Moisture Effect on Properties of Out-of-Autoclave Laminates with Different Void Content

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Abstract   Fabrication of large structures using out-of-autoclave prepreg materials will lead to a great amount of savings in manufacturing costs. In the out-of-autoclave processing method, the presence of voids inside the laminate has been an issue due to the lack of high pressure during manufacturing. This study aims primarily to observe the moisture absorption response of composite samples containing different levels of void. By changing the vacuum level inside the bag during the manufacturing process, three different unidirectional laminates at three levels of void have been manufactured. After immersing the samples in warm water at 60 ℃ for about one year, the moisture absorption level was monitored and then diffusion coefficients were calculated using Fick's law. Results show that the moisture absorption coefficient changes by %8 within the experimental range of void contents. The mechanical behaviour of these laminates has been studied at four different moisture levels by performing dynamic mechanical analysis (DMA) and short beam shear tests. Empirical results indicate that, in general, interlaminar shear strength and glass transition temperature decrease by moisture build-up inside the samples. DiBenedetto equation is proposed to make a correlation between the moisture content and glass transition temperature.

Keywords   Out-of-autoclave materials, Moisture absorption, Moisture diffusivity

1. Introduction

Polymer matrix composites, especially carbon fiber reinforced polymers (CFRP) have become attractive in the last decades in various industries due to their excellent mechanical properties to density ratio. In some of the applications including aerospace, civil infrastructure and marine sectors, these materials are subjected to harsh environments such as water and high humid conditions. Mechanical properties of composite materials are very sensitive to the different environmental conditions such as temperature, humidity, and corrosive environments. These environmental conditions can affect the matrix and matrix-fiber interface properties. To estimate the durability of the material exposed to such environment, it is necessary to study the mechanical properties of them in harsh environments under different loading conditions.

To study the effect of moisture on composite materials, it is essential to have a look at the chemical reactions between the material and water molecules to know how moisture diffuses through the laminate. Due to the fact that the moisture cannot penetrate through the fibers, resin properties become very critical and will be affected by the moisture diffusion [1-2]. There are two factors which affect the diffusion of penetrant molecules into polymeric material: a) availability of molecular size holes between polymer chains and b) bonding forces between penetrant molecules and polymer [3-4]. Availability of molecular size holes depends on various resin characteristics such as polymer microstructure, crosslink density, stoichiometry, and cohesive energy density while the chemical nature of penetrant molecules and polymer determine the essential bonding force between them. As the hydrogen bonding sites in polymer chains increase, the attraction between water molecules and polymer increases [5-6].

Moisture diffusion into the polymer can be controlled by many factors. Movement of a penetrant through the polymer can be different in each polymer because the nature of polymer plays a critical role in moisture transport. The amount of free volume and segmental mobility in the polymer are the factors which affect the diffusion directly. There are some other factors like degree of unsaturation, degree of crosslinking and degree of crystallinity which can affect diffusion indirectly by changing the segment mobility. Moreover, glass transition temperature has a significant influence on moisture diffusion. As T_g reduces in polymers, segment mobility and moisture diffusivity increase. Furthermore as molecular size of polymers increases, segment mobility of the molecular chains decreases

drastically which leads to lower diffusion rate and moisture content [1,7].

In the case of similarity in the nature of polymers and the crosslink density, the nature of the crosslink can affect the diffusion procedure. It is shown that as molecular chain flexibility increases, the maximum level of moisture uptake and diffusion rate increase [7]. Increasing the plasticisers in polymer chains can enhance the rubbery properties and increase the segment mobility of polymer, which can lead to a jump in diffusion coefficient and reduces the solubility of the polymer [7]. Molecular shape and size is another factor that can affect the rate of diffusion in the polymeric matrix. Several studies thus far have linked the size of penetrant with diffusivity in polymers. It has been reported that as the size of penetrant molecules increases, the rate of diffusion decreases [8,9].

The nature of reinforcement, its compatibility with the polymer matrix and also its adhesion properties are the dominant factors in diffusion of a penetrant in filled polymers [7]. When a filler is not compatible with the matrix nature, the amount of void content increases at the interface, which can increase the free volume and also the rate of diffusion. On the other hand, in the case of compatibility of the reinforcement and the polymer system, the filler can go through the void contents and reduce the diffusivity [10]. Moreover, using the fillers can reduce and restrict the mobility of the penetrant, which can again lead to reduction in diffusivity [11-12]. The difference in the coefficient of thermal expansion (CTE) and the coefficient of moisture expansion (CME) between the fibers and the matrix can make the swelling an anisotropic phenomena in composite materials. Swelling decreases by increasing the fiber volume fraction and can be negligible in the presence of a strong interface [13-14].

In most cases, increase in temperature leads to decrease in solubility and increase in both diffusivity and maximum moisture content in the composite materials [15]. Gillat et al, studied the moisture diffusion of T300 graphite/epoxy under different external loadings at 25 ℃, 60 ℃ and 80 ℃ for two different cross-ply laminates. They observed an increase in diffusivity and maximum moisture content as immersion temperature increased [16]. Lv et al, discovered that an increase in diffusivity at a higher immersion temperature is due to an increase in molecular chain relaxation and reduction in T_g and molecular bonding strength at higher temperatures [17].

Composite structures are often cured in an autoclave to achieve the required aerospace grade quality. However, curing large structures requires access to large autoclaves which is limited and expensive. As a result, fabrication of large structures using out-of-autoclave prepreg materials will lead to a great amount of savings in manufacturing costs. In the out-of-autoclave processing method, the presence of voids inside the laminate has been an issue due to the lack of high pressure during manufacturing. This research aims to study the effects of moisture on properties of an out-of-autoclave unidirectional carbon fiber composite material. The main focus is to investigate the moisture absorption behavior (moisture diffusivity) and mechanical properties of the laminates at different levels of void contents. Laminates are made with three different void contents and then subjected to the moist environment for one year. At different moisture level, samples were weighted and then mechanically tested. Effect of moisture on diffusivity coefficient, glass transition temperature, and interlaminar shear strength will be investigated.

1.1. Fick's Law for Moisture Absorption and Desorption

Fickian diffusion is the most common diffusion model to describe the moisture absorption and desorption through polymeric material. This model was described by Adolf Fick in 1855 for steady state (Fick's first law) and non-steady state (Fick's second law) diffusions [18]. The driving force which leads to the molecular transportation is difference in concentration, which moves the penetrant molecules from higher concentration areas to lower concentration areas in the solvent. This movement ends when the polymer reaches the equilibrium mass concentration. Fick's second law explains the moisture diffusion when concentration gradient changes as time goes on. The following equation is the one-dimensional Fick's second law of diffusion equation with the constant diffusivity assumption [19,20]:

$$\frac{\partial c}{\partial t} = D_z \left(\frac{\partial^2 c}{\partial z^2} \right) \tag{1}$$

where c is the moisture concentration which is the absolute amount of absorbed moisture in a material and expressed as the mass of moisture per unit volume. z is the coordinate through-the-thickness and t is time. D_z is the laminate through-the-thickness moisture coefficient of diffusion or moisture diffusivity coefficient. For an infinite plate (width and length >> thickness), Jost derived the following solution [19]:

$$\frac{c - c_i}{c - c_m} = 1 - \frac{4}{\pi} \sum_{j=0}^{\infty} \frac{1}{(2j+1)}$$

$$\sin \frac{(2j+1)\pi z}{h} \exp \left(-\frac{(2j+1)^2 \pi^2 D_z t}{h^2} \right) \tag{2}$$

where c_i is the initial moisture concentration inside the sample (which is zero for dry sample) and c_m is the maximum moisture concentration at the laminate surface. Sample thickness is h and t is the exposure time. The typical moisture concentration at time t is shown in Figure 1.
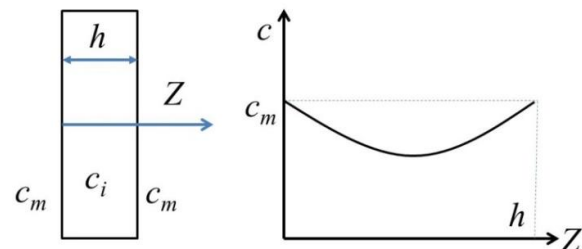

Figure 1.   Typical through-the-thickness moisture concentration at time t

Average moisture content, M (%), is an indication of the average amount of absorbed moisture in a material. It is defined as the ratio of the mass of the moisture in the material to the mass of the oven-dry material and expressed as a percentage. A schematic of perfectly Fickian moisture absorption process is described in Figure 2 based on the moisture content calculation. After finishing the absorption process, when the laminate reaches its maximum level of moisture content, considering the initial linear slope of the absorption curve, the diffusion coefficient of the material can be calculated as follows [20]:

$$D_z = \pi \left(\frac{h}{4M_m} \right)^2 \left(\frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}} \right)^2 \qquad (3)$$

where M_m is the maximum level of absorbed moisture. M_1 and M_2 are moisture contents at time t_1 and t_2, respectively. These two points are selected in the linear region for calculation of the initial slope of the curve.
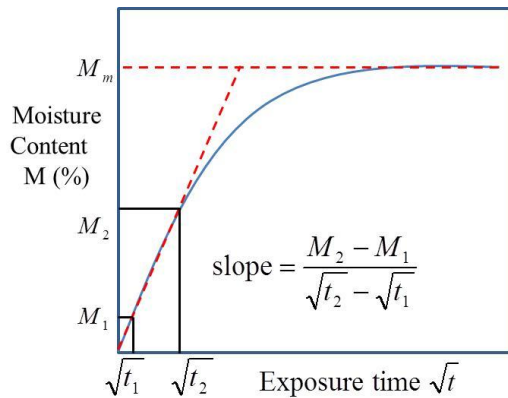

Figure 2. Moisture absorption process based on Fick's law

2. Materials and Manufacturing

Material used to manufacture samples is CYCOM 5320-1 which is produced by Cytec Engineered Materials Inc. This is one of the most common out-of-autoclave materials used in aerospace applications. Material specification is given in Table 1.

Table 1. Material specifications provided by material supplier [21]

Commercial name	UD OOA-CYCOM 5320-1
Manufacturer	Cytec Materials
Fiber	T650-UD carbon
Resin	Cycom 5320
Areal density (g/m^2)	882.34
Resin content (%)	36

One of the most challenging tasks is the ability of producing unidirectional composite laminates with different and acceptable void contents. In hand lay-up process, the applied pressure to the resin and vapour pressure from the trapped moisture between the plies play a critical role in the formation of the voids inside the laminate. During the manufacturing process, when the applied pressure from the

vacuum bag becomes less than the vapour pressure between the plies, porosities can form [22]. To add extra void content, Costa et al [23], produced homogenous void content in the laminate by spraying water uniformly during the layup procedure and having control on applied pressure on resin. To avoid having chemical reactions between the layers, it was decided to control the void content by changing the vacuum level inside the bag during manufacturing process.

Three different laminates containing 24 layers with 200 mm $\times$ 200 mm dimensions were made at different levels of void. Before cutting the prepregs to the desired dimension, the prepreg layers were put at room temperature for about 2 hours. After cleaning the tool, a thin layer of release agent was used to ease the demoulding process after curing. Then, prepregs were stacked up one on top of another. During stacking process, it is common for the air to be trapped between the layers. A roller was used to remove the trapped air and reduce the void content. It has to be mentioned that for the samples with less void content, the roller was used more often. Then the standard bagging method was followed to prepare the material for curing. One thin layer of release film was applied and a bleeder layer was used to absorb the extra resin during the curing process. At the next step, a breather layer was applied which allow the moisture vapours that was forming during the curing process to escape. The last layer was the vacuum bag which covered the laminate surface. The vacuum bag was attached to the mould edges by using sealant tapes. For having three levels of void contents, the vacuum level inside the bag was changed and also frequency of using the roller during the lay-up process was different. The vacuum levels and frequency of using the roller are described in Table 2.

Table 2. Different conditions for manufacturing samples with different void content

| Sample	Vacuum level MPa (in-hg)	Roller usage frequency
A	0.099 (29)	Every layer
B	0.067 (20)	Every 4-layer
C	0.034 (10)	Every 8-layer

A curing cycle suggested by Cycom 5320-1 was followed to have perfectly cured composite laminates [21]. The oven temperature was increased from room temperature to 121 ℃ with a ramp rate of 3 ℃/min and was held for 3 hours to complete the curing stage. Then, the temperature again was increased with the same rate to 177 ℃ and kept for 2 hours for post curing. At this point, the temperature was allowed to cool down to the room temperature.

Three different UD laminates having different levels of void contents were fabricated. To study the moisture absorption behaviour and mechanical properties of laminates under different level of moisture, samples were cut to the required dimensions based on the different standards. Coupon sizes and dimensions based on the related standards are provided on Table 3. To cut the samples, a circular saw machine with coolant was used.

Table 3. A summary of sample dimensions for different tests based on related standards

	Tests		
	DMA	ILSS	Moisture absorption
Sample width (mm)	10	6.5	100
Sample length (mm)	50	20	100
Number of repeats	2	3	2
Related standard	DMA manual	ASTM D2344	ASTM D5229

Void content was measured by doing microscopic observation for three random specimens from each main plate. Microscope's software was used which utilized difference in transparency in each cross section to identify different regions. To have a good image from cross-section, it is required to have a great surface finishing. Therefore, the samples were polished step by step from coarse size grinding paper to the finest size. Figure 3 shows the sample of optical microscopy for all three laminates. After microscopy the amount of 0.2%, 1% and 2% void content for each laminate was calculated.

Sample A	Sample B	Sample C
(0.2% $\pm$0.05% void content)	(1% $\pm$0.05% void content)	(2% $\pm$0.05% void content)
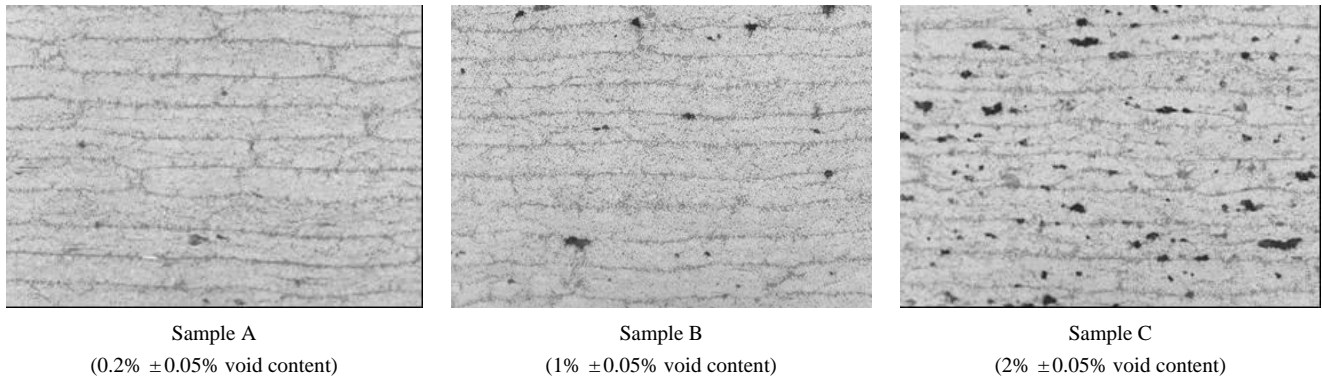
Figure 3. Cross sections of three different samples

3. Experiment

(a)   (b)
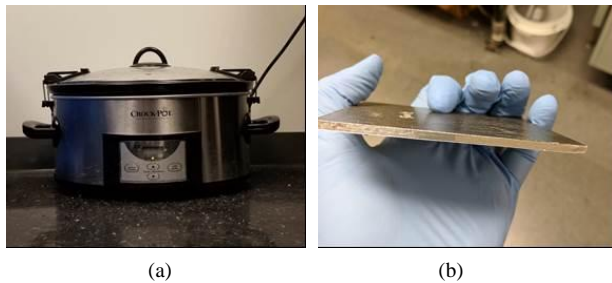
Figure 4. a) Moisturising pot, b) Sample after adding aluminum foil to the edges

After cutting the samples, it is crucial to dry the samples. After measuring the initial weight of the samples, the samples were put in an oven for different times at 60 ℃. During the drying procedure, the weights of the samples were monitored and the procedure was stopped when the weight does not change more than 0.02% as suggested by ASTM standard. At this step, the last monitored weight was labeled as W_d. Since our objective is to obtain the through the thickness diffusivity and moisture absorption behaviour of composite laminates, the samples were bonded by stainless steel foil at the edges (Figure 4). This prevents the penetration of the moisture from the edges and the only passage for moisture to get into the laminate will be from the laminate surface. Before starting the moisturising, the weights of the samples were measured to consider the extra weight the steel foil. This weight is called W_{Al}. A slow cooker pot was used for moisturizing the samples. The pot was filled with the water and the samples were placed inside the pot and temperature was set to 60 ℃ [20,24].

Temperature was monitored through an independent thermocouple which was placed inside the laminate during manufacturing. Every week the samples were removed, cleaned and weighted (W_i) and then put back in the pot.

It has been reported that exposure to the moisture environment can lead to increase in polymer flexibility and ductility while it can reduce the elastic modulus and strength [25,26]. In order to investigate the effect of both void and moisture on the stiffness and strength of the composites, dynamic mechanical analyzer DMA and short beam shear test were used. Dynamic mechanical analysis is one of the most common experiments to measure the mechanical properties of wide ranging materials from low to high temperature. DMA can provide valuable information such as storage modulus, loss modulus, damping factor and glass transition temperature. DMA tests were performed using a TA instrument Q800 using three-point bending fixture. Dimensions of the samples were 50 mm $\times$ 10 mm $\times$ 3.5 mm and the fibres directions were perpendicular to the length of the samples (90 ˚). That is because of the fact that moisture effects to the fibres are negligible and our interest is to study the change of matrix properties as the amount of absorbed moisture increases. In three-point bending mode, the sinusoidal force is applied at the middle of the sample with the frequency of 1 Hz while the samples were heated from room temperature to 220 ℃ with ramp of 5 ℃/min.

Inter laminar shear strength ILSS which is considered as one of the matrix dominated properties is measured using short beam shear test [27]. ASTM D2344 standard test method was followed to conduct the ILSS tests using three point bending mode at room temperature. Samples are cut as the fibers are parallel to the length of the samples (0 ˚). Z5

single column tensile tester was used with the Wyoming three point bending fixture to perform the ILSS tests. According to the ASTM standard and based on the specimen's dimensions, span length of the machine was 14mm and the tests were run with the crosshead speed of 1 mm/min.

4. Results and Discussion

4.1. Moisture Diffusivity Coefficient

The procedure proposed by ASTM was followed to run the moisture tests and to collect and analyze the data [20]. The sample weights of six unidirectional carbon/epoxy laminates at three different void contents were measured and recorded during different intervals within one year exposure time. The provided results are average of weight measurement of two specimens. The moisture content was calculated from change of weight using the following equation [20]:

$$M, \% = \left(\frac{W_i - W_{Al}}{W_d}\right) * 100 \qquad (4)$$

The results are shown in Figure 5. As can be seen, the moisture absorption for all laminates was linear up to almost 1.6% moisture level. After that, there is a change in slope of the moisture absorption which illustrates the laminates are getting saturated. For the laminate with the lowest void content, the weight change is less compare to the laminate with the highest void content. This might be related to the void effect. Up to 1.6% moisture absorption, the resin might get saturated and then the water might start to fill the void areas more.
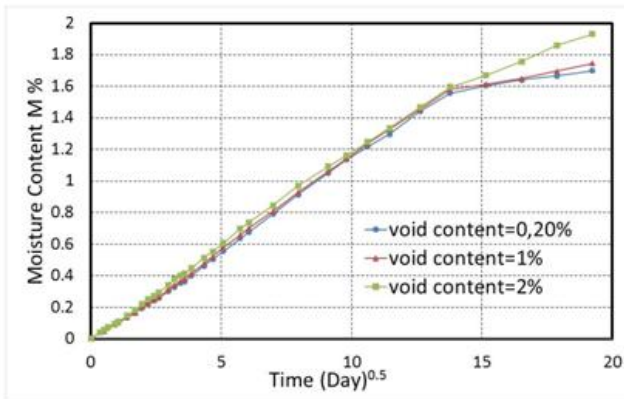

Figure 5. Experimental data from moisture absorption process of composite laminates at three different levels of void

In this research in order to calculate the diffusivity, it was assumed that the last weighted sample after one year represents the maximum level of absorbed moisture due to the time limitation. According to the experimental data, increasing the void volume fraction of laminates led to an increase in the amount of absorbed moisture. Using Fick's law and ASTM standard method, Equation 4 should be used to determine the diffusivity. The second parenthesis in

Equation 4 represents the slop of the experimental results at the linear section of the data. Figure 6 shows the variation of the moisture contents with time for three different void levels at linear section. The slop of the data was calculated using the best linear fit to the data. The slop, thickness, and effective moisture equilibrium content M_m, and diffusivity for each case are shown in Table 4.
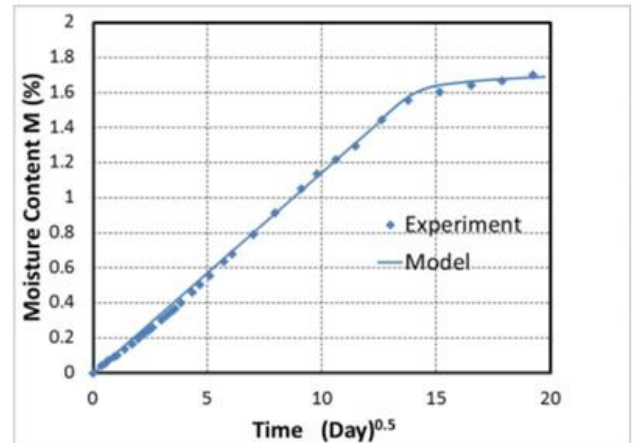
(a)
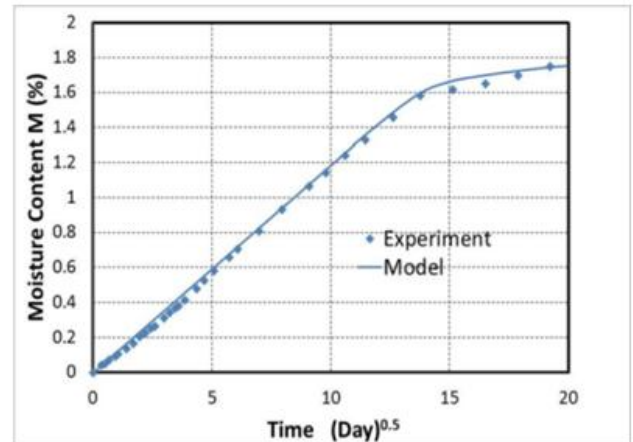
(b)
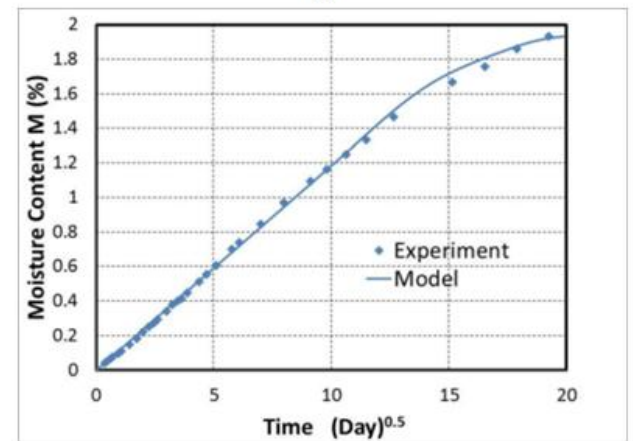
(c)

Figure 6. Moisture content vs time for three different void contents a) 0.2%, b) 1%, and c) 2%

Table 4. Diffusivity for samples containing three different void levels

Void Volume Fraction (%)	Sample Thickness (mm)	M_m (%)	Slope $\left(\dfrac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}}\right)$	Diffusivity D_x (mm²/sec)
0.2	3.4	1.70	3.8x10⁻⁴	1.161x10⁻⁷
1	3.5	1.75	3.9x10⁻⁴	1.202x10⁻⁷
2	3.9	1.93	4.0x10⁻⁴	1.283x10⁻⁷

The results indicate that as the void content increases, the diffusivity increases by 8%. In fact diffusivity will be a function of void level (Figure 7). In order to find a mathematical expression to describe the variation of diffusivity vs void contents more data is required. However, it can be concluded that within the range of experiment, the behaviour can be assumed to follow a linear function as:

$$D_z = 7.24 \times 10^{-7} \, v + 1.13 \times 10^{-7} \qquad (5)$$

where v is the void volume fraction.
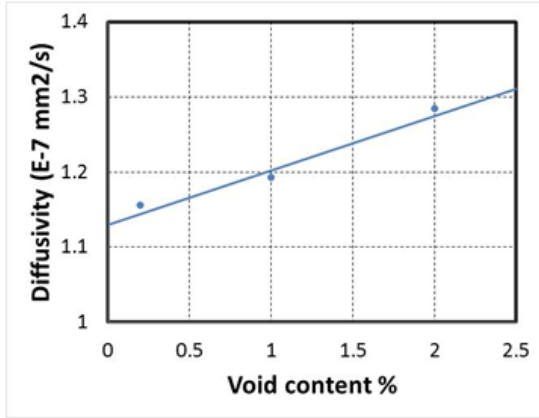
**Figure 7.** Variation of moisture diffusivity and void content

4.2. Interlaminar Shear Strength

Short beam shear test was performed to measure interlaminar shear strength. The load-displacement curve for a dry sample with the minimum void content (0.2%) is shown in Figure 8a. All the samples were examined for the mode of failure based on the ASTM D 2344 standard. Typical sample failure is layer slippage at the layer boundaries as shown in Figure 8b.

For each sample, shear strength is calculated by the following equation [28]:

$$\tau_{ILSS} = 0.75 \times \frac{P_m}{b \times h} \qquad (6)$$

Where τ_{ILSS} is the interlaminar shear strength (MPa), P_m is the maximum load observed during the test (N), b is the measured laminate width (mm) and h is the average laminate thickness (mm). Figure 9 shows how ILSS varies for the composite laminate after exposure of samples to the moisture environment. Results are the average of three (3) specimens. Comparing the ILSS values for two panels clearly indicates the effect of the void on the composite properties. For the dry samples, ILSS reduces by almost %15 from 103 MPa to 87.6 MPa. Similar trend can be seen on the other moisture levels.
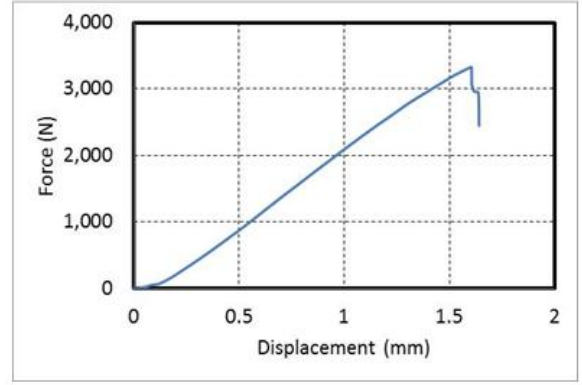
(a)

(b)

Figure 8. a) Force-displacement curve for a short beam shear test, b) Mode of failure
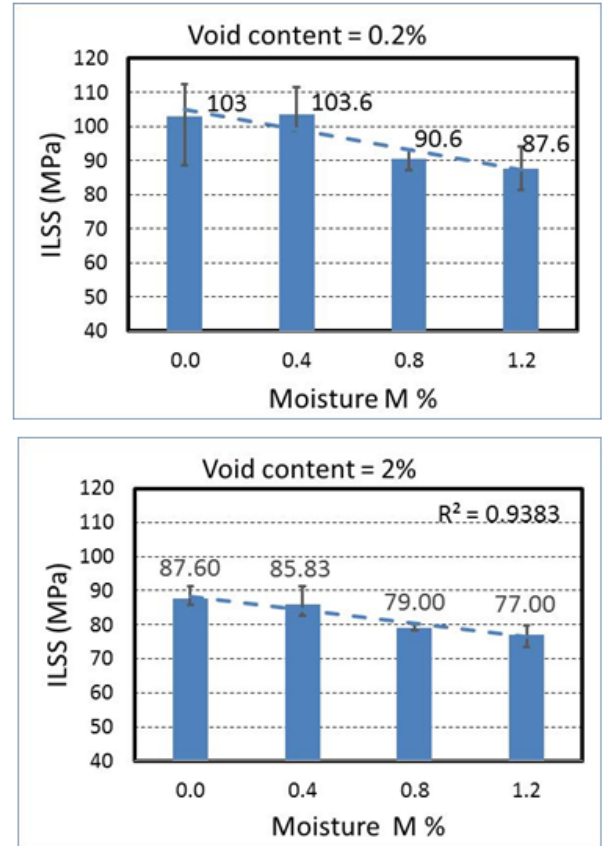
Figure 9. Interlaminar shear strength of unidirectional laminates containing 0.2% and 2% void contents at four different moisture levels

For the samples with the void content 0.2%, the ILSS first increased a bit from 103 MPa to 103.6 MPa after absorbing 0.4% moisture. This might be due to the completion of remaining crosslinking during the moisturising which was

performed by keeping the samples in the hot water at 60 ℃. Then, it dropped sharply about 12.5% to 90.6 MPa after absorbing 0.8% moisture. After absorbing 1.2% moisture, the ILSS drops almost 15% at the end. Examining the results for the panel with 2% void content shows the same behaviour. For these samples, the ILSS drops by 12% at the end at the moisture level of %1.2.

4.3. Glass Transition Temperature

Glass transition temperature is another valuable output from DMA tests. T_g is the temperature which the material goes from glassy state to the rubbery state with a sharp drop in stiffness. The DMA results for sample with minimum void content at dry condition are shown in Figure 10. As temperature increases, the storage modulus of the sample decreases while loss modulus of the sample increases which can cause an increase in damping factor Tan δ . T_g can be determined by the temperature at the peak value of the tan δ curve.

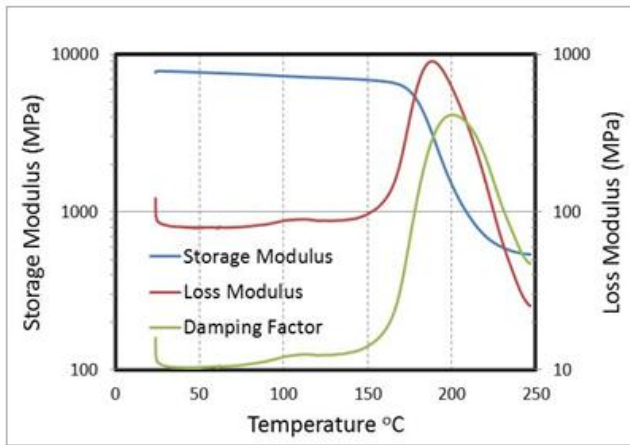**Figure 10.** DMA results for dry sample with 0.2% void content

Damping factor (Tan δ) refers to the ratio of the loss modulus to the storage modulus. Figure 11 indicates the variation of tan δ for the unidirectional laminates at dry condition and three different levels of moisture. After absorbing 0.4% moisture, tan δ dropped by almost 25%. Tan δ continuously reduced as temperature increased and reached at its minimum value at 1.2% moisture level. It can be observed that the shape of the damping factor changes as the moisture level increases. This is an indication of the moisture penetration inside the polymer structure.

Glass transition temperature (T_g) is another remarkable output of DMA tests. Although T_g can be presented using storage modulus, loss modulus, and tan δ plots versus temperature, it is preferred to use Tan δ graphs. The temperature at the first peak of Tan δ is considered as the glass transition temperature. Variation of T_g for all moisture levels are provided in Figure 12. It is clear that T_g tends to reduce continuously with increasing moisture level because water acts as a potent plasticizer [29]. It can be seen that after absorbing 1.2% moisture, T_g dropped by almost 23%. The same trend is reported by other researchers as well [30].
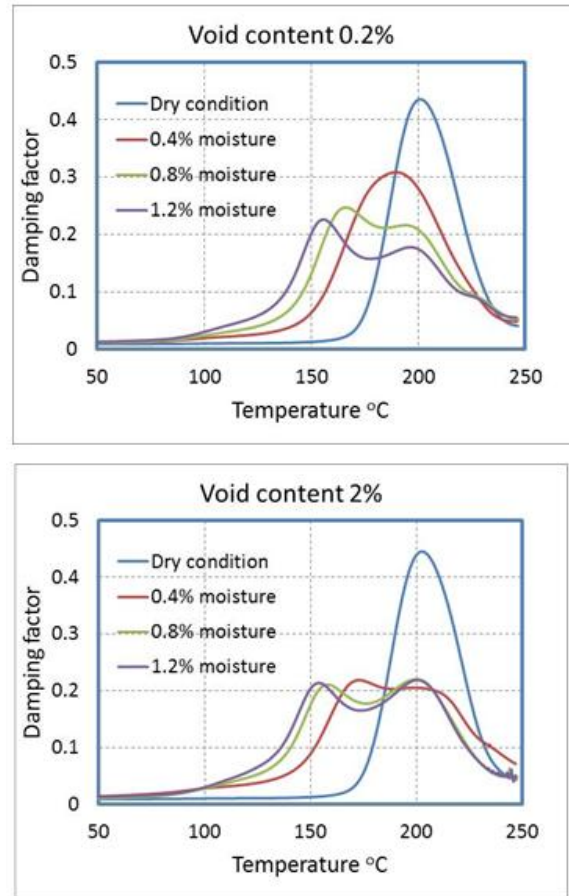

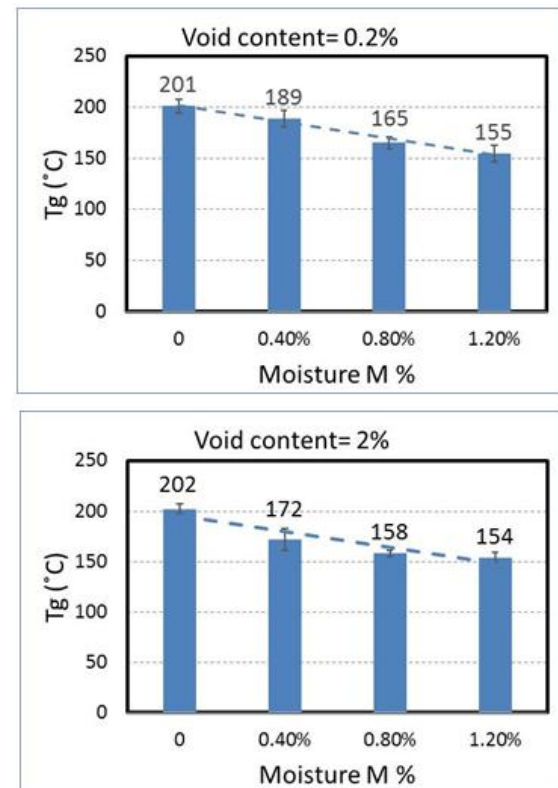**Figure 11.** Variation of Tan δ at different moisture content

Figure 12. Change of glass transition temperature with moisture content

4.4. DiBenedetto Equation

During the manufacturing of thermosetting composite materials, as the degree of cure and crosslinking increases, the glass transition temperature increases. For many thermosetting systems, the relationship between glass transition temperature Tg and degree of cure (α) can be described by the empirical DiBenedetto equation as [31]:

$$\frac{T_g(\alpha) - T_g(0)}{T_g(\infty) - T_g(0)} = \frac{\lambda \alpha}{1-(1-\lambda)\alpha} \quad (7)$$

where $T_g(0)$ is the glass transition temperature of the uncured resin, $T_g(\infty)$ is the glass transition temperature of the fully cured material, and λ is a material constant. This equation is fitted to the experimental data to obtain $T_g(\infty)$, $T_g(0)$ and λ.

When the thermosets absorb the moisture, the presence of water molecule inside the resin molecular structure has softening effect and therefore the glass transition temperature drops. This behaviour is the opposite to the curing process. However, through analogy, the form of the DiBenedetto equation can be adopted to capture the variation of the glass transition temperature vs moisture content (M). The modified DiBenedetto equation can be expressed as

$$\frac{T_g(M) - T_g(D)}{T_g(\infty) - T_g(D)} = \frac{\lambda M}{1-(1-\lambda)M} \quad (8)$$

where $T_g(D)$ is the glass transition temperature of the completely dried resin, $T_g(\infty)$ is the glass-transition temperature of the fully saturated resin, and λ is a material constant. M is the moisture content (%). This equation was fitted to the experimental data to obtain $T_g(D)$, $T_g(\infty)$ and λ (Figure 13). The results are as follows:

$$T_g(D) = 207 \; ^\circ C$$

$$T_g(\infty) = 150 \; ^\circ C$$

$$\lambda = 1.45$$

By having the level of moisture, the glass transition temperature can be predicted.
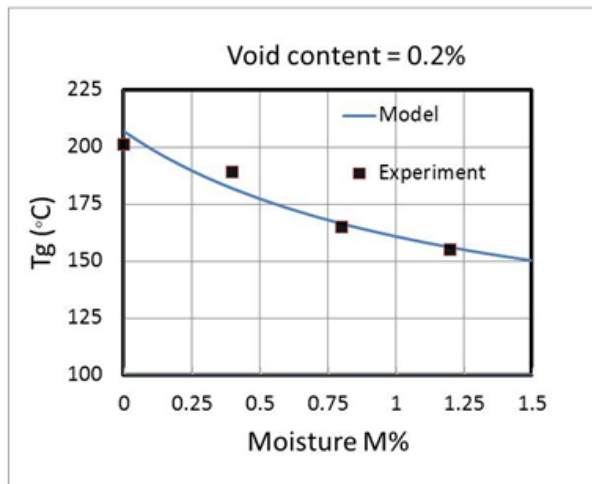
Figure 13. Variation of glass transition temperature vs level of moisture using proposed modified Dibenedetto equation

5. Conclusions

The moisture absorption behaviour of carbon/epoxy unidirectional composite laminates manufactured by out-of-autoclave prepreg material was investigated. Three unidirectional composite laminates containing different levels of void exposed to the hot water at temperature of 60 ℃. Laminates moisture absorption were monitored by weighting the samples at different intervals. Fick's law of diffusion was employed to find the moisture diffusivity coefficient. It was shown that as the void content of the sample increased, the diffusivity of the material also increased. A linear equation was also suggested to relate void content of the laminates and diffusivity. Interlaminar shear strength (ILSS) of the laminates was studied by short beam shear tests. The moisture effect on ILSS was almost the same for all levels of void contents. Moisture reduced the ILSS drastically by about 15% compared to the dry condition. Glass transition temperatures (T_g) for all the laminates were determined by the temperatures related to the peak value of tan δ graph from DMA experiments. It was shown that the moisture generally reduced the glass transition temperature of the laminates. T_g tends to reduce after each moisture absorption level. In all levels of void content, T_g reduced by almost 25% after absorbing 1.2% moisture, compared to the dry condition. DiBenedetto equation was proposed and used to correlate the glass transition temperature to the moisture content.

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