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FAILURE CHARACTERIZATION OF FRP BY SCANNING ELECTRON MICROSCOPE TECHNIQUE

A THESIS SUBMITTED IN PARTIAL FULFILLMENT
OF THE REQUIREMENT FOR THE DEGREE OF

Bachelor of Technology
in
Metallurgical and Materials Engineering

By
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&
PARITOSH UPADHYAY

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CERTIFICATE

This is to certify that the thesis entitled, "FAILURE CHARACTERIZATION OF FRP BY SCANNING ELECTRON MICROSCOPE TECHNIQUE" submitted by Sri SUBRAT KUMAR PATRO & PARITOSH UPADHYAY in partial fulfillment of the requirements for the award of Bachelor of Technology Degree in Metallurgical and Materials Engineering at the National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University / Institute for the any Degree or Diploma.

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CONTENTS

ABSTRACT

Fiber-reinforced composite materials are used extensively in stiffness critical, weight sensitive structures such as those found in aerospace and motor racing. They are characterized by high in-plane strength, stiffness and toughness and low density. The environmental effect on the FRP (fiber reinforced polymer) and the subsequent failure has lead to emphasize on the study of different fracture surfaces and their different modes of propagation. Delamination between layers is an important problem in applications of fiber reinforced composite laminates. This paper describes an experimental study to characterize the crack surface, crack origination and their propagation using scanning electron microscope (SEM). By observing carefully the fracture surface of the composite the factors affecting their respective failure and the type of environment they were subjected to could be determined. SEM micrographs of the fractured surfaces of carbon/epoxy and glass/epoxy composites revealed the failure modes (delamination sites, debonding, fiber pullout regions, crack propagation front, shear cups, hackles, striations, bubble bursting in the matrix).

Chapter 1

INTRODUCTION

1. INTRODUCTION

The advantages of utilizing fiber reinforced polymer (FRP) in structural engineering over conventional materials are well known, i.e. higher strength and stiffness to weight ratio, good resistance to corrosion and fatigue performance [1]. A design life of 10–50 years is required for important areas of application of Fiber Reinforced Polymers (FRP) which include the automotive and aeronautical industry, bridge structures, water and waste systems and more recently in the offshore exploration and oil production [2]. These areas of applications require a better study of effect of temperature (both high and low), moisture, humidity, various loading rates and other environmental effects on FRP's. From a fracture mechanics point of view, if an FRP specimen (e.g. a thin plate) is stressed by an increasing load, the presence of alternative energy dissipation phenomena (debonding, pull-out, fiber bridging and friction) will result in an increase of the toughness [3].

FRP composite structures are often subjected to out of plane loads during manufacturing and service conditions. In such cases, layered composites suffer severely by delamination cracking because of poor interlaminar fracture resistance. On further loading, the interlaminar crack propagates and thus weakens the structure. By introducing small amount of fibers in the thickness direction of the laminate, the damage tolerance and suppression of delamination crack initiation or rate of interlaminar crack growth can be enhanced [4]. Interface between reinforcing fibers and matrix is believed to play an important role in composite properties. The effectiveness of load transfer at the interface depends upon the extent of chemical and mechanical bonding [5]. In the present study the focus is on the origin of the crack so that the proposal of limiting the propagation of the crack could be analyzed and the factors affecting the fracture could be determined.

The mechanical behavior of a composite material is decisively controlled by the fiber-matrix interface or interphase. Its properties influence the integrity of composite behavior because of its role in transferring stress between the fiber and the matrix. The factors affecting the inter face are too complex to be precisely concluded [6].Fibrous composites are increasingly being used in many applications owing to various desirable properties including high specific strength , high specific stiffness and controlled anisotropy. But unfortunately polymeric composites are susceptible to heat and moisture when operating in changing environmental conditions. They absorb moisture in humid environments and undergo dilatational expansion. The presence of moisture and stresses associated with moisture-induced expansion may cause lowered damage tolerance and structural durability. The structural integrity and life time performance of fibrous polymeric composites are strongly dependent on the stability of the fiber/polymer interfacial region [7].References [8-14] has cited a number of possible interactions: selective adsorption of matrix components conformational effects, diffusion of low molecular weight components from the fiber, penetration of polymer molecules into the fiber surface, and the catalytic effects of the fiber surface on polymers. The low molecular weight impurities may migrate from the bulk of the adhesive to form a weak boundary layer at or near the fiber surface [15].The active carbon fiber surface can strongly attract polar molecules of the polymer matrix. This may develop a boundary layer of high cross-link density. This micro structural gradient may promote crack initiation and propagation through this layer[16,17].Moisture interaction with the metal oxides in E-Glass leads to corrosion induced damage and thus results in reduced mechanical strength[18,19].The environmental action, such as high moisture and high temperature can limit the usefulness of polymer composites by deteriorating mechanical properties during service[18].The average bond strength of Epoxy resin with an E-glass fiber (approximately 33 MPa) is lower than with a carbon fiber(approximately 57 MPa) [21].But the anisotropy in carbon fibers limit there usage in various applications. However, particular structural requirements may need materials which have a higher modulus and a higher fatigue strength value than those which can be provided by the glass fiber [22]. Epoxy resins are the most common matrices for high performance advanced polymer composites, but they are also inherently brittle because of there high degree of cross linking. The densely cross linked structures are the basis of superior mechanical properties such as high modulus, high fracture strength, and solvent resistance. However, these materials are irreversively damaged by high stresses due to the formation and propagation of cracks. These lead to dangerous loss in the load-carrying capacity of polymeric structural engineering materials [23-26].Currently the unsaturated polyesters are the most widely used polymer in construction. These are easy to process with the ability to manufacture a good quality product; they are an ambient temperature cured material. However, the increase in styrene content in the unsaturated polyesters results in significant microcracking in resin rich areas and high residual stresses in composites having high volume fractions. Generally the Vinyl esters have good wetting characteristics and bond well to glass fibers. They possess resistance to strong acids and strong alkalis and they can be processed at both room and elevated temperatures. Compared to polyesters, vinyl esters offer reduced water absorption and shrinkage as well as enhanced chemical resistance. It is important to note that irrespective of the cure mechanism used, vinylesters do not completely polymerize, generally reaching a level of cure higher than 95 %, with the last part of cure continuing very slowly. Incomplete cure can result due to environmental conditions, incorrect stoichiometric of resin system components, or the failure to reach a sufficient temperature of cure. This state can adversely affect mechanical properties, moisture absorption and susceptibility to moisture induced degradation of the resin and the fiber matrix interface. Phenolics have good dimensional stability and resistance to acids and have good flame retardant properties, low smoke generation and high heat resistance. In addition, they have high resistance to water vapor transmission and water uptake. They are not stable in ultraviolet radiation [27].

Chapter 2

LITERATURE REVIEW

2. LITERATURE REVIEW

2.1 COMPOSITES

A composite is combination of two materials in which one of the materials, called the reinforcing phase, is in the form of fibers, sheets, or particles, and is embedded in the other materials called the matrix phase. The reinforcing material and the matrix material can be metal, ceramic, or polymer. Composites are used because overall properties of the composites are superior to those of the individual components. For example: polymer/ceramic composites have a greater modulus than the polymer component, but aren't as brittle as ceramics. The following are some of the reasons why composites are selected for certain applications:

- High strength to weight ratio (low density high tensile strength)
- High creep resistance
- High tensile strength at elevated temperatures
- High toughness

Typically, reinforcing materials are strong with low densities while the matrix is usually a ductile, or tough, material. If the composite is designed and fabricated correctly, it combines the strength of the reinforcement with the toughness of the matrix to achieve a combination of desirable properties not available in any single conventional material. The downside is that such composites are often more expensive than conventional materials. The strength of the composite depends primarily on the amount, arrangement and type of fiber (or particle) reinforcement in the resin. Typically, the higher is the reinforcement content, the greater is the strength. In some cases, glass fibers are combined with other fibers, such as carbon or aramid (Kevlar29 and Kevlar49),

to create a "hybrid" composite that combines the properties of more than one reinforcing material. Three types of composites are:

- Particle-reinforced composites
- Fiber-reinforced composites
- Structural composites

2.2 FIBER-REINFORCED COMPOSITES:

Reinforcing fibers can be made of metals, ceramics, glasses, or polymers that have been turned into graphite and known as carbon fibers. Fibers increase the modulus of the matrix material. The strong covalent bond along the fiber's length gives them a very high modulus in this direction because to break or extend the fiber the bonds must also be broken or moved. Fibers are difficult to process into composites which makes fiber reinforced composites relatively expensive. Fiber-reinforced composites are used in some of the most advanced, and therefore most expensive, sports equipment, such as a time-trial racing bicycle frame which consists of carbon fibers in a thermoset polymer matrix. Body parts of race cars and some automobiles are composites made of glass fibers (or fiberglass) in a thermoset matrix. The arrangement or orientation of the fibers relative to one another, the fiber concentration, and the distribution all have a significant influence on the strength and other properties of fiber-reinforced composites. Applications involving totally multidirectional applied stresses normally use discontinuous fibers, which are randomly oriented in the matrix material. Consideration of orientation and fiber length for particular composites depends on the level and nature of the applied stress as well as fabrication cost.

Production rates for short-fiber composites (both aligned and randomly oriented) are rapid, and intricate shapes can be formed which are not possible with continuous fiber reinforcement.

The matrix keeps the fibers in their desired locations, and orientation, separating them from each other to protect the fibers from abrasion, and provides a mean of distributing the load and transmitting the load in between the fibers, without itself fracturing.

The matrix is generally more ductile than the fibers; hence it is the source of composite toughness.

In composites the main causes of failure can be:

(a) Breaking of fibers

(b) Debonding (separation of fibers &matrix).

(c) Microcracking of the matrix.

(d) Delamination

Composites are superior to conventional metals because they have:

(a) High strength to weight ratio

(b) Good dimensional stability (extremely low coefficient of thermal expansion).

(c) Good resistance to heat, cold & corrosion.

(d) Good electrical insulation properties.

(e) Ease of fabrication.

(f) Relatively low cost.

2.3 TYPES OF FIBERS USED IN FIBER REINFORCED COMPOSITES:

1. Glass fibers

2. Carbon fibers

3. Aramid fibers

Glass Fibers

The most common reinforcement for the polymer matrix composites is a glass fiber. Most of the fibers are based on silica (SiO_2), with addition of oxides of Ca, B, Na, Fe, and Al. The glass fibers are divided into three classes -- E-glass, S-glass and C-glass. The E-glass is designated for electrical use and the S-glass for high strength. The C-glass is for high corrosion resistance, and it is uncommon for civil engineering application. Of the three fibers, the E-glass is the most common reinforcement material used in civil structures. It is produced from lime-alumina10 borosilicate which can be easily obtained from abundance of raw materials like sand.. The glass fiber strength and modulus can degrade with increasing temperature. Although the glass material creeps under a sustained load, it can be designed to perform satisfactorily. The fiber itself is regarded as an isotropic material and has a lower thermal expansion coefficient than that of steel.

1. *E-glass (electrical)*

Family of glassed with a calcium aluminum borosilicate composition and a maximum alkali composition of 2%. These are used when strength and high electrical resistivity are required.

2. *S-glass (tensile strength)*

Fibers have a magnesium aluminosilicate composition, which demonstrates high strength and used in application where very high tensile strength required.

3. *C-glass (chemical)*

It has a soda lime borosilicate composition that is used for its chemical stability in corrosive environment. It is often used on composites that contain or contact acidic materials.

2.4 PROPERTIES OF CARBON, GLASS AND KEVLAR

PROPERTY	CARBON PAN-based Type I	CARBON PAN –based Type II	GLASS	KEVLAR-49
DIAMETER(micrometer)	0-9.7	7.6-8.6	8-14	1.9
DENSITY (gm/cc)	1.95	1.75	2.56	45
YOUNG'S MODULUS (GN/m^2)	390	250	76	25
MODULUS(perpendicular to fiber axis)(GN/m^2)	12	20	76	_
TENSILE STRENGTH (GN/m^2)	2.2	2.7	1.4-2.5	2.8-3.6
ELONGATION TO FRACTURE(%)	0.5	1.0	1.8-3.2	2.2-2.8
COEFFICIENT OF THERMAL EXPANSION ($0\text{-}100^0C$)($10^{-6}/^0C$)	-0.5 to -0.12 (parallel) 7-12 (radial)	-0.1 to -0.5 (parallel) 7-12 (radial)	4.9	-2 (parallel) 59 (radial)
THERMAL CONDUCTIVITY(parallel to fiber axis) $Wm^{-1}\ {}^0C^{-1}$	105	24	1.04	0.04

2.5 Analytical Model

Fractographic techniques can be used to study micro-mechanisms of fracture, investigate of failure in laboratory structures, and post-mortem investigation of in-service components. The basic approach is to characterise the fracture morphologies of specimens failed under known (pure) failure modes, and then compare these morphologies to 'unknown' failures.

One of the key issues of laminated composites is that of delamination. The limited through-thickness strength of laminated composites means they are susceptible to out-of-plane loads, such as develop during impact. The general approach to characterising composite delamination resistance is to partition the fracture process into three 'pure' modes; mode I (peel), mode II (shear) and mode II (tearing). Tests are conducted under these pure modes, and under combinations of the modes (mixed-mode). The delamination toughness of a material is then characterised over a range of mode mixities, from which a delamination failure criterion can be developed that can be used in predictive models. The delamination resistance of a material is influenced by a number of factors such as moisture, temperature, ply orientation and even crack length. The increased toughness associated with such increases in resistance (in composites) to crack propagation is traditionally associated with plastic deformation, whereas in laminates the deformations are wholly elastic. The prevalent view is that the increases come from fibers bridging, the delamination and are more extensive, though less stable, in cross-ply laminates.

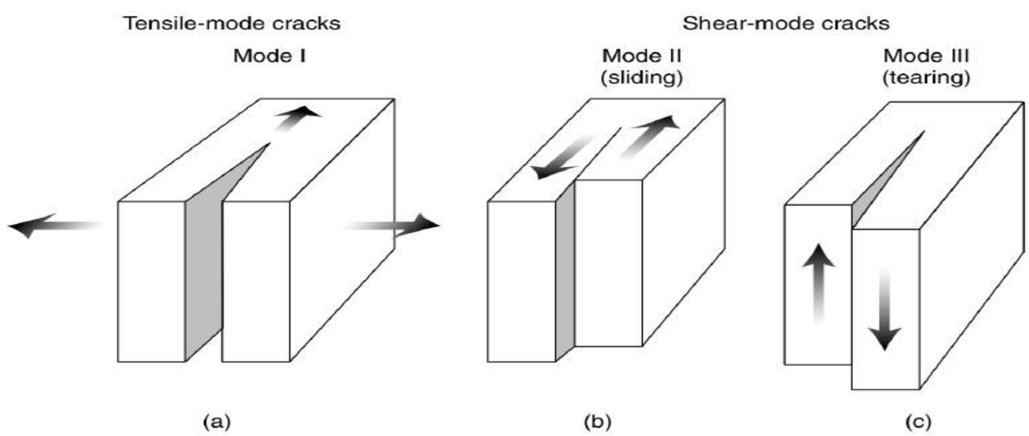


Fig.1 Different fracture opening modes.

Individual modes are well defined along the crack front [28]. Mode I is caused by the out of plane crack opening, Mode II by the shear perpendicular to the straight delamination/crack front and Mode III by the shear component tangential to the front. In composite materials however, the delamination crack growth can occur in any of the above three modes or a combination of them usually referred to as a mixed-mode phenomena [33]. Individual modes of energy release rate along the delamination front are calculated based on the Irwin's concepts of linear elastic fracture mechanics [34] and subsequent developments by Rybicki and Kanninen [35], due to the superimposed thermo-mechanical loading. The energy released by a self-similar propagation of a crack of length 'a' to that of a + Δa due to a sequential thermo-mechanical loading is nothing but the work required to close the crack from a + Δa length to 'a'. For the crack growth configuration as shown in fig.2, the strain energy released associated with the delamination extension is equal to the work required to close the incremental crack.

$$W = \frac{1}{2}\int_0^{\Delta a} [\sigma_M(n) + \sigma_T(n)][\delta_M(n - \Delta a) + \delta_T(n - \Delta a)]\, dn \tag{1}$$

where the subscripts 'M' and 'T' represent respectively the mechanical and thermal effects of the denoted parameters. $\delta(x - \Delta a)$ is the crack opening displacement between the top and bottom delaminated surface (Fig. 3) and $\sigma(x)$ is the stress at the crack front required to close the delaminated area. For a straight-edged crack front, the curvature plane and normal is constant everywhere. So mode definition is intuitive and constant for the entire front.

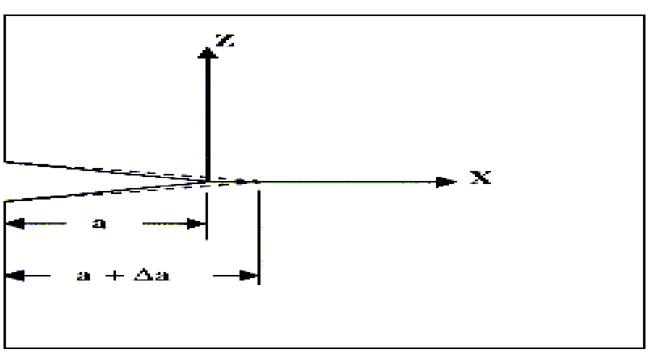


Fig.2 Schematic of crack growth propagation.

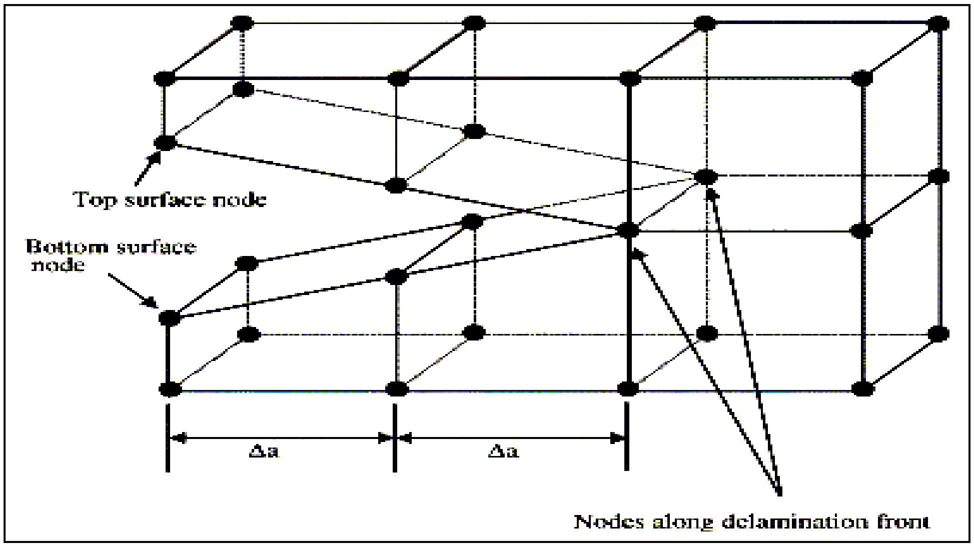


Fig.3 Schematic representation of crack front .

However, as shown in Fig. 4, for a curved crack front the tangent and normal along the curvature varies from location to location. Therefore for an arbitrarily shaped delamination front the mode definition changes along the contour. In the present study, as the crack front is curved, appropriate local coordinate transformations on the crack tip stress and displacement fields have been carried out to obtain the values of strain energy release rate. It is to be emphasized that unlike a cylindrical coordinate transformation as in the case of circular delamination, here an elliptic coordinate (n, t) transformation is needed for the calculation of strain energy released. So the integral in Eq. (1) needs to be evaluated along the normal to the elliptic crack front. This transformation yields

$$W = \frac{1}{2}\int_0^{\Delta a} [\sigma_M(n) + \sigma_T(n)][\delta_M(n - \Delta a) + \delta_T(n - \Delta a)]\, dn \qquad (2)$$

Then the energy release rate is obtained as

$$G = \lim_{\Delta a \to 0} \frac{W}{\Delta a}$$

Referring to Fig. 4, at any point 'P' along the delamination contour a local coordinate [n, t, z] is defined such that 'n' and 't' represent the tangent and normal to the curve at that location in the plane of delamination. The modified crack closure integral has the advantage of mode separation of strain energy release rates for a qualitative analysis of fracture behavior of delaminated surface. The three components of strain energy release rates for Mode I, Mode II, and Mode III are

$$G_{\mathrm{I}} = \lim_{\Delta a \to 0} \frac{1}{2\Delta a} \int_0^{\Delta a} [\sigma_{zz\mathrm{M}}(n) + \sigma_{zz\mathrm{T}}(n)][\delta u_{z\mathrm{M}}(n - \Delta a) + \delta u_{z\mathrm{T}}(n - \Delta a)]\,\mathrm{d}n$$

$$G_{\mathrm{II}} = \lim_{\Delta a \to 0} \frac{1}{2\Delta a} \int_0^{\Delta a} [\tau_{zn\mathrm{M}}(n) + \tau_{zn\mathrm{T}}(n)][\delta u_{n\mathrm{M}}(n - \Delta a) + \delta u_{n\mathrm{T}}(n - \Delta a)]\,\mathrm{d}n$$

$$G_{\mathrm{III}} = \lim_{\Delta a \to 0} \frac{1}{2\Delta a} \int_0^{\Delta a} [\tau_{zt\mathrm{M}}(n) + \tau_{zt\mathrm{T}}(n)][\delta u_{t\mathrm{M}}(n - \Delta a) + \delta u_{t\mathrm{T}}(n - \Delta a)]\,\mathrm{d}n$$

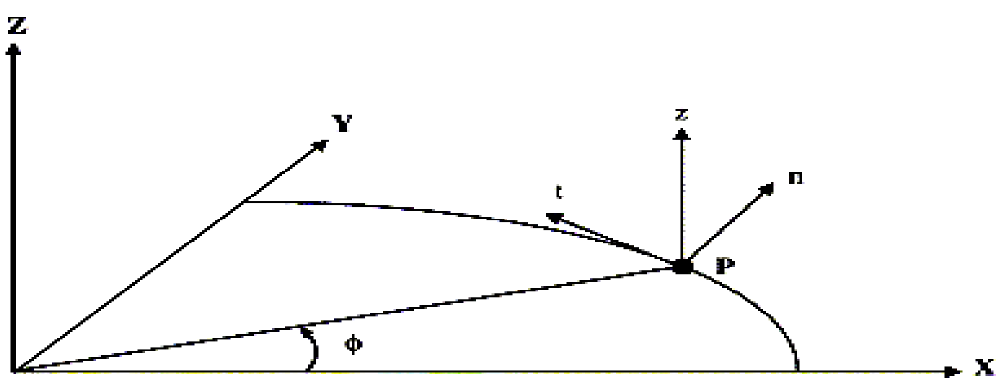


Fig. 4. Contour of curved delamination front.

The [n, t, z] are local coordinate transformations of [x, y, z], where n and t are the normal and tangent directions along the delamination front respectively. Similarly $[\sigma_{zz} \quad \tau_{zn} \quad \tau_{zt}]$ are the elliptical local coordinate transformations of interlaminar stresses $[\sigma_{zz} \quad \tau_{zx} \quad \tau_{zy}]$ in global coordinates. δ_{uz}, δ_{un}, δ_{ut} are respectively the relative opening, normal and tangential displacements of the upper delaminated surface to the lower one along the delamination boundary. The total energy release rate considering the thermal residual stress effects can then be expressed simply as the algebraic sum of the individual modes as follows

$$G_{total}=G_I+G_{II}+G_{III}$$

2.6 Coupling effect of interphase and fibre-bridging on the toughness of FRP

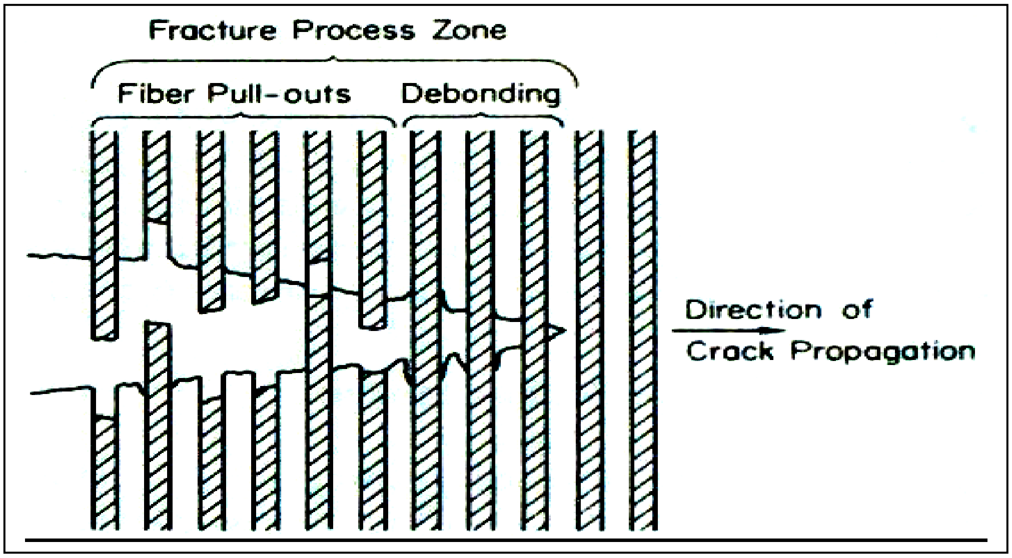
Important factors resulting in an increase of toughness during crack growth :

i. fibre bridging behind a crack tip (bridging zone is shielding the crack tip, thus reducing the net stress intensity factor)

ii. An interphase with a certain thickness could have different behavioural properties such as elastic, elastic–plastic, perfect plastic, viscous, etc. depending on the sizing, the types of fibre and matrix and the curing process.

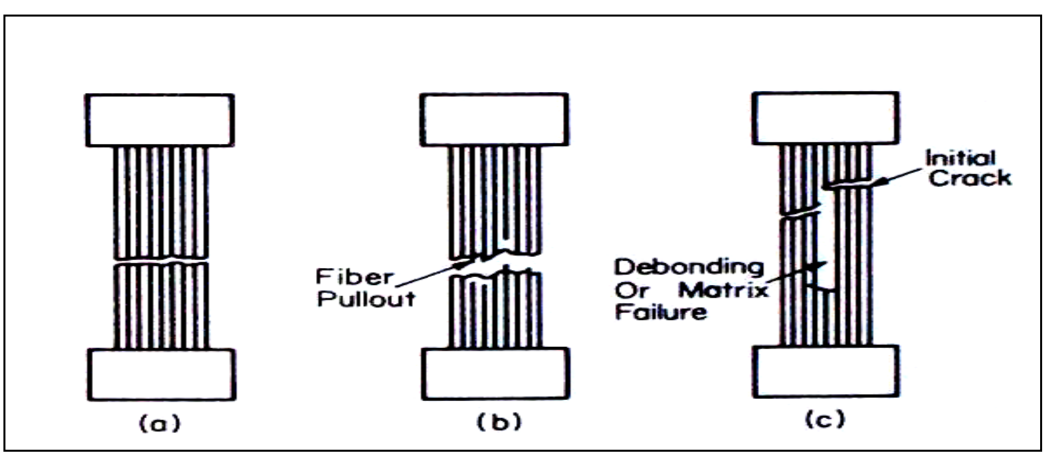
iii. If an external load is applied to the thin composite plate, crack propagation and stress transfer within the interphase will take place.

2.7 Model of crack tip in fiber composites showing local failure events



At some distance ahead of the crack the fibers are intact. In the high stress region near the tip they are broken, although not necessarily along the crack plane. Immediately behind the crack tip fibers pull out of the matrix. In some composites the stress near the crack tip could cause the fibers to debond from the matrix before they break. It is also possible for a fiber to be left intact as the crack propagates [29]. When brittle fibers are well bonded to a ductile matrix, the fibers tend to snap ahead of the crack tip, leaving bridges of matrix material that neck down and fracture in a completely ductile manner. In addition to these local failure mechanisms, on reaching the interface of the two laminae in a laminated composite, a crack can split and propagate along the interface, thus producing the delamination crack.

2.8 Failure modes of unidirectional composite



Subjected to longitudinal tensile load:

a) Brittle failure (V_f<0.4)

b) Brittle failure with fiber pullout (0.4< V_f< 0.65)

c) Brittle failure with debonding and/or matrix failure (V_f>0.65)

Chapter 3

EXPERIMENTAL PROCEDURE

3. Experimental Procedure

The samples were collected from the laboratory of the Guide where a large amount of research is being carried out on FRPs .The samples provided were already fabricated, conditioned and tested. Fabrication was mostly done by Hand lay-up method using glass and carbon fibers and epoxies, polyesters and vinyl esters as matrix materials

The conditioning included various environmental conditions such as

1. Cryogenic treatment using liquid nitrogen (77 K) for different time cycles and different crosshead speeds

2. Hygrothermal treatment at 60^0C for different medium and different time cycles

3. Tested at ambient temperature.

4. Fast thaw and slow thaw conditions at ambient, cryogenic (–50˚ C & –80˚C) at different cross head speeds.

The interlaminar shear strength (ILSS) was measured as follows,

$$ILSS = 0.75p/bt$$

where, ‘p’ is the breaking load, ‘b’ the width, and ‘t’ the thickness of the specimen. An Instron1195 tensile testing machine was used to perform SBS tests and tensile tests in accordance with ASTM D2344-84 standards. The tested samples were then chosen for SEM analysis. The samples were cut into 5mm butts along the length with fractured surface and then sent for the analysis. The SEM used was JEOL JSM 6480LV at NIT Rourkela.

Chapter 4

DISCUSSION & INTERPRETATION

4. DISCUSSION & INTERPRETATION

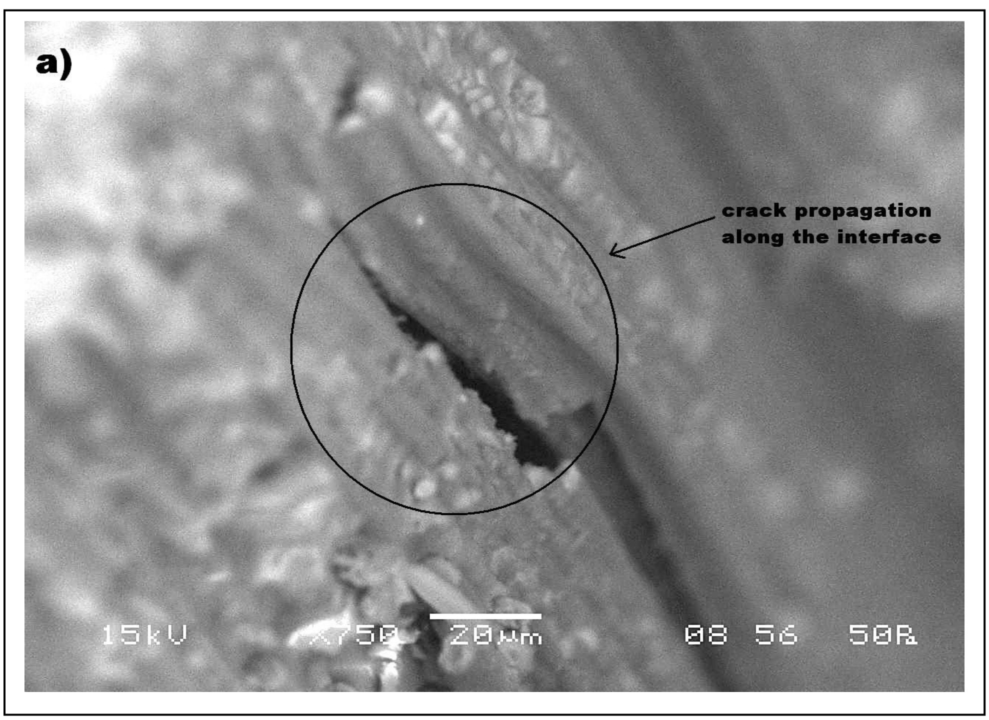
4.1 FRACTURE MODES IN COMPOSITES

Fracture modes in composites can be divided into three basic fracture types

a) Interlaminar, b) Intralaminar, c) Translaminar

When considered on microscale, interlaminar and intralaminar fracture types can be similarly described. In both cases, fracture occurs on a plane parallel to that of the fiber reinforcement. In a similar manner to that described for metals ,fracture of either type can occur under mode I tension ,mode II in-plane shear, mode III anti-plane shear, or any combination of these load conditions. Translaminar fractures are those oriented transverse to the laminated plane in which conditions of fiber fracture s are generated .Our present work is based upon the above characterization.

4.1.1 Translaminar fracture mode



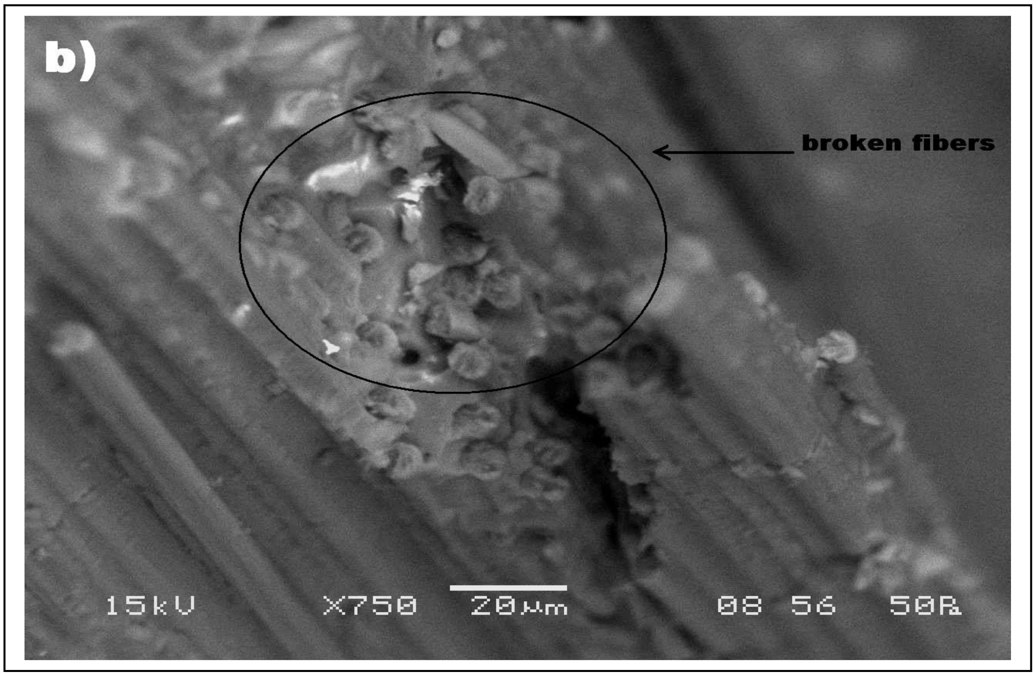


Fig 6. SEM micrograph of cryogenically conditioned (77K) carbon /epoxy composite sample showing crack propagation along the interface at crosshead speed of 100mm/min.

The matrix contraction at cryogenic temperature is resisted by stiff fibers through fiber/matrix interfacial bonding that originates residual stresses. The matrix response to an applied load is temperature dependent and change in temperature can cause internal stresses to be set up as a result of differential thermal contraction and expansion of the two constituents. Increased thermal stresses are the underlying cause of micro cracking in composites at cryogenic temperatures[30].

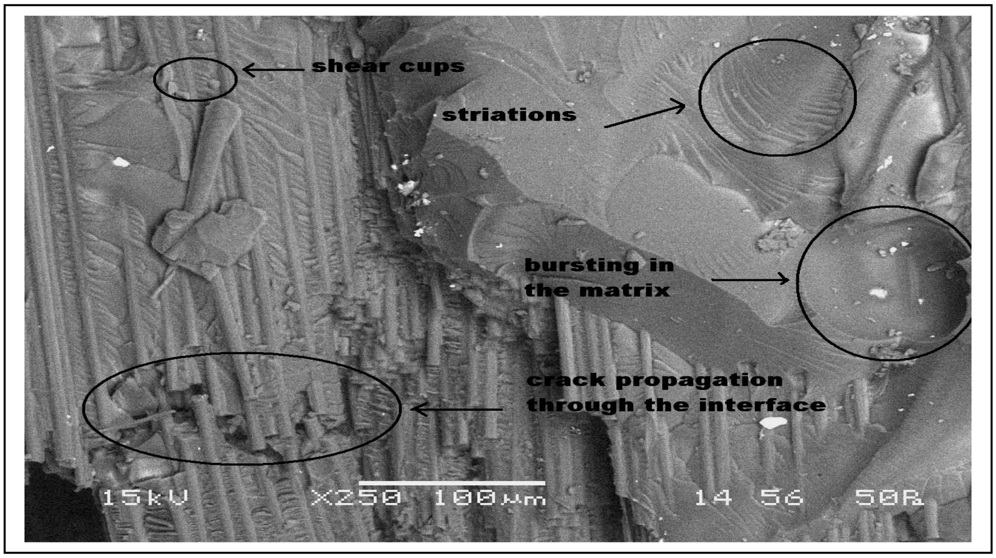


Fig 7. SEM micrograph of cryogenically treated carbon/epoxy composite sample at crosshead speed of 200mm/min.

Fig. 7 shows the formation of rows of cups due to the development of transverse micro-cracks along the interfacial area [30]. Increase in brittleness of the epoxy matrix after cryogenic conditioning causes opening of these micro-cracks easily that develops profile with rows of cups. When these cracks accumulate and merge to form longitudinal cracks along the fiber then failure of the composite results. The damage may begin with the formation of striations/microscopic cracks (crazing) in the matrix or at the fibre/matrix interface [31]. When these cracks develop to a certain density and size, they will tend to coalesce to form macroscopic matrix cracks.

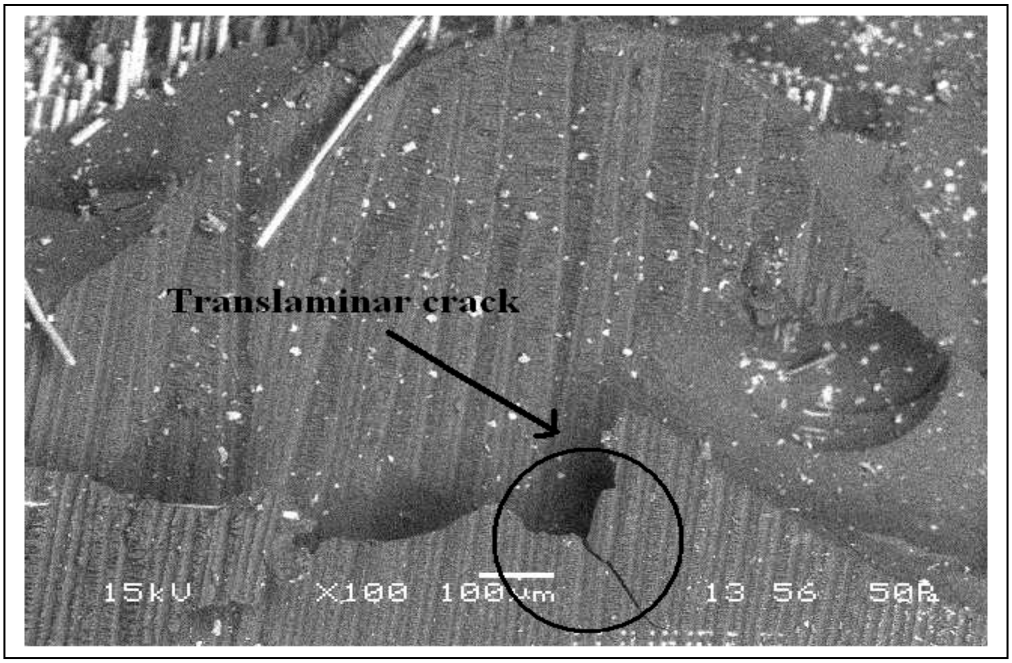


Fig. 8 SEM micrograph of a delaminated glass/polyester composite specimen.

The micrograph in fig. 8 shows failure due to propagation of transverse crack across the interface in a delaminated ply. The clean regions of fiber debonding are also seen in the figure.

4.1.2 Interlaminar Fracture Mode

a)

b)

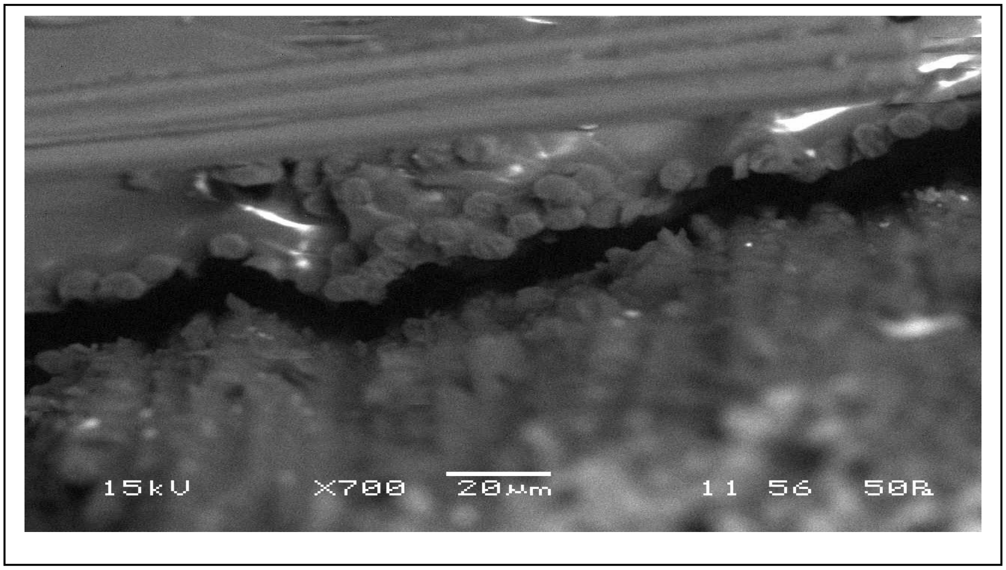


Fig. 9. SEM micrograph of treated carbon/epoxy composite sample at ambient temperature.

The above micrograph fig. 9a shows the recessed pockets as fiber bundle pullouts in the broken cross section of CFRP composite with fiber orientation of $0^0/90^0$. It also shows a delamination crack front propagating along the $0^0/90^0$ interface. Fig 8b shows debonding of the fiber. The propagation of the crack front in fig. 9b is along the interface in which the fibers have different orientations (cross-ply perpendicular to each other) ie. interlaminar crack.

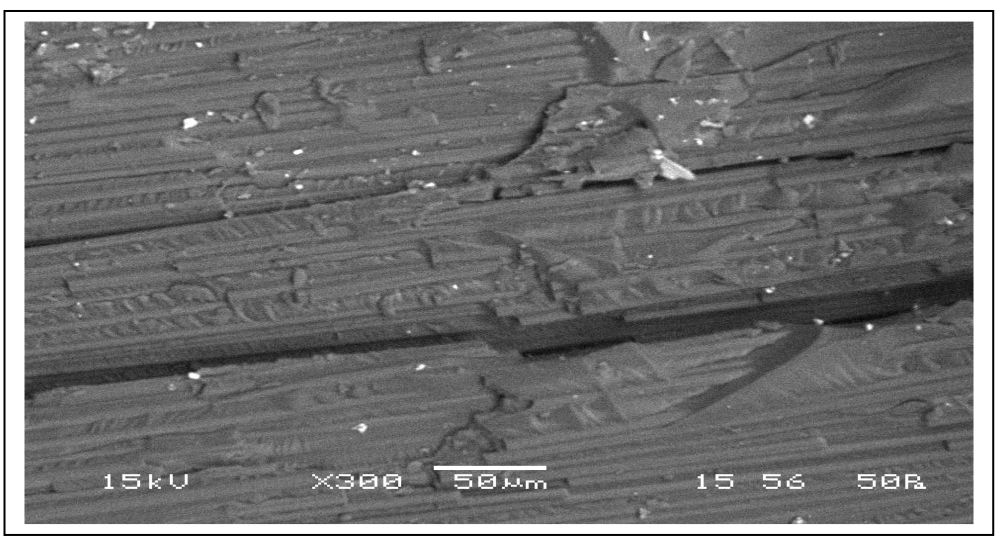


Fig. 10 SEM micrograph of cryogenically treated carbon/epoxy composite sample at crosshead speed of 200mm/min.

Fig. 10 shows a large amount of matrix crackings which may be attributed to brittleness of the epoxy resin at low temperature leading to nucleation of delamination cracks in the weak fiber-matrix interface.

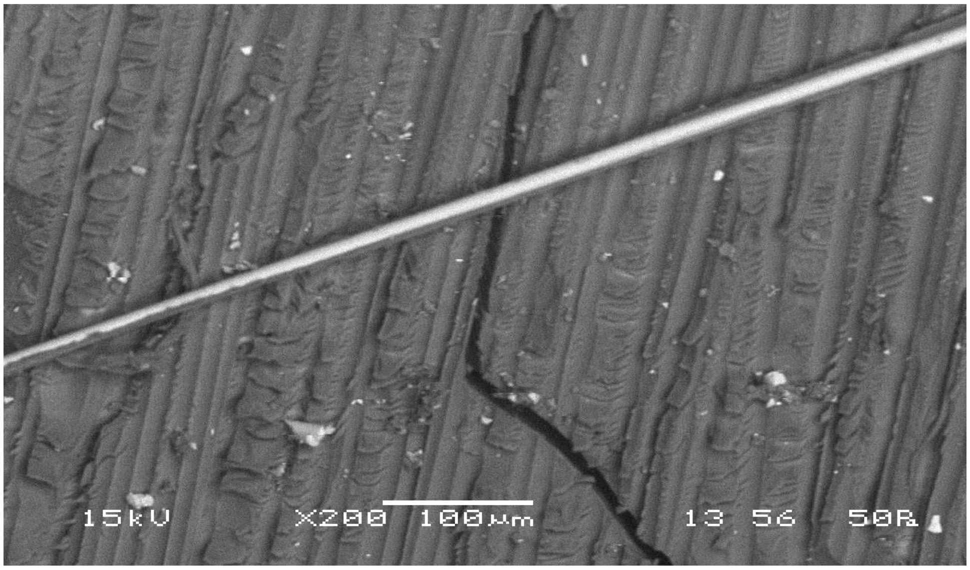
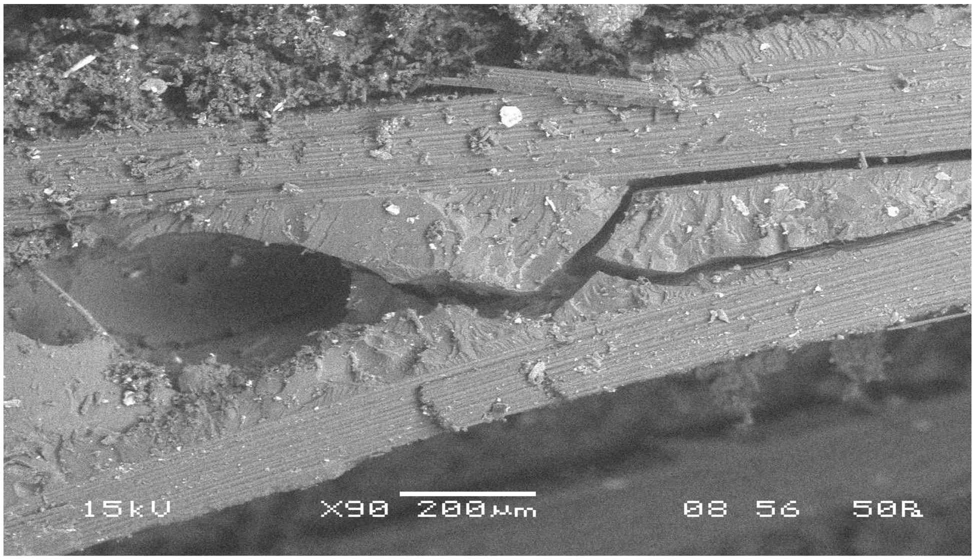


Fig. 11. Fracture surface of glass/polyester composite sample pulled transverse to the fiber surface.

Fig.11 shows a delaminated surface of GFRP composite with a prominent crack propagating in the transverse direction. However, when this propagating crack meets with a weak fiber/matrix interface, shear failure of the weak interface takes place. This in turn has diverted the propagating transverse crack along the fiber direction. Extensive matrix damage is also visible [31].

a)



b)

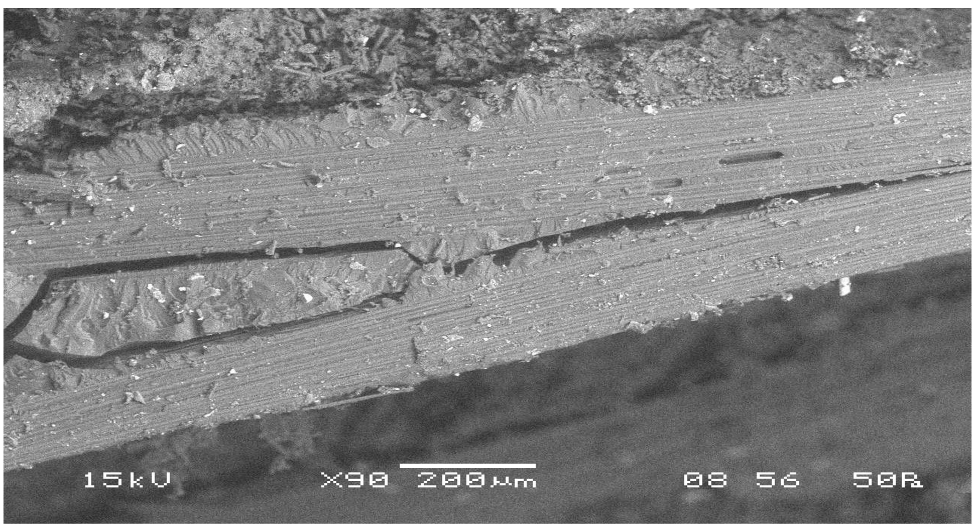


Fig.12 Fracture surface of carbon/epoxy composite specimen failed in 3 Point Bend testing.

The above micrographs (fig.12a & 12b) shows crack propagation the interlaminar plies moving from left side of the fracture surface to the right hand side of the surface which can be inferred form the markings produced by cavity nucleation and growth ahead of the advancing crack front. The crack front changed its path orientation when a ductile matrix inferred in its path.

4.1.3 Mode I Fracture Mode

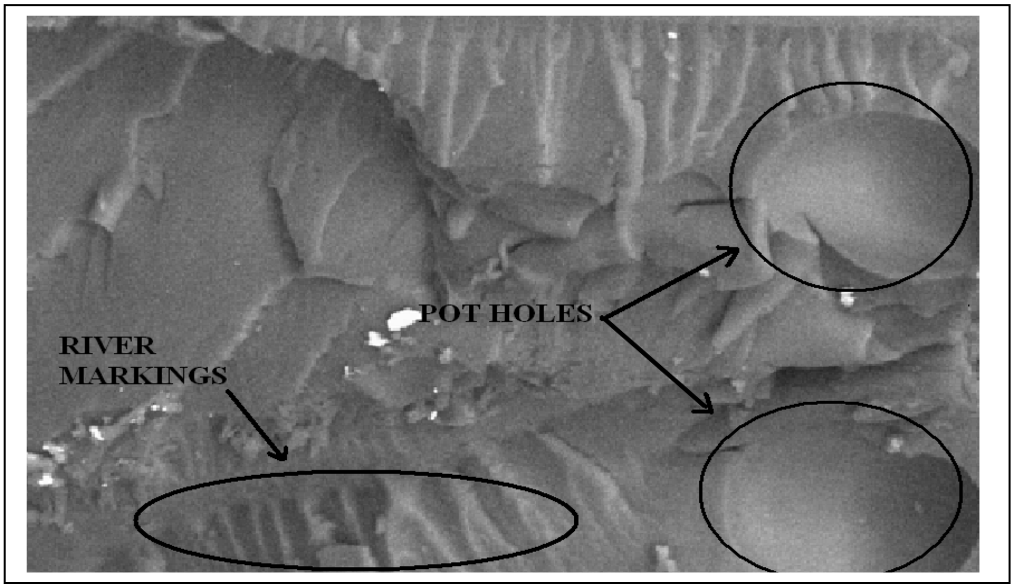


Fig.13. Fracture surface of carbon/epoxy specimen that failed during 3-point bending.

The regions in the above figure.13 shows pot holes that generated may be due to bursting of the entrapped moisture molecule. Mode I tension fractures produced at various angles to the direction of fiber reinforcement typically exhibit river markings. In some cases, the variations can produce relatively large areas of flat-resin fracture with distinct river marks oriented in a consistent direction. Alternatively, extensive amounts of fiber exposure can lead to the existence of extremely localized microscopic areas of fracture. This latter condition results in river marks oriented in a variety of angles across the fiber surface.

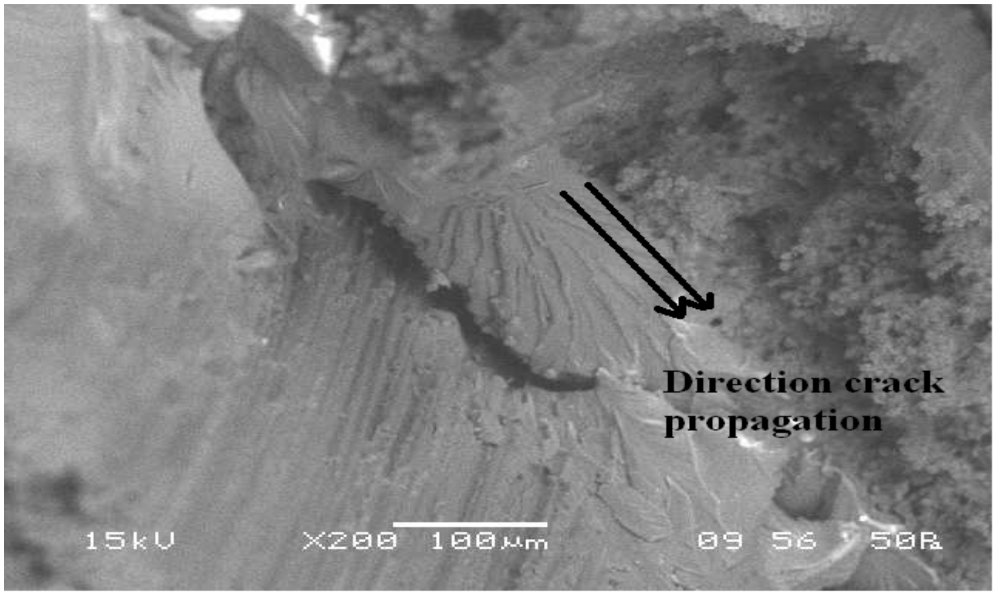


Fig.14 SEM micrograph of carbon/epoxy composite specimen dipped in liquid nitrogen and tested at ambient temperature subjected to flexural loading.

The above micrograph in fig.14 shows the river markings in the matrix. The direction of the river markings shows the crack propagation as shown in the figure. These river marks correspond to fracture ridges formed by minutely displaced failure planes. As crack propagation occurs, these planes link up, resulting in coalescence of this ridge structure to form a river like pattern [36].

4.1.4 Mode II Fracture Mode

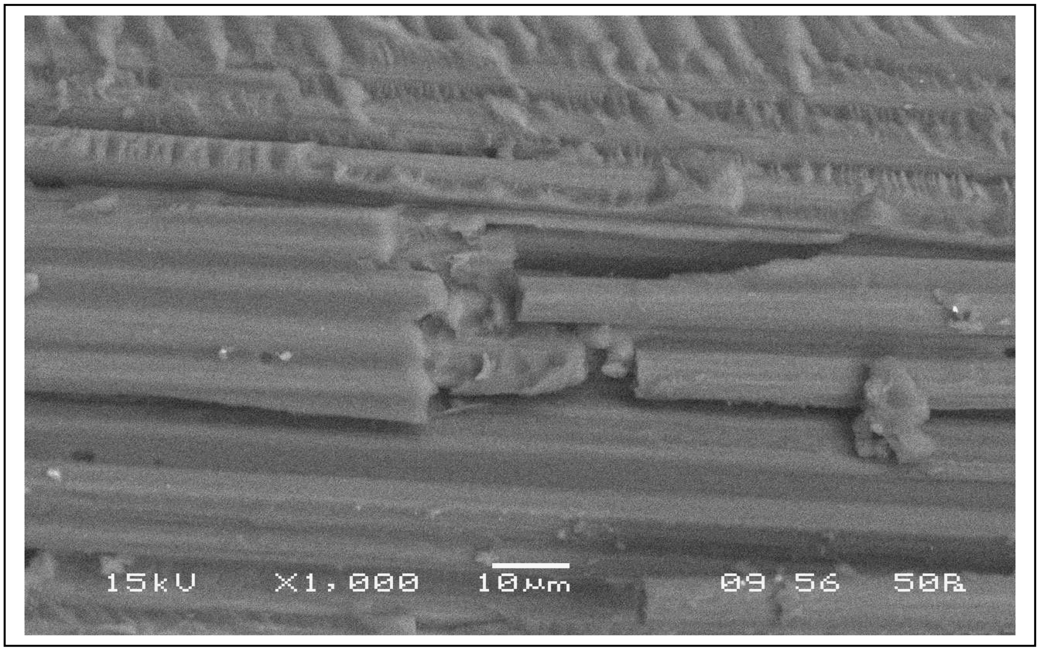


Fig.15 Fracture surface of delaminated carbon/epoxy specimen that failed during 3-point bending.

A few fiber breaks are shown in this fractograph. These are not common in transverse tensile failure surfaces (unless the fibers are somewhat misoriented). The fibers fail at angles to the fracture surface that range from almost perpendicular to very oblique. All the fibers retain a great deal of resin on their surfaces, which indicates that the interfacial bond was strong and that the primary failure mode was resin failure. The top part of the figure shows hackle formation due to shearing [36]. The top region shows the formation of rows of cups [30]. These cups are formed due to the development of transverse micro-cracks along the interfacial area [37, 38]. Increase in brittleness of the epoxy matrix after conditioning causes opening of these micro-cracks easily that develops profile with rows of cups. When these cracks accumulate and merge to form longitudinal cracks along the fiber then failure of the composite results.

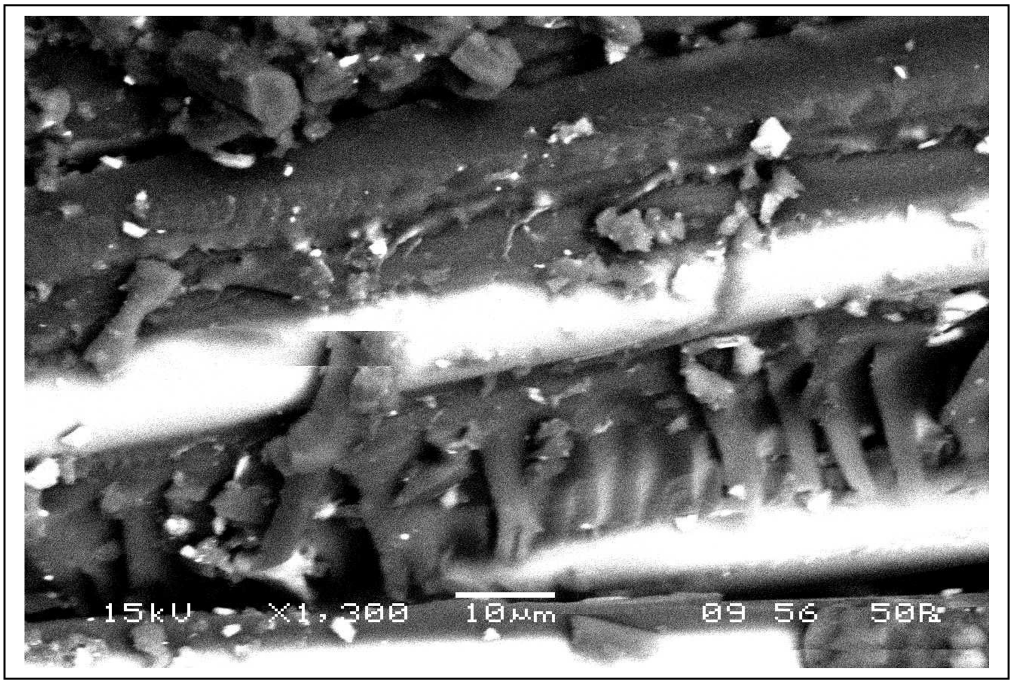


Fig.16 Fracture surface of hybrid (glass and carbon) composite failed in tension.

The properties of hybrid composites are anisotropic. When these are stressed in tension, failure is usually non catastrophic. The carbon fibers are the first to fail, at which time the load is transferred to the glass fibers. Upon failure of the glass fibers the matrix phase will have to sustain the applied load. The fig. 15 shows hackle formation in the matrix phase exhibiting matrix damage.

4.1.5 SOME OTHER FEATURES

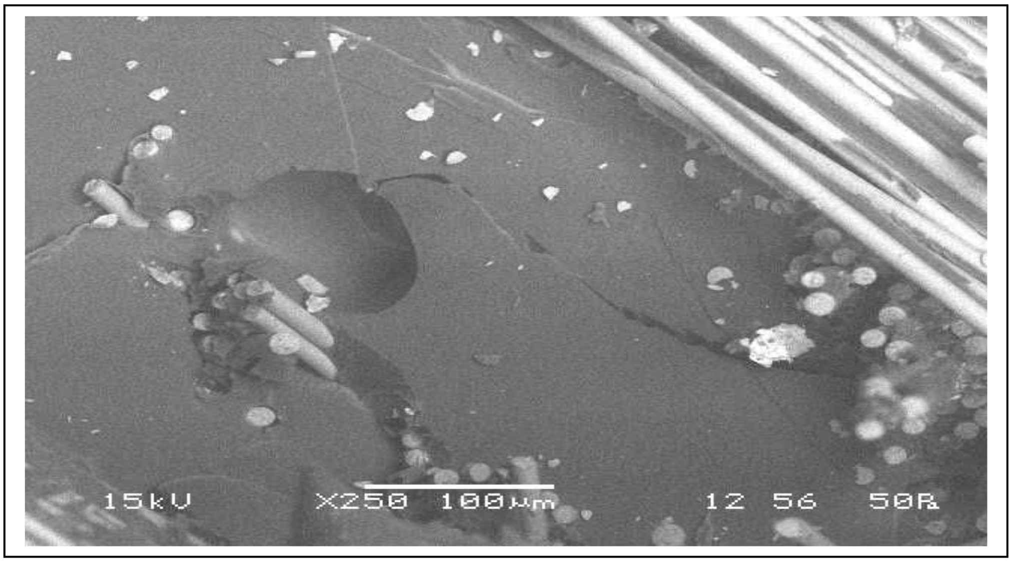
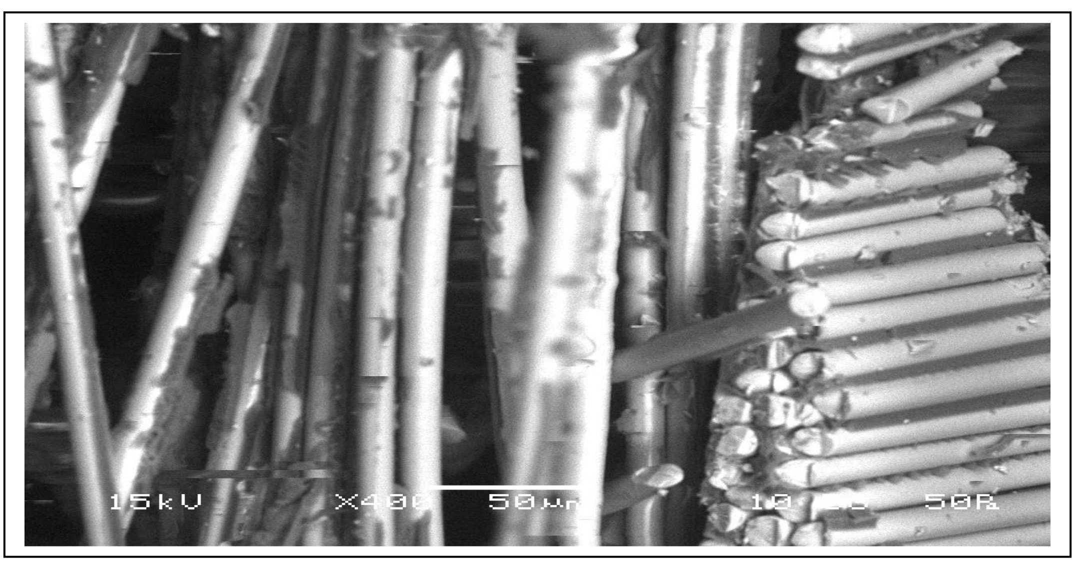


Fig. 17. Fracture surface of glass/polyester composite sample pulled transverse to the fiber surface.

Fig. 17 depicts crack origination from stress concentration region like a bubble in matrix region. The bubble bursts during testing and cracks run in three different directions along the weak interface parallel to fiber.

a)



b)

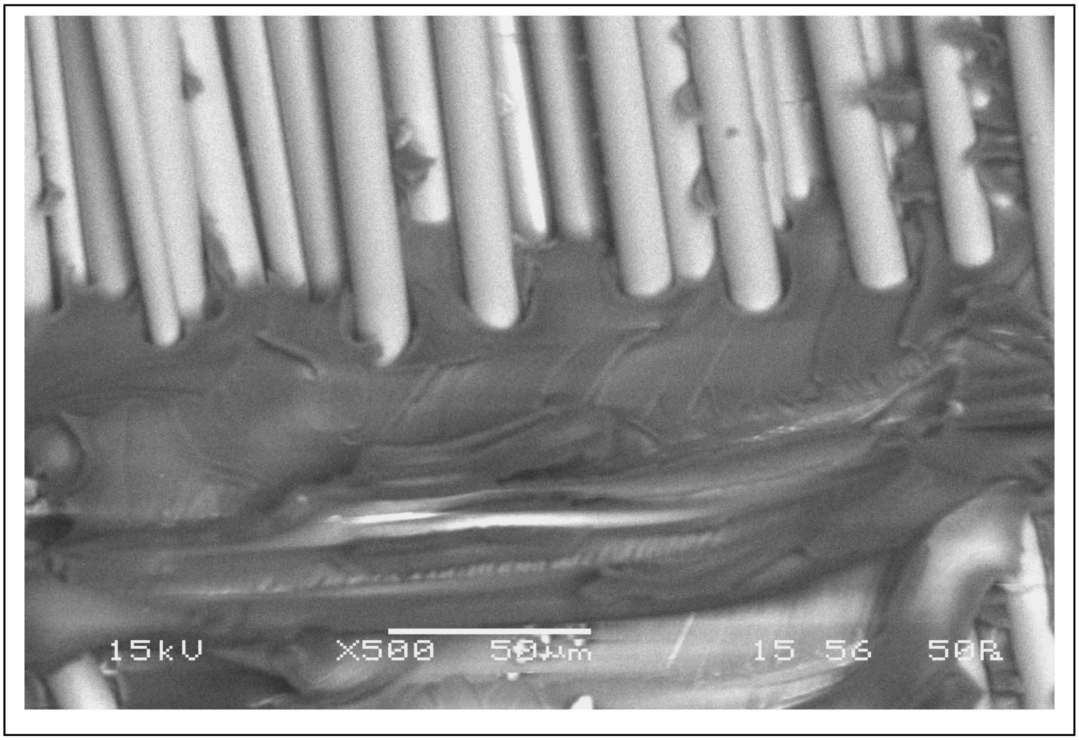


Fig. 18 Fracture surface of longitudinal (0^0) (a) glass/polyester(thermosetting resin) (b)glass/vinylester (thermoplastic resin) composite that failed in tension.

The clean fiber surface of the glass/vinylester(thermoplastic resin) composite shows weak adhesion property between the fiber and the matrix as compared to glass/polyester (thermosetting resin). The reason being, the chemical bonding mechanisms that involve silane coupling agents or other bifunctional molecules apply generally to thermosetting polymers because the organo-functional group is chemically locked into the cross-linked structure of the resin during the chemical curing reactions which change the resin from a liquid to a rigid solid. This type of chemical bonding cannot occur with glass fibers introduced into thermoplastic matrices because the molecules are already fully polymerized.

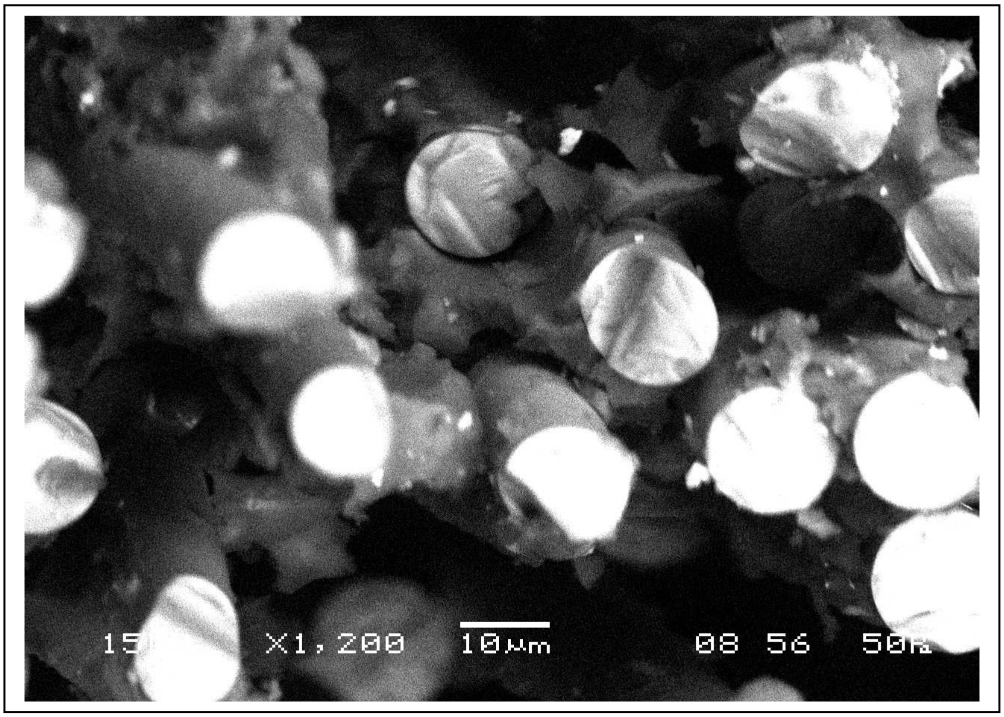


Fig.19. Fracture surface of hybrid (glass and carbon) composite failed in tension.

The above micrograph shows the pull out of the glass fibers in the recessed areas due to weak interface between the glass fiber and the matrix.

Chapter 5

CONCLUSION

5. CONCLUSION

Durability and long life of FRP have been major area of concern. Several models have been developed to explain service failures of composites .However heterogeneous nature of FRP makes the process quite cumbersome. In the present work it’s been tried to explain the failure mechanism actually occurring in the tested samples on the basis of established theories through SEM fractographs . Fracture behavior depends on factors, such as, resin relaxation, state of interfaces, post-curing phenomena, stresses relaxation and development, crazing and cracking in the matrix resin and also micro-void formation because of differential contraction/expansion among constituent phases. Also the micrographs of the multiple failure mechanisms using SEM are developed to determine structural composition and observe defects at the micro-level. By observing the orientation and structure of various primary and secondary features like river patterns, shear cups and hackles, the point of initiation and propagation of insidious crack can be predicted. They also give information about mode of failure and specific response of composite to particular type of loading.. The correlation between the environmental degradation on FRP composite and failure is a vast field for the investigation. By observing carefully the fracture surface of the composite the factors affecting their respective failure and the type of environment they were subjected to could be determined.

Chapter 6

Scope for future work

6. SCOPE FOR FUTURE WORK

The utilization of FRP holds a promising future . The important areas of application of Fiber Reinforced Polymers (FRP) includes the automotive and aeronautical industry, bridge structures, water and waste systems and more recently in the offshore exploration and oil production. These areas of applications require a better study of effect of temperature (both high and low), moisture, humidity, various loading rates and other environmental effects on FRP's. Due to such wide range applications the study of failure mechanisms in specific environment and loading condition has gained importance. Composites can produce complex features exhibiting interlaminar, intralaminar and translaminar planes of separation. Because the occurance of such conditions as interlaminar fracture can often be quite extensive and can occur on multiple planes , the decision as to which surface to examine and how to examine, it often represents one of the most difficult tasks involved in failure analysis. The relationship between the environmental factors, the loading condition and failure of FRP through crack propagation is very complicated so its precise prediction and calculation requires detailed analysis of fractured structure using fractographs and analytical models. The information obtained can be subsequently used for developing better fabrication techniques and preventive measures during service condition for ensuring long life and durability of FRPs.

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