Effective use of transient vibration damping results for non-destructive measurements of fibre-matrix adhesion of fibre-reinforced flax and carbon composites

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Abstract

Fibre-matrix adhesion affects fibre-reinforced composites’ mechanical properties, a process which can be improved by applying an appropriate sizing on the fibre. Transverse bending tests and Scanning Electron Microscopy (SEM) can help quantify this effect. This paper investigates if modal damping measurements are a reliable alternative for quantifying fibre-matrix adhesion. When a composite sample is vibrating, part of the dissipated energy is due to the internal friction inside the material. More internal friction and slipping at the fibre-matrix interface is expected with a weaker fibre-matrix bond, hence increasing the amount of dissipated energy which in turn is proportional with the modal damping value. This paper researches two different cases to validate this hypothesis. In the first case, we will use two composite samples of flax fibre, one with and one without sizing. In the second case, we will compare flax and carbon fibre laminates. If the only variable is fibre sizing, a better adhesion is related to significantly lower damping and higher resonance frequencies. If composite laminates with different fibre and matrix type are compared, a lower adhesion is not necessarily related to increased damping and lower resonance frequencies. However, when combining the damping result with SEM microscopy, it is possible to assess the relative contribution to the internal energy dissipation of the fibre, the matrix and the fibre-matrix interface individually.

Keywords: material damping; SEM; composites; fibre-matrix adhesion; transverse bending

1. Introduction

In fibre-reinforced composites, mechanical properties do not only depend on the properties of the separate constituents and their respective fraction of the composite material, but also on how both the fibre and the matrix material interact with each other. This measure for how the matrix binds itself to the fibres is known as interfacial adhesion. Generally, fibre surface
treatments have been shown to improve interface adhesion compared to their neat-fibre surface. Different measurement techniques exist to evaluate the fibre-matrix interface. These mechanical tests can be subdivided into two main categories, (i) single-fibre tests and (ii) tests on composite specimens [1]. Within the latter category, transverse bending tests and Scanning Electron Microscopy (SEM) are often used to measure fibre-matrix adhesion quality. In the transverse bending test, it is mainly fibre-matrix adhesion and composite laminate’s fibre volume fraction of that determine mechanical stiffness and strength [2-4]. This is independent of fibres’ mechanical properties. With the transverse bending test, maximum strain is located at the outer layers of the specimen so that only a small volume is loaded with critical strain. As such, fracture probability due to voids and other defects in the material is minimized. Because of this, the transverse bending test is preferred to a transverse tensile test because the latter has a uniform strain distribution [2]. SEM microscopy is used for qualitative interpretation of the interface’s quality. With a weak interfacial bond, fibres and resin are separated easily from each other, resulting in a fibre surface which is almost-resin free. In case of a strong fibre-matrix bond, however, the matrix will be attached to the fibre. Strong adhesion is characterized by a fracture across the fibre or the resin, whereas a weak adhesion is characterized by a fracture at the fibre-matrix interface [5]. Measuring the material damping is a method to characterize the effect of modifications to the internal structure of the material [6-12]. As such, fibre-matrix adhesion also affects the specimen’s dynamic properties. Friction and slip at the fibre-matrix interface increase in case of a weak interface bond, leading to higher internal energy dissipation and, consequently, improved composite damping behaviour. In case of a strong fibre-matrix bond, microscopic friction is minimal and results in a lower damping capacity of the material. As an added bonus, modal damping tests are non-destructive, which is advantageous if the sample has to be used for other experiments.
The purpose of this paper is to examine whether modal damping measurements from transient vibration excitation are suited for detecting adhesion and fibre type effects in fibre-reinforced composites. This will be verified with results from a transversal bending and SEM microscopy test. Two different cases were evaluated. In the first case, two composite laminates were produced with the same manufacturing process. One sample was made with coated flax fibres for improved fibre-matrix adhesion whereas the other has neat, uncoated fibres. We will discuss if damping results can replace the two other methods for assessing interface quality. In the second case, composite laminates with flax and carbon fibre were investigated. These samples were made with pre-preg (pre-impregnated) fibre-matrix laminae. The damping results reflect the properties of the laminate itself, which is not necessarily related to the fibre-matrix adhesion only. We will therefore examine if the findings on damping measurements from the first case are still valid if two different composite materials are compared with each other. By combining three independent measuring techniques, we gain access to additional information that can help determine the contribution of the fibre, the matrix and fibre-matrix adhesion to the composite damping.

This research is limited to investigating the fibre-matrix interfacial properties of coupon plate specimens, with rectangular or square geometry. This is required prior to drawing conclusions if the modal damping technique could also be applied to actual geometries and sizes. If so, this technique could be used as an NDT method to detect the fibre-matrix interfacial properties. However, fibre-matrix adhesion is not the solely damping parameter, but also other influences such as boundary conditions, exact excitation method, local variations in part geometry and thickness, play an equally important role, and are very difficult to filter out when using it as an NDT method for real structures.
2. Sample preparation

Fibre-matrix adhesion was characterized by different composite materials and production techniques, the manufactured composite laminates had a unidirectional (UD) fibre direction.

Sample preparation with the RTM (vacuum-assisted resin transfer moulding) production process allows the production of composite laminates with neat and treated flax fibres, the flax fibre material was provided by Lineo and has a density of 150 g/m². The treated flax fibre is, in contrast to the neat flax fibre, coated for improved fibre-matrix adhesion and to prevent water absorption. The RTM process also makes it possible to produce neat epoxy samples, which will later be used for comparison with the composite laminates. For this production technique, the combination of epoxy resin RIMR 135 and hardener RIMH 137 is ideal.

Composite laminates with flax fibre and carbon fibre were compared with each other based on samples made with the autoclave process. The flax fibre pre-preg material was also available from Lineo, with the same fibre density and coating as with the RTM manufacturing process. The epoxy resin was Araldite LY5150, Young’s modulus is 3.52 GPa and the tensile strength is 68-78 MPa, corresponding with a maximum strain of 2-3 %, according to the manufacturer’s specifications. The carbon fibre pre-preg material consists of M55J carbon fibres and the M18 epoxy resin. The epoxy has a Young’s modulus of 3.50 GPa, a tensile strength of 81.1 MPa and failure strain of 3.7 %, as specified by the manufacturer. The carbon fibre surface was treated with sizing 50B from Toray to improve the fibre-matrix adhesion.

Table 1 lists the mechanical properties of the different laminate types. These properties were assessed from tests according to the standards ASTM D3039 and ASTM D3518. In case of plane stress analysis, four engineering constants are sufficient to describe the composite material’s mechanical behaviour: Young’s modulus in the 1- and 2-direction $E_{11}$ and $E_{22}$ (cf. Fig. 2), the Poisson’s Ratio $\nu_{12}$, and the shear modulus $G_{12}$. 
3. Experimental test procedure

To verify if modal damping measurements are suitable for determining the influence of the fibre-matrix adhesion and the fibre type, it was necessary to have a reliable reference that can be related directly to the fibre-matrix adhesion. Therefore, our analysis procedure first evaluates the results from the transverse bending test and the SEM method. These results will then be used for comparison with the modal damping results.

The sample thickness is 2 mm for all tests. The width and the height depends on the selected test method, which will be discussed in the following sections. The samples for the different test methods were cut out from the same base material plate to eliminate scatter on the results due to small variances in the production process.

3.1. Transverse bending test

With the transverse three-point bending test, the composite laminate was positioned as shown in Fig. 1. Direction 1 is parallel with the fibres, direction 2 is perpendicular to the fibres in the laminate’s plane, and direction 3 is perpendicular to the fibres and the plane of the laminate. The sample used two supports at distance $l_S$ and the force was pointed in the 3-direction in the middle of the specimen. The plane defined by the 2-3 directions is the translaminar view of the specimen, the plane defined by the 1-3 direction is known as the interlaminar surface. The tests were executed according to the ASTM D7920-03 standard. The sample thickness $t$ is 2 mm, the width $w$ is 12.7 mm and the length $l$ is 100 mm. The support length $l_S$ is 32 mm, the overlap at each support should be at least 10% of the support length with a minimum of 6.4 mm and. Prior to testing, it was necessary to polish the translaminar view for a better quality of the microscopic analysis (section 4.1.2 and 4.2.2). The finest grain size applied was #4000. It should be noted that quantitative interpretation with bending tests is difficult. The friction at the
supports significantly contributes to the result and the slope at the contact points is possibly too large for correct use of linear elastic theory of bending [13]. As such, the Young’s modulus calculation is omitted from the analysis and bending stress $\sigma$ is calculated following Eq. (1) [14].

$$\sigma = \frac{M}{I/(t/2)} = \frac{F \cdot l_s/4}{w \cdot t^2/6} = \frac{3 F \cdot l_s}{2 w \cdot t^2}$$ (1)

3.2. SEM microscopy: translaminar and interlaminar view

Next, the broken samples from the transverse bending test were used for qualitative interpretation of fibre-matrix adhesion with SEM technology. The laminate’s crack propagation was visualized with the interlaminar and the translaminar view. For SEM microscopy, the test sample was cut to 12.7x12.7 mm$^2$ and a gold coating was sputtered for better conductivity. The translaminar view was visualized with a backscatter electron (BSE) detector for differentiating on chemical composition and the interlaminar view was visualized with the secondary electron (SE) detector for optimum detection of surface topography. The acceleration voltage for collecting the SE and BSE images was 5 kV and 25 kV, respectively.

3.3. Damping

Material damping measures vibration energy dissipation that relates to the material’s internal structure. This energy dissipation is caused by the sample’s internal strain variation. To quantify damping from lightly damped materials (e.g. metals and fibre-reinforced composites), it is important to select the right measuring method and to avoid external damping sources as much as possible. In this paper, we have chosen to measure the sample’s damping capacity when it is vibrating at resonance frequency. The result of this is known as the modal damping ratio and was determined by using a transient time-domain method, as described in [15]. The test setup
is schematically presented in Fig. 2.a. The sample was suspended at the mode shape’s nodal points to avoid contact damping, excitation was carried out with a loudspeaker and responses were measured with a laser Doppler vibrometer (LDV) at the point of maximum amplitude. The LDV’s laser beam was pointed at the location of maximum amplitude for optimum sensitivity of the velocity measurement. Once the sample was in steady-state vibration (at resonance frequency) and has reached maximum amplitude, excitation stops and the steady state vibration amplitude attenuates to zero amplitude. The response resonance decay rate was calculated with the logarithmic decrement $\delta$. The modal damping ratio is then $\zeta = \delta/2\pi$ for lightly damped materials [15] and was determined for the first and second resonance frequency. The mode shapes with the nodal points and the points of maximum amplitude for a square and rectangular UD composite laminate are shown in Fig. 3.

When comparing the modal damping ratio of different materials, we need to consider the internal strain distribution of the sample when vibrating at resonance. The strain distribution depends on the mode shape and the material’s mechanical properties, which leads to another dissipation mechanism. Therefore, comparing modal damping is only valid if the strain distribution is equal for samples with different materials. This strain distribution is calculated as modal strain energy. It is a dimensionless value that relates the individual strain energy components to the total strain energy. The number of individual strain energy components is in analogous to the number of mechanical properties that characterize the material’s elastic behaviour. In case of isotropic materials, this is the Young’s Modulus $E$ and Poisson’s ratio $\nu$. With composite laminates in plane stress loading, four independent engineering constants appear: the Young’s moduli $E_{11}$ and $E_{22}$, the Poisson’s ratio $\nu_{12}$ and the shear modulus $G_{12}$. The corresponding strain energy component is $P_{11}$, $P_{22}$, $P_{12}$ and $P_{66}$ and the modal strain energy ratio is defined as $\frac{P_{ij}}{\sum P_{ij}}$. The modal strain energy is defined as given in Eq. (2) [16].
For each individual strain energy component, the corresponding dissipation component also varies with the frequency. In literature, modal damping tests on composite laminates have shown that damping increases with increasing frequency. Depending on the modal strain energy ratio, this effect varies from negligible to significant [6, 7, 10, 11].

4. Results and discussion

Our results are based on three methods: (i) transversal bending, (ii) fracture analysis with SEM microscopy and (iii) transient vibration damping. In the paragraphs 4.1 and 4.2, the composite laminates with different fibre sizings and two composite materials with different fibre types are discussed respectively.

4.1. Effect of fibre sizing

4.1.1. Transversal bending test

With the test procedure from section 3.1, the bending stress at failure $\sigma_{22}^t$ is calculated. Table 2 shows the mean value and the standard deviation (between brackets) from five independent samples. Fibre sizing positively affects the mechanical properties as bending stress increases considerably when fibre sizing is used. This effect was previously reported by Tran et al. [17], where composite samples with Alkali treated coir and flax fibres showed respectively a 29.5 % and 15.1 % increase for the transverse bending stress, compared with the untreated fibres. This
improvement is also confirmed by Callens et al. [18] where a significantly higher strength is measured for steel fibre/epoxy composites with added fibre surface coating.

4.1.2. Fibre-matrix fracture type with SEM microscopy

Fig. 4.a and Fig. 4.b show the translaminar crack propagation view of the flax fibre laminate without fibre sizing. The translaminar view in the flax fibre laminate with fibre sizing is depicted in Fig. 4.c and Fig. 4.d. The pictures in Fig. 4 on the right hand side are an enlarged view of the white square box of the figure on the left hand side. Generally, flax fibre bundles can be recognized as circular patterns consisting of elementary fibres with an approximate diameter between 10 µm and 25 µm, which are also comprised of micro fibrils. In between two fibre bundles, the cracks propagate through the matrix material. Inside the fibre bundle, the fracture pattern depends on the fibre-matrix adhesion. For the laminate without fibre sizing, the cracks mainly propagate at the fibre-matrix interface without splitting a single fibre. With the fibre sizing, fibres are split in half, with an exceptional crack along the interface. With technical fibres, fibre cracking is practically impossible, whereas with natural fibres, the multi-fibre structure of the elementary fibre possibly initiates the splitting. When fibre and matrix cracking dominate the interface splitting, this indicates a strong interfacial adhesion between the fibre and the matrix. From the translaminar microscopy, it is obvious to qualitatively differentiate between the laminates with and without fibre sizing.

Comparing the interlaminar fracture surface from the laminates without and with fibre sizing shows a clear difference. Without fibre sizing (Fig. 5.a and Fig. 5.b), the surface is smooth. Some microfibers and remaining matrix material are visible but the fibre is generally not unravelled. This indicates that the matrix material could easily separate from the fibre without damaging the fibre. With fibre sizing (Fig. 5.c and Fig. 5.d), the elementary fibre surface is
rough and micro-fibrils are visible. Even if the translaminar view shows fracture propagation at the interface, the interlaminar view confirms good fibre-matrix bonding.

4.1.3. Transient vibration damping

The modal damping ratio values in Table 4 and Table 6 give the mean value, with standard deviation between brackets. The mean value and the standard deviation of the modal damping ratio are calculated from five independent measurements.

The composite UD laminate with and without flax fibre sizing is compared with the neat epoxy material. All test samples have dimensions of 265x265x2 mm³. In the square-sized specimen, the first mode shape of the isotropic and orthotropic material (Fig. 3.a) is dominated by shear energy $P_{66}$ (Table 3). The second mode shape of the UD laminate (Fig. 3.b) is dominated by strain energy in the 2-direction $P_{22}$ and for the isotropic material the strain energy is equally divided in the 1- and 2-direction, with a significant contribution of the Poisson effect, as seen in Table 3. Comparing the damping value between the neat epoxy and the flax fibre laminate for the first mode shape is valid because the strain energy distribution for the first mode shape is approximately equal for both samples. The second mode shape's modal strain energy ratio and its corresponding energy dissipation is different and therefore cannot be used for comparison.

We can observe that the first and second resonance frequency of the composite laminate with coated flax fibre increases by 4 % and 6.9 % compared to the sample with neat flax fibre (Table 4). For the same mass and geometry of the plate and identical fibre-volume fraction, this suggests that a stronger fibre-matrix bond positively affects the material stiffness. From Table 4, we can infer that the neat flax fibre has a significantly higher damping than the coated flax fibre. Since the resonant frequency only slightly varies for both samples, this effect is
expected to be of minor importance when modal damping is compared. The modal damping increases by 68% for the first resonance mode and 36% for the second resonance mode. The larger energy dissipation is probably due to increased slipping and friction at the fibre-matrix interface. When shear strain is the dominant strain component, the effect of fibre sizing on the modal damping ratio is more pronounced than with other strain components. Furthermore, when damping between the neat epoxy sample and the composite laminates is compared, damping is significantly higher for the composite sample. This is due to the friction and slip at the fibre-matrix interface and the fibre itself, but their relative contribution is not known from this analysis.

4.2. Effect of composite material

4.2.1. Transversal bending test

The failure stress $\sigma_{22}^f$ of the flax fibre laminate is more than doubled compared with the carbon fibre laminate (Table 4). It was also observed that the carbon fibre laminate exposed brittle fracture behaviour whereas the flax-fibre laminate exposed initial fracture prior to reaching the maximum bending force. Where the flax fibre laminates in section 4.1.1 have the same fibre-volume fraction, this is not valid anymore for the carbon versus the flax fibre laminate from autoclave manufacturing. De Kok and Meijer [19] found that with increasing fibre-volume fraction the transverse tensile and bending strength increases. The same result was also reported by Gu et al. [20], where they showed that the transverse tensile strength increases with fibre-volume fraction. However, the conclusion that is drawn by these authors is only valid for composites with the same fibre and matrix type. To formulate an answer to why the carbon composite has a lower transverse bending strength, the analysis by the SEM microscopy to study the fibre-matrix adhesion will be required.
4.2.2. Fibre-matrix fracture type with SEM microscopy

Analogous to the flax fibre laminate with fibre sizing from the RTM manufacturing, the flax fibre laminate from the autoclave manufacturing shows the same fracture mechanism. The translaminar view (Fig. 6.a) mainly shows fibre splitting and the interlaminar view (Fig. 6.b) depicts the rough fibre surface with remaining matrix material and micro fibrils attached to the fibres. This contrasts sharply with the carbon fibre laminate, where the translaminar view (Fig. 7.a) shows only fibre-matrix interface fractures and matrix splitting cannot be observed. This is confirmed by the interlaminar view in Fig. 7.b, which shows smooth fibres with very small amounts of remaining matrix material at the fibre surface. It can be concluded that the flax fibre laminate has a more significant fibre-matrix adhesion than the carbon fibre laminate. This also formulates an answer to the observation in the previous section, it is obvious that the weak interfacial strength for the carbon fibre is responsible for the much lower transverse bending strength (Table 2). This effect leads the possible influence of the higher fibre-volume fraction of the carbon fibre laminate, as formulated in the previous section [19, 20]. The higher fibre-volume fraction for the carbon fibre composite is expected when comparing Fig. 8.a with Fig. 8.b, each depicting the translaminar view on a 200 µm scale of the flax and carbon fibre laminate, respectively.

4.2.3. Transient vibration damping

Both samples have dimensions of 445x137x2 mm³. The strain energy ratio for the first and the second mode shape is approximately equal for both laminates (Table 5); the first mode shape is dominated by shear and the second mode shape is dominated by strain in the 1-direction. As such, it is possible to compare both samples’ modal damping (Table 6). The modal damping in both the first and second mode is significantly higher for the flax fibre laminate than for the carbon fibre laminate. This effect is even enhanced by the finding that the resonant frequencies
of the carbon fibre laminate are significantly higher than those of the flax fibre laminate. The higher resonance frequency is expected since the engineering constants $E_{11}$, $E_{22}$ and $G_{12}$ are higher for the carbon than for the flax fibre laminate (Table 1). Extrapolating the damping values of the flax fibre laminate to the same, higher, resonant frequency of the carbon fibre laminate will even result in a higher damping value [6, 7, 10, 11]. These results are in contrast to conclusions from the analysis of the fibre sizing in section 4.1. Here, weak fibre-matrix adhesion, as presented by the SEM analysis and transverse bending test, leads to a lower damping capacity and a higher resonance frequency. This demonstrates that the fibre and the matrix individually have a larger contribution to the internal energy dissipation than the fibre-matrix adhesion.

5. Conclusion

We have investigated if modal damping measurements can substitute the transverse bending and SEM microscopy techniques for measuring the fibre-matrix adhesion in fibre-reinforced composites. Two cases were considered to evaluate this property: (i) the effect of composite laminates with different fibre sizing and (ii) the effect of composite laminates with different matrix and fibre type.

In a first analysis, the effect of fibre sizing was investigated for flax fibre laminates with and without fibre sizing. SEM microscopy indicates that fibre sizing results in improved adhesion. Fibre splitting and a rough fibre surface with remaining matrix material and micro fibrils are observed. The transverse bending test shows that, with fibre sizing applied, the maximum bending stress increases significantly. From the damping tests, it follows that the resonance frequency increases and the damping capacity decreases when fibre sizing is applied.
In our second analysis, two laminates with different fibre and matrix type were investigated. SEM microscopy indicates that the flax fibre laminate has better fibre-matrix bonding than the carbon fibre laminate. This was verified by observing a higher bending stress for the flax fibre laminate than the carbon fibre laminate. The modal damping measurements reveal that the damping capacity of the carbon fibre laminate is significantly lower than the flax fibre laminate, and the laminate’s resonance frequency higher with carbon fibre than with flax fibre.

In general, when the influence of fibre sizing on the fibre-matrix adhesion is investigated, only the damping measurement is sufficient to draw conclusions. The transverse bending test gives no additional information and SEM microscopy only verifies what is already discernible at macroscopic level. Moreover, the transverse bending test is more prone to measuring errors for quantitative analysis and SEM microscopy is relatively time-consuming compared to damping measurements. When two different composite materials are compared, the modal damping value does not yield conclusive results about the fibre-matrix interfacial properties, this test should be combined with a transverse bending test or SEM microscopy. Nevertheless, with modal damping measurements, it is possible to measure which parameter is dominating the dissipation mechanism: the fibre, the matrix or the fibre-matrix adhesion. Here, we have observed that the effects of the fibre and the matrix individually are more important than that of the fibre-matrix adhesion.

Acknowledgements

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### Table 1 Mechanical properties of composite laminates from RTM and autoclave manufacturing for plane stress analysis

<table>
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<th>Engineering constant</th>
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<th>Autoclave manufacturing</th>
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<td>Flax fibre laminate*with fibre sizing</td>
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<td>$E_{11}$ [GPa]</td>
<td>21.2</td>
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<td>$\nu_{12}$ [/]</td>
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<td>$G_{12}$ [GPa]</td>
<td>2.33</td>
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### Table 2 Bending stress from transverse bending tests for different laminate types

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<td>Flax fibre UD laminate*with fibre sizing</td>
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<td>Bending stress [MPa]</td>
<td>129.9 (0.7)</td>
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### Table 3 Modal strain energy ratios for first and second mode shape of neat epoxy and flax fibre UD laminate

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<td>$P_{11}/P$ [%]</td>
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<td>$P_{22}/P$ [%]</td>
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<td>$P_{66}/P$ [%]</td>
<td><strong>94.38</strong></td>
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Table 4 Experimental modal damping values for neat epoxy and flax fibre UD laminates with and without fibre sizing

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<th>Flax fibre UD laminate with fibre sizing</th>
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<tr>
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<td>Frequency</td>
<td>Modal damping [%]</td>
<td>Frequency [Hz]</td>
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<td>Resonance mode 1</td>
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<td>Resonance mode 2</td>
<td>46.9</td>
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<td>62</td>
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Table 5 Modal strain energy ratios for first and second mode shape of flax- and carbon fibre UD laminate

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<td></td>
<td>Resonance mode 1</td>
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<td>$P_{11}/P$ [%]</td>
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<td>$P_{66}/P$ [%]</td>
<td>95.0</td>
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Table 6 Experimental modal damping values for flax and carbon fibre UD laminates

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<th>Carbon fibre UD laminate</th>
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<td>Frequency [Hz]</td>
<td>Modal damping [%]</td>
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<tr>
<td>Resonance mode 2</td>
<td>48</td>
<td>0.890 (0.005)</td>
</tr>
</tbody>
</table>