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FATIGUE OF GLASS-POLYESTER COMPOSITE IMMERGED IN WATER

DJEGHADER D.*, REDJEL B.

Civil Engineering Laboratory, Badji Mokhtar's University, BP 12, 23000 Annaba, Algeria
*Corresponding Author: djameldjeghader@yahoo.fr

Abstract

A composite glass fiber laminated polyester resin have been aged in liquid water at 25°C and tested using cyclic fatigue test with three-point bending. Mechanical analyses have been carried out using Wohler curve before and after immersion in water, the effect of such water uptake with immersion time on laminate fatigue life after fatigue cycling is described. It was found that for various periods of immersion, the composites experienced significant reduction of the life time by deterioration of fiber matrix interface, and increase on the weight by water absorption. These are attributed to the function of the water molecules penetrated in the composites.

Keywords: Fatigue, Composite, Glass-polyester, Immersion, Wohler.

1. Introduction

The increasing use of composite materials reinforced by glass fibers in the various structural applications such as the aircraft industry, auto industry, the storage tanks, construction worries more and more the originators when with the critical effects of the cracks and the defects on their behavior in the various environments considered, such as water, seawater, humidity, high and low temperatures. Durability and sensitivity to degradation in various service environments can be modified by the reaction of the various components of its composites, i.e., fibers, matrices, and the interface/interphase between the matrix and the fibers. However, these materials remain much less reliable than metallic materials in particular when they are subjected to cyclic loads. Thus, studies of trends and modeling of damage caused by repeated loads during the life of structures and composite materials prove an important need is useful for predicting their behavior [1].

The Prediction and characterization of fatigue behavior of composite materials based on glass fiber has been the subject of various studies in order to develop

Nomenclatures

A	Constant of the material
B	Constant of the material
Cr	Correlation coefficient (Fig. 5)
$Fmax$	Maximum level of bending load, N
M_f	Final mass of the sample after immersion in water, g
M_i	Initial mass before immersion in water, g
N_R	Life time, days

Greek Symbols

ρ	The rate of increase in the mass, %

analytic and experimental methods and the creation of reliable models for predicting the mechanical behavior during the lifetime. Laminated composite materials are also very susceptible to damage induced by the environment degrade severely their physical and mechanical properties [2-3].

Aging interfaces glass/polyester in a moist environment at the time service, Gautier et all [4] evaluate the resistance of the glass / polyester interface immersed in distilled water at a temperature of 30°C, they have found that a reduction in the quality of the fiber matrix interface which is about the half after 50 hours of immersion. The use of three-point bending and interlaminar shear tests possible to cause damage within the composite as by soliciting matrix and the interface. Jacquemet [5] found an irreversible drop of 14% to 20% of the breaking stress at the interlaminar shear for a composite material of different polyester resin aged in water at 60°C. The overall behavior and performance of a composite material such as glass / polyester can not be explained only in terms of specific properties of the components, but the interface / interphase between the fibers and the matrix is of great importance [6]. The water penetrates the interface in the composite during aging can modify the interfacial adhesion nature of the system which causes a degradation of the mechanical performance of the composite polyester glass. Moisture damage begins near the surface of the material and spreads inward over time, with cracks tending to grow parallel to the free surface[7]. The penetration of water or moisture in the composite may be by diffusion or capillarity. Water absorption causes a reversible and irreversible change in the mechanical performance of glass / polyester composite materials [8]. Moreover, the conditions of implementation and manufacturing method influence aging through parameters such as fiber content, the presence of vacuum and the matrix cross-linking rate.

The accumulation of damage resulting from repeated impact loads was studied by A.P Mouritz et all [9] who report microscopy revealed the formation of deep craters at the point of application load and the size increase with the number of impacts. Similarly, K. E. Wu , Shyu [10] and M.S Found and Howard [11], show the effect of impact number for different loads on the composite surface. D. Ray and all [12, 13] examining the properties of composite materials in repeated impacts loading using the curves E-Nf (number of impact energy-impact failure fatigue) and report that strength and the number of impacts are the two main parameters that govern the behavior of these materials in fatigue. Furthermore, the effect of moisture or water on properties of reinforced composite materials with glass fibers is an important issue since such environmental factors are generally present in actual service conditions [14]. The water molecules present in a composite material quickly occupy the compound interface between the fiber and the resin due to the capillary phenomenon [15]. The interphase fiber / matrix can undergo a deepening and can be degraded by a hydrolysis reaction of the unsaturated groups in the resin [16-18]. It therefore seems essential that the composite materials retain their mechanical properties with less degradation in a humid environment or exposure to water. The subject of this work is to study the flexural fatigue behavior of composite with polyester matrix and multidirectional reinforcement of short glass in different waste immersion time.

2. Material and Methods

The materiel using in this study is a composite for polyester matrix reinforced with four layers of short multidirectional glass fibers, randomly oriented and manufactured by contact molding method. The molded plates are square of 300 x 300 mm^2, with a thickness of 4 ± 0.5 mm and a volume fraction of 30% for glass fibers. The specimens were cut to 80 mm length and 15 mm width as illustrated in Fig. 1,and as recommended by norm [19].

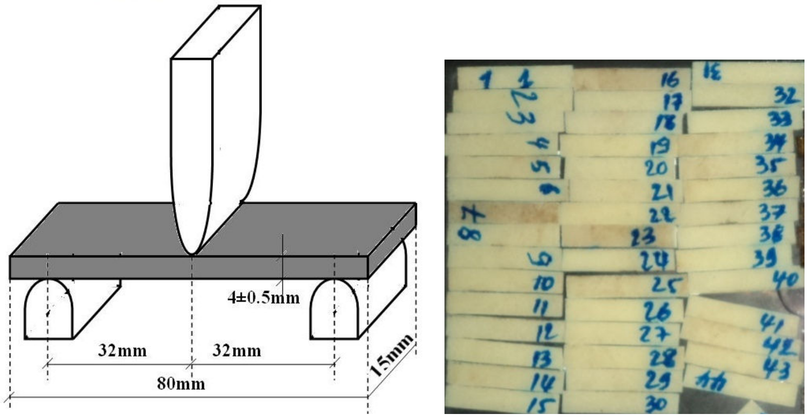


Fig. 1. Dimension specimen glass/polyester test in fatigue loading.

The specimens were immerged in a freshwater tank for 90, 180 and 270 days, respectively. The cyclic tests were carried out in 3-point bending on a machine type Zwick / Roell with a capacity of 20 KN, controlled by computer with software " test expert ", the error percentage of the tests is 1%. The distance between supports is 64 mm. Figure 2 shows the complete device used.

The specimens were loaded and unloaded repeatedly with a sinusoidal signal, a ratio R = 0 between the minimum and the maximum forces. The loading frequency was set to a minimum of 75 cycles/min, (frequency 1.25Hz). The choice of the low frequency avoids any side effects mainly due to the heating of the material. Figure 3 shows a schematic of the signal used.

The test specimen underwent various levels of loading compared to the maximum loading of three points bending test, that is to say, 80%, 70%, 60%, 55%, 45%, 35% and 25%. For each level of load, a minimum of three specimens was tested.

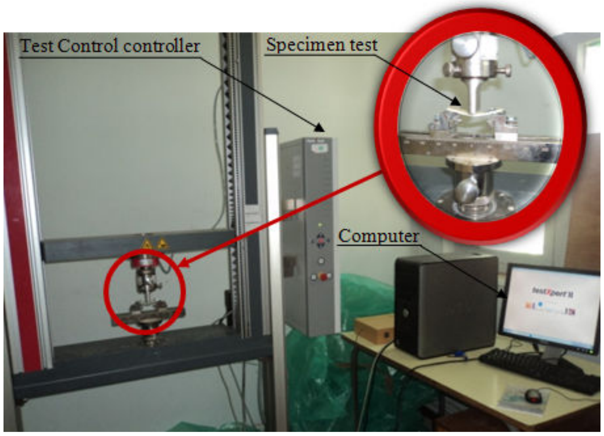


Fig. 2. Complete device in bending test.

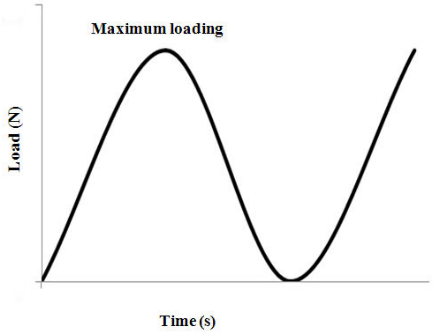


Fig. 3. Sinusoidal sign.

3. Results and Discussion

The glass polyester composite using in this research with important number of sample test provides several significant results.

3.1. Absorption of water

The whole of the test-specimens immerged in water present a rate of absorption varied according to the time of immersion, this variation is according to the chemical nature of the material. The rate of increase in the mass of the samples ρ according to their time of immersion in water was calculated by the following formula: $\rho = (M_f - M_i)/M_f$, M_f represents the final mass of the sample after immersion in water and Mi its the initial mass before immersion in water. Table 1 gathers the results of measurement for the whole of the times of immersion. All the specimens immersed in water present a variety of absorption by the immersion time, this variation is a function of the chemical nature of the material.

Table 1. Rate of increase in the mass of the composite glass/polyester according to the time of immersion in water.

Immersion time (days)	Rate of increase ρ (%)
0	0
90	3.4
180	4.1
270	5.9

The change of specimen weight is apparent. It thus appears that the absorption in a short time as used in this investigation is accompanied a gradual and monotonic increase with increasing days of immersion. Prolonged immersion in water can eventually saturate the specimen. It is still likely that when the compounds are immersed in water, the molecules may be attracted by the hydrophilic groups in the glass fibers and the unsaturated polyester.

3.2. Wohler curve

To get the maximum bending force using in fatigue test, (5) five simple test were tested in three-point static bending. Figure 4 shows an example of loading force - deflection curve obtained by three-point static bending. This curve shows a first low linear part at the beginning of the trial translating the elastic behavior of the material followed by a second slightly smaller slope, but higher, reflecting the evolution of the progressive damage that occurs in the composite form of microcracking of the matrix debonding and fracture of the fiber-matrix interface, chunking and fiber breakage and accumulation will cause breakage maximum load.

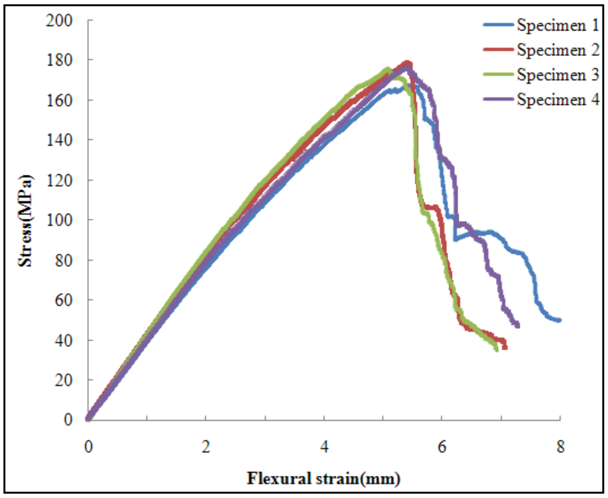
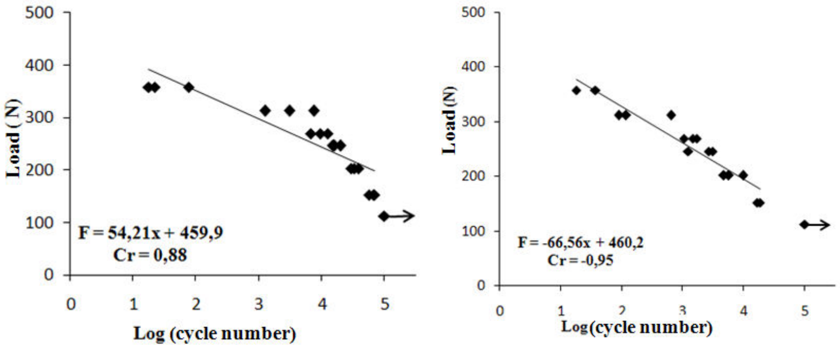
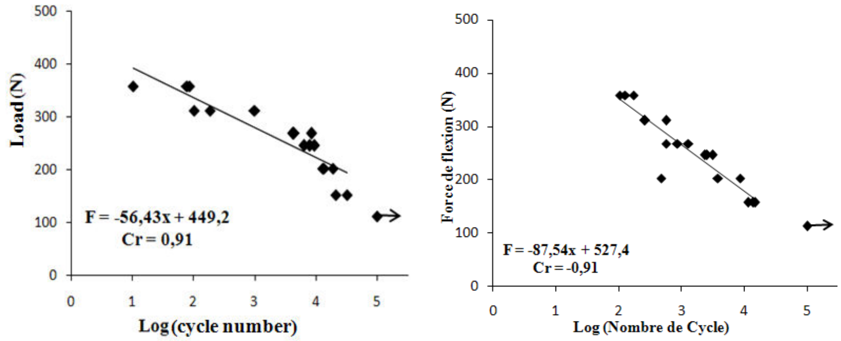


Fig. 4. Stress-strain curve in bending test.

Figure 5 shows the Wohler curves traced as the maximum cyclic bending charge against the logarithm number of cycles. The criterion adopted in this study is complete fracture of the specimen (Fig. 6). However, it should be noted that after 10^5 cycles and for practical reasons in the laboratory, the test is stopped even if the specimen is not broken. We consider that there's no fracture. It is important to note that different models are proposed to describe and represent the results of the fatigue endurance testing as Basquin equation that accounts of an asymptotic branch for high numbers of cycles [20].



(a) Dry environment. **(c) Immersion for 180 days.**



(b) Immersion for 90 days. **(d) Immersion for 270 days.**

Fig. 5. Wohler curve for glass polyester composite.

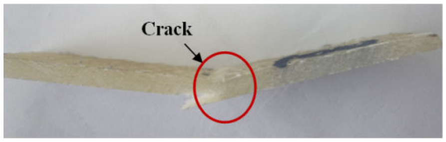


Fig. 6. Damage specimen in fatigue test.

Generally, the mathematical form adapted to describe the fatigue endurance results is the equation given by Wöhler.

$$F_{max} = - B \log (N_R) + A. \qquad (1)$$

F_{max} and N_R are respectively the maximum level of bending load and the life time. A and B are constants of the material. For some authors, the parameter A is the force in static loading. These coefficients are identified for each case of immersion in water by a straight regression performed on the experimental points. Table 2 shows the values of the coefficients *A* and *B*.

Table 2. *A* and *B* parameters values.

Immersion time (days)	*B*	*A*
0	54	460
90	56.5	449
180	66.5	460
270	87.5	527

The correlation coefficients of the Wohler curve for the all cases studied presents a value Cr = 0,88 ~ 0,91 which translate that this type of composite material has a less important heterogeneity, these coefficients reflecting the dispersion nature of the results of life between the specimens and it is the consequence of the heterogeneity of the studied composites. The characteristics of the specimens such as fiber volume fraction and orientation, distribution and the density of defects and the static mechanical strength are still not comparable from the specimen to the other. The damage state fatigue in general is characterized by a combination of density and orientation of micro cracks seem to be affected by the load and the test pieces of packaging conditions. This damage in the case of the studied composites is mainly due to micro cracking mechanisms of the matrix interfacial debonding. The accumulation and development of these progressive degradation mechanisms give the material kinetics of damage that can be conditioned by its microstructure and the type of load, it can be also identified from the experimental results. According to N. Benabdi and all [21] it appears that the stages of evolution of the damage in the case of cyclic loading on composite plate of carbon / epoxy have the same nature as those encountered in static loading. The composites deterioration occurs in the early fatigue loading cycles and increases gradually until the final fracture. It is characterized by three stages of progressive evolution.

It is clear to announce that the dispersion phenomenon makes that the Wöhler curve cannot estimate with a good probability of the lifetime prediction of multidirectional composite materials. However the alignment of this curve can always be used as comparisons corresponding to variations of compositions, variations in rates reinforcements, variations in resin nature, frequency test, parameters of cycling.

By comparing the slopes of the straight regression lines of the Wöhler curves (*B* coefficient in Table 2), it appears that the glass-polyester composite material presents the weakest slope for the dry environment, followed by a respective order of composite material immersed in water during 90 days, 180 days and finally 270 days. This report joined the behavior in statics of these materials indicating the negative effect of water on the long-term resistance of the glass-polyester composite materials.

In addition, the water absorbed by the material causes difference swelling of the fiber between a volume element close to the exposure moister and a volume element away from the exposure moister, so a difference in water gradient concentration causing microcracks to the various interfaces within the material.

Water molecules penetrate the macromolecular network and mitigate the weak interactions between polar groups carried by different polymer chains that may create a partial debonding of the polymer structure and an increase in local movements polyesters channels [22] and accompanied by a decrease of the yield stress and elastic modulus, an increase in the strain to failure, and a decrease of the shear stress [23].

3.3. Degradation:

Water enters the composite by diffusion and attack the chemical structure of the polyester and the glass fiber reinforcement by hydrolysis of the ester link. This penetration causes swelling of the resin and increase the weight of the composite with a degradation of their properties.

The glass fibers are generally composed of metal oxides or alkali dissipated in a silica system, according to the theory of Zachariasen [24], an example of a three-dimensional random system of oxygen tetrahedra (atoms "o" on Fig. 7), whose centers are occupied by Si^{4+} ions and Al^{3+} ions (atoms " ● "); sodium atoms Na+Calcium ca^{2+} and K^{+}potassium, occupy the interstices system (atoms "•") [25].

An oxygen atom is bonded either to two silicon atoms (Si) by covalent bonds, or an atom (Si) (covalent bond) and an alkali metal ion with an ionic bond. Pure water "washing" the glass: the weakly bound alkali ions diffuse to the surface, creating a depletion layer alkaline to a few atomic layers on the surface. In this process, OH^- ions are released:

$$Si - O - Na^{+} + H_2O \rightarrow SiOH + Na^{+}, OH$$

The extraction of alkaline metallic oxides actuate starters of rupture inside by the formation of the hydrogen bond (silanols) [26] which cause a reduction in resistance to the propagation of the cracks of the fiber glass and consequently a degradation of the interface fiber/matrix.

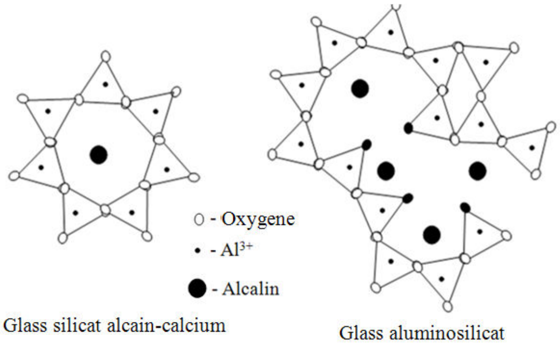


Fig. 7. Structure of a glass fiber.

This behavior may also be highlighted in another way. Indeed, taking the intercept of the straight Wöhler as the break load in three point satatic bending test as reported in the literature, Wöhler equation can also be written [27]:

$$F_{max}/A = 1 - (B/A).LogN_R \quad (2)$$

This form of equation used to draw straight monotone decreasing normalized slope - *(B / A)* from 1 ordered that is to say from 1 cycle corresponding to the fact static bending load (extrapolation ¼ cycle) for all considered environments. This allows to identify a constant degradation rate per cycle of decade for each case, quantified by the slope *B / A* value in absolute value. Table 3 gives the values of the rate of degradation increases with the increase of immersion time.

Table 3. Degradation rate B/A.

Immersion time (days)	B/A (%)
0	12
90	13
180	15
270	17

The water causes a negative effect in the polyester glass composite. The increasing of immersion time allows water to diffuse into the structure of the material and to alter the fiber matrix interface that degrades over time and gradually affecting the performance of the material and reducing the effect of a progressive and diffuse damage.

Moreover, the experimental points couples degradation rates depending on the immersion time (B / A; T) of Table 3 are plotted on a plane arranged approximately in a straight as shown by their tendency illustrated in Fig. 8. It is given with a good correlation coefficient very close to one.

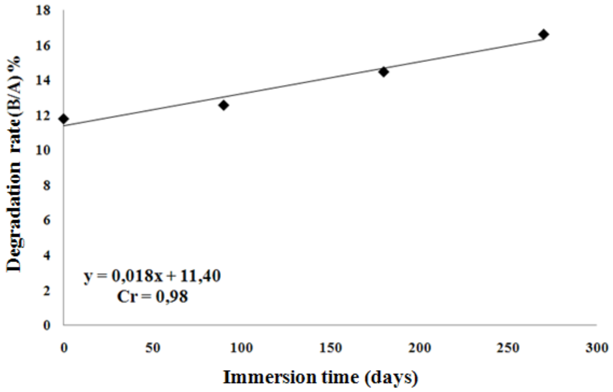
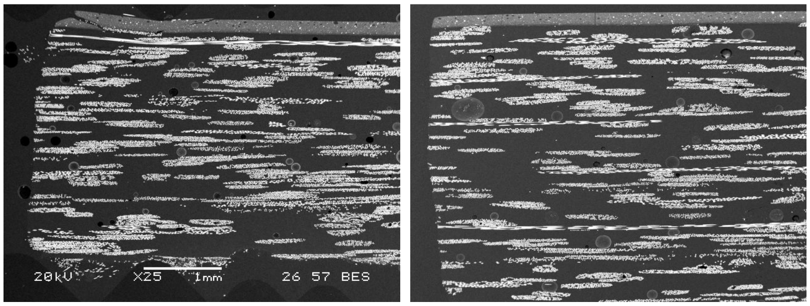


Fig. 8. Degradation rate according to immersion time in water.

Thus the rate of degradation seems to evolve or operate at constant speed. Finally it should be noted that it does not seems to be a specific endurance limit for each case, as in the four cases studied, the samples tested at 25% strain of the static stress as the maximum value are cycling arrivals up to 10^5 cycles without breaking the specimen.

3.4. Fracture surface:

Figure 9 shows the fracture surface of broken specimen of the glass/polyester composite in fatigue test before and after the water immersion. The photos were taken by using the scanning electron microscope.



(a) Before immersion. (b) After 90 day immersion in water.

Fig. 9. Fracture surface of broken section in fatigue test.

With the increase of immersion time, the water molecules enter first into the free space of micro voids formed by cavities and cracks in the matrix, at the same time, the water molecules can rapidly penetrate and spread along the interface due to the capillarity, they increase the weight of the specimen. On may be noted in Fig. 9(b) increasing the thickness of the test which is due to the plastic swelling of the polyester resin causes a widening of unreinforced sections. It is understood that long time water immersion will deteriorate the matrix, the reinforcing material as well as the interface. This would discourage the peeling strength of the composites [28.]

4. Conclusions

These exploratory works have to approach the fatigue study of composite glass reinforcements within a thermoplastic polyester resin. They have sought to observe the behavior of these materials under repeated cyclic loading and the effect of immersion time in the water on the damage evolution and endurance of these composites. The following conclusions can be gotten:

- The result in fatigue test shows dispersion characteristic of these composites in cyclic loading.
- The mass of specimens tested increase with increasing immersion time in the water.
- The results of the cyclic tests (Wöhler curves) are modeled by linear regression lines that give the middle part a pretty good representation.
- These materials are characterized by a constant rate of degradation during their lifetime. This rate appears to increase with immersion time at a constant speed.

Fatigue approach by the mechanics of the damage is to be taken based on the measurement of the evolution of deformation during fatigue history. Such an approach could lead to the creation of a model for prevision the lifetime based on the gradual accumulation of damage that will represent an alternative to unreliable method of Wöhler which will certainly be a very important fundamental result.

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