ON THE TIME-DEPENDANT CHARACTERISATION PROCEDURES OF BIO-COMPOSITE MATERIALS

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The present work aims to discuss the viscoelastic characterization procedures required for laminate composite materials. An analysis concerning the available tests is made, together with their implementability. Equipment requirements and recommendations are also examined. Particularities are presented for a series of creep-recovery experiments conducted on a bio-composite laminate with unidirectional flax fiber reinforcement. The tests were conducted on samples with a fiber direction of 0°. With the help of these experiments, a series of conclusions and good practice measures are drawn for the procedure and for future works concerning the time dependent behavior of fiber reinforced laminates.

Keywords: Bio-Composite, Creep, Flax Fiber, Experimental

1. Introduction

Composite materials are being regarded as the most performant class of materials, whose mechanical properties are tailorable on demand [1].

Out of these, the most interesting ones in terms of strength to weight ratio are polymer-based fiber reinforced laminated composite materials [2]. They are highly used in the transportation industry, where mass is one of the most important design criteria [3]. The expansion of this industry has been a catalyst for the laminate composite market as well which, in recent years, has brought to light two significant disadvantages of synthetical composites: they are not biodegradable and recycling is a challenge [4]. A possible solution to this problem is the use of bio-based composites, whose own nature solves this issue, along with some possible improvements in their processing, such as energy requirements for the fabrication stage [5].

Although they have an excellent mechanical behavior under high loads, an important number of studies have shown that plastic based composite materials present a viscoelastic behavior, caused mainly by the nature of the matrix [6]. Works such as the one of Boyd [7], which concerns glass fiber reinforced composites or the one of Vinet [8], aimed at carbon fiber reinforcement, point out the presence of this phenomenon. It has been observed on all fiber directions, with the exception of the longitudinal one (the force is applied on the direction of the

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reinforcement), where the fiber's rigidity dominates the overall behavior of the composite.

In the case of natural fiber composites, however, it has been shown that even the longitudinal direction presents a pronounced time dependent behavior [9].

For a structure subjected to loads for high periods of time this behavior needs to be considered, starting from the design stage as it has an impact on the structure's possible lifespan. This implies that, whatever reinforced plastic material is to be used, its viscoelastic properties need to be known and an appropriate model needs to be chosen to describe its evolution in time.

The literature shows that there is a lack of consensus on the necessary procedure for the viscoelastic characterization of composite laminates, as details concerning the measurement method, equipment, duration etc. are rather based on the experience of the one conducting the tests.

The present work proposes to analyze the possibilities of viscoelastic characterization, with details from the authors own experiments, but also from the literature, with the intent of providing a guideline for future studies regarding the time dependent behavior of materials.

2. Viscoelastic testing procedure and requirements

If a material exhibits a viscoelastic behavior, its rigidity will vary in time when subjected to a load. For the characterization, in practice, the most often used tests involve imposing a constant stress and measuring the strain response or, the opposite, namely, a constant strain and measurement of the stress response. The first case is called a creep test, schematized in Figure 1, while the second one, relaxation, represented in Fig. 2. In the two figures, a second part of the test is also included, the recovery, where the constant load is eliminated, but the material continues to deform towards its initial state.

Even though other tests may exist, such as ramp loading (where the loading speed is kept constant), the literature points out the predominance of the first two, due to the possibility of long-term tests. Both of them are preferred due to the possibility of discovering the complete nature of the overall material behavior (which might not be limited only to viscoelasticity). The creep type tests tend to be easier to implement, as it is more accessible to measure the material's response in deformation than it is in stress.

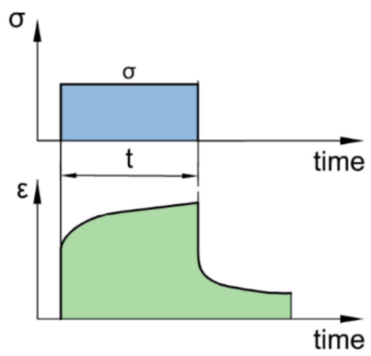


Fig. 1: Creep test

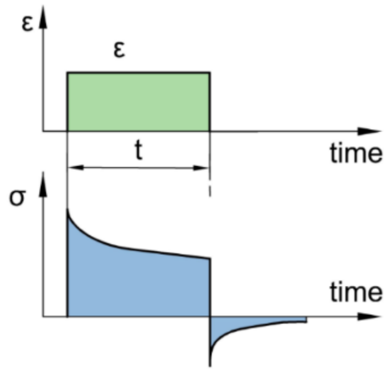
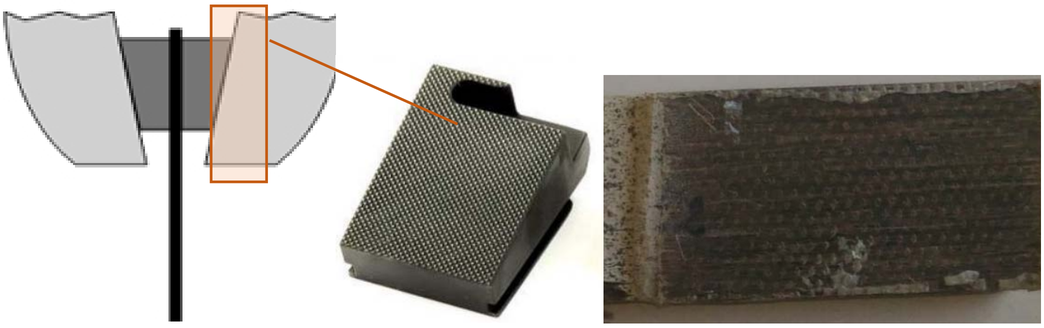


Fig. 2: Relaxation test

A significant number of studies have shown that the viscoelastic behavior depends on several factors. In the case of composite laminates, the most important ones are: stress level [10], temperature [11], humidity [12]. Their influence is most often studied by varying them inside the viscoelastic characterization procedure, which may lead to a significant consumption of time and materials. Experience has shown that the samples being analyzed could be subjected to repeated tests, with the condition of monitoring their integrity. This is generally accepted as, apart the reduction of material required, it tends to reduce errors caused by sample variation.

3. Materials and equipment

The samples that are to be subjected to viscoelasticity tests need to be designed in such a way as to be able to sustain the loads for the desired period of time. This is most easily achieved with a constant stress distribution in the specimen. An example of a geometry which fulfills this requirement is the rectangular shaped sample, with loads being applied on the small side, uniformly. These have the advantage of a simple fabrication procedure, due to their basic shape. It is desired especially when dealing with laminated composites where stress distribution is dependent on fiber orientation as well. The drawback is that, when mounted in the testing installation, the fixtures might damage the sample, as a significant number of them are grip based to prevent sliding (Fig. 3). If this tends to happen in the experiments, tabs of higher rigidity are recommended to be glued on the mounting area, which are to take over the stress concentrators.



a) b) c)

Fig. 3. Grips of a testing machine: a) typical grip schema; b) typical jaw; c) results of clamping on a sample

The testing equipment depends strongly on the type of experiment being conducted. As it has been previously discussed, creep tests tend to be more practical. For this reason, this section will present only equipment for creep type tests.

The demand for a long a long period of testing time (ranging from hours to months), imposes certain constraints on the devices, whether they are for introducing the load or measuring the response.

In the first category, the most appropriate device is a creep testing machine (Fig. 4, a). It comes usually equipped with thermal chambers which allow a good control of the sample's temperature. The drawback, though, is that they are limited to this type of testing sequence, making them rare in laboratories for general purpose material testing.

A more accessible way to conduct a creep test is to mount a weight to the sample, either directly or through a lever (Fig. 4, b). This can be done in any laboratory and can run for long periods of time ([10],[13]). The limit might come from the maximum force that can be applied and, thus, the materials that can be tested or the geometries the samples can have. Apart that, the loading part of the test can lead to shock in the sample (which might damage it), if the load is applied too rapidly.

Another appropriate testing installation is the universal testing machine, UTM (Fig. 4, c). It is versatile and an essential device in every material testing laboratory. In the case of viscoelasticity tests, they can be controlled to maintain a constant force, making them suitable for conducting creep tests, but they can also be used for other procedures which point out the time dependent behavior, such as ramp loading or relaxation type tests. A significant number of research work has been conducted on this type of machines [8],[9], [13]). Their drawback can be considered the fact that they are general purpose and, thus, they need to be especially prepared for long period tests and to have an appropriate software.

In terms of response measurement, seeing that there is a displacement or a strain that needs to be recorded, the devices must be able to read/record them for the desired period of time, but also with satisfactory precision.

The easiest and most affordable means to achieve this is by using a dial gauge (Fig. 5). The measured values have to be recorded manually, when desired, making its use cumbersome. It is, however a stingy and accessible solution.

If a higher precision is required, an extensometer can be used (Fig. 5). Most UTM come equipped with this device, which, to function, is mounted on the tested specimen. As with the UTM, it is very versatile and can be used for measurement, but also for control (if a closed control loop is programed in the machine). Its limitation comes from the fact that it can record only while the testing machine is turned on and it can only measure one sample at a time, making it difficult to record the recovery part of the test, if existent. They also tend to be fragile and costly.

Another device frequently used for deformation measurement is the strain gauge (Fig. 5). It allows reading and recording in an automated fashion, for extensive periods of time. It is highly sensitive to excitations, including parasitic ones. Samples are usually equipped with more than one strain gauge in order to eliminate reading errors. The limitations come from the requirement of auxiliary devices, such as data acquisition boxes (which can be costly), but also, they are consumables (as they are glued to the sample). Cheaper strain gauges also drift in time, thus creating a source of errors. With all of this considered, they tend to be the preferred instrument for time dependent deformation measurements.

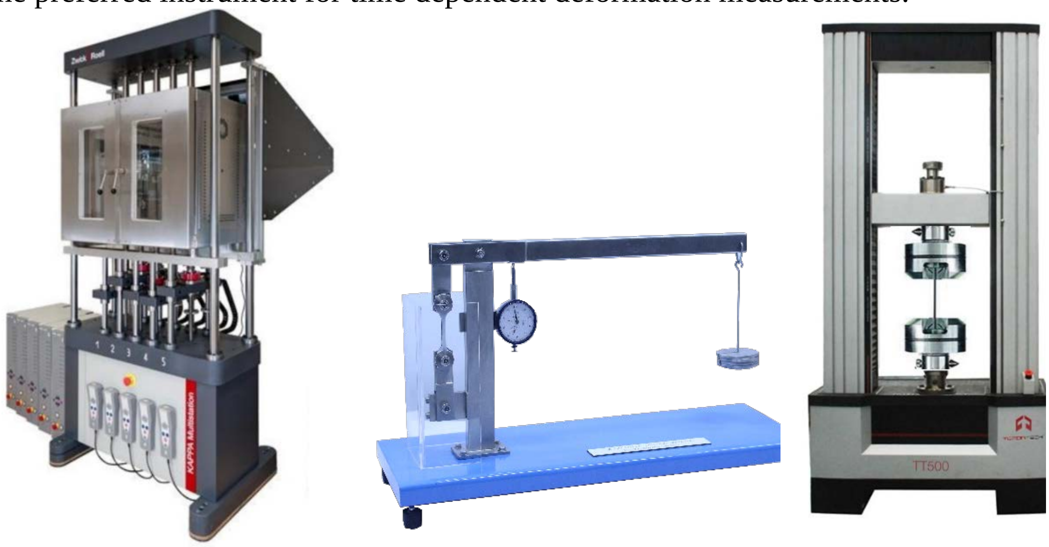


Fig. 4: Creep testing equipment: a) creep testing machine; b) lever mechanism; c) Universal testing machine

a) b) c)

Fig. 5: Instruments for deformation measurement: a) dial gauge; b) extensometer; c) strain gauge

The deformation recording frequency strongly depends on the total period of creep, the instrumentation's performance and, if existent, the viscoelasticity model. Ideally, a high recording frequency is preferred at the beginning of the creep and recovery periods, where the viscoelasticity phenomenon is prominent. However, most data acquisition systems allow only a constant sampling frequency during a recording. Post processing can be implemented if the number of lecture points is excessive.

Apart the two devices, which are primordial to conducting a creep test, extra precautions might need to be taken, particular to the material, so as to ensure good measurements. Examples of such precautions are the conditioning of the samples in a controlled environment before the experimental procedure or monitoring their structural integrity with an acoustic emission system or preamble tests to verify degradations in Young's modulus.

4. Application of creep-recovery type tests on bio composites

The multi load creep- recovery testing method has been applied on bio composites made of an epoxy resin matrix reinforced by unidirectional flax fibers. Previous studies have shown an effect of humidity on the samples, which has prompted the conditioning of the samples prior to testing. This was done in an environment with a constant temperature of 23°C and 50% humidity.

The sample geometry was chosen to be rectangular for all the specimens, of 25 mm in width, with 2 mm in thickness. They were cut from larger plates, obtained through a thermocompression cycle. Aluminum tabs were glued with Araldite on the sample's extremities as it has been proven in previous experiments that it is a good way to reduce gripping effects.

Four load levels of creep – recovery cycles have been applied on samples with fiber directions of 0° (Figure 6). The basic testing conditions were: one hour of creep followed by a minimum of 24 hours of recovery. The load levels were chosen based on preamble tests. Thus, tensile tests were firstly conducted where, with the help of the acoustic emission, the sample's structural integrity was monitored (Figure 7). A damage threshold has been found, of 180 MPa. The creep tests were conceived with the stress levels lower than these values.

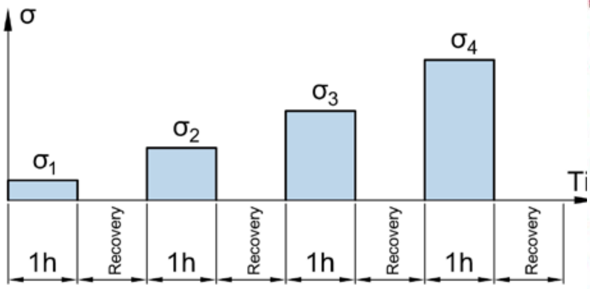


Fig. 6: Testing cycle

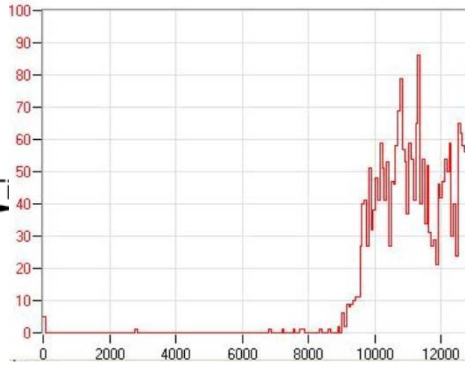


Fig. 7: Acoustic emission results. Damage threshold at 9000 N or approx. 180 MPa

The load during the creep phase of the test has been chosen to be introduced with the help of a universal testing machine. Special attention needs to be paid when mounting the sample in the machine's grips as, when tightened, they can introduce an unwanted load in the sample which will affect the measurements (Fig. 10). The tested sample's structural integrity was continuously monitored during the test, the reason being that it can damage in time as well.

For the strain measurements, strain gauges have been chosen, as the samples were to be tested for a total period between two and seven days (depending on the recovery period), including the recovery periods overnight. They have been mounted in pairs on each side of the specimens with the purpose of eliminating the bending effect present while mounted in the testing installation. The strain gauges were connected to a data acquisition box which allowed reading on multiple channels allowing, thus, multiple samples to be tested in parallel. A sample of the specified geometry fully equipped for testing with end tabs and strain gauges is presented in Fig. 8.

Fig. 8 : Sample with fibers at 0°. The second strain gauge is glued on the opposite face

In Fig. 9, the deformation signal is shown for one the tested samples, with readings from the two strain gauges. It can be seen that the signal differs during

during the creep phase, where one of the gauges records a higher deformation than the other (black line). Similar issues appear during the mounting of the samples in the machine (Fig. 11) and during their unmounting.

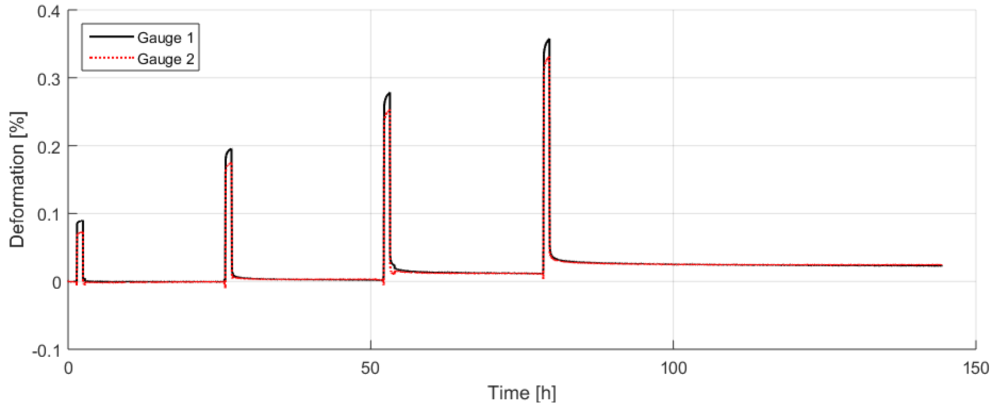


Fig. 9 : Deformation signal during a creep-recovery testing procedure, fiber direction of 0°

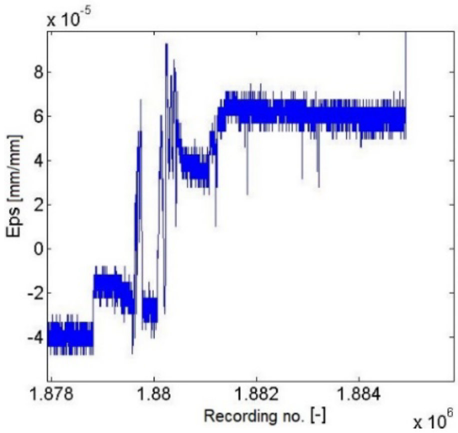


Fig. 10: Strain due to fixture grip

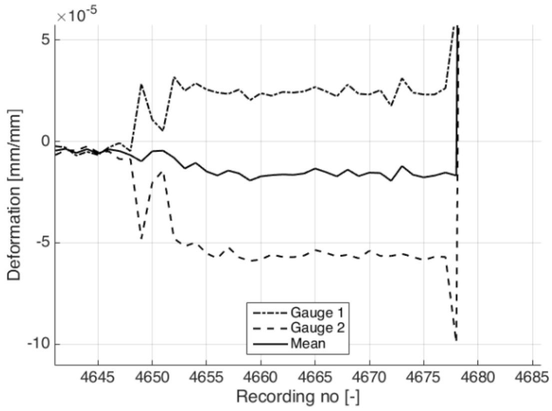


Fig. 11: Strain due to bending

The end of the creep test should be made with a fast opening of only one of the grips, as any other procedure to eliminate the load would introduce unwanted deformations as well, which would affect the recovery period and the subsequent creep tests.

The strain gauge sensitivity makes it that any contact with the sample creates a deformation reading response. Thus, to eliminate these effects at the beginning of the recovery phase, the sample can be left in the testing machine (with only one end gripped) or, if other samples need to be tested, it can be extracted after a couple of minutes, when the prominent part of the recovery deformations has passed, and relocated in a place where it can recover unhindered. Both cases have proven efficient in the presented tests. After the desired period of recovery, the next creep test can be conducted and the procedure repeated. All of the information presented here has been taken into account when conducting the creep-recovery type tests for the given example.

The deformation signals presented in Fig. 9, for the unidirectional flax fiber epoxy resin composite are mediated and analyzed for their clarity in Fig. 12, where the creep part is presented, and in Fig. 13, where the recovery part is shown. It is worth noting that, since the recovery period is uneven, this signal has been limited to 24 hours reading. As the signal tends to stabilize, it has been considered that anything which exceeds this period does not bring any further information.

It is visible during the creep period that the signal tends to increase in a continuous fashion This implies that the material exhibits significant deformation, while a constant load is being maintained. Specifically, 6% for the first load level, 12% for the second, 14% for the third and 12,5% for the last load level.

As for the recovery period, Fig. 13 shows that the signal has a slight variation, but following a continuous path. A possible reason for this variation is the slight change in temperature in the laboratory. Even though the material tends to return to its original state, for the load levels higher than 30 MPa, a significant portion of plastic deformation still remains, suggesting a viscoplastic deformation occurring during the creep phase. The two signals show a predictable evolution of the deformation, during creep and recovery periods giving, thus, the possibility of modeling, based on these results.

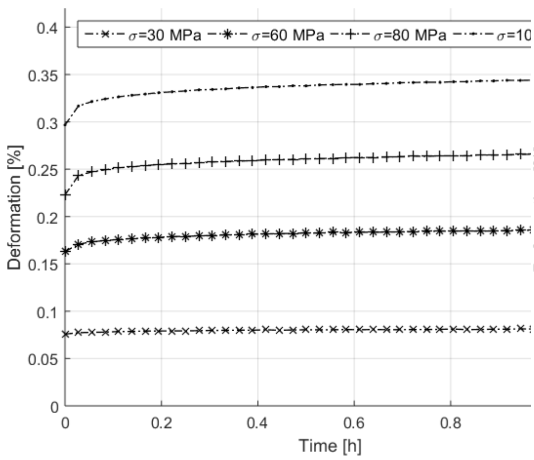


Fig. 12 : Creep period

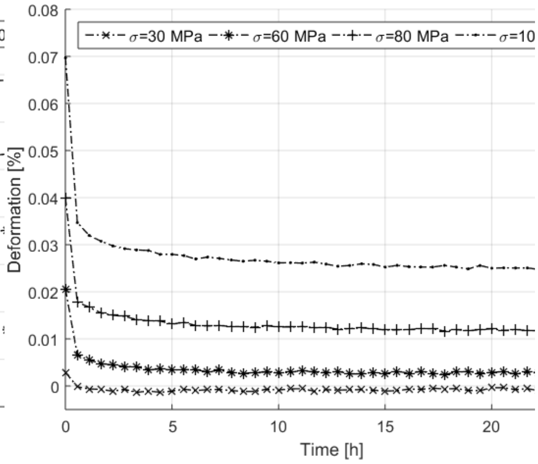


Fig. 13: Recovery period

For bio-composite materials, in particular, studies have shown a significant dispersion of mechanical properties [14]. For this reason, the creep-recovery testing procedure needs to be conducted on more than one sample, in order to verify if the variation is larger than the one in classic tests, such as traction or bending. However, the time required for these tests (in the presented case, a week) makes it cumbersome to repeat the test many times.

5. Conclusions

A method for viscoelastic characterization of composite materials has been presented, with the emphasis being made on the testing conditions. With the help of previous works, it has been shown that a standardized testing procedure for composite materials does not exist yet and that the preferred testing procedure consists of multiple creep-recovery tests, which allow the most thorough analysis of the time dependent behavior of composite materials. The necessary equipment and precautions have also been presented for this testing sequence along with an example conducted by the authors on a laminate composite of unidirectional flax fiber reinforcement, with emphasis being made on the quality of the data obtained from the tests.

Particularities and precautions of the test for this material have been presented, along with a series of recommendations during the testing phase. It is worth noting that the presented procedures have proven to yield useful data for the viscoelastic characterization of flax-fiber composites but, although improvements could be implemented for future work.

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