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► To cite this version:

Quentin Drouhet, Fabienne Touchard, Laurence Chocinski-Arnault, David Mellier, Teddy Fournier.
The yarn/matrix interface in composite materials: fragmentation tests and micro-CT based digital
volume correlation. 21st European Conference on Composite Materials (ECCM21), Centrale Nantes;
Nantes Université, Jul 2024, Nantes, France. pp.262-269. hal-04655150

HAL Id: hal-04655150

<https://hal.science/hal-04655150>

Submitted on 24 Jul 2024

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THE YARN/MATRIX INTERFACE IN COMPOSITE MATERIALS: FRAGMENTATION TESTS AND MICRO-CT BASED DIGITAL VOLUME CORRELATION

Q. Drouhet¹, F. Touchard², L. Chocinski-Arnault², D. Mellier², T. Fournier¹

¹Centre Technologique Nouvelle-Aquitaine Composites & Matériaux Avancés (CANOE), Bât
Cheminov – ENSMAC, 16 avenue Pey Berland, 33600, Pessac, France
Emails: drouhet@plateforme-canoe.com, fournier@plateforme-canoe.com
Web Page: <https://www.plateforme-canoe.com>

²Pprime Institute, Department of Physics and Mechanic of materials, ENSMA, 1 avenue Clément
Ader, 86961, Futuroscope Chasseneuil, France
Emails: fabienne.touchard@ensma.fr, laurence.chocinski@ensma.fr, david.mellier@ensma.fr
Web Page: <https://pprime.fr>

Keywords: natural fibers, hydrothermal ageing, 3D segmentation, damage quantification

Abstract

Competing with glass-reinforced composites, natural fibre-reinforced composites are booming, and are of increasing interest to manufacturers for structural applications. A key issue of this kind of composite is investigated: the yarn/matrix interface behaviour. Different states of ageing and yarn treatments were studied through quantitative damage analysis and evaluation of the interfacial shear strength (IFSS). First, at the macroscopic scale of a hemp-based woven composite, damage rates were calculated from micro-CT image acquisitions before and after mechanical tests for different fibre orientations, matrices and hydrothermal ageing states. Results showed the predominant role of the yarn orientation in the development of interfacial damage. To further investigate the yarn/matrix interface, single yarn composites were manufactured and subjected to fragmentation tests. IFSS values were calculated for treated and untreated hemp yarn. Results showed that plasma treatment carried out at CANOE produced promising results. Finally, a damage scenario for a single yarn composite was established performing a fragmentation test coupled with micro-CT scans and digital volume correlation. This enabled the tracking of the full-field strain evolution at the yarn/matrix interface throughout fragmentation testing and a better understanding of the stress and strain redistribution that occurs when fibre breaks and debonding appear in the gauge length.

1. Introduction

Fibre-reinforced polymer composites take an important part in the transport and energy industry [1]. One of the major subject is still to better understand the behaviour of the yarn/matrix interface which allows transfer of the load from the matrix to the yarns. Macroscopic mechanical tests such as tensile tests on woven composites can be carried out for different fibre orientations to study the adhesion quality at the yarn/matrix interface [2]. At a lower scale, micromechanical tests can be performed such as push-out, microbond test or fragmentation test [3]. The latter was first introduced by Kelly and Tyson in the 1960s [4]. The principle of the fragmentation test consists in loading in tension a single yarn embedded of a matrix material in the shape of a dog-bone sample. During the test, yarn breaks occur when failure strain is reached in a section of the yarn. This implies a redistribution of the stresses after each yarn break. The measurement of fragment lengths in the sample allows the determination of the adhesion quality by calculating the interfacial shear strength (IFSS). The analysis of interface quality by fragmentation test is widely used, whether for synthetic fibres [5,6] or for plant fibres [7,8]. For natural

yarn composite, the study of adhesion quality is essential due to the hydrophilic behaviour of the plant fibres [9, 10]. Some studies focussed on physical or chemical treatment for improving the adhesion quality at the fibre/matrix interface [11].

In this study, all the mechanical tests were combined with micro-CT image acquisition. X-ray micro-CT technique is a powerful tool for the investigation and characterisation of the internal 3D structure of materials [12]. Micro-CT image acquisitions were carried out for woven composites and for single yarn composites to evaluate the damage ratio before and after mechanical tests for different ageing states. An additional fragmentation sample was also scanned to perform digital volume correlation (DVC). As with the widely known DIC method, DVC relies on tracking subvolumes of voxels to determine the relative displacement vectors in space between a reference and a deformed volume [13, 14]. Thus, DVC allows three dimensional strain measurements. To carry out DVC calculation, a 3D speckle is necessary [15-17]. In the present work, small particles (5 μm in diameter) were added in an epoxy matrix of a single hemp yarn composite in order to analyse the 3D strain fields during fragmentation test by combining DVC and micro-CT.

2. Material and methods

2.1. Woven composites

The studied composites are made of 7 plain-woven plies of hemp fabrics and three different polymer matrices : two epoxy systems (Epolam 2020 and Greenpoxy 56) and a MMA-based polymer named Elium 180, produced by the company Arkema. Composites plates were manufactured by the vacuum infusion process and rectangular samples were cut to provide two reinforcement orientations ($\pm 45^\circ$ and $0^\circ/90^\circ$). The overall dimensions of the samples were 140 mm in length and 20 mm in width. The thickness of the coupons was about 3.5 mm. For all the studied composites, to ensure comparability, the fiber mass fraction was around 46%.

2.2. Single hemp yarn composite

To manufacture single hemp yarn composites, hemp yarns were extracted from fabrics used to elaborate the woven composites. The matrices used are the thermoset polymers Epolam 2020 and Greenpoxy 56. After drying in an oven for 48h at 40°C , the yarns were placed in a rectangular mould with a low pre-tension before casting the resin. Sample geometry is dog-bone shaped, which enables to concentrate the strain and stress in the gauge length and around the yarn. Some hemp yarn were preliminary treated to improve the adhesion quality. A plasma treatment was performed at the CANOE platform and single hemp yarn composites were then manufactured as described above.

Finally, an additional single hemp yarn composite was manufactured for micro-CT based DVC calculations. Here, the matrix (Epolam 2020) was filled with an Al_2O_3 powder of 5 μm in diameter allowing the creation of a 3D speckle in the material [18]. It made it possible to apply the correlation method but it has to be noticed that it also can slightly modify the matrix behaviour [17].

2.3. Tensile tests

Tensile tests were performed using an Instron 5982 testing machine equipped with a load cell with a capacity of 100 kN. All the samples were loaded in the longitudinal direction with a crosshead speed of 0.5 mm/min.

2.4. Fragmentation test

The principle of the fragmentation test is to apply a longitudinal stress on a single yarn embedded in a polymer matrix. Tests were carried out using an Instron 5982 tensile machine with a crosshead speed depending on the matrix.

For the single yarn composite used for DVC calculations, a step-by-step fragmentation test was carried out with the crosshead speed corresponding to the hemp/Epolam 2020 material ($5 \cdot 10^{-3}$ mm/mim). At each step, the sample was unloaded and scanned by micro-CT. A first micro-CT scan was registered at the initial state, before loading. Six progressive steps were then performed, before the complete failure of the specimen at the 7th one.

2.5. Micro-CT

Micro-Ct image acquisitions were performed using an UltraTom CT scanner from RX Solution. A Hamamatsu micro focus sealed X-ray tube operates at an amperage range of 0-500 μ A and a voltage range of 20-150 kV with a maximum power of 75 W. The parameters used depend on the scanned section of the specimen. Table 1 shows the image acquisition parameters and the spatial resolution obtained for each case study.

Table 1. Micro-CT image acquisition parameters for each type of sample and corresponding spatial resolution.

Specimen Type	Amperage (μ A)	Voltage (kV)	Scanned zone dimensions (mm)	Acquisition time (hours)	Spatial resolution (μ m)
Woven composite	250	60	40*20*3.5	3	15
Single hemp yarn composites (IFSS)	90	50	6.0*3.0*2.5	8	1.5
Single hemp yarn composites (DVC)	90	50	3.3*3.1*2.9	4	3

2.6. Digital volume correlation

Digital volume correlation based on micro-CT scans were performed on the single yarn composite sample made of epoxy filled with Al_2O_3 powder and a hemp yarn. The correlation calculation requires a unique random pattern, recorded after each step of the fragmentation test. The first picture taken at the initial state is the reference image which is compared to an other one taken at a deformed state. The first picture is divided into small subsets, each one being a measurement point, characterised by its grey level distribution. The principle consists in seeking, for each subset of the reference image, the most similar one in the deformed image, using a correlation function. Figure 1 shows a scheme of the principle of the DVC calculations.

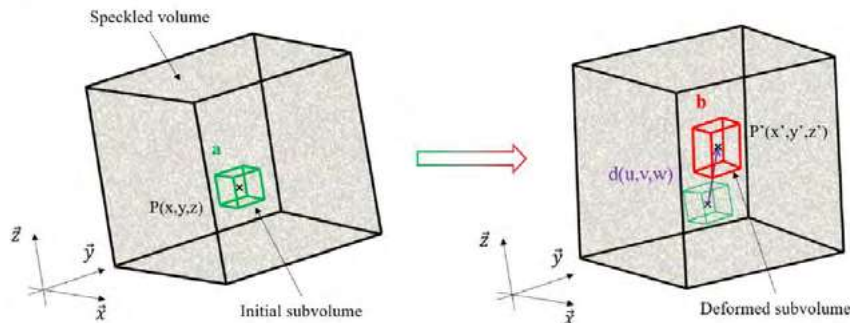


Figure 1. Schematic principle of the DVC process.

In this study, DVC calculations were performed using the open source Matlab program called Augmented Lagrangian Digital Volume Correlation (ALDVC) [19]. ALDVC is a hybrid DVC algorithm

which combines fast computation times and the advantages of finite element-based global methods. At each step of the fragmentation test, 400 tomographic slices in the XY plane containing $1112 \text{ pixels} \times 1037 \text{ pixels}$ were considered, representing about 460 million voxels. Due to the large amount of data, the calculations were carried out on the laboratory's computing machines equipped with a processor Intel Xeon Gold 5218 2.3 GHz and a RAM memory of 256 GB. One DVC calculation between two steps of the fragmentation test lasted about 12 hours.

3. Results and discussion

3.1. Damage rates in woven composites

Hemp-reinforced woven composites were loaded in tension until failure before and after 30 cycles of hydrothermal cycles. One cycle consisted in immersing samples in water at 60°C until reaching the maximum water uptake and drying them in an oven at 40°C for 2 days. The maximum water uptake was reached after 12 immersion days. Figure 2 shows the volumetric reconstructions of micro-CT scans for hemp/Epolam 2020 samples before and after mechanical testing and before and after ageing. In the micro-CT scans, the hemp yarns oriented at $+45^\circ$ and -45° are clearly visible. In the segmented images, the damage is shown in red and the rest of the sample in grey to facilitate analysis. For the "Ambient Storage" sample, before the tensile test, no damage is visible, with the exception of a few porosities created during the manufacturing process. After mechanical test, some damage essentially localized near the fracture zone and globally oriented at 45° can be observed. After 30 hydrothermal cycles, the succession of water absorption and desorption phases led to the creation of damage throughout the sample. Then, after the tensile failure, it can be seen that this damage has developed considerably, not just near the fracture zone, but in the whole sample. The same type of observations was made for other hemp-reinforced composites studied.

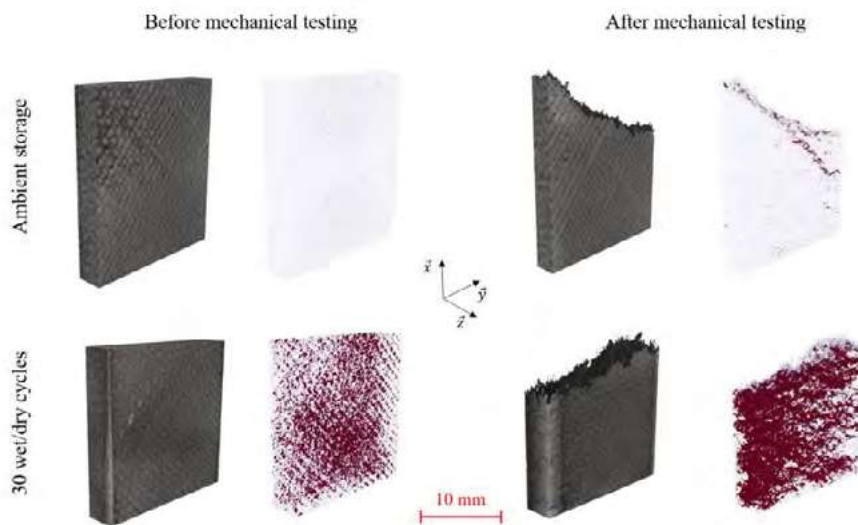


Figure 2. 3D reconstructions of micro-CT scans and segmented volumes for the $\pm 45^\circ$ hemp/Epolam 2020 samples before and after mechanical testing for the "Ambient storage" condition and after 30 hydrothermal cycles.

3.2. Single yarn composites

To better understand the creation of damage at the yarn/matrix interface, single yarn composites have been studied. At this lower scale, damage rate as a function of the ageing state was evaluated, values of interface shear strength (IFSS) were calculated for different pairs of yarn/matrix and a step-by-step

fragmentation test coupled with micro-CT scans and digital volume correlation (DVC) calculation was performed.

3.2.1. Damage rates in fragmentation samples

In this part, single hemp yarn composites made of Epolam 2020 were investigated and the effect of hydrothermal ageing was analysed. Samples were scanned by using micro-CT set-up and images were segmented in order to identify damage such as debonding between yarn and matrix. The hydrothermal cycles were the same as those applied to woven composites. Figure 3 shows a plot of the damage rates as a function of the number of hydrothermal cycles. The segmented volumes are placed next to the corresponding points, with the damage indicated in red and the yarn in blue. For “Ambient storage”, some red voxels can be seen, due to small lacks of impregnation of the matrix. Then, After the first ageing cycle, half of the voxels analysed are associated with damage, and the damage rate curve appears to reach a plateau after 10 hydrothermal cycles. This high increase in the damage rate from the first wet/dry cycle has also been observed for woven composites.

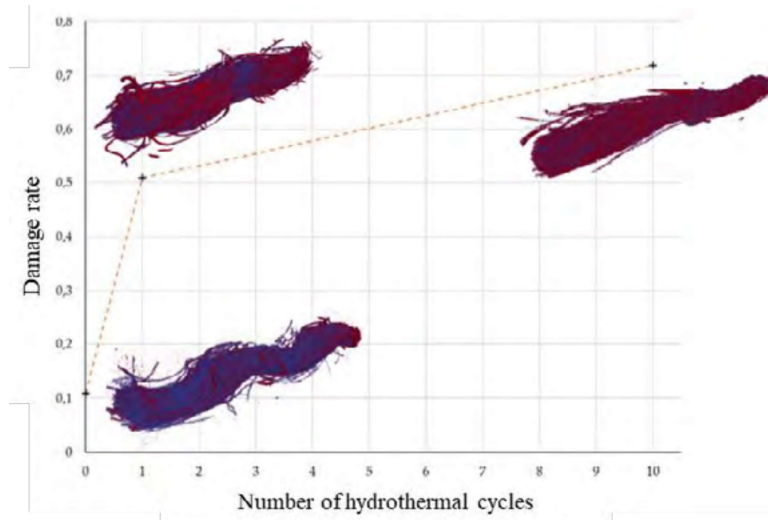


Figure 3. Damage rate as a function of the number of hydrothermal cycles for hemp/Epolam 2020 single yarn composites and associated segmented images.

3.2.2. Interfacial shear strength (IFSS)

First, two pairs of hemp/epoxy samples were analysed: hemp/Epolam 2020 and hemp/Greenpoxy 56. The second matrix is made of a partially bio-based epoxy. Fragmentation tests were carried out using a crosshead speed of $5 \cdot 10^{-3}$ mm/mim for hemp/Epolam 2020 samples and $7.5 \cdot 10^{-4}$ mm/mim for hemp/Greenpoxy 56 ones. Equation 1 enables to calculate the interfacial shear strength with ϕ the mean diameter of the yarn, l_c the critical length, depending on the length of all the fragments obtained after fragmentation tests and $\sigma_f(l_c)$ the maximum stress of a hemp yarn having a length equal to l_c [4,20]. The determination of l_c and $\sigma_f(l_c)$ is based on a Weibull distribution law [21].

$$IFSS = \frac{\phi \times \sigma_f(l_c)}{2 \times l_c} \quad (1)$$

Results show differences between IFSS values calculated for the two hemp/epoxy samples: with Epolam 2020, the IFSS value was found equal to 44.6 ± 13.0 MPa and the IFSS value of hemp/Greenpoxy 56 samples was equal to 26.4 ± 7.7 MPa. Even if the two matrices are epoxy polymers, results show that little chemical modifications are enough to modify the adhesion quality.

In order to quantify the effect of the interfacial bonding at the yarn/matrix, plasma treatment was then performed on hemp yarns at the CANOE platform and IC2MP lab. Samples of hemp/Epilam 2020 were studied, some with plasma-treated hemp yarns and others with untreated yarns. Results showed that the IFSS value is multiplied by 3 after plasma treatment [8].

All these results reveal the complex behaviour of the yarn/matrix interface. To gain a better understanding of the interfacial behaviour, a fragmentation test coupled with micro-CT and DVC calculations was carried out.

3.3. 3D full-field strain maps

At first, in order to better track the yarn break development, 3D segmentation of a micro-CT sub-volume was performed at each step of the fragmentation test. Figure 4 presents the results obtained for step 0 (before loading), steps 5 and 6. The hemp yarn, made of several fibres, can be seen in grey colour and the voids are represented in red colour. Some porosity can be observed at the initial state (Figure 4a). In Figure 4b, the three yarn breaks (YB) are clearly visible, the one that has spread the most being the one that is centred and appeared first (YB1). After the sixth interruption of the fragmentation test, the matrix crack that started from YB1 propagated so widely that it reached the limits of the segmented zone, which explains its square shape (Figure 4c). It can be noticed that the yarn breaks lead to axisymmetric matrix cracks centred on the yarn, which is consistent with the fragmentation test theory.

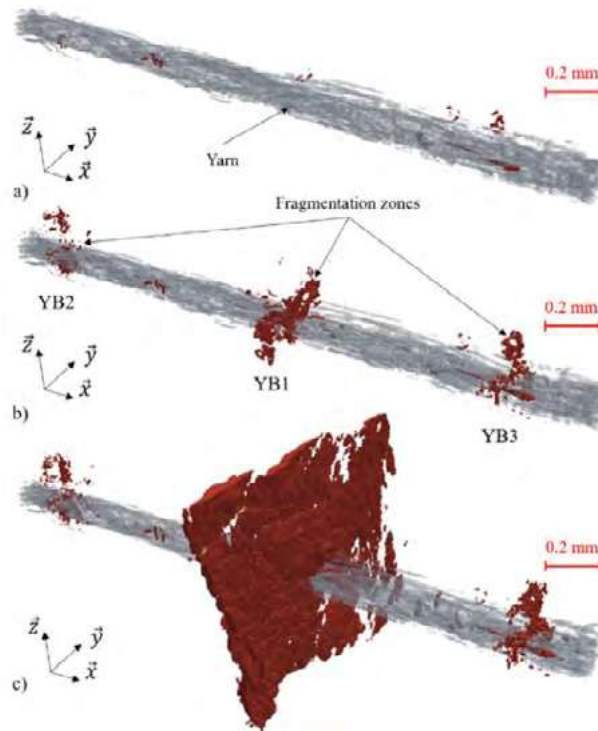


Figure 4. 3D segmentation of micro-CT scans of the single hemp yarn composite after a) step 0 (initial state), b) step 5 and c) step 6 of the step-by-step fragmentation test.

DVC calculations were then performed at each step of the fragmentation test of the single hemp yarn composite. Correlation calculations were realised between the micro-CT scans registered before loading (initial state) and the scans corresponding to step i . At each step i , the micro-CT scan was performed after unloaded the sample. Therefore, the measured strains are the residual ones, after unloading. Figure 5 presents the residual axial strain fields calculated by DVC in all the directions at the fourth, fifth and sixth steps, using the same color scale. Strain fields are shown in three YZ slices, located at

each yarn break and in two XY and XZ planes crossing the yarn. It allows to better understand how the development of damage takes place during the fragmentation test, with an axisymmetric shape of the matrix cracks around each yarn break and a $\pm 45^\circ$ strain redistribution in the matrix.

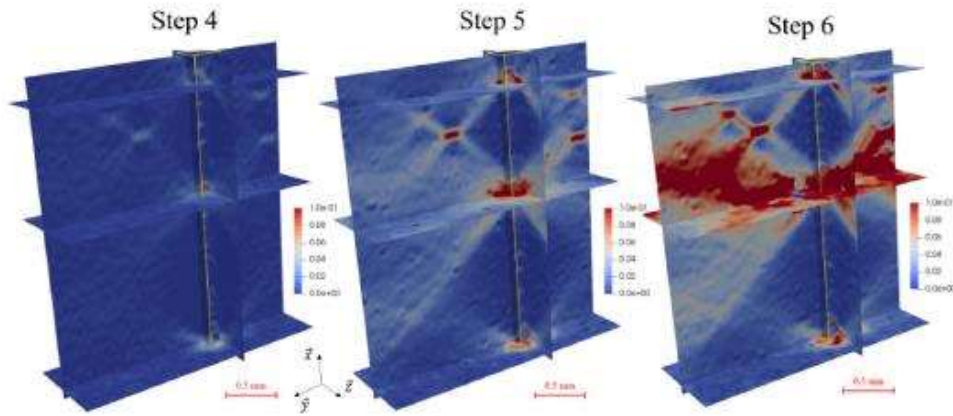


Figure 5. Volumetric residual axial strain calculated by DVC from micro-CT scans at step 4, step 5 and step 6 of the fragmentation test on the single hemp yarn composite.

4. Conclusion

At different scales, this study shows the importance to analyse the yarn/matrix interface behaviour. Micro-CT image acquisition is a powerful technique to observe internal volumetric material architecture and damage. It enabled also to evaluate the influence of hydrothermal ageing on the development of damage inside different composite materials. An in-depth analysis of the yarn/matrix interface, by using fragmentation tests, shows that the interface can be improved by plasma treatment of hemp yarn or by careful selection of the yarn/matrix pair. Micro-CT combined with DVC calculations revealed the volumetric strains at different steps of the fragmentation test. The analysis of the 3D strains showed how the strains were redistributed along $\pm 45^\circ$ directions in the matrix and around the yarn breaks. This study demonstrates the feasibility of DVC based on micro-CT for 3D strain measurements and the wealth of data collected. It paves the way for a comprehensive volumetric analysis of strain and damage mechanisms in composite materials.

Acknowledgments

Computations were performed on the supercomputer facilities of the *Mesocentre de calcul SPIN* Poitou-Charentes. This work was partially funded by the French Government program “Investissement d’Avenir” (EQUIPEX GAP, reference ANR-11-EQPX-0018) and by the CPER FEDER project of Région Nouvelle-Aquitaine. It pertains to the French Government program “Investissement d’Avenir” (EUR INTREE, reference ANR 18-EURE-0010).

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