4D-XCT and Digital Volume Correlation of Flax Fibre Composites Under Compressive Load After Relaxation

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Abstract
In-situ testing capabilities of lab-scale industrial computed tomography are rapidly improving. In the present study, the compressive deformation behaviour of a flax fibre-reinforced epoxy composite is studied in an *in-situ* compression test with stepwise loading, during 3D imaging with X-ray Computed Tomography (XCT). The 3D volumes are processed with Digital Volume Correlation (DVC) to identify the deformation of each loading step. The natural variability of the flax texture served as an adequate speckle pattern for DVC. The precision and accuracy of the DVC were investigated prior to the analysis of the deformation volumes by digitally deforming the reference undeformed volume with a known strain of 2 %. The calculated global strain was in good agreement with the digitally applied strain. Subsequently, DVC was applied to the acquired reference and deformed volumes and the analysis was focused on the 3D strain field evolution between the consecutive loading steps. Strain localization regions were observed in the axial strain ε_{zz} and the in-plane strain ε_{xx}. In the subsequent loading steps, the onset of compressive fibre-kinking is observed as a rotating fibre inside the magnified strain region. The 3D strain field evolution allows for a better understanding of the deformation and failure mechanisms at the meso/micro-scale in natural fibre reinforced composites under compressive loads.

Keywords: Bio-composites, 4D-XCT, Digital volume correlation, *In-situ* testing

1 Introduction

High-performance fibre reinforced composites are widely used in advanced applications due to the inherent advantages of combining different material properties, usually resulting in stiff and strong materials with low density. Despite their excellent performance, the traditional reinforcing fibres, mainly carbon and glass fibres, are highly energy-intensive to manufacture and with limited end-of-life recycling options. As an alternative, the use of natural fibres as the reinforcing element of composites has gained high interest in recent years due to their more sustainable and recyclable profile, resulting from their natural origin [1–3]. Among the family of natural fibres, flax fibres have attracted the attention of both the academic and industrial fields, primarily due to their stiffness similar to glass fibres and at the same time approximately a 40 % lower density [4,5]. This results in higher specific stiffness, which coupled with the environmental benefits can outperform traditional materials in engineering/structural applications.

In many applications, axial compression is the limiting designing factor for fibrous composites due to their weak compressive strength, compared to the tensile strength, caused by geometrical instabilities of the fibre geometry. Those geometrical instabilities lead to fibre kinking and hence the introduction of premature failure. Flax is no exception and its generally poor compressive performance has been observed with a ratio of compressive to tensile strength of 30 – 60 % [6–8]. In addition, the significant stiffness mismatch between the axial stiffness of the flax fibres and the polymer matrix further complicates the assessment of the micro-scale strain development in these natural fibre composites. A detailed understanding of the mechanical behaviour is key in designing performant natural fibre composite products.

The continuous improvements to industrial XCT in recent years have led to a rapid increase in the popularity of *in-situ* stepwise and dynamic XCT measurements: 4D-XCT [9]. Many processes in structural materials are time-dependent and can be studied properly only when time is factored in as the 4th dimension. A thorough understanding of the deformation mechanisms of heterogeneous materials, such as fibre reinforced composites, requires *in-situ* techniques that allow tracking the evolution of the (micro-)structure as a function of time. One potential approach is to measure the volumetric deformation by expanding the well-established Digital Image Correlation (DIC), for surface deformation capturing, to the three-dimensional (3D) domain in DVC

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[10,11]. The DVC method shares its working principles with DIC for the measurement of the displacement field. A 3D random speckle pattern with high contrast is thus a necessity within the examined material.

The speckle pattern can be achieved by embedding microparticles during specimen manufacturing [12] or by the natural microstructure of the material itself, as for example in bone [10]. Lee et al. [13] investigated the deformation behaviour occurring inside a carbon fibre laminate under tensile load by means of *in-situ* synchrotron XCT imaging. It was the first time that the microstructure of the laminate itself provided the random speckles required to perform DVC. They concluded that although the correlation steps were satisfactory (correlation coefficient > 0.7), a few erroneous correlations were present. These are caused by the repetition of the microstructural pattern, in their highly organized material. Mehdikhani et al. [11] expanded the utilization of DVC in carbon fibre composites into the area of damage analysis by observing the local strain magnification in the DVC strain fields, corresponding to matrix cracks in transverse plies, interface delamination and ply splits.

In this study, we aim to obtain a better understanding of the currently poorly understood compressive deformation behaviour of flax fibre-epoxy composites. Here it is, to our knowledge for the first time, investigated if DVC analysis can be applied to natural fibre composites and used to capture strain localizations, study the onset of deformation, fibre rotation and the growth into a kink-band. DVC is applied to the XCT volumes of an *in-situ* stepwise compressive loading experiment. The natural texture in these composites, originating from the inherent variability of the natural fibres and the attenuation contrast between fibres, matrix and voids, serves as the speckle pattern.

2 Materials and methods

2.1 Materials

For the manufacturing of the composites, a unidirectional flax fibre fabric (FLAXTAPE©) with an areal density of 200 g/m^2, supplied by Lineo NV (Belgium), was used as the reinforcing material. The fabric layers consist of long, hackled and aligned fibres which were pre-dried in an oven at 60 °C prior to the manufacturing of the composites. Apart from drying, no further treatments were used on the flax fabric. For the matrix material, the epoxy system Epikote 828 LVEL together with a 1,2-diaminocyclohexane (Dytek DCH-99) hardener from Hexion was used with a 100/15.2 mixing ratio following the manufacturer's datasheet. After mixing, the epoxy/hardener system was degassed under vacuum for 20 min to remove any potential air bubbles produced during the mixing.

2.2 Composite production

The unidirectional composite plate was manufactured using vacuum-assisted resin infusion with a target volume fraction of 40% and a thickness of 2.4 mm which was controlled using steel spacers. The impregnation of the flax fabric with epoxy was performed at 45 °C, followed by curing at 70 °C for 1 h and post-curing at 150 ºC for 1h. After slowly cooling down to room temperature, a rim of approximately 2 cm was removed from the plate to avoid edge effects.

Two plates were manufactured, and in one of both, air bubbles were introduced to investigate the effect of void content in the correlation quality. Specimens of 32×10×2.4 mm^3 were subsequently cut using a diamond saw, followed by dry-polishing of the edges. This smoothening helps to avoid potential critical defects that are created during the cutting. The dimensions were selected based on previous macro-scale experimental work [8]. The authors list the required high thickness is needed for reducing the bending ratio below ±10 %, as indicated in the ASTM D3410 [14] of shear loading unsupported gage compression test of composites. The higher thickness strengthens the specimen's stability against bending, thus the specimens were subjected primarily to compressive loading and the contribution of bending was minimal and below the required ratio in agreement with the testing standard.

The unsupported gage length was set to 10 mm while the rest of the 12 mm from each side of the specimen was fixed between the supporting clamps (Figure 1) to constrain the lateral movement of the sample. Then, the produced specimens were left for at least one month at standard conditions of 20 °C / 50 % RH, and the weight was monitored continuously until it reached a stable value. The latter step is critical due to the plasticizing effect of moisture on the highly hydrophilic flax fibres which affects the stiffness of the composite.

2.3 *In-situ* compressive 4D-XCT after relaxation

The specimens were fixed in the clamps of an in-house modified CT5000 DEBEN loading stage with a 5 kN load cell. The modifications to the DEBEN rig include (1) adaptable support tube length, allowing to work at a smaller source-to-object distance for faster scanning or higher resolution, and (2) the incorporation of modular spacers in the design - providing flexibility for sample length, from 5 mm to 35 mm in compression mode, and 10 mm to 50 mm in tensile mode.

The DEBEN stage was mounted in a TESCAN UniTOM XL scanner, equipped with a 230 kV/300 W microfocus X-ray tube, with a tungsten reflection target, and a detector of 2856 × 2856 pixels, with a 150 µm pixel pitch (Figure 1). Six stepwise displacement-controlled loading steps of 0.1 - 0.2 mm were made, at a motor speed of 0.1 mm/min, followed by a 10-minute pause, to allow for sample relaxation after the interruption of the loading, and avoid movement during the subsequent XCT scan

(Figure 2b). The scans were taken at 60 kV and 15 W, with a voxel size of 6 µm. In total, 2878 radiographic projections were acquired over a 360° angle, each with an exposure time of 900 ms and frame averaging set to 3, resulting in an acquisition time of 139 minutes. Cross-sectional imaging reconstruction was thereafter performed by applying a filter back-projection algorithm in the TESCAN reconstruction software Panthera.

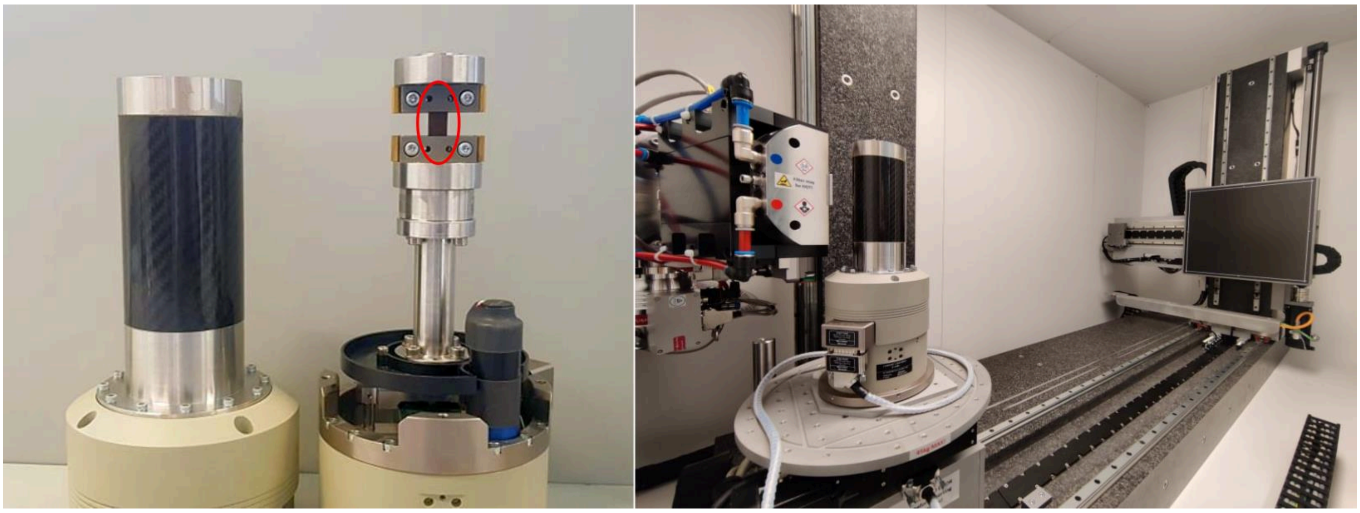

Figure 1: Sample fixed (encircled) in the Deben clamping system (left) and installed inside the TESCAN UniTom XL scanner during *in-situ* testing (right).

2.4 Post-processing & digital volume correlation workflow in Avizo

Image analysis and DVC were performed in Avizo v2020.1 (ThermoFisher). Prior to the correlation of the volumes, noise filtering was applied, using a 3D median filter with the neighborhood connectivity set to 18 and 2 iterations. The median filter reduces the photon noise in the scanned volume. Next, a 3D rigid body registration was performed in Avizo to register all acquired volumes into the same coordinate system, with the metric set to normalized mutual information. The Avizo DVC approach follows a two-step procedure. First, a subset-based (local) approach is used to capture large displacements on a coarse regular grid resulting in a time-efficient correlation, but generally with low precision. The main purpose of the local DVC step is to provide a good initial guess for the second DVC step, which is a robust Finite-Element-based (FE-global) DVC approach on a fine mesh, resulting in a high accuracy displacement field. FE-global DVC requires higher computational power and is characterized by a longer processing time.

The DVC in this research is performed using a cubic subset of 80 px³ for the local step, while the FE-global DVC is performed using tetrahedral elements of 60 px³. The subset size of the reference volume was chosen based on the calculated microstructural range in the autocorrelation versus distance plot in Avizo. The local DVC is applied using a correlation-metric of 0.7. After completion, the resulting displacement field serves as input for the FE-global DVC, which follows an iterative process to calculate the displacement field until the displacement convergence criterion of 0.001 px is reached. For each loading step, the displacement was analyzed with respect to the reference volume. Furthermore, the method allows the feed of the displacement field from a preceding step to the following to improve the precision of the algorithm.

2.5 Digital deformation

A preliminary analysis was conducted to investigate whether the quality of the speckle pattern (in this case the natural texture of the fibres) was sufficient for a high precision correlation as well as to assess the DVC parameters. A 2 % “virtual” compressive strain was applied to the reference scan by applying a uniaxial re-sizing of the volume, after which DVC was performed on the digitally deformed volume and the reference scan. Since the applied digital strain is known, it is possible to estimate the accuracy of the calculated global strain. Additionally, the quality of the displacement field and the resulting strain map can be assessed. They are expected to be nearly uniform, in accordance with the applied digital deformation.

3 Results and discussion

3.1 Sample characterization

The XCT representation of the complex microstructure of the flax fibre composite is shown in Figure 2a. The reference (undeformed) XCT volume was used to analyze the phase composition and the overall microstructure of the material. Due to the strong phase contrast in the images, greyscale-based thresholding was sufficient to segment the phases in the composite material, i.e., the matrix, fibers and voids. Additionally, the complex natural structure of the flax fibres can be observed, as well as the tubular inherent porosity in the center of the fibre, called lumen. The natural intrinsic variability of the fibres provides a more random speckle pattern compared to synthetic fibres, which is beneficial for the DVC precision.

Quantitative image analysis of the segmented phases learned that the produced natural fibre composite had a void and fibre volume fraction of 0.1 % and 40 %, respectively. High fibre volume fraction and low porosity are critical for DVC analyses, because voids represent areas with no speckling for correlation, while a higher fibre content provides a denser speckle pattern. The applied displacement for each loading step and the corresponding relaxation after each interruption are shown in the force-displacement diagram (Figure 2b).

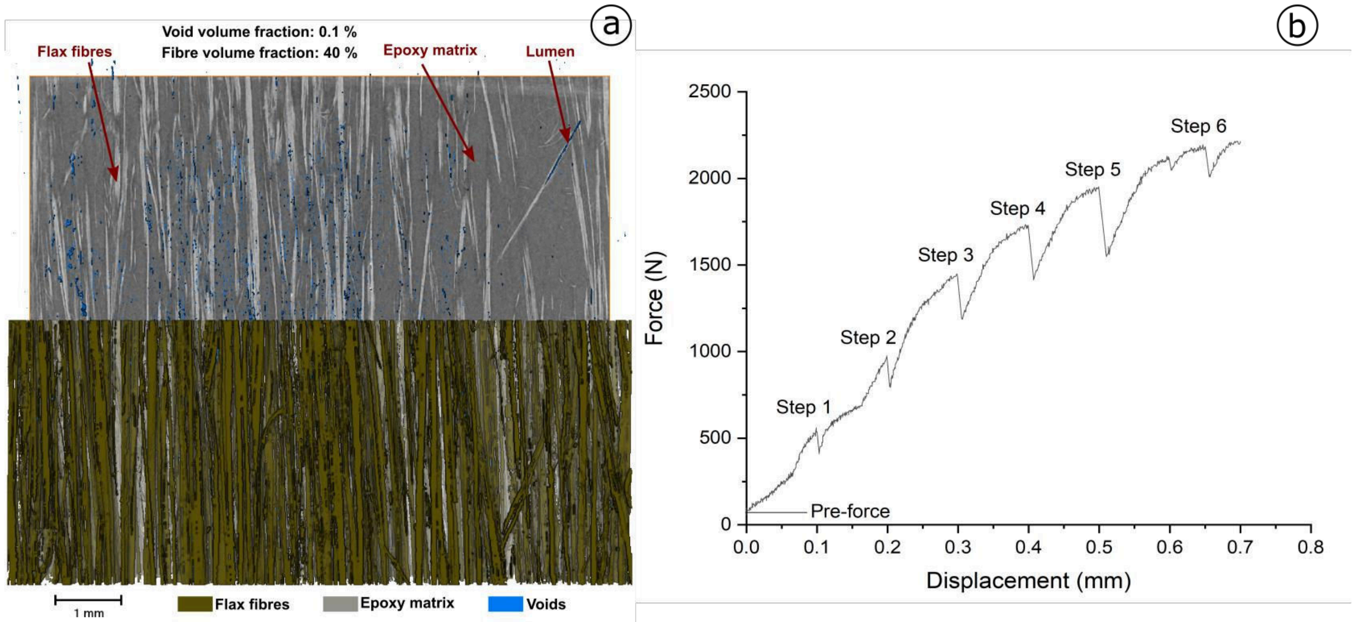

Figure 2: a) The segmented phases of the flax-epoxy material (bottom half of the image) and a 2D cross-section and visualization of the voids (top half of the image), b) The force-displacement curve of the stepwise *in-situ* loading and relaxation.

3.2 Digital deformation analysis

The preliminary analysis of the digitally deformed volumes (Figure 3a and 3b) shows a largely uniform strain field and a good agreement between the virtually applied axial strain (-2 %) and the calculated axial strain in the FE-global DVC (-1.969 % ± 0.027 %). Furthermore, the plot of the mean, maximum and minimum strains versus the Z-coordinate coupled with the representation of the strain map shows a distribution deviating from the uniform profile of the applied strain depending on the respective location in the volume. The internal regions show good agreement with the mean value while the borders of the analyzed volume are subject to larger strain variations. At the borders, the maximum and the minimum strain values deviate from the mean value significantly. The lower correlation quality for the borders can be attributed to the lower number of neighbours available for elements close to the border of the mesh [11].

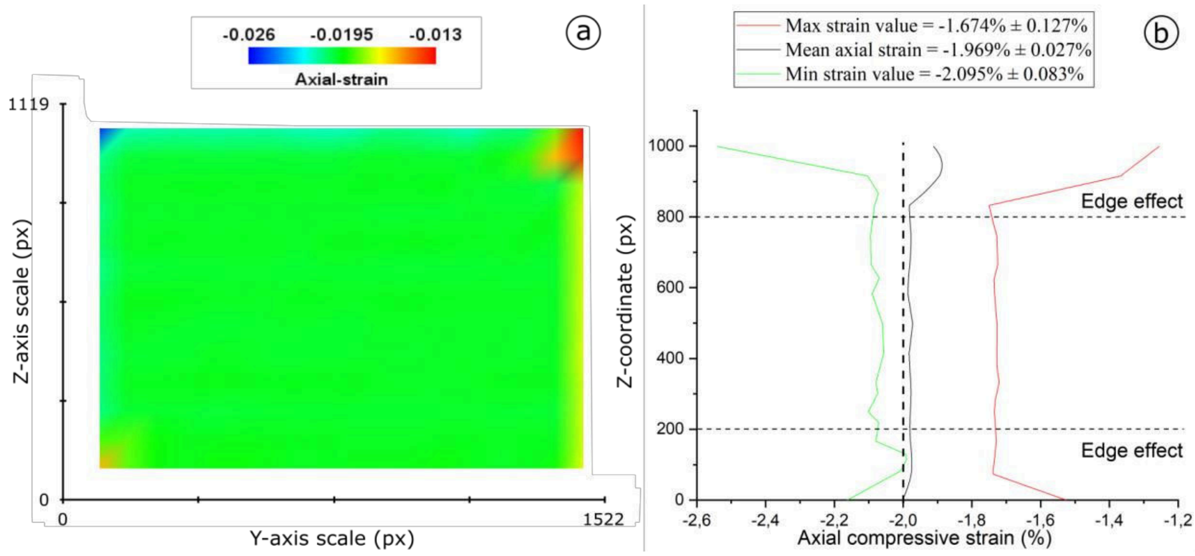

Figure 3: a) Strain map of the axial strain (ε_{zz}) between the reference and digitally deformed volume and b) plot of mean, max and min ε_{zz} value against the Z-coordinate. Weaker correlation zones are observed near the borders of the mesh.

Furthermore, this edge correlation quality is further negatively affected in the real volumes because of the speckling pattern moving in and out of the border elements of the mesh when displacement is applied. Therefore, prior to the interpretation of the correlated volumes, three layers of surface elements were removed from the top and bottom edge of the XCT volumes, where this effect is primarily observed in response to the compressive load.

The effect of the chosen mesh element size on the average axial strain and standard deviation for the FE-global DVC of the digitally deformed volume is shown in Figure 4. A smaller element results in a finer strain map resolution, but as mentioned above, comes at a computational cost and exponential increase in the processing time. In this case, the 60-px element mesh is identified as the minimum size with a reasonable standard deviation. Larger mesh sizes yield similar average axial strain values. Hence, depending on the desired analysis details, increasing the mesh size can be beneficial in terms of processing time. Overall, the digital deformation and DVC form an ideal case study that excludes imaging noise or local strain magnifications. It is an efficient method for the assessment of the DVC input parameters and can provide insight into the precision of the correlation.

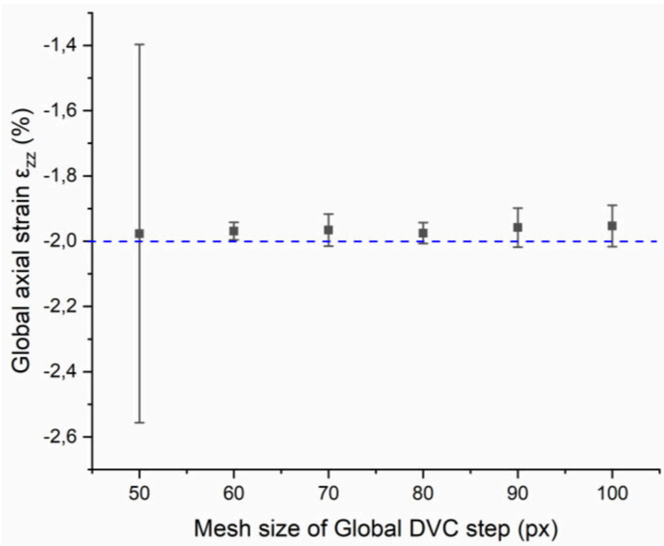

Figure 4: Effect of the DVC mesh size on the average global strain and the standard deviation.

3.3 *In-situ* compression DVC analysis

The influence of the void volume fraction on the DVC correlation was evaluated using two samples with considerably different void contents, 0.1 % and 10 %, respectively. The resulting average correlation metric of > 90% in the former is indicative of a good correlation, while the latter showed a lower average correlation metric (~80 %). The resulting out-of-plane 3D strain fields, mapped on the segmented fibres, are presented in Figure 5, for displacement steps of 0.1 mm, 0.3 mm and 0.5 mm, showing the strain distribution due to the inhomogeneous complex structure of the composite. In the 0.1 mm displacement step, a strain distribution can already be observed in the early loading stage. Enlarged strain localization and larger strain ranges are observed in the subsequent 0.3 mm and 0.5 mm displacement steps.

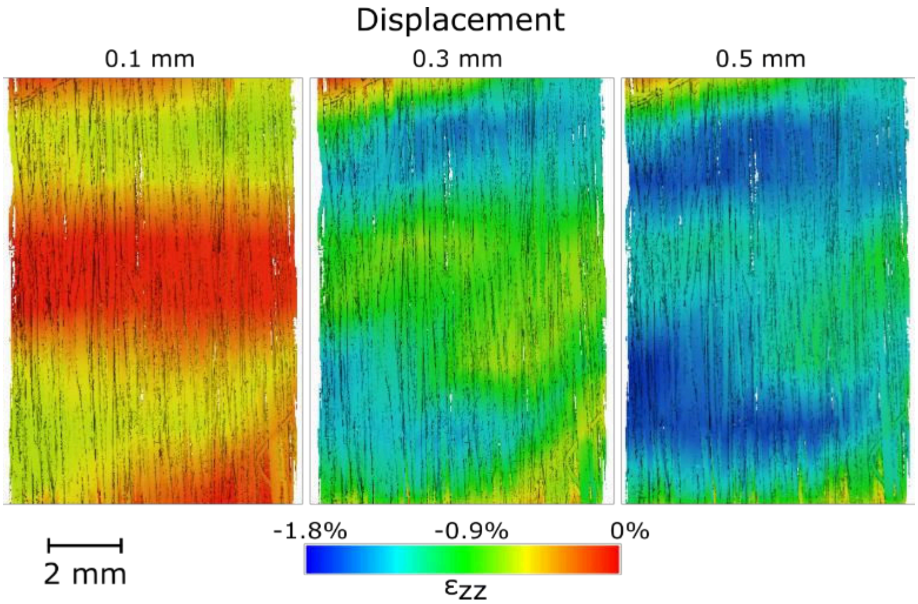

Figure 5: Strain field of ε_{zz}, mapped on the segmented fibres for the 0.1 mm, 0.3 mm and 0.5 mm displacement steps.

This behaviour in compression is likely related to the significantly less organized structure of natural fibres compared to highly organized and aligned synthetic fibres. The less aligned fibres can result in a locally degraded mechanical response, due to the reduced contribution of the reinforcing fibre to applied external stresses [15]. Strain localization areas during compression are of particular importance because they are indicative of compliant fibres during loading and likely locations for the initiation of damage. The dominant damage formation mechanism in uniaxial compression of these composites involves the rotation of those compliant fibres, initiating a kink band, where the fibres inside this kink band are rotated with respect to the fibres on the outside of the kink band. The strain localization is studied in more detail before (1950 N) and after (2200 N) damage formation (Figure 6). The displacement vector field in the 0.5 mm displacement step (Figure 6a) confirms that the compressive behaviour is captured in the DVC analysis. The transversal out-of-plane strain $\varepsilon_{\chi\chi}$ (Figure 6b) yields insignificant and uniform transversal strain. On the strain localization area that took most of the deformation (ε_{zz} = -0.18%), however, there is a significant local strain build-up, which possibly relates to the tendency of the natural fibres to locally rotate due to the applied compressive load and the non-linear shear behaviour.

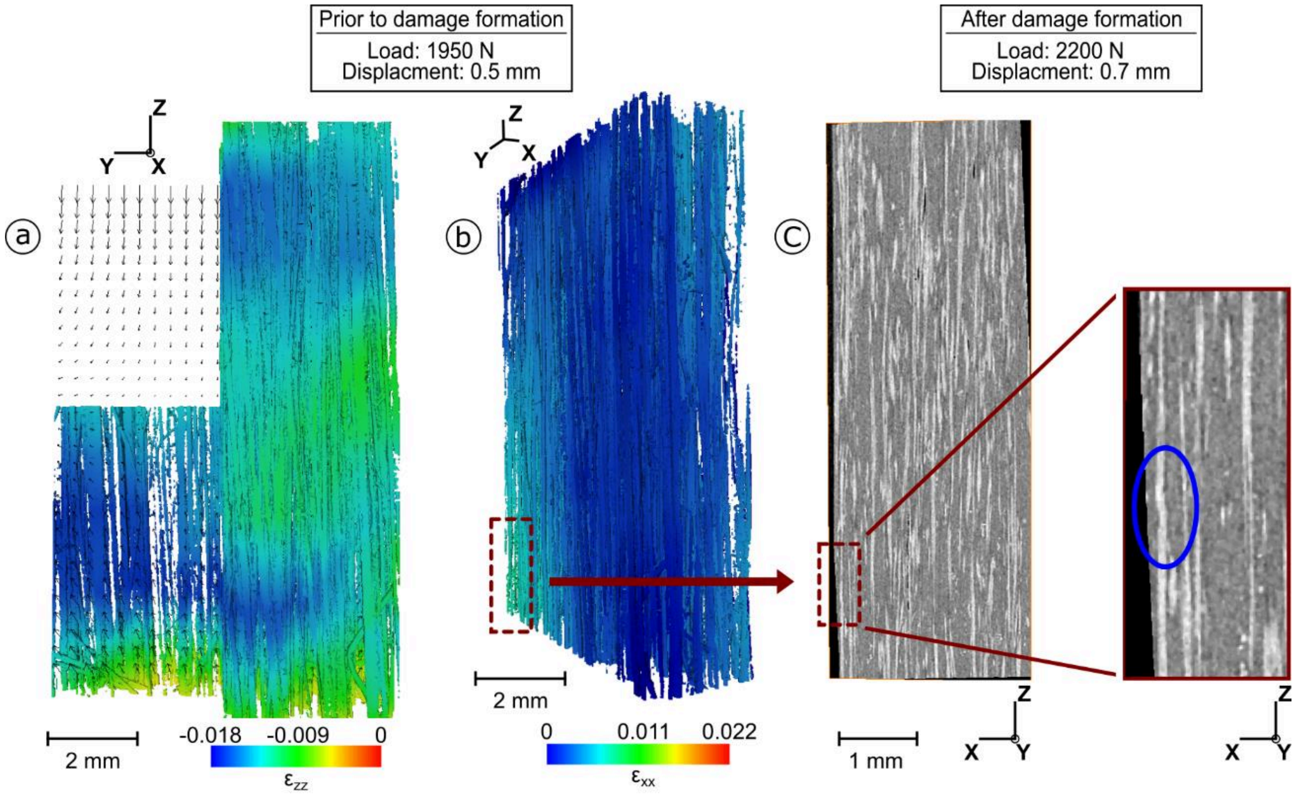

Figure 6: (a) Strain map ε_{zz} partially cropped to show the displacement vector field in the loading direction prior to damage initiation, (b) Transverse strain ε_{xx} prior to damage initiation and (c) fibre damage initiation in a later loading stage.

The location of the first fibre kinking, observed after 0.7 mm displacement (Figure 6c), is predicted by the strain localization region in the earlier loading steps, where no kink was present yet.Additionally, the initiation of fibre kinking is observed in the fibres closer to the sample surface. Fibre kinking relates to geometrical instabilities, and since the fibres close to the surface are less supported than the inner fibres, they are more prone to kinking under compressive stresses. The information on the progressive deformation of the composite and the onset and growth of fibre kinking is crucial for the modelling of the compression behaviour of flax fibre composites. The 4D-XCT voxel data can serve as input for, and help to finetune existing models, where a misalignment factor is introduced and often continuous fibre rotation is assumed from the starting point of the loading [16,17].

4 Conclusion

The digital deformation precision analysis and the high average correlation metric in the *in-situ* compression DVC analysis illustrate that the natural texture in the studied natural fibre composite and the attenuation contrast between the material phases are adequate for volume correlation purposes. This microstructural variability is even higher than for synthetic fibre composites. DVC was able to capture the strain evolution throughout the analyzed volume and show the importance of strain localization leading to the initiation of a fibre rotation in the subsequent loading steps. This is particularly beneficial for modelling realistic structures and predict the kink band initiation and growth in flax fibre composites under compressive loads.

High-resolution industrial 4D-XCT opens many new research pathways, since many processes studied today are time-dependent and can only be assessed with time factored in as the fourth dimension. Lab-scale XCT scanners provide more accessibility to perform both *in-situ* stepwise testing, where relaxation occurs, and *in-situ* dynamic loading experiments compared to synchrotron facilities. Industrial 4D-XCT helps to understand and predict the composite material behaviour in response to external stresses. The 4D-XCT experiments followed by DVC analysis have a strong potential to investigate progressive enlargement of strain localization inside the volume of the material leading to onset fibre rotation and the progression into the formation of a kink-band. In future research, the focus is to investigate the reason behind the strain localization, primarily by performing a detailed analysis on the local microstructure and the fibre alignment in those regions. Further straining of the sample will allow to study the growth of the kink-band up until the point of a fully formed kink. The technique has strong potential in tailoring the materials' design and the development of new natural fibre composite materials towards specific applications.

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