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Temperature effect during humid ageing on interfaces of glass and carbon fibers reinforced epoxy composites

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ABSTRACT

Weight change behavior of fiber-reinforced polymer composites in humid and thermal environments appears to be a complex phenomena. The state of fiber/matrix interface is believed to influence the nature of diffusion modes. A significant weakening often appears at the interface during the hygrothermal ageing. It effects the moisture uptake kinetics and also the reduction of mechanical properties. The importance of temperature at the time of conditioning plays an important role in environmental degradation of such composite materials. An attempt has been made here to evaluate the deleterious effect of temperature on shear strength of carbon/epoxy and glass/epoxy composites during hygrothermal conditionings. Mechanical tests were conducted at room temperature to assess the effectiveness of the relaxation process in the nullification of environmentally-induced damage in the composites.

Key words: Polymer composites; Adhesion; Diffusion; Interfaces; Ageing; Mechanical properties

1. Introduction

The degree of environmental degradation that occurs in a fiber reinforced polymer composite structure is linked directly with the amount of moisture that is absorbed. But the moisture absorption kinetics of epoxy resins differ widely and also change with physical ageing [1]. 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 the stresses associated with moisture-induced expansion may cause lowered damage tolerance and structural durability. The structural integrity and lifetime performance of fibrous polymeric composites are strongly dependent on the stability of the fiber/polymer interfacial region.

Moisture may penetrate into polymeric composite materials by diffusive and /or capillary processes [2-4].The interactions between the fiber and the matrix resin are important rather complex phenomena. Both reversible and irreversible changes in mechanical properties of thermoset polymers are known to occur as a result of water absorption. Plasticization ans swelling are among the adverse consequences of absorbed water. Plasticization iduces plastic deformation in addition to lowering the T_g. Whereas swelling is related to the differential strain which is created by the expansion force exerted by the liquid while stretching polymeric chains [5]. These two damage mechanisms are believed to generate capillarity in polymeric composites. Moisture absorption in polymer composites leads to changes in the thermophysical, mechanical and chemical characteristics of polymer matrix by plasticization and hydrolysis [6]. The amount of moisture absorbed by the matrix resin is significantly different than that by the reinforcement fiber. This results in a significant mismatch in moisture induced volumetric expansion between the matrix and the fibers, and thus leads to the evolution of localized stress and strain fields in fibrous composites [7, 8]. The literature [9-15] has cited a number of possible interactions: selective adsorption of matrix components, conformational effects, diffusion of low molecular weight components from the fiber, penetration of polymer molecules into the fiber surface, and the catalytic effects of the fiber surface on polymers. The low molecular weight impurities may migrate from the bulk of the adhesive to form a weak boundary layer at or near the fiber surface [16]. The active carbon fiber surface can strongly attract polar molecules of the polymer matrix. This may develop a boundary layer of high crosslink density. This microstructural gradient may promote crack initiation and propagation through this layer [17, 18]. Moisture interaction with the metal oxides in E-glass leads to corrosion induced damage and thus results in reduced mechanical strength [19, 20]. Many applications reveal a sensitivity of resin matrix and fiber/matrix interface to both environmental degradation, with interactions between various mechanisms being possible [21]. With the increasing applications of these materials, more and more assessmnet is needed to get a better understanding of interfacial bonding of the materials. The environmental action, such as high moisture and high temperature, can limit the usefulness of polymer composites by deteriorating mechanical properties during service.The present study focuses the effect of temperature on interlaminar shear strength (ILSS) of carbon fiber/epoxy and glass fiber/epoxy laminates during moist and thermal environment conditioning. It is reasonable to state that this strength is effected during such exposure. An attempt has been made here to evaluate the effect of thermal stress on shear strength of the moisture–induced-swelled composites. One of the key features of this material class is their damage initiation and propagation behavior which is spatially distributed in nature and comprises a variety of mutually interacting damage modes. The most common damage modes are matrix cracking, delamination growth and fiber fracture [22].

There has been a pressing need to quantify the degree of environmental degradation on the deviation of mechanical properties of fiber/polymer composites. Impact of environmental factors such as temperature and humidity on composite materials behavior are of significance concern for the aircraft industry since storage and operating conditions vary considerably and can add to the wear and tear of structural components [23]. The average bond strength between an epoxy resin and with an E-glass fiber (≈ 33 MPa) is lower than with a carbon fiber (≈ 57 MPa) [24]. The microstructural gradient between the weak boundary layer [16] at the fiber/matrix interface and the bulk of adhesive matrix may promote the initiation of interlaminar failure and/or propagation of crack through this layer. The nature of interfacial phenomena is strongly influenced by the presence of residual stresses [25]. This may also result in microvoids or cracks [26]. Differential coefficients of thermal expansion between fiber and polymer further develop residual stresses at the interface. These different natures of stresses may weaken the brittle thermoset epoxy resin and/or the interfacial region of the laminate. The mechanisms of interfacial degradation due to hygrothermal ageing range from the reduction in bond strength [27], to creation of osmotic cracks [28], to the lowering of the glass transition temperature of the epoxy resin [26]. The present investigation aims to study the effect of temperature on moisture uptake kinetic and also on interlaminar shear strength (ILSS) of glass/ epoxy composites at 95% relative humidity (RH) atmosphere. It is found that temperature has a dominating effect in changing the nature of absorption kinetic curve at higher temperature conditioning. It is also evident that the reduction in ILSS value is more at higher conditioning temperature for the almost same absorbed moisture inside the laminate compared to lower temperature of conditioning. Water pick-up kinetics and mechanical test (ILSS) are supposed to be indicative of evaluating adhesion chemistry at fiber/matrix interfaces and integrity of composites.

The absorbed water molecules in polymer composites are known have significant effects on their final performance of composite structures especially in their long-term utilization. The resulting hygrothermal forces and residual stresses combined with each other may be sufficiently large enough to influence the failure of laminated composite and thus should not be neglected in modern design analysis and lifetime estimation [29]. Moisture diffusion into a laminated polymer composite is a matrix-dominated phenomena [30]. Failure in many cases occurs in the interface region due to chemical reaction and/or plasticization when impurities (commonly water) penetrate the interface [31]. The stress transfer efficiency from the matrix to the fibers, the stress build-up in broken fibers and the redistribution of the stresses in the neighboring intact fibers are all controlled by the interfacial strength and integrity [32, 33]. Fiber reinforced polymer composite structures are expected to experience a range of hygrothermal environmental conditions during service life. Since absorbed moisture can alter the stress state and degrade the interface, understanding of hygrothermal behavior is critical for predicting structural performance [34].

Epoxy resins are the most common matrices for high performance advanced polymer composites, but they are also inherently brittle because of their high degree of crosslinking. The densely crosslinked structures are the basis of superior mechanical properties such as high modulus, high fracture strength and solvent resistance. However, these materials are irreversibly 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 [35-38]. A number of properties of amorphous materials including fatigue, fracture and component performance are governed by the magnitude of strain fields around inhomogeneities such as voids and cracks [39]. Regardless of the application, once cracks have formed within polymeric materials, the integrity of structure is significantly compromised. Microcracking induced by environment is a long-standing problem in polymer composites. Cracking leads to mechanical degradation of fiber reinforced polymer composites [40-41].

2. Experimental procedure

2.1 Material systems

2.1.1. Carbon fiber/epoxy

The pre-impregnated unidirectional carbon fiber and epoxy resin (XAS/914) were used to fabricate the laminate in this experiment. No sizing was applied on the fiber surface. Laminated composites were prepared by vacuum bagged technique.The weight fraction of fiber in the composites was approximately 0.60. Carbon/epoxy composite specimens were obtained from M/s Hindustan Aeronautical Limited, Bangalore, India to required sizes and specificatiobs.

2.1.2. Glass fiber/epoxy

The fabrication was carried out with woven roving E-glass fibers (FGP, RP-10) and epoxy resin (Ciba-Geigy, araldite LY-556 and hardener HY-951). There was no sizing treatment given on the fiber surface. The weight fraction of fiber in the laminated composites was approximately 0.60. The laminate was fabricated by wet lay-up method. They were allowed to cure for 24 hours at room temperature.

2.2. Experimental method

A microprocessor controlled Brabender climatic chamber was used for the hygrothermal conditionings. The short beam shear (SBS) specimens were prepared by diamond cutter. The dimension of specimens was maintained at per with ASTM standard (D2344) for the 3-point bend test. All the specimens were dried in a oven at 50˚C temperature for the time as long as the weight of each specimen became stabilized.

One lot of carbon/epoxy short beam shear (SBS) test specimens were exposed to 60˚C temperature and at 95% relative humidity (RH) atmosphere. The other lot of the same samples were treated in 70˚C temperature and at 95% RH environment. They were exposed up to about 1200 hours at those conditions.

The one lot of glass/epoxy SBS specimens was exposed to 50˚C temperature and 95% RH and for another lot it was 70˚C temperature and 95% RH environment for about 260 hours. The moisture uptake kinetics were measured at different intervals of the conditioning time.

The weight gain was calculated according to the following equation,

$$\text{absorbed moisture (\%)} = (m_w - m_d / m_d) \times 100 \qquad (1)$$

where m_d is dry weight and m_w is wet weight of the specimen.

Then 3-point bend tests were carried out to determine the ILSS values of the carbon/epoxy and glass/epoxy laminates. Figure 1 shows the schematic diagram of the test geometry with dimensions of specimen. The test was performed with Instron universal testing machine (Model 1195). The testing was done at a crosshead speed of 1 mm/min. There were 5 to 7 samples tested at each point of an experiment and then average value was reported. The ILSS value was determined as follows,

$$\text{ILSS} = 0.75p/bt \qquad (2)$$

where p is maximum load, b is width, and t is thickness of specimen.

3. Results

3.1. Carbon fiber/epoxy

Figure 2 shows the effect of temperature on moisture absorption characteristics of carbon fiber/epoxy composites at 60˚C temperature and 95% RH and also at 70˚C temperature and 95% RH environments. It is clear from the figure that at a higher temperature, the moisture uptake rate is higher. This is due to the higher diffusion rate. Here there is no evidence of anomalous Fickian type absorption rate at higher temperature.

The variations of ILSS values for the both types of conditioned specimens are plotted against the percentage of absorbed moisture in Figure 3. It is clear that the degree of degradation in shear values is more at higher conditioning temperature for almost the same amount of absorbed moisture inside the carbon/epoxy laminates for the longer exposure time (i.e. more absorbed moisture). The probable cause for such behavior may be due the adverse effect of a higher degree of thermal stress at the higher temperature. This higher amount of thermal stress may promote crack initiation and propagation through the boundary layer of high crosslink density at the fiber/matrix interface. The initial rise in ILSS values in both the cases may be due to strain free state in the laminates [26,42]. The curing shrinkage stress is released here by the hygroscopic swelling stress in the initial stage of moisture absorption.

3.2. Glass fiber/epoxy

The moisture gain characteristics of glass/epoxy laminated composites for both the conditioning environments are plotted against square root of exposure time in Figure 4. It is evident that the moisture uptake rate is higher at higher conditioning temperature for the same humid condition. It is also clear that the linear nature of the curve starts deviating at higher rate after about 100 hours of exposure for the 70˚C temperature conditioning. This could be attributed to the ageing of easily degradable weak bond at the interface of glass fiber/epoxy composites.

The 3-point bend test was carried out with the conditioned specimens at room temperature. The interlaminar shear values were determined from SBS test data. Here the specimen may fail by fiber rupture, microbuckling or by interlaminar shear cracking [27]. The variations of shear values are plotted against the percentage of absorbed moisture for both the conditioned specimens in Figure 5. The figure shows that except for the initial period of conditioning, the shear strength decreases with the exposure times for both the conditioning temperatures. The values are less for longer conditioning time for same amount of absorbed moisture with higher exposure temperature. This could be due to more interfacial degradation at higher temperature.

4. Discussion

4.1. Carbon fiber/epoxy

The absence of the anomalous Fickian nature of moisture absorption kinetics may be attributed to the surface-induced crystallization in the epoxy matrix [24] at or near the polar adherent (e.g., carbon fiber surface). The stronger interfacial bond for carbon fiber/epoxy does not show this kind of deviating nature of absorption rate in such type of environmental exposure. That could be the reason that the moisture is not penetrated into the composite by capillary process but by the diffusive route only.

The amount of moisture absorbed by the epoxy matrix is significantly greater than that by the reinforcement carbon fibers. This results in a mismatch in the moisture-induced volumetric expansion between the fiber and polymer matrix. That leads to the evolution of localized stress and strain fields in the composite. The moisture absorption most often leads to changes in the thermophysical, mechanical and chemical characteristics of the epoxy matrix by plasticization and hydrolysis [6]. Integrity of polymer composites in terms of matrix cracking and/or fiber/matrix debonding/discontinuity by humid ageing may be reflected by moisture absorption and interlaminar shear strength studies.

4.2. Glass fiber/epoxy

The coupling effect of swelling stress and thermal stress is higher for the higher conditioning temperature. The anomalous Fickian behavior can be related to the creation of additional free volume by viscoelastic effects [25], by hydrolysis [43] or by a combination of the two. The hygrothermally generated porous and weaker interface may allow capillary flow of moisture in the composites at higher conditioning temperature. It also may be reasonably confirmed that the crack formation and fiber/resin debonding [44] may contribute to such phenomenon in glass/epoxy system.

The probable reasons for such degradation may be related to the weakening effects of higher thermal and moisture induced swelling stresses at the interface and/or in the matrix resin. It may also be hypothesized that this conditioning environment could result in either breakdown of chemical bonds or secondary forces of attraction at the interface. A previous research report [27] suggests any partial regeneration of bond strength is recovered for glass/Epon composites by drying. The initial exception is due to the strain-free state of the composites. The increase may be related to the release of curing stress by the swelling stress.

4.3. Comparison of fiber systems

Figure 6 shows the comparison of ILSS values with the absorbed moisture for both carbon/epoxy and glass/epoxy laminated composites at two hygrothermal conditions. Critical observation may highlight the presence of polymer matrix adherence to the carbon fibers and epoxy matrix damage (Figs. 7(a) to 7(c)). The cleaner fibers and interfacial cracking are prevalent in fractured surface of aged glass/epoxy composites (Figs. 8(a) to 8(c)). The study reveals that the fiber/matrix adhesive damage and a loss of interfacial integrity are dominating mechanisms in polymer composites during environmental ageing.

The diffusion process is complex in fibrous composites. It depends on the diffusivities of the individual constituents, their relative volume fractions, constituent arrangement and morphology. Thee rate of moisture diffusion is controlled by the diffusivity. It is a strong function of temperature and a weak function of relative humidity [45]. Moisture can potentially cause debonding at the fiber/matrix interface not only through chemical attack and reaction, but also through mechanochemical effects such as osmotic pressure [20]. The mechanism of attack at the interface is decisively governed by the chemistry, structure, morphology and modes of failure at that interface. Recent FTIR images suggest that there is a chemical gradient in the structure of the epoxy matrix from the fiber surface to the bulk polymer due to different conversions. This often leads to change in the stoichiometry [46].

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 interfacial bonds. Mechanical degradation is a function of matrix swelling strain [47-49]. The fiber/matrix interfacial adhesion is most likely to control the overall mechanical behavior of fiber reinforced polymer composites. An interfacial reaction may result in various morphological modifications to polymer matrix microstructure in proximity to the fiber surface [50].

5. Summary

The study leads to the conclusion that the higher temperature during hygrothermal ageing not only increases the moisture uptake rate but it may also modify the local stress threshold required for delamination nucleation. The higher temperature acts like an activator of the diffusion of the water molecules through the composite. The less value of ILSS for the almost same level of absorbed moisture at higher temperature could be attributed to the pronounced degradative effect of temepratture. The room temperature test results further reflect the irreversible nature of damage at the interface due to temperature. It is reasonable to conclude that the interfacial adhesion in the carbon/epoxy and glass/epoxy composites is more effected by hygrothermal ageing at higher conditioning temperature and for more exposure time (i.e. more absorbed moisture). The reduction in ILSS values is significant here in both the systems for the same level of absorbed moisture at a higher conditioning temperature. It is not only the absorbed moisture but also under what conditions, it diffuses into the specimen characterizes the interfacial degradation phenomena.

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Figure captions

Figure 1 Schematic diagram of the SBS test geometry with specimen dimensions.

Figure 2 Moisture absorption kineties of carbon/epoxy composites at 60˚C temperature and 95% RH, and at 70˚C temperature and 95% RH.

Figure 3 Variation of ILSS values of carbon/epoxy composites with the absorbed moisture at two different hygrothermal conditions.

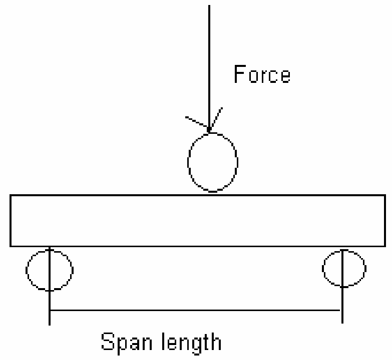
Figure 4 Moisture absorption kineties of glass/epoxy composites at 50˚C temperature and 95% RH, and at 70˚C temperature and 95% RH.

Figure 5 Variation of ILSS values of glass/epoxy composites with the absorbed moisture at two different hygrothermal conditions.

Figure 6 Comparison of ILSS values of carbon/epoxy and glass/epoxy composites with the absorbed moisture at two different hygrothermal conditions.

Figure 7(a) to 7(c) Scanning electron micrograph shows matrix cracking and fiber damage in carbon/epoxy composites**.**

Figure 8(a) to 8(c) De-adherence and interfacial cracking are evident in aged glass/epoxy laminated composites.



Length = 12 mm (carbon), 42 mm (glass)

Thickness = 2 mm (carbon), 6 mm (glass)

Width = 6 mm (carbon), 6 mm (glass)

Figure 1.

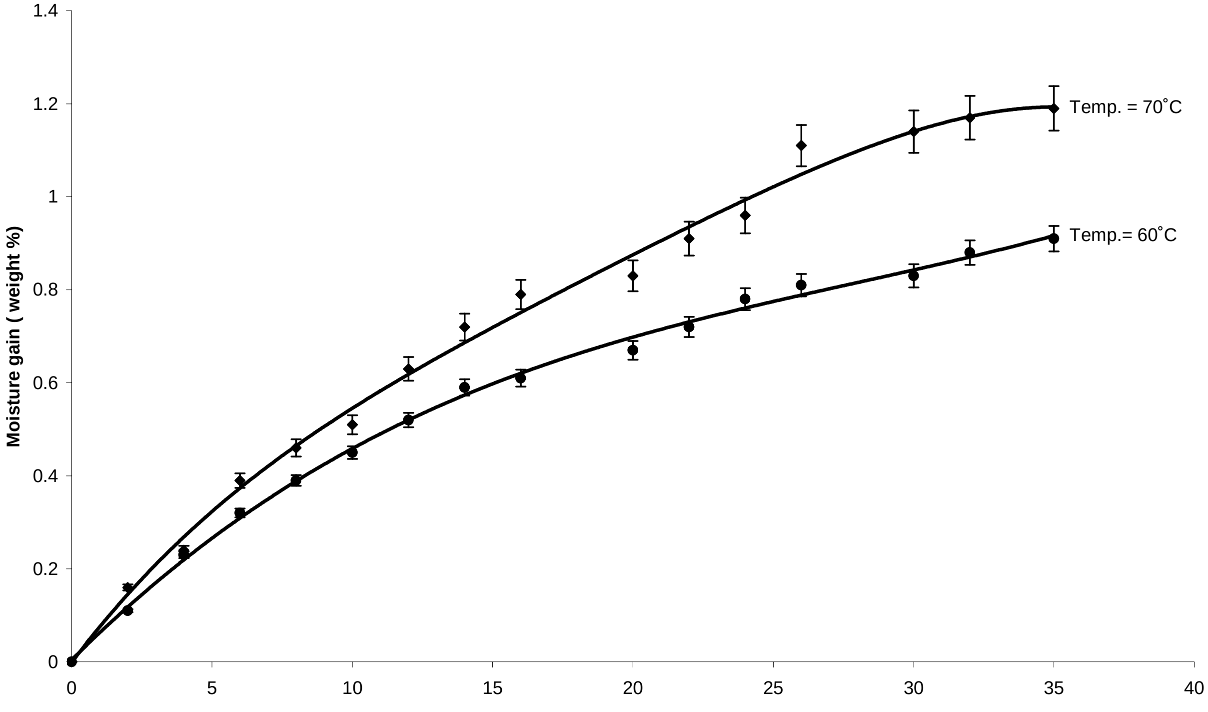

1.4
1.2
1
0.8
0.6
0.4
0.2
0
Moisture gain (weight %)
Temp. = 70˚C
Temp.= 60˚C
0
5
10
15
20
25
30
35
40
Square root of conditioning time (hours)

Figure 2

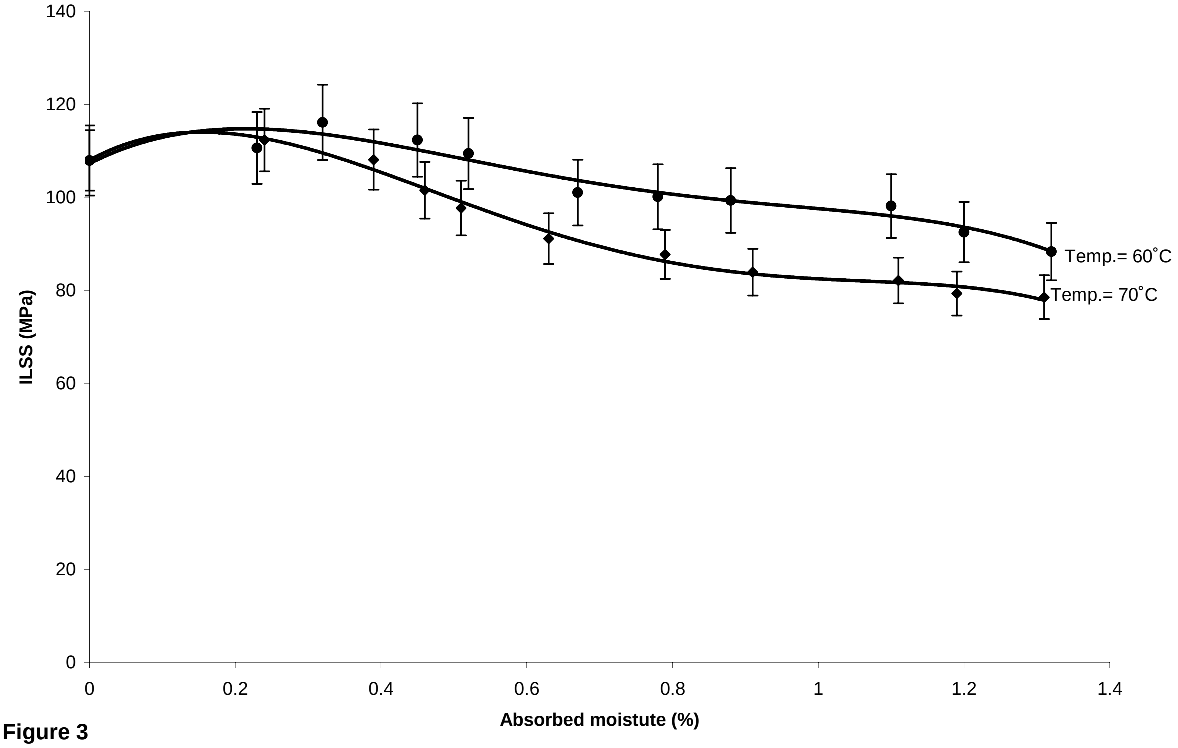
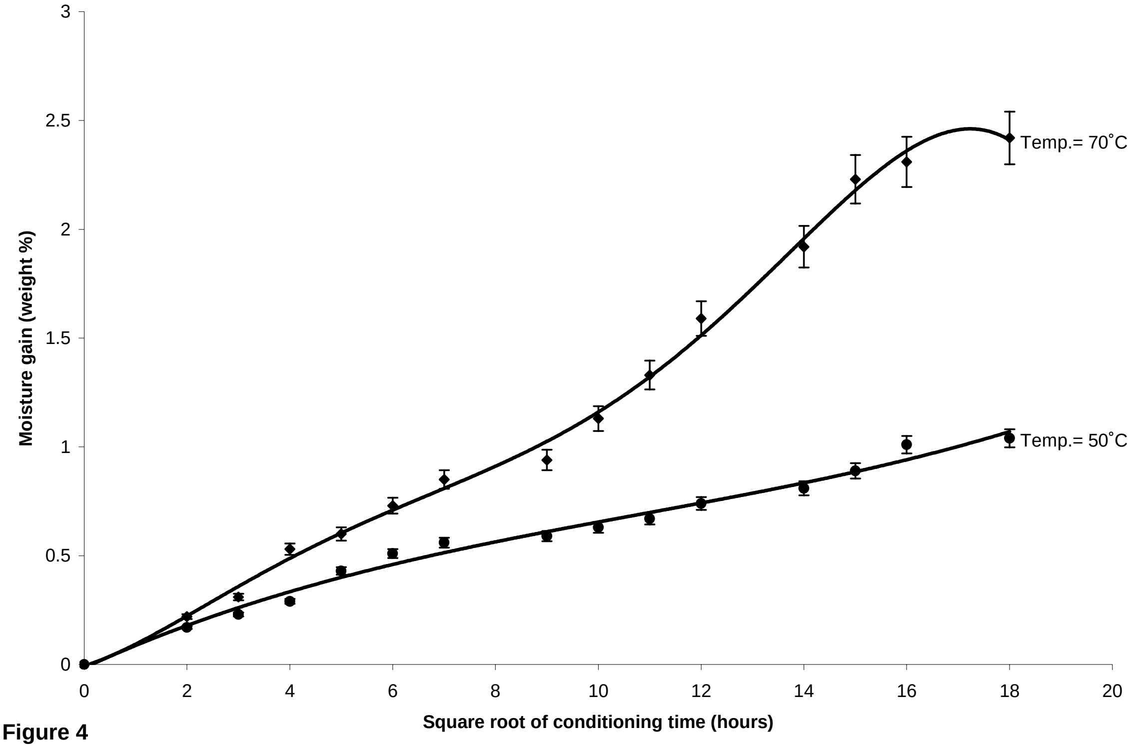
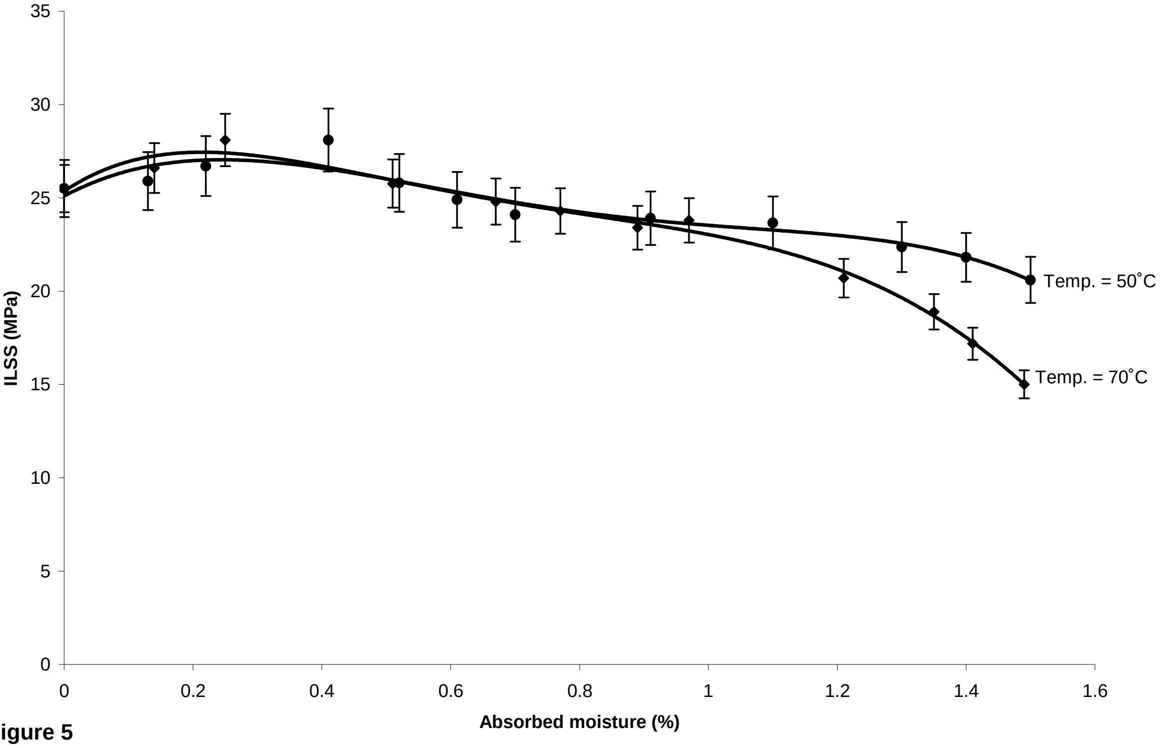

140
120
100
80
60
40
20
0
ILSS (MPa)
0
0.2
0.4
0.6
0.8
1
1.2
1.4
Absorbed moistute (%)
Temp.= 60˚C
Temp.= 70˚C

Figure 3



3
2.5
2
1.5
1
0.5
0
Moisture gain (weight %)
0
2
4
6
8
10
12
14
16
18
20
Square root of conditioning time (hours)
Temp.= 70˚C
Temp.= 50˚C

Figure 4



35
30
25
20
15
10
5
0
ILSS (MPa)
0
0.2
0.4
0.6
0.8
1
1.2
1.4
1.6
Absorbed moisture (%)
Temp. = 50˚C
Temp. = 70˚C

Figure 5

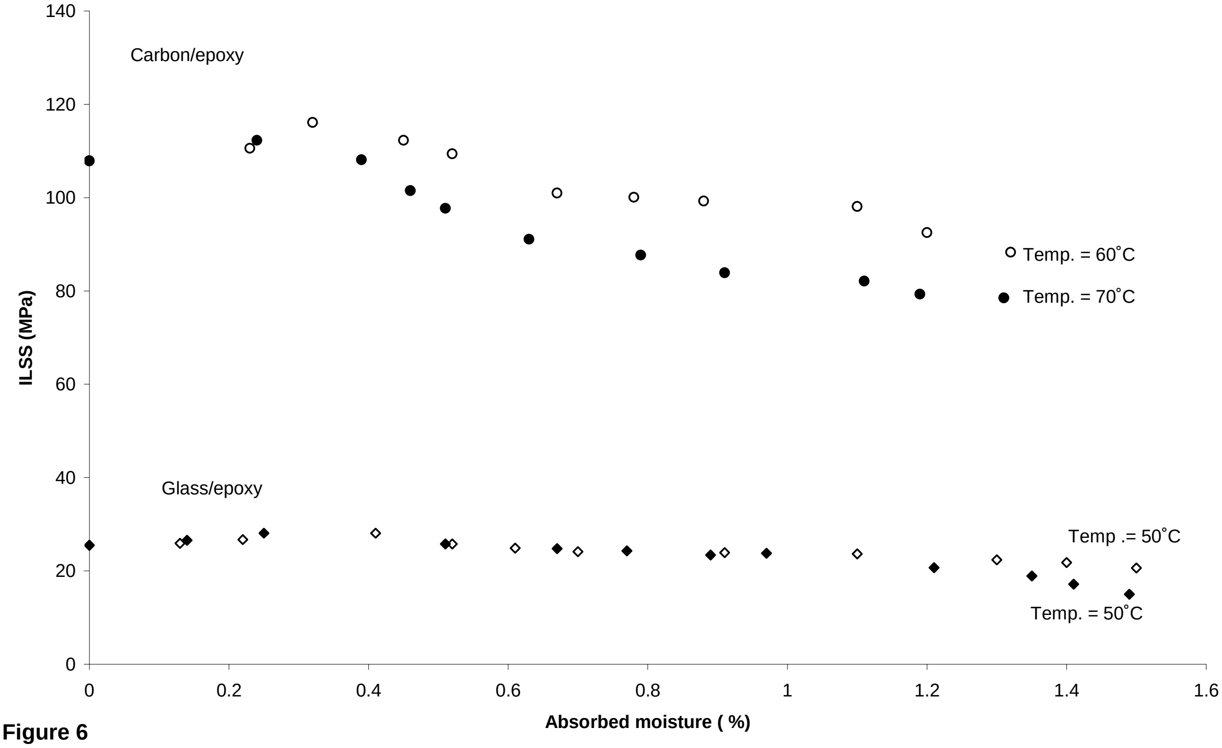

Carbon/epoxy
Glass/epoxy
ILSS (MPa)
Absorbed moisture (%)
○ Temp. = 60˚C
● Temp. = 70˚C
Temp .= 50˚C
Temp. = 50˚C
0
20
40
60
80
100
120
140
0
0.2
0.4
0.6
0.8
1
1.2
1.4
1.6

Figure 6

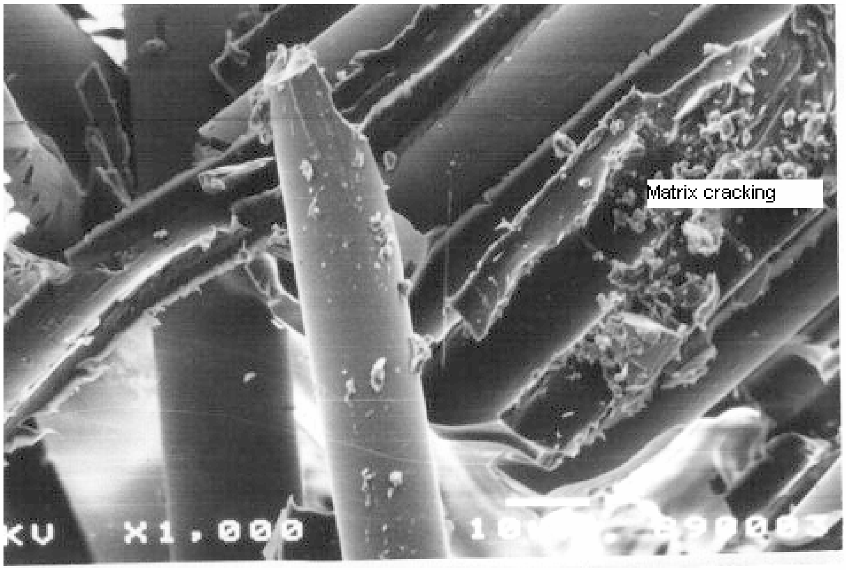


Figure 7(a)

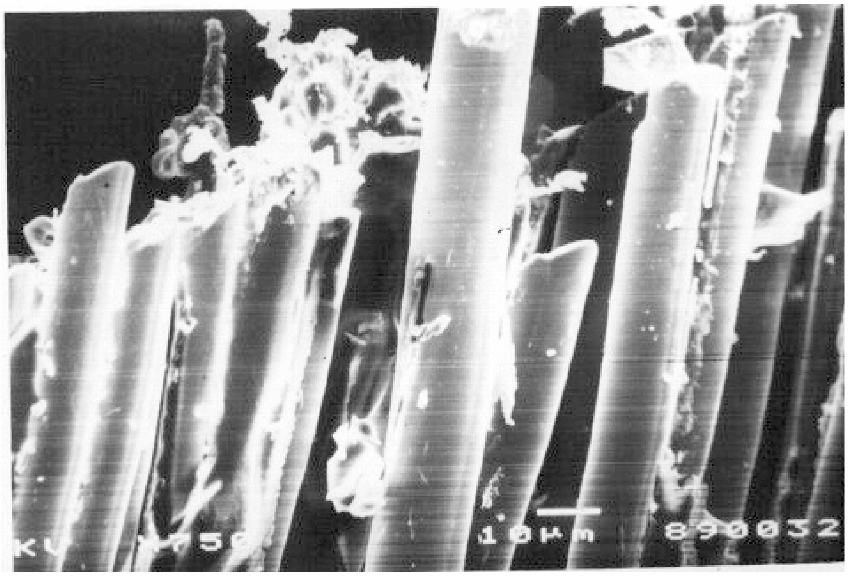


Figure 7(b)

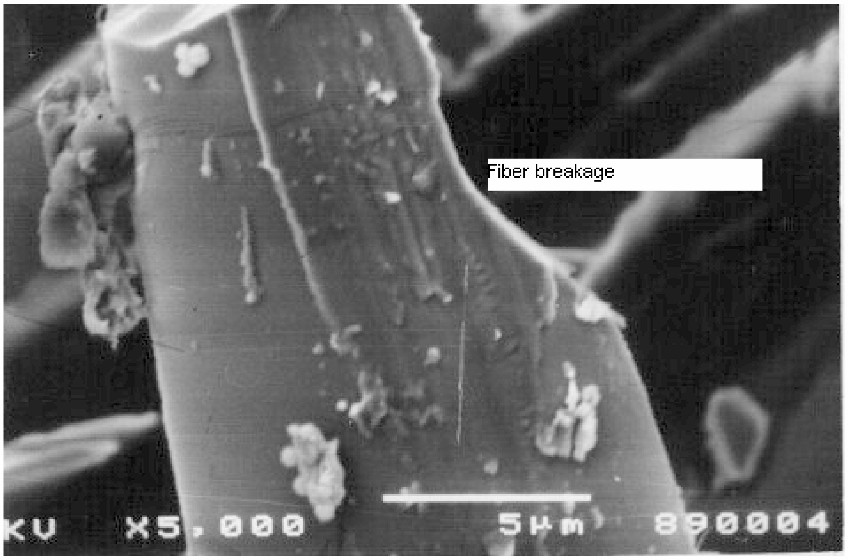


Figure 7(c)

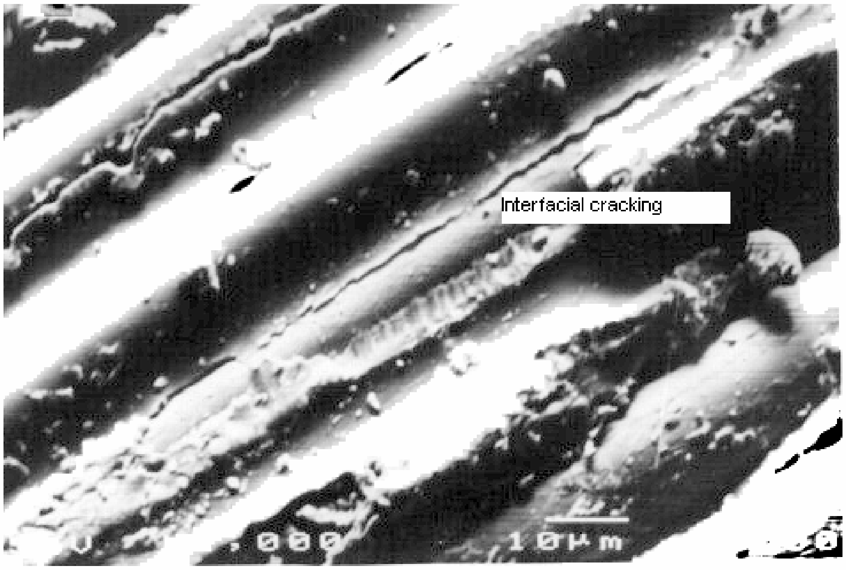


Figure 8(a)

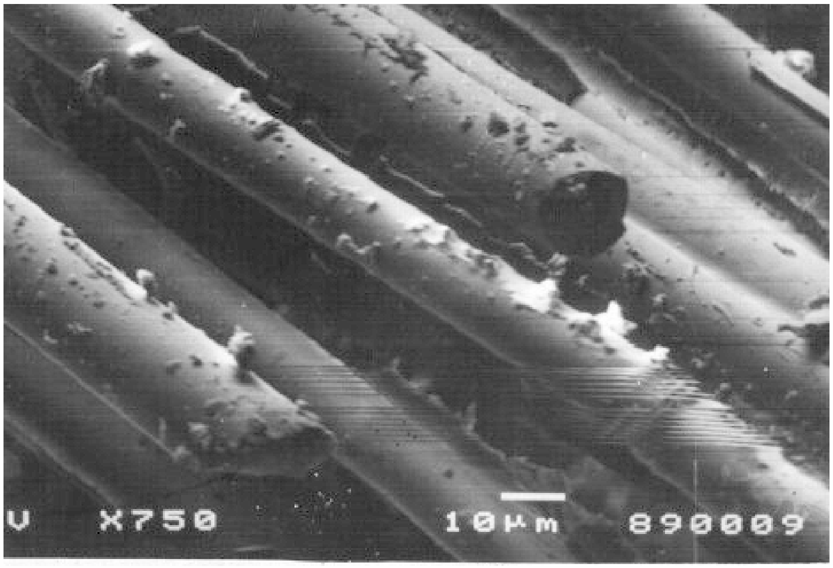


Figure 8(b)

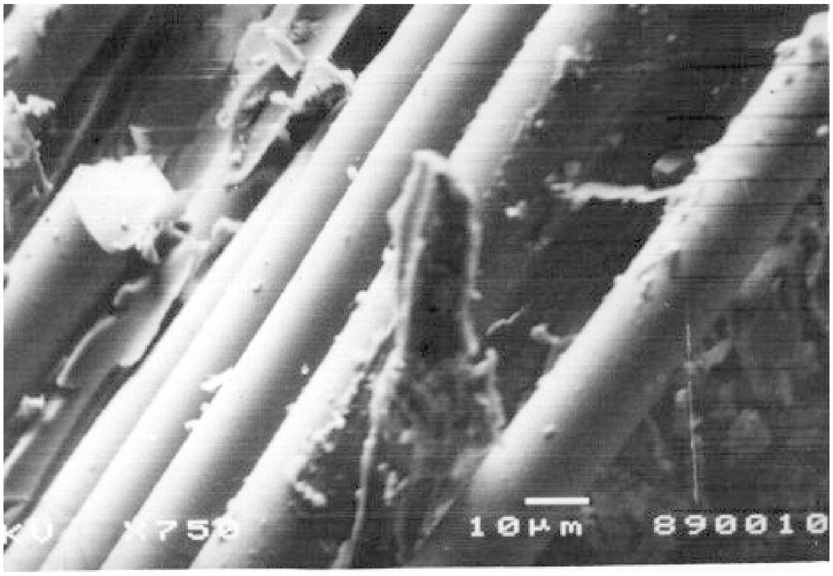


Figure 8(c)