# An assessment of mechanical behaviour and fractography study of glass/epoxy composites at different temperatures and loading speeds

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## Abstract

Effect of loading rate on fracture and mechanical behavior of autoclave cured glass fibre/epoxy prepreg composite has been studied at various loading (striking) rates (0.01-$10^3$mm/min). The maximum load carrying capacity and strain at yield continuously increases with increasing loading speed. The interlaminar shear strength (ILSS) value is high at low loading speed and becomes low at high loading speed with the transition of loading rate at approximately 300 mm/min. The formation of steps, welt interfacial failure and cleavage formation on matrix resin i.e. localized plastic deformation processes were dominating mechanisms for specimens tested at low loading rates, while brittle fracture of fibre, fibre pull-out and impregnation were dominating mechanisms for specimens tested at loading rates of 800 mm/min or higher.

**Key Words**: Glass/epoxy composites; Loading rate; Fractography; Interlaminar shear strength; Environment

## 1. Introduction

Over the past years, prepreg glass/epoxy composites have been successfully used as precursors in the manufacture of high-performance composites such as airplane components, fishing rods, motor vehicles, sporting goods and water skies [1-3]. The epithet prepreg refers to a preimpregnated lamina (viscoelastic material) comprised of aligned fibres embedded in an uncured or partially cured resin. The development and experimental characterization of these marvelous materials, held together primarily through Vander Walls interactions and entanglements, have been the focus of significant research over the past decades. Despite the challenges and expenses inherent in polymer composite processing, applications remains a major topic of interest due to their potential for high strength to weight ratio, specific strength and structural integrity. These properties are translatable to the macroscale through the incorporation of glass fibre reinforcement into epoxy matrix resin and composites. However, these materials while subjected to certain environmental conditions that may prove damaging to installed wraps. These include moisture, UV radiation, thermal effects, alkalinity, humidity and underwater [4, 5]. At high and low temperature exposure plays a dominant role during its short term and long term service life. The interface between the fibre reinforcement and matrix resins can increase resistance to fracture, join materials of different character, make them deform more easily and provide motility. While they represent only a tiny fraction of the overall volume, interfaces are essential for the integrity and durability of the overall composite material [6].

During their service period it comes across many type of loading rates as static to dynamic with wide range of loading rates (striking rates). Few literatures have been addressed the mechanical behavior and fractography at different temperatures [7, 8, 9]. Regarding chemistry of this composite material Huang et al [10] present the degree of impregnation of prepreg through NIR spectra, describing the flow of resin matrix through fiber tows. Grunenfelder et al [11] addressed the effect of voids on both autoclave and VOB precede laminates they observed that the high pressures during autoclave processing were sufficient to restrain the void formation. Boey et al [12] studied use of high pressure of up to 7000 KPa by means of isostatic press without vacuum application the reduction of void levels as 3%. Considering all the research works we followed autoclave curing cycle for the fabrication of composites materials which was discussed in experimental part. On the other hand we know severe environmental exposure affects physical and mechanical properties of polymeric composite materials resulting in an undesirable degradation. As the material is heterogeneous and anisotropic in nature, damage and degradation of material often be strongly influenced by local processes. The anisotropy contributes to more complexity in the assessment of the damage mechanisms and in their impact on the composite responses. An aggressive progress is needed, specifically in the aerospace industry to tailor thermal expansion together with the low material density. Optimization of structural component through improving the prepreg composite material the resin and the fiber is a challenge for the material scientist as well as the process engineer. Therefore, scaling concepts need to be applied in the development of new products to aid the formulation and processing of these heterogeneous, anisotropic and viscoelastic material. Okoli [13] studied the 3-point bend test of glass/epoxy laminates with increasing strain rates to find a relationship between energy to failure and strain rate. They reported a change in failure modes observed as strain rate increased. Shah Khan [14] studied the resistance of glass fiber reinforced polymer composites with increasing loading rates (2 to 1000 KN/sec) and compressive strain rates (10-3/sec to 102/sec). They found that in-plane elastic modulus and strength increases with strain rate and then decreased at higher strain rates. In the present study 3-point bend tests were conducted on glass/epoxy laminates at increasing loading rates to ascertain the relationship between ILSS and loading rate. Very limited work has been devoted related to ILSS with loading rate, which is the fundamental knowledge to understand the reliability and structural applications of GFRP composites. To meet this challenge, researchers and designers must give way to new paradigms, guided by microstructural characterization of complex phenomena that are discovered and then reported in a timely way.

## 2. Experimental procedure and processing

### 2.1 Experimental details

Glass/epoxy (GFRP) prepreg composites which contain the reinforcement as E-glass fiber and matrix as epoxy were prepared. After synthesis of GFRP prepreg composites it was planned to cure with autoclave following the cure cycle shown in Figure 1. GFRP prepreg consists of 60 wt% E-glass fiber and 40 wt% matrix epoxy with a 0/90º weave. The prepreg material used in the testing was the epoxy prepreg made by HEXCEL, supplied by Hindustan Aeronautics Limited (HAL) having specification Hexply 913-37%-7781-1270, containing a proprietary curative and glass cloth filter with a 0/90º weave. This prepreg is unique as it can be stored at room temperature for 24 days.

### 2.2 Processing of the laminates

Glass/epoxy composite laminates were cured by autoclave method following 5 steps of the cure cycle which shown in Fig 1. Here first dwell time at 75ºC for 45 minutes. Whereas second time at 135ºC for 65 minutes. Here pressure is 2.5 bar and vacuum is 0.8 bar. After curing, the laminate was cut into the required size for 3-point bend (Short- Beam Shear) test by diamond cutter. Then stability test was done for the composite laminates. Here the laminates were weighed. One batch of sample was treated with +50 ºC temperature for thermal conditioning whereas another batch for -50ºC for cryogenic treatment tests. For comparison one batch for ambient +28 ºC temperature testes sample. The samples were kept inside the furnace at +50ºC temperature and allowed to stay at that temperature for 10 min as soaking period. Similarly for cryogenic temperature the samples were kept at -50º C temperatures and allowed to stay at that temperature for 10 min as soaking period with help of blowing liquid nitrogen gas.

The GFRP prepreg laminates were then cured by autoclave. The material manufacturer's recommended cure cycle was employed for autoclave processing which is shown below.

1. Heat from room temperature to 30°C at 1-2ºC/MIN
2. $1^{st}$ Dwell at 75ºC±5ºC for 45 MIN
3. Heating rate 1-2ºC/MIN
4. $2^{nd}$ Dwell at 135°±5ºC for 65 MIN
5. Cooling rate 2-5ºC/MIN

The cured panels fabricated from cure cycle outlined above were subjected to conditioning. The number of test specimens employed for each test is given in section 2.3.

## 2.3 Characterization equipment and procedures

### 2.3.1 Flexure strength: Instron 5967 with environmental chamber (insitu testing)

Instron 5967 is a servo-control and signal conditioning electronics instrument for material testing applications. It has fine position adjustment thumbwheel with 0.004mm resolution for precise positioning of crosshead while testing. Specimen protect also applied to the specimen outside a set threshold-protecting to overcome unwanted damage. Bluehill 3 software helps to get the data from the attached computer.

The test coupons of different sizes were cut from the laminates for physical and mechanical characterization. ILSS testing were conducted on an Instron 5967 test apparatus using three-point bend jig according to ASTM: D 2344-13 shown in Fig 2

The dimension of the ILSS specimen was 60× 40×4 $mm^3$. Thirty six specimens were tested from two conditions panels with six loading speed (3 specimens each) for each test. The results were then compared with the data obtained from unconditioned specimens. The flexural methods are applicable to polymeric composite materials. A testing machine with controllable crosshead speed with environmental chamber is used in conjunction with a loading fixture. The shear stress induced in a beam subjected to a bending load is directly proportional to the magnitude of the applied load and independent of the span length. Thus the support span of the short beam shear specimen is kept short so that an inter-laminar shear failure occurs before a bending failure.

This test method is defined, which specifies a span length to specimen thickness ratio of 5 for low stiffness composites and 4 for higher stiffness composite. The loading arrangement is shown in a span length of 40 mm. The tests were performed with 6 increasing crosshead speed ranging from 0.1, 1, 100, 300, 800 and1000 mm/min at different temperatures. For each point of test 3 specimens were tested and the average value was taken. ILSS was calculated by equation (1)

$$ILSS = 0.75 * P/bt \qquad (1)$$

Where, P = maximum load, b = width of specimen, t = thickness of specimen

### 2.3.2 Scanning electron microscope (SEM)

The scanning electron microscope (SEM) has been a well accepted tool for many years in the examination of fracture surfaces. The prominent imaging advantages are the great depth of field and high spatial resolution and the image is relatively easy to interpret visually. To study the different failure mechanisms of the tested samples micrographs of the failure samples was carried out using a JEOL-JSM 6480 LV SEM. The samples were loaded onto the sample holder and placed inside the SEM, adjusting the working distance and hence the spot size the chamber was closed and vacuum was applied.

## 3. Results and discussions

### 3.1 Mechanical properties (3-point bend test)

Fig 3(a) represents the short beam shear test curves based on different loading rates. The ILSS vs. crosshead speed curves gives different ILSS values at different speed of loading. It was found that ILSS values depend on working temperature of the material. Here we considered 3 modes of loading phase while correlating with interlaminar shear strength, Mode A (1 to 500) mm/min, Mode B (500 to 1000) mm/min and Mode C (0.01 to 1000) mm/min. When the specimen at ambient subjected to 3-point bend test the ILSS values increases with increasing with loading rate in each loading phase. In contrast to this, when the specimen treated with +50°C temperature, the strength had decreases in each modes of loading speed but the ILSS value is more than ambient temperature. In Mode A, it decreases by 16.14% (Max.), Mode B 9.75% (Max) and Mode C 6.05%(Max) was observed. This may be due to spreading of process zone (PZ) in large depth (softer resin) in epoxy resin matrix resin which leads to increase in degree of fiber bridging. In this condition, fiber/matrix debonding is critical and also exhibit increased deformation which shown in Fig 3 at low and high loading rates. When sample is loaded there is widespread microscopic damage arises throughout the laminate. Large damage can be sustained to a critical value at which failure occurs by the propagation of cracks. These cracks are much more complex in nature than cracks in homogeneous materials. The failure of a composite involves the fractures of the load-bearing fibers and the matrix as well as a complex combination of cracks propagated along the interfaces [15]. Thus ILSS values decreases in every mode of loading.

While at -50°C temperature, the ILSS values increases by 51.09% (max.) in Mode A, in Mode B it decreases by 18.12% (max) again in Mode C it decreases by 3.72% (max). There is a noticeable decrease of ILSS values in higher cross head speed. In contrast to above, at ambient temperature ILSS value is increased by 27.07% (Max.) in Mode A, in Mode B it decreases by 16.42% again in Mode C it is increased by 8.37%. The reason behind the reduced values of ILSS at low temperature after Mode A loading duration (medium cross head speed), loading time is less than the mechanical relaxation time i.e. time is not sufficient for plastic deformation to take place at the crack tip thus matrix becomes brittle and fracture strain decreases. Thus fiber/matrix bond strength is well tailored and it is unusual for bare fibers to be exposed. From fractographic prospective of sample reveals that failures manifest from matrix microcracking to fiber fracture and fibers pull-out which shown in Fig 3(b). However, increased high cross head speed (Mode B) the fracture stress and strain increases and matrix become more tough and ductile. Here fracture starts to preferentially occur at fiber/matrix interface region. Heat capacity at matrix is less, thus an appreciable temperature rise occurred in front of the crack tip even small value of inelastic energy at low temperature. It is anticipated as by existing of adiabatic heating i.e. unstable crack propagation [16]. In this state, rate of heat generation is lower than for its removal (thermally active zone to passive zone). Thus ILSS value decreases at higher cross head speed which shown in Fig 3(b). This may also be hypothesized of preexisting microcracks, notches, debris i.e. with a stable element (low-energy crack growth). This fracture may be arrested and then reinitiated, or may lead to initiation and growth of secondary mode of failure.

## 3.2 SEM fractographic analysis

The failed specimen surfaces were viewed under SEM to study how changing failure modes affect the ILSS value as loading rate increased. Identification of the cause of fracture through fractography has become a standard investigation technique. The large depth of focus and the fact that the actual surface can be examined make the SEM, an important tool for research and for failure analysis. Microscopic material failure is defined in terms of crack propagation and initiation. Such methodologies are useful for gaining insight in the cracking of specimens and sample structures under well-defined global load distributions [17]. Microscopic failure considers the initiation and propagation of a crack. Fig (4, 5, 6, and 7) shows impact fracture surfaces of the prepreg composites from which progressive changes in the locus of failure can be clearly identified as a result of change in test temperature for a given loading condition. Fig (4) shows uniform propagation of matrix microcracking at -50ºC temperature. The locus of failure is not exactly at the interface region but is significantly above it. However, there is some fiber imprint are visible on the surface. This is because of development of internal residual tensile stresses in the matrix resin at low temperature. As matrix behaves as brittle manner at low temperature the molecular motion will be abysmal; molecular motion will be small and re-orientation modes will be relatively simple [18]. Similarly the deportment of interface region of glass/epoxy at low temperature doesn’t undergo any significant change which tends to high adhesion bond strength. Thus very tiny interfacial failure observed at low loading speed (Mode

A). Here cohesive failure of the bulk matrix plays the dominant role. At higher crosshead speed 800mm/min brittle failure of fiber observed. Brittle fibers have low fracture strain and low energy absorbing capability [19]. Fibers can fracture early at the weak cross section point, which is not necessarily the direction of crack propagation.

At low temperature (-50°C temperature) when specimen subjected to 1000 mm/min cleavage marking on the matrix surface and fiber pull-out observed. As the applied loading rate increases 1000 mm/min the fracture proceeds, the broken fibers will likely pullout from the matrix which observed in SEM micrograph. When the matrix behaves as brittle, the energy of fracture is fairly low and there is little step marking formed during failure, which is refereed to cleavage marking [20]. The characteristic feature of cleavage fracture is flat facets which generally are about the increasing size of 9.22μm to 20.6μm. Cleavage fracture represents brittle fracture occurring in the matrix region, which shown in Fig 5.These are indications of the absorption of energy by local deformation. The direction of the river pattern represents the direction of crack propagation. Quasi-cleavage fracture is related but distinct to cleavage fracture. It is observed chiefly low temperature. The term quasi cleavage is used because the facets on the fracture surface are not true cleavage planes. Quasi-cleavage fractures often exhibit dimples and tear ridges around the periphery of the facets [21].

In contrast to the foregoing phenomena, fracture surface of ambient samples subjected to 1 mm/min, 800 mm/min and 1000 mm/min was shown in Fig (6) display fiber pull-out, steps and welts, tiny nodules on the matrix surface, matrix cracking, and progressive interfacial debonding. In addition, there are significant changes in the morphology of the matrix fracture. Besides that, other important matrix fracture is scraps and ribbons. When multiple microcracks are formed and begin to propagate in several planes, and they subsequently converge onto one plane. Ambient temperature signifies the beginning of the steps and welts as well as macromatrix cracking. Both the things are parallel to the fracture propagation direction. This failure mode was observed at low loading speed. The steps are identified by toughened matrix or smooth region, while welts are identified by ribbons. If the crack planes overlap before they coalesce to form scarp [22]. When the loading rate increases 800 mm/min density of crack plane increases and macomatrix cracking plays the dominant role.

Above discussions associated with matrix failure modes at high as well as low temperature, which are responsible for integrity and durability of polymer matrix composites.

Fig (7) shows fiber/matrix debonding as well as cohesive failure of matrix at low and high loading rate respectively at +50º C temperatures. Damage initiation for cohesive failure observed at the matrix resin where severity of stress state occurred.

## 4. Conclusions

In this investigation, an experimental study was carried out on the failure of GFRP composites in thermal and cryogenic environment. Parameters such as temperature and loading rate were considered to assess and evaluate mechanical behavior. The present study may possibly reveal the following conclusions:

1) The percentage of ILSS value decreases during above-ambient temperature testing in every mode of loading rate ranges because of thermal conditioning effect which leads to spreading of process zone in the matrix resin which impart high fiber/matrix debonding.
2) In contrast to, at sub-ambient temperature the percentage of ILSS value increases in Mode A, phase and decreases in Mode B and C due to unstable crack

propagation (adiabatic heating) at the crack tip. This yield matrix microcracking to fiber fracture and then fiber pull-out.

3) In comparison when a virginal sample is tested the percentage of ILSS value increases in each mode of loading speed which may be due to preexisting stable element (low-energy crack) which divulge steps and welts along with matrix cracking in the matrix region.
4) The load carrying capacity and the strain at yield increases with increasing the loading rate in each environmental conditioning treatment.

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## Figure captions

Fig 1: Curing cycle for glass/epoxy composite followed within autoclave.

Fig 2: Instron 5967 with environmental chamber used during the insitu testing of sample.

Fig 3(a) Loading rate with ILSS of GFRP samples at +50°C and +28°C temperatures.

Fig 3(b): Loading speed vs ILSS curves of glass/epoxy composite at -50°C and +28°C temperatures.

Fig 4: Matrix micro cracking and brittle fracture of fibre at-50°C temperature at 1 mm/min and 800 mm/min.

Fig 5: Cleavage marking and fibre/matrix debonding at -50°C temperature at 1000 mm/min.

Fig 6: Scanning electron micrograph at ambient 1 mm/min and 800 mm/min shows steps and welts as well as matrix cracking respectively.

Fig 7: Thermal conditioning sample at 1 mm/min and 800 mm/min showing fibre/matrix debonding and macromatrix cracking.

## Figures with captions

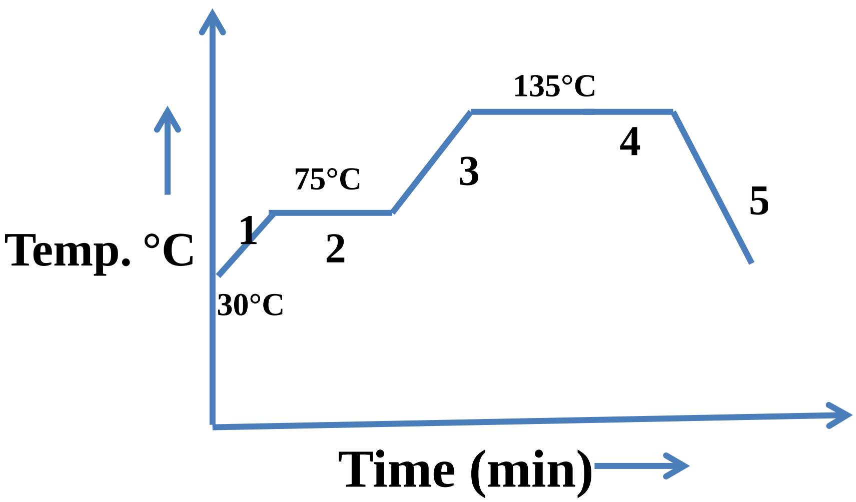


**Fig 1**: Curing cycle for glass/epoxy composite followed within autoclave.

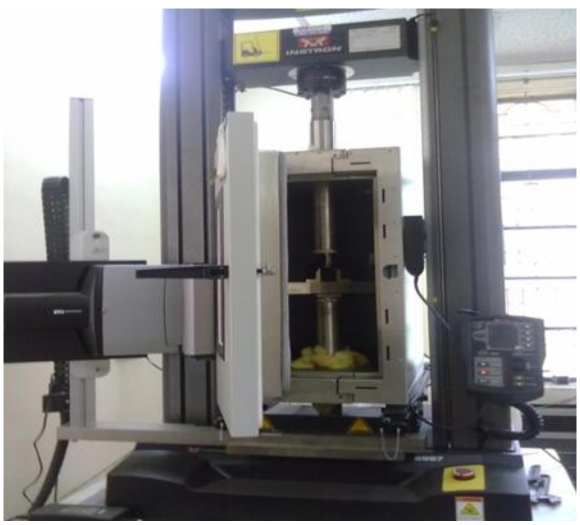


**Fig 2**: Instron 5967 with environmental chamber used during the insitu testing of sample.

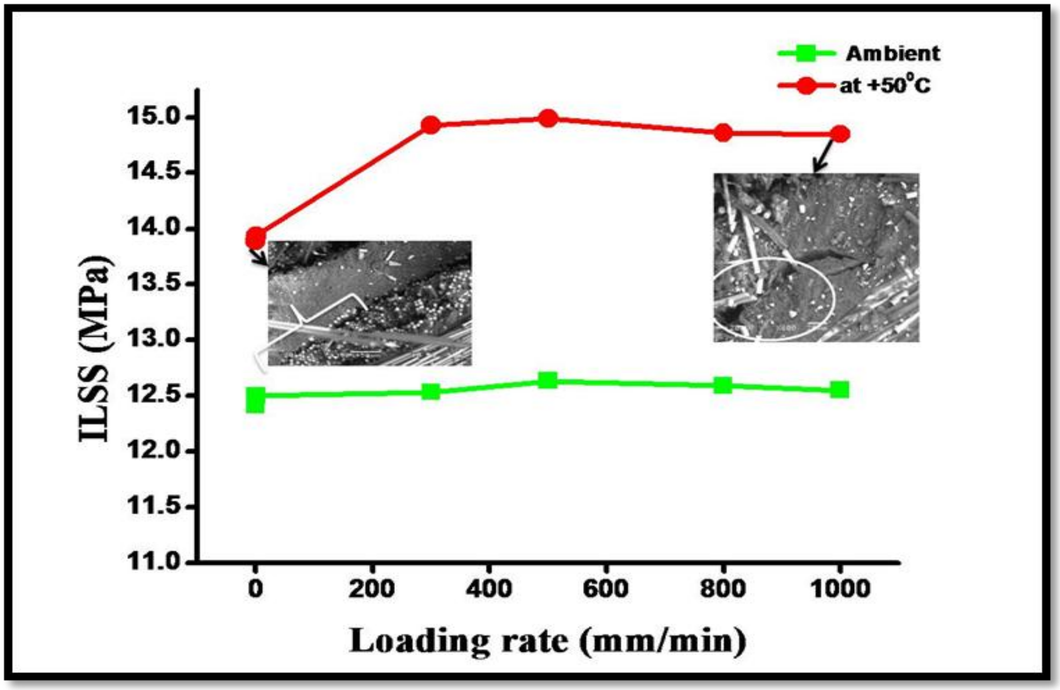


**Fig 3(a)** Loading rate with ILSS of GFRP samples at +50°C and +28°C temperatures.

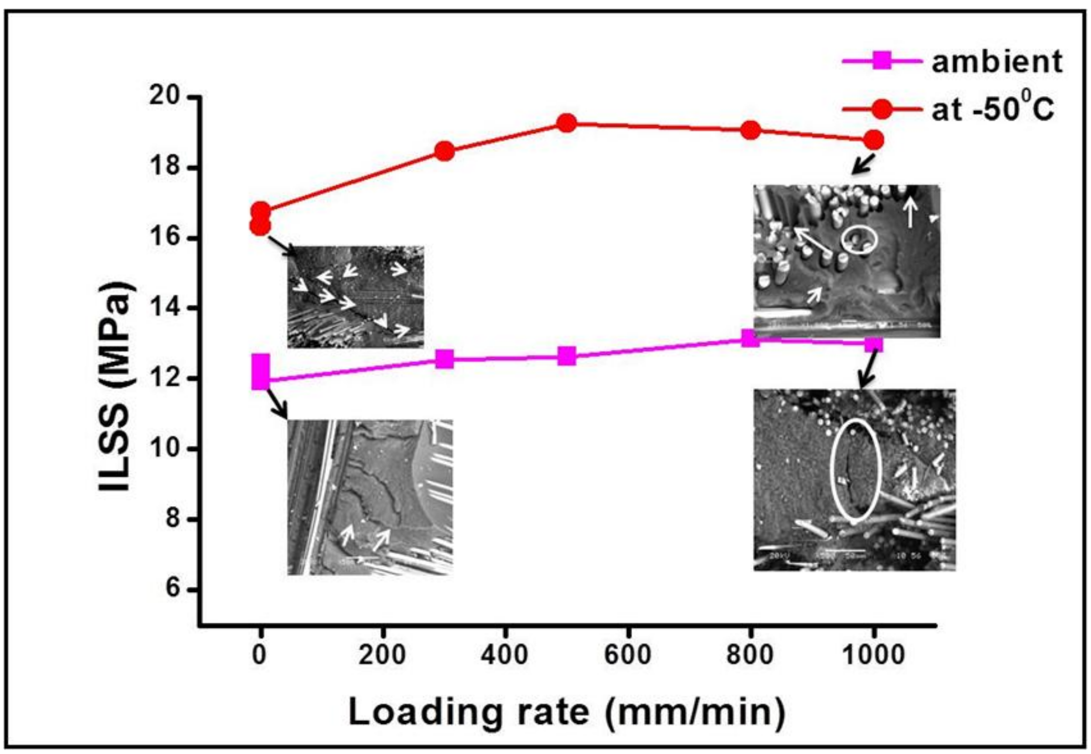


**Fig 3(b):** Loading speed vs ILSS curves of glass/epoxy composite at -50°C and +28°C temperatures.

-50°C temperature  degree 1 mm/min

800 mm/min

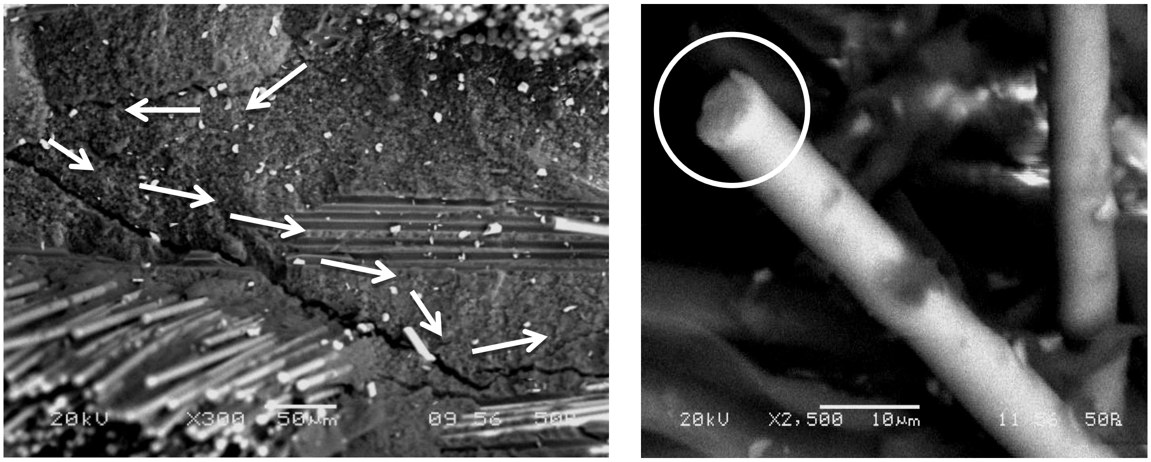


**Fig 4**: Matrix micro cracking and brittle fracture of fiber at-50°C temperature at 1 mm/min and 800 mm/min.

-50°C temperature 1000 mm/min

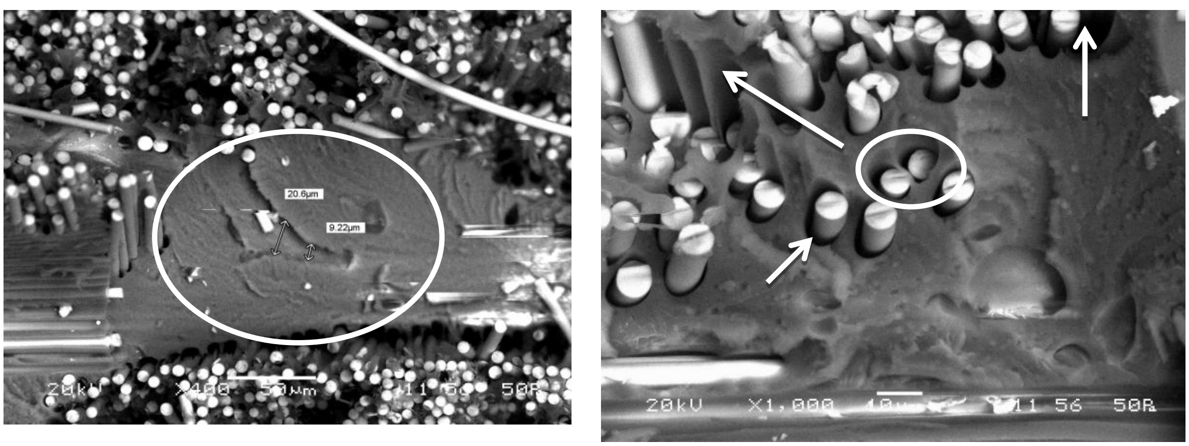


**Fig 5**: Cleavage marking and fibre/matrix debonding at -50°C at 1000 mm/min.

Ambient 1 mm/min

Ambient 800 mm/min

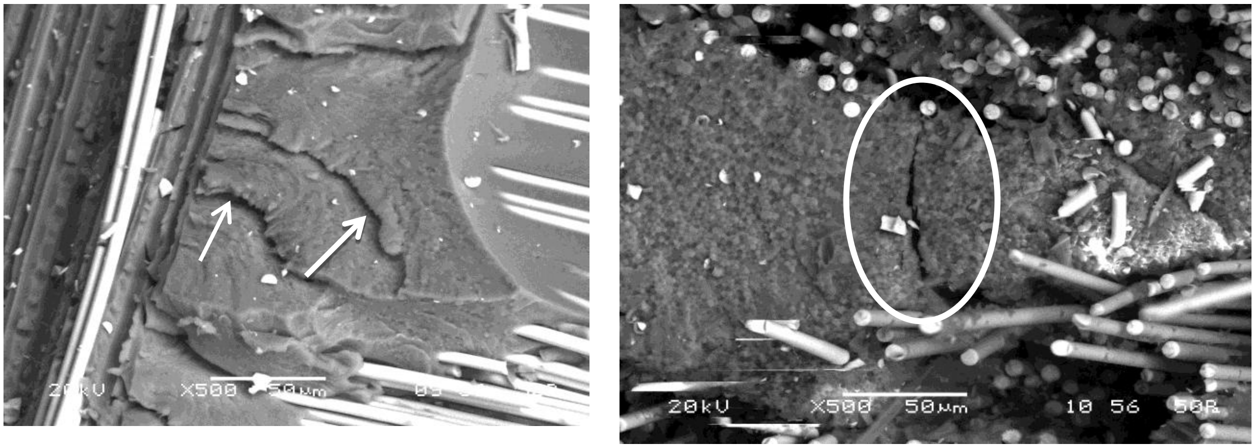


**Fig 6**: Scanning electron micrograph at ambient 1 mm/min and 800 mm/min shows steps and welts as well as matrix cracking respectively.

+50°C temperature 1mm/min

800mm/min

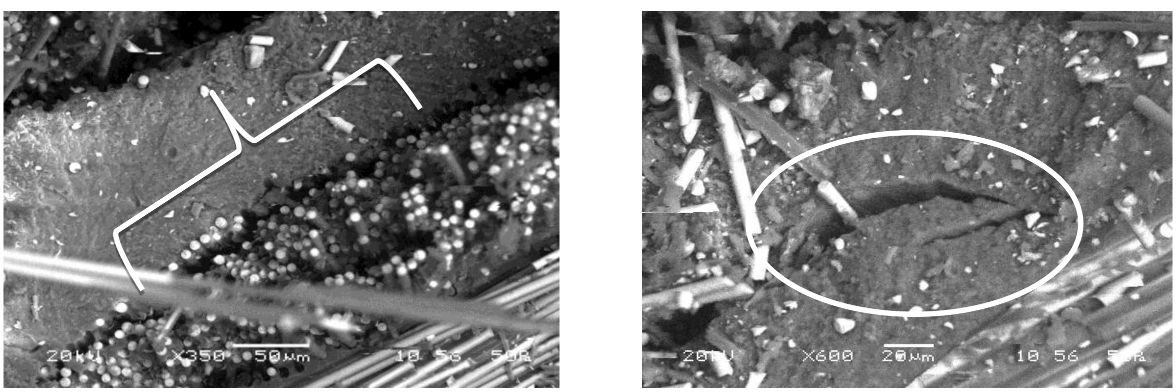


**Fig 7**: Thermal conditioning sample at 1 mm/min and 800 mm/min showing fibre/matrix debonding and macromatrix cracking.