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A comparative study of the fatigue and post-fatigue behavior of carbon–glass/epoxy hybrid RTM and hand lay-up composites

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Abstract The paper presents a study of the fatigue and post-fatigue behavior of a hybrid carbon–glass biaxial fabric reinforced epoxy composite manufactured by the resin transfer molding (RTM) and the hand lay-up (HL) processes, with the main objective of assessing whether a material characterization run at the prototype level of a handicraft technology could be significant for a mass production technology and whether a comparison on static properties (a viable task at an industrial level) could ensure the same level of agreement for the fatigue life and residual properties. Tensile and flexural static tests as well as displacement-controlled bending fatigue tests (*R* ratio of 0.10) were conducted on two sets of standard specimens, having fiber orientation parallel to the loading direction (on-axis specimens) and at 45° to the loading direction (off-axis specimens). Specimens were subjected to different fatigue loading, with the maximum load level up to 60% of the average ultimate flexural strength, and damage in the laminate was continuously monitored through the loss of bending moment during cycling. After 10^6 cycles, the fatigue test was stopped and residual properties were measured. Micrographs of sample sections revealed some voidage for HL specimens while resin rich areas were observed for RTM specimens. Results of the static tensile and flexural tests pointed out lower mechanical properties for the RTM specimens when tested on-axis and slightly higher properties when tested off-axis. Regardless of specimen fiber orientation, the fatigue and post-fatigue performance of RTM samples was inferior to that of HL specimens with the gap increasing for increasing fatigue load levels. The result was ascribed to the presence in RTM samples of resin-rich areas, which are reported to have limited influence on the laminate static properties but which may act as initiation sites for fatigue cracks.

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Introduction

In the development of new products, a very concerning factor for companies is the understanding of how representative a prototype will be with respect to the production models. Prototypes for experimental testing are manufactured with processes and technologies that usually differ from those of mass production. This is due to economical reasons; mass production requires very expensive tooling with high potential for automation and labor reduction; whereas prototypes are often built with handicraft technologies, which are cheaper for small productions and more suitable for manufacture changes in the phase of experimental assessment. In the present work two technologies have been compared, hand-lay up (HL) and resin transfer molding (RTM), which are probably the most common processes among handicraft and mass production technologies, respectively. Apart from costs and work health and safety issues, the main advantages and disadvantages of the two techniques, as known and perceived by field experts, are very low void contents with possible drawback of unimpregnated areas for RTM, and a quality of laminates very dependent on the skills of laminators for HL.

In the literature, the few available papers that look into direct comparisons among different production techniques are not very recent and mainly report results of static tests performed on on-axis specimens. In [1], tensile, flexural and interlaminar shear strength material tests were used to compare physical properties of cross-ply glass fiber epoxy composites produced by wet lay-up with autoclave consolidation and RTM. The superior compaction pressure of the autoclave molding technique assured higher flexural and tensile strength and modulus, while the better matrix quality and lower void content of RTM specimens led to higher interlaminar shear strength. In [2], microscopic examination of the architecture of E-glass/epoxy 3D multilayer woven composites obtained by RTM revealed that the binder yarn arrangement and compaction pressure have a strong influence on the distribution of fibers and resin-rich areas. However, the presence of resin rich areas did not affect the static tensile strength and modulus in both warp and weft direction. In [3], static tensile tests were performed on unidirectional and woven fabric laminates made with different types of reinforcing fibers manufactured by bag molding, resin transfer molding and vacuum assisted resin transfer molding. Results show that, regardless of fiber type and orientation, the laminate properties were always lower for vacuum assisted molding. One single paper was found on the comparison of fatigue performance. In [4] tension–tension fatigue tests were performed on cross-ply glass fiber in phenolic resin composites produced by HL and pultrusion method. The lower fatigue strength of pultruded specimens was ascribed to a dissimilarity in the main failure mechanism as evinced through SEM observations.

To the author's best knowledge, no comprehensive comparative study is available on the static, fatigue and post-fatigue strength of composite laminates where on-axis and off-axis loadings are considered. Owing to the fact that in a component of complex shape, fibers can not be aligned to the loading direction at all points, comparative studies which consider only on-axis laminates could not correctly take into view the role of the better matrix quality usually associated to the RTM technique. Indeed, microscopic studies [5] have shown that, while for on-axis specimens the fatigue behavior is primarily affected by the stochastic breakage of the brittle fiber bundles, for off-axis specimens the fatigue behavior is strongly influenced by inelastic shear deformation and crack propagation of the ductile polymer matrix.

Moreover, comparisons available in the literature are usually made on specimens which are manufactured in the laboratory under accurately controlled process parameters, whereas the main idea behind the present work was that of verifying whether a material characterization run at the prototype level of a handicraft technology could be significant of a mass production technology and if a comparison of static properties (which is a practicable task at an industrial level) could ensure the same level of agreement for the fatigue life and residual properties.

Materials and methods

The material under study is a hybrid laminate made of layers of biaxial fabrics of Toray T400 6k carbon, E-glass and hybrid Toray T400 6k carbon/E-glass fibers in a matrix of epoxy resin. The matrix is a two-part system of LY-564 resin and HY-2954 hardener which is intended for both RTM and HL applications.

The stacking sequence of the laminate consists of ten layers of biaxial fabrics: CBX400/CEBX180/EBX400/CEBX180/EBX400//symmetric. Details on the layers are given in Table 1. Biaxial fabrics consist of two layers of long fibers held in place by a secondary non-structural stitching tread (polyester). One main advantage over woven fabrics is the improvement in mechanical properties, primarily from the fact that fibers are always straight and not crimped.

Eight laminated plates 1000×1000 mm^2 were manufactured: four plates using HL with vacuum bagging and four plates using the RTM technique. While the plates manufactured by HL were produced by one of the end-users participating in the project, the plates manufactured by RTM were commissioned to an external source employing the same layer fabrics and resin type used for HL.

For HL, the plates were prepared so as to have a fiber volume fraction of 55%. The volume fraction was estimated by the manufacturer by the usual weighing method. The plates mean thickness was 3.20 mm as measured with a caliper. The plates manufactured through RTM were quite thicker (4.20 mm). Considering that the layer fabrics

Table 1 Material properties of the biaxial fabric layers used for the hybrid composite

Layer type	CBX 400	CEBX 180	EBX 400
Material	Carbon	Hybrid E-glass carbon	E-glass
Fiber orientation	Biaxial ± 45°	Biaxial ± 45°	Biaxial ± 45°
Weight in each axis (g/m^2)	200	52 (carbon) 38 (E-glass)	200
Total weight (g/m^2)	400	180	400
Dry thickness (mm)	0.45	0.20	0.43

provided to the external source were those employed in the HL manufacturing process, the fiber volume fraction for RTM plates was calculated in relation to the measured mean thickness and was estimated to be around 42%. The one mm difference leads to a 23.8% difference in thickness and fiber fraction between the two techniques which is obviously quite significant but it is of the same order of the difference reported in [1] between RTM and wet lay-up with autoclave consolidation.

Micrographs of representative sample sections were taken for each one of the two laminates to gain information on the microstructure. Sections containing some voidage were observed for HL specimens, with voids having a 50 μm maximum diameter (Fig. 1a); however, the overall void content was estimated to be below 4% [6]. In RTM samples no significant voidage was observed but rather the presence of resin rich areas (Fig. 1b). This last effect is consistent with the lower fiber volume fraction achieved in RTM samples [1].

The composite plates were cut into individual specimens using a laser beam. Cutting parameters were carefully chosen to avoid possible burning of both the matrix and the fibers. Two different fiber orientations were considered with respect to the loading direction: a cross-ply [0/90] and an angle-ply [±45] laminate were examined. The lay-ups were chosen to represent two fundamentally different stress states: bending of [0/90] specimens results in a quasi one-dimensional loading of the laminate, with large stresses along the longitudinal fiber direction; in the [±45] specimens, bending originates a combined state of normal stresses in the two orthogonal fiber directions of the fabric and shear stresses.

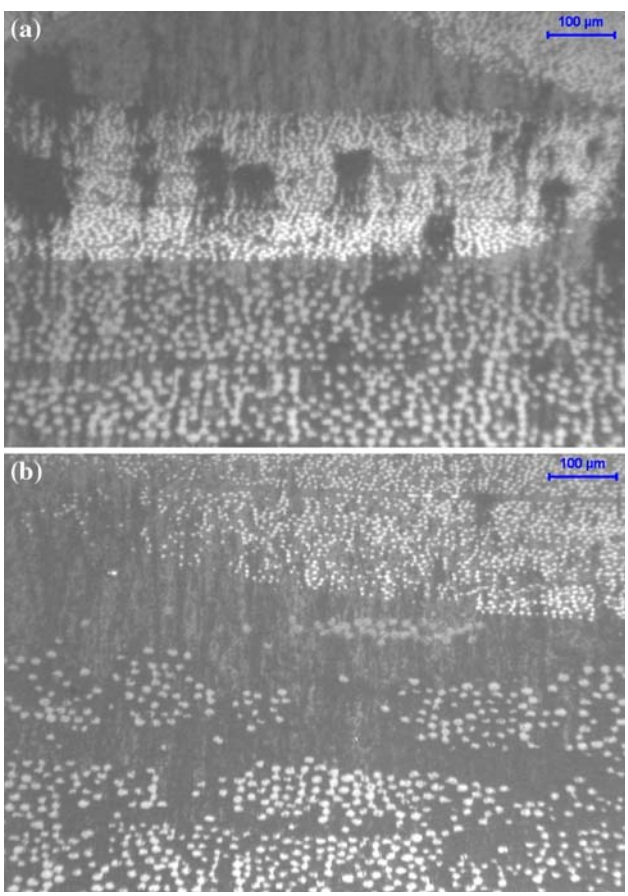

Fig. 1 (**a**) HL micrograph at 200× magnification. (**b**) RTM micrograph at 200× magnification

Static tensile tests were run on 210 × 20 mm^2 beam-like samples and performed in accordance with the ASTM D3039 standard [7]. Specimens were loaded to fracture on an universal electro-mechanical testing machine at a cross-head speed of 1 mm/min. Squared-off 90° tabs were bonded on the specimen ends to strengthen the specimen at the machine grips. A three grid rosette was applied on each specimen face to investigate possible out-of-plane bending.

Four-point static flexural tests were run on 210 × 20 mm^2 beam-like specimens [8] as well as on the waisted specimens used for bending fatigue tests (Fig. 2), as the testing program includes static flexural tests on pre-cycled specimens. The testing fixture was mounted on the servo-hydraulic testing machine. The hydraulic actuator was electronically controlled in order to perform constant velocity tests at 6 mm/min. For the waisted specimens (Fig. 2), the upper span was 16 mm and the lower span was 65 mm.

Four-point bending fatigue tests were run at room temperature on a Schenk-type bending fatigue machine (a schematic drawing is shown in Fig. 2). The machine is displacement controlled through a crank-linkage mechanism and gives place to a sinusoidal waveform. Details on the data acquisition system are given elsewhere [9]. In a constant amplitude test, the amount of load required to deflect the sample may progressively reduce due to material degradation. Therefore, stiffness reduction is sharp at first, as substantial matrix degradation occurs, and then quickly tapers off until only small reductions occur. Failure of the specimen is often not achieved. However, this type of tests is quite suitable to investigate stiffness degradation with cycling as it is representative of what happens at a local level on a real structure, where continuous redistribution of stress and a reduction of stress concentrations occur, as a consequence of local deterioration of the material around the damage areas. Fatigue bending experiments were preferred over tension–compression tests considering that the stacking sequence of the hybrid laminate tested in the paper was specifically selected to enhance the flexural properties of the laminate.

Fatigue experiments were performed with different values of the imposed displacement, i.e. at various initial

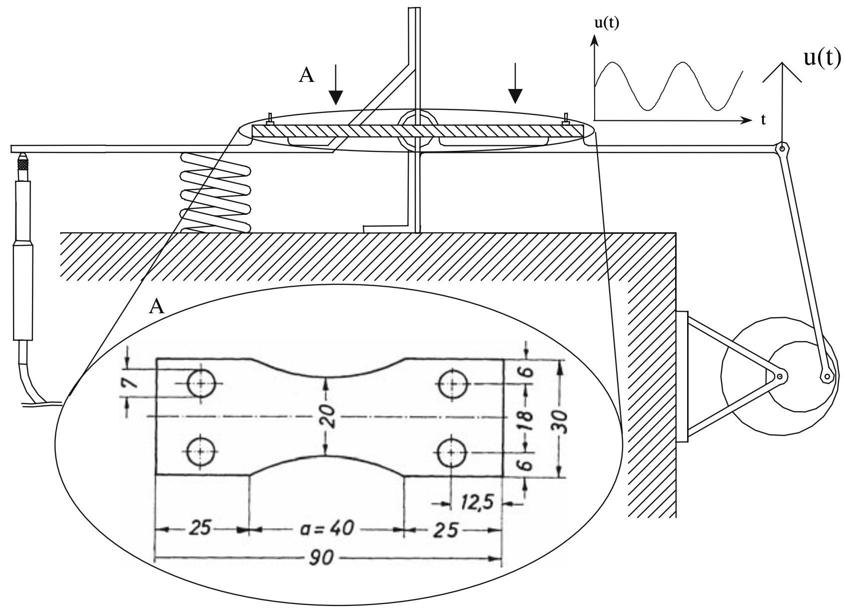

Fig. 2 Schematic drawing of the instrumented testing machine for displacement-controlled bending fatigue. Specimen geometry

stress levels. The level of the imposed displacement was set so that the maximum stress in the first cycle was taken as a percentage of the laminate average ultimate flexural strength (UFS), as obtained in static flexural tests. Fatigue tests were run up to 10^6 cycles. The minimum to maximum displacement ratio was fixed at 0.1. The testing frequency was set at 10 Hz. As shown in past testing programs [9], no significant effect of the test frequency was evident for the material under the test conditions, at least in the 4–10 Hz range. After 10^6 cycles the fatigue tests were stopped and static flexural tests were performed on the pre-cycled specimens to measure the laminate residual flexural strength and residual flexural modulus.

Results and discussion

Static tests

Table 2 summarizes the average value and the standard deviation of the apparent laminate properties as determined in tensile static tests. Five tests were run for each lay-up configuration. Data are calculated with reference to the specimen symmetry axes (x, y). The x-axis is parallel to the load direction. In accordance with the ASTM standard D3039 [7], the ultimate tensile strength (UTS) was calculated by dividing the maximum load prior to failure by the average cross-sectional area. The Poisson's ratio (ν_{xy}) obtained on the two specimen faces was consistent, showing that, as expected, no significant out-of-plane bending occurred during the test. Narrow standard deviations show repeatability of the experimental results.

Results obtained in flexural static tests are reported in Table 3. Six tests were run for each lay-up configuration. Once more, data are calculated with reference to the specimen symmetry axes (x, y), with the x-axis taken to be parallel to the load direction. Standard deviations are again quite narrow and confirm repeatability of the experimental results.

The ultimate flexural strength (UFS) was calculated using a homogeneous beam theory [8]:

$$\sigma = \frac{3Pa}{bd^2} \tag{1}$$

where P is the maximum applied load, a is the distance between opposing supports, b and d the width and thickness, respectively, of the beam-like specimen.

Table 2 Apparent laminate properties: average value and standard deviation obtained in static tensile tests on beam-like specimens

		RTM		HL		RTM % difference
		Average	SD	Average	SD	
Off-axis	UTS (MPa)	107	2.5	96	7.0	+11.5
	E_x (GPa)	8.7	0.20	8.3	0.20	+4.8
On-axis	UTS (MPa)	464	28.0	509	26.8	–8.8
	E_x (GPa)	27.7	1.50	36.4	1.49	–23.9

Table 3 Apparent laminate properties: average value and standard deviation obtained in static flexural tests on beam-like specimens

		RTM		HL		RTM % difference
		Average	SD	Average	SD	
Off-axis	UFS (MPa)	150	2.1	145	5.6	+3.5
	E_x (GPa)	9.8	0.30	8.9	0.13	+10.1
On-axis	UFS (MPa)	455	10.5	516	16.8	–13.4
	E_x (GPa)	33.8	1.40	43.5	4.11	–22.3

For the on-axis fiber orientation, evaluation of the maximum of the load–displacement curve poses no troubles (Fig. 3). For the off-axis fiber orientation, the load at failure is harder to define as there is a sort of ''plateau'' in the curve, where the displacement is increasing without hardly any increase in the load (Fig. 3). For calculating the UFS, the maximum load value was used. It was in any case verified that the UFS values calculated using the minimum load in the ''plateau'' region fall within the experimental scatter.

The longitudinal flexural modulus E_x was calculated from the initial slope of the load–deflection curve (Fig. 3):

$$E_x = \frac{a^2(3L - 4a)}{bd^3}\frac{P}{\delta} \quad (2)$$

where L is the lower support span, δ is the displacement under the points of application of the two $P/2$ loads on top.

Results reported in Tables 2 and 3 point out that, given the manufacturing technology, for the on-axis fiber orientation the apparent elastic modulus measured in flexural tests is noteworthy higher than that measured in tensile tests, while no significant difference is found between the ultimate tensile and flexural strength. The difference in elastic modulus can be explained by the specific stacking sequence of the laminate with carbon fibers in the outer layers. The similarity in the ultimate strength suggests that the laminate strength is dominated by the outer carbon fibers also in the case of tensile tests [10].

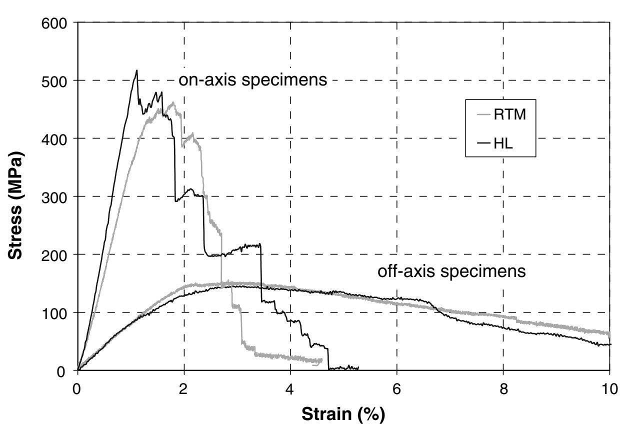

Fig. 3 Representative stress-strain curves for static flexural tests

Looking into the manufacturing technology, it is worthwhile noticing that for the off-axis fiber orientation RTM specimens exhibit slightly higher properties than HL specimens (both in terms of elastic modulus and of laminate strength), while for the on-axis fiber orientation higher stiffness and strength properties are found for HL specimens. Results obtained for the off-axis fiber orientation may be ascribed to the adequacy of the RTM process in terms of good matrix quality, with a lower void content in comparison to laminates obtained through handicraft technologies (Fig. 1). Results obtained for the on-axis fiber orientation owe to the lower fiber volume content of RTM specimens. In particular, the estimated 23.8% fiber volume reduction well agrees with the measured stiffness reduction observed in tensile and flexural tests (23.9% and 22.3%, respectively). The reduction in strength, even if noteworthy, is limited to about 10%. A similar correlation between the reduction in fiber volume content and laminate properties was observed in [1].

Fatigue behavior

Results obtained in displacement controlled four-point bending fatigue tests run up to 10^6 cycles are reported in Figs. 4–6. As often reported in the literature [10] for similar experimental studies, stiffness degradation during cycling was evaluated by measuring reduction in the Relative Bending Moment (RBM). The RBM parameter is defined as the ratio between the bending moment applied at the Nth cycle (B_N) and the bending moment applied at first cycle (B_1).

Figures 4 and 5 depict how the RBM parameter changes during cycling for different values of the maximum initial bending moment. The initial maximum stress levels selected were 10%, 20%, 30%, 40%, 50% and 60% of average ultimate flexural strength (UFS). The 60% UFS corresponds to the maximum loading capacity of the test apparatus. Data for maximum stress levels of 10% and 20% UFS are not reported as no significant stiffness loss was observed during cycling. Curves drawn in Figs. 4 and 5 are for RTM specimens while the shadowed area correspond to the scatter band obtained for HL specimens.

For the off-axis fiber orientation (Fig. 4), data are well superimposed for stress levels up to 40% UFS. At 50%

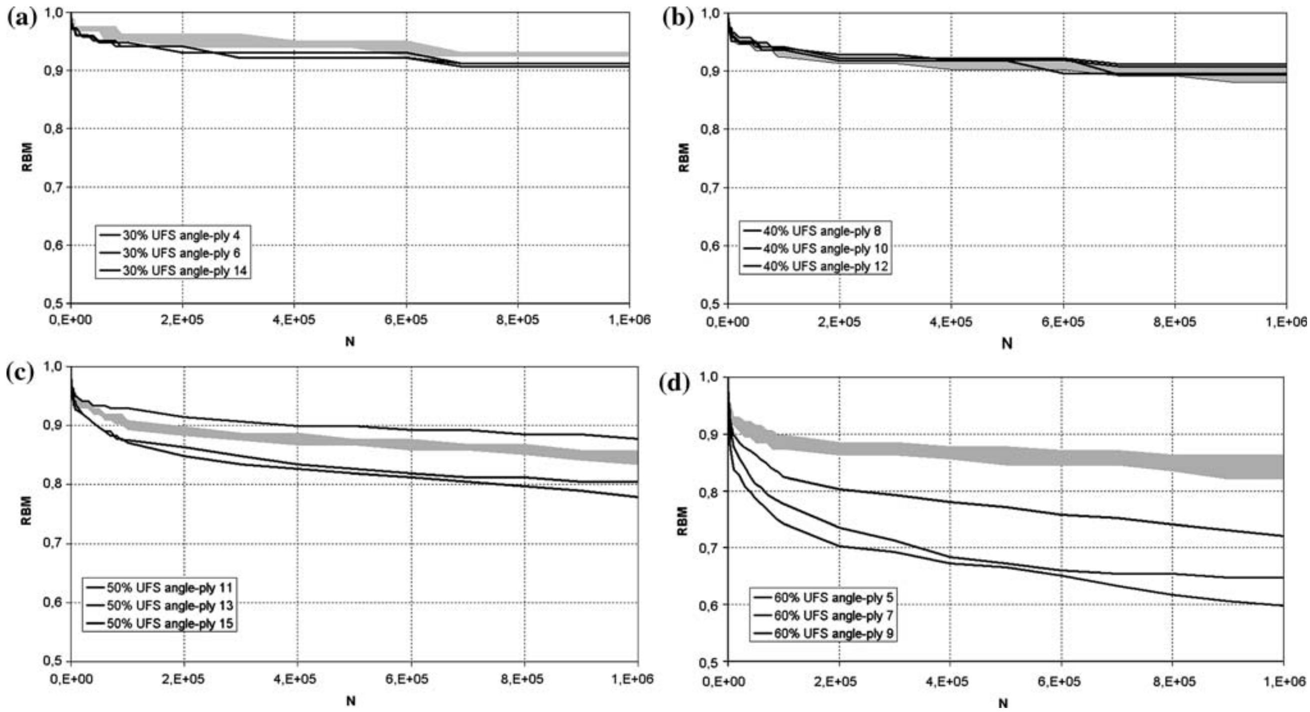

Fig. 4 Loss of relative bending moment (RBM) versus number of cycles. *Drawn curves* are for RTM, while the *shadowed area* corresponds to the scatter band for HL. Tests run on angle-ply $[\pm 45]_{5S}$ laminates—off-axis specimens

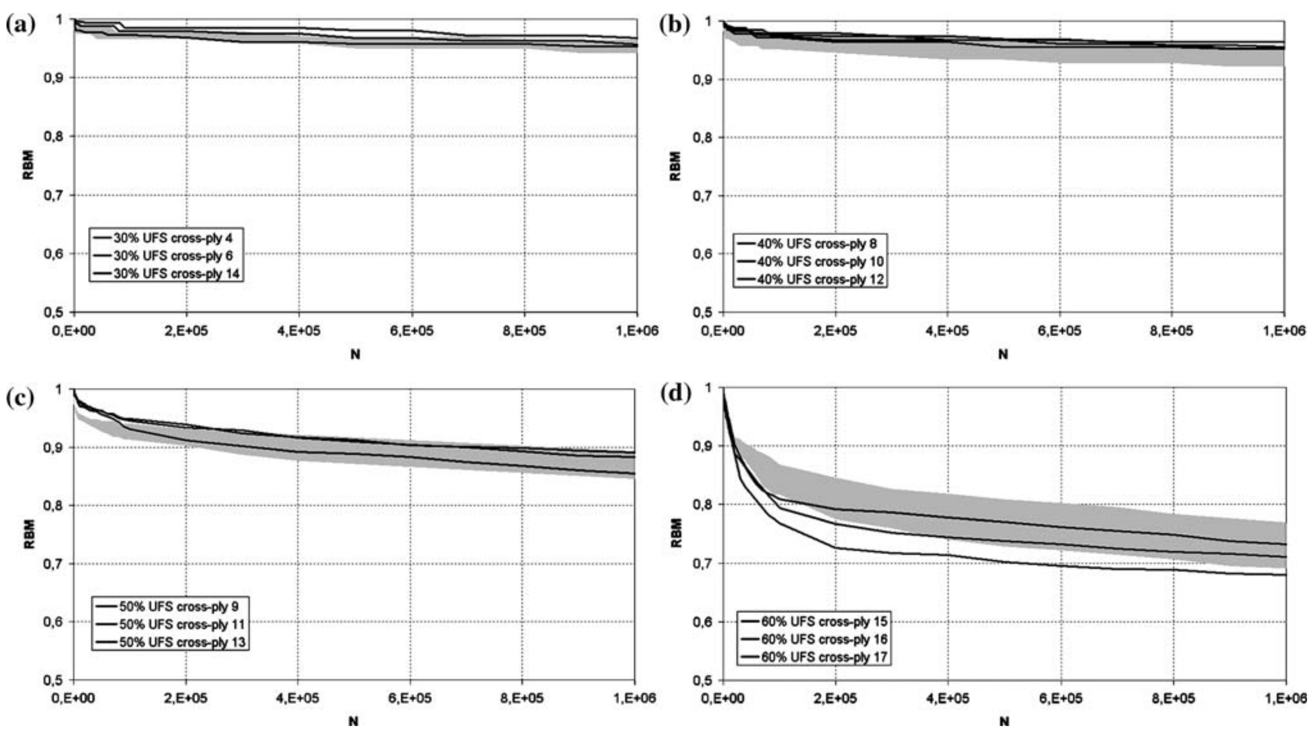

Fig. 5 Loss of relative bending moment (RBM) versus number of cycles. *Drawn curves* are for RTM, while the *shadowed area* corresponds to the scatter band for HL. Tests run on cross-ply $[0/90]_{5S}$ laminates—on-axis specimens

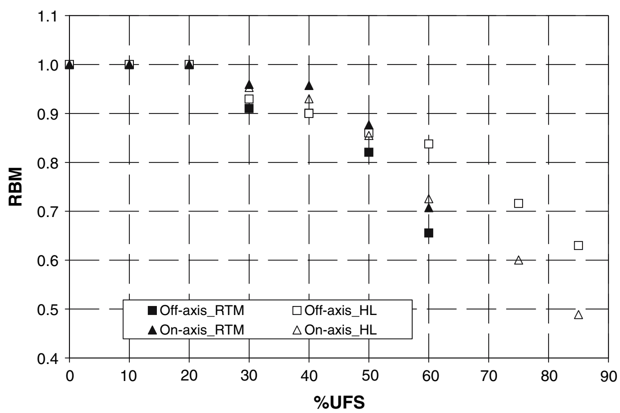

Fig. 6 Loss of relative bending moment (RBM) at 10^6 cycles versus level of fatigue loading

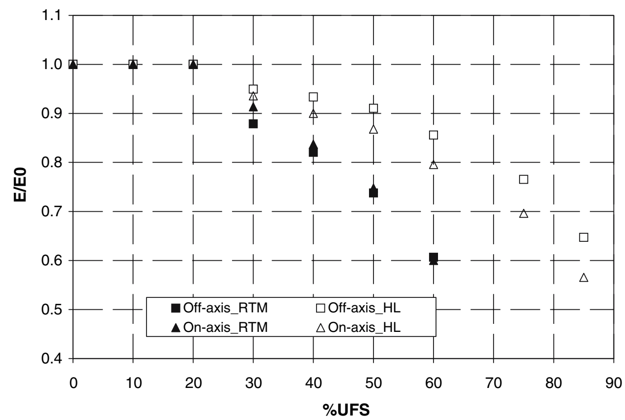

Fig. 7 Normalized residual modulus at 10^6 cycles versus level of fatigue loading

UFS (Fig. 4c), the three curves obtained for RTM specimens are quite spread, with two curves below the scatter band of HL specimens. At 60% UFS (Fig. 4d), the stiffness reduction for RTM specimens is clearly more pronounced. For the on-axis fiber orientation (Fig. 5), data are well superimposed up to 50% UFS. At 60% UFS (Fig. 5d), one of the three curve is actually below the scatter band of HL specimens, signaling a possible turning point from which the stiffness loss for RTM specimens becomes higher than for HL specimens. Unfortunately, no tests for stress levels higher than 60% UFS could be run on RTM specimens as the maximum loading capacity of the testing machine was reached.

In Fig. 6, the average RBM value at 10^6 cycles is plotted against the level of fatigue loading, summarizing the pieces of information given in Figs. 4 and 5. For maximum fatigue stress levels of 10 and 20% UFS, RBM values of one are reported as no significant stiffness loss was observed during cycling. In the case of HL specimens, data points were available from a previous experimental program [9] and are fully reported up to 85% UFS. Due to the lower thickness of HL specimens, higher stress levels were achieved before the maximum loading capacity of the apparatus was reached. As it can be observed from Fig. 6, up to 50% UFS no significant difference can be evinced between RTM and HL specimens regardless of fiber orientation. At 60% UFS data points are more spread: in particular, off-axis RTM specimens exhibit the lowest RBM value.

Residual properties

Residual properties of specimens pre-cycled at 10^6 cycles are shown in Figs. 7 and 8. To avoid confusion only average data points are drawn. The level of data scattering was in any case limited for both sets of specimens. For sake of comparison between RTM and hand-lay up specimens,

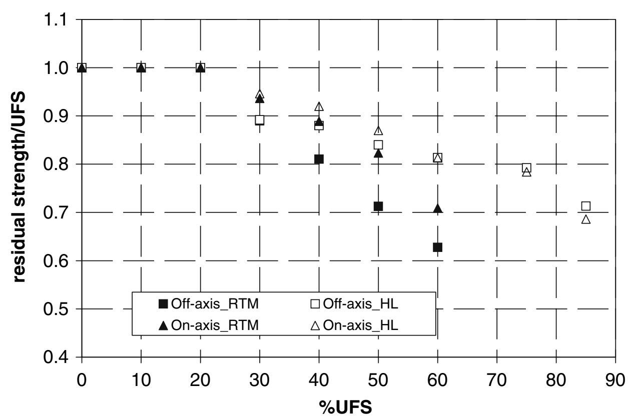

Fig. 8 Normalized residual strength at 10^6 cycles versus level of fatigue loading

the residual properties are normalized by the average material properties of the virgin specimen.

When looking at the data reported on Figs. 7–8, it is quite evident that the post fatigue performance of the RTM specimens is inferior to that of HL specimens and that the gap between the two sets of specimens increases for higher stress levels. The difference is more pronounced for the off-axis fiber orientation, but clearly exists also for the on-axis fiber orientation for which similar values of the RBM at 10^6 cycles were observed (Fig. 6). The poorer performance of RTM samples is associated to the presence of resin rich areas, which are reported to have limited influence on the laminate static properties [2] but which may degrade the laminate fatigue performance as they act as initiation sites for cracks [1, 2].

A comment worth making is that the greater thickness of the RTM samples might have played a detrimental role, taking into consideration that the fatigue tests are performed in bending. Unfortunately, the potential ''size effect'' could not be singled out in this study considering that the two sets of samples also differed for the achieved fiber volume fraction. On the other hand, it is interesting to note that the dissimilarity in static properties between RTM and hand lay-up samples (Tables 2–3) is of the same order regardless of the type of static test being performed (tensile and flexural), suggesting that, at least for the static properties, it is the lower fiber volume fraction to be the main responsible for the reduced laminate static strength and stiffness of the RTM samples.

From Figs. 7–8, it is evident that for maximum stress levels of 10% and 20% UFS fatigue cycling causes no degradation in the flexural strength and modulus. The result may be interpreted as the existence in bending of a ''fatigue threshold stress'' [9, 11]. Interestingly such threshold value, when normalized by the static average strength, is the same regardless of manufacturing technology and fiber orientation.

One last comment can be made regarding the fact that, even if similar trends can be envisaged, there is no exact correlation between the loss in modulus as measured at the end of the fatigue test and the loss in modulus (and strength) as measured in static tests on pre-cycled specimens. The dissimilarity in the two values of the flexural modulus was already observed in [12] and it is acknowledged to exist in materials which do not have elastic nature till fracture. The fairly good agreement between the loss in residual modulus and in residual strength is in accord with the model of Van Paepegem and Degrieck [11].

It is in any case worthwhile noticing that the material parameter which appears to most effectively distinguish between the two manufacturing technologies is the residual flexural modulus, for which the relationship with the level of fatigue loading is rather smooth. Data of residual modulus may be employed to calculate the so-called macroscopic damage variable $D = 1 - E/E_0$, often used in the literature to assess the level of material damage. As it can be seen from Fig. 9, for fatigue load levels above the

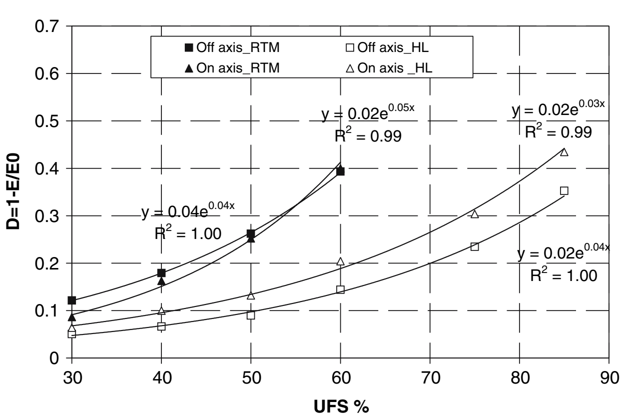

Fig. 9 Macroscopic damage variable at 10^6 cycles versus level of fatigue loading

''fatigue threshold stress'', data points are well interpolated by an exponential regression curve. On the parameter values of the regression curves, the influence of the manufacturing technology appears more significant than the fiber orientation.

Conclusions

The fatigue and post-fatigue behavior of carbon–glass biaxial fabric reinforced epoxy composites manufactured by RTM and HL has been experimentally evaluated with the main objective of verifying whether a material characterization run at the prototype level of a handicraft technology as HL could be significant of a mass production technology as RTM and if a comparison of static properties (which is a practicable task at an industrial level) could ensure the same level of adequacy for the fatigue life and residual properties.

With the purpose of investigating different stress states, standard specimens having on-axis and off-axis fiber orientation were considered. The experimental results point out the following conclusions:

- For the HL samples, a 55% fiber volume content was achieved. Thickness measurements pointed out a lower fiber volume (around 42%) for the RTM samples. Micrographs of sample sections revealed some voidage in the HL specimens while resin rich areas were observed in the RTM samples;
- Tensile and flexural static tests show slightly higher stiffness and strength for RTM specimens when tested off-axis owing to the better matrix properties and significantly higher stiffness and strength for HL specimens when tested on-axis owing to the achieved higher fiber volume fraction;
- Regardless of the manufacturing technology, the similarity in the ultimate tensile and flexural strength suggests that the strength of the considered hybrid laminate is dominated by the outer carbon fibers also in the case of tensile tests;
- No significant loss in stiffness during cycling nor in residual properties was observed when the maximum fatigue stress was below 30% of the average ultimate flexural strength, regardless of the manufacturing technology and sample fiber orientation The result was interpreted as the existence in bending of a ''fatigue threshold stress'';
- Regardless of specimen fiber orientation, the fatigue and post-fatigue performance of RTM samples was inferior to that of HL specimens with the gap increasing for increasing fatigue load levels. The result was ascribed to the presence of resin rich areas in RTM samples, which act as initiation sites for cracks.

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