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Accelerated hygrothermal cyclical tests for carbon/epoxy laminates

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The paper deals with the design of reasonable accelerated test conditions to assess polymer matrix composite durability when it is subjected to supersonic flight-cycles. The study is closely linked to novel application of carbon fibre polymer matrix composites in supersonic aircraft primary structures, leading to substantial weight saving and stiffness improvement. A supersonic flight can result in surface temperatures close to 130 °C, inducing severe thermal gradients and drying which are quite new for this type of materials, now used in primary structures of subsonic jets operating at low subsonic flight-temperatures. Therefore, the particular effect of the drying on the long-term behaviour of composites should be investigated. Numerical simulations based on Fick's law confirm that the supersonic flight-cycles induce a material drying on the long-term and a significant moisture uptake occurs during the aircraft maintenance periods. Then, particular accelerated cycles are proposed to approach the effect of the drying and moisture uptake during service life. First experiments showed that the long-term hygrothermal fatigue can induce significant changes in the material properties and a drop in the glass transition temperature of about 20 °C.

Keywords: Supersonic flight-cycles

1. Introduction

Due to their high specific properties, carbon fibre polymer matrix composites are good candidates to design components of primary structures of the future supersonic aircraft. In such particular application, the materials are exposed to harsh environments since the structures are subjected to high amplitude temperature variations between −55 °C—subsonic flight—and +130 °C—supersonic flight, resulting in through-the-thickness thermal gradients and drying of the materials. These conditions are far from those prevailing during subsonic flights where the temperature remains low (−55 °C). Therefore, it is absolutely necessary to approach the in-service behaviour of composite materials under the specific conditions of a supersonic flight. More particularly, we investigate here the effects of the supersonic drying and moisture uptake on the material properties on the long term. As a matter of fact, the supersonic aircraft being subjected to maintenance operations, the aircraft will be periodically grounded for periods of 3 months and therefore it will uptake some moisture during this period of time.

In this current study, the material will be subjected to various hygrothermal environments, which are known to strongly affect the properties of polymer matrix composites [1]. Moisture absorption may induce severe mechanical and physicochemical changes in polymer matrix or fibre/matrix interphase: polymer chains can undergo a reversible plasticization process, which lowers the glass transition temperature, be subjected to irreversible hydrolysis [2,3] and the fibre/matrix interphase can be damaged due to the coupling with internal stresses for instance [4]. The moisture diffusion process is highly dependent on the temperature and relative humidity [5–7].

In this paper, as an introduction we verify that the moisture diffusion process is in general agreement with the classical Fick's law, which will provide the basic spatial–temporal model to compute the moisture concentration fields. Special emphasis is put on the in-service

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Nomenclature

a	(dimensionless) constant depending only on the material
b	(dimensionless) constant depending only on the material
c_∞ (%)	maximum moisture content
$c(t)$ (%)	moisture content at the time t
$c_i(z)$ (%)	initial space-dependent moisture concentration
$c(z,t)$	moisture concentration inside the plate at the time t and point z
D (mm^2/s)	coefficient of diffusion in the thickness direction
D_0 (mm^2/s)	constant
e (mm)	the plate thickness
e_0 (mm)	extend of the fluctuating part
E_a (kcal/mol)	activation energy
R (kcal l/mol K)	constant of perfect gas
t (s)	time
T (K)	material absolute temperature
z	position in the thickness direction
Φ (%)	ambient relative humidity
τ (s)	period of the hygrothermal cycle

material internal state along the succession of high temperature drying and maintenance operations (low-frequency cycles), the specific hygrothermal fatigue due to the flight-cycles (high-frequency cycles) not being considered in this approach. The design of the specimen thickness and the environmental conditions to meet the in-service material internal state in short time are then tackled. Finally, samples are subjected to the accelerated cycles to characterize the material properties and to reveal possible damages induced by the high temperature drying.

2. Position of the problem

A wing component made of Carbon/Epoxy IM7/977-2 quasi-isotropic 30 ply laminate is considered. It will be regarded as an infinite 4 mm thick plane laminate subjected to cyclical conditions of both the temperature and the moisture (Fig. 1). Every flight-cycle includes the pre-flight time of the aircraft on the ground, the takeoff, the flight comprising subsonic and supersonic modes and the landing finally [8]. The flight-cycles have been simplified here by neglecting the low temperatures when landing, which have no significant effect on the water concentration and temperature fields (Fig. 1).

After N successive flight-cycles, the supersonic aircraft would be grounded for a maintenance operation of 3 months (Fig. 2). This 3-month maintenance duration simply results from the industrial experience of the project partners who consider this time as reasonable. During this period of time, moisture uptake occurs at ground conditions. At the end of this 3-month period, the material reaches the so-called *pseudo-humid* state. Then, the aircraft is subjected to a succession of N flight-cycles and it will finally reach the so-called *pseudo-dry* state, before the next maintenance period starts. Hence, the material internal state periodically varies from the *pseudo-humid* to the *pseudo-dry* state and conversely during the whole aircraft service-time between every maintenance stop (Fig. 2). The problem here is to design simple accelerated tests, which can be performed in laboratory conditions, to simulate those low-frequency cycles, the time-period being the time between two maintenance stops, and to study the drying of the material on the long term. The characterization of the hygrothermal fatigue due to the flight-cycles (high-frequency cycles), involving the computation of transient stress gradients, is tackled in an other reference [9]. The question only addressed here is to quantify the concentration gradients occurring in those two basic pseudo-humid and pseudo-dry states. Then, the design of the accelerated tests by reproducing these two internal states in shorter time will be tackled and first data analysed.

3. Moisture diffusion modelling

According to the one-dimensional Fick's law, the distribution of moisture concentration through-the-thickness of an infinite laminate is a function of time t and position z in the thickness direction. It is given by

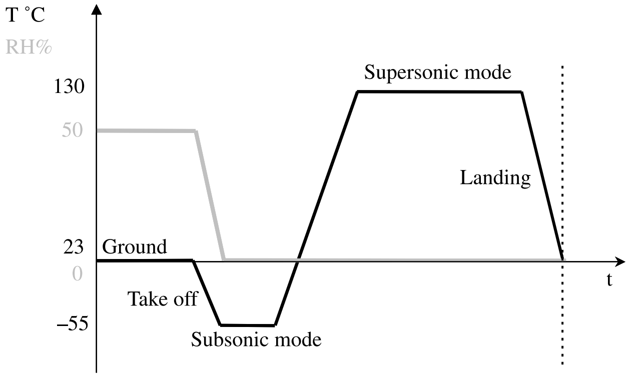

Fig. 1. Supersonic hygrothermal model flight-cycle.

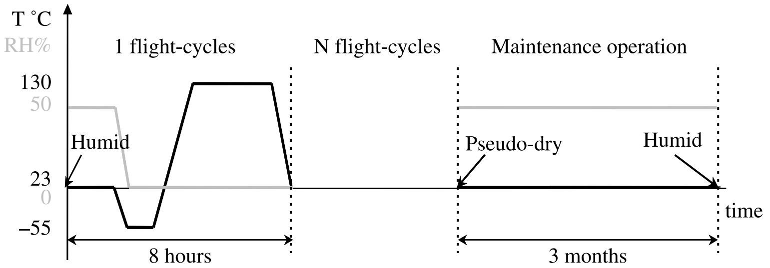

Fig. 2. In-service conditions: flight-cycles and maintenance. Details of the humid and pseudo-dry states.

the following equation, e being the plate thickness:

$$\frac{\partial c(z,t)}{\partial t} = D\frac{\partial^2 c(z,t)}{\partial z^2}, \quad \forall\, 0<z<e \text{ and } t>0;$$
$$c(z,t) = c_i(z), \quad \forall\, 0\leq z\leq e \text{ and } t<0;$$
$$c(z,t) = c_\infty, \quad \forall\, z<0,\ z>e \text{ and } t>0 \qquad (1)$$

The Fick's response of the plate is governed by two parameters: c_∞, the moisture content set at the laminate surface, which is asymptotically and uniformly reached after a long period of exposure at constant temperature and relative humidity, and the coefficient of diffusion D, which controls the diffusion rate through the thickness.

The material is assumed to be homogeneous regarding the moisture diffusion process, meaning that the diffusivity does not depend on space variables and is only temperature-dependent (2). The concentration at the surface depends on the relative humidity at time t only (3) [5]:

$$D = D_0 \exp\left(-\frac{E_\mathrm{a}}{\mathrm{RT}}\right) \qquad (2)$$

$$c_\infty = a\Phi^b \qquad (3)$$

The well-known solution of the one-dimensional Fick's problem is given by relation (4) which determines the water concentration within the material thickness as a function of time, at constant temperature and relative humidity [5]:

$$c(t) = c_\infty \quad 1 - \frac{8}{\pi^2}\sum_{j=0}^{\infty}\frac{1}{(2j+1)^2}\exp \quad -\frac{(2j+1)^2\pi^2 Dt}{e^2}\Bigg)\Bigg) \qquad (4)$$

In order to predict the concentration gradient within the IM7/977-2 quasi-isotropic laminate, the moisture content at saturation c_∞ and the diffusivity D must be determined (4). Moisture uptake tests are performed to control the moisture absorption mechanism following the Fick's law and to measure these parameters in a particular case of interest. The complete test procedure is reported below:

- *Selection of the specimen geometry*. Three test specimens are cut from an IM7/977-2 quasi-isotropic plate laminate of thickness 2 mm. The selected specimen is twice thinner than the original 4 mm thick plate to reduce the conditioning time, proportional to the square of the plate thickness [5]. Moreover, such thin specimen minimizes the water diffusion through the edges and keeps the same ply lay-up. Finally, the specimen dimensions are $200\times 10\times 2$ mm^3.
- *Specimen drying*. Specimens are completely dried in a vacuum oven over a duration of 3 weeks at 130 °C, which is the supersonic flight-temperature. The dry weight of each specimen is controlled with an accuracy of 0.1 mg.
- *Moisture uptake tests*. Specimens are kept at 80 °C and 80% RH and their weights are recorded as a function of time. This special environment has been selected to speed up the moisture uptake process since the kinetic of moisture diffusion is temperature-activated (2). Furthermore, the moisture content at saturation is a growing function of the relative humidity (3). Moisture uptake test duration was about 10 weeks. Moisture content is then plotted as a function of the square root of time (Fig. 3).

The identification method to derive the diffusion parameters is based on the adjustment of the analytical solution of Fick's problem (4) and the average experimental mass variations of each specimen [5,10]. The standard deviation between the analytical solution (4) and the experimental moisture uptake (Fig. 3) is minimized to identify the set of two parameters (c_∞, D) given in Table 1. These identified parameters are in agreement with previous data obtained under other conditions by the project partners, those data obtained at different temperatures and relative humidity lead to the identification of the parameters of Eqs. (2) and (3).

In conclusion, experimental results clearly confirm that the diffusion process within the IM7/977-2 quasi-isotropic laminate is in agreement with the Fick's law. Thus, it will be adopted in the following to estimate the moisture concentration field through the laminate thickness.

4. Hygrothermal supersonic low-frequency cycles

The internal state of the in-service material (Carbon/Epoxy IM7/977-2 quasi-isotropic 30 ply laminate) moves

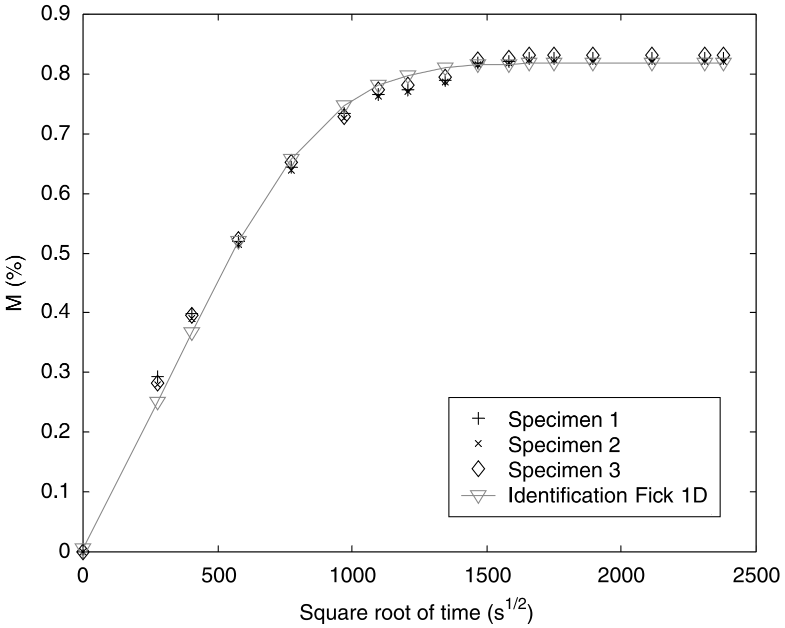

Fig. 3. Experimental and identified moisture uptake as functions of square root of time.

periodically from the *pseudo-humid* state, after every maintenance operation, to the *pseudo-dry* state after N successive flight-cycles and conversely, during the whole aircraft service-time (Fig. 2). The objective of this section is first to characterize these two internal states of the real material.

The finite difference method is used to solve the one-dimensional Fick's equation to derive the moisture gradient within the laminate for every hygrothermal cycle. Relations (2) and (3) are numerically implemented and c_∞ and D are given for any value of temperature and relative humidity.

During the maintenance period, moisture uptake occurs. At the end of the period, the 30 ply laminate reaches the *pseudo-humid*, which will be regarded here as the initial state of the material. The numerical simulations show that this initial state is basically characterized by a non-uniform concentration field, because the maintenance time is too short for the material to reach the saturation state (Fig. 4). Then, the water concentration gradient within the laminate is computed at the end of every cycle after a given number of successive flight-cycles (Fig. 5). We consider that the *pseudo-dry* state of the laminate is achieved when the amount of desorbed water per cycle becomes negligible if the number of cycles increases. In the special case of the study, the *pseudo-dry* state is reached at the 300th cycle (Figs. 4 and 5). The maintenance period results in a moisture uptake whereas the supersonic flight-cycles leads to material drying (Figs. 4 and 6).

The water concentration through-the-thickness obviously presents two distinct regimes (Fig. 5) as shown elsewhere [6,11]: a transient regime inside an internal zone and a fluctuating regime near the surfaces (Fig. 5). The extent of the fluctuating part is characterized by e_0 which depends on the material properties and the hygrothermal cycle. Its value is approximated by the following expression [12,13]:

$$e_0 = 2\sqrt{\pi \int_0^{\tau} D(t)\mathrm{d}t} \tag{5}$$

It is interesting to emphasize the fact that the initial water concentration, corresponding to the *pseudo-humid* state, is not uniform over the laminate thickness and it may induce an *apparent* departure from Fick usual water mass versus square root of time evolution although the phenomenon is basically of Fickian nature (Fig. 6).

In conclusion, straightforward numerical simulations based on classical Fick's law prove that the succession of in-service supersonic flight-cycles induces a material drying on the long-term. The numerical simulation of the concentration gradients over the laminate thickness leads to the characterization of the two key in-service material states: the *pseudo-humid* and *pseudo-dry* states. Therefore, it is now possible to design accelerated hygrothermal cycles simulating those long time periodical changes of the material moisture content to study possible effects of wet-drying cycles on that type of structural materials.

Table 1
IM7/977-2 identified hygroscopic properties at 80 °C and 80% RH

Material	D (mm^2/s)	C_∞ (%)
IM7/977-2	9.64×10^{-7}	0.82

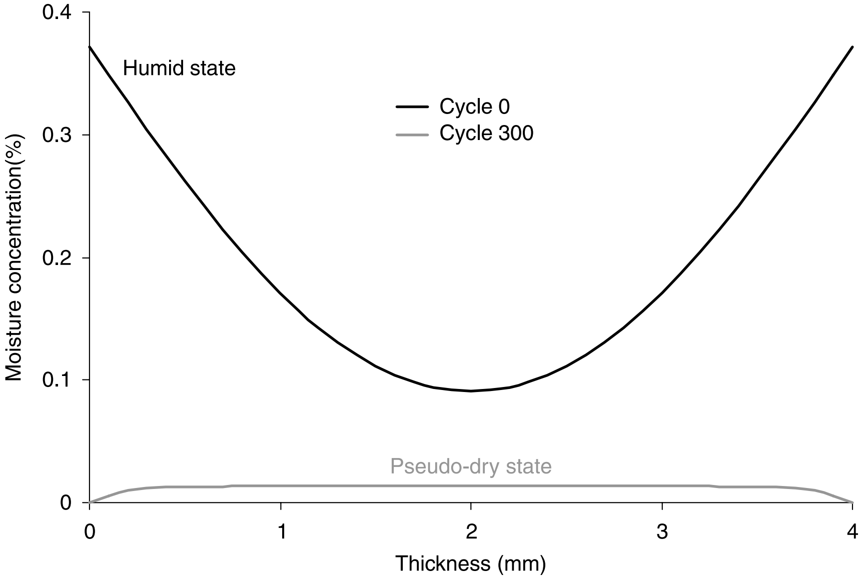

Fig. 4. Humid and pseudo-dry states induced by the in-service supersonic conditions.

5. Accelerated hygrothermal cycles

5.1. Design of the accelerated cycles

The challenge in this section is to design reasonable and suitable accelerated hygrothermal cycles to be carried out in laboratory. The supersonic aircraft has an expected service lifetime of 80,000 h, it is therefore unrealistic to expect to approach the environmental response of the material in real time. So, accelerated tests suited to the particular in-service conditions are necessary and detailed below.

5.1.1. Simplification of the in-service conditions

The hygrothermal conditions undergone by the aircraft can be summarized as follows: the aircraft is basically subjected to the *low-frequency cycles*, each one including a maintenance operation followed by N supersonic flight-cycles, so-called *high-frequency cycles*. Thus, during *low-frequency cycles* the material internal state moves from the *pseudo-humid* to the *pseudo-dry* state periodically. One way to simplify the experimental conditions is first to consider every *low-frequency* cycle as comprising two successive steps of moisture uptake and drying. Now, the objective is to achieve the in-service *pseudo-humid* and *pseudo-dry* basic

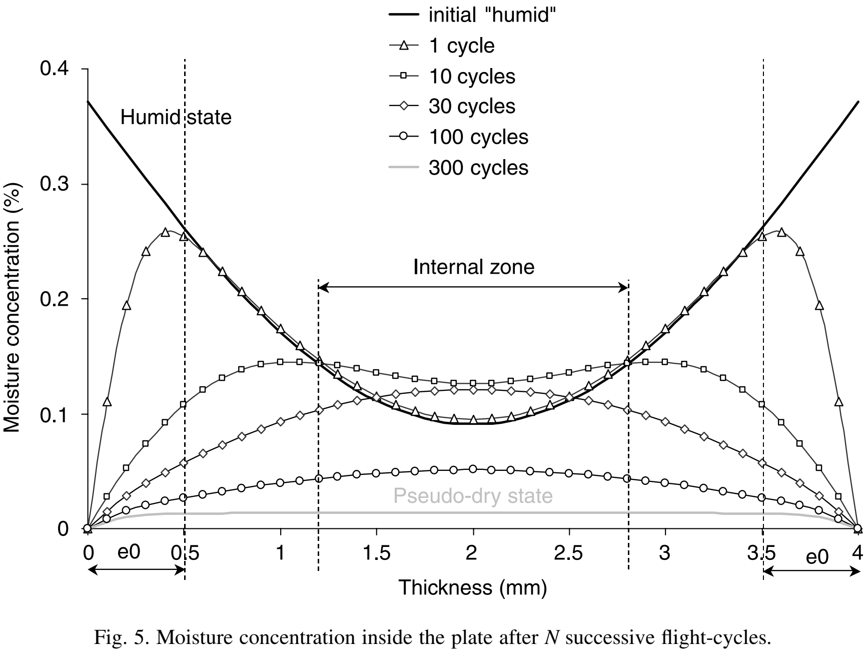

Fig. 5. Moisture concentration inside the plate after N successive flight-cycles.

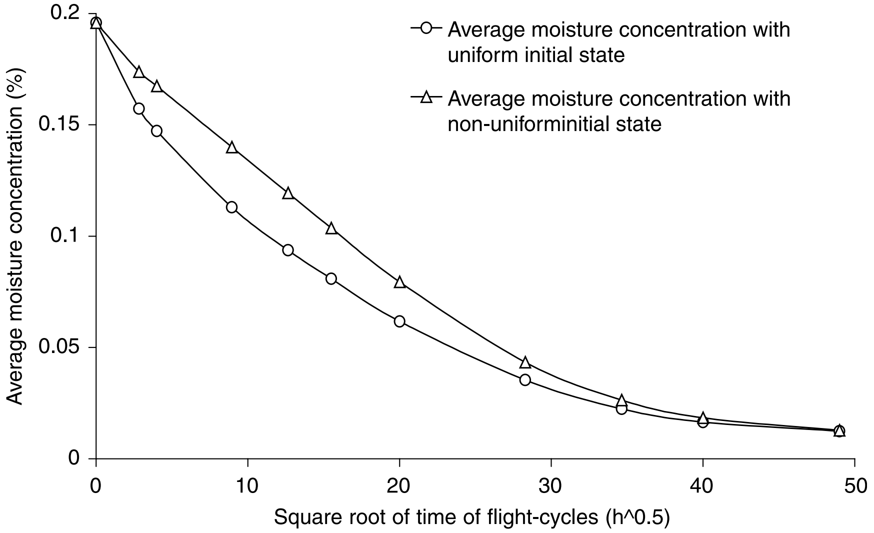

Fig. 6. Average moisture concentration as a function of square root of time during the supersonic drying with an uniform and a non-uniform hygroscopic initial state.

states, characterized by concentration gradients, in a time suitable for tests to be carried out in laboratory. The current approach focusing on the specific effect of the drying has been decided to disregard the high-frequency cycles here, although those cycles induce stress time-dependent gradients in the vicinity of the outer laminate surfaces, and only consider constant by piece temperature and relative humidity evolutions. Effects of high-frequency cycles have been modelled in previous papers [9].

5.1.2. Reproducing the in-service basic states in a shorter time

In this section, we keep the original laminate thickness constant (4 mm). Assuming that the supersonic flight-cycles can be approximated by successive drying and moisture uptake steps (Figs. 4 and 6), we first apply a constant conditioning temperature of 130 °C, the material being initially in the *pseudo-humid* state, to dry the sample and achieve the *pseudo-dry* state, as it would be if subjected to 300 successive flight-cycles. We can easily estimate the time necessary to reach the *pseudo-dry* state. The drying temperature has been set at 130 °C because it is the real in-service temperature. In that case, numerical simulations show that the time necessary to dry the material in the same way as the flight-cycles has been reduced by 3, 5 for the original 4 mm thick laminate. We could perform a stronger drying by applying temperatures higher than 130 °C, but such temperatures will not have a clear meaning regarding the in-service conditions; moreover they might activate some degradation phenomena, such as thermo-oxidation [14], which are not significantly active under the present flight-cycles.

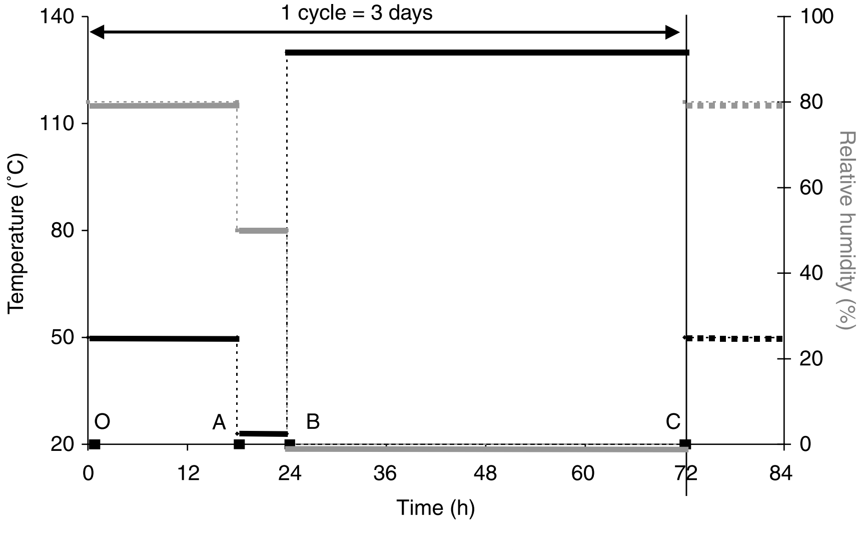

Fig. 7. Accelerated hygrothermal cycles for a 1-mm thick plate.

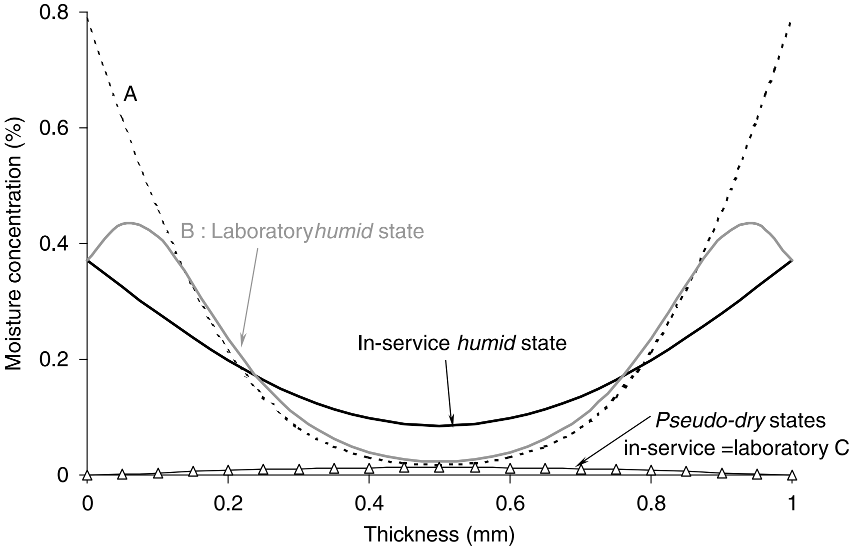

Fig. 8. Comparison of the pseudo-humid and pseudo-dry states induced by the in-service conditions and accelerated cycles.

In order to accelerate the moisture uptake induced by the maintenance, it is necessary to change the environmental conditions. As a matter of fact, if we do not take care, the new *pseudo-humid* state will not necessarily have a similar moisture concentration profile as the in-service one. The objective is to design accelerated cycles in such a way that the induced *pseudo-humid* and *pseudo-dry* states have the same moisture concentration gradients, at least the same average concentration, as those generated by the in-service conditions. Those particular accelerated cycles will approach the real evolutions in a closer way than usual, where conditions are mainly dictated by time saving only.

5.1.3. Approximating the in-service states

Usually, the conditioning time can be reduced by increasing the temperature (2) and relative humidity (3) as well as by reducing the sample thickness [5]. Since the emphasis is put on the desorption and absorption process, the time has been reduced by modifying the environmental conditions together with reducing the specimen thickness [15]. Numerical simulations have been performed to select reasonable accelerated conditions such as the resulting *humid* and *pseudo-dry* states have the same concentration gradients and average moisture concentration as those induced by the in-service conditions.

We first choose to determine accelerated cycles for 1 mm thick specimens. In fact, the conditioning time being proportional to the square of the laminate thickness, the duration associated with the 1 mm thick plate should be 16 times faster than the one associated with the original 4 mm plate, without changing anything else. The procedure to accelerate the moisture uptake and drying for the 1 mm thick specimens (Fig. 7) is detailed below:

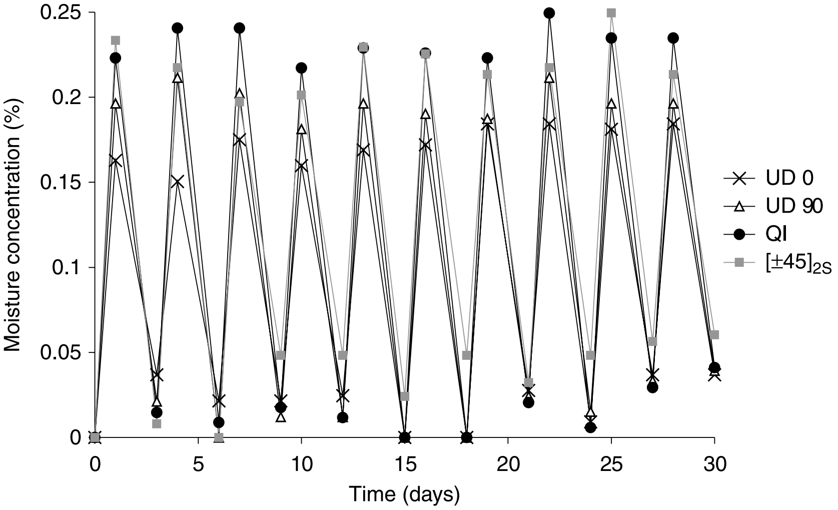

Fig. 9. Moisture uptake and drying during the accelerated cycles for different stacking sequences specimens.

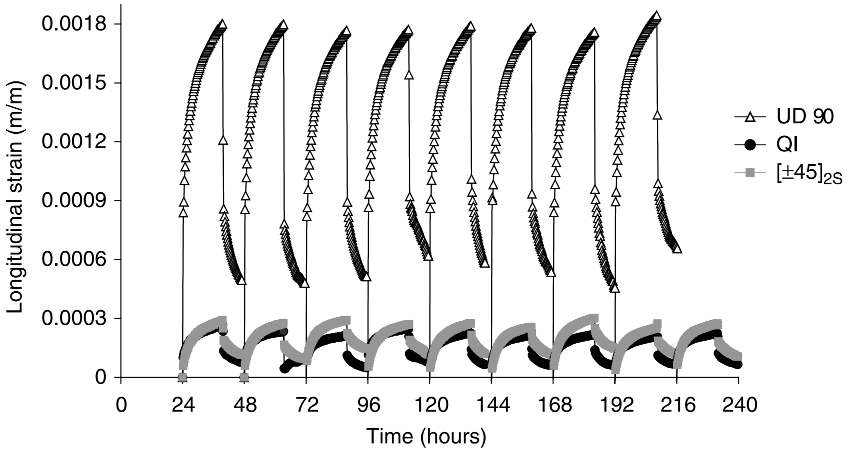

Fig. 10. Measurement of the longitudinal hygrothermal strain during moisture uptake step of accelerated cycles for different stacking sequences specimens.

- *Moisture uptake step*. The plate is first exposed to 50 °C and 80% RH during 18 h (point A in Fig. 7), then to 23 °C and 50% RH during 6 h (point B in Fig. 7). According to Fig. 8, the material releases moisture mainly close to the surfaces during this period. The laboratory *pseudo-humid* state has the same average moisture concentration as the in-service one and a close concentration gradient (Fig. 8).
- *Drying step*. As explained before, the supersonic cyclical drying along the 300 flight-cycles will be approximated by an isotherm drying at 130 °C. The laboratory *pseudo-dry* state is reached at point C (Fig. 7) after 48 h of conditioning time, the specimen achieved a similar concentration gradient as in the in-service state (overlapped curves in Fig. 8).

In conclusion, the previous laboratory cycle, applied to 1 mm thick specimens, is 60 times faster than the in-service conditions acting upon the 4 mm thick plate and the moisture concentration gradients of *pseudo-humid* and *pseudo-dry* material states have been completed.

5.2. Experiments and results

The objective is first to characterize some basic material key-properties and analyse the effects of the low-frequency hygrothermal fatigue upon those properties. The accelerated cycles (Fig. 7) are repeated 10 times to reproduce the number of maintenance operations seen by the aircraft during its whole service-time. Different stacking sequences specimens have been tested: unidirectional $[0]_8$, $[90]_8$, angle-ply $[\pm 45]_{2S}$ and quasi-isotropic QI.

Specimens are first completely dried in a vacuum oven during 3 weeks at 130 °C, then weighed at each *humid* and *pseudo-dry* state, according to the accelerated cycles (Fig. 9). Fig. 9 shows moisture uptake and desorption during accelerated cycles for the various ply layups. If we focus on the *pseudo-dry* state, we note that moisture content increases mainly for the last cycles. This phenomenon is due to the gradual evolution of the matrix properties. Moreover, this figure reveals that the ply layups have a significant effect on the diffusion characteristics. The unidirectional specimens tend to absorb less moisture than the cross-plies during the same period of time. The change of the moisture concentration with time is linked to the ply lay up and specimen geometry. Let us only focus on the difference between UD and QI, since the fibre volume fraction is constant, the difference is mainly due to the stacking sequence. For UD laminates, the manufacturing residual stresses are equal to zero at the ply scale; however, in the case of QI or cross-ply laminates the plies sustain a tension transverse to the ply fibres below the curing temperature. Therefore, we can understand that having a larger space, moisture diffusion and final saturation concentration can reach higher values for QI laminates. This first qualitative

Table 2
Tensile tests before and after the hygrothermal fatigue associated with the accelerated cycles for different stacking sequences specimens

	UD 0		UD 90		QI		[±45]	
	Before	After	Before	After	Before	After	Before	After
E (GPa)	175	180	8.5	9	57.5	55.4	17.5	16
Ultimate strength (GPa)	1.9	1.7	0.03	0.06	0.8	0.8	0.19	0.2
Ultimate strain (%)	1.1	1	0.4	0.7	1.4	1.4	4.8	9

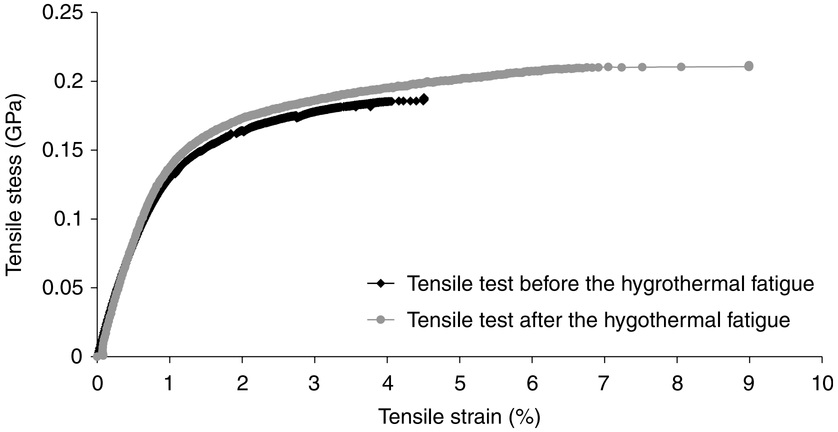

Fig. 11. Tensile stress–strain curves for pseudo-dry $[\pm 45]_{2S}$ specimens tested before and after the hygrothermal fatigue.

approach should be strengthened by a refined analysis in the future.

The longitudinal strain due to the hygrothermal conditioning is measured along the cycles by using unidirectional gauges bonded on the specimens (Fig. 10). Only the strain due to the first part of the accelerated cycles O, A, B (Fig. 7) are reported in Fig. 10. The evolutions of the strains result from both temperature and relative humidity changes. The gradient of concentration depends on time; finally the strains show time-dependent variation, which should not be confused with any viscoelastic effect. These cycles will be further analysed to detect any evolution of coefficients of thermal and moisture expansions induced by chemical or physical changes of the polymer matrix.

Tensile tests are carried out on three specimens of each stacking sequence at room temperature, before and after the hygrothermal fatigue. Specimens are at the same *pseudo-dry* state before and after the tests. Mechanical properties shown in Table 2 are average values of three tested specimens. It is clear in Fig. 11 that the toughness is increased after the hygrothermal fatigue for the $[\pm 45]_{2S}$ specimens and the ultimate strain is approximately twice larger after the fatigue test. According to Fig. 12, ultimate strength and strain for $[90]_8$ specimens are twice larger after the fatigue test and the toughness is increased as well. The hygrothermal fatigue seems to be beneficial for the material since it enhances the toughness of $[\pm 45]_{2S}$ and $[90]_8$ specimens. This phenomenon is due to the strong plasticising of the matrix behaviour. In order to strengthen these results, T_g the glass transition temperature is measured by DSC before and after the hygrothermal fatigue. We note that the decrease in T_g is approximately 20 °C after the fatigue test that reveals a significant effect of the moisture.

6. Conclusion

In the present paper, a methodology is proposed to design accelerated hygrothermal cycles representative of the supersonic flight-cycles. The new point in this study is

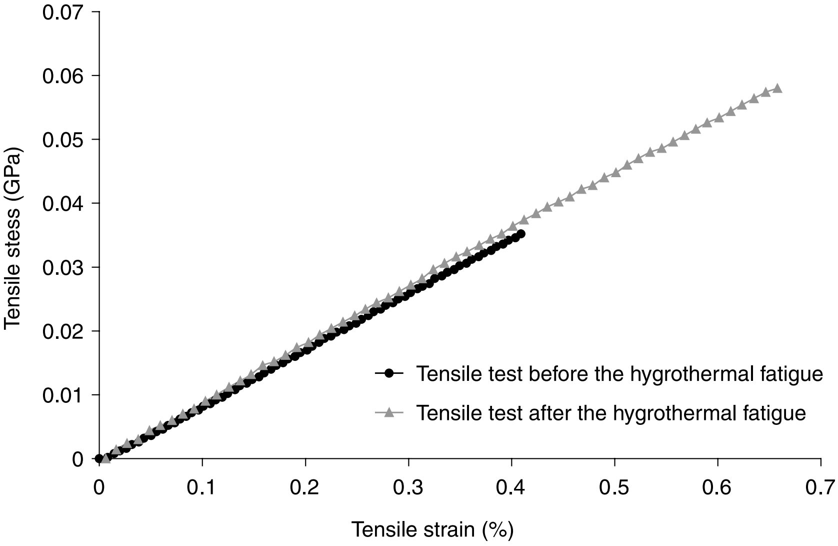

Fig. 12. Tensile stress–strain curves for pseudo-dry $[90]_8$ specimens tested before and after the hygrothermal fatigue.

the investigation of the effect of the high flight-temperature of 130 °C on the long-term material behaviour. According to the in-service conditions, two characteristic material states have been identified (the *pseudo-humid* and the *pseudo-dry* states) by using the Fick's law, previously verified. The principle used to design accelerated cycles is to make moisture uptake and drying faster than the in-service conditions in such a way that typical concentration gradients remain similar. This is done by defining specimen geometry and tuning the conditioning parameters. Thus, accelerated cycles 60 times faster than the in-service ones have been designed.

These accelerated cycles are applied in the laboratory on different stacking sequences specimens. We notice that the hygrothermal fatigue increases the toughness for $[90]_8$ and $[\pm 45]_{2S}$ specimens and causes an evolution of the matrix properties since T_g decreases of approximately 20 °C.

But still there are some differences between accelerated and in-service cycles, since during the supersonic drying, the material goes by negative temperatures during every flight-cycle. These temperatures are not taken into account in the accelerated cycles we defined. Thus, one next step would be to define new accelerated cycles by using the same methodology and introducing these negative temperatures into new accelerated cycles.

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