Cost FP 1004 – STSM

Scientific Report

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1 Purpose of the STSM

Numerous innovations in wood technology and construction base on the application of hardwoods. Reliable designs presume durable adhesive joints that maintain their performance during the whole lifetime. In hardwood designs, this challenges adhesive systems, since they mostly were designed for softwood applications. Hardwoods generally have a more distinct hygroscopic behaviour than softwoods, resulting in higher internal stresses with climatic changes, what finally may lead to delamination in or at the bond lines of such structural elements. With this in mind, the STSM shall contribute to a better understranding of the delamination processes of bond lines in hardwood elements. The goal was to measure the adhesion of a melamine urea formaldehyde (MUF) resin and one-component polyurethane (PUR) adhesive onto beech wood (*Fagus silvatica* L.) on the cellular level by means of nanoindentation (Fig. 1).

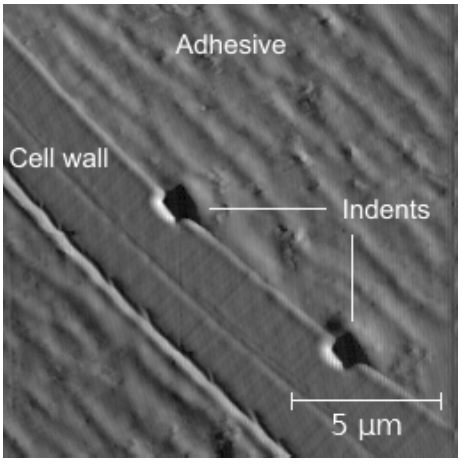

Fig. 1: Indentation spots after adhesion measurement at spruce/PUR interface (SPM image).

2 Work Carried Out

Routine pre tests (tensile lap shear test according DIN EN 302-1 [1]) showed bad quality of the MUF bonded samples. It was decided to use phenol resorcinol formaldehyde (PRF) resin as alternative. This adhesive system is – like the MUF – water based and well established in the industry. Therefore it was found legitimate to replace the MUF with PRF and no further drawbacks originated.

The specimens were subjected to the treatment A5 from DIN EN 302-1 Tab. 1 [1], i.e. boiling in water and redrying afterwards, as kind of a simple ageing process. In addition, samples that were exposed directly to the weather during the last three years were analysed to highlight the effects of weathering and compare them with the treatment A5. As reference for the adhesion analysis, surface treated samples with reduced wettability (water) were produced. The surfaces were silylated according to Mohammed-Ziegler et al. [2] with octadecyltrichlorosilane and chlorotrimethylsilane. Water based adhesive systems like MUF or PRF show heavily reduced adhesion in such samples. How the PUR reacts on such surfaces had to be determined.

In Tab. 1 an overview of the samples and treatments is given. Due to a lack of time it was not possible to analyse the A5 treated PRF samples.

Table 1: Analysed samples

	untreated	silylated	A5	weathered
beech / PUR	•	•	•	•
beech / PRF	•	•		
spruce / PUR	•	•	•	
spruce / PRF	•	•		

Weathered samples were only available in the combination beech/PUR. Beech/MUF would have been possible but omitted because of the cancellation of the regular MUF samples and for lack of time.

Generally the adhesion onto beech wood was measured at the tertiary wall in the lumen of the vessels. In the untreated and silylated beech/PUR samples the adhesion was also measured at the tracheid cell wall to distinguish possible differences.

In all tested samples the modulus of elasticity (MOE) and hardness were measured additionally in the wood cell wall as well as in the pure adhesive. These measurements ensure the comparability of the results and no biasing effect originated by possible variability of the cell wall material. All measurements were done according to Obersriebnig et al. [3] under the guidance of Mr. Obersriebnig and Assoc.Prof. Konnerth.

3 Main Results

For a serious analysis of the results the precision of each individual indent position has to be checked within the next weeks. As the procedure is not finished yet the data shown here exibits slightly higher scattering of the results than expected for the final version.

Generally it can be said that no treatment affects the hardness or MOE of the individual materials (see Fig. 2 and Fig. 3). Only PRF seems to slightly increase the wood cell wall characteristics. This observation corresponds with Konnerth et al. [4], where they conclude that PRF components penetrate the wood cell wall, but PUR does not.

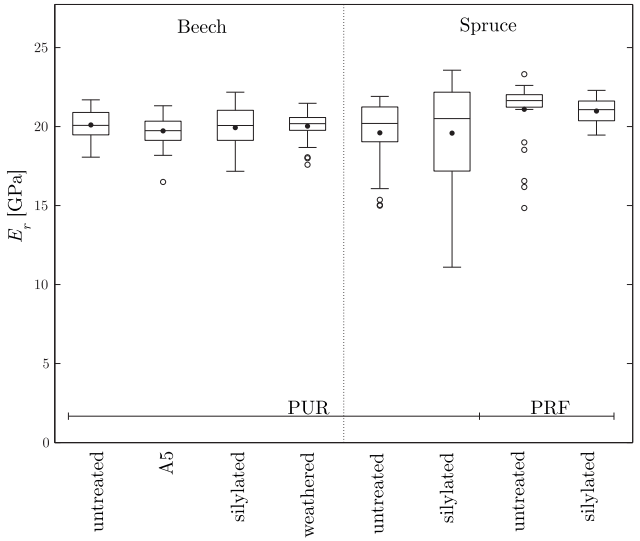

Fig. 2: MOE of the wood cell walls.

With the individual bond line components being unaffected by the treatments, the results from the adhesion measurements (Fig. 4) can be compared. For the spruce/PUR system the following conclusions can be drawn:

- The system is not significantly affected by any treatment.
- The adhesion is slightly lower than in the beech/PUR system.

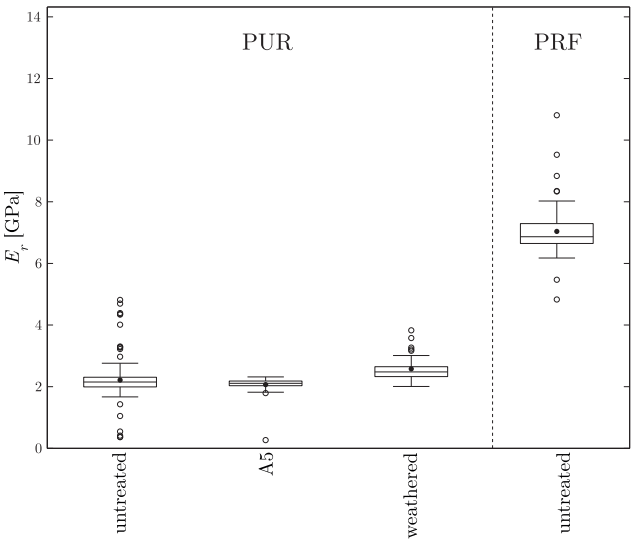

Fig. 3: MOE of the pure adhesives.

For the beech/PUR system the following conclusions can be drawn:

- A5 increases the adhesion, without changing the adhesive itself (Fig. 3).
- Weathering has a similarly positive effect like A5 treatment.
- The adhesion is potentially higher at the vessels than at the tracheids.
- The silylation seems to only influence the adhesion at the tracheids.

As expected the reduction of the wettability by silylation heavily influences the performance of the PRF bond lines. Other than within the PUR samples, the silylated PRF samples allow to extract the adhesion work from the complete indentation work.

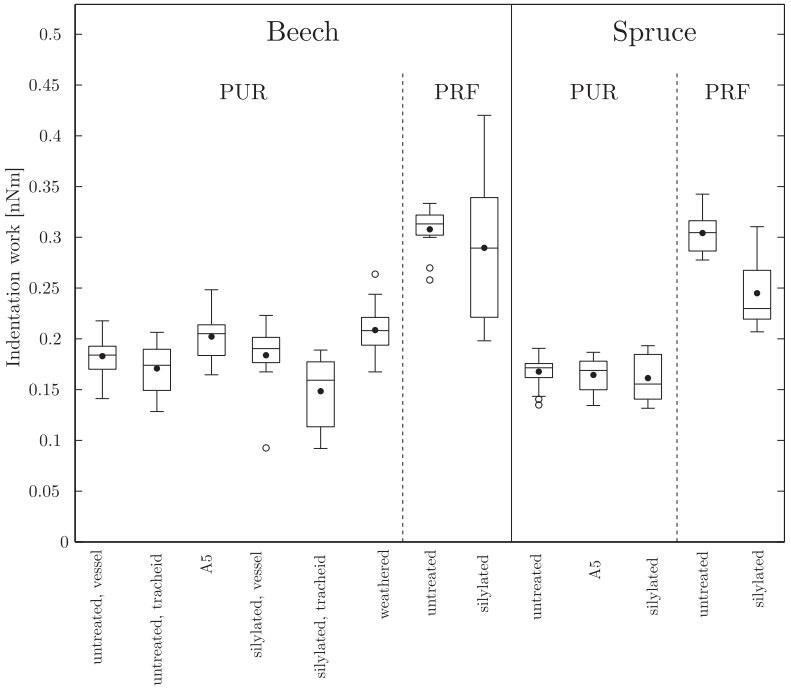

Fig. 4: Results of the adhesion easurements.

These experiments show the feasibility of adhesion measurements by means of nanoindentation directly at the interface of the adhesive and beech wood cell wall (S3), a testing setup that was successfully tested in spruce wood by Obersriebnig et al. [3].

In contrast to the water based PRF, PUR seems to be insensitive to surface treatments of the beech wood. Weathering and A5 treatment does not influence the pure PUR or beech wood, but a tendency to an increased adhesion is noticeable.

4 Future Collaboration

In addition to the STSM a second short stay in Vienna is considered. The measurements that could not be done during the STSM because of the lack of time could be made up to complete the data set. It has not yet been decided wether additional measurements are necessary or not.

In autumn our research groups will together have a colloquium concerning the ongoing wood research. It is mainly for the doctoral students to discuss their research and for an exchange of ideas.

5 Planned Publication

An article based on the results of the STSM will be published in a scientific journal.

At the *International Conference on Wood Adhesives* in Toronto, taking place in October 2013, the experiments will be mensioned in a presentation and in the paper for the proceedings. At this conference the colleagues from Vienna will also participate and talk about this testing method. Therefore these results will not be the main content in our conference contribution.

6 Confirmation by Host Institution

In the name of the host institution, the University of Natural Resources and Life Sciences (BOKU), Vienna, the undersigned Assoc.Prof. Dipl.-Ing. Dr. Johannes Konnerth confirmes that Samuel Ammann has undertaken the proposed STSM.

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References

[1] DIN EN 302-1. Adhesives for load-bearing timber structures – Test methods – Part 1: Determination of longitudinal tensile shear strength, 2011.

[2] I. Mohammed-Ziegler, Z. Hórvölgyi, A. Tóth, W. Forsling, and A. Holmgren. Wettability and spectroscopic characterization of silylated wood samples. *Polymers for Advanced Technologies*, 17(11-12):932–939, 2006.

[3] M. Obersriebnig, J. Konnerth, and W. Gindl-Altmutter. Evaluating fundamental position-dependent differences in wood cell wall adhesion using nanoindentation. *International Journal of Adhesion and Adhesives*, 40:129–134, Jan. 2013.

[4] J. Konnerth, D. Harper, S.-W. Lee, T.G. Rials, and W. Gindl. Adhesive penetration of wood cell walls investigated by scanning thermal microscopy (SThM). *Holzforschung*, 62 (1):91–98, Dec. 2007.