Recueil De Mécanique
El Wancharissi University
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Research Paper DOI : 10.5281/zenodo.5048314 Open access

Study of changes of mechanical properties of an aged composite by digital image correlation

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ARTICLE INFO

Article history:

Received 04 April 21

Received in revised form 21 May 21

Accepted 02 june 21

Keywords:

Composite, fiberglass, orthotropic, digital image correlation, aging

ABSTRACT

The orthotropic fiberglass composites are of great interest in different contemporary industrial fields. During their use and under the effect of external stresses, such as heat and mechanical stresses, these materials can undergo structural changes that can alter their physical and mechanical properties. In the present work, mechanical characterizations were carried out on orthotropic composite plates in polyester fiberglass subjected to a heat treatment in ovens at different temperatures. This shows how aging can affect the mechanical properties of the composite material such as longitudinal modulus of elasticity, stress and strain at break.

Analysis of the results of different characterization methods such as tensile test with extensometer and also digital image correlation by 7D software allows the understanding of degradation mechanisms mainly based on changes in internal structure, geometry, boundary conditions and external constraints that are applied.

1. Introduction

In their environment of application, polyester fibreglass composites are often subjected to significant thermal stresses. However, as for most of the plastic materials, these composites are sensitive to the environmental external factors such as sunlight, heat and mechanical stresses. To anticipate or prevent failure of the material and to estimate its service lifetime under its aspect of manufactured product, aging studies can be regarded as a good method to understand these different aspects. Hence, during an aging protocol, test pieces can be picked-up at regular intervals to perform characterization and to estimate the progress of aging. The evaluation of fibre reinforced polymer (FRP) resistance to thermal aging at elevated temperatures and relatively long processing times are conducted using unidirectional tensile stresses.

An image analysis performed using the 7D software developed in the SYMME laboratory is used during the experimental tests. The method is combined with a classical extensometer and the use of these two techniques reveals the variations of the mechanical properties of the composite depending on treatment temperature. Therefore, the aim of this work is to show the changes in terms of longitudinal Young's modulus, strain and stress at fracture and thermal properties of a polyester fibreglass compositee aged thermally and to highlight the effects of the catalyst concentration on the aging rate. Hence, the analysis of the results got by these different characterization techniques allows understanding how aging affects the molecular and morphological structure, but even more, what are their effects on the mechanical properties. Finally, one can understand or highlight the relationship existing between structure and properties.

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2. Methodology

2.1. Molding method used for the preparation of the composite material

Contact molding (Fig. 1) is the process used to make the composite [1]. The reinforcement is manually placed in a mold previously lined with a gel coat. Then, the resin is added making sure to penetrate the reinforcement. The resin is compacted with a roller to remove air bubbles. This type of process makes it possible to process a small number of series of large pieces. Nevertheless, the ability of the operator influences the quality of the product. The way in which one deposits the reinforcement in the mold and applies pressure on the roll during the compaction of the whole greatly influences the final properties of the piece.

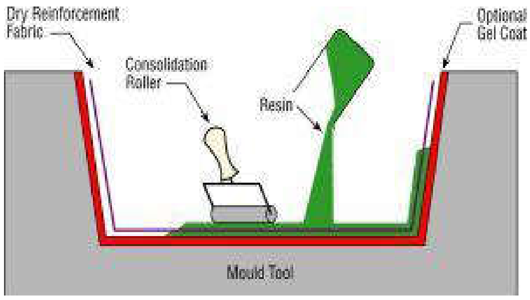


Fig. 1 – Principle of contact molding [1].

Unidirectional cross-ply composites are made by contact molding. Gel coat (to facilitate demolding after drying) is deposited on the surface of the mold, and then placed sheets of pre-impregnated fibers [2, 3]. Hardening is done at room temperature. The percentage of fibers and resin [40% volume of glass fibers (E = 73 GPa), 60% volume of resin (E = 3.53 GPa)] to constitute the composite was calculated so as to optimize the properties of the laminate.

The gel coat used has the trade name MOLD-WIZ F57 NC. The molds are made in aluminum and measure 500 x 500 mm² (Fig. 2). The fiberglass is marketed in roll form, so for each plate to develop six sheets of 380 x 250 mm² are cut. The molding consists of successively depositing on one side of the mold a layer of resin, then a reinforcing layer of fiberglass. Impregnation of the reinforcement is a manual operation using a steel toothed wheel roller and a brush (de-boiling and rolling) to remove larger bubbles [4]. This operation is repeated as many times as there are reinforcing layers until the desired plate thickness is obtained. In our case, 6 layers of 2 materials polyester resin and fiberglass (from 0 ° / 90 ° and 0 ° / 45 ° / 90 ° / -45 ° / 0 ° / 90 ° to the directions of the fibers) were thus bunk. The counter-mold is put in place after the laying of these folds and without any tightening.

Fig. 2 – Preparation of the composite: weighing of the hardener, coating of the gel-coat mold, then deposition of the fiberglass.

After remolding the material, it is allowed to dry either in ambient air or preferably in an oven at 40 ° C for 2 hours. The material developed for this study is presented in Figure 3.

Fig. 3 – Composite material of the study.

2.2. Mechanical analysis of material

Mechanical testing of composites was performed on an INSTRON 5569 tensile machine with a 50 kN force cell (Fig. 4) and a 12.5 mm wide extensometer to measure strains. At least three specimens were solicited under identical conditions. This precaution makes it possible to calculate the dispersion and therefore to represent our results with standard deviations. In order to refine our results, the specimens with too much dispersion were eliminated and the test renewed.

2.2.1. Uniaxial tensile test with image correlation

The standard NF EN 2747 sets the test conditions with a test speed of 2 mm / min and at an ambient temperature of 22°C. The tensile test with imaging has the advantage of acquiring the strains in two different ways, one with the extensometer and the other one by image analysis. Access to mechanical quantities is therefore done by these two methods.

Fig. 4 – INSTRON tensile machine.

Unidirectional tensile tests on specimens tested in the transverse direction tend to stress more particularly the fiber-matrix interface of the composite. The Young's modulus, maximum stress and strain at fracture are respectively denoted E_T, σ_T and ε_T. Tensile tests in the longitudinal direction preferentially solicit the fibers and give access to the same quantities respectively denoted E_L, σ_L and ε_L [5]. The simplest expressions of the modulus of elasticity of the longitudinal or transverse directions are those given by the law of the mixtures. It is assumed for E_L a uniform and identical deformation of the fibers and the matrix (Equation 1) and for E_T that the stress in the transverse direction is transmitted to the fibers and to the matrix, ie the equality of the stresses in the fibers and in the matrix (Equation 2):

$$E_L = \Phi v_f E_f + (1 - \Phi v_f) E_m \quad (1)$$
$$E_T = E_m / [1 + (E_m/E_f - 1)\, \Phi v_f] \quad (2)$$

Where E_m is Young's modulus of the matrix, E_f Young's modulus of the fiber and Φv_f is the volume percentage of fibers. The relation of the law of the mixtures for the longitudinal modulus is verified by the experiment whereas an empirical model (Equation 3) seems in better agreement with the experiment for the transversal module in the case of Polyester systems according to Barrere-Tricca .

$$E_T = [E_m + 0{,}2\ \Phi v_f (1 - \Phi v_f)\ E_f (1 - E_m/E_f)^2] / [1 - \Phi v_f (1 - E_m/E_f)] \quad (3)$$

The specimens are cut from the plates made with a diamond disk under a water jet according to the dimensions recommended by ISO 524.4 for traction. Fig.5 illustrates the dimensions of the tensile test piece of this study.

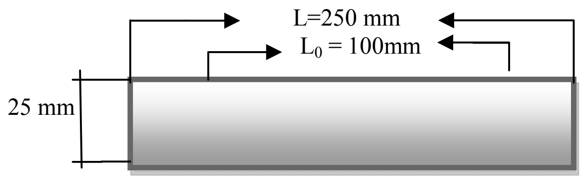


Fig. 5 – Composite tensile specimen with a thickness of 3.2 mm.

2.2.2. Experimental method of imaging

Sample preparation: In order to obtain a surface state that can be used for digital image correlation method (random gray pattern, unique and small enough depending on the resolution of the results you want to obtain) a speckle is placed on each specimen. It is a black acrylic paint mixed with fine white polymer particles of about 7 µm in size. Fig. 6 shows an example of speckling obtained on the surface of a fiberglass / polyester composite test piece.

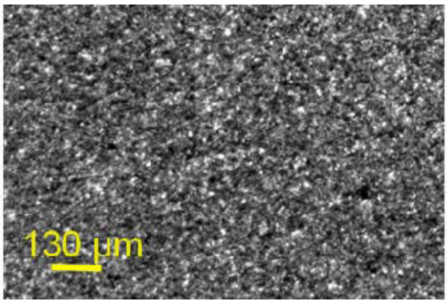


Fig. 6 – Speckling on a specimen.

Taking pictures: The acquisition of images is carried out with a high definition camera of 10 Mpixels (4004 pixels x 2670 pixels) with a constant focal length of 80 mm (Fig. 7), fixed on a plate allowing to make focusing with micrometer screws. The specimen is illuminated by a ring of white LEDs to prevent gray scale variation. The magnification obtained is 2.6 µm for 1 pixel. The images taken by the camera cover an area of 10.6 mm x 7.1 mm.

Fig. 7– Ultra-fast imaging camera.

3. Results & discussions

3.1. Tensile test results for specimens aged at 100 ° C, fiber direction 0 ° / 45 ° / 90 ° / -45 ° / 0 ° / -45 °

The tensile tests on the fiberglass / polyester composite aged at 100°C were carried out with the same tensile machine and the same test speed of 2 mm / min, at ambient temperature of 22°C. The strain measurements are carried out using an extensometer attached to the center of each test piece. The curves stress versus strain for tensile test for samples with 2% catalyst is shown in Fig 8.

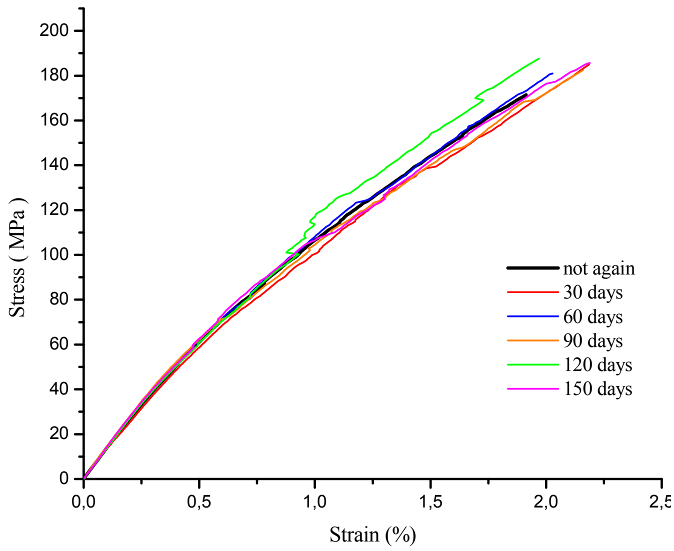


Fig. 8 – Stress as a function of strain of the specimens aged at 100°C of the polyester fiberglass composite of 2% of catalyst.

There is a variation in the results, but there is no order in the curves with respect to aging time; this may be due to the dispersion of the results that can be observed on the composites (very heterogeneous materials).

2.2 Results of 7D image analysis on tensile test

2.2.1. Presentation of the 7D software:

"SevenDi" or "7D" is digital image analysis software which main function is to measure displacements to calculate strains on plane surfaces. The simple recording of two digital images of the same object whose flat surface has a contrasting pattern is sufficient to determine displacement maps in pixels between these two images. The search for a correspondent between two points (matching principle) is obtained by comparing the gray levels. This information in terms of displacements is analyzed by the software, either to define strain fields, or to carry out 3D scans in the case where there are the presence of two cameras [6].

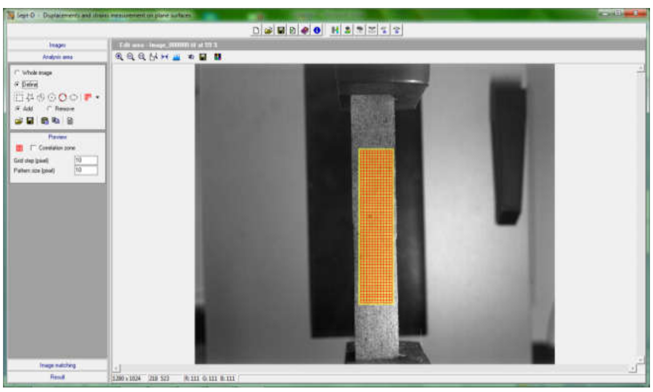


Fig. 9 – Choice of the number of mesh elements for image analysis.

The surface analysis operation with the 7D software, which calculates the displacements and strains of the specimen following the mesh (Fig. 9), for all the images taken during the test according to the 1st image [6]. These analyzes allowed us to find different results: the displacement along the Y axis (axis of the test piece) which is presented in Fig. 10, the displacement vectors which are well in the axis of the specimen, but also the maximum and minimum strains.

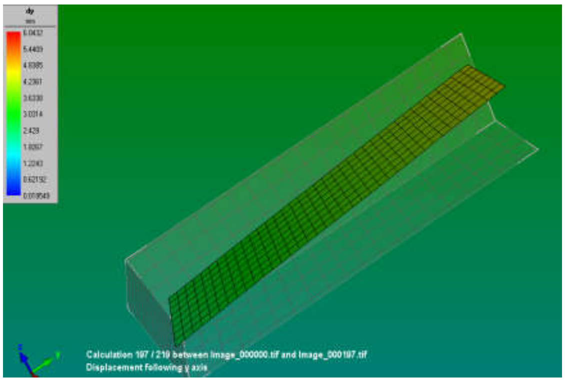
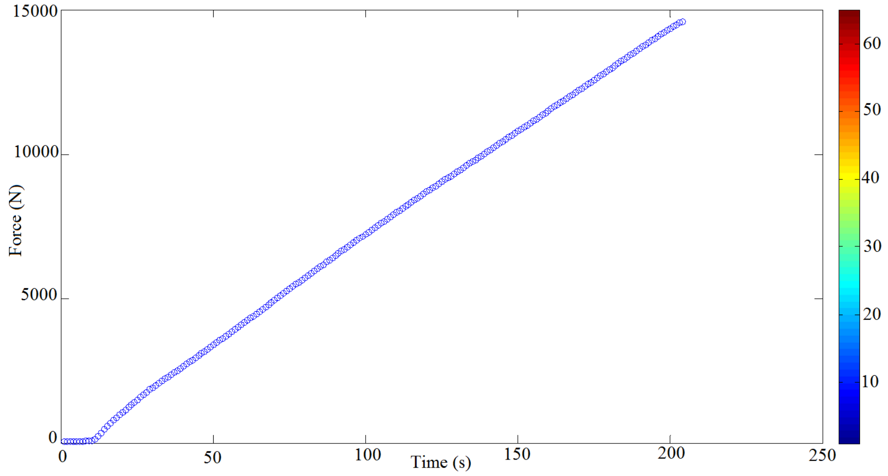


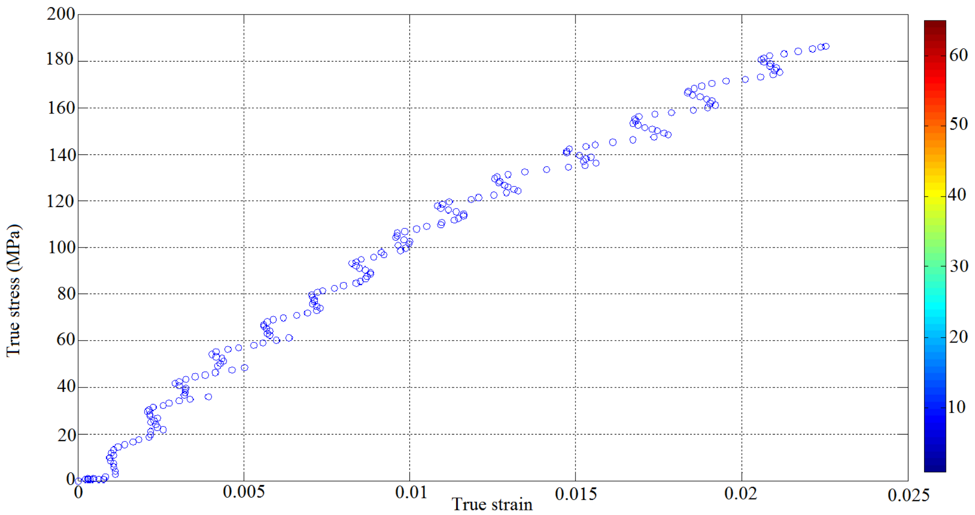
Fig. 10 –Displacement along the Y axis analyzed by the 7D software.

2.2.2. Results of the analysis of the specimens at 2% of catalyst and for the directions of fibers at 0 ° / 45 ° / 90 ° / -45 ° / 0 ° / 45 °

The results (Fig. 11) found after analysis by the software 7D, and especially the axial deformation part in the direction of the Y axis, have regular waves because of the nature of the composite material which is very resistant to strain. Also, the orthotropic and non-plasticity of the composite and the change of directions of the fibers in the layers of the reinforcements inside the material are other causes of the non-linearity of the curves found [7].



b)



a)

Fig. 11– a- Time force curve of the 3% composite, unaged, b- True stress curve / true strain analyzed by 7D of the 3% composite, unaged.

Figures 12 to 14 show the tensile test image analysis results for the 2% polyester fiberglass composite for the 0 ° / 45 ° / 90 ° / -45 ° / 0 fiber directions. ° / 45 °, treated at 100 ° C and for aging conditions of up to 150 days. On each figure, there is the superposition of 3 curves:

- Exp.: nominal stress curve / nominal strain obtained with extensometer
- 7D: true stress / true strain curve obtained with
- Poly. : Interpolation of the 7D curve.

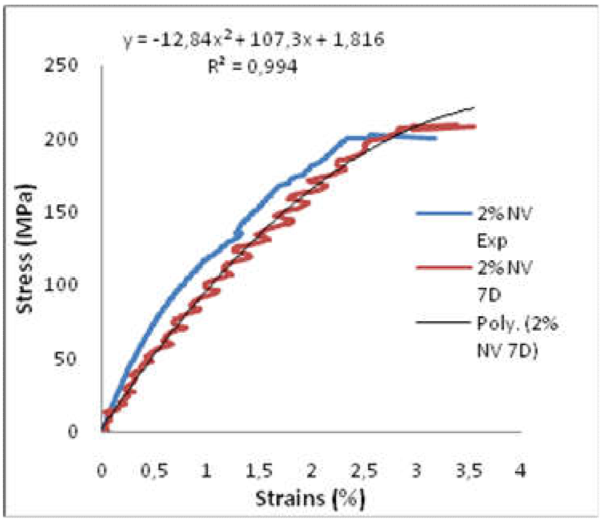


Fig. 12– Stress curves - extensometer strains and 7D analysis and interpolation of a tensile test of polyester fiberglass at 2% catalyst, and not aged.

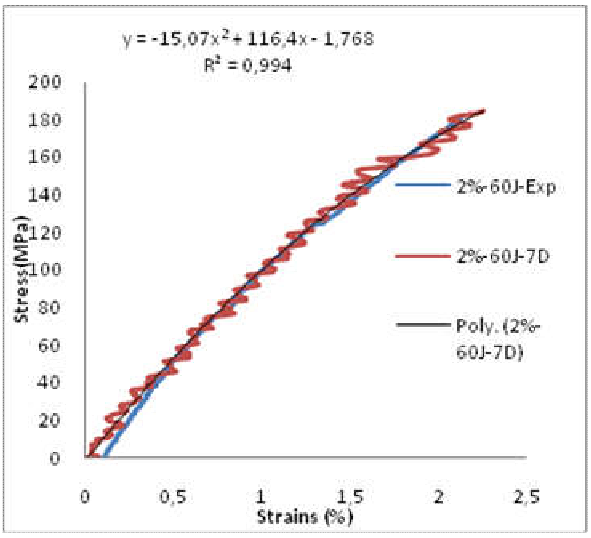


Fig. 13 – Stress curves - Strains extensometer and 7D analysis and interpolation of a tensile test of polyester fiberglass at 2% catalyst, and aged for 120 days.

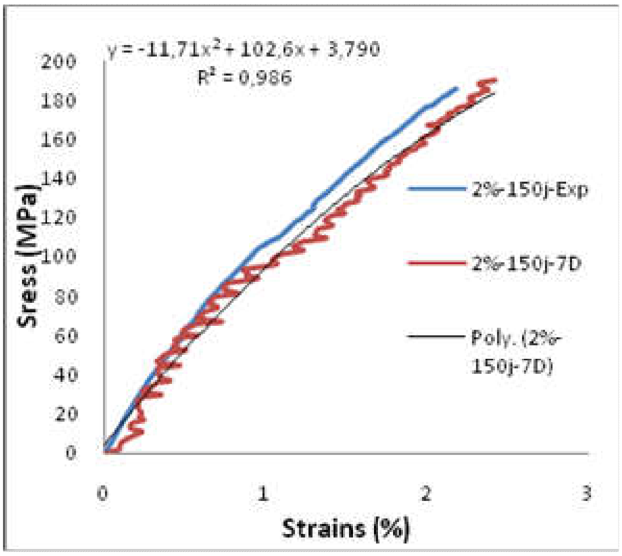


Fig. 14 – Stress Curves - Extensometer Strains and 7D Analysis and Interpolation of a Tensile Test of 2% Catalyst Glass Fiber, and Aged for 150 days.

These curves give ranges of approaches that vary between small and medium between the two experimental results of strain measurements of the material with extensometer and 7D image analyses. The experimental results found with the extensometer were measured in one section of the specimen (over the length of the extensometer, ie 12.5 mm), while the results found by image analysis are distributed on a very narrow cross-section in which the specimen deforms the more, and thus gives more accurate strains because more local strains.

Table 1 gives the different mechanical characteristics found with extensometer and 7D image analysis for the 2% catalyst material.

Table1 - Mechanical parameters for tensile test for 2% catalyst material, V = 2mm / Min, Temp. = 22°C

Composite of 2% of catalyst	Max stress (MPa) Exto	Max true stress (MPa) 7D	Max nominal strain (%) Extenso	Max true strain (%) 7D
Not aged	202.61	207.67	3.18	3.55
Aged 30 days	185.04	188.77	2.29	2.47
Aged 60 days	181	184.87	2.135	2.224
Aged 90 days	182.48	186.69	2.15	2.38
Aged 120 days	187.35	193.26	1.96	2.25
Aged 150 days	185.67	190.02	2.11	2.42

These results make it possible to do an analysis which gives the variation of the mechanical characteristics with respect to the thermal aging time at 100°C, and so an analysis of the behavior of the material. Referring to Fig. 15, the variation of the two stresses at fracture: nominal and true as a function of aging time.

In Fig. 16 shows the variation of the 2 tensile strains of the composite material at 2% of catalyst as a function of the aging time: maximum nominal strain obtained with extensometer method and true strain obtained with 7D method.

The variation of Young's modulus following the X axis E_L versus aging time is shown in Fig.17; we can notice an increase in longitudinal Young's modulus during the 90 days of thermal aging after that, we notice a stability of this module by compared to the time of exposure to heat.

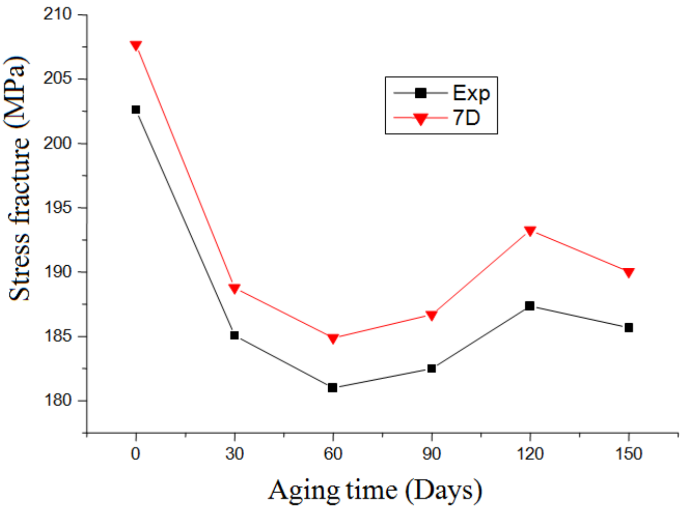


Fig.15 – Variation of the nominal and true maximum stresses with respect to aging time.

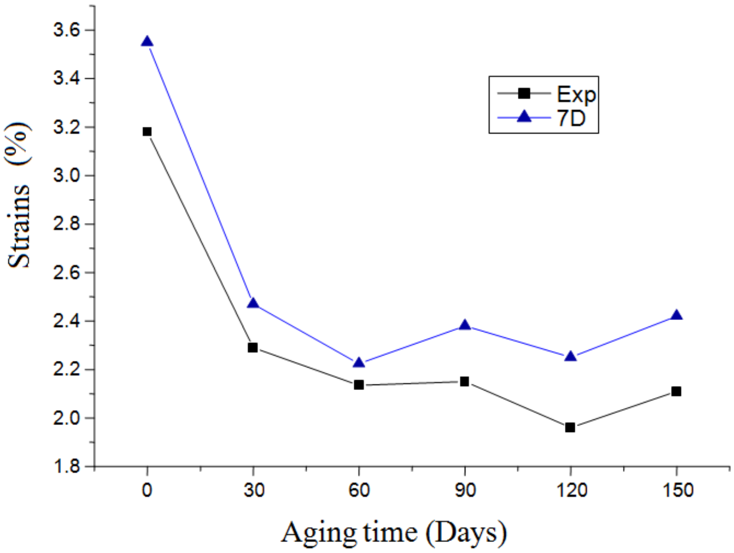


Fig. 16 – Max strains variation with respect to aging time.

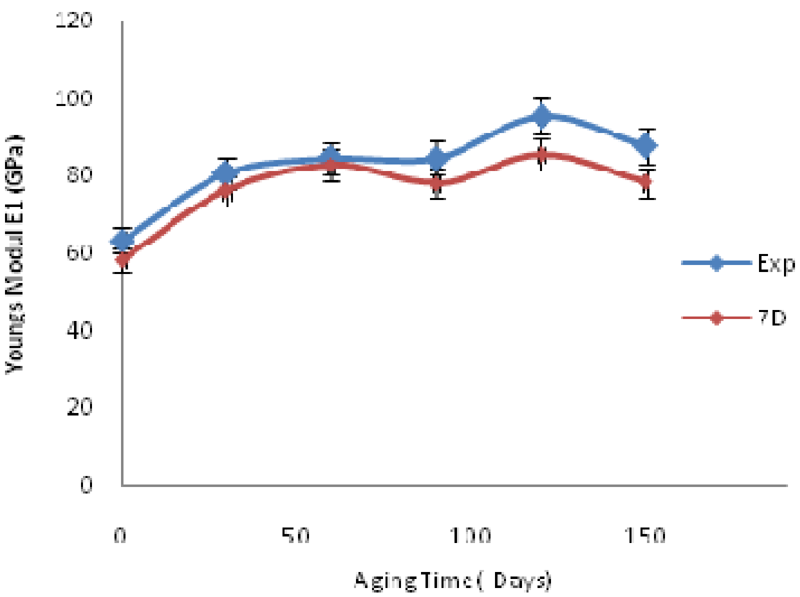


Fig. 17 – Young's modulus E_L versus aging time.

These results show that there is a decrease in the properties of the composites after 60 days of thermal aging at 100°C. This is characterized by a decrease in the maxi stress and the maxi strain. Beyond this duration, we note a stability of the mechanical characteristics which varies between 180 and 195 MPa for nominal and true breaking strengths and of 1.9 to 2.4% for the maximum strains. This variation is probably due to the molecular stability of the matrix reinforcement interfaces. Besides of this work, we obtained very similar results with 3% catalyst composite [8].

3 Conclusion

The heat treatment at 100°C of the composite material shows an evolution of the mechanical properties: a decrease in the quantities at break during the first 60 days is observed then the evolution fluctuates randomly beyond (sometimes decrease, sometimes reinforcement). In the same way, mechanical measurements made in 3-point bending vary, as for tensile test, with a regular decrease up to 60 days and then fluctuate beyond. It remains difficult, however, to explain the phenomenon of the reinforcement of the mechanical properties, but it is very likely that it is due to the microstructural changes of the polymer matrix which is the object of cross linking reactions favored by the rise in temperature. The image analysis performed using the 7D software during the tensile tests for strain measurements obtained experimentally with an extensometer confirms the experimental results [9]. These measurements made it possible to highlight the phenomena of deformation over the entire length of the surface of the test pieces. Also, it appears that the material loses enormously of its elasticity, contrary phenomenon for the Young's modulus which practically increases. Maybe some SEM images could give some explanations of what happens inside the resin and fibers according the thermal treatment.

In perspective, work to improve the mechanical and physical properties of fiberglass composites will be carried out, changing various parameters such as the percentage of hardener and glass fibers in the composite, in order to better resist the stresses exterior such as high temperature and erosion.

ACKNOWLEDGMENT

I would like to thank Professor Laurent Tabourot for his invitation at SYMME laboratory in Annecy. Our heartfelt thanks go to everyone who helped us to achieve this research work.

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