Experimental study on mechanical behaviour and microstructural assessment of Kevlar/epoxy composites at liquid nitrogen temperature

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Abstract

This present investigation has emphasised on understanding the mechanical behavior and progressive failure modes of Kevlar/epoxy reinforced laminated composites subjected to 3-point bend test at liquid nitrogen temperature (77K temperature). The tests were conducted on the woven fabric laminates with different loading speeds and characterized by scanning electron microscope (SEM). The effect of liquid nitrogen temperature on interlaminar shear strength (ILSS) of Kevlar/epoxy composites with various loading speeds was observed. Results indicate values of ILSS were increased at higher cross head speeds as compared to ambient temperature. Fibrillation progression and matrix failure (riverline marking) was observed by SEM. A change in cross head speed may result in variation of failure modes.

Key words: Kevlar/epoxy composites, ILSS, Liquid nitrogen temperature, SEM

Introduction

Polymer composite materials are an important branch of structural and engineering materials with wide applications in a range from aircraft components, automobile and cryogenic equipment [1-3]. Kevlar fiber reinforced polymer (KFRP) woven laminates are used mainly in armor, boat hulls, and various forms of ballistic armor and many cryogenic environments. With the ever-increasing demand for KFRP woven laminates as structural members, considerable effort has been focused in recent years on their mechanical behavior and microstructural characterization at low temperatures. Kevlar 49 has been specifically engineered for polymer reinforcement and is intended more for the aerospace industries, primarily to achieve significant weight reduction without compromising performance [4, 5]. In addition it has excellent toughness, high impact strength, stress rupture life, fatigue strength, damping characteristics and is easy to process [6]. The degree of environmental degradation that occurs in a fiber reinforced polymer composite structure is linked directly with these components as fiber, matrix and fiber/matrix interface region. The interface is defined as a region which is manifested as a result of bonding and reactions between the fiber and the matrix. This region is the site of synergy in composite materials and its influence to the overall mechanical properties is significant [7-9]. From a macroscopic perspective, poor fiber/matrix bond strength relates to brush-like failure [10, 11]. The interfacial bond strength between Kevlar/epoxy composites is weaker than what is experienced in carbon/epoxy composites [12-15]. Delamination, matrix cracking and other failure modes are frequently observed when these materials are subjected to low temperature. Hence, these failure modes have a synergetic effect on the mechanical properties of composite materials. The present investigation aims to study the mechanical behavior and microstructural characterization of Kevlar/epoxy composites at liquid Nitrogen temperature. Kevlar fiber has been specifically engineered for polymer reinforcement and is intended more for space craft where low temperature systems are present. These materials are support materials for endurity the large accelerations and severe vibrations during launch. The support system must also provide excellent thermal isolation to maximize the performance of the cryogenic system [16]. Kevlar fiber shows essentially no embrittlement or degradation at temperature as low as -196°C (77K).The high radial expansion coefficient of Kevlar fiber causes weakening of interfacial adhesion under the influence of temperature gradient. However, the weak interface may readily allow crack deflection along the interface and improves the energy-absorbing capacity [5]. Impact energy level and temperature were found to have significant effects on the impact behavior of fiberglass and combinations of fiberglass with Kevlar [17]. However, the fracture behavior and mechanism of the laminate composites at low and high temperature levels are complicated when compared with those of the composite at room temperature [18-21]. In the present investigation, an attempt has been made to study the mechanical behavior of Kevlar epoxy composites at liquid nitrogen temperature and correlate the failure modes which are responsible for the degradation of composite materials.

Materials and Experimental Methods

2. Experimental

2.1 Materials and Methods

An epoxy interpenetrating network system was developed by using resin Laptox L-12, which is an unmodified liquid resin based on Bisphenol A, along with Hardener K-6 (aliphatic primary amine). Kevlar fibers with 60 weight percentage were used for Kevlar/epoxy composites fabrication. The calculated percentage of epoxy and hardener is thoroughly mixed at room temperature.

2.2 Fabrication of composite laminates

The laminate is prepared by hand lay-up techniques using Kevlar woven fabric, pieces of size 20*20 cm. The preweight mixture of epoxy and curing agent (hardener) is applied over a fabric sheet using brush. Rolling was carried out with uniform pressure in order to remove the air pockets. Same procedure was carried out for 8 layers. The material is kept one over the other in the releasing agent sprayed mould. They were cured for 24 hr. at room temperature. The laminates were cut into desired dimensions as per ASTM standard 2344-10.

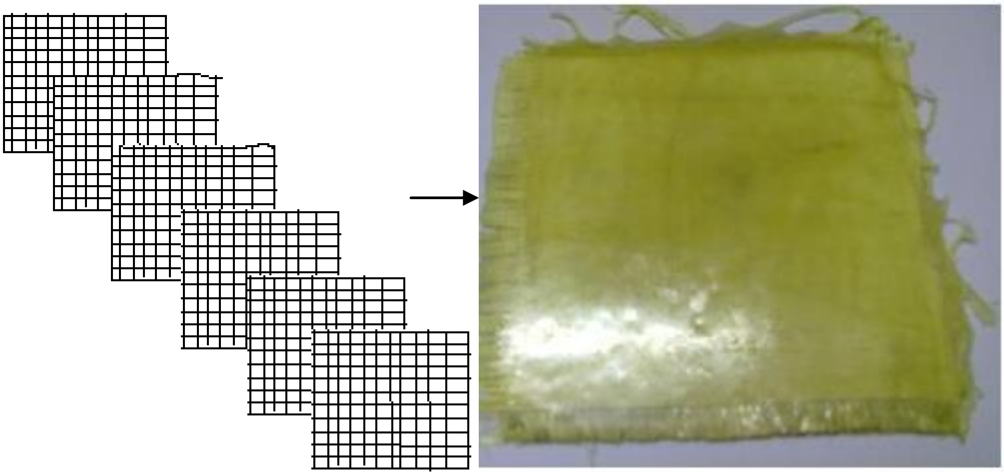

Fig 1: Kevlar/epoxy laminates after fabrication

2.3 Experimental methods

Flexural test (short-beam shear test)

The flexural methods are applicable to polymeric composite materials. A testing machine with controllable crosshead speed 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 does. This test method is defined by ASTM D 2344-10, which specifies a span length to specimen thickness ratio of five for low stiffness composites and four for higher stiffness composite. The short beam shear (SBS) tests are performed on the composite samples at the conditioned temperature and room temperature to evaluate the value of inter-laminar shear strength (ILSS). The SBS test is conducted as per the ASTM standard D2344-10 with an Instron 1195 testing machine. The loading arrangement is shown in a span length of 40 mm. The tests were performed with five increasing crosshead speed ranging from 1, 10,100, 200 and 500 mm/min. For each point of testing 4 to 5 specimen were tested and the average value was taken. The inter-laminar shear strength was:

$$ILSS = 0.75 * P/bt$$

Where P is the maximum load, b the specimen width and t the specimen thickness.

Scanning electron microscope (SEM)

The scanning electron microscope (SEM) has been a well-accepted tool for many years in evaluation of fracture surfaces. The prominent imaging advantages are the great depth of field and high spatial resolution, as well as the image is relatively easy to interpret visually. To study the different failure mechanisms of the tested samples micrographs of the failure samples were taken using a JEOL-JSM 6480 LV SEM at 20 Kv. The samples were loaded onto the sample holder and placed inside the SEM, adjusting the working distance and the spot size, the chamber was closed and vacuum was applied.

Experimental Procedure

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 until a constant weight was reached. One batch of sample was treated with LN whereas another batch was tested in ambient temperature. The specimens were dipped into the LN_2 for 15 min and then immediately underwent a 3-point bend test.

3. Results and discussions

3.1 Mechanical properties and loading rate

Fig 1 shows the effect of liquid nitrogen (LN_2) temperature on ILSS value of Kevlar/epoxy composites at different loading speed, (1, 10,100,200,500 mm/min). The research showed that the increase of laminate strength at low temperatures depends mainly on the fiber/matrix interface strength. A slight improvement in shear strength with each loading speed was observed at low temperature. A large percentage of the interfacial area was strongly affected at low temperature. Kevlar fiber show high strength under axial tension. The specimens were first tested at ambient temperature at different loading rates. Thereafter, liquid nitrogen treatment was followed with the same loading rates. There is a slow drop in ILSS value at 10mm/min for LN_2 temperature which may be due to less adhesion level at this loading speed. The variation of ILSS in this case is the net result of interfacial interaction shown in SEM. The low temperature is likely to change the chemistry at the fiber/matrix interface region. At low temperature the polymeric matrix becomes stiffer and stronger but also less ductile. These phenomena may result to better adhesion at the interface.

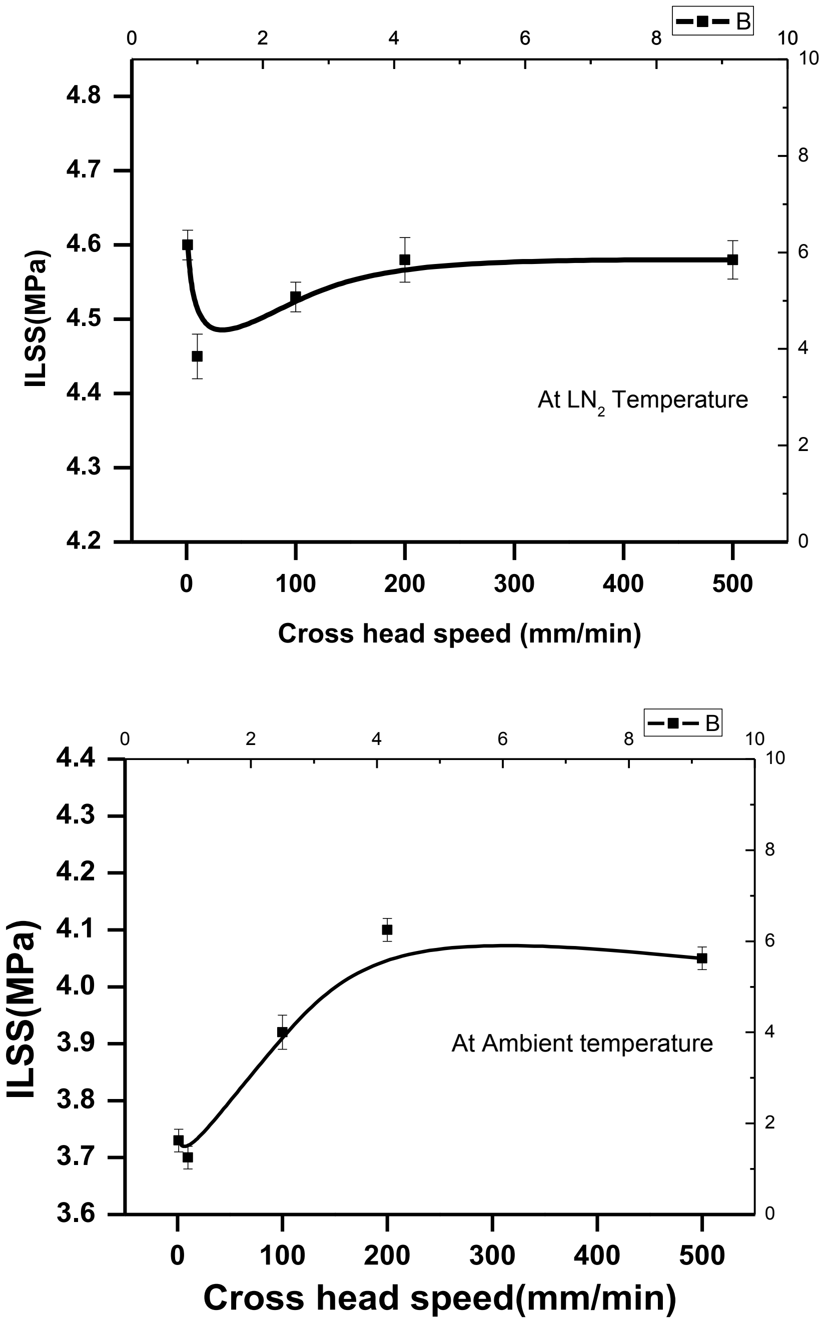

Fig 2: a) Variation of ILSS with cross head speed at LN_2 temperature b) Variation of ILSS with cross head speed at ambient temperature

The improved interfacial adhesion is likely to be counteracted by the adverse contraction of aramid molecules. This good adhesion of interface at low temperature may be due to the reduction of radial or transverse contraction of Van der Waals or hydrogen-bonded aramid molecules compared to the isotropic polymer matrix. Because of this good adhesion, the ILSS value increases as compared to ambient tested samples.

Fig 2 shows the variation of the ILSS for Kevlar/epoxy composites with crosshead rate at ambient temperature. At ambient temperature the shear strength value was less as compared to the treated one. This may be attributed to failure of Kevlar fiber in a fibrillation manner. FRP composites generally contain microvoids, microcracks with statistically distributed sizes. A weaker interfacial bond may result in a lower flexural strength of the laminate. But with increasing loading speed it shows higher shear strength for almost all loading speeds. At lower cross head speeds the polymer gets more time for relaxation due to which, there is less gross plastic deformation, thus resulting in enhancement of ILSS values [6].The failure in tension brings into play the covalent bonding along the axis, which ultimately leads to chain scission and /or chain sliding or a combination thereof. However, they have poor properties under axial compression, torsion and in the transverse direction. Kevlar fibers have the highest tensile strength-to-weight ratio of any commercially available reinforcement fibers [22]. This fiber excels in composite toughness or damage tolerance applications. The lower the stress concentration factor, the greater the resistance of the laminate to crack propagation. The structural integrity losses at higher cross head speed increase the cracks density. Accordingly, the transverse, shear strength and stiffness are very low. The transverse stiffness of this fiber is similar to that of an isotropic polymer at low temperature. Table 1 and Table 2 show the test results of the Kevlar/epoxy composites at ambient and LN_2 temperatures. Damage tolerance includes both the ability to resist penetration during impact and the retention of properties after a given level of impact. Toughness is related to the energy absorbing ability of the material which is shown in Table 1 and Table 2. Along with good impact resistance and damage tolerance, fiber has high fracture toughness or resistance to crack propagation.

Table 1 ILSS values and modulus of ambient Kevlar/epoxy sample.

Specimen Number	Cross head speed (mm/min)	Strain at peak (mm/mm)	Modulus(MPa)	Interlaminar shear strength (ILSS) at ambient (MPa)
1	1	0.0688	4528.1	3.726
2	10	0.0847	5084.3	3.70
3	100	0.0344	23020.0	3.98
4	200	0.0584	12850.1	4.32
5	500	0.2303	4794.6	4.039

Table 2 ILSS values and modulus of Liquid nitrogen treated samples.

Specimen Number	Cross head speed (mm/min)	Strain at peak (mm/mm)	Modulus(MPa)	Interlaminar shear strength (ILSS) after LN treatment (MPa)
1	1	0.0473	5499.1	4.59
2	10	0.0419	6646.2	4.12
3	100	0.0410	6112.5	4.10
4	200	0.0363	5561.0	4.19
5	500	0.1215	4103	4.172

The rigid linear molecular chains are highly oriented in the fiber axis direction, with the chains held together in the transverse direction by hydrogen bonds. The strong covalent bonds in the fiber axis direction provide high longitudinal strength, whereas the weak hydrogen bonds in the transverse direction result in low transverse strength.

3.2 Fractographic results

Considering the influence of low temperature on the fracture micromechanisms in composites, the high radial expansion coefficient of fibers causes residual tensile stresses in the matrix. Regarding the fracture morphology of interlaminar (intralaminar) fracture at very low temperature, resin embrittlement dominates and thus increases the ILSS value as compared to ambient temperature. Fig.3 represents very thin riverline marking, toughened matrix and potholes after LN_2 treatment of the samples. Local failure may initiate along a line defect, such as fiber and spread into the surrounding matrix. This phenomenaon leads to important fractographic features as riverlines**.** This is the most valuable features for crack growth directions which are observed sharply on SEM images. The convergences of pairs of planes from the tributaries of the rivers ultimately converge into one crack; therefore, the direction of riverlines markings is the direction of crack propagation of the matrix plane [23]. One of the most important phenomena of matrix fracture is the process by which multiple fractures initiate along the crack front, begin to propagate on several slightly different planes, and the subsequently converge onto one plane. The morphology of the matrix rollers (Fig. 5) is strongly dependent on the matrix type, interface strength as observed in SEM. As the matrix toughness increases the rollers become more elongated, exhibiting increasing plasticity. This may be the reason for the increase of the ILSS value in each loading speed. The stressing conditions and the environments that a composite is subjected to play a key role in determining its failure process [24-27].

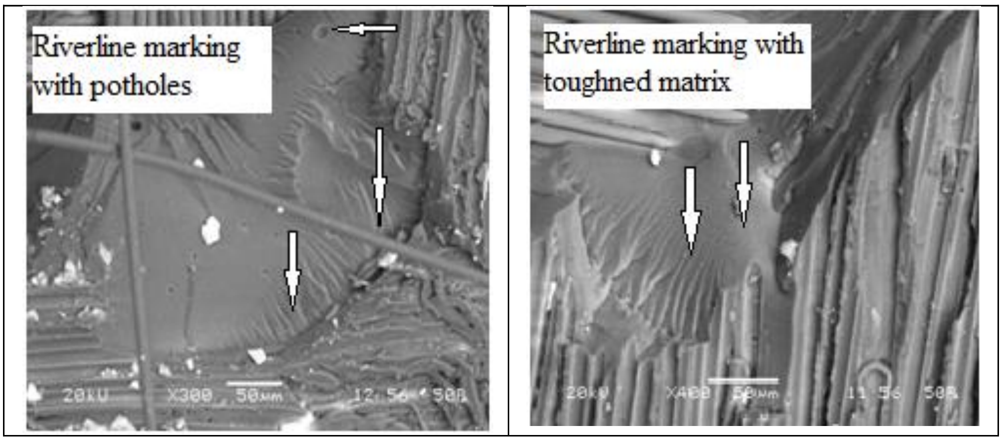

Fig 3: SEM micrographs of LN_2 samples shows riverline marking with potholes and toughened matrix

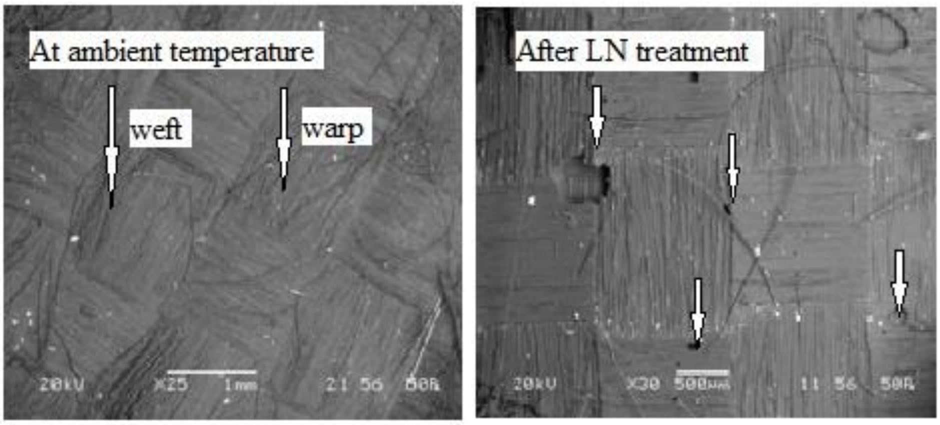

Fig 4: SEM micrographs shows a comparison between at ambient and after liquid nitrogen treatment.

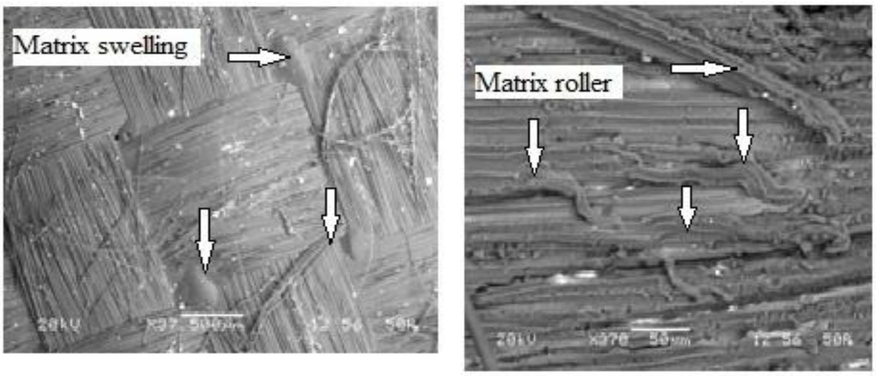

Fig 5: SEM micrograph of LN_2 samples shows matrix swelling and matrix roller

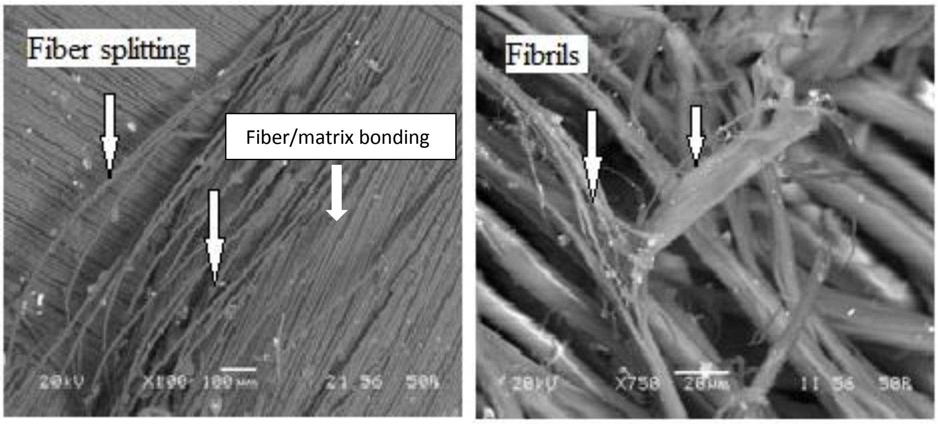

Fig 6: SEM micrograph shows fiber splitting and fibrils region at ambient temperature

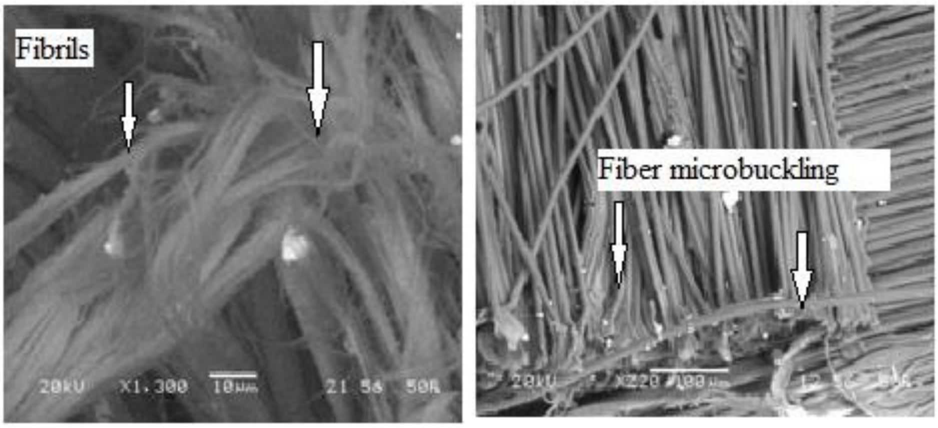

Fig 7: SEM micrographs of LN_2 samples shows fibrils and fiber microbuckling

Fig. 4 represents the warp and weft condition of the woven fabric Kevlar/epoxy composites at ambient temperature and after LN_2 treatment. Highly oriented aramid fiber fail in a fibrillar fashion. Fibrillar fracture observed in Figs. 6, 7 signifies that the fracture surface is not transverse to the axis but runs along a number of weak planes parallel to the fiber axis, its axial tensile modulus increases but the shear modulus decreases. Examination of the fiber ends of a compression fracture shows further evidence of the microbuckling failure mechanisms shown in Fig. 7. The fractographic features shown on the fiber ends exhibit morphology particular to laminate compression failure. Across each individual fiber end is a line, which represents the neutral axis of the fiber as it undergoes bending. Microbuckling may occur on several planes giving rise to a series of steps on the fracture surface, each step being a multiple of half the buckling wavelength. During failure involving compressive stresses, fibrillation occurs, which results in a large degree of new surface area. This fibrillation process results in high-energy absorption during the process of failure.

Conclusions

This experimental study investigated the loading rate sensitivity of woven fabric Kevlar/epoxy laminated composites at ambient and LN_2 temperature conditions. The ILSS with loading rate was plotted for both cases, at increasing loading rate to ascertain the relationship between ILSS to loading rate. The failure specimen surfaces were also examined under SEM to show how the changing failure modes affect the ILSS value as loading rate is increased. The significant findings from the present investigation are as follows:

The ILSS value of Kevlar/epoxy laminated composites at room temperature increases with increasing loading speed, whereas at LN_2 temperature no significant change in ILSS was observed. The interlaminar shear strength value at LN_2 temperature was increased as compared to ambient temperature at each loading rate due to good interfacial bond between fiber and matrix observed in SEM. By considering damage transition of laminate composites, it was observed that at LN_2 temperature toughened matrix, very thin riverline marking and matrix roller are the dominant failure modes. In contrast to this, fiber splitting, fibrils and fiber micro buckling failure modes are the dominant role at ambient temperature.

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