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MICRO-MECHANICAL MODELING OF THE PRESSURE DEPENDENT FAILURE OF HIGHLY CROSSLINKED EPOXY RESIN

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ABSTRACT

In order to improve the modelling and the prediction of the mechanical behaviour of fibre reinforced polymers, a more detailed description of the constitutive behaviour of their constituents is necessary. A new fracture criterion of the industrial grade epoxy resin RTM 6, widely used in aerospace applications as a matrix of fiber reinforced composites, is presented. The fracture criterion explains the brittle fracture of the resin both in uniaxial tension and compression, despite a much larger ductility in the later case, by the existence of internal small size defects which induce local tensile stresses in their vicinity. Therefore, finite element analyses were carried out in order to compute the stress levels under different loading conditions at the instant and the location corresponding to the onset of failure. The comparison of the stress levels computed in uniaxial tension of notched and unnotched specimens and in pure compression enables an estimation of the aspect ratio of the critical microdefects and of the critical principal tensile stress. Fracture mechanics allows a determination of an upper bound of the size of defect, whose nature remains to be determined.

1 INTRODUCTION

The present study is performed in the context of the increasing use of fibre reinforced polymers to reduce the weight of aircraft structures. The proper modelling and prediction of their mechanical behaviour is often limited by the insufficiently detailed descriptions of the constitutive behaviour of the matrix.

The failure of the industrial grade epoxy resin RTM 6, widely used in aeronautical applications, is investigated in this work. While other studies in the literature were already dedicated to the characterization of the visco-plasticity, creep and thermo-mechanical behaviour of the RTM 6 [1], and of other epoxy resins [2], studies on the failure process and failure criteria in neat epoxy resins are less widespread. Here, experimental results, involving pure compression and tensile tests on notched and unnotched specimens, are used in order to compute the stress levels in RTM 6 specimens under different loading conditions, at the instant and location corresponding to the onset of fracture, using finite element analyses. A brittle type fracture criterion based on the stress concentrating effect of small internal microstructural defects is proposed. The fracture criterion relies on lower scale simulations made on 3D unit cells of RTM 6 containing a defect, which allow the aspect ratio of the critical defects to be determined, while a superior limit of its size is defined thanks to fracture mechanics arguments.

2 MATERIALS, PROCESSING AND TESTING METHODS

2.1 Material

The material considered is the monocomponent HexFlow RTM 6 epoxy resin, supplied by Hexcel. The RTM 6 epoxy resin has been developed in order to be used in the aeronautic and space industry

applications, where it is usually utilized as a matrix in carbon fiber reinforced composites. It is supplied as a premixed system composed of tetra-glycidylmethylenedianiline (TGMDA) epoxy polymer and of two amine curing-agents M-DEA and M-DIPA.

The RTM 6 resin has a high glass transition temperature $T_g = 220$ [°C], extracted from differential scanning calorimetry (DSC), which guarantees good thermal stability and makes it suitable for use up to 180 [°C].

2.2 Processing

The supplied mixture was first degassed during 75 [min] at 90 [°C] under vacuum. Then, the resin was poured into release-agent coated moulds that were pre-heated beforehand. The curing phase was decomposed in two cycles. First, the mixture was heated from 90 [°C] to 130 [°C] at a heating rate of 2 [°C/min], then a low-temperature curing cycle was made by maintaining the temperature at 130 [°C] during 3 hours. This first step is necessary to avoid local over-heating in the slabs and was followed by a three hour post-curing step at higher temperature (180 [°C]), with a heating ramp equal to 2 [°C/min]. The samples were cooled down to room temperature at a cooling rate of 10 [°C/min].

The entire curing process enables the attainment of a high cross-linking percentage (>95%), which is required for aeronautical certification.

2.3 Machining and mechanical testing

Cylindrical resin slabs were machined into small cylindrical specimens for uniaxial compression tests and into cylindrical dog bone specimens for the uniaxial tensile tests. Notched specimens were also produced by machining notches with different radii (1, 1.5, 2, 3 and 5 [mm]) into the dog bone specimens. Single-edge notched specimens were prepared from larger rectangular slabs.

Tensile, compression and fracture toughness tests were carried out on a screw-driven universal testing machine (Zwick-Roell with an external loading cell of 250 [kN]). For all tests, the standard crosshead speed was 1 [mm/min] and the reference temperature was ambient. The strain was measured by extensometer or by a compliance corrected crosshead displacement method, depending on the specimen geometry and loading configurations. The friction and the induced barreling effect during the compression tests could be eliminated thanks to PTFE films inserted between the platens of the testing machine and the specimens.

2.4 Finite element modeling

The finite element simulations were carried out using the commercial finite element code Abaqus. Each mechanical test carried out on the resin specimens was modeled in order to accurately determine the local stress states at the onset of failure. At this stage, the mechanical response of the resin is modeled by providing the macroscopic true stress - true strain curve obtained by a uniaxial compression test at a strain rate of $1.6 \cdot 10^{-4} \text{ s}^{-1}$. The curve was extrapolated up to very large equivalent true strain in order to avoid unrealistic perfectly plastic behavior, if the equivalent true strain becomes locally larger than the macroscopic strain when failure occurs. Therefore, as a first approximation, the material is assumed to be elastic-plastic, strain rate independent and the pressure dependence on plastic yielding is neglected.

In order to study the stress concentrating effect of inherent defects in the material, simulations were carried out on a representative volume element (RVE), which contains a defect. The defect is modeled as an ellipsoidal void, whose volume fraction is small enough not to influence the global mechanical response of the RVE. Furthermore, the volume fraction of the voids is considered to be low enough for not favoring shear yielding in the ligaments between them. It is assumed that inside each macroscopic point there is statistically always a defect oriented such as to maximize the principal stress reached in its vicinity. This assumes that the defects are very small in such a way that a sufficiently large population of defects corresponds to a volume size over which the stress and strain are macroscopically constant or, at least, marginally varying. Fig. 1 shows the critical orientation of the defect with respect to the direction of the applied load (only one eighth of the geometry is modeled).

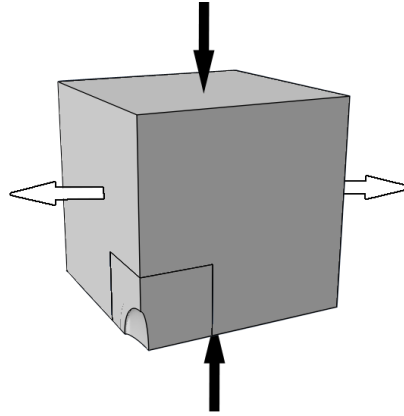


Figure 1: A 3D unit cell of RTM 6 resin containing an ellipsoidal defect. The black (white) arrows show the direction of the applied compressive (tensile) load with respect to the orientation of the defect, maximizing the principal stresses.

Second-order tetrahedral elements have been chosen for the uniaxial compression tests, in order to avoid hourglassing problems when large compressive strain are applied, while a structured mesh of first-order hexahedral elements was preferred around the defect in case of tensile loadings.

3 RESULTS AND DISCUSSION

3.1 Uniaxial tension and compression tests

Uniaxial tensile tests carried out on the cylindrical dogbone specimens exhibit a brittle fracture mode of RTM 6 corresponding to a true tensile stress at failure slightly over 100 [MPa] and a true strain of 0.07 (average values).

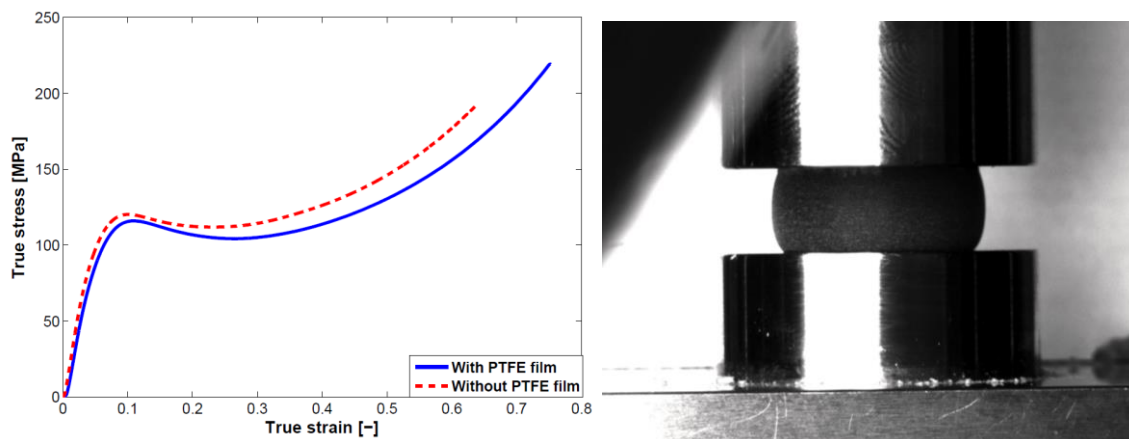


Figure 2: a) Influence of the friction between the platens and the specimen during a uniaxial compression test on the mechanical response of the material, b) illustration of the barreling effect when no PTFE film is used.

Fig. 2a shows the true strain – true stress curve of RTM 6 from a uniaxial compression test. It also illustrates the effect of the friction between the platens and the specimen by showing curves corresponding to tests with and without using a PTFE film. It is clearly observed that both the stress and the strain to failure are significantly reduced in the presence of friction. In both cases, the specimens exhibit brittle type fracture process and surfaces.

Fig. 2b illustrates the detrimental effect of friction as it shows the barrel like shape of the specimen at the end of the test. This barrelling effect leads to significant tensile stresses at the edge of the specimen, which consequently accelerates the occurrence of the first crack in the material, explaining the lower failure stress and strain. Nevertheless, the finite element analyses carried out in order to quantify the magnitude of these tensile stresses showed that they were actually lower than the ones leading to failure in the tensile tests. Therefore, it can be assumed that the overall compressive stress development in the material remains the major cause of failure but that the local tensile stresses accelerate the fracture process by favouring an earlier appearance of the first crack. This statement is supported by the brittle fracture observed even when friction is minimized, that is when no barrelling develops. In that case, the maximum principal stress (MPS) is lowered toward zero and cannot explain the failure of the material. This result was one of the main motivations for a fracture criterion based on a population of pre-existing small size defects.

3.2 Tensile tests on notched specimens

Tensile tests on notched specimens were carried out in order to determine the sensitivity of the RTM6 to the notch radius. Several tests were made for each of the five different notch radii (1, 1.5, 2, 3 and 5 [mm]). Fig. 3a) shows reference true stress – true strain curves from the tensile tests performed on each specimen geometry. The results were used to compute the stress states right before failure occurs, thanks to the finite element analyses. Fig. 3b) shows the profiles of the MPS near the notch (along the ligament perpendicular with respect to the loading direction), for each notch radius, using the average values of the forces at fracture. The MPS reached in the notched specimens are very similar and close to the one reached in uniaxial tensile tests on unnotched specimens. Hence, from a macroscopic view point, it is a first good approximation to assume that the fracture of the RTM 6 specimens is controlled by a critical MPS value. However, this criterion will, of course, not work on pure compression specimens.

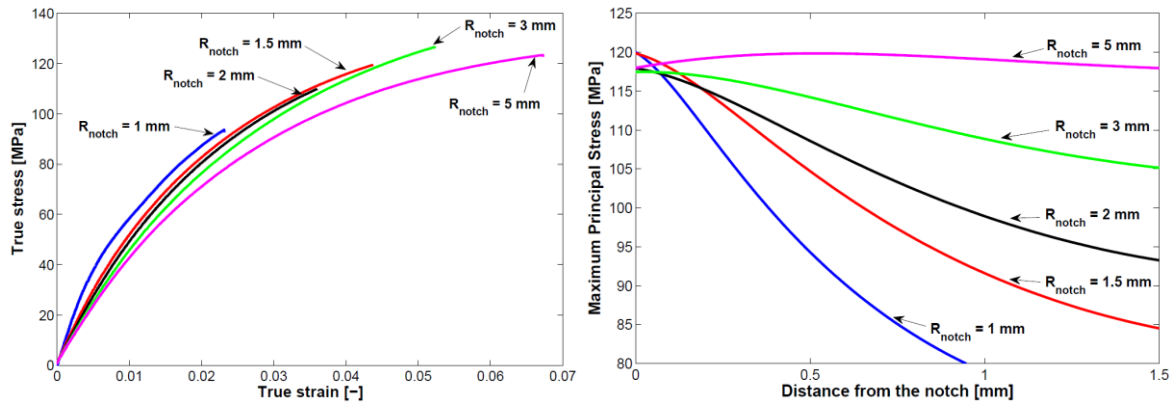


Figure 3: a) Reference true stress – true strain curves of the tensile tests on notched specimens; b) variation of the maximum principal stress as a function of the distance to the notch root right at failure, computed by FEA.

3.3 Finite element analysis of microscale 3D unit cell with defect

The brittle fracture observed both in tension and compression, despite the much larger strain and absolute stress to failure reached during compression tests, hints that the fracture has the same physical origin. In order to reunite both the overall tensile and compression loading conditions, the hypothesis is made that the fracture of RTM 6 is initiated, in all cases, when a critical principal stress is reached around an internal defect of the material. This defect acts as a stress concentrator and will induce the appearance of a local tensile stress at its periphery even under uniaxial compression. Fiedler et al. [3]

and Hobbiebrunken et al. [4] already suggested a weakening effect of the intrinsic defects in the RTM 6 resin, as they showed the increase of its strength when the tensile specimens size was made very small. More interestingly, they also showed a drastic increase of the scatter in the strength data of the fibers. Such an increase points towards the presence of a population of small internal defects in the specimens.

The defect is modeled as a spheroidal void. It is assumed that inside each macroscopic point, there is statistically always a defect that is positioned, with respect to the applied load, as to maximize the principal stress in its vicinity. In order to identify the parameters entering the fracture criterion, the first step was to find out the aspect ratio of the small internal defect that leads to the same local MPS when fracture occurs, both in uniaxial compression and uniaxial tension. To do so, the experimentally observed local stresses to failure were applied to the 3D unit cell represented in Fig. 1, following the direction of the black and white arrows for the uniaxial compression and tension, respectively. While the volume of the defect was kept constant, the aspect ratio A_r was varied. The local MPS in the finite element unit cell was then extracted for each different case. The aspect ratio of the critical defect was then found by simply calculating the difference between the MPS reached in tension and in compression ($= \Delta MPS$), at the corresponding aspect ratio. A ΔMPS larger than zero means that a larger principal stress is attained in tension than in compression, and reversely. The best aspect ratio corresponds to $\Delta MPS = 0$. Fig. 4 shows the variation of ΔMPS with respect to the aspect ratio of the defect. An aspect ratio of the critical defect between 0.24 and 0.30 provides the best predictive capability for this new fracture criterion.

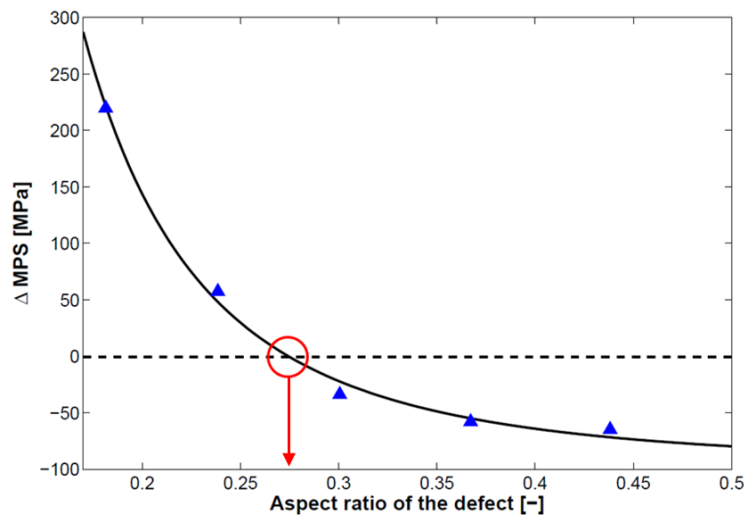


Figure 4: Variation of ΔMPS with respect to the aspect ratio of the defect

Then, the results obtained on the notched specimens can be used to assess the new fracture criterion. The stress state at the location of the MPS in the specimen is chosen to be applied to the unit cell. Fig. 5 shows the MPS attained at the edge of the microscale defect (in the 3D finite element simulation) for each notch radius, and for an aspect ratio of the defect equal to 0.24.

Fig. 5 also illustrates what the statistical variation of the MPS at the edge of the defect would be if it followed the exact same variation as the experimentally measured force at failure of the notched specimens. Despite the scatter of the experimental results of the tensile tests on the notched specimens, it actually proves that the local MPS attained at the tips of the defect in the highly stressed region of the notched specimens is very similar to the one in the unnotched specimens. The idea of a critical defect (that is a defect with an aspect ratio between 0.24 and 0.30, and oriented as to maximize the MPS) located in the vicinity of the notch is thus valid. As the results shown on Fig. 5 are computed for an aspect ratio of 0.24, the observed critical MPS, which is around 290 [MPa], is an upper bound of

the actual critical MPS. Results obtained for an aspect ratio of 0.30 set a lower bound at 220 [MPa].

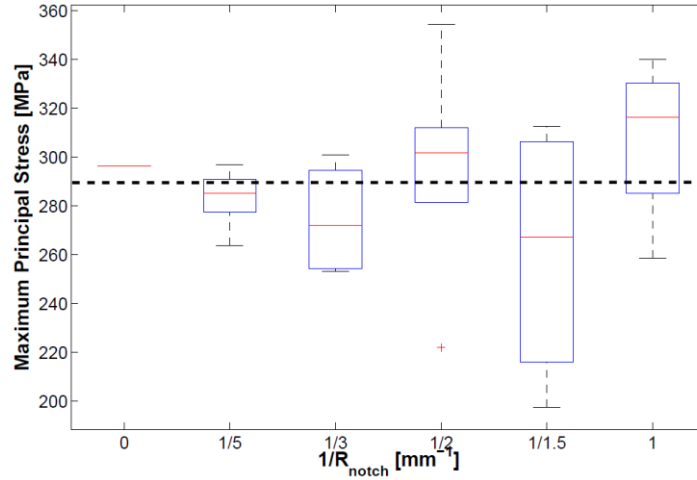


Figure 5: Maximum principal stress at the edge of the microscale defect for each notch radius for a macroscopic loading corresponding to fracture, and illustration of the experimental variability of the tensile tests on the notched specimens over that maximum principal stress; the dashed line gives an approximation of the critical principal stress.

3.4 Size of the microscale defects

A major question that remains is about the size and the nature of the defect. At this stage, only preliminary arguments can be formulated on this topic. First an upper bound of the defect size can be given by considering the concept of crack tip opening displacement (CTOD or δ). Indeed, it is known that a critical δ_c can be estimated thanks to [5]:

$$\delta_c = d G_{Ic} / \sigma_y \quad (1)$$

with $G_{Ic}=200$ [J/m²] and $\sigma_y = 110$ [MPa] for RTM 6 [1]. δ_c of a macroscopic precrack is close to 1 [μm]. As it is assumed that statistically there is always a critical microdefect in the process zone, whose average distance to the crack tip is approximated by δ_c , it can be concluded that the microdefects that initiate the cracking process of a macrocrack are much smaller than 1 [μm]. This statement is also supported by the fact that fractographic analyses of RTM 6 specimens have not highlighted the presence of critical defects that would have a detectable size larger than a micron. This upper limit of the defect size also justifies the fact that the stress state seen by the defect is actually homogeneous and that a same stress state can be applied to the entire unit cell. Now, the exact nature of the defects and whether they are made of nanocracks or simply weaker zones remains to be found.

4 CONCLUSION

The epoxy resin RTM 6 exhibits a much larger ductility in compression than under tensile loadings. However, the brittle fracture in both cases suggests that the larger strain to failure in compression has little to do in the fracture process of the resin. Indeed, in view of the failure mechanics, it is the tensile principal stresses reached locally in the material that is responsible for the final failure of the material. While reaching such a stress under tensile loadings is obviously straightforward, it can only be explained by the presence of intrinsic defects in the material when it is subjected to a uniaxial compression. The proof is the brittle fracture observed in pure compression even when the friction with the platens has been nullified by using a PTFE film as a lubricant.

Therefore, experimental results on various geometries have been used to allow the computation of the stress states in the material right at failure. First, the results of uniaxial compression and tension were used to apply the failure stress to a 3D unit cell of RTM 6 containing a spheroid void to model

the presence of a critical defect, oriented as to maximize its stress concentrating effect. The aspect ratio of the critical defect was determined by finding the shape of the defect that induces the same MPS in both cases. In order to assess the validity of the identified shape of the microdefects, experimental results on notched specimens under tensile loadings were used to extract the stress states at the location of the MPS in the specimen when the fracture occurs. After applying these stress states to the 3D unit cells, it is concluded that the critical shape of the defect remains valid. In other words, the proposed failure criterion is able to encompass stress states varying between pure compression and moderately high triaxialities in the notched specimens, with only two parameters: a local maximum principal stress and a defect aspect ratio. Still, it remains to be seen if the criterion can be applied to other load conditions like at the tip of a pre-crack or involving shear contributions.

Fracture mechanics allows the determination of an upper bound of the size of the defect, which confirms that the defect sees a homogeneous stress field, even in the vicinity of notches. However, the nature of the defect remains to be determined. Even though it has been modeled by a void so far, it must be described more generally as a weak region in the epoxy network, such as a less cross-linked region, for example.

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