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Marina Pecora, Olga Smerdova, Marco Gigliotti. In-situ characterization of the local mechanical behaviour of polymer matrix in 3D carbon fiber composites by cyclic indentation test. *Composite Structures*, 2020, 244, pp.112268 -. 10.1016/j.compstruct.2020.112268 . hal-03490452

HAL Id: hal-03490452

<https://hal.science/hal-03490452>

Submitted on 22 Aug 2022

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IN-SITU CHARACTERIZATION OF THE LOCAL MECHANICAL BEHAVIOUR OF POLYMER MATRIX IN 3D CARBON FIBER COMPOSITES BY CYCLIC INDENTATION TEST

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Keywords: Instrumented indentation, Polymers, Polymer-matrix composites

ABSTRACT - *In this work, a cyclic indentation test is employed for the in-situ characterisation of the local mechanical properties of a PR520 epoxy resin matrix in a 3D carbon fiber composite. Since the evolution of indentation response with time is studied, the cyclic loading allows to characterise both the elastic and the time dependent, viscoelastic, mechanical response of the material simultaneously. The 3D carbon fiber composite used in this study contains a number of large resin pockets between fiber bundles (mesoscopic scale) with a characteristic dimension ranging from several hundred of micrometers to several millimeters. The mechanical behaviour of the polymer matrix is investigated on the external surface and in volume of the composite and compared with the neat polymer. The instantaneous elastic modulus from reloading, the energy ratio and the residual depth are determined from the cyclic material response and compared through a Student t-test based statistical analysis. Results show that there is at least 95% of probability that the neat and in-situ polymer matrix material belong to different populations. However, this difference is rather small (between 1 and 2.5%) and almost constant with cycles. Moreover, a difference between composite core and surface was measured.*

1 – INTRODUCTION

3D textile composites with complex architecture have attracted significant interest of researchers recently, since these materials can be employed for the realisation of advanced aircraft and aerospace structures, due to their good resistance to impact and fatigue [1].

The mechanical behaviour of these materials is usually modelled by using the material properties measured on its elementary constituents, neat polymer matrix and fibres, separately. However, curing of polymer matrix around the fibres, as in Resin Transfer Moulding process, may introduce heterogeneity of the matrix crosslinking, local thermal residual stresses leading to heterogeneous matrix mechanical behaviour and different local (in-situ) mechanical behaviour compared with that of the correspondent neat resin. Any change of mechanical properties of the constituents could be crucial for the failure prediction. One of few experimental techniques allowing for the in-situ characterization of the local mechanical behaviour of the matrix in the composite is the instrumented indentation. The classical protocol associated to this technique consists in penetrating a diamond tip into the material surface and studying the evolution of the displacement as a function of the applied load during a load-unload cycle, then applying the Oliver and Pharr analysis method [2] to calculate Hardness and Elastic modulus from load-displacement indentation curves. In order to apply this protocol to the matrix in the composite, the composite material must have sufficiently large matrix pockets to allow the diamond tip penetrating into the material without any constraint from the surrounding reinforcement fibers. Previous work conducted on various polymer-matrix composite systems [3-10] showed that the main difficulty in matrix characterization with instrumented indentation is to decouple the constraint effect of surrounding fibres from a proper change of polymer matrix properties. Two-dimensional finite element simulations of elasto-plastic composite matrix showed that, to avoid any constraint effect, the resin pockets should be at least 50 times larger than maximal penetration depth [4-5]. Very few experimental studies are available in the

literature aiming at comparing the local in-situ properties of polymer matrix composite materials and the neat resin. In the particular case of the 997-3 epoxy resin / carbon fibre composite system, Gregory and Spearing [4] found that in-situ modulus is 20-30% higher than neat modulus while there was virtually no difference in the measured hardness values. Hardiman et al. [3] found on co-cured neat and in-situ 6376 epoxy resin that, even for unconstrained values, the in-situ modulus depends on matrix pocket radius. That was probably caused by a far-field interphase effect and/or by a difference in local curing conditions near fibres. He concluded that the difference between mean values of unconstrained in-situ modulus and neat modulus of 6376 epoxy resin is about 10% while the difference in hardness is about 6%. In both cases, tests were conducted on unidirectional carbon fibre reinforced composites cured in autoclave, in the direction parallel to fibres. Moreover, the analysis method of nanoindentation data used in previous studies provides only elastic and plastic material properties omitting viscous nature of polymer [11].

The literature lacks of research concerning the in-situ characterisation of the local mechanical properties of a polymer matrix in 3D carbon fiber composites.

The aim of this work is to employ a cyclic indentation test for the in-situ characterisation of the local mechanical properties of a PR520 epoxy resin matrix in a 3D carbon fiber composite. Since the evolution of the cyclic indentation behaviour parameters with time is studied, this kind of loading allows evaluating both the elastic and the time dependent mechanical response of the material. The 3D carbon fiber composite used in this study contains a number of large resin pockets among fiber bundles (mesoscopic scale) with a characteristic dimension ranging from several hundred of micrometers to several millimeters. The mechanical behaviour of the polymer matrix is investigated on the surface and in the volume of the composite and compared with that of the neat material through a Student t-test based statistical analysis carried out on the parameters extracted from cyclic indentation load-displacement curves.

The paper is organised as follows: Section 2 presents the material, the experimental setup and method. Section 3 presents the characterisation of the in-situ cyclic indentation behaviour of the matrix in the 3D composite and the comparison with the neat resin; an analysis of the constraint effect related to the in-situ measurement and a statistical analysis to compare and discuss the results. Finally, Section 4 presents conclusions and perspectives.

2 – MATERIAL, EXPERIMENTAL SETUP AND METHOD

A scheme of the 3D fiber architecture of a composite material similar to the one used in our study is represented in Figure 1 [12]. In Figure 1, the location of resin pockets on the external surface, in the core on the plane perpendicular to the weft direction and in the core on the plane perpendicular to the warp direction have been highlighted.

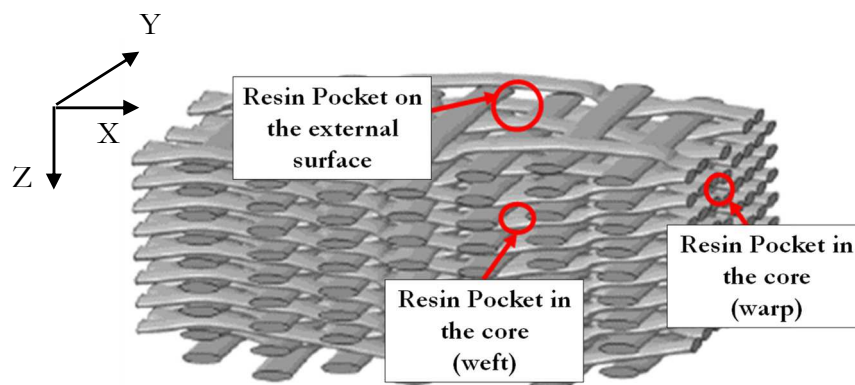


Figure 1. Scheme of a 3D interlock carbon-fiber reinforcement similar to the one used in this study [12].

The polymer material used as a matrix of the composite is the PR520 epoxy resin ($T_g \approx 150^\circ\text{C}$, Tensile Young's Modulus = 4GPa [13]). The samples of neat (bulk) polymer and polymer composite were injection-moulded in large thick plates, which were cut on small pieces and prepared for microindentation by grinding and finishing. The neat polymer samples were tested in the area that corresponds to the core of the initial injected thick plate.

Both neat and in-situ PR520 epoxy resin have been tested using a cyclic indentation procedure on the force controlled Ultra-Micro Indenter Fischerscope H100C equipped with a diamond Vickers tip. The testing protocol (Fig. 2a) consists in 40 triangular wave load-unload cycles between a minimum force of 0.5 mN and a peak force of 10mN with a loading/unloading rate of 2 mN/s. A hold phase at 0.1 mN was introduced at the beginning of the test to perform thermal drift correction on the load-displacement data.

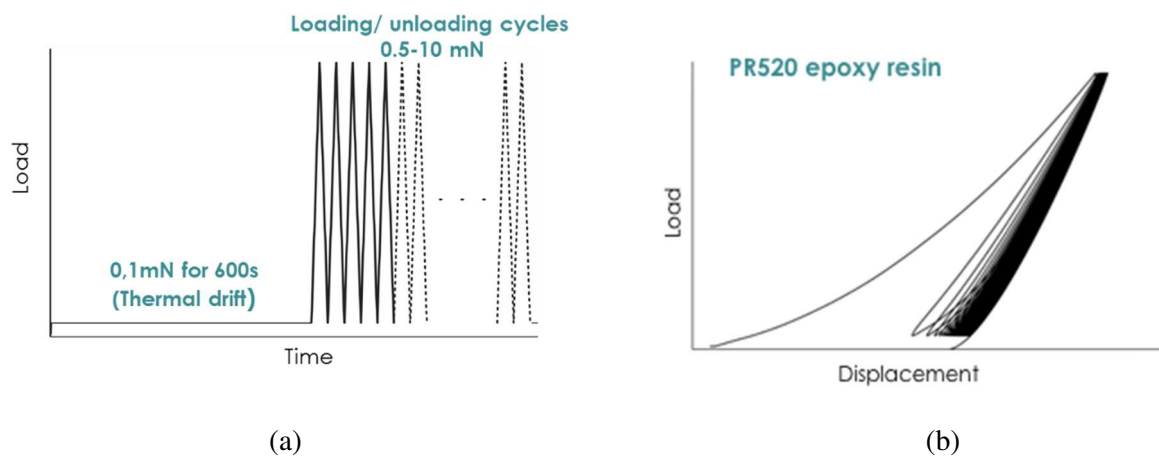


Figure 2. Cyclic indentation test protocol used in this study to evaluate time dependent indentation response of polymer and polymer matrix composite (a) and typical result of cyclic indentation test on PR520 epoxy resin (b).

The load-displacement curves obtained on the PR520 epoxy resin (Fig. 2b) are characterized by large hysteresis loops that evolve with cycles. More details on cyclic indentation and on the response of polymers to this kind of solicitation can be found in reference [14]. This response suggests that a large amount of energy is dissipated in each indentation cycle and that the material behavior is not purely elasto-plastic. The analysis of hysteresis loops is based on the assumption that the indent is formed plastically during the first loading, while all following cycles highlight viscoelastic behaviour. It is therefore possible to define following parameters to describe the mechanical response:

- the instantaneous elastic modulus at each cycle, E_{load}^* , calculated as

$$E_{\text{load}}^* = \frac{1}{\frac{1}{E_{\text{r_load}}} - \frac{1 - \nu_i^2}{E_i}} \quad (1)$$

where ν_i and E_i are, respectively, the Poisson's ratio and Elastic modulus of the indenter, $E_{\text{r_load}}$ is the reduced modulus of the tested material calculated as

$$E_{\text{r_load}} = \frac{\sqrt{\pi}}{2\beta\sqrt{A_p}} S_{\text{load}} \quad (2)$$

where A_p is the projected area of contact, S_{load} is the reload stiffness and β is the shape correction coefficient, equal to 1.012 for Vickers indenter. Unlike usual Oliver & Pharr procedure [2], the instantaneous elastic modulus is evaluated from reloading rather than unloading step. Among many advantages of this choice detailed in our previous paper [14] is a better contact definition between the indenter and the material with presumably conforming surfaces, and a minimization of time-dependency effect on the purely elastic modulus, known as “nose effect”.

- the residual depth after each cycle, h_{min} . This parameter describes the long-term viscoelastic and plastic material response.
- the energy ratio η , which is defined as the ratio of the energy dissipated at the cycle $W_{\text{irr } i}$, to the total work of the cycle $W_{\text{tot } i}$, as

$$\eta_i = \frac{W_{\text{irr } i}}{W_{\text{tot } i}} = \frac{\int_{h_{\text{min } i-1}}^{h_{\text{max } i}} F dh - \int_{h_{\text{max } i}}^{h_{\text{min } i}} F dh}{\int_{h_{\text{min } i-1}}^{h_{\text{max } i}} F dh} \quad (3)$$

The loop area in load-displacement indentation curves W_{irr} is also known as the irreversible work of indentation [15]. For elasto-plastic materials, it has been shown that the energy ratio η is proportional to the ratio between hardness and elastic modulus [16]. According to our assumptions, the energy ratio of the first cycle is associated with plastic and viscoelastic dissipation, while the following cycles dissipate energy only by viscoelasticity.

In order to establish if any apparent local property change observed on measurement performed on different resin pockets in the composite and on neat polymer material is significant and representative of a true property change, a statistical analysis of experimental data has been carried out using the Student t-test [17]. The Student t-test consists in calculating the value of the Z parameter from the mean values (\bar{X}), standard deviations (σ) and number of tests (N) of two data sets, as

$$Z = \frac{|\bar{X}_1 - \bar{X}_2|}{\sqrt{\frac{\sigma_1^2}{N_1} + \frac{\sigma_2^2}{N_2}}} \quad (4)$$

The calculated value of Z is compared to its critical value Z_{crit} found in tables [18] for the chosen level of confidence (99.9% in this case) and for the number of degree of freedom d_f

$$d_f = N_1 + N_2 - 2 \quad (5)$$

The comparison between the two values of Z allows to accept ($Z > Z_{crit}$) or to reject ($Z < Z_{crit}$) the null hypothesis that the two data sets are drawn from different populations with a probability that depends on the level of confidence chosen. The percent difference between two mean values is then calculated as

$$\Delta = \left| \frac{\bar{X}_1 - \bar{X}_2}{\bar{X}_1} \right| \times 100\% \quad (6)$$

3 – RESULTS AND DISCUSSION

3.1 – Fibre constraint effect on in-situ measurements

As reported in the Introduction, previous works carried out on unidirectional carbon-fiber polymer-matrix composites showed that the main difficulty in matrix characterization with instrumented indentation is to decouple the constraint effect of surrounding fibers from a proper change of polymer matrix properties [3-10]. Two-dimensional finite element simulations of elasto-plastic composite matrix of unidirectional carbon fiber reinforced composites showed

that, to avoid any constraint effect, the resin pockets should be at least 50 times larger than the maximal penetration depth [4-5]. To discard this effect in the present study, the distance between the indents and the surrounding fibres was verified by optical microscopy.

Figure 3 illustrates an example of the resin pocket located on the YZ plane, perpendicular to the weft direction. The black circles highlight 9 indentation prints performed in the middle of the pocket. The image shows clearly that the pocket is large enough to consider these measurements unconstrained. Moreover, for the four pockets tested in this plane, the minimal distance from fibres varies from 72 to 376 μm . Microscopic observations were also performed in the zones on YZ plane, perpendicular to the warp direction, and on XY plane, on the external surface of the composite sample. Overall, it is noted that the indentation prints in XY and YZ planes are very far away from the fiber bundles with the lowest distance over 500 μm . Since the maximal penetration depth was less than 1.5 μm in all tests presented here, these observations confirm that the indentation tests are unconstrained according to Gregory and Spearing criterion [4].

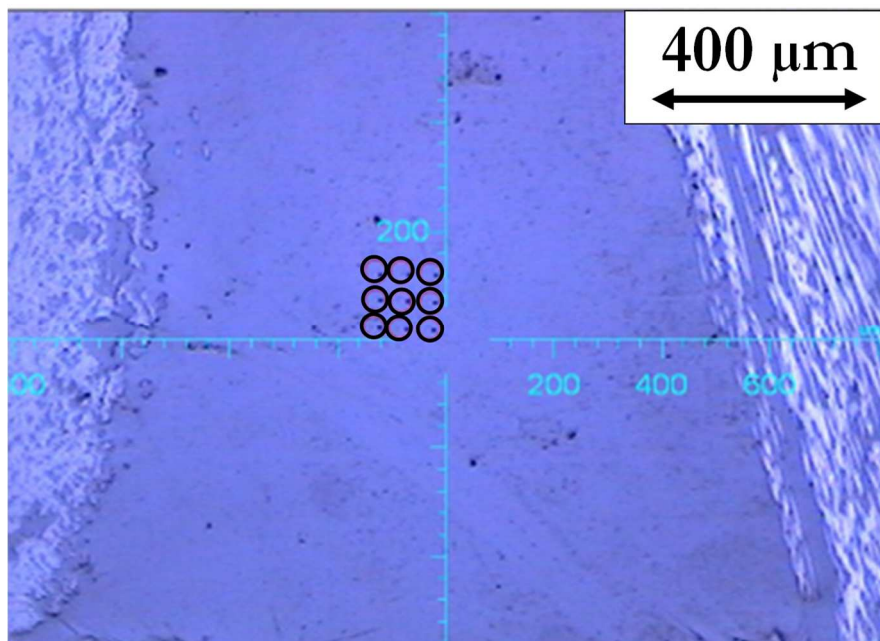


Figure 3. Microscopic image of a resin pocket located in the YZ plane, perpendicular to the weft direction.

However, in the 3D interlock composite used in our study, the constraint effect might be due not only to fibre bundles surrounding resin pockets, but also to the bundles that could pass below the tested surface. Therefore, a second verification was performed by analysing the indentation loading curves.

As reported by Gregory and Spearing [4], unconstrained load-displacement curve should follow a power law, also known as Kick’s law

$$F = Ch^n \tag{7}$$

in which C is a material constant and $n = 2$ for a conical indenter in the case of elasto-plastic materials, while it depends on loading rate in the case of polymers. Since tests performed on neat resin are unconstrained, any deviation of the in-situ loading curve fitting parameters (n and C) from that of neat material is, according to the authors [4], due to the fiber constraint effect. Gregory and Spearing [4] concluded that a test is unconstrained if the deviation between neat and in-situ fitting parameters is lower than 10%. In order to verify if properties that we have measured on the external surface and volume in both warp and weft direction of the composite sample are not affected by any constraint effect, the first loading curves have been fitted with Kick’s law (Eq. 7) and the fitting coefficients C and n have been compared to those of the neat resin. The results are summarized in Figure 4 in which error bars represent \pm one standard deviation.

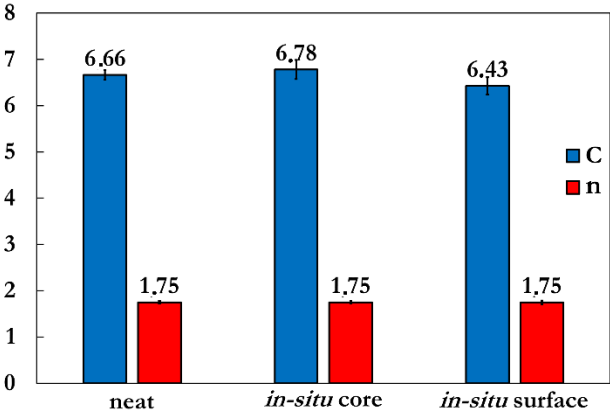


Figure 4. C and n fitting coefficients of the (first) loading curves for neat resin and composite core and surface.

Figure 4 reveals that there is no difference between neat and in-situ exponent n values and a slight difference on curvature C values that, however, does not exceed 5.5%. We can thus consider that properties measured in the composite are unconstrained and that any deviation observed between different in-situ and neat polymer data sets or between several in-situ data sets is due to a proper material difference.

Another possible factor influencing indentation results is the tilt of material surface. For instance, matrix shrinkage near the fiber bundles due to cutting and polishing procedures of samples can induce a tip/sample misalignment in indentation tests. In the present study, composite surface topography measured with interferometry technique revealed that the shrinkage was negligible in the middle of the resin pockets where the indentation tests were performed.

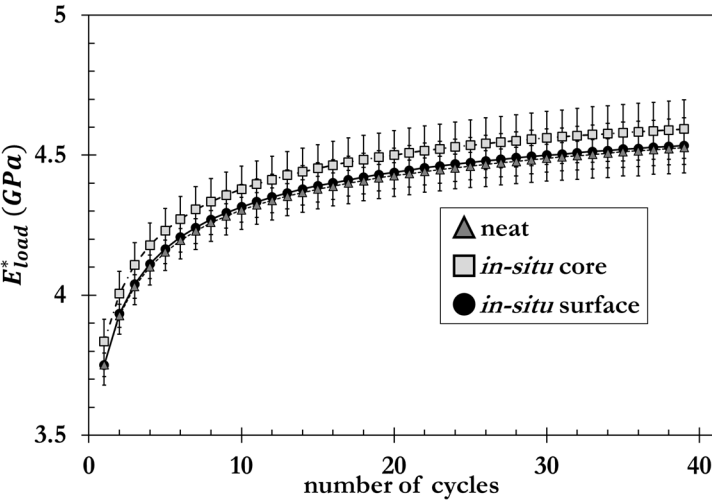
3.2 – Heterogeneity of polymer matrix in the composite

The first step of this study, highlighted in this section, was to study the homogeneity of polymer matrix inside the 3D composite. To do so, several cyclic indentation tests were performed on different resin pockets between fibre bundles on the external surface and in the core of the sample.

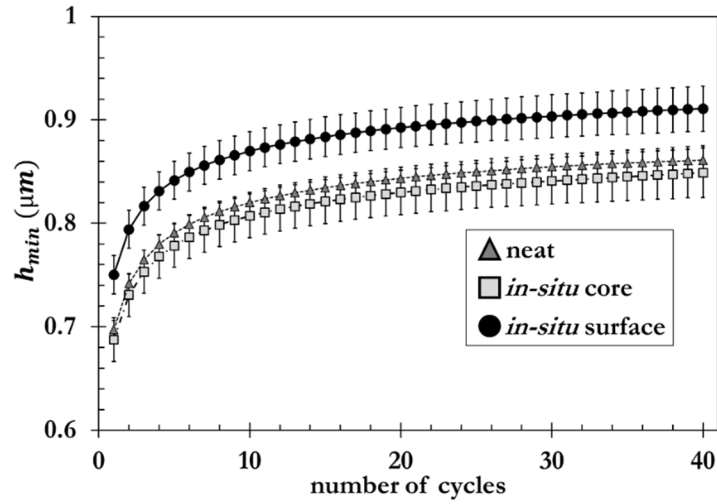
Firstly, the cyclic indentation results obtained on 4 pockets in YZ plane were compared by applying Student t-test on the mean values of 9 indentation between each pair of tested pockets. The test revealed no statistically significant difference for an interval of confidence of 99.9%. The application of the Student t-test for the comparison of mean values of the results obtained in each pair of pockets on the XZ surface (5 pockets, 9 prints par pocket) showed no statistically significant difference with a probability of 99.9% as well. A similar result was obtained from the comparison of the mean value of all pockets on the YZ plane with that of all pockets on the

XZ plane. These results suggest that the polymer matrix in the middle of the pockets is homogeneous in the composite volume. Moreover, the comparison of each pair of pockets on the external surface of the sample (3 pockets, 30 prints per pocket) through the Student t-test for an interval of confidence of 99.9% has not revealed statistically significant difference, showing that the matrix is homogeneous also on the surface.

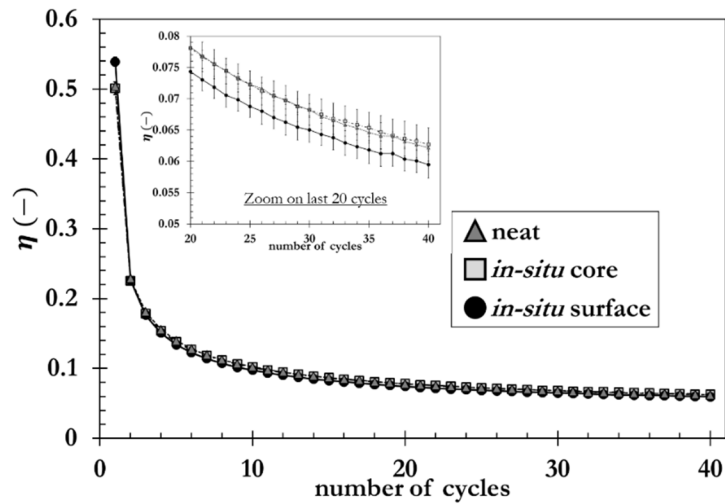
Finally, the average of all resin pockets in the volume and the average of all resin pockets on the external surface were compared. The evolution with cycles of instantaneous elastic modulus, residual depth and energy ratio of in-situ measurements in the core (squares) and on the external surface (circles) is represented in Figures 5a, 5b and 5c respectively. Error bars represent mean value \pm one standard deviation. In Figure 5, the behaviour of the neat resin (triangles) is also illustrated.



(a)



(b)



(c)

Figure 5. Evolution of (a) instantaneous elastic modulus, (b) residual depth and (c) energy ratio with indentation cycles measured on the neat polymer (Δ) and in core (\square) and on the external surface (\circ) of the composite.

Figure 5 shows that, while cyclic behaviour is similar, a slight difference between core and surface mean values is observed. In particular, instantaneous elastic modulus is higher in the sample core, residual depth is higher on the surface while the energy ratio is higher on the surface for the first cycle, and slightly higher in the core for further cycles. The Student t-test was used to compare the two data sets. The values of Z calculated for each parameter are represented in Figure 6 as a function of the cycle number, together with the critical values of Z

for an interval of confidence of 99.9% (upper red dashed line) and of 95% (lower red dashed line).

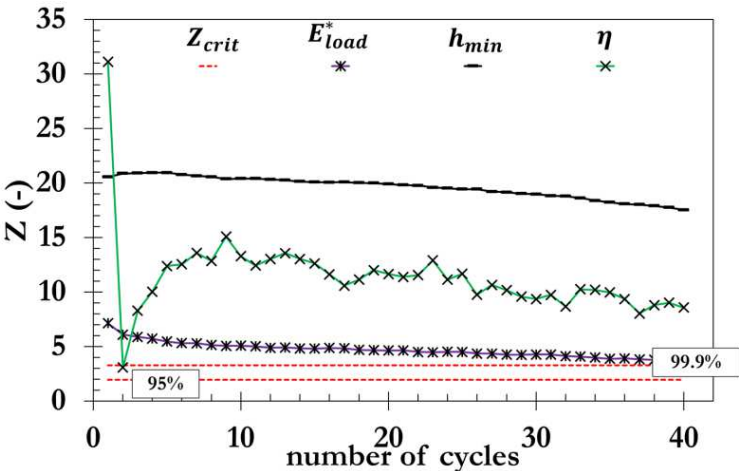


Figure 6. Evolution with cycles of the parameter Z calculated from composite core and composite surface of: instantaneous elastic modulus (*), residual depth (-) and energy ratio (x) and of Z_{crit} for an interval of confidence of 99.9% (upper red dashed line) and 95% (lower red dashed line).

From Figure 6, it appears that there is a probability of 99.9% that the measurements performed on the external surface and in the core of the composite belong to different populations. The percent difference of all parameters between external surface and core in-situ mean values has been quantified. The results for 1st, 10th, 20th, 30th and 40th cycle are reported in Figure 7. It is appropriate to clarify that, in the case of indentation modulus, calculated from the reload curves, the first value corresponds to the first reload, namely the second loading cycle.

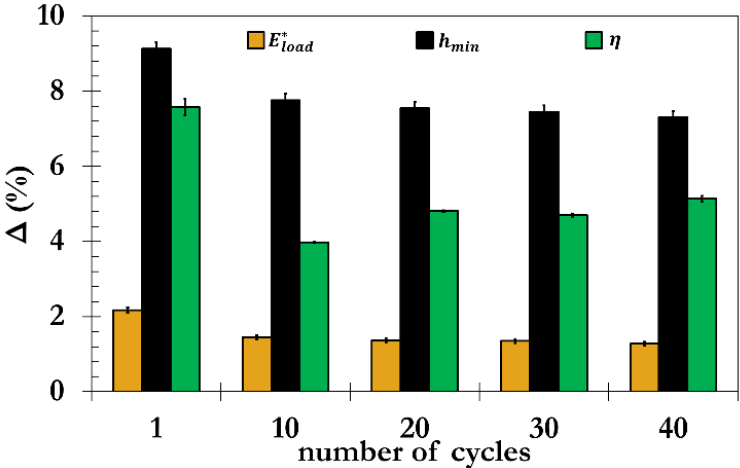


Figure 7. Histogram representing the percent difference between composite core and composite surface mean values of instantaneous elastic modulus (yellow), residual depth (black) and energy ratio (green) for 1st, 10th, 20th, 30th and 40th cycle.

From Figure 7, we can observe that, except for the energy ratio, the percent difference between the two data sets is almost constant with cycles, confirming that the cyclic behaviour of the two materials is similar. This difference is about 2% on instantaneous elastic modulus and 8% on residual depth. The difference on the energy ratio at the first cycle is about 8%, while for the subsequent cycles it is between 4 and 5%. These small variations of the energy ratio for second and further cycles are most likely due to the accuracy in the calculation of this parameter due to trapezoidal integration. Indeed, the zoom in Figure 5c shows that the loop become very thin with cycles.

3.3 – Neat vs. in-situ properties of PR520 epoxy resin

Once the heterogeneity of the composite matrix is established, the indentation response of the composite matrix external surface and core will be compared with that of the neat polymer material manufactured by the same method but without fibres.

For this analysis, 25 cyclic indentation tests have been performed on the polished surface of a neat polymer sample cut from the thick polymer plate. The evolution with cycles of instantaneous elastic modulus, residual depth and the energy ratio of neat PR520 epoxy resin is represented in Figure 5a, 5b and 5c by triangles.

Figure 5 reveals that the evolution with cycles of the different parameters is similar for the three materials, however a slight difference in mean values exists. In particular, the instantaneous elastic modulus of the neat polymer is rather close to composite surface and slightly lower than in the sample core. The residual depth of the neat polymer is slightly higher than the composite core and slightly lower than the composite surface. Finally, the energy ratio at the first cycle of the neat polymer is close to the composite core and lower than the composite surface. From the

second cycle, the energy ratio is close between the three materials, although small difference persists (cf. zoom in Fig. 5c).

In order to evaluate if this difference is significant from a statistical point of view, the Student t-test was applied again. The properties of the neat polymer were compared at first with the mean properties of all pockets in the composite core. The values of Z calculated at each cycle are represented in Figure 8 as a function of the number of cycles for all the parameters. The dashed red lines represent the values of Z_{crit} for an interval of confidence of 99.9% (upper line) and 95% (lower line).

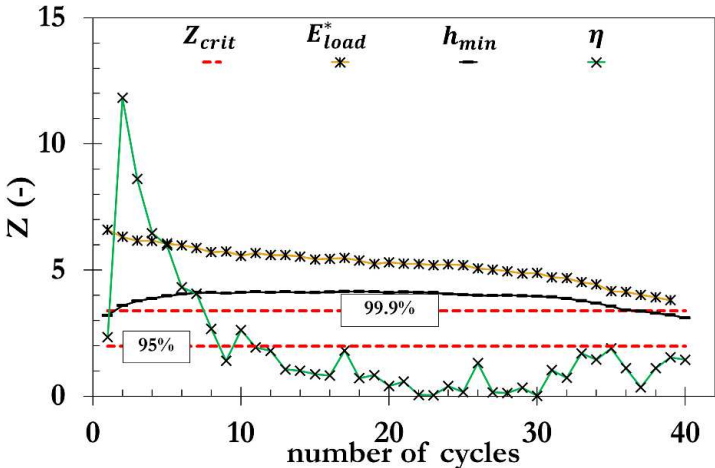


Figure 8. Evolution with cycles of the parameter Z for the comparison between neat polymer and composite core mean values of instantaneous elastic modulus (*), residual depth (-) and energy ratio (x) and of Z_{crit} for an interval of confidence of 99.9% (upper red dashed line) and 95% (lower red dashed line).

The result of statistical test suggests that, except for the energy ratio, there is a 99.9% of probability that the values of the parameters neat and in-situ in the core belong to different populations. The percent difference has been quantified and the results are reported in Figure 9.

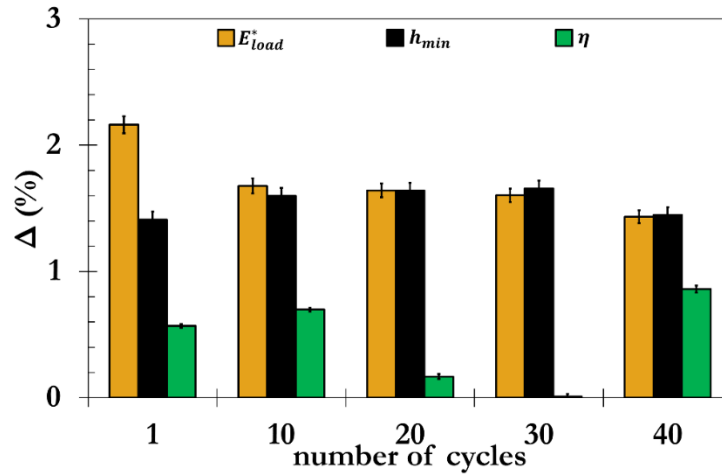


Figure 9. Histogram representing the percent difference between neat polymer and composite core mean values of instantaneous elastic modulus (yellow), residual depth (black) and energy ratio (green) for 1st, 10th, 20th, 30th and 40th cycle.

In Figure 9, we can observe that the percent difference between neat polymer and composite core mean values is rather small, it lies between 1% and 2.5%. This difference is almost constant with cycles, except for the energy ratio, which is again affected by the accuracy of its calculation.

The last comparison that was considered is between the neat polymer and the external surface of the composite. The values of Z of all parameters as a function of the number of cycles are represented in Figure 10.

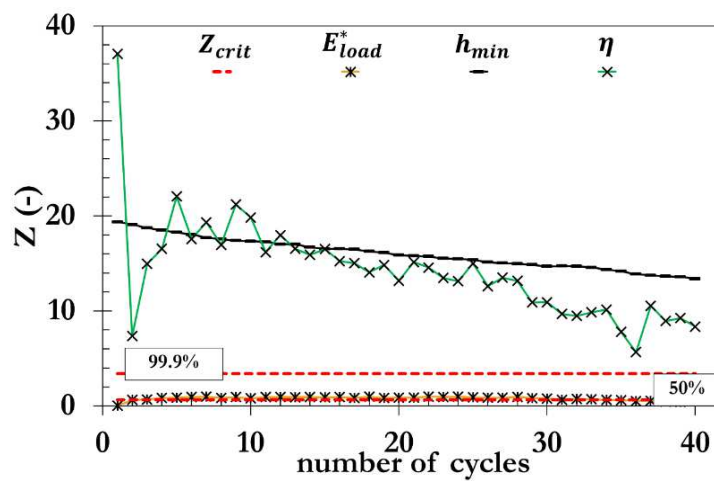


Figure 10. Evolution with cycles of the parameter Z for the comparison between neat polymer and composite surface mean values of instantaneous elastic modulus (*), residual

depth (-) and energy ratio (\times) and of Z_{crit} for an interval of confidence of 99.9% (upper dashed red line) and 50% (lower dashed red line).

In this case, there is no statistically significant difference on instantaneous elastic modulus as its Z values are close to the Z_{crit} corresponding to an interval of confidence of 50% (lower red dashed line). The other parameters belong, however, to different populations for an interval of confidence of 99.9% (upper red dashed line). Their percent difference is about 4% for residual depth, 7% for energy ratio at the first cycle and 5% for energy ratio from the second cycle as represented in the histogram in Figure 11.

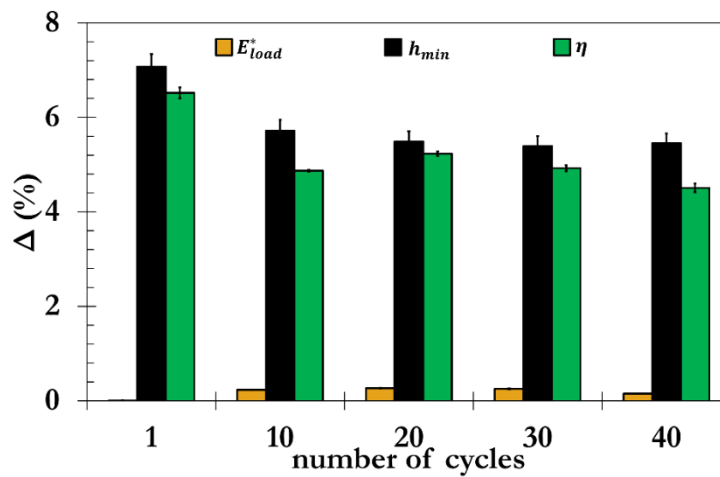


Figure 11. Histogram representing the percent difference between neat polymer and composite surface mean values of instantaneous elastic modulus (yellow), residual depth (black) and energy ratio (green) for 1st, 10th, 20th, 30th and 40th cycle.

3.4 – Summary and discussion

All results from the analyses presented in Sections 3.2 and 3.3 are summarized in Table 1. In this table, the maximum values of percent difference on E_{load}^* and h_{min} between all cycles are given and the percent difference on energy ratio is separated on the first cycle and the maximum of further cycles. The data presented in this table highlights that although small but statistically significant difference exists between the polymer and the composite core, the highest discrepancy is between the composite surface and both its core and neat polymer. The closeness of neat polymer to the composite core rather than its surface can be explained by the fact that

the tested surface of the neat polymer was in the core of the thick polymer plate. Whether the surface of polymer plate is different from the composite surface is a matter of future research.

As supported by Figs. 7, 9 and 11 and Table 1, the difference of the instantaneous elastic modulus is small in all cases and nearly constant with cycles. Although the slight variations with cycles might be from the material itself, it is also possible that they are due to the accuracy of fitting process, from which this parameter is determined. The percent differences measured on the residual depth and energy ratio are significantly higher and do not lie in the possible measurement error range. According to the framework of our analysis, the difference in energy ratio at the first cycle means that plastic and short time viscoelastic behaviour are affected. A decrease of the percent difference between the 1st and 10th cycle, observed for composite surface – polymer and composite surface – composite core analyses in Fig. 7 and 11, may be interpreted as a change in intermediate time viscoelastic response. Once this difference is established, the constant gap between data in further cycles suggests that the long time viscoelastic behaviour between the surface and the core of composite or the neat polymer is not different. This behaviour is confirmed by the evolution of the residual depth, which is the most accurately determined parameter since it is directly measured by the depth sensor.

	E_{load}^*	h_{min}	η^{1st}	η
composite core - composite surface	2%	9%	8%	5%
neat polymer - composite core	2%	1.5%	<1%	<1%
neat polymer- composite surface	<1%	7%	7%	5%

Table 1. Maximum percent difference from cyclic response of polymer matrix in 3D composite and the neat resin in different configurations.

The small observed gap between surface and core properties can be ascribed to the so called “skin-core” structure observed in injected thermoplastic parts [19-23]. In the case of thermoset resin, it is possibly due to slightly different crosslinking degree in different zones of the composite samples caused by thermal gradients in the mold.

4 – CONCLUSIONS

In this paper, a cyclic indentation test was employed for the in-situ characterisation of the local mechanical properties of a PR520 epoxy resin matrix in a 3D carbon fiber composite. Since the evolution of the cyclic indentation behaviour parameters with time is studied, this kind of loading allows evaluating both the elastic and the time dependent mechanical response of the material. The 3D carbon fiber composite used in this study contains a number of large resin pockets among fiber bundles (mesoscopic scale) with a characteristic dimension ranging from several hundred of micrometers to several millimeters. The mechanical behaviour of the polymer matrix is investigated on the surface and in the volume of the composite and compared with that of the neat material through a Student t-test based statistical analysis carried out on the parameters of the cyclical indentation test, namely the instantaneous elastic modulus, the energy ratio and the residual depth. The main conclusions that arise from this analysis are:

1. Far from fibre bundles, the polymer matrix is homogeneous in the core as well as on the sample surface;
2. The in-situ local properties measured in the composite sample core are slightly different from those measured on the composite sample surface, but significant from a statistical point of view. This difference has been attributed to a phenomenon similar to the “skin” effect observed on the injected thermoplastic parts, possibly due to slightly different crosslinking degree in different zones of the composite samples.

As a further research study, it would be interesting to compare the surface of the neat polymer with the surface of the composite injected by the same process.

Acknowledgements

The authors would like to acknowledge SAFRAN – SNECMA (now SAFRAN Aircraft Engines) for providing the material. The work subject pertains to the French Government program “Investissements d’Avenir” (LABEX INTERACTIFS, reference ANR-11- LABX-0017-01; EQUIPEX GAP, reference ANR-11-EQPX-0018).

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