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**DURABILITY ASSESSMENT OF A HIGHLY REINFORCED
THERMOPLASTIC COMPOSITE : EXPERIMENTAL AND
MICROSTRUCTURAL SIMULATIONS OF THE TRANSVERSE DAMAGE
KINETICS.**

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ABSTRACT

abstract – no more than 10 lines

INTRODUCTION AND CONTEXT

Thanks to their potential to offer high mechanical properties, low weight and a resistance to cyclic loadings, composite materials are increasingly used for high pressure equipments such as stationary or on-board gaseous storage vessels or for pipes. Such engineering structures are submitted to complex multiaxial stresses which are locally amplified due to the microstructural variability. Long term safety of composite materials involves a deep understanding of damage kinetics at the material scale. Indeed, microstructural details of a material – which often determine its properties and especially its durability – have long been ignored by continuum mechanics approaches and inductive modelling. Deployment of microscopic and tomographic observations now gives access to more detailed knowledge of microstructures and thus feed deductive approaches.

Unidirectional (UD) composite materials are known to have highly anisotropic properties, with excellent stiffness and strength characteristics in the fibre direction. While numerous studies have been undertaken for many years on the longitudinal performances of UD, very few studies address the transverse behaviour which is nevertheless often the site of the first cracking and affect the load transfer between fibres. If this is often negligible for monotonic loads, this phenomenon becomes significant in the long term for the durability or lifetime assessment. It is particularly true with a thermoplastic matrix,

which is more sensitive to hydrostatic pressure so that the damage mechanisms are very different from a composite material manufactured with thermosetting resin.

This work is thus focused on the transverse damage kinetics of composite materials and is based on an understanding of the physical phenomena occurring at the microscale. Confined to this length scale, mainly due to fibre contacts at high volume fractions, polymeric matrix exhibit unexpected properties apparently different from their bulk state [17]. This “interphase” behaviour is far from universally accepted and it is quite difficult to obtain reliable experimental data. Nevertheless, some of the differences observed come from the local mechanical loading and the hydrostatic pressure effects undergone by the polymer in a confined state between rigid fibres.

MATERIAL OF STUDY AND CHOSEN APPROACH

The composite of the study exhibits a high volume fibre fraction (>60-65%), glass fibres and a PA11 thermoplastic matrix. Owing to this matrix, the composite features a progressive damage – discussed thoroughly in [4] – in contrast with a reinforced thermosetting resin material. The figure 1(a) displays a cross section of the material at issue. This highlights two inevitably manufacturing defects:

- Matrix defects: micro and macro porosities (evidenced in figure 1(b)) and which will explain *a posteriori* the matrix behavior law implemented
- A microstructural variability *e.g.* fibre dispersion, diameter distribution (underlined in figure 1(c)), matrix-rich areas...

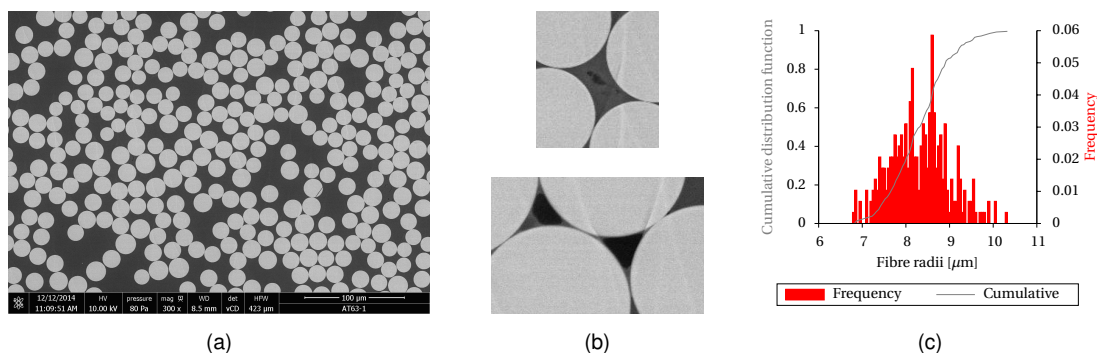


Figure 1 – (a) Composite of the study ; (b) Micro and macro voids ; (c) Fibre radius dispersion

A multiscale top-down (global/local) experimental and numerical approach has been developed pursuing the thesis works carried by Cayzac [4]. The multiscale analysis proposed in this contribution attempts to relate the macroscopic behaviour to microscopic physical processes. And a particular effort has been made on the understanding of the influence of microstructural variability. To this end, studying microstructures seems relevant to investigate the mechanics and kinetics of damage that take place at the fibre section level. Indeed, while the macroscopic elastic effective properties of a UD composite are not dependant of the fibre arrangement, the stress distribution is, on the contrary, highly affected by the fibre layout [15].

An important issue, when representing a material at its microscale, is the choice of the RVE (Representative Volume Element). The idea of an RVE is well documented in

the literature since the early works of Hill (1963) and justifies its use to predict material properties.

Finite elements is the most commonly used numerical method used as far as RVE and micromechanics are concerned. Of course, this requires a meshed microstructure. Two possibilities are left to the authors. On the one hand, an experimental method consists of digitizing a real microstructure from SEM image analysis of a sample [4]. On the other hand, a complete numerical method generates artificial microstructures (cited below) and it only remains to mesh them afterwards. The latter method is the subject of many recent publications which provided inspiration for our methodology explained hereafter.

METHODOLOGY AND TOOLS DEVELOPED

The generation of microstructures is often tackled when studying reinforced polymer material. A UD fibre-reinforced composite transverse cross-section outlines a non-overlapping distribution of circular disks. We will only focus here on the generation of 2D microstructure cells.

State of the art

Two conditions have to be met: the disks cannot intersect each other and a certain area fraction (or volume fraction if we consider a 3D UD fibre-reinforced composite) has to be reached. Several algorithms can be found in the literature to this extent. The most straightforward and common are the Strauss *hard-core* and Random Sequential Adsorption (RSA) algorithms. Unfortunately they both present a low peak volume fraction, they are respectively limited to 55 and 30%. Further improved versions of the RSA method [13] [22] lead to higher volume fraction but increased the computational costs.

Several authors particularly focused on high volume fractions. For instance, Desmond *et al.* [5] who introduced a method of random close packing between confining walls. Others, in [26], started from a regular periodic packing and disturbed this arrangement. Melro *et al.* [16] proposed a combination of earlier mentioned algorithms capable of generating fibre distributions with a volume fraction up to 70%.

Lastly, Lubachevsky *et al.* [14] put forward an algorithm based on molecular dynamics. The method can be used to create up to the theoretical dense packing volume fraction and requires low computational resources. Moreover, it exhibits the particular and important asset of generating periodic cells. Ghossein *et al.* recently implemented the code in MATLAB[®] in three dimensions in their works [9].

Microstructure generation tool

In this paper, we focused on the works carried by Ghossein *et al.* for several reasons: the permitted very high volume fraction, the periodicity of the generated cell, the processing speed of the calculations and the possible improvements we imagined. These assets are in accordance with our approach and material of study. The code was implemented in MATLAB[®], like Ghossein *et al.* did, and was inspired by the publication [9]. Furthermore, the reader is referred to [9] for its detailed description.

The main steps can be summarised as follows. The cell size is set and a chosen number of points (disks with a null radius) are randomly assigned in its area. Each point has a position vector, an initial random velocity vector and a radius growth coefficient (time-dependant). The points are set in motion as soon as the calculation and time start. The collision time between every particles with each other or with the cell boundaries are computed. From there, the minimal calculated time is considered: the disks are moved

forward to this time, the radii are updated. If it corresponds to a collision between disks, their respective velocity vectors are computed observing the conservation of kinematic energies. If a disk and one or several cell boundaries are in contact, a cloned disk is added to the opposite side in order to guarantee the geometric periodic conditions. The next collision time is then calculated and the same steps go on. The overall computation stops when the volume fraction overshoots the objective. The last stage consists of readjusting the disk radii to strickly meet the targeted volume fraction.

We then used Gmsh [7] to mesh the cell since it provides a powerful and yet user-friendly tool. In this way, the mesh – periodic and regular – can be used as input geometry file to Z-set [1] in order to proceed with Finite Element calculations.

This microstructure generation tool can be used to create various cells with for instance: high volume fractions, a wide range of disk radii, matrix-rich zones or cells with numerous fibres such as shown in figure 2.

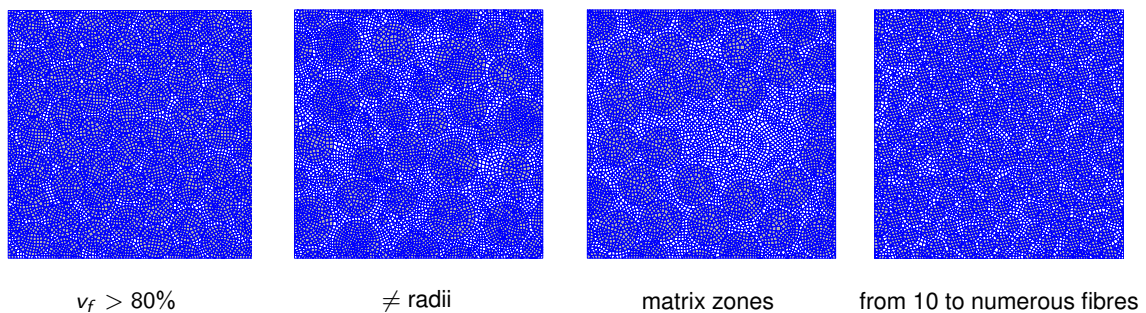


Figure 2 – Meshing and microstructure generation possibilities

Finally, the geometry was extruded over several elements in the out-of-plane direction to take into account the third dimension since our material is a UD fibre reinforced composite. We are currently working on generalised plane-strain periodic elements conciliable with large strain formulation.

Statistical spatial descriptors

While several cells of a microstructure can be created, one needs to prove here that they are representative of the real microstructure. The fibre volume fraction is obviously the first statistical parameter that comes to mind. But there are numerous different statistical and spatial descriptors chosen in the litterature.

The statistical and spatial descriptors of the microstructures implemented to our code are listed below.

- The fibre radius distribution
- The first and second neighboring fibre distance distributions [16] [25] [21][15]
- The first neighbour orientation distribution [16] [25] [21] [8] which displays the presence of preferred orientations (anisotropy) between neighboring fibres in the microstructure.
- The Voronoï polygon area distribution [16] [25] [8] which helps revealing the regularity (or disorder) of the spatial arrangement.
- The covariance functions [12] [11] [25] [8] which point out, according to a direction of observation, the geometrical dispersion of fibres in the cell.

- Ripley's K function (or second-order intensity function) [20] [8] [16] [21] [23] which, compared to the Poisson (complete randomness and permitted overlaps) point pattern, gives information about vacuity or clustering of the arrangement of fibres.
- Ripley's L estimator [16] which indicates clustering (or regularity) for positive values (or negative values respectively) corresponding to the distance on the abscissa axis of the graph.
- The pair correlation function [15] [25] [21] [23] which gives the intensity of occurrence of the distances between the fibre centres.

Let us consider a cell with 56 fibres and a 60% fibre volume fraction. To illustrate several microstructure generations and only one statistical parameter, we decided to constrain the distance between each fibre. We obtained several cells from an hexagonal pattern (set as prime example) to a completely disorderly arrangement by progressively increasing the first neighbor distance. The microstructures generated and their respective first neighbour orientation impulse polar graph are represented below on figure 3. We can reasonably imagine that induced strains and stresses under a mechanical load will definitely not be equivalent in these different cells.

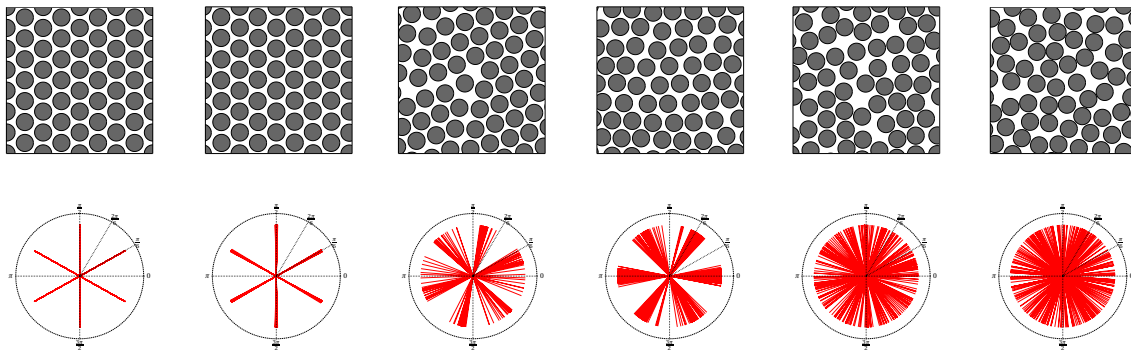


Figure 3 – Cell generation - from hexagonal pattern to completely random

SET UP OF THE FE CALCULATIONS

Any cell with a regular pattern or with a random arrangement of fibres can, from now on, be generated and investigated with FE modelling. The framework of this work could lie in the large family of homogenization schemes or multiscale approaches. The goal is to capture the behavior of the material (from a macro or meso point of view) while being able to localize phenomena in the microstructure (damage). This study will help to assess the durability (creep or fatigue tests for instance) of a material taking into account the variability of the real microstructure. The chosen FE formulations and the behavior laws used will be explained hereafter.

Material behavior laws

We suppose in our first approach that there are no interphase and the matrix of the composite of this work behaves like the bulk material. Previous studies on the PA11 [2] [3] lead to the identification of a non-linear porous media behavior law: a modified GTN model. f is an intern variable quantifying the void in the matrix. The initial porosity, f_0 , is set to 0.1%. The chosen behavior law will be used in all our calculations and it necessitated the large strain formulation available in Z-set [1]. As for the glass fibres, we will use

a linear elastic law, relevant enough, considering the role of the matrix.

Micromechanical problem, boundary conditions and FE formulations

The cells were built with a periodic mesh on purpose. We suppose that they contain all the necessary statistical information, in accordance with our descriptors, and that the ergodic hypothesis holds. Owing to the standard homogenization method, we can introduce the macroscopic (also called overall or averaged) strain $\underline{\mathbf{E}}$ and the macroscopic stress $\underline{\boldsymbol{\Sigma}}$, accordingly to the Hill (1967) and Suquet [24] definitions. These macroscopic quantities come from the microscopic strain $\underline{\boldsymbol{\varepsilon}}$ and the microscopic stress $\underline{\boldsymbol{\sigma}}$ defined by the following equations. Let the cell with the body V and the boundary ∂V , we have:

$$\underline{\mathbf{E}} = \langle \underline{\boldsymbol{\varepsilon}} \rangle = \frac{1}{V} \int_V \underline{\boldsymbol{\varepsilon}} dV \quad ; \quad \underline{\boldsymbol{\Sigma}} = \langle \underline{\boldsymbol{\sigma}} \rangle = \frac{1}{V} \int_V \underline{\boldsymbol{\sigma}} dV$$

We particularly focused on periodic boundary conditions, well documented in the literature [6] [10] [19] [18] [24]. The periodic conditions can be summed up with:

- The traction vector $\underline{\boldsymbol{t}}$ fulfills the anti-periodic conditions : it is opposite on facing sides of ∂V . Also with,

$$\underline{\boldsymbol{t}}(\underline{\boldsymbol{x}}) = \underline{\boldsymbol{\sigma}}(\underline{\boldsymbol{x}}) \cdot \underline{\boldsymbol{n}} \quad \forall \underline{\boldsymbol{x}} \in \partial V$$

- The local strain $\boldsymbol{\varepsilon}(\underline{\boldsymbol{u}})$ is split into its average and a fluctuation term. We then look for a displacement field of the following form (where $\underline{\boldsymbol{u}}_{\#}$ is periodic):

$$\underline{\boldsymbol{u}} = \underline{\mathbf{E}} \cdot \underline{\boldsymbol{x}} + \underline{\boldsymbol{u}}_{\#} \quad \forall \underline{\boldsymbol{x}} \in V$$

Thus, the periodic formulation makes it possible to prescribe either macroscopic stresses or strains over the cell.

RESULTS

We will explain hereafter several of the calculations that the cell generation can enable with all the used specifications *i.e.* periodic fields, non-linear GTN behavior matrix and large strain formulation.

Computations of void evolution under biaxial tension test of chosen cells

We impose an uniaxial numerical tension test, either on the hexagonal pattern or random arrangement cell, along the horizontal direction, accordingly with the top figure in 4. The tests are pursued until failure, the evolution of damage is characterised by the void growth in the matrix. In the same figure 4 are displayed on the left side the stress-strain curve (filled circles) compared with the void growth in the matrix (empty circles). The contour map (in the left side of 4) shows the void in the matrix. It clearly demonstrates that, while the void is well spread in the hexagonal pattern cell matrix, only few of areas are concerned in the random cell. The proximity of fibres – confined matrix – is a concentrator of triaxiality. Other things being equal (*i.e.* prescribed load, constitutive materials), the void increases significantly faster in these confined areas. Inevitably, the first broken elements (where the calculations are stopped on purpose) quickly appear at lower strain on random arrangement cell which displays closer first neighboring fibres than hexagonal pattern cells.

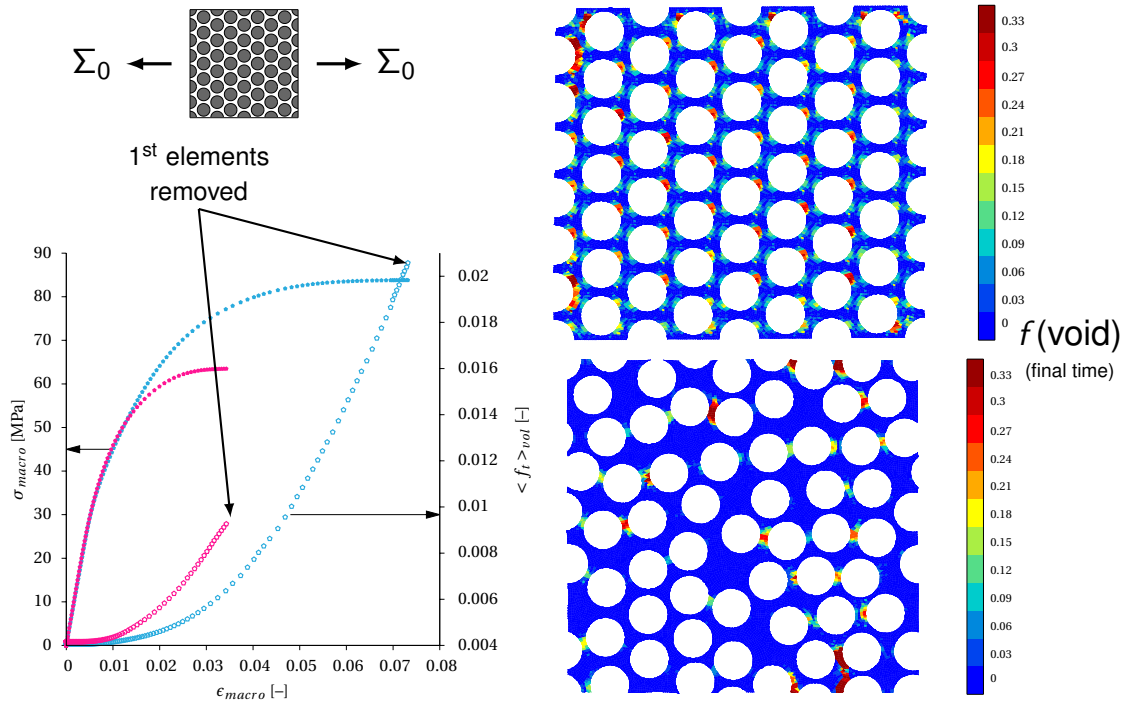


Figure 4 – Void contour map for hexagonal pattern or random cell before failure

In addition, let us consider an in-plane biaxial tension test of a cell, illustrated in figure 5(a). We prescribe the following macroscopic stress over the cell:

$$\underline{\underline{\Sigma}} = \Sigma_0 \vec{e}_\theta \otimes \vec{e}_\theta$$

We compute the tension tests for several angles between the uniaxial tests along the \vec{e}_1 and the \vec{e}_2 axes. The contour maps of the void evolution in the cell matrix are shown in figure 5(d) and can be compared with the polar graph of the first neighbour fibre in the cell in figure 5(c). The loaded hexagonal pattern cell highlights specific evolutions of the void depending on the orientation of the tension test. On the contrary, the loaded random fibre arrangement cell shows an isotropic evolution of porosity.

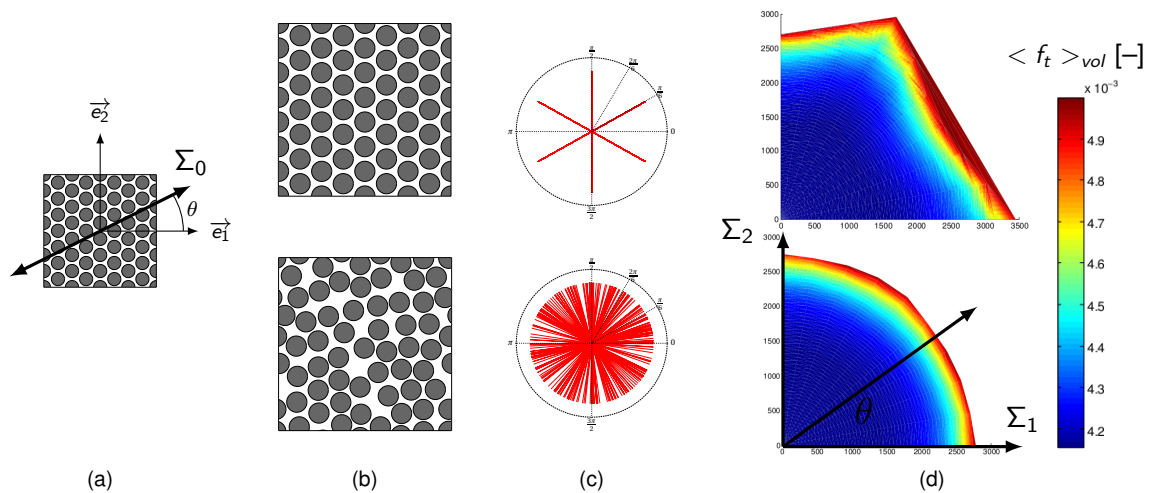


Figure 5 – Void growth in the whole matrix of the cell for a planar biaxial numerical test

Durability investigation

As a last example, we present here several numerical creep tests on the hexagonal pattern cell. The latter is loaded until a certain macroscopic stress level and is maintained from there (shown in figure 6 on the left side). The figure 6 on the right side displays the evolution of the void as a function of the time. The slope of the void growth depends on the macroscopic stress level maintained over the cell and on the time. This means that even a light macroscopic stress held can damage the matrix over the time. The lifespan of the material under a creep test can thus be determined.

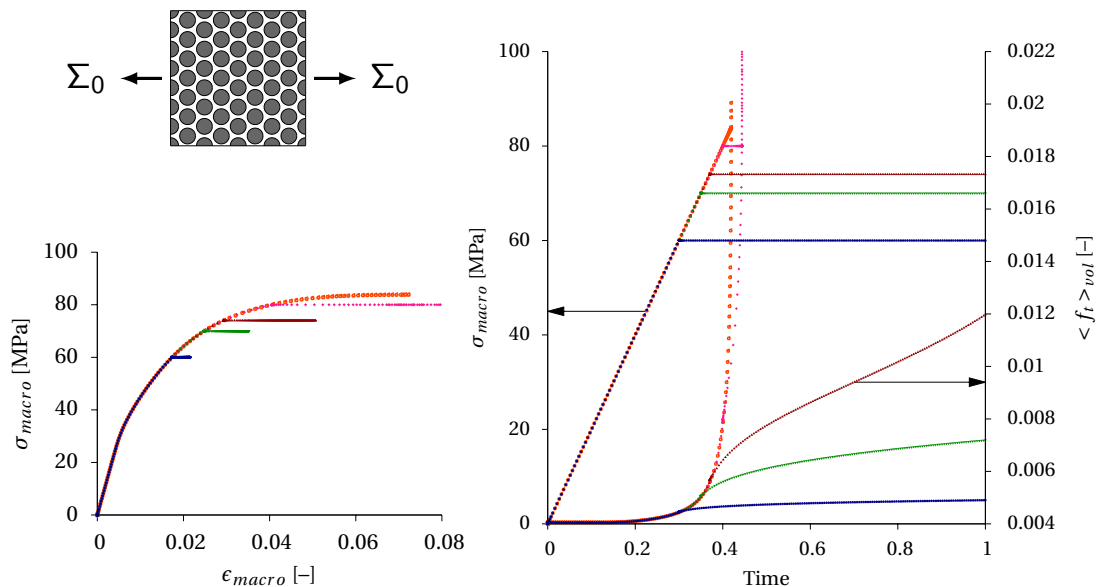


Figure 6 – Durability - Numerical creep tests on a hexagonal pattern cell

CONCLUSIONS

In this proceeding, we introduced the works carried on transverse microstructure numerical tests. To fulfill this goal, the study of microstructures is inescapable to understand the mechanics and kinetics of damage of reinforced thermoplastic composite materials. Our approach relies on generation and FE computations of periodic microstructures where the fibre arrangements can be controlled to fit the real microstructure. Using this method, we can investigate the influence of the variability of the microstructure. Each parameter *e.g.* fibre radii dispersion, fibre arrangement, matrix-rich zones, macroporosity or distance between neighboring fibres can be treated independently. Not only are mechanical computations possible with this cell and mesh generation but heat diffusion problems were also processed in parallel of this work.

The current works tackle the scale transitions: from micro to macro. The final goal lies in the industrial effort to accurately predict the properties and evolution of damage of a material within a structure, only knowing the constitutive materials and its microstructure. It would furthermore be relevant to study “interphases” (as defined in [17]) in a second approach to enrich our works. Several experimental tests will also be performed with tomographic observations in order to confirm the whole numerical investigation.

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