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On the Fatigue Behavior of a Triphasic Composite

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Abstract**—**This paper presents the results of an experimental characterization of a glass fibre-epoxy composite. The behavior of the traditional two-phase composite has been compared with the one of a new three-phase composite where the epoxy matrix was modified by addition of a 3% weight fraction of montmorillonite nano-particles. Two different types of nano-clays, Cloisite® 30B and RXG7000, produced by Southern Clay Products Inc., have been considered. Three-point bending tests, both monotonic and cyclic, were carried out. A strong reduction of the ultimate flexural strength upon nano-modification has been observed in quasi-static tests. Fatigue tests yielded a smaller strength loss. In both quasi-static and fatigue tests a more pronounced tendency to delamination has been noticed in three-phase composites, especially in the case of 30B nano-clay, with respect to the standard two-phase glass fiber composite.

Keywords**—**Bending fatigue, epoxy resin, glass fiber, montmorillonite.

I. INTRODUCTION

DIFFERENT recent studies show that the mechanical properties of polymers may be significantly be improved by the addition of small quantities of nano-particles [1], [2]. From one side, this improvement is not observed in all cases, since it depends in a not completely understood manner from several factors, being the quality of the dispersion of the nano-particles in the polymer the most important one, from the other side it opens new perspectives on the field of traditional polymer matrix composites.

In fact, for these materials an improved resistance and stiffness of the matrix, that is the weakest part of the composite, may lead to the improvement of several mechanical properties dominated by the matrix characteristics, such as the resistance to traction in the transversal direction respect to the fibers, to longitudinal compression or to inter-laminar shear.

The majority of the studies available in the scientific literature regard two phase composites in which the nano-reinforcement is embedded in a neat resin.

Limited information, on the other hand, is available on the mechanical behavior of three phase composites in which the biphasic composite is used as matrix for a traditional fiber reinforced composite [3]-[10]. In particular, the number of studies on the fatigue response of these new materials is very small [11]-[13].

This work summarizes the results of an experimental test campaign on the fatigue behavior of glass fiber reinforced epoxy resin modified by nano-montmorrillonite particles.

II. MATERIALS AND METHODS

The base material utilized in this study was a laminate composite constituted by 8 layers of 2x2 twill woven fiber in the $[(0/90)]_8$ configuration. The fiber was E-Glass (VV-350 T provided by G. Angeloni s. r. l.), with nominal mass of 350 g/m^2; the resin was an epoxy (EC 157 provided by Elantas Camattini S. p. A.). The panels from which the specimens were later machined were produced by infusion with a post curing cycle at 60°C for 15 hours as suggested by the resin producer.In the same way, other panels containing two kinds of nano-charges, Cloisite® 30B e RXG7000, provided by Southern Clay Products Inc., were manufactured. The dispersion of 3% in weight of the nano-particles in the resin (without hardener) was performed by mechanical stirring followed by sonication. A further stirring was done to mix the hardener to the previously obtained mixture. The subsequent infusion was done in a similar way to the one used for the base material, except a different pressure in the pump due to the higher viscosity of the nano-charged resin.

The thickness of the so obtained panels ranged from 3.6mm in the case of the resin reinforced by RXG7000 to 3.9mm for the base material and the one reinforced by 30B. From the panels, 25mm width specimens, like the one visible in Fig. 1, were cut by means of a diamond saw.

Fig. 1 Three point bending device

Tests were performed on a 50 kN servo-hydraulic machine with a three point bending device designed and manufactured

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to the purpose (Fig. 1.) following procedures similar to the ones used in [14].

The distance between supports was 50mm and it was chosen in order to obtain a relatively small span to thickness ratio so that a significant shear effect was expected, in addition to normal bending stresses. This was made to enhance the effects of the nano-modification of the matrix on the resistance to inter-laminar shear. The supports were constituted by rollers in order to minimize friction and the radius of the rollers and of the cylindrical loading nose was 7mm.

Preliminary quasi-static displacement controlled tests were performed at a nose speed of 0.01mm/s to compare the bending resistance of the different materials.

To avoid the loss of unilateral contact between specimen and nose, and the following repeated impact, during the fatigue cycling done in load control, a load ratio $R = \sigma_{min}/\sigma_{max}$ of 0.1 was used.

To limit the material heating, the loading frequency was set at 1 Hz after preliminary tests at different frequencies in which the surface temperature was monitored by means of an infrared device. The run-out life limit was set to one million cycles for that reason.

During the tests, the nose position and the applied load were acquired at regular intervals for one second at a frequency of 100Hz in order to reconstruct hysteresis cycles and analyze their change during the test.

III. RESULT AND DISCUSSION

The values of ultimate strength obtained in the preliminary tests, calculated as mean and standard deviation on three tests, are reported in Table I. They take into account of the real dimensions of each specimen measured with a precision ±0.01 mm and they are referred to the maximum stress reached on the outermost surfaces calculated with the elementary beam theory, where F is the applied load, L is the span, b and h are width and thickness respectively.

$$\sigma = \frac{3FL}{2bh^2} \tag{1}$$

TABLE I
MAXIMUM TENSILE STRENGTH AND STIFFNESS IN QUASI-STATIC TESTS

Material	Base	30B 3 %	RXG7000 3 %
Maximum Strength (MPa)	748 ± 5	501 ± 6	471 ± 11
Stiffness (kN/mm)	1.193 ± 0.046	1.043 ± 0.016	1.068 ± 0.007

A sharp reduction of the resistance due to the introduction of nano-charges may be noted (33 % and 37 % for nano-clay 30B and RXG7000, respectively) in comparison to the values obtained for the base material.

The reported stiffness (defined as the force necessary to obtain a unit inflexion) was calculated directly from the displacement and the load measured by the machine. To take into account the actual width and thickness of the specimens the values were scaled according to the formula:

$$k_{ref} = k\frac{b_{ref}}{b}\frac{h_{ref}^3}{h^3} \tag{2}$$

referring them to an ideal specimen with h_{ref} = 3.85mm and b_{ref} = 25.00mm.

The values were slightly lower in presence of nano-reinforcement (12% less for the 30Band 10% for the RXG7000). This result is in agreement with the reduction of Young modulus of the composite, previously measured in compression tests on specimens instrumented by strain gages, from 30.0 GPa for the base material to 26.6 and 27.4 GPa for the 30B and RXG7000 respectively.

Twelve fatigue tests were performed for each material and the results are reported in Figs. 2 and 3. For all the data sets the results are not much dispersed and the classic Basquin interpolation

$$\sigma = AN^b \tag{3}$$

represents a good empirical description of their fatigue behavior in all the range considered, that in terms of fatigue life goes 10^3 to 10^6 cycles). From the datait is not possible to determine the existence of a fatigue limit.

Plotting data within ordinate the absolute values of the maximum stress (calculated from (1)) reached during the load cycle (Fig. 2.) the points relative to the base material remain higher than those pertinent to the nano-charged materials, while it is not possible to distinguish between them, apart from a slightly higher slope for the RXG7000. The A and b values according to (3) obtained for the three materials are reported in Table II.

TABLE II
PARAMETERS OF THE BASQUINS-N INTERPOLATION

Base		30B 3 %		RXG7000 3%	
A (MPa)	*b*	*A* (MPa)	*b*	*A* (MPa)	*b*
910	-0.097	610	-0.068	720	-0.086

Due to its high slope, the line relative to the base material intersects those of the two triphasic materials at a very high number of cycles. This intersection is located outside the range considered in this study, in particular as RXG7000, so in condition in which the validity of the interpolation is not proved. As a consequence, it is not possible to conclude on the basis of the data that a zone in which the fatigue resistance of the nano-reinforced composite is higher than the one of the base material exist. On the contrary, at a lower number of cycles the lowering of the fatigue properties due to the introduction of the nano-fillers is evident. For example, we have a life reduction of three times at al load level of 300 MPa, or a resistance reduction of 15 % for a fatigue life of 10^4 cycles.

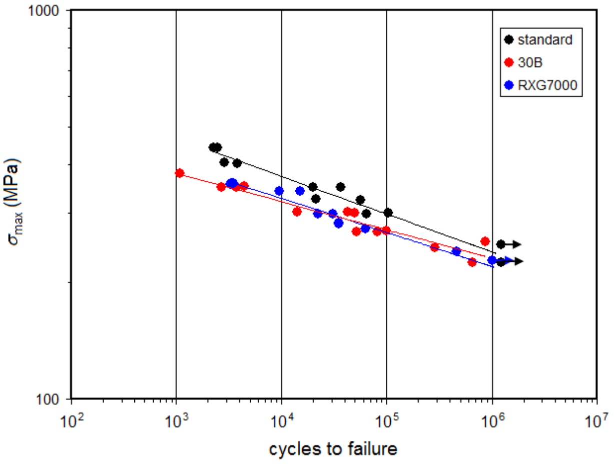

Fig. 2 σmax-N diagram

Reducing data differently and plotting the ratio between the maximum stress in the fatigue cycle and the ultimate static stress of each (Fig. 3), the situation is opposite. Taking as reference for example a life of 10^4 cycles, the base material is able to resist to 50 % of its ultimate stress, while the two triphasic materials can sustain 65% (30B) and 70% (RXG7000).

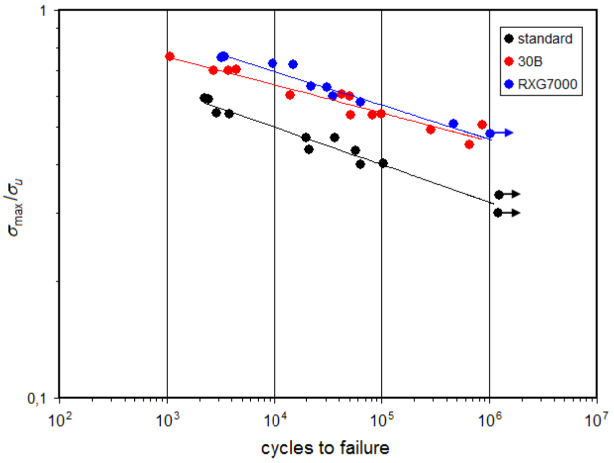

Fig. 3 σmax/σu-N diagram

Evidences of a different mechanical behavior were found also from the analysis of the fracture mechanisms. The only characteristic common to all the specimens (base and modified materials) was the presence of an elastic instability zone on the loaded surface, near the nose, where the maximum compressive stresses are present. This kind of failure happened regularly in the last part of the tests few cycled before the final collapse. Marked differences were noted in the specimens after failure, as regards the tendency to delamination (Figs. 4-6). While the base material showed a small delaminated zone, centered under the loading nose, even when the specimen was nearly broken in two pieces (Fig. 4), the two nano-charged materials (Figs. 5, 6) presented always a wider delaminated zone, especially in the case of nano-clay 30B, in which it went till the lower support of the specimen, in many cases non symmetric respect to the position of the nose.

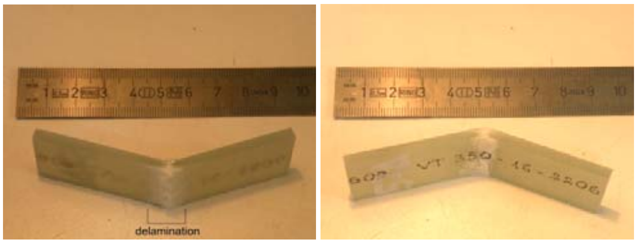

Fig. 4 Example of base material specimen after fatigue failure

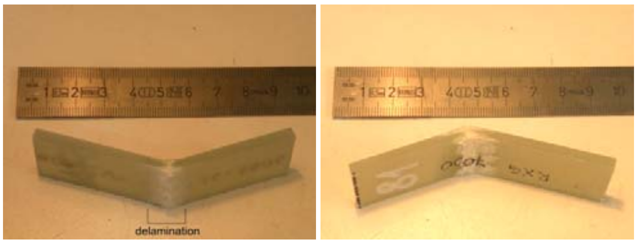

Fig. 5 Example of nano-clay RXG7000 modified matrix specimen after fatigue failure

Fig. 6 Example of nano-clay B30 modified matrix after fatigue failure

This observation suggests that the propagation of the delamination was unstable also in the fatigue test as it was noted in the quasi static ones by visual observation. Probably the unstable propagation of delamination in one direction reduces the available energy impeding the propagation in the other direction. Since the geometrical configuration of all the experiment was the same (in particular the span to thickness ratio), this different behavior was due to a change in the matrix properties or in the interfacial properties between glass fibers and epoxy resin due to the addition of montmorillonite.

It is very likely that the addition of the nano charge reduced the inter-laminar shear strength of the epoxy matrix [15].

The analysis of the data acquired during fatigue test does not show appreciable differences between the three materials. As it can be seen in Figs. 7-9, in which three examples relative three specimens with similar fatigue lives, three phases may be singled out.

In the first phase, a nearly linear decrement of the stiffness was observed, while the energy absorbed during the cycle (calculated by numerical integration of the mechanical hysteresis cycle) was practically constant with fluctuation that are within the measurement error of this quantity.

The linear increment of the mean displacement of the cycle (except in the first cycles where it is quicker, probably because the loading nose and the support may indent progressively the outer most resin layer) indicate a regular translation of the cycle itself due probably to ratcheting.

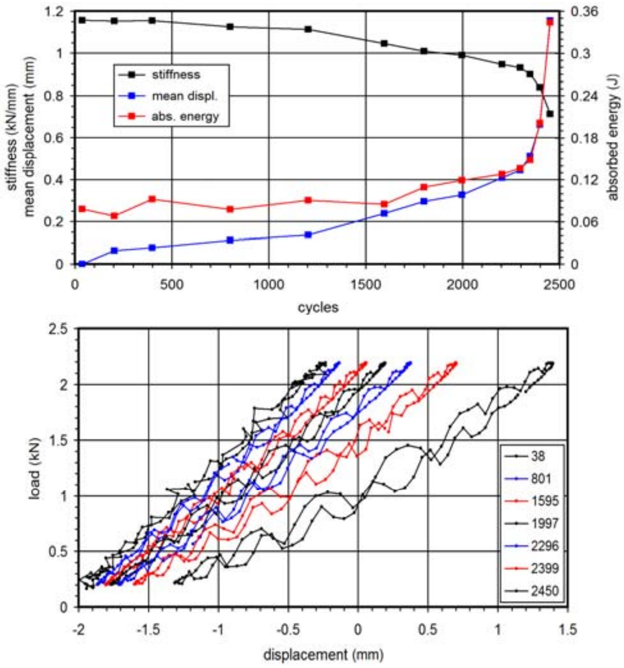

Fig. 7 Stiffness, mean displacement in the cycle per cycle absorbed energy and hysteresis cycles for the or base material (failure at 2451 cycles)

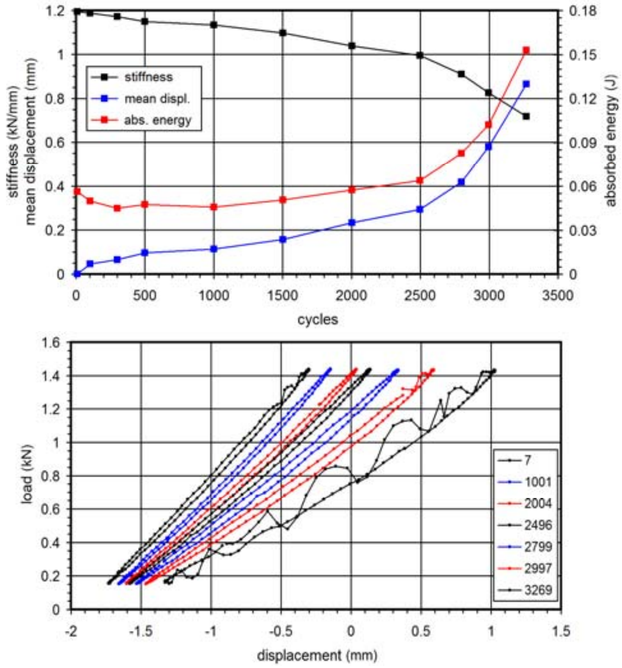

Fig. 8 Stiffness, mean displacement in the cycle per cycle absorbed energy and hysteresis cycle for the RXG7000 modified material (failure at 3447 cycles)

Approximately in the middle of the test a second phase was observed, similar to the first one, but with an energy absorption slowly increasing; in parallel also the velocity of the translation of the cycle and of reduction of the stiffness was increasing. The difference respect to the first phase seemed higher for both the nano-charged materials, while it was barely visible for the base material and this was the only difference.

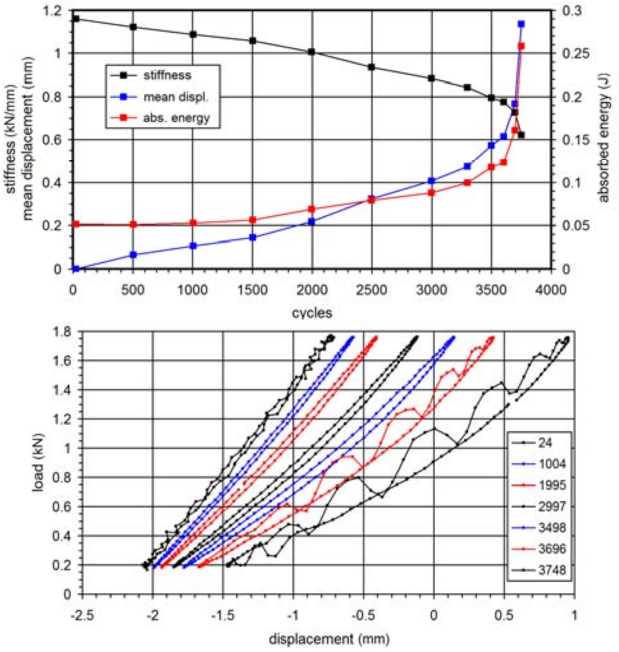

Fig. 9 Stiffness, mean displacement in the cycle per cycle absorbed energy and hysteresis cycle for the B30 modified material (failure at 3748 cycles)

The third phase, always limited to the last part of the test, was characterized by a reduction of stiffness and a translation of the hysteresis cycle with an increasing rate and by a noticeable increase of the absorbed energy up to the final failure.

The higher compliance of the specimen provoked difficulties in the load control of the servo-hydraulic machine.

And this is the cause of the oscillations observed in the last cycles. These oscillations, in any case, were limited and did not influence the measure of stiffness and cycle area that were calculated basing on a high number of points. The stiffness was evaluated in the increasing load part of the cycle.

As regards the meaning of the results presented in Figs. 7-9 it is worth pointing out the following.

The measure of the stiffness may be considered a valid estimate of the damage of the specimen. In fact, even if the displacement was measured by means of the transducer of the piston of the machine, so including the deformation and clearances of all the components of the machine in the load chain, these spurious effects were small because the load was much lower than the machine capacity and most of all they used to vary in a limited way from on test to the other, since the load interval considered was limited as well (from 1.0 to 2.2 kN). The same may be said for the mean displacement.

The absorbed energy, on the contrary, for the same reasons

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includes in a way difficult to quantify all the dissipative effects of the system together with the one related to damage processes of the material.

In addition, even in an ideal machine, the absorbed energy would include frictional effects due to the contact of the damaged internal surfaces.

For this reason, the measured absolute values cannot be treated as quantitative measures of the energy necessary to form fracture surfaces. However within the single test the value of the absorbed energy may be considered an important empirical parameter.

IV. CONCLUSION

Three point bending tests have been done on a two triphasic composites obtained by nano-modification of an epoxy resin by means of two different montmorrillonite nano-particles Cloisite® 30B e RXG7000, produced by Southern Clay Products Inc. reinforced by glass fiber. The results have been compared with those of the biphasic glass/epoxy base material.

Quasi-static monotonic tests showed a lower stiffness in the specimens in which nano-clays were added, 10% less for 30B and 12% less for RXG7000, respectively. This result is compatible with an analogous reduction of the elastic modulus, previously observed.

Moreover, the introduction of the nano-particles reduces dramatically the resistance of the composites by 33% and 37% in the two cases.

This different was found also, even if less pronounced, in the fatigue tests.

In absolute terms, the two triphasic materials showed a lower fatigue resistance in all the range of duration explored, from 10^3 to 10^6 cycles. The reduction in resistance was around 15% for 10 10^4 cycles, higher for lower lives and lover for higher number of cycles.

In relative terms, reducing the applied stress with the static strength for each material, a better result was found for the nano-modified materials due to their poor static properties.

The analysis of the cycles did not show appreciable differences among the three materials, except a modification in the failure mechanism of the two nano-modified materials that are more prone to delamination both in the quasi-static and in the fatigue tests.

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