EFFECTS OF HYGROTHERMAL HISTORY ON THE STRUCTURAL PERFORMANCE OF AEROSPACE COMPOSITE MATERIALS: PRELIMINARY EXPERIMENTS AND MASS DIFFUSION MODELS

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ABSTRACT

Experimental research was conducted to characterise the effects of hygrothermal history on the structural performance of Hexcel™ IM7/8552 composite laminates.

The laminates were first conditioned with a series of absorption/desorption cycles at accelerated hot/wet condition (70 ºC in water). Gravimetric techniques have been used to measure the weight variation and consequently record the moisture content and the material saturation.

After various cycles of absorption and desorption, changes to key structural properties such as stiffness, glass transition temperature (T_g), and tensile strength of the materials were evaluated via Differential Scanning Calorimetry (DSC), resonant vibration testing and tensile testing. Scanning Electron Microscopy (SEM) was also conducted to inspect for physical damage to the composite.

Gravimetric test results have clearly shown Fickian diffusivity and saturation to increase with each hygrothermal cycle. The effective diffusivity was also found to increase. This suggests that a potential permanent change in the material can occur in a composite laminate when subjected to hygrothermal history, whilst stiffness and tensile strength are virtually unaffected.

Besides experimental work, Finite Element modelling was performed to study the complex moisture diffusion in thick laminates containing ply-drops. The model is complementary to the experiments as it allows the analyst to understand the "preferred paths" of moisture diffusion and to understand which parts of the structure are likely to saturate first.

1 INTRODUCTION

Composite materials are widely used in the aerospace industry because of their high specific stiffness and strength. They have been used both in the airframe and in the aero-engine fan blade and casing designs. Even if composite materials are inherently corrosion resistant, they can suffer from electric current damage or moisture absorption [1]. Moisture absorption may lead to matrix plasticisation, resulting in a degradation of structural performance [2], especially when loaded in compression [3]. Fracture toughness [4], fatigue life [3] and stiffness [5] may also be negatively affected. As moisture can reduce the structural performance of composite structures, it is important to understand the moisture content and distribution as a function of time and as a result of multiple absorption/desorption cycles. Composite materials potentially used in aero-engines, furthermore, may be subjected to cyclic hygrothermal ageing, which is the accumulated long-term damage of the composite due absorption and desorption cycles at elevated temperatures.

Traditionally, in order to study moisture absorption and the induced changes in structural properties, test coupons are first saturated in a moisture chamber (85% relative humidity) at high temperature (generally not higher than 70 ºC, otherwise chemical changes may be induced in the matrix) and then structurally tested. However, there appears to be a lack of understanding/data on the effects of hygrothermal history on the structural performance of composite materials. The term "hygrothermal history" is used here to describe the series of absorption/desorption cycles applied to the composite material before performing a structural test or measurement.

The microstructure of the polymer matrix of an organic composite material is susceptible to changes due to moisture absorption, especially at high temperatures. Moisture can permanently or temporarily alter the chemical structure by inducing matrix plasticisation, crazing or hydrolysis. At a macro-level, hygrothermal expansion may lead to excessive stresses due to the anisotropic nature of the hygrothermal expansion coefficients of fibre composites. Such prolonged residual stresses can lead to permanent interface damage. The combination of the above-mentioned micro and macro-scale accumulated damage may degrade the composite and thus its mechanical performance.

The aim of this project is to characterise the moisture absorption and desorption behaviour of a carbon fibre epoxy composite subjected to different moisture cycles in terms of moisture content, saturation and structural performance degradation (variations in stiffness and resonance frequencies).

In this research, the Hexcel™ IM7/8552 cured pre-preg samples have been cyclically conditioned in 70 ºC water and in a hot air oven. Upon each saturation (at each cycle), the samples were examined for changes in the polymer matrix through differential scanning calorimetry (DSC) and scanning electron microscopy (SEM). The mechanical structural properties were tested by frequency sweep vibration tests followed by destructive tensile testing. Finally, the complex moisture diffusion in thick laminates containing ply terminations was investigated via ply-level finite element analysis (FEA) using the software Abaqus.

2 MOISTURE ABSORPTION/DESORPTION IN COMPOSITES

The phenomenon of moisture diffusion in composite materials can be divided into two different regimes: the so-called 'Fickian diffusion' and a long-term absorption [7]. An example of weight uptake due to moisture absorption is shown in Fig. 1 for composite panels of thickness 1 mm, 2 mm and 4 mm [7].

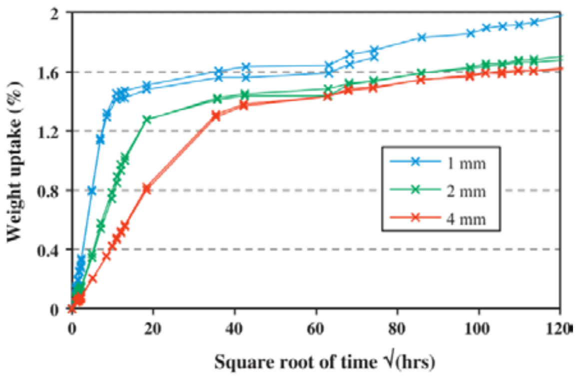

Figure 1: An example of moisture absorption for laminates of different thickness [7].

The Fickian regime is governed by a differential equation which for a unidimensional case is,

$$\frac{\partial C}{\partial t} = D\frac{\partial^2 C}{\partial x^2} \tag{1}$$

where C is the moisture concentration, D is the isotropic diffusion coefficient, t is time and x is the spatial coordinate.

The *diffusivity coefficient* determines how quickly saturation is reached, and *saturation* is the maximum amount of moisture that the composite material may contain. Both measures are thought to be functions of temperature and relative humidity. Different diffusivity coefficients are generally measured during the absorption, desorption and re-absorption phases. For pre-preg continuous-fibre composite materials, three unique diffusivity coefficients can be attributed to the fibre direction, in-plane transverse and out-of-plane directions.

The degree of ageing of a composite subjected to a hot-wet environment also has an impact on hygrothermal activities. The curves in Fig. 1 are generic gravimetric readings which represent the bulk absorption behaviour. The two regimes described above are easily observed, namely a linear region at the beginning of absorption (Fickian regime) followed by a non-linear region occurring after the saturation limit. Note that the duration of hygrothermal conditioning required to reach the non-linear region is relatively long as the horizontal axis of the graph is in square root hours. Therefore, the study presented here was limited to the Fickian region only.

3 EXPERIMENTAL METHODS

Test specimens consisting of 8 plies of Hexcel™ IM7/8552 have been manufactured at the University of Bristol to undergo hygrothermal aging (70 ºC water when absorbing, 70 ºC hot air oven when desorbing) and mechanical investigations (tensile test and natural frequency measurements). The lay-up of each specimen was the cross-ply $[0, 90]_{2S}$. With dimensions 200 mm × 30 mm × 1.048 mm, the specimen was designed to reach the moisture content corresponding to Fickian equilibrium within a relatively short period of time (as the panes were only about 1 mm thick).

The procedure adopted in this work – absorption/desorption cycles, mechanical testing and inspection – is summarised in Fig. 2.

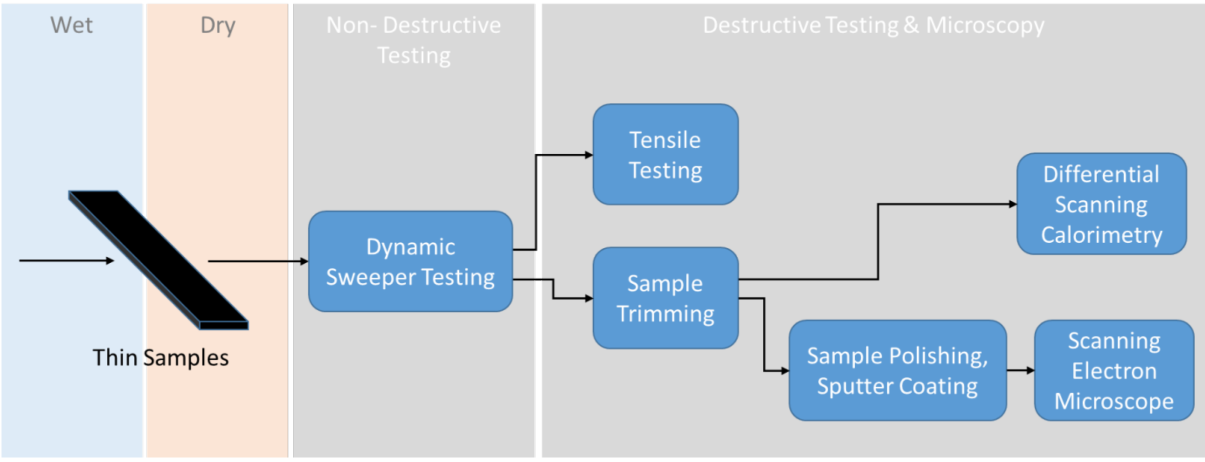

Figure 2: Schematic representation of the experimental work performed.

3.1 Gravimetric Testing

The gravimetric tests follow the ASTM D5229/D5229M standard for moisture absorption properties and equilibrium conditioning of polymer matrix composite materials. The composite specimens have been initially immersed in a container with water at 70 ºC until the average moisture content of the material was observed to change by less than 0.02% over two consecutive measurements. Their weight was regularly monitored until saturation was reached. Once saturated, the specimens were dried in a fan-assisted oven at 70 ºC. Also in this case, the weight was monitored at regular intervals of time. The process was repeated three times to yield three sets of samples as shown in Fig. 3. At the end of each hygrothermal cycle, mechanical tests were performed.

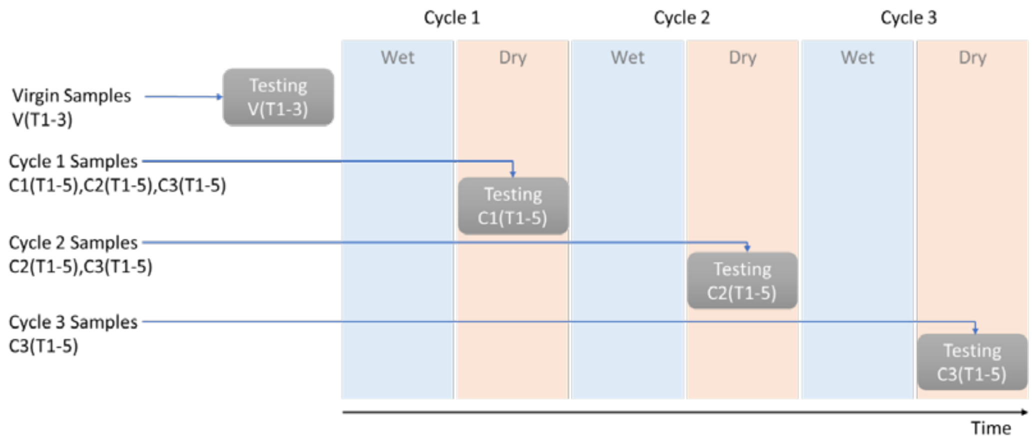

Figure 3: Schematic representation of the three absorption/desorption cycles. At the end of each cycle mechanical tests were performed.

3.2 Dynamic Frequency Sweep

Dynamic frequency sweeps were used to measure the resonance frequency of the specimens as they underwent hygrothermal ageing. Natural frequency is highly sensitive to small changes in stiffness caused by moisture absorption and material ageing. Resonance frequencies were measured using the dynamic frequency sweeper in the Modal Validation Facility in the University of Bristol. The set-up of the equipment is shown in Fig. 4. The sample was clamped along its centre resulting in a dual-cantilever configuration. A vertical oscillatory force was applied by means of an electromagnetic shaker, and a laser Doppler vibrometer measured the response of the specimen at a point near one of its ends.

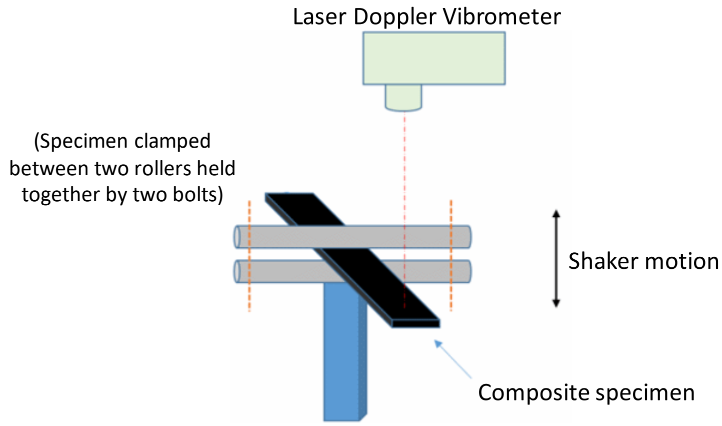

Figure 4: Test setup for the dynamic frequency sweep.

3.3 Tensile Testing

Quasi-static tensile tests were performed on the specimens using an Instron™ 100 kN universal testing machine, with a grip pressure of 500 psi and a cross-head speed of 1 mm·min^{-1}. A 'clip-gauge' contacting extensometer was used to measure strains.

3.4 Differential Scanning Calorimetry

A Differential Scanning Calorimetry (DSC) has been used to study the changes of the polymer after hygrothermal ageing. The thermal response of the epoxy resin was studied by comparing the glass transition temperature (T_g), the degree of crosslinking and melting point of aged samples with the ones of pristine samples.

Changes to the mechanical behaviour of the thermoplastic phase contained in the matrix material were expected to be negligible. Consequently, a Modulated Differential Scanning Calorimetry (MDSC) test was performed as opposed to the more traditional DSC. This option resolved the issue where the transition temperature of interest is overlapped or overshadowed by a more dominant feature such as residual cure, which prevents the accurate interpretation of results.

3.5 Scanning Electron Microscopy

Lastly, Scanning Electron Microscopy (SEM) was used to visualise any micro-structural changes that may have occurred due to hygrothermal ageing. The objective of the SEM, in other words, was to search for physical evidence of damage due to the absorption/desorption cycles.

A secondary electron beam was used to study the surface topography and examine micro-cracks. Back-scattered electron (BSE) beam was used to search for any chemical alterations due to prolong moisture saturation. The Hitachi™ *TM3030Plus* SEM was used, set to a 15 KeV beam and a working distance of 5.6 mm.

4 RESULTS

The results of these various techniques are presented in this section. They concern both the absorption/desorption processes (moisture content), as well as tensile and vibration tests.

4.1 Gravimetric Testing Results

Absorption data (for the three cycles) are given in Fig. 5. The plot shows the average weight uptake of all the specimens conditioned as a function of $\sqrt{t}$, where t is expressed in hours.

Figure 5: Averaged weight uptake interpolation as a function of $\sqrt{t}$ for all the conditioned specimens.

Absorption/desorption cycles can be seen to affect both the diffusivity and saturation of the material. In other words, the time needed to saturate the material and the maximum moisture concentration are strongly dependent on hygrothermal hitory. The saturation value of the material is given by the last data point of the graph, this value is defined and recorded as the point where two consecutive readings were measured to contain less than 0.5% change from the previous measured value.

The change in diffusivity and saturation are a strong indication of potential permanent damage to the matrix material. The “bulk” diffusivity value (due to omnidirectional absorption) of the composite material is given by [9],

$$D_{eq} = \pi \left(\frac{h}{4M_\infty}\right)^2 \left(\frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}}\right)^2 \quad (2)$$

where:

D_{eq} is bulk diffusion coefficient (not taking into account) the directional differences;

h is the thickness of the coupon;

M_∞ is the weight at full saturation;

M_i is the weight at time point i;

t_i is the elapsed exposure time at time point i.

Diffusivity and saturation data for the three cycles of the experiment are given in (Table 1).

Table 1: Hygrothermal cycles *vs.* diffusivity and maximum saturation weight.

Hygrothermal Cycles	Diffusivity [$mm^2 \cdot s^{-1}$]	Maximum saturation weight [g]
1	3.8460×10^{-6}	9.595
2	3.5098×10^{-6}	9.628
3	1.2615×10^{-6}	9.717

4.2 Vibration Testing Results

As previously mentioned, the natural resonance frequency was used as a measure of stiffness/mass changes of specimens subjected to hygrothermal history. The natural frequencies of each sample were averaged and plotted against the number of hygrothermal cycles in Fig. 6.

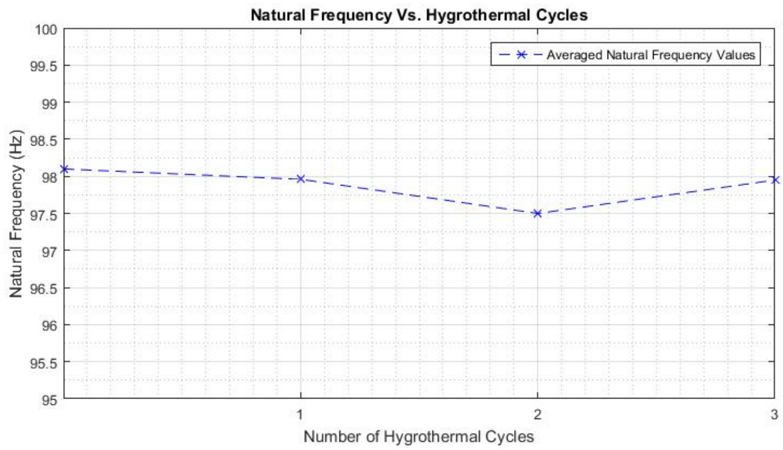

Figure 6: Changes in natural frequency with respect to hygrothermal history.

It can be seen that, although the saturated mass of the specimen changes with the number of hygrothermal cycles, the resonance frequency is not strongly influenced. However, a consistent trend was not observed. It can be deduced that the stiffness variation induced by the hygrothermal history was negligible, at least for the small number of cycles performed in this piece of work. As future work, it would be interesting to increase the number of hygrothermal absorption/desorption cycles to explore further potential stiffness degradation mechanisms due to ageing.

4.3 Tensile Test Results

Fig. 7 shows the ultimate tensile strength (UTS) of each individual sample. Scatter of results was observed throughout the experiment, and no conclusive trend could be observed with respect to changes in hygrothermal history. This is an indication that moisture absorption and desorption had no significant impact on the tensile properties of the material. This is expected as the tensile load were carried by the 0º plies (parallel to the loading direction), while damage to the material is expected to be matrix-dominated rather than fibre-dominated.

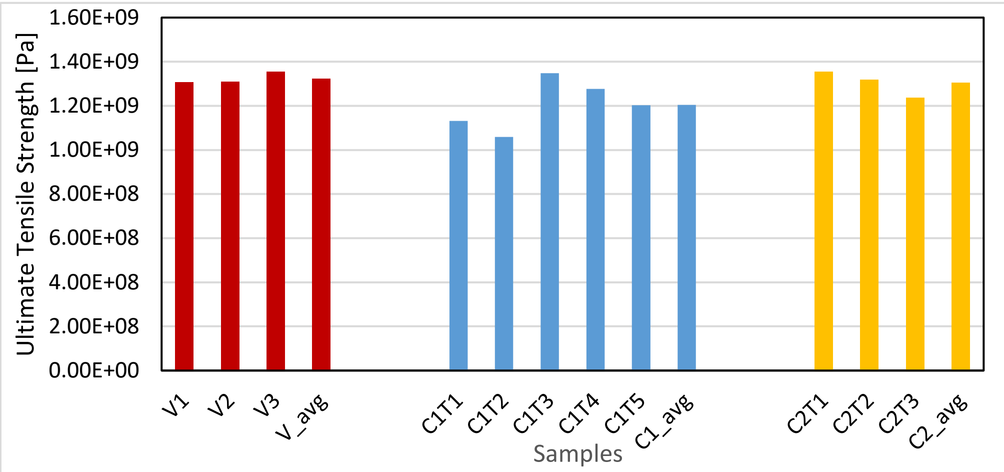

Figure 7: Ultimate tensile strength of individual samples.

Alternatively, Fig. 8 shows stress-strain graphs from tensile test. An elastic region can be identified for each sample with failure occurring at approximately 0.014 strain. This typical failure behaviour and limit is expected in IM7/8552 composites. No significant changes in the Young's modulus of the material were observed with increasing hygrothermal cycles.

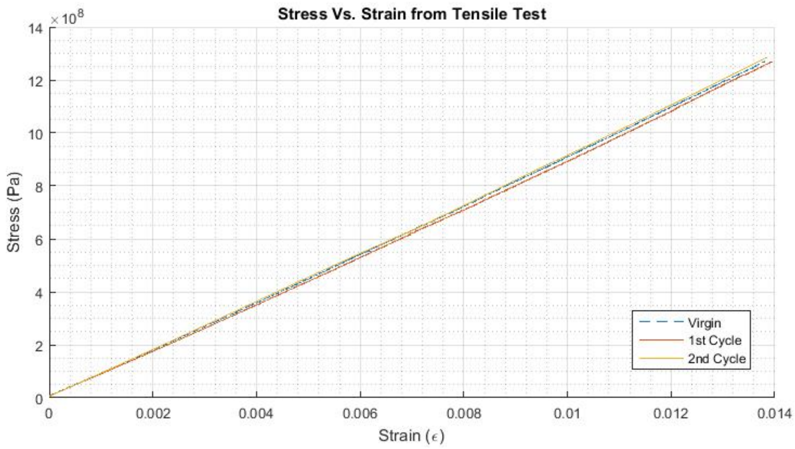

Figure 8: Stress and strain graph of samples with different hygrothermal history.

It can be deduced that the tensile strength is not noticeably affected by the hygrothermal history for the small number of cycles applied.

4.4 Differential Scanning Calorimetry

Modulated Differential Scanning Calorimetry (MDSC) was performed on the samples after destructive testing. Using a 3 ºC·min^{-1} heating rate with a 1 ºC modulated ramp, the specimen was brought from 25 ºC to 350 ºC to observe the thermal properties of the polymer in the composite.

The correlation between the number of hygrothermal cycles and the glass transition temperature range of the specimens is shown in Fig. 9. A positive correlation was observed where increased absorption / desorption cycles lead to a shift in T_g., indicating that more energy is required to transform the polymer from a glassy state to a rubbery state. This result was confirmed by the analysis of the end of T_g range, Fig. 9b, where the values also shifted in a similar manner. It can be concluded that there is a significant difference between pristine samples and hygrothermally-cycled samples.

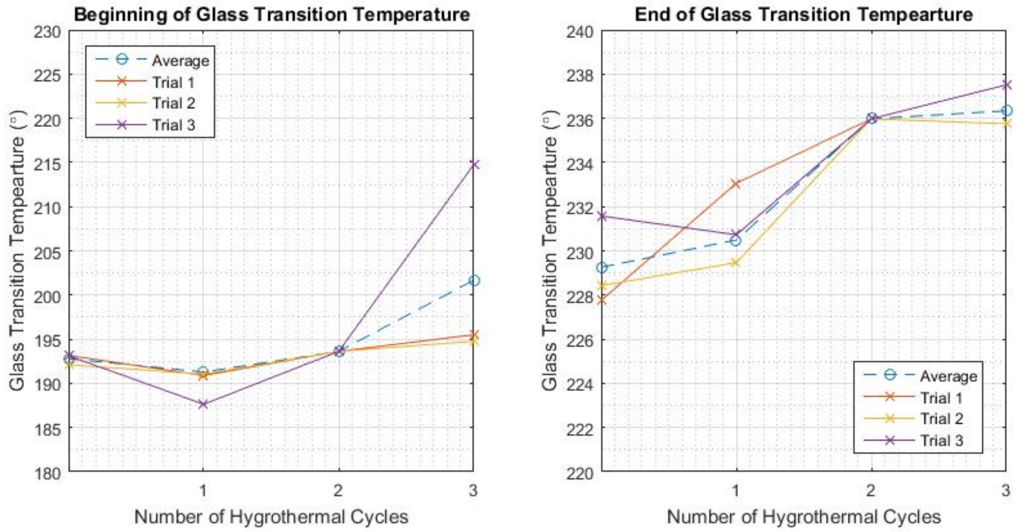

Figure 9: (a) Beginning of T_g with respect to hygrothermal cycles; (b) end of T_g with respect to hygrothermal cycles.

4.5 Scanning Electron Microscopy

Scanning electron microscopy (SEM) was used to study the microstructure of the composite after each hygrothermal cycle. Attention was paid to regions of interests, including fibre-matrix interfaces, interlaminate regions, and resin pockets. The objective was to find evidence of moisture-induced damage due to hygrothermal aging.

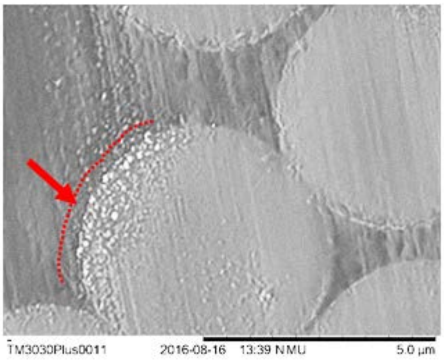

Figure 10: SEM at 16,000× magnification showing potential evidence of fibre-matrix interface damage.

A SEM image of a specimen after two hygrothermal cycles at 16,000× magnification is shown in Fig. 10. Details of the fibre-matrix interface can be observed in this image. It is known from literature that hot-wet testing of composite materials can lead to severe damage along the fibre-matrix interface [12]. Severe cracking phenomena could not be observed in this image; however, signs of fibre-matrix interface debonding were observed and are highlighted by the red arrow. The scale of delamination is minor (no stiffness degradation recorded at the macro-structural level), but it could potentially lead to the onset of crack formation on a greater scale with increasing hygrothermal cycles and applied load.

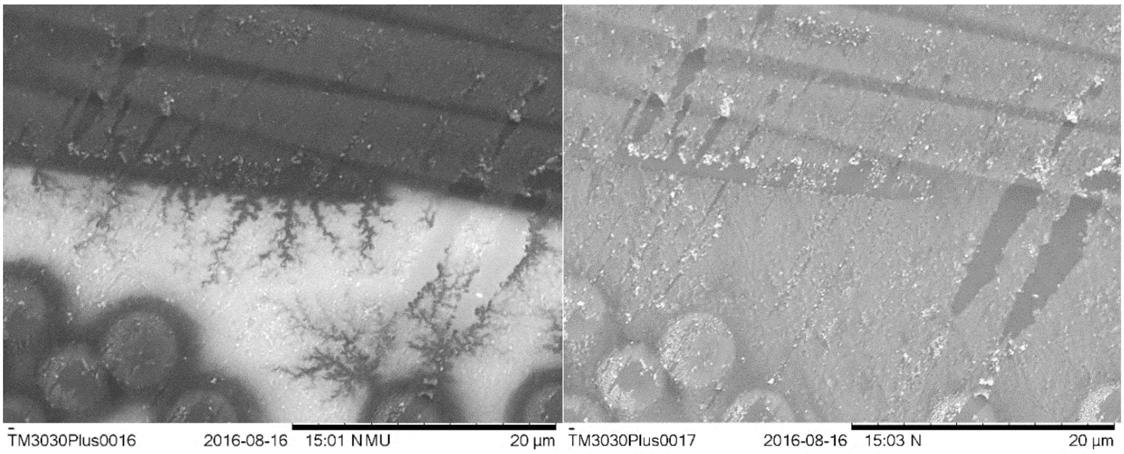

Figure 11: (a) SEM at 5,000× magnification, fractal pattern (b) BSE at 5,000× magnification of the same region of the specimen.

At high magnifications of up to 5,000×, 'fractal' patterns can be observed in secondary electron microscopy as shown in Fig. 11a. The patterns expand out of existing dark shaded regions around the fibre matrix interface. The SEM was switched to the BSE mode and the exact same location was imaged as shown in Fig. 11b. The fractal patterns were not observable under BSE settings. This is an indication that the pattern is a topographical feature instead of an elemental phase change. Therefore, it can be hypothesised that the fractal patterns could potentially be micro cracks formed by hygrothermal history.

5 FINITE ELEMENT MODELLING

5.1 Moisture mapping

Finite Element (FE) modelling was conducted in the software Abaqus (v6.14) to study moisture diffusion in complex composite laminates including internal ply terminations ('ply-drops'). The objective was to simulate the absorption / desorption behaviour with three-dimensional spatial resolution.

Gravimetry experiments can help understand weight uptake, but they do not give an indication of how the moisture concentration is distributed. In other words, plots like the one shown in Fig. 5 are useful to understand the moisture weight uptake, but they do not provide any information of the moisture distribution within the composite (especially if the Fickian saturation is not reached).

To accurately depict the diffusion behaviour of a sample, a tapered laminate made of the same IM7/8552 material was investigated. The stacking sequence of the thick section was $[0_2, (90, 0)_4]_S$ while at the thin section it was $[0, (90, 0)_4]_S$, *i.e.* the laminate contained two 0^o ply terminations. The ply-drop region was polished and scanned under an optical microscope and is shown in Fig. 12. The features of the cross-section, including interlaminar regions, resin pockets, ply interfaces and ply terminations were traced out using a CAD software. The sketch was then used to create a 3D mesh by extruding the boundaries along the width direction. The mesh could then be imported into Abaqus for analysis, as shown in Fig. 13.

The material properties of IM7/8552 carbon fibre and pure epoxy resin were given as input in the FE analysis. The orthotropic diffusivity was computed by using the values determined experimentally by Choi *et al.* [10]. The saturation values were taken from the gravimetric tests described in previous sections.

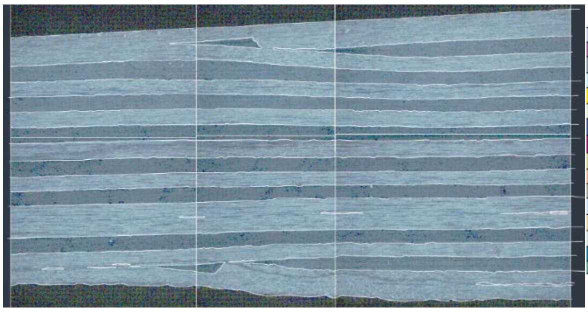

Figure 12: Optical microscope image of the composite ply drop region.

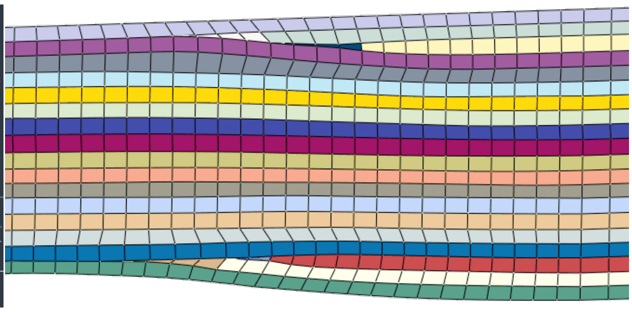

Figure 13: 3D Finite Element mesh for diffusion analysis.

The boundary conditions for the analysis were the weight uptake at all external surfaces, which were time-dependent. The output of the analysis is the nodal mass concentration (in this case a weight uptake) as a function of the simulation time. An example of output at the first desorption cycle is shown in Fig. 14.

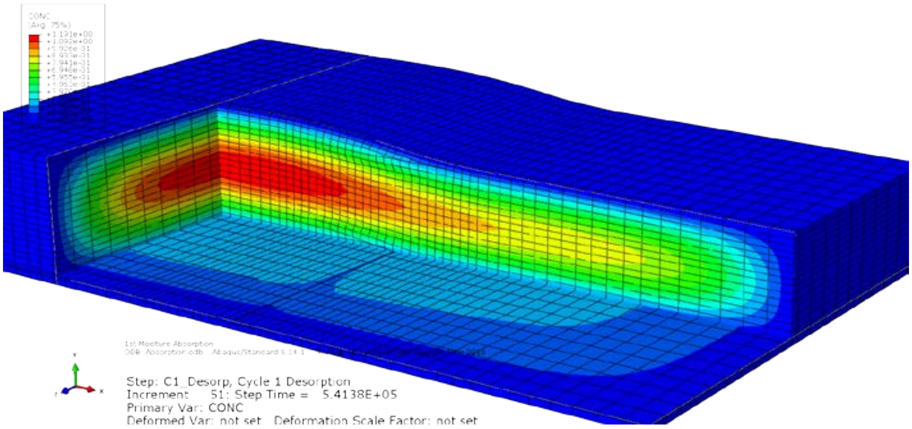

Figure 14: Moisture concentration during the first desorption cycle.

This numerical modelling exercise helped to understand the “preferred paths” of moisture diffusion during the process of absorption and desorption. When looking at the internal regions of the laminate, resin pockets introduced by ply terminations were found to behave as ‘channels’ conducting moisture more rapidly than other regions. Moisture absorption and desorption were observed to always initiate at the edges of the composite regardless of the history of the material (the edge is closer to the applied boundary conditions). When the material is fully saturated after the absorption process and desorption starts, moisture in the internal portions of the laminate take longer to vanish. When re-absorption was applied before all the moisture content was lost, saturation was reached earlier as less time was required to saturate the specimen.

5.2 Micromechanical model

A shown in Fig. 15, a micro-mechanical mass diffusion model was developed as an attempt to estimate orthotropic diffusivity values for the composite material using a representative volume element (RVE). This model is currently being used to estimate the effective diffusivity coefficients along the three orthogonal material directions based on weight uptake measurements.

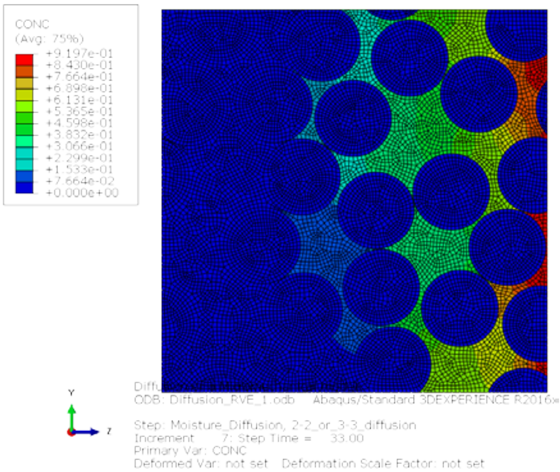

Figure 15: Directional diffusion via a micro-mechanical model.

6 CONCLUSIONS

Samples of IM7/8552 carbon fibre epoxy composites were manufactured, subjected to hygrothermal aging, and tested for changes in mechanical properties and microstructure. The moisture absorption / desorption behaviour was characterised by gravimetric testing. Vibration testing (natural frequency sweep) and tensile testing were conducted to quantify the changes in mechanical properties. The effects of hygrothermal aging on the polymer matrix were validated by differential scanning calorimetry (DSC) and examined using scanning electron microscopy (SEM). Lastly, an in-depth study of moisture flux within the composite was conducted via finite element (FE) analysis.

The results indicate that the hygrothermal history has a strong impact on the mechanical performance of aerospace-grade composite laminates, and that the absorption-desorption behaviour in the presence of internal features such as ply terminations and resin pockets can be analysed successfully by detailed ply-level finite element analysis. Further conclusions include:

- With an increasing number of absorption / desorption cycles, an irreversible hygrothermal 'history' is applied to the composite. Gravimetric testing revealed that the Fickian linear behaviour remained unchanged. However, an increase in diffusivity and maximum saturation was observed with increasing numbers of cycles. This suggests that the behaviour of the material is altered due to microstructural changes that affect the material's absorption / desorption behaviour.
- Literature studies suggest that composites subjected to moisture are vulnerable to polymer plasticisation which leads to reductions in strength and stiffness. Therefore, changes in bending stiffness were attempted to be measured via vibration testing. Results did not show statistically significant changes in natural frequencies for the relatively small number of hygrothermal cycles investigated here.
- The ultimate tensile strength and axial stiffness of the composite after hygrothermal aging were obtained by mechanical tensile tests. Validated by statistical analysis, the results revealed that hygrothermal aging did not affect the axial tensile properties of the composite significantly. In hindsight, attention should be paid to the shear and compressive properties of the material instead, since hygrothermal degradation is matrix-dominated, whereas axial tensile properties are mostly fibre-dominated.
- The modulated DSC results agree with theory as the glass transition temperature shifts positively with each increasing hygrothermal cycle. This suggests that the polymer structure changes with respect to the hygrothermal history of the composite.
- SEM revealed several features unique to the aged samples. Firstly, a dark region surrounding every fibre strand was observed in all samples, which was hypothesized to be fibre-matrix

interface degradation. Secondly, at nano-scale magnification, minor fibre-matrix debonding could be observed in samples with high cycles of hygrothermal aging. Lastly, peculiar ‘fractal’ patterns were found within interlaminar regions of the composite. these patterns were found to be topographic features instead of elemental changes, and the further work is required to fully understand the formation of such features.

- Moisture diffusion in tapered laminates was studied by ply-level FE modelling, and moisture maps containing spatio-temporal information were generated. The time to saturation predicted by the model was in good agreement with experiments, suggesting that the methodology is valid for the analysis of more realistic laminates.
- The FE model revealed that resin pockets introduced by ply terminations behave as ‘channels’ that conducts moisture rapidly. This results in high mass flux within the pockets themselves and their proximity. Moisture absorption and desorption was observed to always initiate at the boundary or edges of the composite regardless of the history of the material. An observation was made that the mass flux is driven by the potential between dry and wet regions; therefore, when the concentration of trapped moisture deep within the composite is low, the time required to diffuse the moisture outwards is long.

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