Energy-absorbing wood composite for improved damage tolerance inspired by mollusc shells

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Keywords: Bionics, Crossed-lamellar-structure, damage tolerance, wood composite

Abstract

The crossed lamellar structure (CLS) found in mollusc shells is an excellent example for nature’s ability to form complex hierarchical microstructures with a remarkable balance between strength and toughness. The CLS has become the subject of numerous studies focusing on the replication of the unique microstructure using synthetic composites. The present study proposes a wood composite replicating the CLS' middle layer microstructure and investigates the mechanical properties using three-point bending tests. The morphology of the failure mechanisms is recorded using digital microscopy and the experimental data are compared to those from ply- and solid woods. The results show a successful replication of the dominating failure mechanisms of crack deflection and crack bridging. While strength decreased significantly by ~60%, toughness increased remarkably by ~70% compared to plywood and was in the range of solid wood. The small data scattering from the wooden CLS samples compared to solid wood further hints on a stable failure mechanism and uniform energy-absorption. The results document that wood can be used to design an energy-absorbing composite based on the CLS-inspired ductile microstructure.

1. Introduction

Complex hierarchical microstructures found in natural materials like bones, mollusc shells or wood are an inspiration for manmade composite structures due to their excellent mechanical behaviour [1–4]. These natural microstructures exhibit different strategies at various hierarchical levels to increase damage tolerance and simultaneously an improved strength, thus increasing the chances of survival of the respective organisms [5].

In the case of wood, the stem-branch junction is a good example for a complex microstructure exhibiting a very high toughness before failure [6–8]. As described by Müller et al [6], the tree forms a so called 'sacrificial tissue' in the upper part of the branch which serves as a potential crack path in case critical stresses are exceeded. If cracks form, however, its path is deflected in a zig-zag way leading to high energy absorption, protecting the vital connection between stem and branch. Similar strategies can be found in the crossed lamellar structures (CLS) of mollusc shells [9]. Natural CLS are a composite of brittle calcium carbonate (calcite or aragonite) in a soft protein matrix [10]. According to Ji and Gao [11] the main contributing factors to its high toughness and strength are large aspect ratios and a staggered alignment of the inorganic crystallites. The fracture toughness is further enhanced due to the hierarchical microstructure, consisting of different macroscopic layers and three orders of lamellae [2] further depicted schematically in figure 1.

The macroscopic layers are arranged perpendicular to each other leading to a 0° (O), 90° (M) and 0° (I) build-up on a macroscopic scale. Within the middle layer the first-order lamellae are turned ±90° degree from one lamella to the next. The second-order lamellae are tilted 45° in regards to the principle x-axis leading to a ±45° build-up within the first-order lamella. The third-order lamellae in turn are arranged parallel within the second-order lamellae causing the same ±45° directions in relation to the first-order lamellae. In case yield
stresses are exceeded the highly twisted structure deflects the induced cracks multiple times and therefore increases the toughness without losing too much initial strength. This mechanisms of controlled crack propagation as well as crack branching were fundamentally described by Cook and Gordon [14]. They concluded that the mismatch of adhesive strength and general strength of the solid is crucial in order to increase overall toughness of usually brittle materials.

This display of nature’s capability to form highly complex structures achieving a remarkable balance between strength and toughness has become the focus of various research trying to imitate CLS in manmade composites [12, 15–22]. The common materials used to mimic the inorganic phase are ceramics [16, 18, 20] or synthetic polymers [12, 19, 22]. Few literature was found using natural materials to replicate CLS in a manmade composites and materials. Hou et al [15] used bamboo to manufacture a simplified model of the twisted lamellae found in the first-order of natural CLS. They found that the alignment angle of second-order lamellae represents the key factor when mimicking CLS and further concluded that crack deflection, bridging and branching as well as fiber pull-out are the main toughening mechanism in natural CLS. Based on an extensive literature research, no attempt has been made to utilise CLS as inspiration for a biomimetic composite structure based on wood.

The aim of the present study is to design, build and characterize a wood-based composite inspired by the crossed lamellar structure of mollusc shells. In a first approach, the middle layer (as seen in figure 1) of the macrostructure is reproduced using birch veneers and a polyurethane adhesive. The main hypothesis answered in this study were:

H1. The dominating failure mechanisms based on crack deflection found in natural CLS can be reproduced using wood.
H2. The utilization of the crossed lamellar structure leads to an increased work of fracture (WOF) compared to plywood and solid wood indicating an improved toughness.
H3. The introduction of staggered lamellas leads to a decrease in strength compared to plywood and solid wood.

2. Materials and methods

2.1. Structural design and material selection

The dimensions reported by Kamat et al [3] were used as baseline to derive the size ratios for the structural design of the wooden CLS samples. Wood itself can be considered as composite of fibrous cellulose embedded in amorphous hemicellulose and lignin [23]. The wood fibers within the veneer were therefore already considered as the third-order lamellae while the veneer strips mimic the second-order lamellae (figure 1). Lastly the glued veneer strips represent first-order lamellae. Additionally, as described by Hou et al [15] a certain mismatch in elasticity between the inorganic phase and organic matrix is desired to match the relations in natural CLS. Birch wood shows excellent mechanical properties paired with medium high density [24]. 1C-PUR adhesives usually have relatively long open times and allow for a long processability due to their ready-to-use state [25]. The mechanical properties of the adhesive were investigated by Nenning [26] by means of in situ and ex situ tests. The
1K-PUR adhesive was chosen because of its low stiffness, which gives a stiffness contrast to wood parallel to the fibre, corresponding to the stiffness contrast between the organic and inorganic substance in the mollusc shells. Therefore, 1 mm thick, grade A birch veneers (sourced from Koskisen Oy Thin Plywood Industry, Finland) where chosen to mimic the inorganic phase and 0.15 mm thick 1C-PUR (Collano Semparoc 60 sourced from Collano AG, Switzerland) glue lines take on the role of the organic matrix. The final dimensions of the CLS samples are further described in figure 2.

2.2. Fabrication

In order to guarantee reproducibility of the wooden CLS sample an 8-step production process was developed which is illustrated in the appendix (figure A1). The manufacturing process includes multiple steps of gluing, pressing and cutting. In a brief overview: (1–2) production of unidirectional laminated wood, (3) cutting of individual strips, (4–5) gluing of strips to first order sheets and grinding, (6) manufacturing of cross laminated boards, (7) Cutting of CLS specimens at a 45° angle and formatting to the final dimension. Prior to each bonding step the veneer surfaces were grinded (P100 grit) either by hand or in a wide-belt sanding machine. In each bonding step (steps: 1, 2, 4, 6 and 7) 220 g m⁻² of 1C-PUR adhesive (Collano Semparoc 60 sourced from Collano AG, Switzerland) was applied in order to guarantee a glue line of about 0.15 mm in the final sample. After each surface gluing (steps: 1, 2 and 7) a uniform pressure of 0.5 MPa was applied at room temperature in a veneer press (Langzauner LZT-160-SFB, Maschinenfabrik Langzauner GmbH, Austria) for 180 min. In order to guarantee a solid bond line between the edges of the sheets, the sheets where pressed at room temperature in a vacuum press (at a vacuum of 750 hPa) for 180 min after each edge gluing (steps: 4 and 6).

Reference solid wood samples (SWB) were produced using defect free straight grained birch wood. Reference plywood samples (PLY) were produced using grade A birch veneers (supplied by J.u.A. Frischeis GmbH, Austria) and the same 1C-PUR adhesive. Plywood was chosen as it is a common veneer based engineered wood product with a comparable structural design.

2.3. Experimental characterization

2.3.1. Mechanical properties

The bending strength (σ_b), the bending modulus (MOE) as well as the work of fracture (WOF) were determined in three point bending according to standard EN 408 [27] by using a universal testing machine (Z100, Zwick/Roell, Ulm, Germany) with a cell capacity of 100 kN (resolution of 0.06 N). Prior to testing, the samples were stored under standard climate conditions at 20°C ± 2°C and 65% ± 5% (RH) [28] until constant mass was reached. In order to prevent indentation of the samples during testing the samples were placed on trapezoidal bearings. This enabled a constant bearing surface during testing, especially during the high deflections of the CLS specimens. The samples were pre-loaded with 20 N before the test was started and then loaded at a constant displacement rate of 15 mm min⁻¹. Deflection was recorded using a mechanical extensometer (Makrosense, Zwick/Roell, Ulm, Germany). The test was stopped after a 95% load reduction of the peak force was reached. The strength as well as the modulus of elasticity (MOE) were calculated according to standard EN 408 [27]. The work of fracture (WOF) was determined as the work until a residual load of 90 N obtained from the load-displacement curves in relation to the initial cross-section (A) of the sample. Additionally, the energy per unit volume (ω) was calculated using the dimensions of the samples and the corresponding loads.
2.3.2. Microscopic inspection

In order to investigate the failure mechanisms, optical images from CLS fracture surfaces were taken with the digital microscope DSX 1000 (Olympus, Shinjuku, Tokyo, Japan) using bright field observation mode at a total magnification of 42 × (objective lens DSX10-XLOB3X). To capture in-focus images exceeding the microscope’s field of view, processing modes for vertical and horizontal image compilation using the software DSX10 (version 1.1.2.2, Olympus, Shinjuku, Tokyo, Japan) were applied. Via 3D acquisition mode the entire sample cross section (see figure 2) was scanned in vertical direction. Horizontal image stitching was performed with an overlap of ten percent on each side.

2.3.3. Statistics

Experimental data were processed using Excel 2016 (Microsoft, Redmond Washington, USA). This also included descriptive statistics (mean, minimum and maximum values, coefficient of variance) as well as the creation of the boxplots. Statistical tests (variances, Mann-Whitney U-test) were carried out using SPSS 24 (IBM SPSS Statistics version 24.0, IBM, New York, USA). The error probability for the statistical tests was set at a threshold of $p \leq 0.05$.

3. Results

3.1. Failure mechanisms

Figure 3 shows a representative CLS sample after 95% load reduction was reached. Additionally, the fracture pattern of the same sample is further depicted in figure 4. Crack initiation occurred on the tension side of the sample. The cracks propagated along the $\pm 45^\circ$ second order lamellae as seen in figures 3(A) and 4(C)–(D). The right-angled design of the first order lamellae caused the cracks to branch out in $\pm 90^\circ$ directions to each other (indicated by the white arrows in figures 3(B) and 4(A)–(B)). The propagation along the $\pm 45^\circ$ second order lamellae was halted at the transition zone between tension and compression (figure 3(A)). This crack deflection between every other first order lamellae confirms H1. The dominating failure mechanism found in natural CLS [2, 11, 15] can be reproduced using wood. Furthermore, fibre-bridging between first order lamellae (black

![Figure 3. Observed failure mechanisms I. A: Front view of a representative CLS sample after 95% load reduction and before de-loading. Black arrows indicate areas where crack-bridging occurred. B: Detail isometric view (tilt angle: $\sim 40^\circ$) of a representative CLS sample. Black arrows indicate the same areas of fibre-bridging as in A. White arrows indicate areas of crack initiation.](image-url)
arrows in figures 4(C)–(D)) as well as within second order lamellae (black arrows in figure 3) was observed for some of the CLS samples. In addition, plastic deformation due to compression of second order lamellae as well as the adhesive is indicated by grey arrows in figures 4(E)–(F).

3.2. Mechanical properties

The work of fracture of the CLS and the reference samples (figure 5(A)) as well as the corresponding variabilities (figure 5(B)) are depicted in figure 5. The WOF of 15 CLS samples ranged from 58.09 kJ m\(^{-2}\) up to 100.99 kJ m\(^{-2}\) with an average value of 77.11 kJ m\(^{-2}\) and a CoV of 16%. The 9 valid plywood samples exhibited values between 34.98 kJ m\(^{-2}\) and 53.58 kJ m\(^{-2}\) with an average of 46.07 kJ m\(^{-2}\) and a CoV of 13%. The 10 solid wood samples resulted a WOF between 28.63 kJ m\(^{-2}\) and 101.57 kJ m\(^{-2}\) with an average of 65.01 kJ m\(^{-2}\) and a CoV of 39%. Comparing the work of fracture partly confirms H2. While there is a significant improvement of the CLS work of fracture over plywood (exact Mann-Whitney U-test: \(U = 0.000, z = -4.025, p = 0.000\)), there is no significant improvement over solid wood (exact Mann Whitney U-test: \(U = 103.000, z = -0.870, p = 0.474\)).

The \(\omega\) of 15 CLS samples ranged from 184.42 kJ m\(^{-3}\) up to 320.61 kJ m\(^{-3}\) with an average value of 244.79 kJ m\(^{-3}\) and a COV of 16%. Results for 9 valid plywood samples ranged from 97.2 kJ m\(^{-3}\) up to 148.90 kJ m\(^{-3}\) yielding an average of 127.99 kJ m\(^{-3}\) and a COV of 13%. The \(\omega\) for 10 solid wood samples ranged from 95.46 kJ m\(^{-3}\) up to 338.63 kJ m\(^{-3}\) with an average of 216.72 kJ m\(^{-3}\) and a COV of 39%. The results for energy per unit volume supports the statement made for H2.

All CLS as well as reference ply- and solid wood samples were tested up to a force drop of 95% relative to the ultimate load and failed in the area of highest bending moment. Levels of maximum force were around 450 N \(\pm\) 50 N for CLS (figure 6(C)), around 900 N \(\pm\) 100 N for plywood (figure 6(B)) and around 1500 N \(\pm\) 400 N for solid wood (figure 6(A)), which correspond to the bending strength mentioned in the upcoming section. A representative load-displacement curve for CLS as well as for ply- and solid wood is depicted in figure 6(D).
The typical failure curve for CLS does not show brittle fracture behaviour when yield stresses were surpassed. From the point maximum force was reached to 95% load drop the CLS sample showed completely ductile fracture behaviour. The typical failure curve for ply- and solid wood deviated from the CLS behaviour. After yield stresses were exceeded and cracks were initiated, brittle failure in the tension zones leaded to an abrupt force drop for plywood as well as solid wood. Fracture was halted in layers of higher strength. This combination of crack propagation and crack arrest continued on steadily lower stress levels until 95% load reduction was reached. This behaviour in bending of solid wood goes in line with literature [29–31].

The determined bending strength of the CLS as well as the reference samples is depicted in figure 7(A). \( \sigma_{bl} \) of 15 valid CLS samples ranged from 37.94 MPa up to 42.00 MPa with an average of 40.36 MPa and a CoV of 3%. The plywood samples resulted values between 77.73 MPa and 94.17 MPa with an average of 88.18 MPa and a CoV of 6%. The bending strength of solid wood ranged from 89.77 MPa up to 141.63 MPa with an average of 119.53 MPa and a CoV of 13%. Comparing the bending strength confirms H3. There is a significant decrease of the strength of CLS compared to plywood (exact Mann Whitney U-test: \( U = 0.000, z = -4.025, p = 0.000 \)) as well as solid wood (exact Mann Whitney U-test: \( U = 0.000, z = -4.160, p = 0.000 \)). Additionally, the
determined MOE is depicted in figure 7(B). The MOE of the CLS samples ranged from 2.13 GPa up to 2.34 GPa with an average of 2.29 GPa and a CoV of 2%. Plywood exhibited values between 8.36 GPa and 9.44 GPa with an average of 8.98 GPa and a CoV of 3% and the MOE of solid wood ranged from 11.97 GPa up to 16.14 GPa with an average of 14.36 GPa and a CoV of 8%.

4. Discussion

As depicted in figures 3 and 4, respectively, the simulated CLS structure made out of wood showed the common known failure mechanisms of crack deflection and crack bridging which are associated with the middle layer (M, figure 1) of the mollusc shells [2, 11]. Cracks were initiated at multiple locations on the tension side of the sample leading to a multitude of processing zones. The initiated cracks were deflected along the ±45° second order lamellae. The ±90° build-up of first order lamellae led to crack bridging in opposite directions and hindered rapid growth of the crack along the z-axis (depth of the sample). Similar results were reported by Häsa and Pinho [12] utilizing a synthetic carbon fibre composite. Additionally, the combination of deflection and bridging prevented brittle failure usually associated with wood depicted in figure 6 and described in literature [30, 32]. The predetermined propagation path, given by the ±45° built-up of second order lamellae, prevented the crack from being arrested by areas of higher strength which is often exhibited in failure of plywood and solid wood [30]. However, compared to plywood and solid wood the load carrying capabilities of the wooden CLS remained on a much higher level after yield stresses were succeeded (figure 6) due to the increased number of processing zones.

The WOF (figure 5(A)) as well as σ obtained from the same load-displacement curves as depicted in figure 6 indicate that the utilization of CLS in a wood composite successfully increased the energy-absorbing capabilities compared to plywood and solid wood. While the increase of the wooden CLS compared to plywood was statistically significant, the increase compared to solid wood did not show statistical significance. However, the wooden CLS did show the highest average as well the highest minimum value. Additionally, the scattering of the obtained values was much lower compared to solid wood which is further illustrated in figure 5(B). This goes in hand with literature [33, 34] describing that each additional processing step and decrease in size of the constituents of a wood composite further increases homogeneity and therefore decreases scattering of the mechanical properties. This size effect is particularly important when dealing with cracks and the toughness of materials [35]. The low scattering further proofs that failure and energy-absorption occurred much more stable compared to solid wood. Similar results have also been presented by Häsa and Pinho [12] for synthetic CLS. The current design is therefore competitive with solid wood and with further improvements in regards to yield strength the application of CLS promises to support a completely new high energy-absorbing wood composite.

As described in previous research [36] one domination factor influencing strength and stiffness [32] of wood is the fibre orientation. Therefore, the absence of parallel fibres in the structural design of the current wooden CLS significantly decreased strength as well as stiffness (figure 7). The introduction of CLS in a sandwich composite utilizing face layers with parallel fiber orientation should significantly increase yield strength. In contrast to natural CLS [37] the outer layers would therefore consist solely of third order lamellae i.e. longitudinally oriented wood plies. This adaption differs from the structural design of natural CLS and should increase yield strength and overall toughness while maintaining low levels of scattering. The expected improvement is further depicted in figure 8.

Yet, a trade-off between strength and toughness will remain as both are mutually exclusive [38, 39]. Additionally, the current fabrication process proposed in figure A1 requires multiple steps making the
production of CLS more complicated than e.g. plywood. Therefore, the process needs to be simplified in order to enable cost-efficient production. A different approach to improve cost-efficiency would be a simplification of the structure while maintaining the achieved fracture behaviour.

Possible applications should be found in areas where a balance of strength and toughness is demanded. Especially within the mobility sector different structural components e.g. bumpers or side impact beams often need to have high energy-absorbing capabilities in order to maximize passenger safety [40, 41]. Most of the current structural components used in automotive applications are either based on polymer composites [42] or different metal alloys [43]. Additionally, the increasing demand for environmental friendly products has led to the emergence of different natural composites including natural fibres [44–46] as well as wood [40, 29, 47, 48].

According to Ashby [49], wood and wood-based composites provide highly desired mechanical properties such as high stiffness and strength at a very low density. However, the quasi-brittle failure of solid wood [30] diminishes the crashworthiness compared to other structural composites. The here presented biomimetic composite based on the natural CLS opens a new front for the development of crashworthy bio-based composites for high-performance applications. However, the quasi-static tests performed in the present study does not account for the strain rate dependency of wood [50, 51], influencing the mechanical behaviour under dynamic loading. Subsequently, additional research needs to be conducted in order to enable cost-efficient production and improve initial strength as well as further increase overall toughness, especially under dynamic loading.

5. Conclusions

The presented work proofed that the failure mechanisms found in natural crossed lamellar structure of mollusc shells can be replicated with the bio-composite wood leading to an increased toughness compared to ply wood and competitive levels compared to solid wood. The dominating failure mechanisms crack deflection as well as crack bridging were successfully implemented using the same hierarchical build-up as observed in natural CLS. The established 8-step fabrication method enabled effective reproduction of the complex structure. The structural design of the current wooden CLS leads to a significant decrease in overall strength and stiffness. The absence of ±0° unidirectional layers within the design compared to ply- and solid wood was identified to be the main reason for the decrease. However, scattering of strength and stiffness was much lower compared to solid wood. This low scattering further hinted on a much more stable failure and energy-absorption compared to solid wood. Possible improvements to the current structural design in order to increase strength and further improve toughness were proposed and should be investigated in future research.

Acknowledgments

The results presented in this study are part of the research project ‘Austria Biorefinery Centre Tulln’ (ABCT). The financial support by Amt der Niederösterreichischen Landesregierung (K3-F-712/001-2017) and Weitzer Parkett GmbH & CO KG is gratefully acknowledged. Additionally, the authors are thankful for the financial support by the Austrian Research Promotion Agency (FFG, 861421), Styrian Business Promotion Agency (SFG, 1.000.054.442), Standortagentur Tirol (FGS861421) and from the companies DOKA GmbH, DYNAmore GmbH, EJOT Austria GmbH, Forst-Holz-Papier, Holzcluster Steiermark GmbH, IB STEINER, Lean...
Management Consulting GmbH, Magna Steyr Fahrzeugtechnik AG & Co KG, MAN Truck & Bus AG, MATTRO Mobility Revolutions GmbH, and Volkswagen AG.

Appendix

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