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Effects of crosshead velocity and sub-zero temperature on mechanical behaviour of hygrothermally conditioned glass fibre reinforced epoxy composites

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Abstract

Experimental studies have been carried out to study the effects of thermal and moist environments (hygrothermal) on mechanical behaviour of glass fibre/epoxy composites. The hygrothermal conditioning impairs the fibre/matrix interfacial and/or interphasial chemistry, which plays a predominantly important role in determining the mechanical properties, especially the matrix dominated one, of a polymer composite. The hygrothermallly conditioned laminated composites are further treated at $-6\,^{\circ}C$ temperature to freeze the absorbed moisture in the composites. The inter-laminar shear strength (ILSS) is found to be affected by this conditioning and it is also noticed that the further degradation occurs in the frozen state. The present paper also investigates the effect of variation of loading speeds on the degradation behaviour of polymer composites. A change in loading rate may result in variation of failure modes. The lower value of ILSS at lower crosshead speed may be due to higher ductility or failure strain of the epoxy resin.

Keywords: Composites; Hygrothermal; Strain rate; Residual stresses; Sub-zero temperature; Inter-laminar shear strength

1. Introduction

Composite materials offer a number of potential advantages in the aerospace application, for example high strength to weight ratio, life-cycle cost reduction and greater tolerability over the properties such as strength and fatigue performance of the materials. The mechanical properties of the components of the composite, i.e. the fibre, matrix and interphase or interphasial region, determine the mechanical behaviour of composites on a macroscopic scale [1]. The hygroscopic nature of polymeric systems, however, necessitates a complete understanding of the interaction between structural integrity and hygrothermal environments. Hygrothermal exposure reduces the glass transition temperature of polymers and also causes plasticization, resulting in the reduction of the strength properties, especially dominated by matrix characteristics of polymeric composites [2]. Aircraft during their service life and many other space vehicle components during their fabrication, storage and testing may experience extreme humid and temperature conditions. A fundamental understanding of interfacial properties and characterisation of interfacial adhesion strength may help in evaluating the mechanical behaviour of composite materials during humid ageing. The capabilities and performance of fibre reinforced composites is controlled by the state of fibre/matrix interface [3]. It may be assumed that the interfacial strength is the net result of different contributions to fibre/resin adhesion. These possibly includes chemical bonding, secondary forces of attraction, residual thermal compression forces due to differential shrinkage and also mechanical keying factor at the fibre/matrix interface. Epoxy resin is known to suffer a substantial loss in shear strength on environmental ageing [4,5]. Moisture can penetrate into polymeric composites by diffusive and/or by capillary processes [6–9]. Humid ageing is accepted as one of the prime causes of failure of an organic matrix-based composites. Studies have shown that the presence of moisture may modify the mechanical properties of fibre reinforced polymer composites [10–12]. Hygrothermal exposure may often lead to matrix degradation, fibre/matrix debonding and delamina-

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tion in polymer composites. The lev adhesion between matrix and fibre affects the mechanical behaviour in the off-axis and also parallel to the fibre [13,14]. Environmental exposure results in reduced interfacial stress transmissibility due to matrix plasticization, chemical degradation and mechanical degradation. Matrix plasticization reduces matrix modulus. Chemical degradation is the result of hydrolysis of the bonds at the interface. Mechanical degradation is a function of matrix swelling strain [15,16]. The strain pulls the matrix away from the fibre. The present investigation deals with some aspects of studies with reference to environmental exposure. The variation of loading speeds during testing, results in noticeable change of inter-laminar shear strength (ILSS) values for same amount of absorbed moisture of hygrothermally conditioned specimens. A further study has also been carried out by freezing the absorbed moisture inside the composites at sub-zero temperature. Here also the effect of strain rate is found to have pronounced impact in the ILSS values. The mechanical response of materials is sensitive to the rate at which they are tested. A limited information is available with regard to the effect of strain rate on the response of fibrous composites [17]. The probable failure mechanisms in composite laminates under high loading rate are a complex combination of energy absorption mechanisms. They possibly include delamination by mode 11 shear, matrix cracking by transverse shear and translaminer fracture by fibre rupture and/or by kinking [18]. The material variables, loading and environmental conditions dictate the above fracture processes. One of the most important roles is played by the interface properties in determining damage resistance of composites [19–21]. When the moisture inside the materials freezes, this may lead to a further increase of residual stresses. Thus, the freezing treatment turns out to be more deleterious to ILSS values for the same amount of moisture [22]. It is generally believed that environmental ageing on E-glass/Epon 828 system more often results in irreversible hydrolysis of chemical bonds or breakdown of secondary forces of attraction. Therefore, only partial regeneration of bond strength is achieved by subsequent drying [4].

2. Experimental

2.1. Material

E-glass fibre woven roving (FGP, RP-10) have a density of 0.36 kg/m^2 and epoxy adhesive (Ciba-Geigy LY-556 Araldite, HY-951 hardener) were used to fabricate composite laminates.

2.2. Fabrication of composites

The glass fibre/epoxy composite laminates were fabricated by wet lay-up method; the glass fibre woven cloth of required dimension was laid over a mould and then catalysed epoxy resin was poured, absorbed over the reinforcement. The wet composite was rolled, to distribute the resin and to remove air pockets. The sequence was repeated until the desired thickness was obtained. The layered structure was allowed to harden on cure. It was cured at room temperature for 24 h. After curing, the laminate was cut into the required size for 3-point bend (short-beam shear) test by diamond cutter.

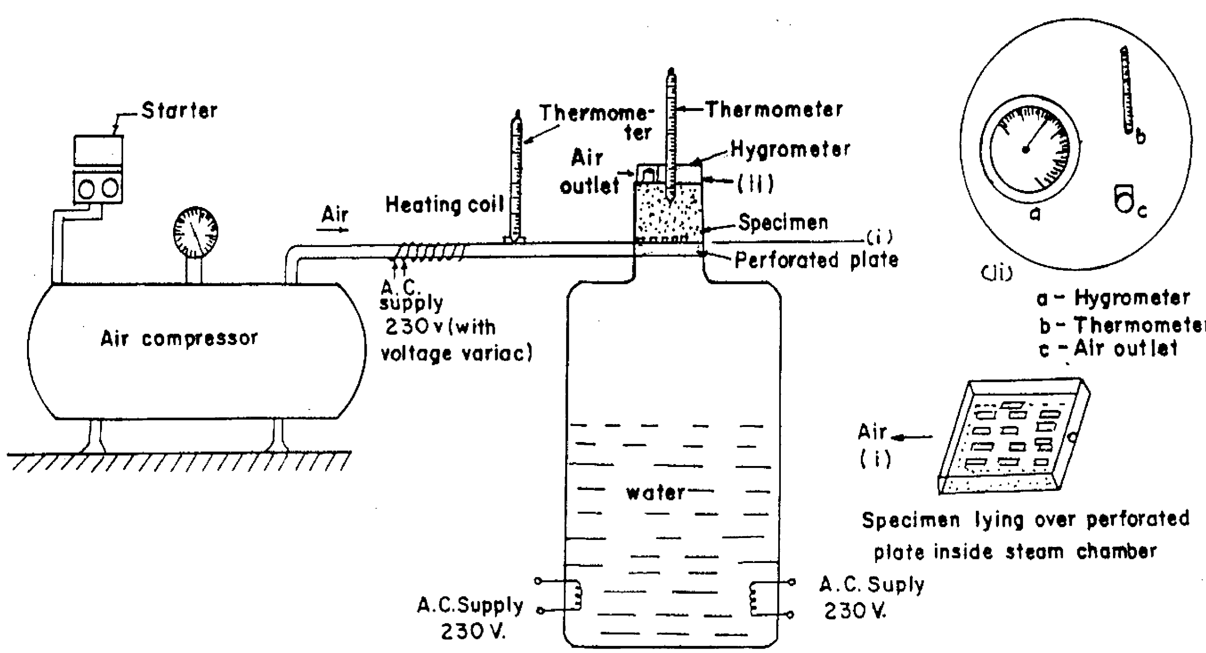

Fig. 1. Hygrothermal chamber.

2.3. Environmental conditioning

2.3.1. Hygrothermal chamber

The hygrothermal chamber was designed and fabricated as shown in Fig. 1. The mild steel water container had inserted within it two immersion heaters to boil the water. The hygrometer for measuring the relative humidity and a thermometer for measuring temperature was fixed in the lid. The air from an air compressor was allowed to pass through a tube, which was wrapped with a heating coil. The heating coil was attached to the voltage variac to control the air temperature. There was also a thermometer attached in the tube to monitor the air temperature. The temperature and humidity were controlled by regulating the air temperature. The composite laminates placed on the perforated plate and kept in the neck region of the chamber. The composite laminates were conditioned at temperature of 60 °C temperature and a relative humidity (%RH) of 95% for different lengths of time.

2.3.2. Freezing treatment

This treatment was accomplished by placing the sample inside a deep freezer. The temperature was measured by a thermometer. The hygrothermally conditioned specimens were kept here at a temperature of −6 °C for 24 h.

2.4. 3-Point bend test

The 3-point bend tests at each stage of absorbed moisture level for both types of conditioned (plain moist and frozen moist) specimens were carried out with an Instron tensile testing machine. The tests were performed at a crosshead speed of 1 and 10 mm/min for each level of exposure/conditioning time. The tests were conducted at room temperature. There were five to nine samples were tested at each stage of conditioning. The inter-laminar shear strength was measured as follows.

$$S_{\mathrm{H}} = \frac{0.75 P_{\mathrm{b}}}{bd}$$

where S_{H} is the ILSS, P_{b} the breaking load, b the width of specimen, d is the thickness of specimen.

3. Results and discussion

The ILSS values at 1 mm/min and also at 10 mm/min crosshead speeds are plotted against the exposure time in Fig. 2. Both curves indicate the reduction of shear strength with the increase of exposure time, i.e. with more amount of absorbed moisture. It was reported that the moisture absorption kinetic increases in Fickian and non-Fickian mode with more conditioning time in glass fibre/epoxy composite [23]. The comparative curves show that the value of ILSS is decreased more for the same exposure time at a crosshead speed of 1 mm/min than that of 10 mm/min. It may be assumed that the failure mechanisms are loading rate sensitive phenomenon. The more time available during lower loading speed may cause the absorbed moisture to move through the cracked channel, developed during testing, and thus causing further moisture/matrix interaction. This results in more deterioration and thus lower values of ILSS are observed. Hygrothermally conditioned at 70 °C temperature and 95% RH polymer composites behaved almost in similar manner [22].

The hygrothermally conditioned at 60 °C temperature and 95% RH specimens were treated at −6 °C temperature for 24 h to ensure complete freezing of absorbed moisture. Fig. 3 corresponds to the change of ILSS values with exposure time for the frozen moisture level specimens. The test was conducted at room temperature and for two different crosshead

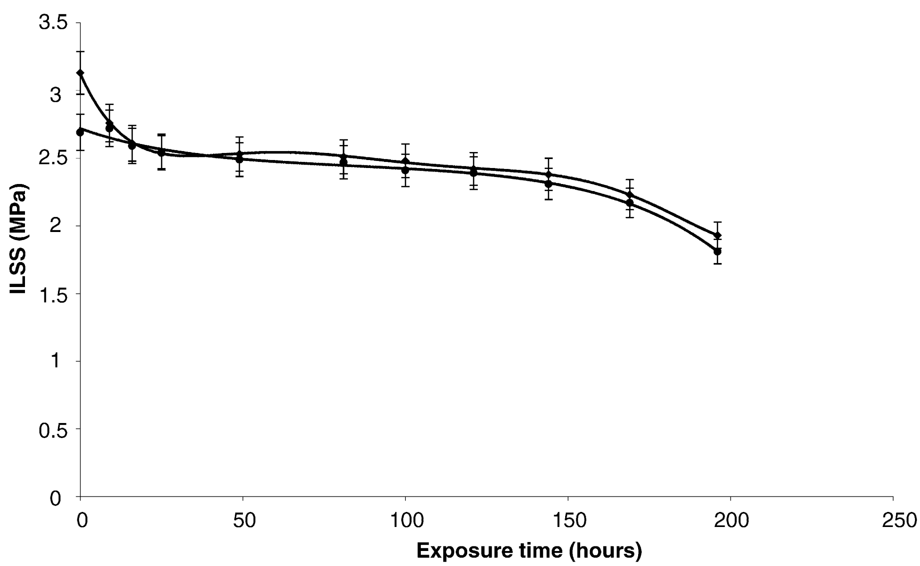

Fig. 2. Variation of ILSS values with exposure time of glass fibre/epoxy composite for different loading speeds ((●) 1 mm/min and (◆) 10 mm/min).

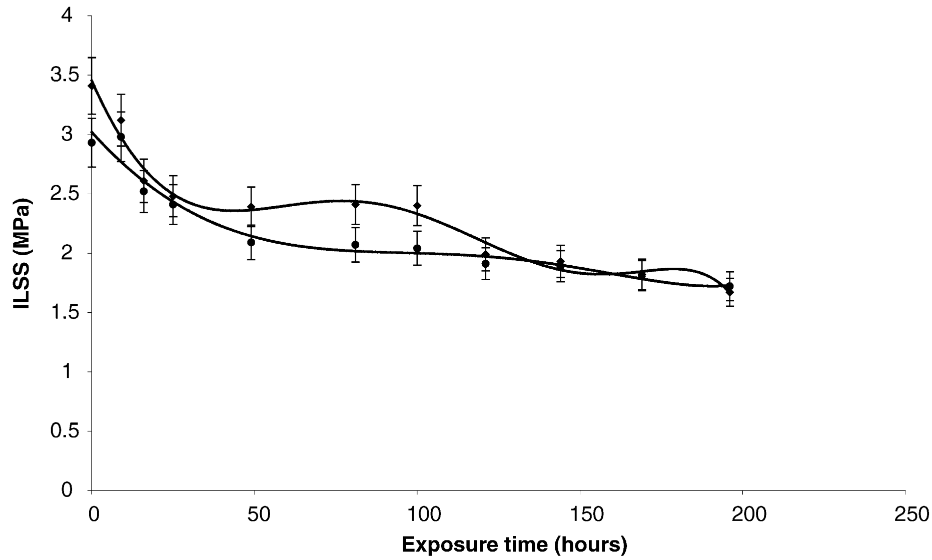

Fig. 3. Variation of ILSS values with exposure time of frozen hygrothermally conditioned glass fibre/epoxy composite for different loading speeds ((●) 1 mm/min and (◆) 10 mm/min).

speeds, i.e. 1 and 10 mm/min. Here, also the overall nature of both the curves show the decline of ILSS values with more exposure time. The ILSS value, here also, at lower loading speeds is reduced more compared to that of higher crosshead velocity for the same time of exposure.

There is a indication that the curves are becoming convergent at a higher level of exposure time in both the conditioned cases (Figs. 2 and 3). This nature of behaviour may be attributed to the fact that at higher levels of absorbed moisture, the degradation of epoxy matrix and interfaces/interphases is colossal. That is why the effects of loading speeds is not noticeable at that conditioned situation.

The ILSS values of both plain moist and frozen moist specimen are plotted against the exposure time at a crosshead speed of 1 mm/min (Fig. 4) and at a crosshead speed of 10 mm/min (Fig. 5). The shear strength value of frozen moisture conditioned specimen is less for the same time of exposure compared to that of only hygrothermally conditioned specimen for the both types of loading speeds, as it is indicated in Figs. 4 and 5. The moisture pick-up leads to the development of hygroscopic residual stresses along with other damaging effects. When the absorbed moisture gets frozen inside the composite specimen, this may lead to volumetric expansion of absorbed moisture, and consequently further swelling stress development in the polymer composite.

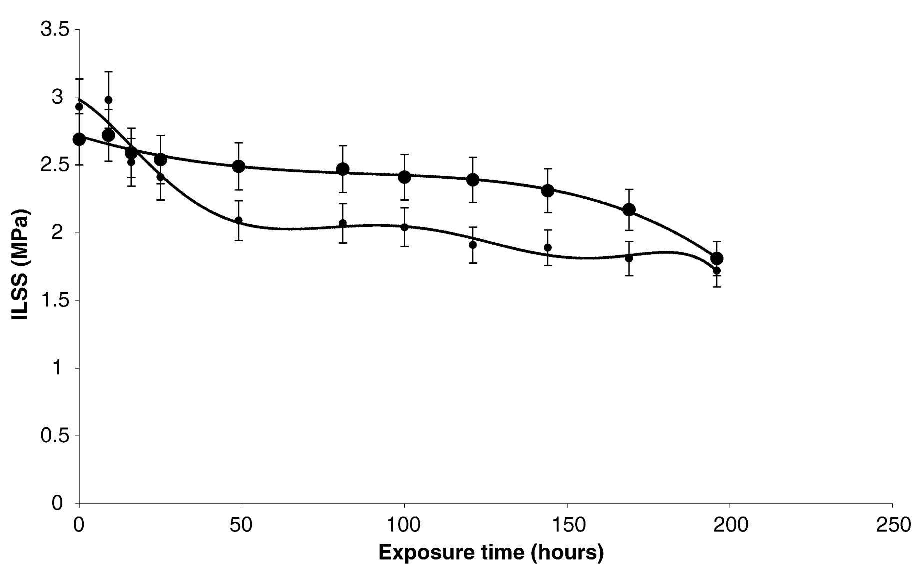

Fig. 4. Comparison of ILSS values of plain moisture and frozen moisture conditioned glass fibre/epoxy composite at 1 mm/min crosshead speed ((•) frozen moisture and (●) plain moisture).

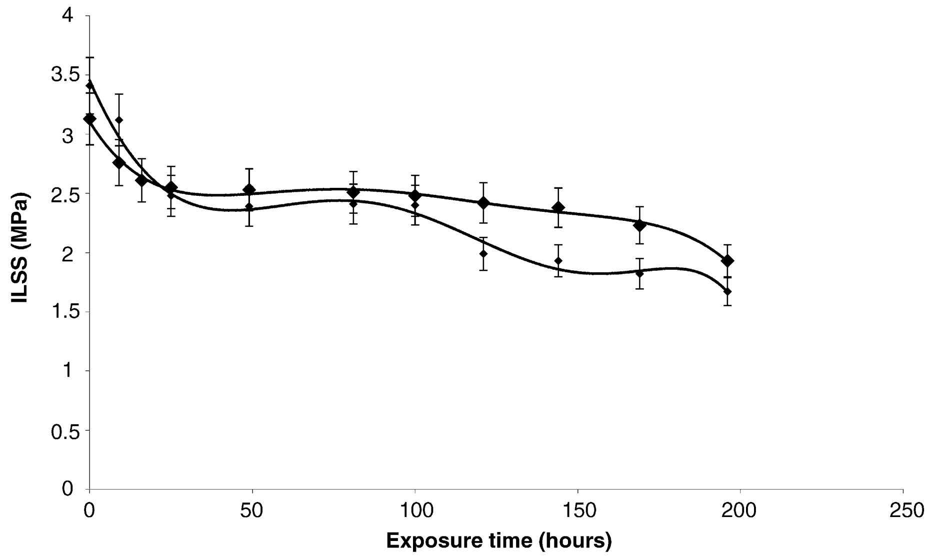

Fig. 5. Comparison of ILSS values of plain moisture and frozen moisture conditioned glass fibre/epoxy composite at 10 mm/min crosshead speed ((◆) frozen moisture and (⬩) plain moisture).

The moisture is absorbed by the resin matrix causing it to swell. This may result in microvoids or cracks in the matrix and resin/fibre interface. The shear failure mechanism is generally explained by a combination of resin swelling, plasticization and lowering of glass transition temperature of epoxy resin as it absorbs moisture [24]. There is also an embrittlement linked to the degradation of macromolecular skeleton by hydrolysis, osmotic cracking, hygrothermic shock for the change of water level and localised damage at the fibre/matrix interface [9]. The more deleterious effect of freezing treatment is irreversible in nature. The sensitivity of composite to strain rate is driven by the resin behaviour [17]. It was observed that the increasing strain rate leads to the increase of failure strength. It may possibly be accompanied by a reduction in matrix ductility [25]. Epoxy resins and glass fibers are known to be highly loading rate sensitive [26,27]. A direct correlation between the loading rate dependency of composites and those of the constituent phases may be difficult or rather complicated [28]. A laminate behaves like a rigid beam and thus less susceptible to bending at higher loading speed. Epoxy resin is more ductile at low strain rate but the failure strain of a matrix resin at high speed may become a limiting factor for the composite strength [29]. It is generally known that a major mechanism of shear strength reduction in fibre reinforced composites upon exposure to various environmental parameters is partial debonding and/or weakening of the interfacial bond. The fibre/matrix interfacial bond greatly influences the mechanical behaviour of composite materials [4,30].

4. Conclusions

It is shown here that the environmentally conditioned glass fibre/epoxy composite is sensitive to loading speed. The strain-rate sensitivity is less pronounced at higher conditioning times. The freezing treatment results in further damaging effect. The degradative effect of further freezing treatment is more evident at lower loading speed. The more detrimental effect because of frozen state of absorbed moisture is permanent. The state of fibre/matrix interface after humid ageing may introduce more complications in evaluating the strain rate sensitivity of fibre reinforced composites.

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