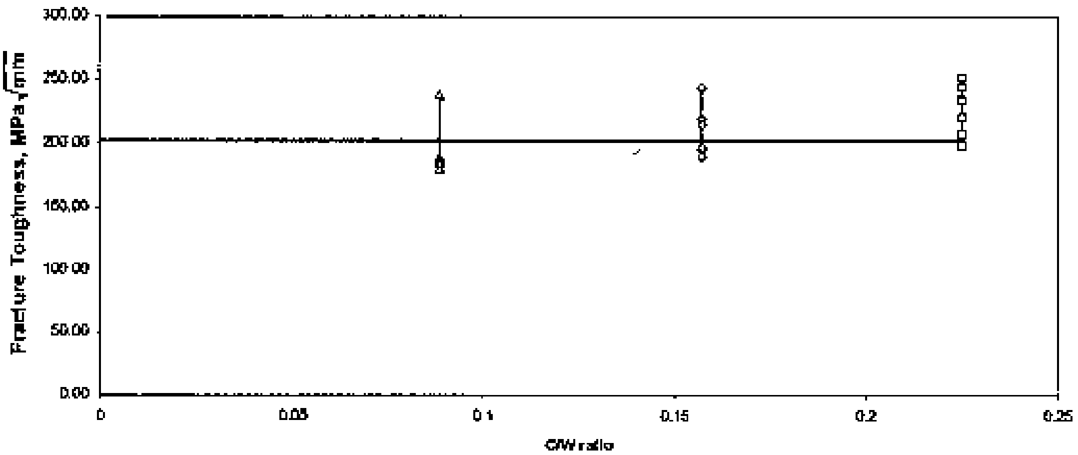

Figure A.10 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into low temperature(ice) after 200 hours

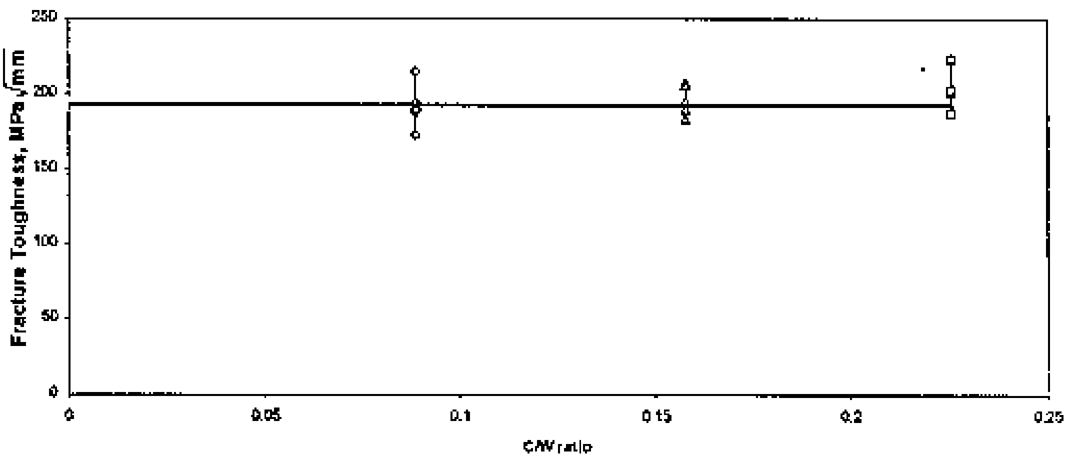

Figure A.11 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into low temperature(ice) after 300 hours

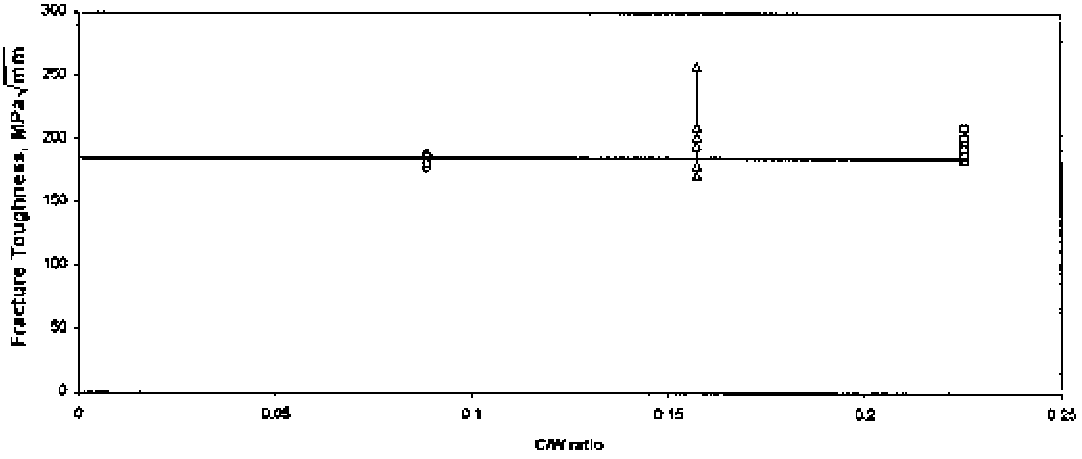

Figure A.12 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into low temperature(ice) after 400 hours

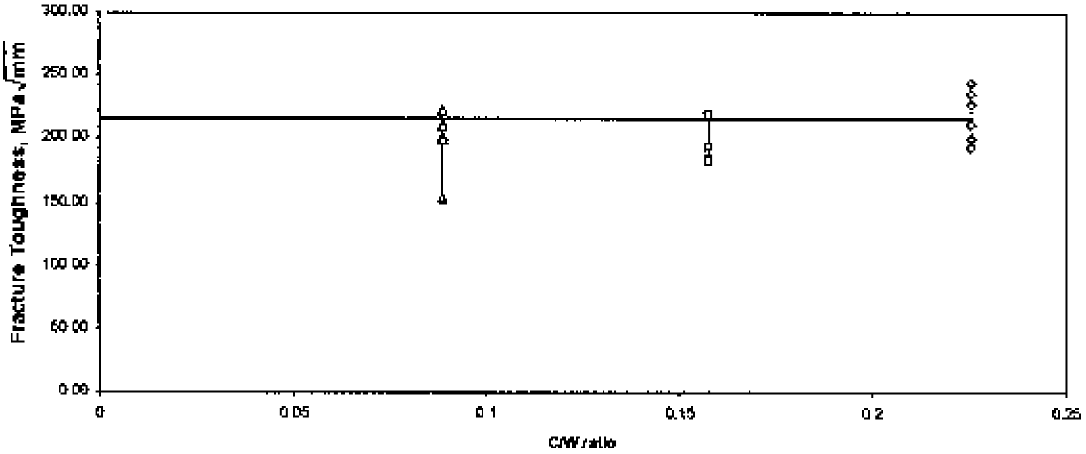

Figure A.13 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into High temperature(80° C) after 100 hours

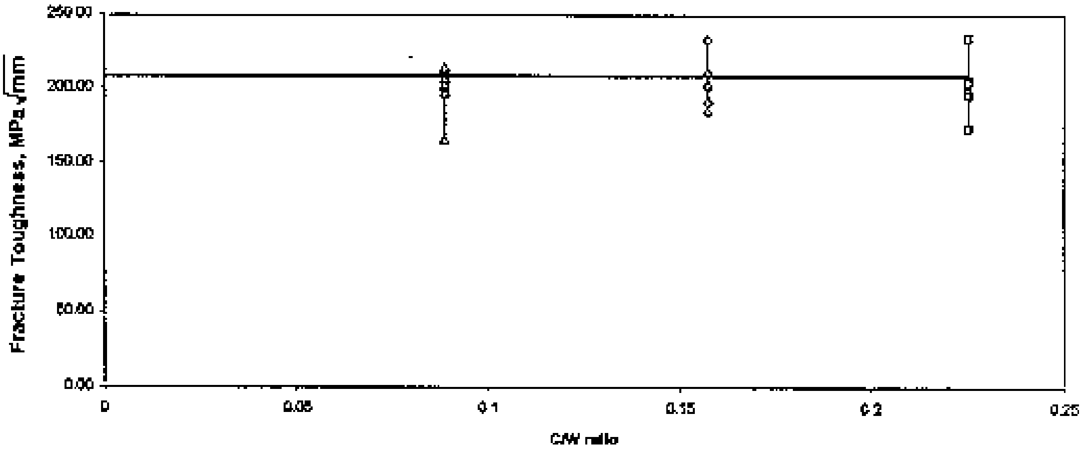

Figure A.14 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into High temperature(80° C) after 200 hours

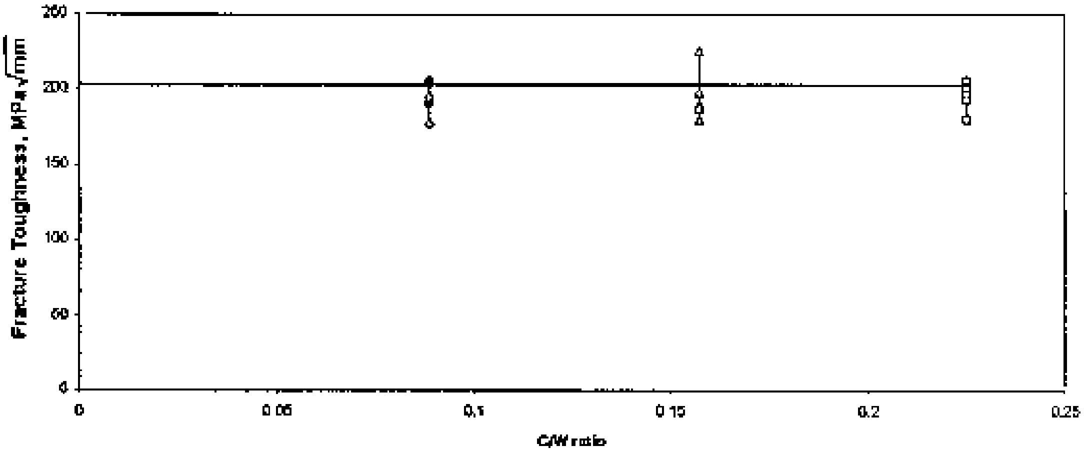

Figure A.15 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into High temperature(80^0 C) after 300 hours

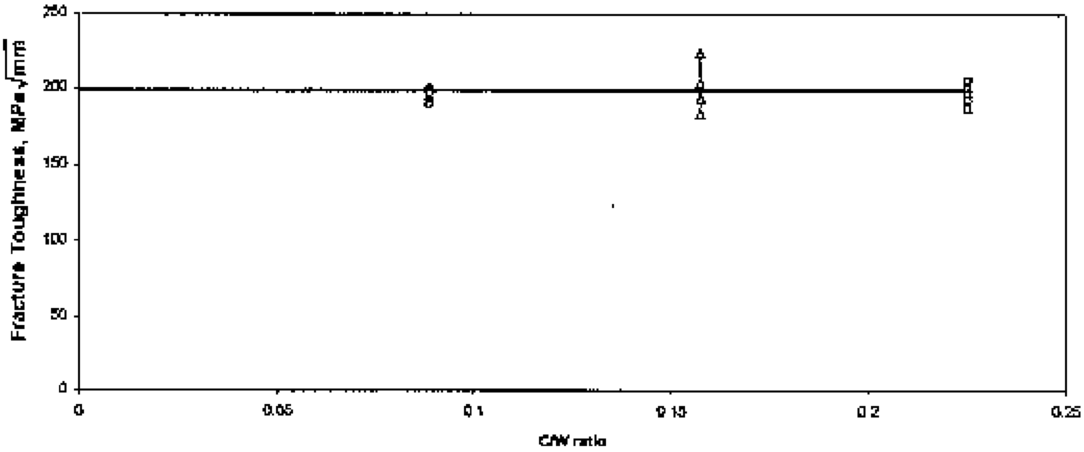

Figure A.16 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into High temperature(80^0 C) after 400 hours

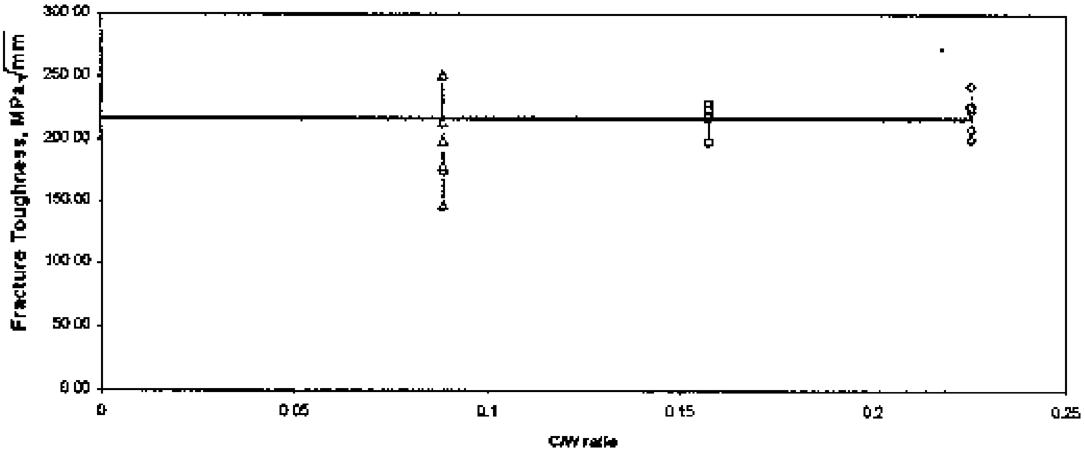

Figure A.17 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into organic fuel after 100 hours

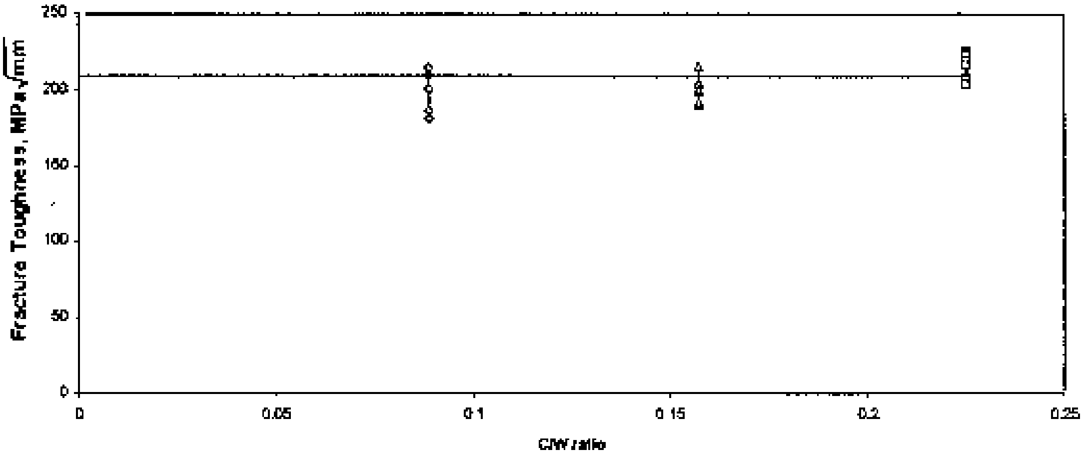

Figure A.18 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into organic fuel after 200 hours

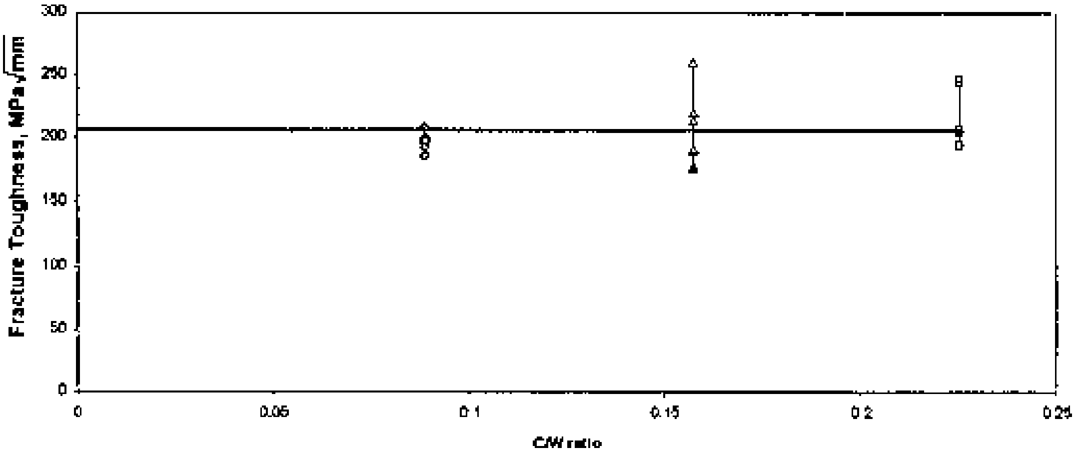

Figure A.19 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into organic fuel after 300 hours

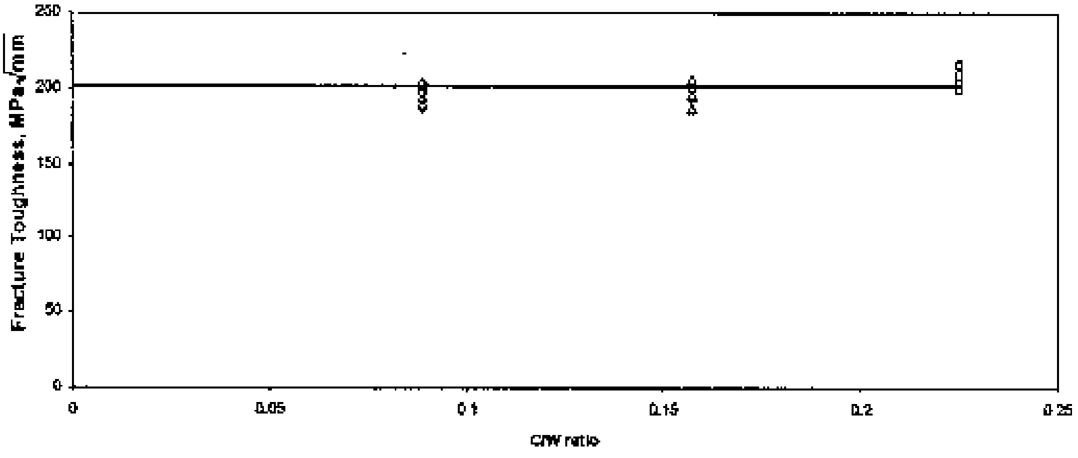

Figure A.20 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into organic fuel after 400 hours

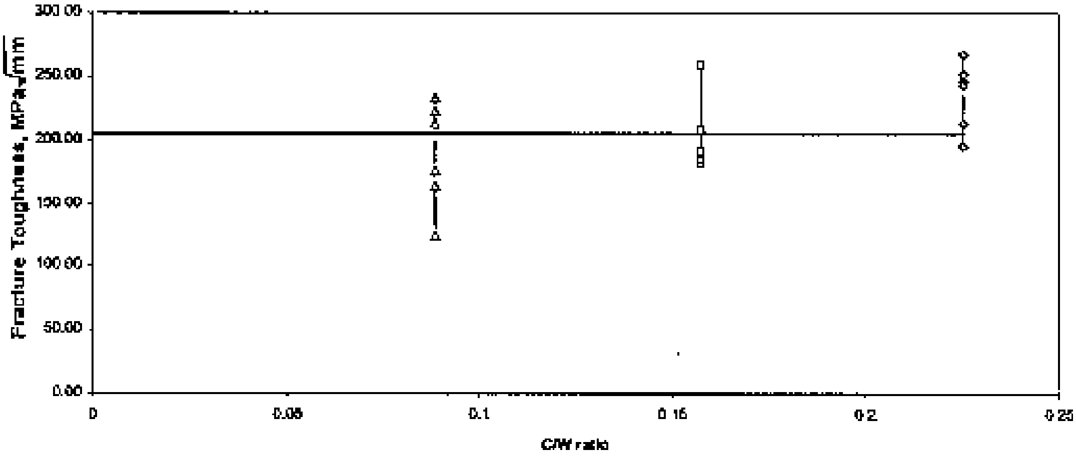

Figure A.21 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into acidic water after 100 hours

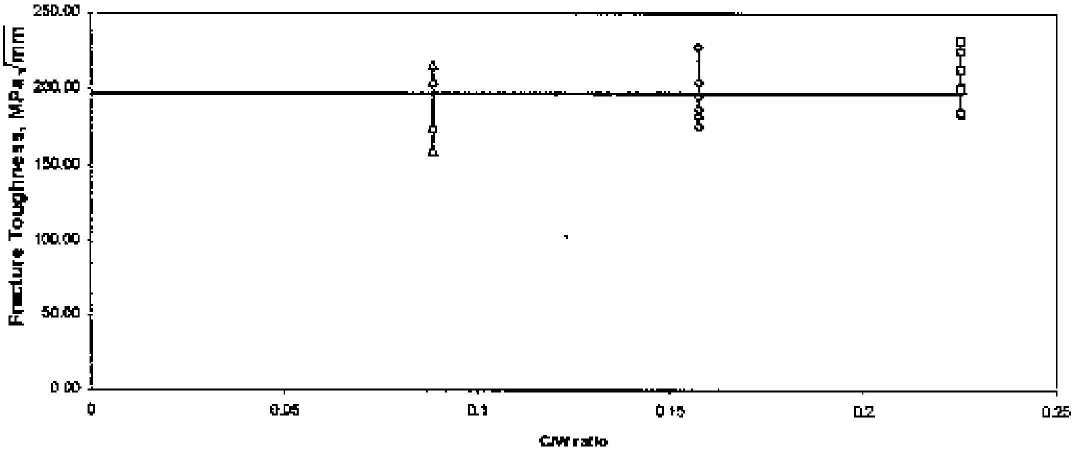

Figure A.22 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into acidic water after 200 hours

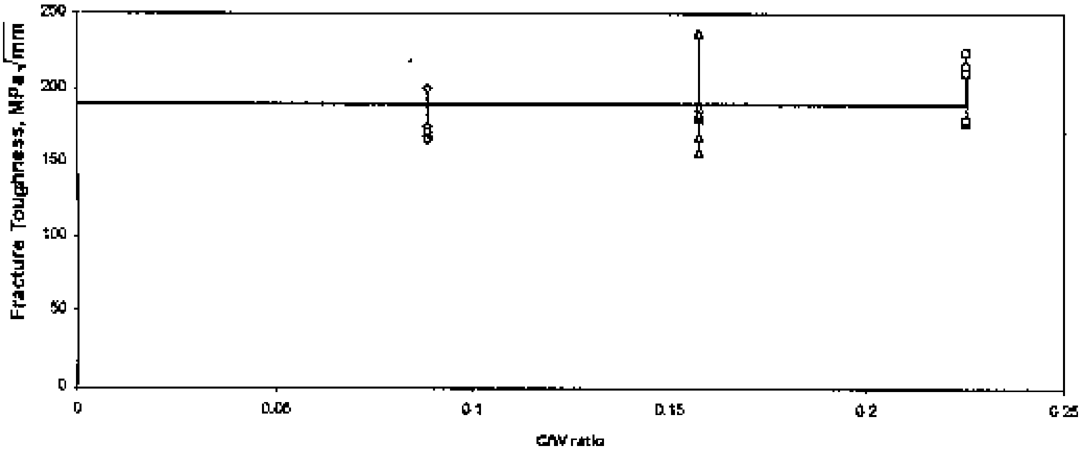

Figure A.23 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into acidic water after 300 hours

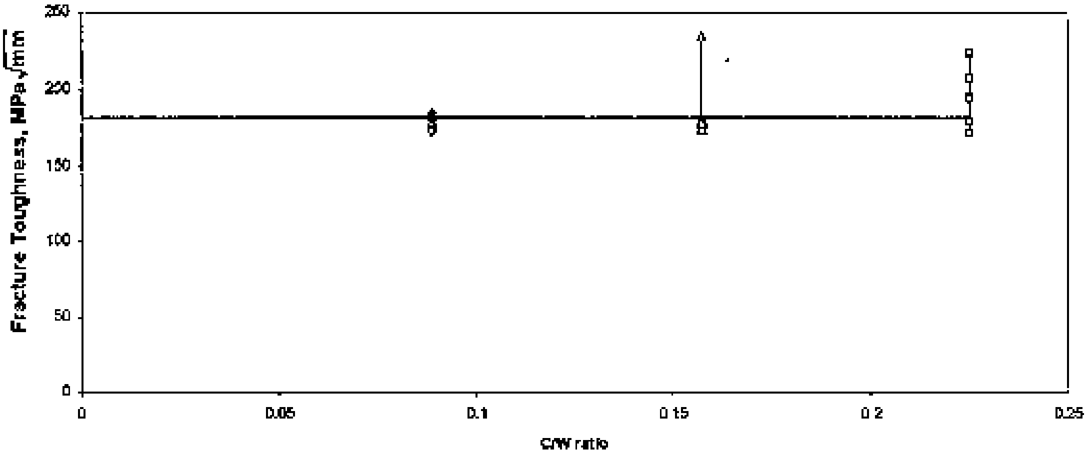

Figure A.24 Fracture toughness strength of Chopped strand mat Glass Fiber/Polyester composite after exposure into acidic water after 400 hours