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THE ELASTO-VISCOPLASTICITY AND FRACTURE BEHAVIOUR OF THE RTM6 STRUCTURAL EPOXY AND IMPACT ON THE RESPONSE OF WOVEN COMPOSITES

X. P. Morelle[1*], F. Lani[1], M. A. Melchior[1], S. André, C. Bailly[2], T. Pardoen[1]

[1]*Institute of Mechanics, Materials and Civil Engineering, Université catholique de Louvain, Place Sainte-Barbe, n°2, L5.02.02, 1348 Louvain-la-Neuve, Belgium*
[2]*Institute of Condensed Matter and Nanosciences, Université catholique de Louvain, Croix du Sud n°1, L7.04.02, 1349 Louvain-la-Neuve, Belgium*
*xavier.morelle@uclouvain.be

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Abstract
A better understanding and modelling of the epoxy matrix behavior is required in order to correctly model the non-linear deformation, damage and failure mechanisms of fibre-reinforced composites under loading conditions involving moderate to large strains in the matrix, such as during creep, impact or tensile test at 45° fibre-orientation. The mechanical behaviour of the industrial grade HexFlow RTM6 epoxy resin, widely used in aerospace composite applications, has therefore been investigated following the local approach of fracture methodology.

1 Introduction
The context of this work is related to the ever-increasing use of high performance composites to reduce the weight of commercial aircraft structures over the last decades. If the use of these materials is now taken for granted, we still lack of a complete understanding of the link between the constituents properties and the overall performances of the reinforced material, especially the plasticity and damage evolution of the resin.
The present study intends to provide some answers regarding the effect of the epoxy matrix intrinsic behaviour on the overall composite response.

A better understanding and modelling of the viscoplasticity and damage mechanisms of the epoxy matrix could potentially be essential for predicting the composite response for loading conditions involving large deformation.

The mechanical behaviour of the industrial grade epoxy resin HexFlow RTM6, widely used in aerospace composite applications, has therefore been investigated following the local approach of fracture methodology.

2 Material, processing and testing methods
2.1 Material
In the present paper, the material under consideration is the mono-component HexFlow RTM6 epoxy resin, certified for aeronautics where it is usually used as a matrix for CFRP

composites. It is an amine-cured epoxy resin supplied by Hexcel and consists of a premixed system composed of tetra-glycidylmethylenedianiline (TGMDA) epoxy polymer with an epoxy equivalent weight of 116 [g/eq.] and of two amine curing agents M-DEA (158 [g/eq.]) and M-DIPA (186 [g/eq.]).

The RTM6 has a high glass transition Tg = 220 [°C] determined by differential scanning calorimetry (DSC). It provides good stiffness and thermal stability.

2.2 Processing

The resin mixture was first degassed under vacuum, at 90 [°C] and for 75 [min], before being poured into a release-agent coated and pre-heated mould. Then, a low-temperature curing cycle (3 hours at 130 [°C]) was applied to avoid local overheating, especially for the production of thicker slabs, and eventually followed by a post-curing step at 180 [°C] for 3 hours with a heating ramp of 2 [°C/min].

This overall curing and post-curing cycle enables to reach a high degree of conversion (>95%) required for aeronautical certification. Different types and size of specimens are processed from thin cylindrical samples to thick rectangular slabs.

2.3 Machining and mechanical testing

Dog-bone tensile specimens, small cylindrical specimens for uniaxial compression and cylindrical tubes specimens for torsion tests were machined from the same cured and post-cured resin to ensure identical thermal history. Single-edge notched bend (SENB) specimens were also prepared from thicker rectangular slab of plain resin.

Tensile, compression and fracture toughness tests were all carried out on a screw-driven universal testing machine (Zwick-Roell with an external loading cell of 250 [kN]), while the torsion tests were performed on a servo-hydraulic torsion-tension testing machine (MTS, type MTS793). For all tests, the standard crosshead speed was 1 [mm/min] and the reference temperature was ambient. The strain was measured by strain gauge, LVDT (linear variable differential transformer) or by compliance corrected crosshead displacement, depending on the specimen geometry and loading configurations.

3 Results and discussion

3.1 Large strain deformation behaviour

Compression and torsion tests have been performed in order to characterize the deformation behaviour at large strains.
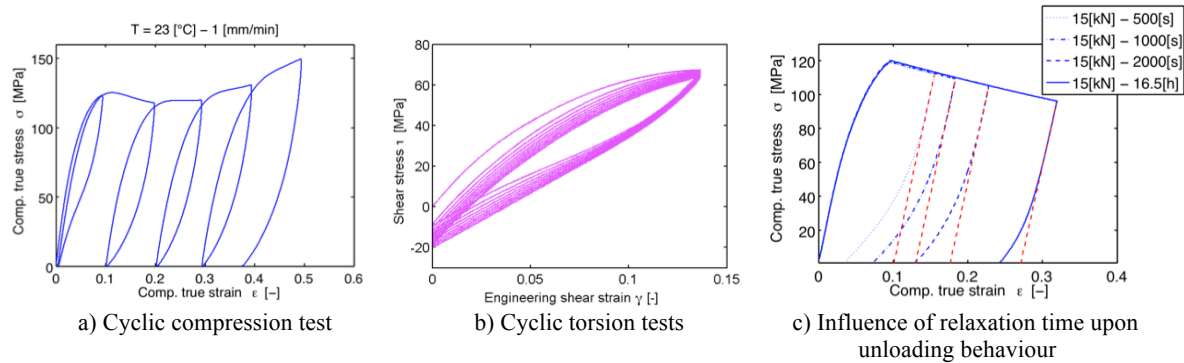
First, we have studied the effect of strain rate, temperature and applied load using compression and creep tests (relaxation at constant applied load), see **Figure 1**.

a) Compression at different strain rate and temperature   b) Creep tests for different applied loads at room temperature
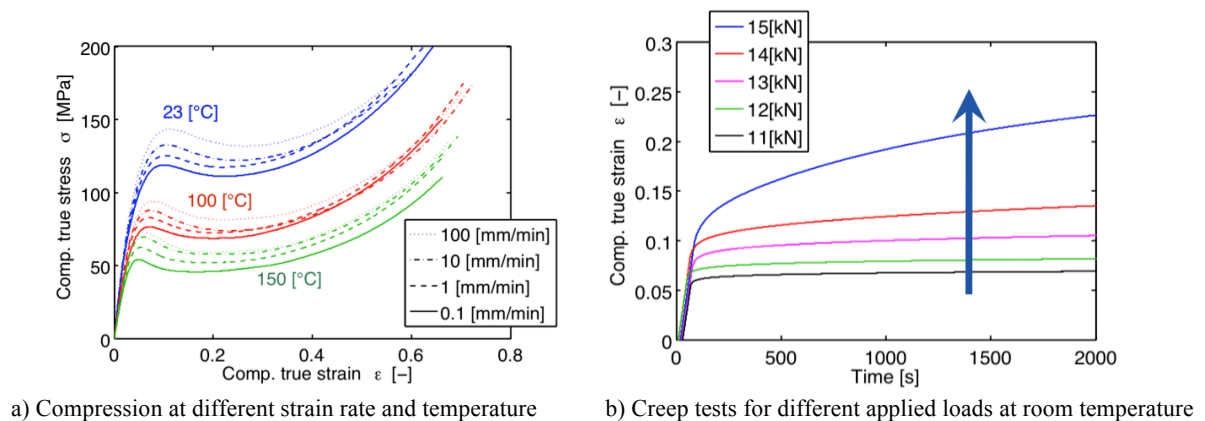
Figure 1. Mechanical characterization of *elasto-viscoplastic* behaviour of RTM6 epoxy resin.

Figure 1a shows that the RTM6 epoxy exhibits a classical rate and temperature dependent behaviour, with yielding followed by *strain softening* and *re-hardening* at larger strains. The elastic modulus changes only weakly with rate and temperature indicating negligible viscoelastic effects compared to the important viscoplastic effects, illustrated on **Figure 1b**.

As shown in **Figure 2**, cyclic tests were performed at different level of plastic strain and for different types of loading (**a)** compression and **b)** torsion). The effect of stress relaxation on the unloading response was also studied by performing creep holding tests before unloading, see **Figure 2c**.

| a) Cyclic compression test | b) Cyclic torsion tests | c) Influence of relaxation time upon unloading behaviour |

Figure 2. RTM6 *unloading behaviour* characterization under low cycle fatigue conditions and after constant load relaxation tests.

Figure 2 indicates a strong *kinematic hardening* during unloading. Indeed, the unloading curve is strongly non-linear showing *reversed plasticity* while still in overall tension. The analogy with the Bauschinger effect observed in metals can thus be made, even though the underlying physical mechanisms are obviously different. From now on, we will more simply call this phenomenon: *back stress* upon unloading.

This back stress develops over the entire range of plastic strains but decreases significantly with creep relaxation as shown on **Figure 2a** and **c**. Note that this back stress effect has also been observed for other polymer systems.

The origin of the *back stress* is that the deformation mechanisms are actually strongly heterogeneous. Indeed, plastic deformation occurs in a very localized way through *shear bands* or similar mechanisms, with a characteristic size, distribution and number of heterogeneities that depend on the chemistry involved [1-2]. Furthermore, regions next to crosslinks are much stiffer and resistant to plasticity than other regions, inducing strong *stress heterogeneities*. Therefore, when the overall load is relieved, zones are loaded differently in the material, giving rise to the observed back stress upon unloading. The effect of local heterogeneities on the mechanics of polymers has been recently discussed and reviewed by Rottler [3].

The back stress can be significantly reduced after long relaxation time at constant load as stress relaxation occurs leading to a "re-homogenization" of the material, as shown on **Figure 2c**. Similar results and explanations for *thermally activated stress relaxation* mechanisms in glassy polymers were also made by M. Rink and al. [4].

*3.2 Damage and fracture mechanisms*

The fracture toughness has been measured using three point bending tests on single edge notched bend (SENB) specimens, following the ASTM standard [5]. The results obtained for the neat resin are given in **Table 1**.

	K_{1c} [MPa.$\sqrt{m}$]	G_{1c} [J/m^2]
neat RTM6	0.79 ± 0.075	216.3 ± 32

Table 1. Fracture toughness K_{1c} and G_{1c} measurements of RTM6 through 3point bending test.

These results confirm, as expected, the relatively brittle behaviour of the RMT6 epoxy. We can also extract, from the fracture toughness and yield stress of the resin, an estimate of the size of the plastic zone. The plastic zone size is around one micron, which is quite small and is thus in agreement with the relatively brittle behaviour.

Tensile tests have been performed on notched samples with different notch radius in order to investigate the effect of stress state on fracture strain. The results are synthesized on **Figure 3**, next to the fracture strains obtained from compression, torsion and Iosipescu tests.
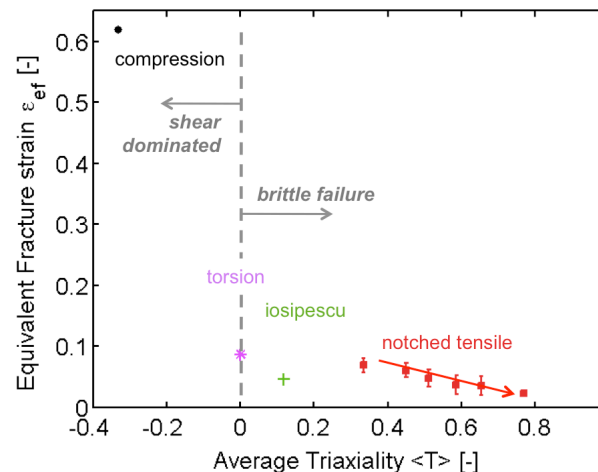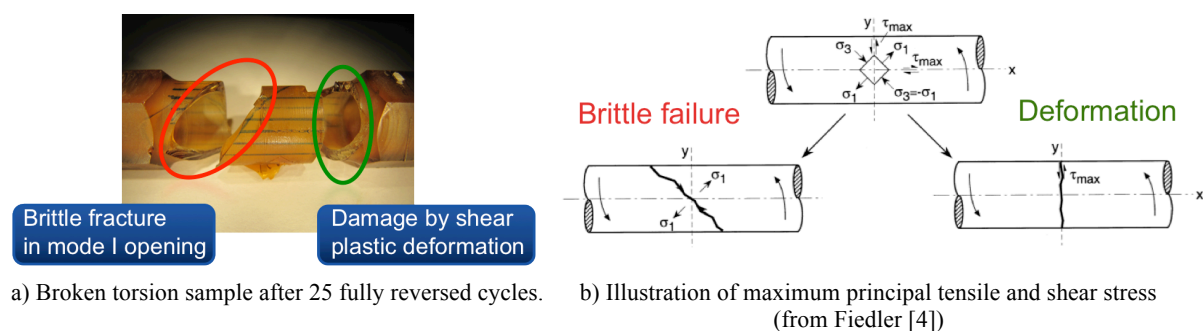

Figure 3. Influence of stress state over fracture strain.

Figure 3 shows that the fracture strains of the notched tensile tests decrease with increasing stress triaxiality, defined as the ratio of hydrostatic stress over equivalent stress.

As illustrated on **Figure 3**, two *independent* and *competing* failure mechanisms were observed during the different types of testing mode. For low triaxiality level, large strain deformation can be achieved before fracture; while brittle fracture occurs quickly at higher stress triaxiality. Indeed, if large strain can be achieved through shear deformation mechanisms, this is possible only when the criterion of brittle fracture is avoided, such as in compression tests. Otherwise, if the geometry of the test specimen gives rise to local stress concentration, the fracture will occur much earlier even though shear deformation is promoted, such as for Iosipescu testing.

By chance, we were able to observe both competing mechanisms in a same torsion specimen, as shown on **Figure 4a**. In this case, the distinction between maximum principal *tensile* stress and maximum shear stress is well observable with respectively 45° and 90° fracture lines, as illustrated on **Figure 4b**.


a) Broken torsion sample after 25 fully reversed cycles.
b) Illustration of maximum principal tensile and shear stress (from Fiedler [4])

Figure 4. Two independent and competing mechanisms for deformation and fracture in RTM6.

4

The fracture surface analysis of both fracture paths confirms the two different mechanisms, with mirror-like surface and river lines pattern for the brittle fracture at 45°, and rough damaged area due to important shear strain for 90° fracture surface. These observations were also reported by Fiedler [6] for another epoxy matrix system.

4 Modelling

In order to make the transition to the composite scale, the constitutive response at the ply scale has been determined by FE simulations on the unit cell of a five harness (5HS) carbon fibre composite with an idealized representation of the yarn morphologies (cf. **Figure 5**).
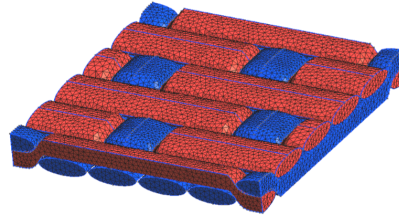

Figure 6. FE Simulation of a representative volume element (RVE) of a 5 harness single ply.

The response of the yarn is predicted using non-linear mean-field homogenization (Mori-Tanaka), as well as for the entire RVE response. Carbon fibres are modelled as ellipsoids with very high aspect ratio and are considered as fully elastic. The matrix response, on the other hand, is modelled at this stage with simple J_2 flow theory. Note that detailed description of the modelling procedure will be given elsewhere [7].

Comparison between experimental and simulated stress-strain curves for tensile tests on a composite with ± 45° of fibre-orientation are shown on **Figure 6**.

Figure 6a illustrates that a simple *elasto-plastic* model for the resin behaviour performs well at low strains ($\varepsilon_{eq} < 0.025$) but does not provide a good prediction of the composite mechanical response at larger strains. The model fails to predict the rehardening: indeed, in addition to neglecting the effect of matrix strain hardening, the significant composite fibre alignment along the loading direction is not properly accounted for. The relative contribution of each mechanism to the composite rehardening will be studied in future work. Moreover, **Figure 6b** shows that the same elasto-plastic model is not capable of reproducing the kinematic hardening of the composite upon unloading, even at low strains. This indicates that the kinematic hardening of the resin response is at the origin of the non-linear unloading behaviour of the composite and must be taken into account.

a) Composite "softening" and "re-hardening"   b) Composite "back stress"
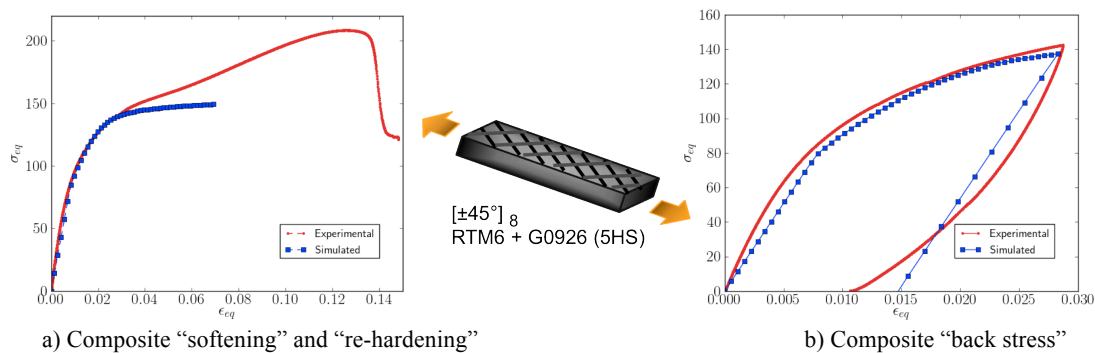
Figure 6. Comparison between model predictions (with a simple elasto-plastic model for the matrix response) and experimental data of a fibre reinforced composite under tensile test at 45∘.

The mechanical response of the overall composite is thus *dominated,* under given loading modes, by the epoxy matrix behaviour. Therefore, a better insight and proper modelling of the intrinsic response of the matrix will lead to a better modelling of the overall composite.

5 Conclusion and perspectives

This study shows that the mechanical behaviour of the RTM6 is complex, involving rate and temperature dependent plasticity, strong kinematic hardening upon unloading and two possible failure modes.

All these effects can potentially play an important role on the composite mechanical behaviour depending on the loading configuration, the geometry and temperature of solicitation.

Future works will focus on identifying more physical and more complex constitutive models involving the different key features discussed above, and which will be used to simulate the response of the composite RVE.

Acknowledgments

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