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REVEALING OF FAILURE MODES OF FRP COMPOSITE BY MICROSCOPIC TECHNIQUE

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

Bachelor of Technology
in
Metallurgical and Materials Engineering

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CERTIFICATE

This is to certify that the thesis entitled, "REVEALING OF FAILURE MODES OF FRP COMPOSITES BY MICROSCOPIC TECHNIQUES" submitted by SUSMIT KUMAR DEB AND CHIRANJEEVEE 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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B.Tech

Metallurgical and Materials Engineering

ABSTRACT

Fiber-reinforced composite materials are used extensively in stiffness critical, weight sensitive structures such as those found in aerospace and motor racing where strength to weight ratio is very much important. 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. The presence of moisture and the stresses associated with the moisture induced expansion may cause lowering of damage tolerance and structural durability. In case of water absorption there are both reversible and irreversible changes in the mechanical properties of the thermoset polymers. Delamination between layers is an important problem in applications of fiber reinforced composite laminates. This paper is an attempt to study the cracked surface and reveal the failure mechanism that has occurred using microscopic techniques. 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 glass/epoxy and jute/epoxy composites under various environmental conditions were studied revealing the failure modes (delamination sites, debonding, fiber pullout regions, crack propagation front, striations and bubble bursting in the matrix). AFM micro graphs were studied to give a precise outlook of the behavior of composites to the changing environmental conditions being exposed to such as moisture absorption etc.

CONTENTS

1. INTRODUCTION

1.1 OVERVIEW:

Fibre composites were originally developed for the aerospace industry and have found their way into a much wider range of applications including transportation, sports, medical science and more recently the building and construction industries.

The application of the fibre composites in the construction industries has created opportunities for a new and innovative approach to structures that have undergone little change over the past. The unique mechanical and chemical characteristics of the fibre composites combine to challenge the supremacy of conventional materials such as steels, timber and concrete particularly in areas that are weight and corrosion sensitive.

A major advantage of GFRPs is that composite properties can be optimized for a specific application by varying the design factors [1], such as fibre volume content, fibre architecture, type of resin, and the chemical nature of the sizing applied to the surface of the fibre.

A composite consists of fibres embedded in or bonded in the matrix with distinct interfaces between the constituent phases. The fibres are usually of high strength and modulus and serve as the principal load carrying members. The matrix must keep the fibres in a desires location and orientation, separating fibres from each other to avoid mutual abrasion during periodic straining of the composites. The matrix acts as the load transfer medium between fibres. The matrix is more ductile than the fibres so also it is the source of composite toughness.

Fibres, in general, possess higher tensile strengths compared to the tensile strengths of the same material in the bulk form which is due to one or more of the following reasons:

(a) Absence of defects of a critical size,
(b) Non-equilibrium structures obtained during preparation of fibres,
(c) Orientation of molecules along the fibre direction in the case of molecularly anisotropic materials like polymers, and
(d) Favorable residual stresses introduced during the fibre manufacturing process.

The higher strength of materials when they are converted to fibres has been the main driving force behind the development of composites. Further the glass fibre composites will also have other desirable properties like greater resistance to impact damage.

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, and 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 fibre composites.

The main advantages of composites are:

- To increase stiffness, strength or dimensional stability
- To increase mechanical damping
- To increase toughness (impact strength)
- To modify electrical properties
- To reduce costs
- To increase chemical wear and corrosion resistance
- To improve design flexibility

The light weight of the composites brings down the fuel consumption dramatically thereby increasing the overall engine efficiency. That is why composite materials are making inroads in aero and auto industries.

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 [3]. 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. 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. The mechanical behavior of a composite material is decisively controlled by the fiber-matrix interface. Its properties influence the integrity of composite

behavior because of its role in transferring stress between the fiber and the matrix.

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. The environmental action, such as high moisture and high temperature can limit the usefulness of polymer composites by deteriorating mechanical properties during service.

For all the wonderful properties that are possessed by the composites they have a major drawback. There is a degradation of material property during its service life as it is often subjected to environments of severe changing parameters.

1.2 THE MATERIALS:

1.2.1 THE FIBRES:

(a). GLASS FIBRES-

There are basically five varieties of glass fibres used in composites4. These are E-glass, S-glass, R-glass, AR-glass and Z-glass (zirconia containing glass fibres). E-glass fibres are by far the most widely used glass fibres. These are used in resin matrix composite for structural and electrical applications. S-glass and R-glass fibres have superior mechanical properties than E-glass fibres. They are generally used in Defenses and aeronautical applications. AR- and Z-glass fibres possess good resistance to alkaline environments and are generally used as reinforcements in cement matrix composites.

(b). CARBON FIBRES-

Carbon fibres are prepared by carbonization of a precursor fibre in inert atmospheres at high temperatures (1600 to 2200 °C). The precursor can be an organic polymer fibre like rayon or polyacrylonitrile, or it can be petroleum or coal tar pitch fibre. The structure and properties of carbon fibres depend on the nature of the precursor and the conditions of carbonization. Carbon fibres from rayon precursors do not possess high strength. The tensile strength of these fibres can be improved by employing a high temperature stretch graphitization. This is mainly due to the fact that

cellulose materials yield lower percentage of carbon on heat treatment resulting in higher porosity. The carbon obtained on heat treatment of cellulose is glassy carbon which is difficult to graphitize.

Pitch-based carbon fibres have structures very close to that of graphite. Pitch being a mixture of a variety of high molecular weight compounds made up of fused benzene rings, yields soft carbons which graphitize readily at high temperatures. The highly graphitized structure of pitch-based carbon fibres results in high tensile modulus of the fibres. Because of the high modulus, the fibres become extremely sensitive to the presence of defects. Thus the strength of the pitch-based fibres are primarily determined by the number and criticality (size) of the flaws. The critical size of the flaw in high modulus pitch-based fibres is estimated to be around 45 to 60 nm.

(c). ARAMID FIBRES-

Aramid fibres are synthetic organic fibres prepared from aromatic polyamides. These are high strength and high modulus fibres with properties suitable for use in composite materials.

1.2.2. THE MATRIX-

The materials used for matrix are epoxy, unsaturated polyester and vinyl ester. 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. 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. 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.

2. LITERATURE SURVEY

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.

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.

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 and glasses. 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. Body parts of race cars and some automobiles are composites made of glass fibers (or fiberglass) in a thermo set matrix. 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.

2.3 THE INTERFACE-

The interface is the main field of interest when it comes to composites. The integrity of the composite as a whole depends upon the ease and effectiveness with which a load can be transferred within the composite. The interface is the boundary across which load is transferred and is of so such importance.

2.4. THE MECHANISM OF BONDING-

The nature of bonding is not only dependent on the atomic arrangement, the molecular conformation and chemical constituents of the fibre and the matrix but also on the morphological properties of the fibre and the diffusivity of elements in each constituent [3]. The interface is specific to each fibre-matrix system.

(a). Adsorption and wetting—

Good wetting of fibres by matrix materials during the impregnation stages of fabrication is a prerequisite to proper consolidation of composites. Bonding due to wetting involves very short range interactions of electrons on an atomic scale which develop only when the atoms of the constituents approach within a few atomic diameters or are in contact with each other. The surface energy of the reinforcements in composites must be greater than that of the matrix resin for proper wetting to take place.

(b). Interdiffusion—

A bond between two surfaces may be formed by the Interdiffusion of atoms or molecules across the interface. A fundamental feature of the interdiffusion mechanism is that there must exist a thermodynamic equilibrium between the two constituents. The bond strength in the polymer composites will depend on the amount of the molecular entanglement, the number of molecules involved and the strength of the bonding between the molecules. This may be promoted by the presence of solvents and the amount of interdiffusion will depend on the molecular conformation, the constituents involved and the ease of molecular motion.

(*c). Electrostatic attraction-*

A difference in the electrostatic charge between constituents at the interface may contribute to the force of attraction bonding. The strength of the interface will depend on the charge density.

(d). Chemical bonding—

Chemical bonding method is based on the formation of a primary bond at the interface. This type of adhesion is a two part process-the first part is the removal of a weak layer from the fibre surface particularly at low levels of treatment and the

second part is chemical bonding at the sites. A bond is formed between a chemical group on the fibre surface and another compatible chemical group in the matrix, the formation of which results from usual thermally activated chemical reactions.

(e). Mechanical bonding—

Mechanical bonds involve solely mechanical interlocking at the fibre surface. It is promoted by surface oxidation treatments which produce a large number of pits, corrugation and large surface area of the carbon fibre is a significant mechanism of bonding in carbon fibre polymer matrix.

2.5. The Environmental Conditions:

(a) Effect of moisture:

Most FRPs are likely to be exposed to rain, humidity and moisture or diffused solutions through other substrates during their service life. It has been observed that [12] water accumulated at the fibre matrix interface contributes significantly to the loss of shear strength of the material. The ingress of water through the voids present in a composite can cause plasticization of the matrix. Plasticization occurs in a number of ways including reduction in the glass transition temperature of the matrix, lowering of operating temperature and reduction in operating temperatures and reduction in stiffness and strength properties. Particularly in case of glass fibres, it leaches alkali oxides (sodium and potassium) from the fibre surface, forms surface micro cracks, slow decomposition or dissolution of the glass fibre,

permanent loss of strength and this could even accelerate with increasing temperature and stress level.

The presence of moisture at the interface can modify the interfacial adhesion thereby affecting the mechanical properties of the FRP composites. Moisture absorption in the composites introduces dilatational stresses [20]. During moisture absorption, the outside ply of a composite laminate is in compression. This results from the outer ply trying to swell, but being restrained by the dry inner plies. Similarly, on desorption, the outer plies try to shrink, but are restrained by the wet swollen inner plies. This results in tensile stresses in the outer plies. Consequently the mechanical properties and long term durability show a marked deterioration.

(b) Effect of alkaline environments:

The alkaline solutions can cause degradation to the main constituents of the composites. Particularly with bare glass fibres, a reaction with an alkaline solution forms expansive silica gels. However the composite material as a whole show superior performance and durability characteristics than more conventional constructional materials. The use of a suitable polymeric resin for the matrix becomes thus an important criterion to save the fibres from such attack providing a protective barrier.

(c) Effect of UV radiation:

Solar UV radiation is deleterious to organic materials. The wavelength of the rays that reach the earth's surface almost coincides with the dissociation energy of most polymers. On prolonged exposure of the composite to the sunrays, the matrix hardens and colour change and pigment loss can also occur. But they are constricted to the top few layers only.

(d) Effect of cryogenic conditions:

Cryogenic temperature is the state of utmost low temperature. The FRP composites are adversely affected by such a low temperature. The matrix hardens and the difference in the elastic modulus between the fibre and the matrix creates an environment of residual stresses to be developed in the matrix. The fibres do not allow the matrix to contract as they are in close contact. The failure of the matrix is hence a prominent observable phenomenon.

(e) Effect of acidic treatment:

In case of FRP composites when subjected to acidic treatment, it was found that the inter laminar shear strength was decreased when exposed to concentrated acidic solution due to the stress corrosion cracking of the laminates as well as the micro cracks developed in the matrix [9]. The amount of water absorbed by the matrix was though lesser for high concentration acidic solution. Further as the cross head speed was decreased, the stress induced cracks were found to be low as more time is now available for redistribution of load.

2.6. THE FAILURE ANALYSIS:

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 characterize the fracture morphologies of specimens failed under known (pure) failure modes, and then compare these morphologies to 'unknown' failures.

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.

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 fractures are generated.

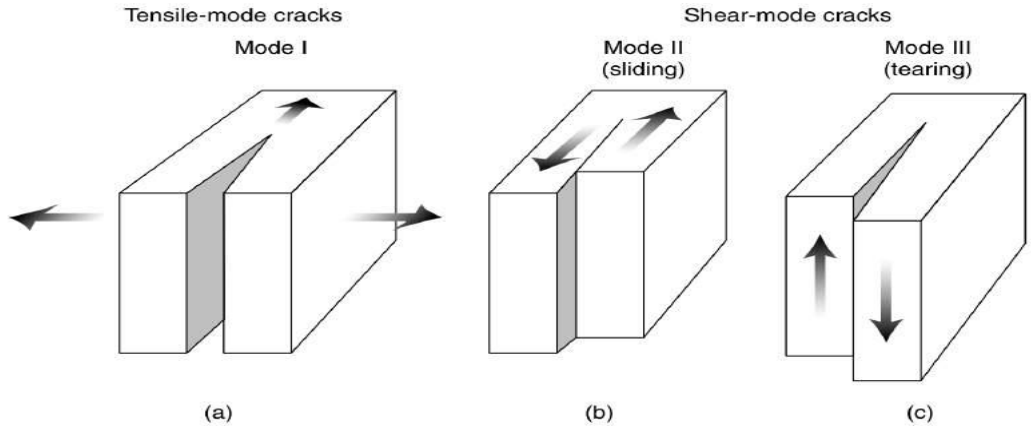


Fig 1. Crack opening modes.

(a) DELAMINATION:

Delamination is a critical failure mode in composite structures, not necessarily because it will cause the structure to break into two or more pieces, but because it can degrade the laminate to such a degree that it becomes useless in service. The interfacial separation caused by the delamination may lead to premature buckling of the laminate, excessive vibration, intrusion of moisture, stiffness degradation and loss of fatigue life [10]. The delamination though in some cases may provide stress relief and actually enhance the performance of the component.

Delamination may be introduced during processing or in service conditions. It may result from low velocity impact, from eccentricities in the structural load path or from discontinuities in the structures which may induce a large out of plane stress.

Even in the absence of such discontinuities delamination may also result from in plane compressive loading causing global or local buckling.

In addition to mechanical loads, the moisture and temperature may also induce interlaminar stresses in a laminate. These may be the results from the residual thermal stresses caused from cooling from processing temperatures and residual stresses created by the absorption of moisture. The delamination may lead to redistribution of stresses which would eventually promote gross failure.

Individual modes of energy release rate along the delamination front are calculated based on the Irwin's concepts of linear elastic fracture mechanics and subsequent developments by Rybicki and Kanninen, 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.

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

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 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. Then the energy rate is calculated as [5]:

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

The three components of strain energy release rates for Mode I, Mode II, and Mode III respectively are:

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

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

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

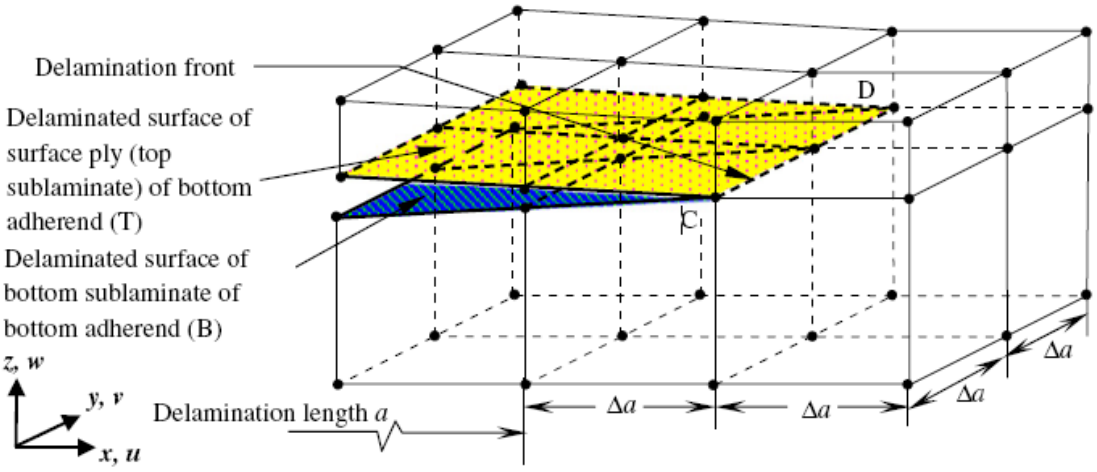


Fig 2. Schematic representation of a crack front.

(b) FIBRE PULL OUT AND DEBONDING:

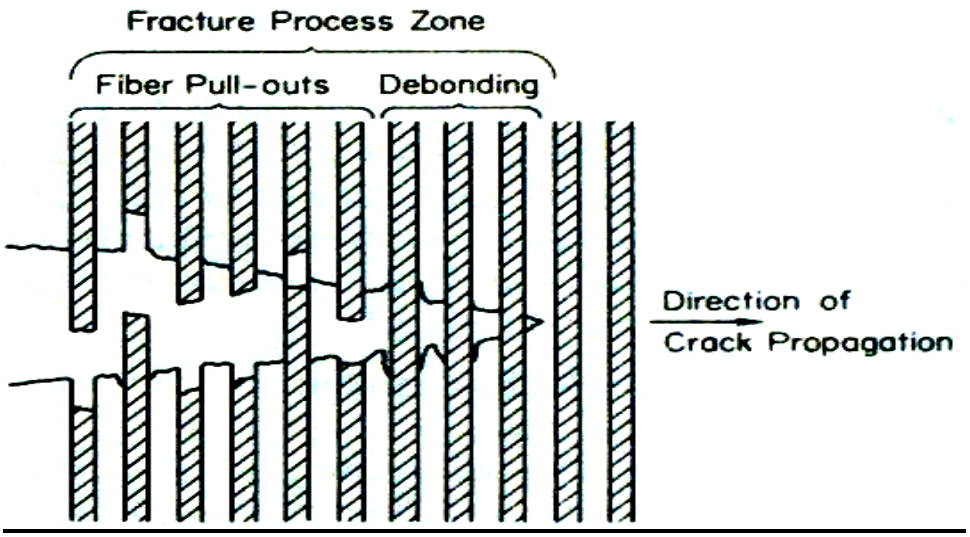


Fig 3. Crack tip showing local failure events.

At some distance ahead of the crack the fibers are intact. In the high stress region near the tip, they are broken, 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. When brittle fibres 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 laminate in a laminated

composite, a crack can split and propagate along the interface, thus producing the delamination crack.

The most significant property improvement in fibre reinforced composites is that of fracture toughness. Toughness is quantified in terms of the energy absorbed per unit crack extension and thus any process which absorbs energy at the crack tip can give rise to an increase in toughness. In metallic matrices, plastic deformation requires considerable energy and so metals are intrinsically tough. In fibre reinforced materials with both brittle fibres and brittle matrices, toughness is derived from two sources. Firstly, if the crack can be made to run up and down every fibre in its path the there will be a large amount of new surface created for a very small increase in crack area perpendicular to the maximum principal stress - INTERFACIAL ENERGY - and in order to get the fibres to break they have to be loaded to their fracture strength and this often requires additional local elastic work, and secondly if the fibres do not break and therefore bridge the gap then work must be done to pull the fibres out of the matrix - FIBRE PULLOUT[23]. Using simple geometric models we can estimate the contribution of each of these processes to the overall toughness of the composite.

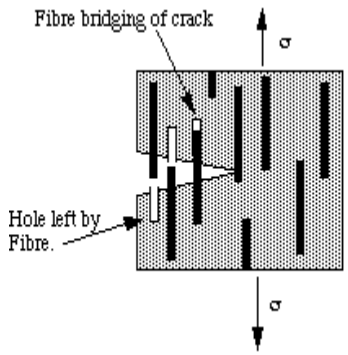


Fig 5 showing a crack front

W is the work done in pulling out a single fibre is the integration of f(x)dx over a distance l_0.

$$W = \int_0^{l/2} F(x)dx$$

$$? = \int_0^{l/2} \pi d \frac{\sigma_m}{2} x dx = \left[\frac{\pi d \sigma_m x^2}{4} \right]_0^{\frac{l}{2}}$$

$$? = \frac{\pi d \sigma_m l^2}{16}$$

The total work done in extending the unit crack area is:

$$G = \pi d \sigma_m \frac{l^2}{16} \times \frac{4f}{\pi d^2}$$

$$? = \frac{f \sigma_m l^2}{4d}$$

$$G_{max} = \frac{f \sigma_m l_c^2}{4d} = \frac{fd}{4} \left(\frac{\sigma_f^2}{\sigma_m} \right)$$

The symbols here denote usual meanings unless stated.

The longest fibre that can be pulled out is the critical fibre length, l_c, which in turn depends on the fibre fracture strength, s_f. Thus a combination of strong fibres in a relatively weak fibre/matrix interface gives the best toughness.

(c) MATRIX MICROCRACKING:

The first form of damage in laminates is often matrix micro cracking. They are intralaminar or ply cracks that traverse the thickness of the ply and run parallel to the fibres of the ply. The most common observable micro cracking is cracking in the 90^0 plies during axial loading in the 0^0 direction. These micro cracks are transverse to the loading direction and are often termed as the transverse cracks. Micro cracks may be observed during tensile loading, during fatigue loading, during changes in temperature and during thermo cycling. Micro cracks can form in any plies but predominantly they are found implies off axis to the loading axis. The immediate effect of the micro cracks is to cause degradation in the thermo mechanical properties of the laminate including changes in all effective modules, Poisson's ratio and thermal expansion coefficients. Another detrimental effect of the micro cracks is that they nucleate other forms of damage such as induction of delamination, fibre breakage or provide pathways for the entry of corrosive liquids. Such damage modes may subsequently lead to laminate failure.

The first micro crack causes very little changes in the thermo mechanical properties of the laminate. Continued loading however normally leads to additional micro cracks and additional micro cracks and continued degradation in the thermo mechanical properties. A change in temperature induces residual stresses between the plies and hence can lead to micro cracking. Due to the presence of moisture as well can induce residual stresses that can subsequently influence micro cracks.

3. EXPERIMENTAL PROCEDURE

The samples were collected from the laboratory of the Guide. The samples were already fabricated, conditioned and tested.

Fabrication was mainly done by hand lay out process using glass and jute fibres with epoxy as the main resin to be used as matrix.

The conditions include:

- Cryogenic treatment at a temperature of 77K for different time cycles and different crosshead speeds.
- Hygrothermal treatment at 60^0C at a humidity level for 25 hrs.
- Ambient temperature.
- Ultraviolet rays treated for different crosshead speeds.

The samples were then taken for the SEM analysis for the study of fracture morphology. The surfaces to be examined were cleaned and baked for 2 hrs at a temperature of 50^0C for removing any moisture or dirt that might be present.

The samples were taken for a brief AFM analysis. It was mainly done for the testing of the effect of moisture absorption on the samples.

4. RESULTS AND DISCUSSION

4.1. SEM ANALYSIS:

(a) Glass Fibre Composites:

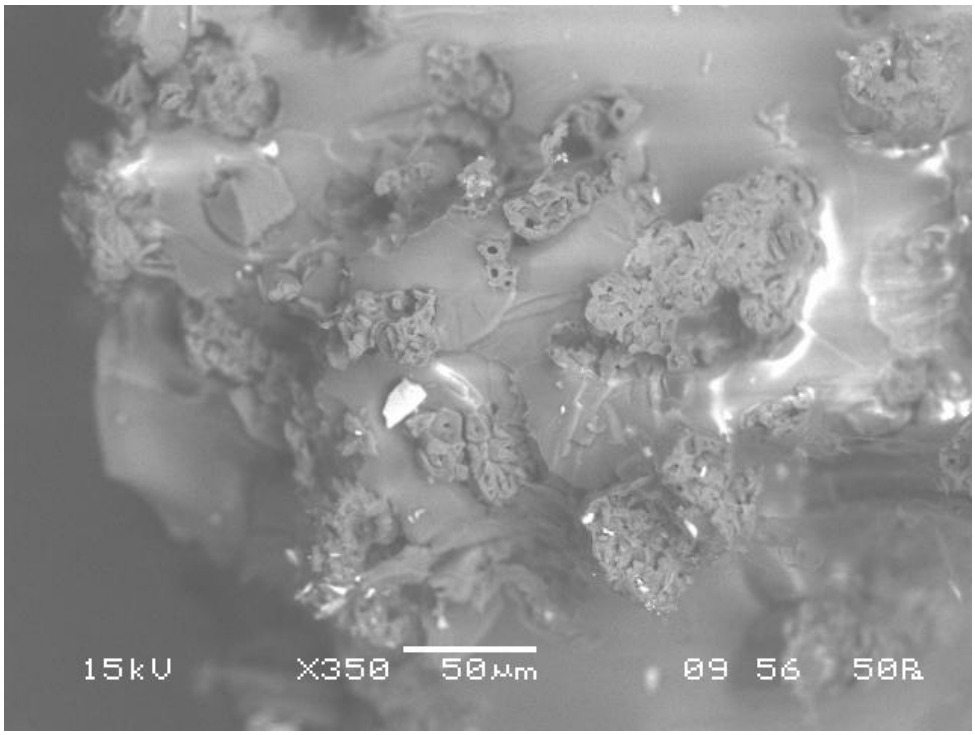


Fig 6(a).

The glass fibre composite in fig (a) has shown enormous amount of fibre pull out. This was mainly because of the weak adhesive bonding between the constituent glass fibre and the epoxy resin matrix. The fabrication process that was used could be the reason for uneven distribution of the stress being applied. The prominent reasons could be those to be present during the curing process as well as the weak interfacial bonds between the constituent phases.

The fig 6 (b) shows the weak bonding between the matrix and the fibres for glass fibre epoxy composite. It shows the cracking of the matrix as well as the debonding between both the phases. This could be due to the residual stress present while curing as well as due to the fabrication techniques used. Small amount of moisture if present can reduce the bond strength between the phases resulting in differential strain which is created by the expansion force exerted by the liquid while stretching polymeric chains and induce addition residual stresses.

Fig 6 (b).

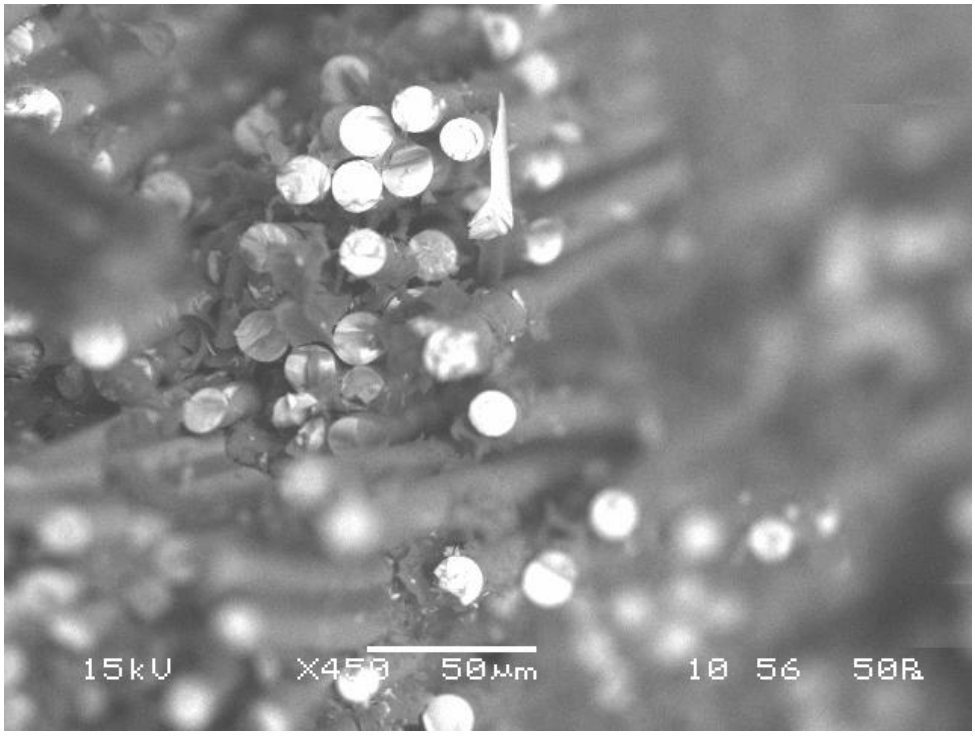


Fig 6(c).

The fig 6(c) shows the large extent of the fibre fracture. The amount of stress being applied was unable to be sustained by the fibres when distributed on them by the matrix due to the inability of the formation of strong interfacial bonds. The glass fibres were in aptly held by the matrix which was followed by the rupture of the fibres which may be due to the localized stress and strain fields in the fibrous composite.

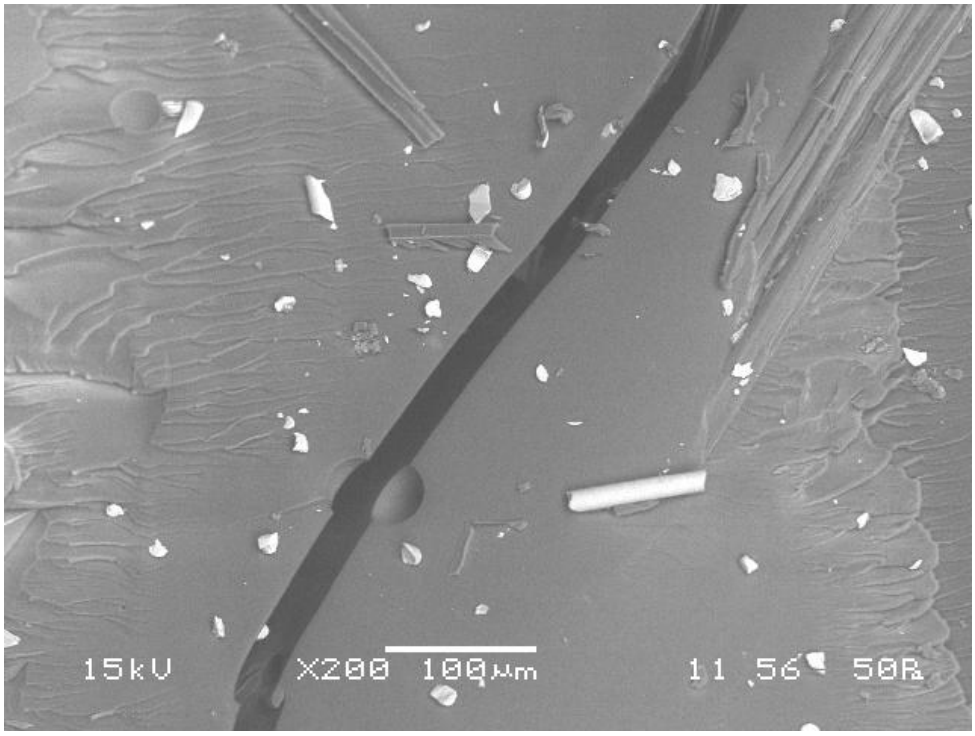


Fig 6(d).

The fig 6(d) shows the propagation of a delaminated crack front along the interface. This could be a result of the manufacturing defects, the generated out of plane stresses and also the laminate geometry. There are as well a large number of striations being seen as smooth lines on the matrix. The damage may begin with the formation of striations/microscopic cracks (crazing) in the matrix or at the fibre/matrix interface. When these cracks develop to a certain density and size, they tend to coalesce to form macroscopic matrix cracks.

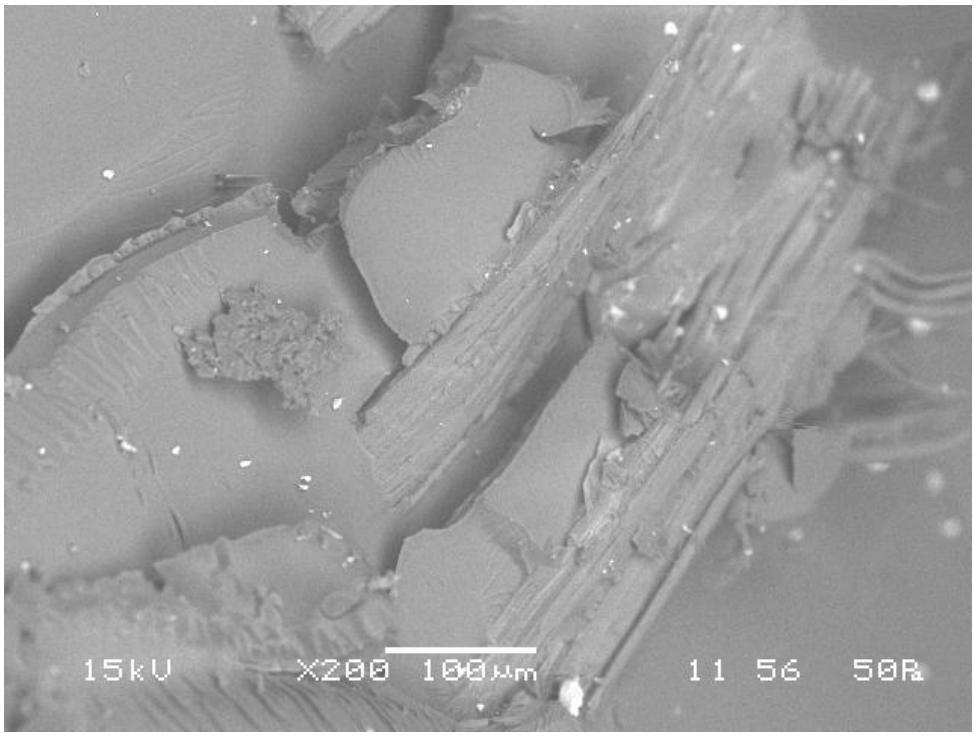


Fig 6(e).

The fig 6(e) shows the bursting of matrix due to the sudden collapse of any entrapped water molecule. The low molecular weight impurities formed by water absorption may migrate from the bulk of the adhesives to form a weak boundary layer at or near the interface. Matrix micro cracking may also lead to such a burst.

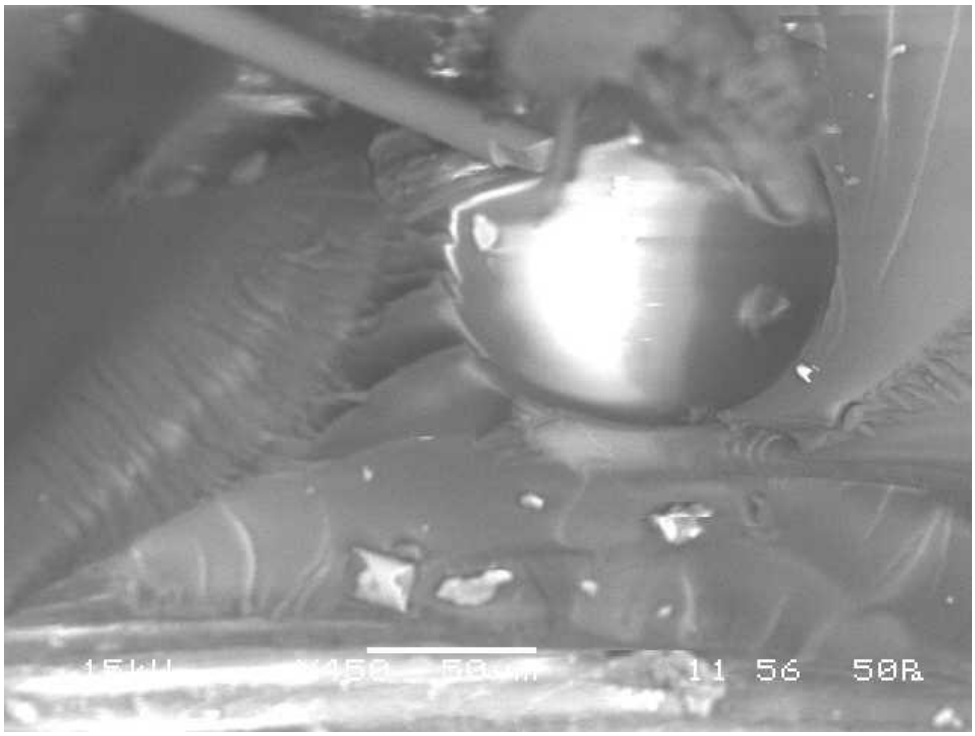
(b) JUTE FIBRE:

Even in case of jute fibre cryogenetically treated for 3 hrs, one could see the large amount of matrix fracture and micro cracking [fig 7 (a)] which may be due to brittleness of the epoxy resin at low temperature leading to nucleation of delamination cracks in the weak fiber-matrix interface.

An appreciable amount of fibre pullout is also observed during such treatment [fig 7(b)]. There is a debonding due to the formation of complex stresses set up as a result of stiffening of the matrix or mismatch of the coefficient of thermal expansion of fibre and matrix. The ILSS is decreased.

In case of jute fibre composite being alkali treated, there is also appreciable amount of fibre pullout [fig 8 (a)] due to the absorption of the alkali in form of solution which may render the jute fibres to be weak as compared to the matrix on preferential absorption of the alkali solution and hence on loading they may not be able to bear the stress generated. At such low temperatures, there is brittle failure of the epoxy matrix.

Fig 8 (b) shows the delamination crack fronts running across the interface meeting the weak interfacial bonds where shear failure takes place. The stress transfer efficiency from matrix to the fibres decreased due to decrease in ILSS.


11 56 50R

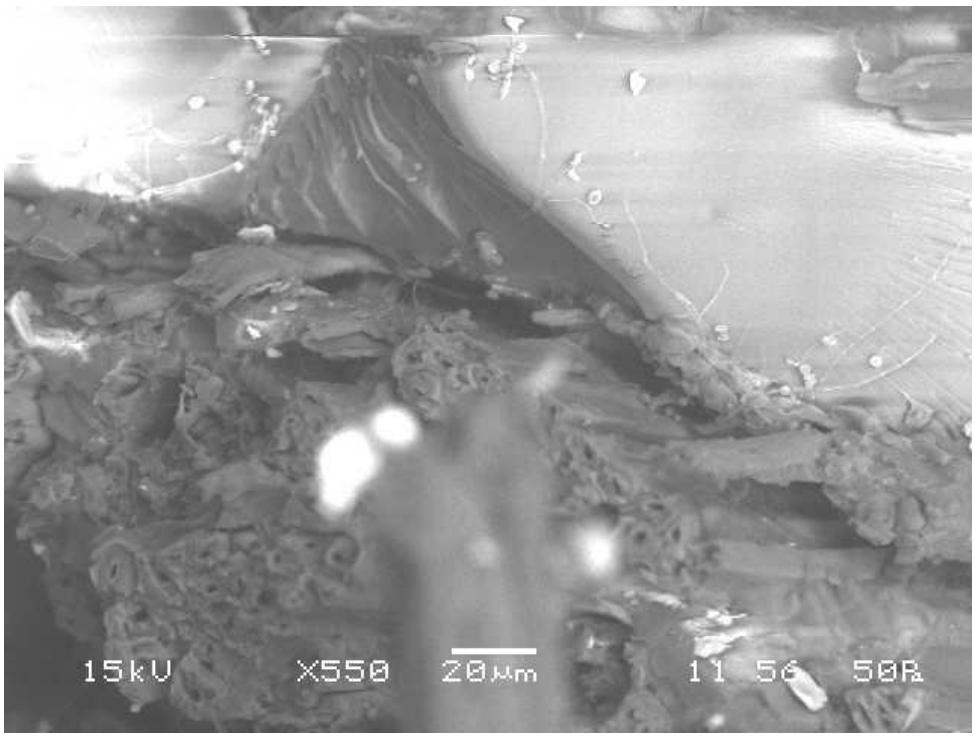

15kV X550 20µm
11 56 50R

Fig 7 (a) and (b). SEM of cryogenic treated jute fibre composite.

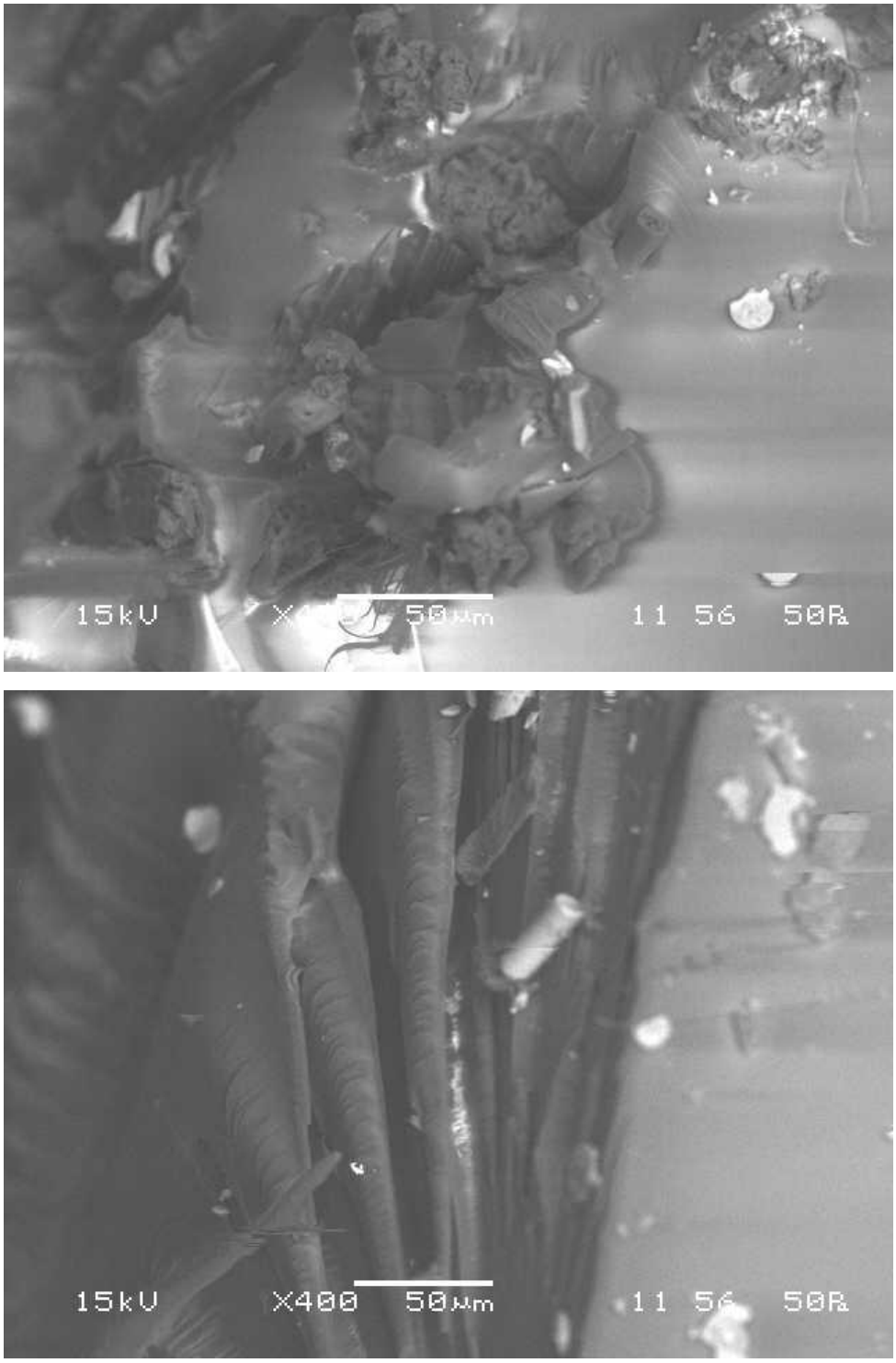


Fig 8 (a) and (b). SEM of alkali treated jute fibre composite.

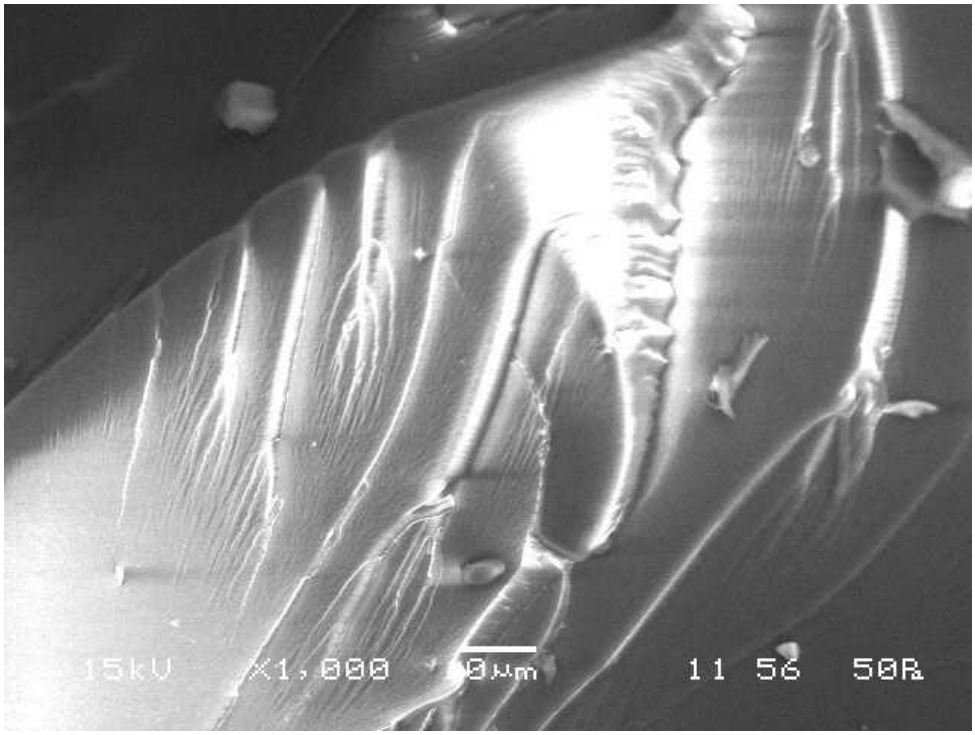


Fig 9 (a). SEM of untreated jute fibre composite.

The fig 9 (a) shows the large number of striations through the matrix caused by the fatigue of the sample. These small cracks join under appreciable energy favored kinetics to form larger cracks to lead to further failure of the matrix.

In case of jute fibre alkali treated under various crosshead speeds, it was observed in fig 10 (a) and (b) that fibre pullout was prominent among all the speeds of 2, 50 and 100 m/sec. and the extent increased with increasing speed.

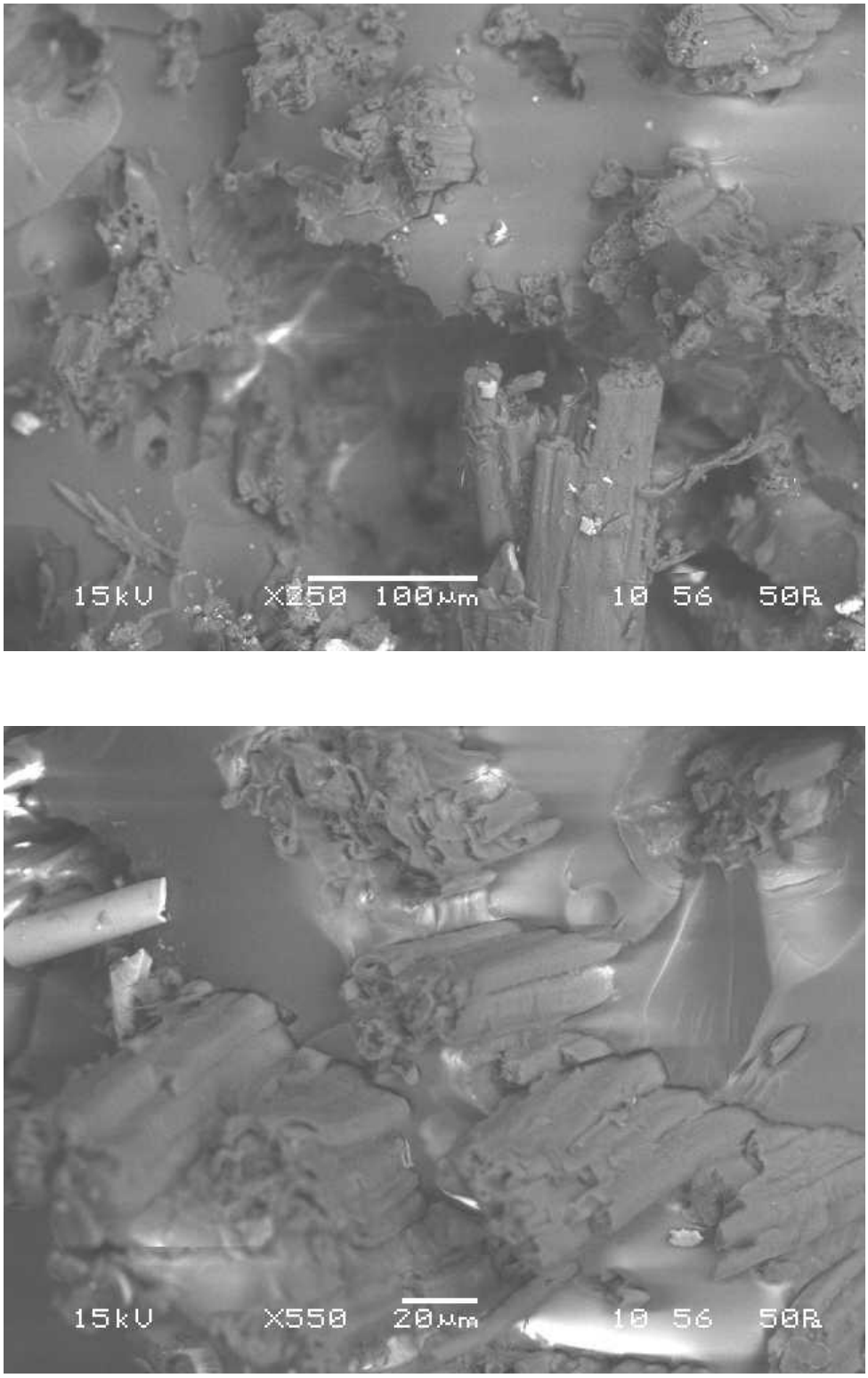


Fig 10(a) and (b). SEM of alkali treated jute fibre composite for 2m/sec and 100 m/sec cross head speed.

The results for untreated jute fibre composite show that there is fibre pullout as well but the extent were comparatively less as in fig 11 (a). The matrix is shown to crack in the tranlaminar direction across the interface.

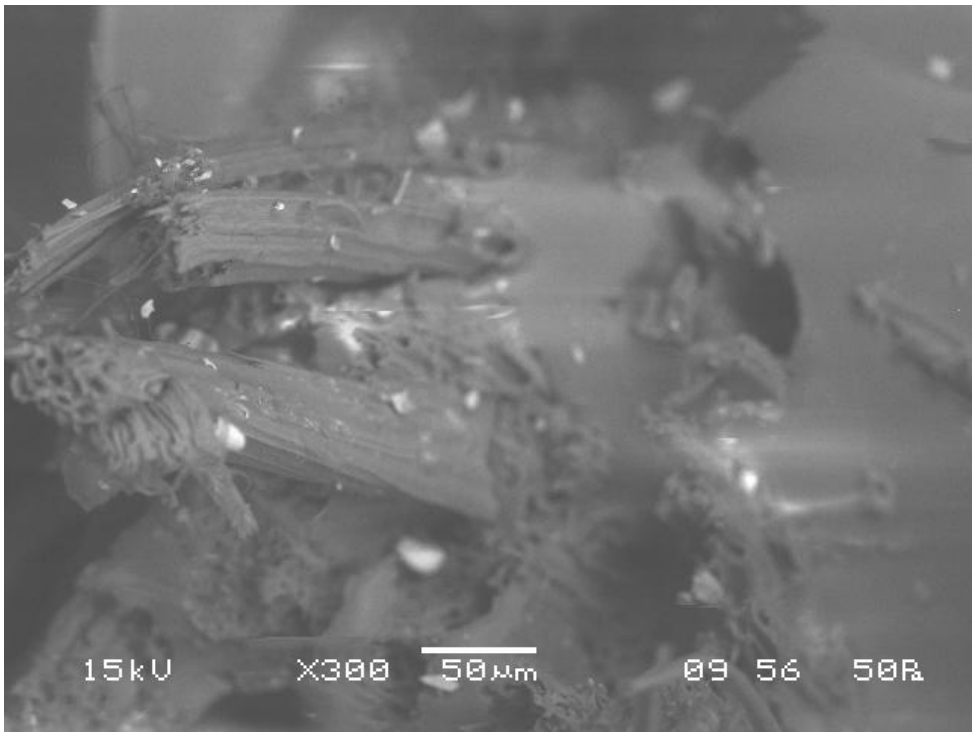


Fig 11(a). SEM of untreated jute fibre composite.

4.2. AFM ANALYSIS:

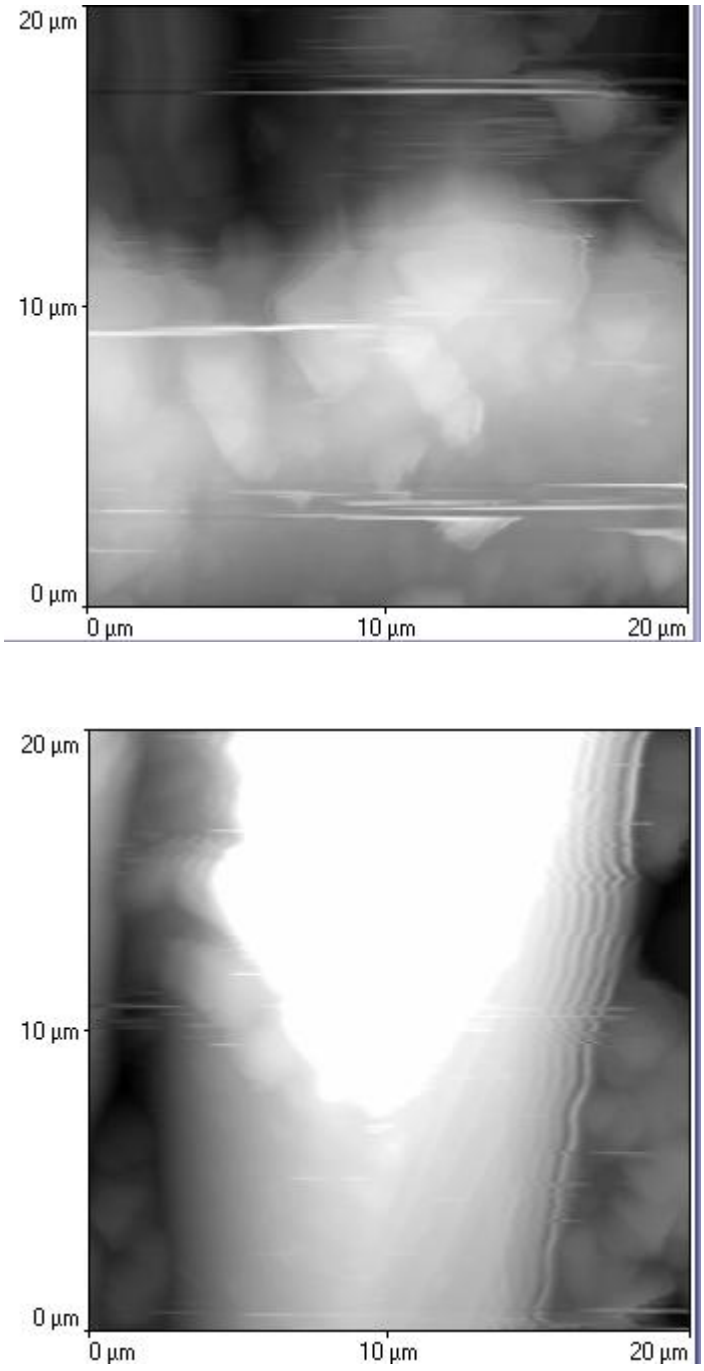
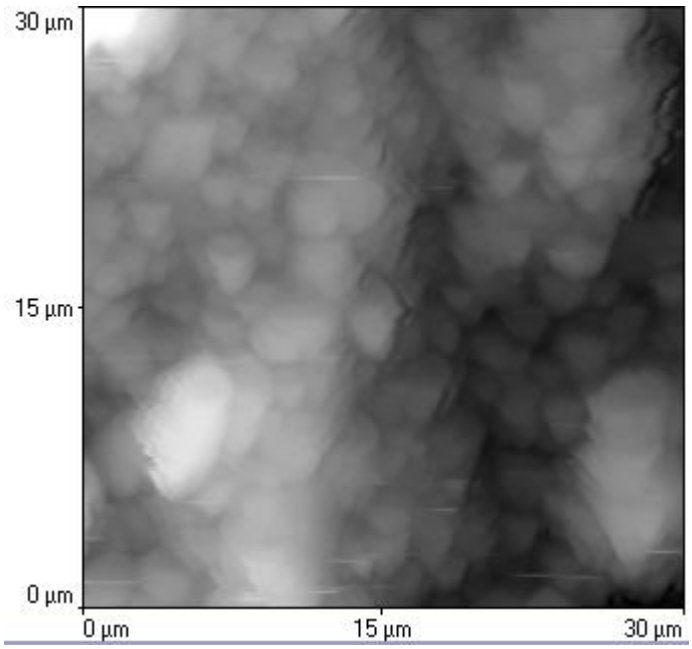


Fig 12 (a) and (b). AFM analysis for glass fibre composite in untreated and hygrothermally treated respectively.

The contrast in the images clearly indicates the swelling of the matrix due to moisture absorption. The rings are more prominently seen with fig 12 (b) which might be attributed to the moisture absorbed. The possible explanation is the plasticization of the matrix due to the moisture effect which facilitates the movement of glass fibres to better orientations. These changes seem to concentrate at the interface area which further implies that degradation of the composite is likely to occur at these interfaces.

The region between the brighter fibre phase and the dull matrix phase is a region of interphase (rather then the conventional interface) which is an area of altogether different chemical and mechanical nature. The chemical reactions taking place between the matrix and the fibre leads to a differential chemistry of the interphase formed. One could also see the layered structure of the matrix near the interface due to preferential moisture absorption by the matrix.



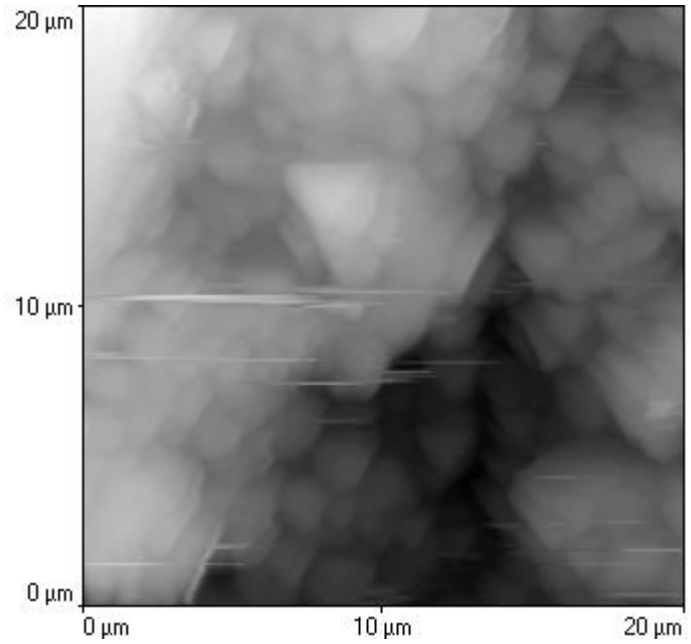


Fig 13 (a), (b) showing untreated and hygrothermally treated carbon fibre.

In case of carbon fibre composite as well there is a contrast in the images showing pronounced matrix and fibres. The weak interfacial bond strength

between carbon fibres and epoxy matrix results in the initiation of interlaminar failure and/or propagation of cracks through this layer.

5. CONCLUSION

Durability and long life of FRP composites 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. They also give information about mode of failure and specific response of composite to particular type of loading. 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.

6. SCOPE FOR FUTURE WORK

Over the last forty years the FRP composites have found widespread applications as an important structural material over their metallic counterparts. However their increasing use in automobile and aerospace industries has also made their durability in the exposed atmosphere a reason of concern. Plasticization and swelling are among the adverse consequences of absorbed water when a composite is exposed to humid atmosphere in operation. The study of the performance of such parts under the effect of the conditions has made the failure analysis of such utility. The FRP composites can be damaged in a number of ways say for example interlaminar, intralaminar or translaminar or a mixed mode of failure. Hence the fractographic study of such materials is so very important. The failure modes can be predicted and the designing can be made such that these can be counteracted.

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