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CREEP TESTING OF EPOXY RESIN-FLAX FIBER COMPOSITES

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Abstract: *The present study proposes an experimental approach into determining the viscoelastic characteristics of a composite made out of an epoxy-resin matrix with long flax fiber reinforcement. In a first instance, the fabrication process will be discussed in detail, which consists of resin impregnation in the dry fiber and a thermo-compression. Afterwards, creep-recovery tests are performed at room temperature on the obtained samples in two sets of cyclic ramp loadings by varying, for the first one, the ramp load whilst keeping the creep time constant for each loading step and for the second one, a fixed load and a variable creep time. Reading of the elongation is made with the help of strain gages glued on the tested specimens for the entire duration of the experimenting session. Acoustic emission tests were performed during the creep phase for each load step in order to verify that there are no internal failures which might introduce plastic deformations. The samples are unidirectional, 0°, 90° and ±45° strips cut from the fabricated composite plates.*

Key words: *Creep, Flax Fiber, Fabrication, Composites, Epoxy, Bio-Material.*

1. INTRODUCTION

The introduction of flax fibers in composite materials is an idea which, even though a few decades of age, is being thoroughly analyzed at the moment from a mechanical point of view [1–3] and a conceptual one [4–6]. More and more articles are appearing with the suggestion of replacing glass fiber based composites with the vegetable based flax fiber, the arguments being that the latter is renewable, already has a developed industry, albeit for other uses, is environmentally friendly, easier to work with, for it is not abrasive to tools ,the shrapnel is not toxic for the workers handling it and it has a lower density than the already established glass fiber [7], making the components lighter, a big factor for transport industry.

From a mechanical point of view, however, flax fiber has proven to have a lower ultimate strength, comparable though with its artificial counterpart and a wider dissipation of values, caused by its organic nature [7]. Even so, it’s specific strength, thanks to the big difference in densities makes up for the inconvenient of lower ultimate strength.

The two mechanical factors only make the study of its properties all the more important for, the EU at least, are demanding an increase in bio materials use by introducing legislation with the purpose of reducing waste and the use of non-recyclable materials [9 and 10] making this inconvenient only a step necessary to overcome as soon as possible.

Along with the research conducted toward the discovery of potential applications and material properties, flax fiber based composites have proven to have a pronounced behavior in terms of time-dependent loading [11]. More specifically, a constant load applied for a longer period of time will keep on deforming the structure, the phenomenon being called creep. The present study proposes to analyze this behavior by testing unidirectional composite samples obtained through thermo-compression. Multiple ramp creep-recovery tests have been conducted by varying independently fiber direction, sample load and creep duration. The recovery period was approximately 48 hours for each loading step. Previous tests on other types of composites have shown that the same sample might be used more than once if there are no internal defects [12].

2. SAMPLE FABRICATION

The epoxy resin system used for the fabrication was Sicomin 8200 with a Sicomin 8205 hardener in a ratio of 100:31. It was used to impregnate the dry flax fibers, which were provided in a roll of 400 mm width and held together by cotton bands. These are considered to be of a too low proportion of the roll to have any significant influence to the overall mechanical properties of the composite.

The roll was cut to the wanted dimensions with the help of an electric scissors and then the edges were taped with duct tape so as not to unravel them during the impregnation or the fabrication process.

This mixture was then put in a mold which in turn was placed in the press for a curing cycle of 8 hours under a temperature of 60°C and a pressure of 4 bars. In this fashion, multiple 12 plied 400 × 400 mm plates were

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Fig. 1. One of the fabricated plates.

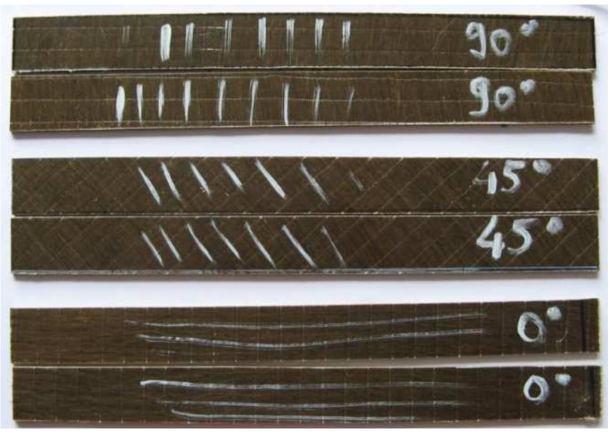

Fig. 2. 250 × 25 mm strips.

obtained for the 0° and the 90° directions (Fig. 1). Because of how the fiber mat was supplied, it was impossible to cut with the same size, in the case of the 45° samples and thus, three 400 × 120 mm plates were fabricated instead of a 400 × 400 mm one.

Two sample types were cut with the help of an electric saw, with multiple roles to play. First of all, 250 × 25 mm (Fig. 2) composite strips were cut, as many as possible from a single plate and as well 40 × 25 mm (Fig. 3) strips. The first type was used for viscoelastic characterization and the latter for volume fraction calculus of the plate of provenance and specimen tabs. After serving as samples for volume fraction determination, the tabs were glued with araldite on the specimens which were to be used in the tests. In order for the araldite to cure, a second curing cycle of an hour, at a temperature of 60°C was applied to the specimens. This second curing cycle does not affect the already cured composite specimens.

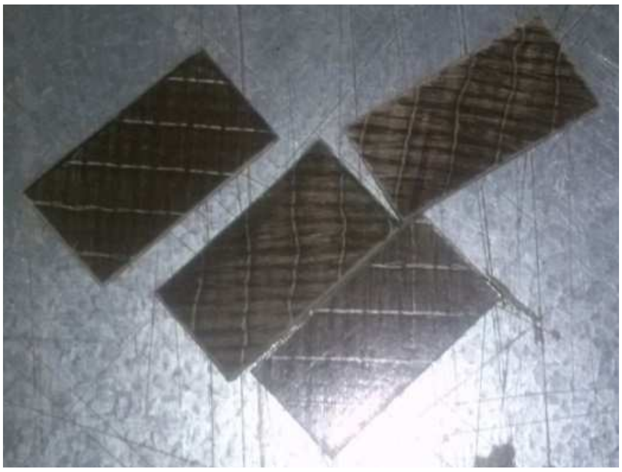

Fig. 3. Samples used for volume fraction and tabs.

It is worth mentioning that tab's fiber direction was the same as the specimen's, ensuring the same rigidity for the two. The reasoning behind using tabs from the same material and the same direction is that, in previous tests where aluminum ones were used, failure tended to occur close to the extremities. It was theorized to be caused by stress concentrators in that area because of the great difference in rigidity between the composite material and the tabs.

In order to read the elongation, each of the specimens used for testing was equipped with a strain gauge, glued in its middle.

The specimens used for testing can be seen in Fig. 4.

Fig. 4. One of the equipped specimens.

3. PRELIMINARY TESTS AND PREPARATIONS

As mentioned before, more than one sample was cut from the fabricated plates. In order to determine which ones are the best suited for the creep-relaxation tests, all of the strips were measured, before being fitted with gauges and tabs, with the help of a micrometer (for the sample thickness) and caliper (for the length and width). The reasoning behind these measures is connected with error minimization, by having samples with as small as possible variations in size and as close as possible to each other. A total of six samples were chosen, two for each fiber direction and were further equipped.

For the volume fraction determination, the tab's dimensions and the fiber mat's surface area were measured so as to determine the proportion of masses. In order to transform it into volumetric proportions, the densities, provided by the data sheets of the composite's constituents were used.

Also, traction tests on the samples have shown a clean failure zone in the middle of the gage length for all the tested specimens, confirming that stress concentrators might appear by using aluminum tabs.

4. EXPERIMENTS AND EQUIPMENT

Multi load creep – recovery tests have been conducted on the six prepared specimens as follows: a set of 0°, 90° and ±45° were subjected to an augmenting load for each load step, with creep duration of one hour, followed by 48 hours of recovery. The other set was subjected to a constant load, whilst augmenting the loading time and the same 48 hours recovery period. Details for each sample are shown in Table 1. The sample notation is as follows: [fibre_angle]_variable, followed by the parameter's value for the current load step.

The 48 hours period of recovery was chosen because of the big number of specimens to be tested and the length of the tests interference between the creep periods would have appeared for a lower duration.

As can be seen in Table 1, the number of load steps differs from one sample to another. In the case of specimen [90°] _ σ, at the next load level, of 22.5 MPa, the sample failed and it was thus impossible to use it any further. As for the others, with more than 10 charges, they were usable even after the initially proposed number of load steps and were considered viable readings.

The reading of the strain gauges was made with the help of a National Instruments PXI 1033 data acquisition box with 8 channels, which recorded for the entirety of the period, with an acquisition frequency of 5 Hz during experimentation days and 1 Hz during the night and the days with no experiments. The testing was made with the help of a MTS Criterion tensile testing machine, capable of adapting the test program to the desired conditions. The load, during the creep phase, was read with the help of a force cell of 10 kN of the testing rig. In order to translate the force in MPa, the read value was divided to the specimen's cross section, measure beforehand.

The raw data, read by the data acquisition box was then fed into a Matlab code, written for this session of experiments which, in a first step glued together all of the values read by the box, which are presented later on in this paper. It then divided the complete intervals into creep-recovery periods and load level intervals.

During the creep phase, the acoustic emission tests were performed on the specimens, by adding two microphones at the extremities of the gage length. Its role was to ensure that the load would not induce cracks or other material failures that could lead to plastic deformations to the specimen which would render load step data useless and the specimen as well for further use. It was considered that the sample would be damaged without the possibility of being reused after 100 events read on the acoustic tests.

Table 1

Specimen test conditions

Load step	[0°] _σ	[90°] _ σ	[45°] _ σ	[0°] _time	[90°] _time	[45°] _time
	σ [MPa]	σ [MPa]	σ [MPa]	t [min]	t [min]	t [min]
1	10	2.5	3	20	40	20
2	24	5	6	40	60	40
3	40	10	9	80	80	60
4	50	12.5	12	100	100	80
5	70	17.5	15	140	120	100
6	80	20	18	160	140	120
7	90		21	180	160	140
8	100		24	200	180	160
9	110		27	1356	200	180
10	140		30		2611	200
11			33			374
12			39			1416
13			45			

5. RESULTS AND DISCUSSIONS

The volume fraction calculus revealed that the samples had close values of 44±1%, considered acceptable for further investigation.

The acoustic emission tests performed during the creep period showed a negligible number of events, confirming that whatever plastic deformations might occur, they were not caused by internal failures.

The variable duration creep-recovery tests were designed to verify the existence of plastic deformation dependent of the creep period. Such a connection was not visible during the testing and thus, these results will not be disseminated.

However, as can be seen in Table 1, the last load step for each fiber direction has a great period of creep time, introduced in order to determine if the time frame was too small to produce any significant results. Neither this duration of more than 6 hours presented any plastic deformations greater than the reading error, let alone specimen failure.

Preliminary data showed that the three directions have a different behavior during the recovery phase, with the [0°] _σ specimen behaving the closest the smooth curve that was expected. In Fig. 5, the evolution in time can be seen. From load level 4 onward, a plastic deformation can be observed, which is considered bigger than the reading error. Another thing to note is that, after a certain period of time, the recovery seems to stabilize, phenomena more visible for the lower loading levels where, the deformation returns fast to 0 or close to it.

From a technical point of view, as can be seen in the same figure, the recovery period was not always 48 hours, the reason being that a long experimenting period as this one, which spanned over several weeks passed through weekends and vacation days, when it was impossible to do the experiment. This meant that the initial schedule had to be modified. As such, the recovery periods were between 48 and 72 hours, period which did not affect the overall results.

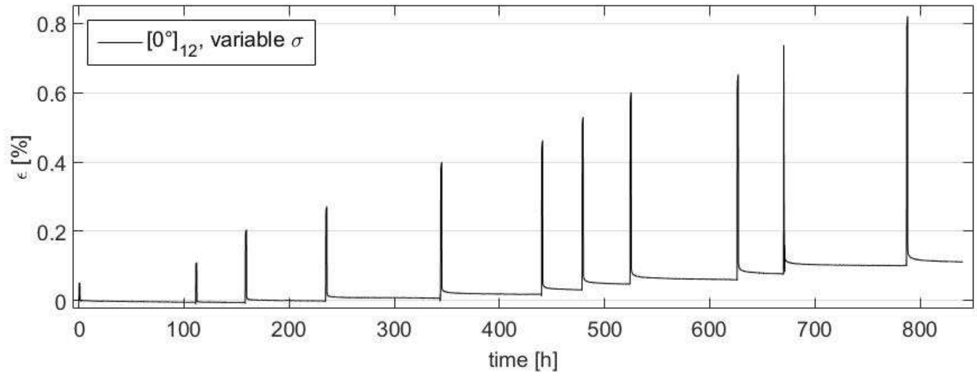

Fig. 5. Creep recovery periods for the [0°] _σ specimen.

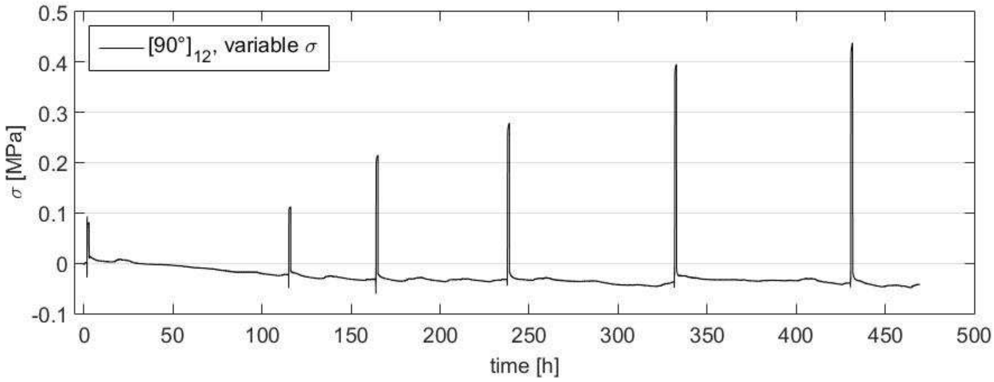

Fig. 6. Creep recovery periods for the [90°] _σ specimen.

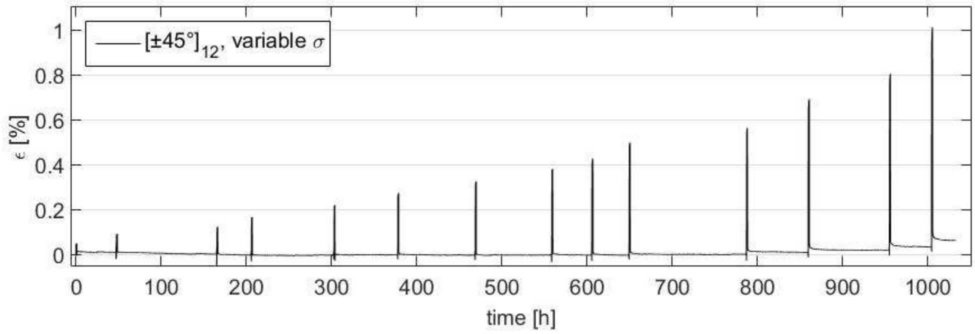

Fig. 7. Creep recovery periods for the [±45°] _σ specimen.

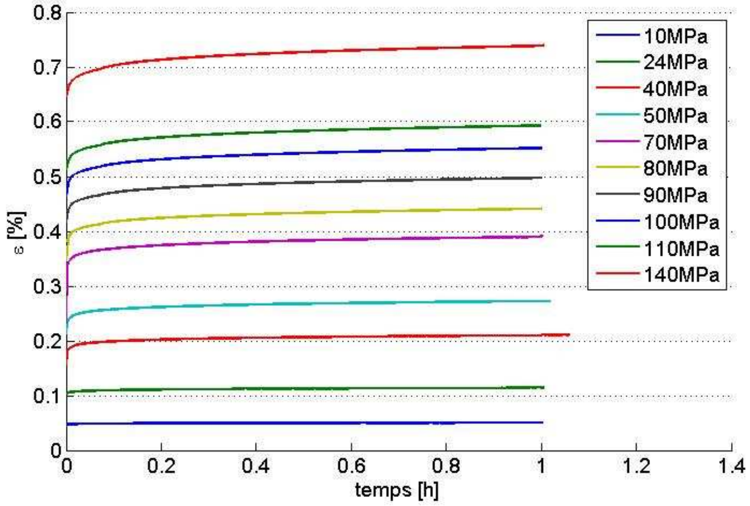

Fig. 8. Creep periods for [0°] _σ.

Figure 6 shows the creep recovery period for the 90° subject to a variable load. It can be seen that its behavior during the recovery period is far from a clean evolution and a return towards 0. A definite explanation was not found, the main theory for this behavior being that the material structure is unstable on this direction from a creep-recovery point of view. This behavior needs to be analyzed further in order to understand the mechanisms involved.

Finally, the specimen with a ±45° fiber direction is shown in Fig. 7. Its behavior is stable during the entirety of the testing period. The residual deformation tends to 0 for the majority of the load levels, except for the last 3, where a bigger than the reading error value can be seen. It is the main reason for which this specimen was used for more than 10 initial load steps. However, time could not allow increasing load until failure, for which no prediction has been made.

The creep period for all the load steps, for specimen [0°] _σ is shown in Fig. 8. It can be seen that, by increasing the load level, the deformation increases, both the instant one (at the beginning of the curve) and the transient one (during the creep period).

The last two load levels have been increased over the initial planned 100 MPa, for the specimen presented no defects and was thus tested until failure. This arrived at a value of 150 MPa, after a creep period of approximately 26 minutes.

6. CONCLUSIONS AND FUTURE DIRECTIONS OF RESEARCH

This paper presents the fabrication and experimental procedures for the creep characterization procedures conducted at the Superior Institute of Automotive and Transports, Nevers, France. It is the first part of a study which contains a rheological modeling with the help of Zener and Burger models.

The work has shown that flax fiber composites present a pronounced viscoelastic behavior, even at room temperature, highlighted with the help of creep-recovery tests on specimens with 0°, 90° ±45° fiber direction.

Depending on how the components are presented, certain precautions have to be taken into consideration, regarding the fiber unravel, desired volume fraction (which dictates the necessary resin quantity and curing cycle pressure), experimental procedure scheduling.

Although the creep period had a predictable variation, the recovery one seems to differ from one direction to another, as seen especially in the case of the 90° fiber direction.

First thing needed to be done in succession of this study is to analyze the reasons behind this inadvertence between the fiber directions. Once this is established, the desire is to use this experimental data for a model which could be used to design, from a time and load dependent point of view, load bearing structures made out of materials which present this kind of behavior.

As well, the influence of environment temperature needs to be taken into consideration for the glass fiber composites in use today are subject to values greater than the normal conditions in a laboratory and the epoxy resin systems tend to modify their behavior depending on this factor. An example application is that of glass fiber panels from the racing car bodies.

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