ON THE EFFECT OF THERMAL AGEING IN CFRP MECHANICAL PROPERTIES

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Abstract

Highly Accelerated Life Testing (HALT) of any product consist in applying several types of loads for accelerated reliability analysis in order to find weaknesses during both design and development stages. Different environmental stress conditions like high rate thermal cycling and vibration are generally introduced during HALT.

In this work, thermal cycles have been performed between -50 and 150°C for aeronautic composite materials in non-reactive and oxidative atmospheres. The thermal fatigue tests applied to polymer fibre reinforced composites involve matrix oxidation and cracking due to the mismatch of the thermal expansion coefficient between the carbon fibres and the epoxy matrix.

In order to correlate the composite degradation with mechanical properties, fatigue and quasi-static tensile tests have been carried out in reference and post-ageing conditions. Microstructural characterization using X-ray computed tomography and light optical microscopy have been performed as well for a better understanding on damage mechanisms. Combined effect of thermal cycling and mechanical load tests on CFRP structural parts (T profile) were also performed.

1. Introduction

HALT methodology is aimed to improve the products reliability during its service life. It is intended to be applied during the development of prototype phases. The main objective is the detection of flaws and weaknesses coming from the design and manufacturing processes. It is based on the application of different types of stresses, such as thermal cycles, mechanical loading and humidity cycles [1]. Although this methodology is commonly applied to electronic devices, it can also be used to study the degradation and combined effect of stress in CFRP mechanical properties.

In the last decades there has been a higher interest in carbon fibre composites materials, mainly in aerospace industry, due to its low weight and considerable improved mechanical properties against metals. The use of composites in aerospace structures is one of the keys in the design of new aircrafts, where weight saving is essential. In this way, the environmental ageing and its effect on CFRPs mechanical properties is a matter of concern for the aeronautic industry.

In this work, the effects of thermal cycles applied to carbon fibre-epoxy composites in different atmospheres are investigated. The combination of thermal oxidation at high temperature [2] and thermal fatigue, due to mismatch of the thermal expansion coefficient between the carbon fibres and the epoxy matrix, involves the material degradation [3-6]. Loss of mechanical properties is analysed by means of quasi-static tensile and long life fatigue tests, both performed in a reference and post-ageing status. Matrix cracking development is also studied during the ageing process.

Finally, HALT methodology is also applied to CFRP structural parts (T stiffeners) by the application of thermal cycling and static mechanical load simultaneously. Effects are evaluated by means of static mechanical test up to failure, and post-ageing tomographic inspections.

2. Experimental

2.1. Materials

AS4/8552 pre-preg composite material is commonly used in aeronautics structures, as well as widely analysed in different investigations. For this study, the following stacking sequences were selected: $[0_3/90_3]_s$ for samples exposed to thermal cycling, and $[+45/-45/0/90]_{2S}$ for specimens tested under the combination of thermal and mechanical loading (T stiffeners). Curing conditions for both types of laminated were 3 hours long cycle at 180°C, with a 7 bar pressure. Additionally, a drying cycle, at 80 °C, 200 mbar and 22 hours stabilization was applied before and after ageing tests in order to homogenize humidity conditions for all of the samples.

Three different specimens dimensions were considered for this study: 25 x 35 mm (type M), 160 x 12 mm (type F) and 180 x 18 mm (type T), for Light Optical Microscopy (LOM) & x-ray inspections, tensile-tensile fatigue and static tensile tests, respectively. The direction of the 0° fibres was aligned to the largest dimension of the specimens, with exception of type M samples (there were machined parallel to the shortest dimension). All samples were extracted from panel of 1,050 x 850 mm and 2.21 mm thickness.

T stiffener specimens were machined as shown in Figure 1. The nominal panel thickness was 3 mm, and the 0° fibres are parallel to the largest dimension.

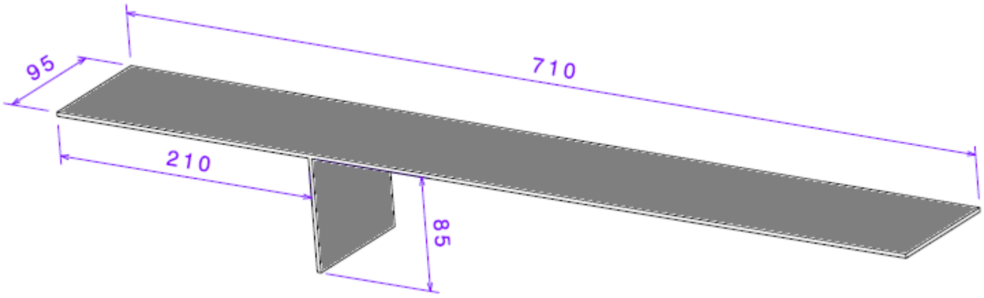

Figure 1. CAD 3D model of T stiffener part with dimensions [mm].

2.2. Microstructural characterization

LOM inspections were carried out by means of an Olympus BX41RF-LED microscope for microstructural crack characterization.
3D characterization has been performed by means of a“VJT-225 µ-CT” X-ray computed tomography (XCT) system manufactured by VJ Technologies. XCT investigations were based in characterizing the internal material structure, defects/inclusions, and crack evolution. The selected inspection parameters are listed in Table 1. In addition, a dye penetrant, based on zinc iodide solution, was used to enhance cracks and delamination detectability [7].

Parameters	Value
Voltage (kV)	60
Amperage (µA)	400
Voxel resolution (µm)	17.1

Table 1. X-ray CT inspection parameters.

2.3. Mechanical testing

Loss of mechanical properties is studied by means of three different types of tests, performed before and after environmental ageing. First of all, tensile properties are determined by standard tensile test, force controlled with a selected speed of test of 2 mm/min until failure of the specimens. Young´s modulus was evaluated using an extensometer. Tests were performed using universal testing machine Zwick Z100.

In order to investigate thermal cycling effects in CFRPs fatigue properties, tensile-tensile fatigue tests were carried out. In this case, the equipment selected was a hydraulic testing machine MTS-810. All tests have been performed at maximum stress 800 MPa, stress ratio 0.1, cycling frequency of 7 Hz, sine wave form and force controlled until failure of the specimens.

T stiffeners were mechanically tested in a universal testing machine Instron 5866. A view of specimen in testing rig is provided in Figure 2a. Force and displacement (for control issues) were recorded in each test. Strain measurements were monitored near to the joint (by means of a strain gauge), where maximum deformation records are experimented (Figure 2b).

In order to get more accurate displacements, Digital image Correlation (DIC) measures were performed by PONTOS-5M system (GOM GmbH).

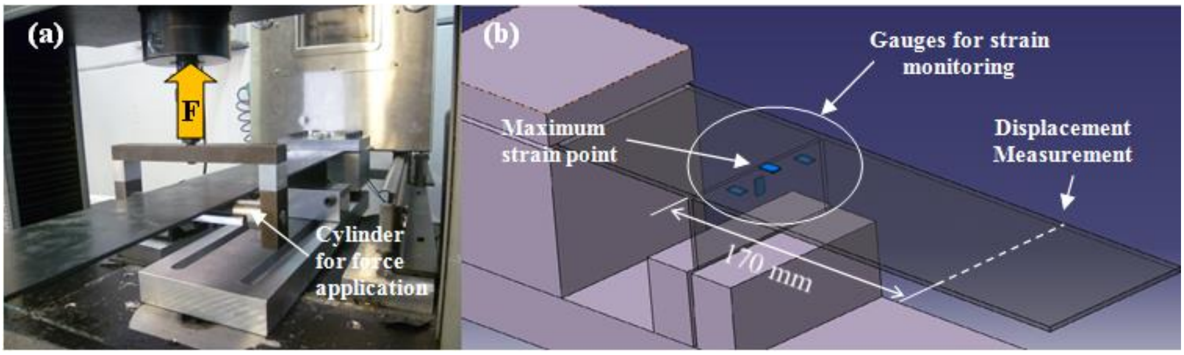

Figure 2. View of T stiffener (a) on testing rig; and (b) location strain gauge for test monitoring.

2.4. Ageing

500 thermal cycles were applied to investigate thermal fatigue effects. Ageing process was stopped at 100, 300 and 500 cycles for samples characterization. This test is performed under two thermal/ambient conditions (Table 2): (i) *Inert*: N_2 atmosphere; and (ii) *Oxidative*: dry air. The high and low temperatures were set at 150°C and -50°C respectively for both conditions. Figure 3a shows the specially designed chamber for controlling atmosphere composition, located inside HALT system (AngelAntoni UHS1400).

A T stiffener was aged by the combination of 140 thermal cycles between -50 and 150°C, heating/cooling rate of 15°C/min, 5 min dwell time, in no controlled atmosphere. Mechanical load by 7.4 kg weight was applied at 170 mm from the T joint (Figure 3b). Maximum strain of 2,732 µε was measured at gauge position (theoretical max. strain region).

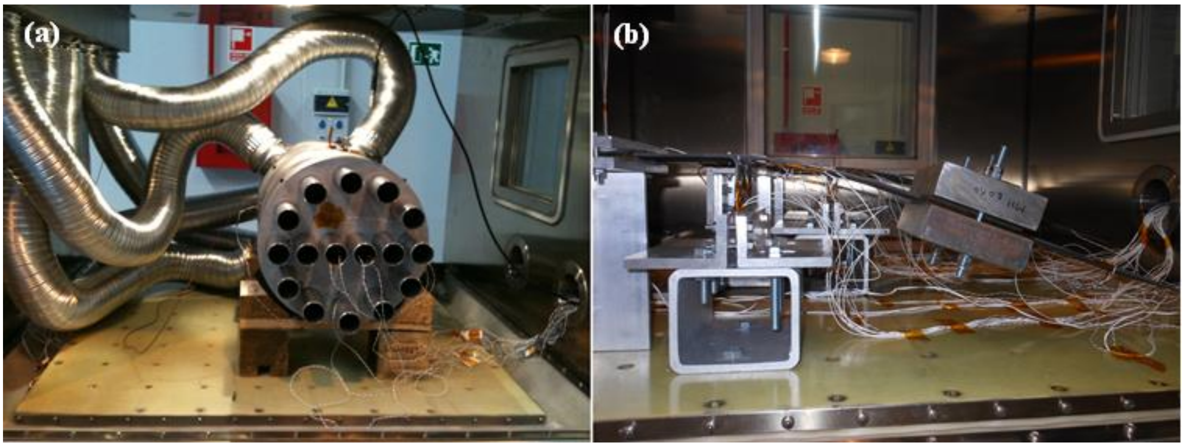

Figure 3. Environmental testing tools for (a) thermal cycles and (b) thermal and mechanical stresses application.

Atmosphere	Gas composition	Min/Max Temperature [°C]	Ramp [°C/min]	Dwell Time [min]
		Sample level		
Inert	Nitrogen	-50/150	15	5
Oxidative	Dry air	-50/150	15	5
		Structural part		
Inert	Free (N_2/air mixture)	-50/150	15	5

Table 2. Selected parameters of the thermal cycling testing conditions.

3. Results & Discussion

3.1. Tomography & LOM

LOM inspections with information about microstructural degradation during thermal cycling are presented in Figure 4. Matrix cracking development due to thermal fatigue in oxidative atmosphere is clearly observable. The damage mechanism is similar to the one observed by mechanical fatigue [8]. Specimen failure is not reached after the application of thermal stresses.

Samples exposed to 100 thermal cycles show transverse cracks on internal layers. In addition, after 300 thermal cycles, the apparition of cracks parallel to external layers (delamination initiation) is observed. Inspections carried out on samples exposed to 500 thermal cycles show the apparition of new cracks and delamination growth. Although the matrix cracking could also be observed on samples aged in inert atmospheres, delaminations were only detected in specimens exposed to oxidative conditions.

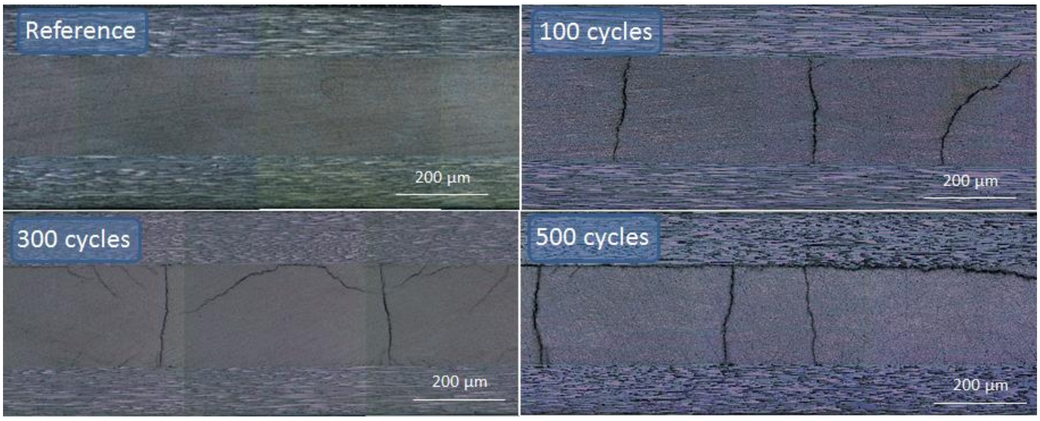

Figure 4. Sample microstructure with matrix cracking development and delaminations (after 500 thermal cycles) during thermal fatigue in oxidative atmosphere (-50/+150°C).

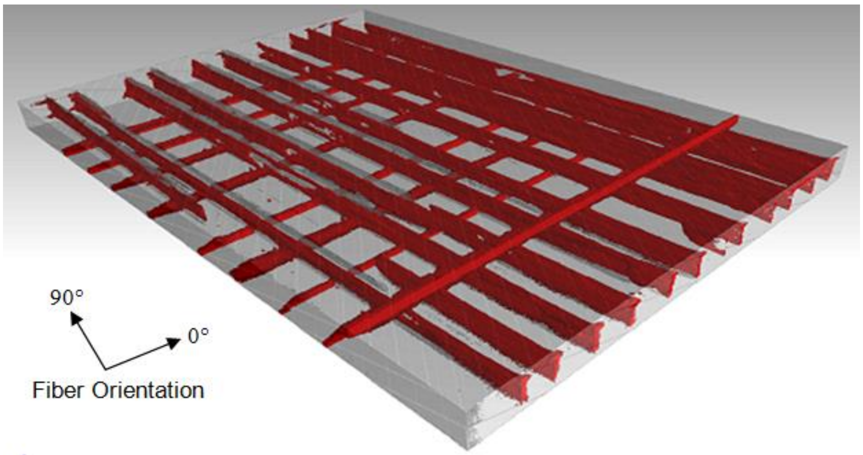

Figure 5. XCT view with three dimensional representation of specimen subjected to 400 thermal cycles (-50/+150°C) at inert atmosphere.

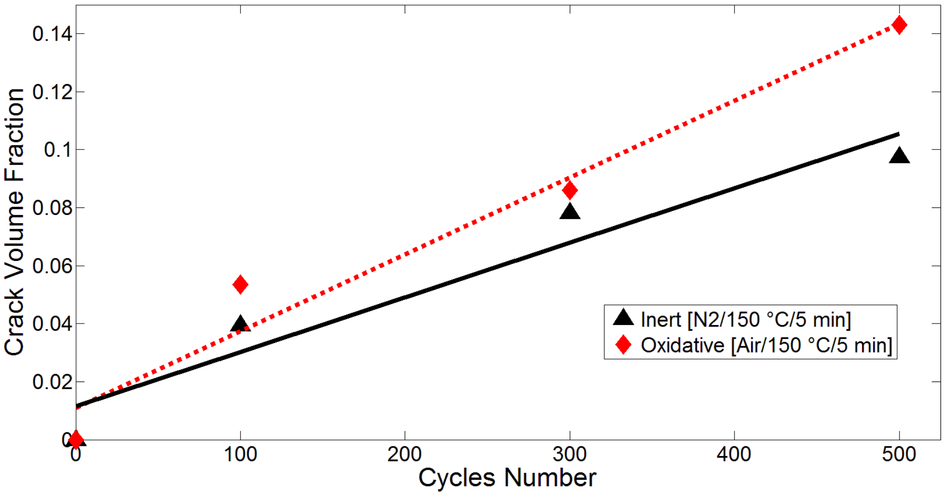

Figure 6. Crack volume fraction evolution against thermal cycling for insert and oxidative atmospheres.

Alternatively, Figure 5 shows inspection example by computed tomography on specimen exposed to 400 thermal cycles in inert atmosphere. Cracks in both orientations (parallel to 0° and 90° layers) are observed. Most of them are fully developed at this ageing stage. From the tomographic view, crack volume fraction can be estimated as crack to sample volume ratio. The evolution of the last, for the different number of cycles applied, is presented in Figure 6 for both inert and oxidative atmosphere. Samples exposed to inert condition exhibit in general lower crack densities. Maximum crack fractions are observed after 500 cycles applied, amounting 0.14 and 0.10 for oxidative and inert atmospheres.

3.2. Mechanical properties

The stiffness of the investigated materials, through Young´s modulus modification, has been studied for both inert and oxidative atmosphere (Figure 7). Related with the microstructural issues reported previously, a slight reduction of the E modulus is observed. After 500 thermal cycles, the Young´s modulus decreases from 71.5 GPa (un-aged condition) to 67.8 and 65.4 GPa (5.2 and 8.5%) for inert and oxidative atmosphere, respectively. It can be noticed that Young´s modulus decreases suddenly after 100 thermal cycles (about -4.5 % less than not aged samples). This issue is observed for both aging atmospheres and mainly related to material damage produced by mismatch of the coefficient thermal expansion of the matrix and fibres.

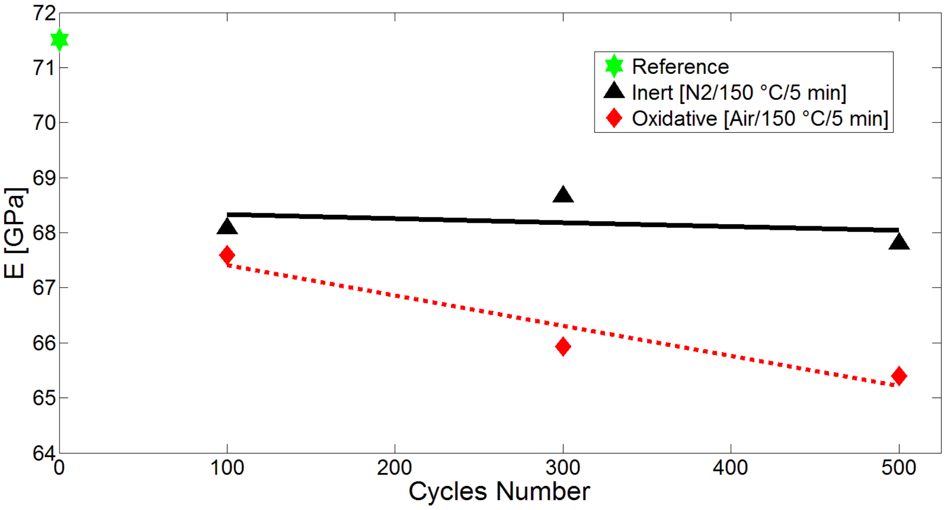

Figure 7. Loss of stiffness against thermal cycles at inert and oxidative atmosphere.

The fatigue behaviour of the investigated materials is presented in Figure 8, where the number of cycles to failure at different ageing conditions is evaluated. A similar behaviour as described previously in static regime is observed: samples tested after 500 thermal cycles and in oxidative atmosphere present an important variation of their fatigue life. Only $2 \cdot 10^5$ cycles are registered while in the reference condition (before ageing) the number of cycles to specimen failure is even larger than $1.5 \cdot 10^6$ (almost one order of magnitude larger). On the other hand, no significant variations are observed on samples after 500 thermal cycles under inert atmosphere.

The summary of the mechanical behaviour of T specimen at the reference state and aged condition is depicted in Table 3. A reduction of maximum load by 31% is recorded after thermal cycling of the specimen (from 291.1 to 200.5 N). The same effect can be observed for maximum strain and displacement, with reductions in max. strain and displacement from 0.761 to 0.526% and 54.01 to 36.69 mm, respectively.

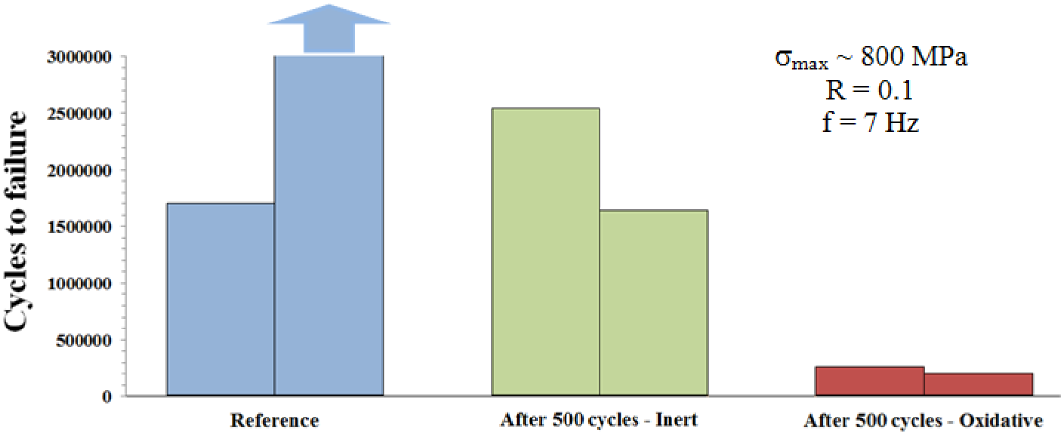

Figure 8. Number of cycles to failure for different aging conditions.

Status	Max. Force (N)	Max. Strain (%)	Max. Displacement (mm)
Reference	291.1	0.761	54.01
Aged	200.5	0.526	35.69

Table 3. T profile mechanical tests results before and after ageing.

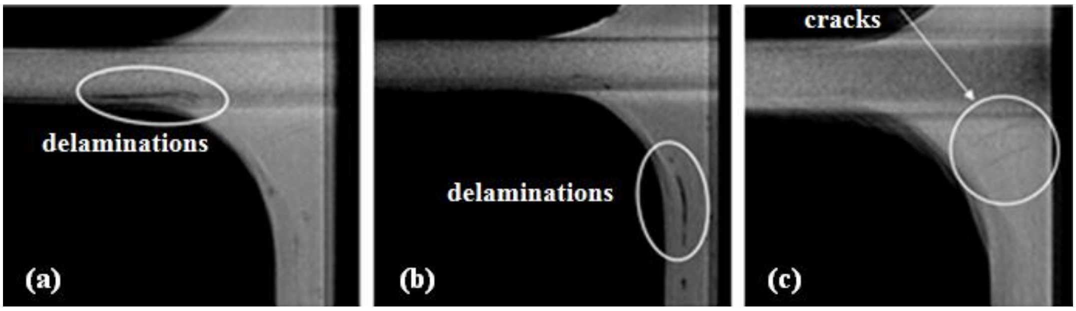

Figure 9. X CT cross section of T profile after HALT testing: View of (a),(b) delaminations and (c) cracks. Large pores are even observable in (b).

Figure 9 depicts tomographic cross sections on T profiles in the aged condition. It can be clearly noticed the appearance of delamination and cracks after thermal cycling and mechanical loading (7.4 *kg*, see section 2.4). These defects together with matrix degradation could be responsible of the lower mechanical behavior of the specimen.

4. Conclusions

Crack appearance, quantification and effect of defects on mechanical properties have been evaluated after thermal cycling.

A large number of cracks are developed in the investigated material after 500 thermal cycles in both inert and oxidative atmospheres (with volumes fractions up to 0.14). For the last one, the crack volume density is increased by 40% due to the presence of oxygen. Thermal cycling under oxidative conditions involves greater damage in CFRPs due to the combination of thermal fatigue and matrix oxidation.

The stiffness of investigated samples is slightly affected during thermal aging and related to crack evolution. This effect is more important in oxidative atmosphere, where degradation of CFRP matrix is also observed [6]. As summary, the E modulus decreases from reference condition (before aging) around 9% and 5% for specimens after 500 cycles under oxidative and inert atmospheres, respectively. It should be notice the effect of CTE mismatch, responsible of a reduction by 4.5% of the Young's modulus at the early stages of thermal cycling.

Regarding fatigue behaviour, after 500 thermal cycles under oxidative conditions number of cycles to failure decreases about 90% from the reference status. Contrary, fatigue life is not hardly affect under inert atmosphere. Presumably, matrix oxidation has an important role on this behaviour (not observed at inert condition [6]).

Finally, HALT tests applied on T stiffeners showed that internal cracks and delamination can appear after 140 thermal cycles at non-controlled atmosphere (N_2/air). These internal damages involve loss of mechanical strength, up to 31% against the reference status. This testing methodology could be useful for weaknesses detection during design and manufacturing stages of CFRP components.

5. Acknowledgements

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