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Green Steel
Process-controlled optimization of the tensile strength of bamboo fibre composites for structural applications

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Process-controlled optimization of the tensile strength of bamboo fibre composites for structural applications

At the Advanced Fibre Composite Laboratory in Singapore, a new mechanical processing for raw bamboo has been developed, which leads to a fibrous material with physical features that are mainly defined by the bamboo species. This material is used as a natural fibre source for the production of a high-tensile fibre reinforced composite material aiming for the construction industry. Thereby, controlling the parameters of the underlying hot press fabrication process turned out to be crucial for a systematic tuning of the tensile capacities of the resulting composite materials.

In recent years, building industries around the globe compete to develop new lightweight materials with extreme tensile strength capacities. These materials find their applications in aerospace industries, in specialized building industries like mining and tunnel construction and lately also in car industry. All of those materials have one thing in common: they are composites. Usually, industrially produced synthetic inorganic fibres are mixed with an adhesive agent to form a symbiotic constellation, which results in a high-strength but lightweight material. However, there are also other common features: composite production is tedious and therefore extremely expensive and the raw materials such as glass or carbon fibres are hard to handle or require high amounts of energy for their production. These are drawbacks that limit the access of such materials to developed nations, which unfortunately represent only a small percentage of the world's population. Hence, action has to be taken in order to find ways, how to make reinforced composite materials more available in terms of their cost and processing techniques as well as more sustainable.

One very promising approach is to use natural fibres as reinforcement instead of the synthetic inorganic fibre materials. Some of the advantages in comparison to synthetic fibres are their abundance, renewability,
Fig. 01 Newly developed bamboo composite material
biodegradability, and their lower costs. Moreover, natural fibres from plants such as pineapple, jute, sisal and bamboo have already been proven to be effectively utilized in load-bearing composite materials. We would like to contribute to the development and improvement of natural fibre based composite materials by presenting an entirely mechanical processing technique for raw bamboo into an easy to handle fibrous material, which is then fabricated into a high-tensile strength composite using the hot press fabrication method. Moreover, we would like to show that by varying and controlling the process conditions (pressure, temperature, time) of the fabrication method we are able to tune the mechanical properties of our “natural” composite material into regimes that might in the future compete with tensile capacities of synthetic composites at significantly lower costs.

The bamboo species employed for all tests within this study was a 5-year-old Phyllostachys edulis, also known as Moso bamboo, which was harvested in the Guangning area of China. At this age the performance of Moso bamboo is known to be at its highest level. What makes this bamboo species so strong is its very fine grain due to its small vascular bundles. Another characteristic is the high concentration of these vascular bundles at the outside surface of the culm walls.

The average density of Moso bamboo with a moisture content of around 10% is 520 kg/m$^3$. The mature bamboo culms were cut and mechanically split into slats, which are then processed into strips with a thickness of several mm. At this thickness the fibres stay intact. Fig. 02 – top shows macroscopic photographs of the raw-bamboo strips. The raw material represents an average fibre collection from upper, middle and lower sections of the bamboo culm in nearly equal ratios. Moreover, it depicts the properties of an assembly of all possible layers from a bamboo culm.

A two-component binder system, whose components are sourced from biological sources, was employed as a matrix. To assess the reinforcing quality of the fibres, the unidirectional alignment of the strips into a layered structure was assured before placing them into the mould of a hot press. Subsequently, the test specimens were subjected to different pressures (between 15 and 25 MPa) and temperatures (between 80 °C and 140 °C) at various press/hold times to afford highly compressed composite samples with a volume fraction of more than 70% (Fig. 02 – bottom). Finally, the composites were cured.

The unidirectional composite specimens were prepared according to the ASTM D3039-08 Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials into dog-bone shapes. This standard has been chosen for this study due to the lack of a specific standard for testing the mechanical properties of bamboo composite materials (Fig. 03). In order to be able to control the limiting values of the mechanical properties of the composite it is important to know the tensile strength of the reinforcing material.
Fig. 02  Top: macroscopic photograph of the oven-dried bamboo strips used for the fabrication of the composites. Bottom: corresponding composite specimen

Fig. 03  Photograph of the dog-bone-shaped test specimens for the tensile tests after tensile failure
Therefore, the tensile strength of the raw oven-dried bamboo strips has been evaluated, as well. Tensile tests were carried out using a Shimadzu AG-IC 100 kN machine in accordance with the ASTM D3039-08 standard at a strain rate of 1 mm/min. The tensile strength was calculated from the ultimate load and the cross-sectional area of the test specimen. From each composite sample at least five specimens have been tested and results exceeding a 10% standard deviation range, which has been statistically set as confidence interval, have been discarded.

The surfaces and cross sectional areas of the raw bamboo strips as well as the tensile fracture surfaces of the composite samples were analysed with a Carl Zeiss SteREO Discovery.V12 optical stereomicroscope and then coated with silver for a field emission scanning electron microscopy (FESEM) analysis with a JEOL, JSM-6700F.

Mechanical properties of raw bamboo strips

The average longitudinal tensile strength of Moso bamboo single fibres ranges from 1.43 to 1.69 GPa, which is significantly higher than nearly all published data for timber fibres. However, due to the natural brittleness of the lignin network that the fibres are embedded into the tensile capacity of fibre bundles drops to about 300–400 MPa. Since the processing of the raw bamboo is purely mechanical and does not involve any chemical, the fibre bundles of the employed strips are further encompassed by significant amounts of holocellulose and water-soluble polysaccharides. This further decreases the tensile capacities as compared with single fibres. Our tests have shown that tensile failure of the raw oven-dried strips occurs already between 120 and 160 MPa without nodes. Here, it is worth mentioning that earlier studies have demonstrated that heat and pressure treatment as present when applying the hot press method may to a certain extent vary the natural strength of the fibrous material by affecting the fibre matrix. Thus, considering the high pressures and elevated temperatures applied throughout this study, the tensile features of the raw strips do not necessarily constitute the limiting factor for the ultimate tensile strength of the resulting composite material. Furthermore, it should be noted that all the composite samples were prepared from bamboo strips with nodes.

Tensile strengths of bamboo reinforced composites

Table 01 summarizes the tensile strengths of the composites for different pressures and temperatures of the hot press fabrication process. Table 01 reveals the highest tensile strengths when applying pressures of 20 MPa and 25 MPa at temperatures of 100 °C and 80 °C, respectively. Notably, with the 25 MPa/80 °C setting the results were less consistent. At a pressure of 25 MPa and temperatures above 100 °C increasing carbonization of the bamboo strips and the decomposition of the test samples has been observed. These samples have been tested for their tensile strength as well but the very broad mean variation of the results did not allow for any meaningful conclusions. At 140 °C the bamboo fibers started to carbonize already at a pressure of 15 MPa. We have attempted to test the specimen produced with a pressure of 20 MPa but the results are obviously very inconsistent.
Hence, in the following we concentrate on the analysis of the test specimens subjected to pressures between 15 and 25 MPa and temperatures between 80 °C and 120 °C. A correlation between the applied pressure and temperature is found when comparing the tensile strengths obtained for the given pressures and temperatures. In general, at 15 MPa and 20 MPa, the tensile strengths follow the same trend, i.e. a maximum tensile strength is found at 100 °C. At 25 MPa the maximum tensile capacity can already be obtained at 80 °C while at 100 °C it drops to a comparable value that is obtained at the 20 MPa/120 °C setting. Assuming that at 25 MPa the maximum tensile capacity is achieved at 80 °C and that at 60 °C the strength would decrease again, implies that increasing the pressure by 5 MPa lowers the processing temperature for obtaining the maximum tensile strength by 20 °C.

Such correlation suggests that it is possible to tune the mechanical properties of the composite by either varying the temperature or the pressure within a specific range. Thereby, the maximum tensile strength holds not only for one single pressure/temperature combination but may be also obtained when lowering the temperature and simultaneously increasing the pressure or vice versa. A maximum is found at 100 °C for both 15 and 20 MPa and at 80 °C for 25 MPa insinuating a shift of the 25 MPa graph to lower temperatures.

<table>
<thead>
<tr>
<th>P / T</th>
<th>15 MPa</th>
<th>20 MPa</th>
<th>25 MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>80 °C</td>
<td>135 MPa SD: 9%</td>
<td>147 MPa SD: 7%</td>
<td>182 MPa SD: 8%</td>
</tr>
<tr>
<td>100 °C</td>
<td>150 MPa SD: 7%</td>
<td>181 MPa SD: 3%</td>
<td>163 MPa SD: 4%</td>
</tr>
<tr>
<td>120 °C</td>
<td>132 MPa SD: 5%</td>
<td>158 MPa SD: 6%</td>
<td>/</td>
</tr>
<tr>
<td>140 °C</td>
<td>/</td>
<td>92 MPa SD: 54%</td>
<td>/</td>
</tr>
</tbody>
</table>

Table 01  Tensile strengths of the Moso composites with the corresponding standard deviations (SD) in per cent as obtained for different pressures and temperatures with a press/hold time of 15/15 min. At 140 °C and at 120 °C with a pressure of 25 MPa the samples started to carbonize and it was difficult to obtain meaningful results

<table>
<thead>
<tr>
<th>Time (press/hold)</th>
<th>5/5 min</th>
<th>15/15 min</th>
<th>30/30 min</th>
<th>60/60 min</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>167 MPa SD: 10%</td>
<td>181 MPa SD: 3%</td>
<td>175 MPa SD: 3%</td>
<td>180 MPa SD: 4%</td>
</tr>
</tbody>
</table>

Table 02  Tensile strengths of the Moso composites with the corresponding standard deviations (SD) in per cent at different press/hold times as obtained at a pressure of 20 MPa and a temperature of 100 °C
Notably, varying both temperature and pressure at a time significantly alters the mechanical properties of the composite. This is related to variations of the viscosity of the binder at higher temperatures and therefore variations of the fibre wetting and/or different binder-network structures formed under different temperature/pressure conditions. Finally, increasing the temperature above 100 °C favours the carbonization of the fibres – especially at elevated pressures – which consequently leads to a modification of the intrinsic fibre matrix and a loss of tensile strength.

Conclusively, the processing temperature most likely controls the binder chemistry, which is well in line with the fact that the matrix requires a certain activation temperature to form its solid network and variations of the curing time and temperature have a strong impact on the binder properties. The pressure, on the other hand, affects the interactions between the bamboo fibres and the matrix as well as controls the infiltration of the binder into the fibres. Further insights were provided by microscopic studies (see below).

The variation of the press/hold time between 5/5 min and 60/60 min (Table 02) appears to affect the tensile strength of the composites only in a time range below 30 min. After the hot press processing the samples were all equally cured for 12 h in order to achieve full strength. Therefore, it is safe to assume that the press/hold time has – if any – only a minor influence on the curing of the binder but rather affects the wetting of the bamboo fibres and the penetration of the binder inside the microstructure of the bamboo.

Evidently, the interactions formed between the bamboo fibres and the binder in the 5/5-min press/hold composites are weaker as for 15/15 min samples and the tensile stress is less favourably transmitted within the fibres and the binder matrix, which is reflected by the lower tensile strength. After 30 min processing time, however, no significant improvement of the tensile strength is achieved by elongating this time.

Conclusively, a certain time is required to activate the formation of the binder matrix, which is associated with a certain activation energy that has to be delivered to the system. This energy amount is required for the binder to fully react with the hardener and develop all potential interactions with the fibres. Hence, it is possible to tune the press/hold time by varying the temperature. In other words, increasing the temperature increases the energy amount per time delivered to the system and therefore accelerates the matrix formation of the binder.

Finally, it should be pointed out that in the composite the intrinsic tensile strength of the bamboo strips is not only preserved but also enhanced. In other words, the binder forms a network, which is able to spatially distribute the locally applied tensile stress over the entire composite structure and, thus, enhance its load bearing capabilities. The reason for this is the homogeneous distribution of the binder within the strips as well as within the bamboo microstructure.
Microstructure of bamboo reinforced composites

The optical micrographs of the traverse sections in Fig. 05 reveal the unidirectional and parallel alignment of the fibre bundles within the strips (left) and the lack of any voids or fibre contact in the longitudinal direction (right) that might contribute to a loss in composite strength.

The optical micrographs of the fractured surfaces of the composites produced at different pressures are displayed in Fig. 05. A clear difference is observed when considering the coverage and the consistency of the binder on the fibres. The samples produced at 100 °C and 20 MPa (Fig. 05b) disclose that the surface of the composite with the highest tensile strength is rather smooth and homogeneously covered with the binder. Hence, at this pressure and temperature the network forms a very thin layer, which efficiently interacts with the surface of the bamboo and most likely penetrates the fibres well due to its viscosity. Due to the high pressure the outer surface of the strips seems to be completely infiltrated with the binder. Reducing the pressure to 15 MPa (Fig. 05a) results in the formation of rather large binder beads on the bamboo surface, which in turn reduces the wetting and homogeneous coverage of the fibres.
In comparison with 20 MPa a pressure of 15 MPa appears too low for the binder to infiltrate the fibres. This is well in line with the reduced tensile properties of the composites shown in Fig. 05a. Eventually, increasing the pressure to 25 MPa (Fig. 05c) has apparently two effects: On one hand, the binder forms even larger crystal-like beads than those found at 15 MPa and, on the other, the infiltration seems to be inhibited by a propagated carbonization of the fibres. The latter is proposed by the significantly darker colour of the composite and the strips.

Similarly, at higher temperatures – 120 °C and 140 °C – the increasing carbonization of the fibres is responsible for the lower tensile strengths of the composite (Fig. 06). At 140 °C the fibres and the binder become extremely brittle, which is represented in Fig. 7a by the dark colour of the fibres and the localized, large binder beads. At 120 °C (Fig. 06b) both effects are present to a slightly lower extent. For a more detailed comprehension of the above-mentioned observations field emission scanning electron microscopic (FESEM) studies were performed on the composite specimens and the raw bamboo.

Fig. 07 shows the cross-sectional areas of the raw bamboo. The images show a rather fine grain structure with highly concentrated round and regular vascular bundles. On the other hand, the FESEM images of the cross-section of the fractured surface of the composite fabricated at 20 MPa and 100 °C reveal a significant infiltration of the binder into the microstructure and a distortion of the round shape of the vascular bundles (Fig. 08a and b). At higher magnification, it is possible to distinguish that some of the larger pores are completely filled and some partially with the binder (Fig. 08c and d). This hints to a rather deep penetration of the binder into the bamboo microstructure. The infiltration within the microstructure highly depends on the viscosity of the binder and therefore on the processing temperature and pressure. Hence, differences in tensile strength may safely be attributed to the degree of infiltration of the fibres by the binder at different processing conditions.
Similar assumptions hold for the FESEM analysis of the lateral fractured surfaces of the composite specimens. As seen in Fig. 09, the surfaces show a similar morphology regarding the coverage of the fibers by the binder as the cross sections.

Interestingly, the lateral fractured surfaces of the composites produced at 100 °C and 15 or 25 MPa exhibit a significantly rougher surface than those fabricated at 20 MPa, which again correlates well with the optical microscopic studies. Fig. 10 displays the FESEM images of the surfaces corresponding to 15, 20 and 25 MPa. The 20 MPa sample shows a very smooth surface with a homogeneous adhesion of the binder to the fibres. At the fracture position single fibres are seen that have been pulled out of the binder. This indicates nearly perfect bonding between both and confirms the infiltration of the binder into the vascular bundles (Fig. 11a). On the other hand at 15 and 25 MPa – apart from an inhomogeneous wetting – a debonding of the binder from the fibres is observed at higher magnification (Fig. 11b and c). The presence of beads and binder residues on the fibre surface implies a lower degree of infiltration as compared with the 100 °C/20 MPa specimens.

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**Fig. 07** FESEM micrographs of the fractured cross-sections of raw Moso bamboo at different magnifications displaying the vascular bundle microstructure

**Fig. 08** FESEM micrographs of the fractured cross sections of the composites produced at 100 °C with a pressure of 20 MPa at different magnifications
Conclusion

High-strength bamboo strip composite materials with a maximum tensile capacity of ~180 MPa were fabricated using the hot press method. Processing conditions such as temperature, pressure and pressing/holding time, have been varied and evaluated for their impact on macroscopic mechanical properties of the composites and microscopic interactions at the interface between the binder and fibre surface. We were able to demonstrate that it is possible to use an easy-to-handle naturally available raw material such as bamboo to produce rather high-strength composite materials. The achieved maximum tensile capacity of 180 MPa is still in the low range of high-performance composites but our findings helped to evaluate the process parameters, such as temperature, pressure and press/hold time and correlate them with the macroscopic mechanical properties of the composite, as well as with the microscopic interactions at the interface between the binder and the bamboo fibres.

In particular, we demonstrated that a purely mechanical processing technique for raw bamboo into strips provides a material in which the natural fibre alignment of bamboo is easily controlled throughout the production of the composite. Moreover, the elevated pressures and temperatures during fabrication turned out not only to preserve the tensile strength of the raw material but also slightly enhance its properties in the composite. Importantly, within a specific temperature/pressure regime a maximum tensile strength was obtained, which was tuneable by adjusting the pressure.
and temperature. Microscopic analysis provided insights into the wetting behaviour and infiltration of the binder into the fibres, which strictly depends on the fibre–binder interface. In conjunction with the degree of carbonization these factors were proven to determine the ultimate tensile capacity of the resulting composites.

Hence, understanding and subsequent optimization of the process parameters – especially at the microscopic level of the fibre–binder interfaces – is crucial for controlling and improving the mechanical properties to reach regimes that might in the future compete with tensile capacities of synthetic composites at significantly lower costs. However, the lack of uniformity of natural materials in comparison to synthetic products is certainly drawback for our composites as well and needs to be controlled. We are currently addressing this issue by optimizing the processing technique and carefully choosing the raw material. Moreover, we are investigating different methods of how to control the interfacial interactions between binder and bamboo by employing various pre-treatment schemes to the raw material.

References


Image Credits

Fig. 01-03: Carlina Teteris
Fig. 04-07, 10: AFCL, Singapore
Fig. 08,09: NTU School of Mechanical Aerospace Engineering

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Colophon

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