

3D Characterization of Adhesive Penetration into Wood by Means of Synchrotron Radiation

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Abstract

μ -tomographic investigations on bonded wood samples were conducted using synchrotron radiation to characterize the pathways for adhesive penetration in beech wood. Single growth rings were analyzed to obtain the porosity and the pore size distribution of the vessels over the growth-ring width and to characterize the connectivity of the vessel network in a three-dimensional way. It could be shown that porosity and pore size distributions are similar for growth rings of various widths. The wood rays force the vessels to weave in the tangential direction. As a result, during penetration, regions at quite different distances perpendicular to the bondline are filled instantaneously. We conclude that investigations of single cross-sections rarely provide sufficient information on the penetration process of an adhesive into wood.

Introduction

The development of a proper bondline is crucial for the strength of a wood adhesive bond (Kamke and Lee 2007). If too much adhesive is transported into the porous network of the adherend, the bondline starves, reducing its strength. A starved bondline has the same effect as if not enough adhesive had been applied in the first place. But also too much adhesive in the bondline will have

negative effects on the strength or the toughness of the bond. Most of the investigations on adhesive penetration into wood are based on micro slides, which were used to characterize the penetration behavior of an adhesive by measuring the penetration depth (e.g., Sernek et al. 1999, Kamke and Lee 2007). This parameter describes the perpendicular distance between the bondline and the most distant adhesive-filled pore in a cross-section. This method bears several difficulties in the interpretation of the results. One is the influence of the sample preparation. Micro slides are usually produced after a special treatment of the wood—extreme ways of treatment are plasticization in boiling water, saturation with epoxy resin, deep freezing (cryo)—which may affect the bondline. On the other hand, the penetration process has to be regarded as a 3D phenomenon. By reducing the investigation to a 2D-analysis, it is evident that information is lost and no conclusions on the influence of the wood anatomy or the volumetric distribution of the adhesive can be drawn. This is demonstrated in **Fig. 1** on the bondlines of a polyurethane adhesive in beech (*Fagus silvatica* L.) [**Fig. 1** (left)] and spruce wood (*Picea abies* karst.) [**Fig. 1** (right)]. There are obvious differences between the bondlines in different wood species. In softwood, the bondline is more or less an interconnected

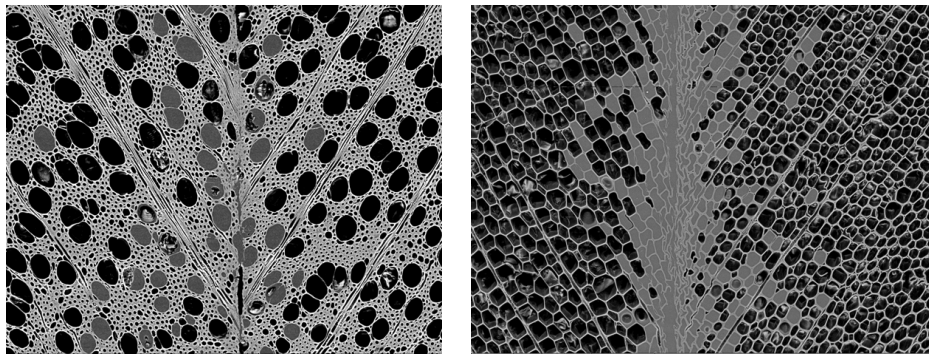


Figure 1. ~ PUR-bondlines in wood; left: beech; right: spruce.

area, where in hardwood, many vessels are visible which appear to be isolated from the bondline. A reason for this distinct appearance may lie in the anatomical differences between the wood species.

To describe the penetration processes in porous media, it is crucial to analyze the pore space of the medium in which the penetration takes place. For hardwood, this means a characterization of the vessel network. A volumetric description of the vessel network of beech has been carried out in (Bosshard and Kucera 1973). They prepared series of micro slides that provided a stack of cross-sections as vertical layers of the specimen and followed the three-dimensional distribution and position of the pores. Clearly this method is very time and labor intensive. With modern tomography methods, an analysis of the volumetric configuration of wood is more practicable. However the majority of the investigations in this field use the technique only for qualitative purposes (e.g., Mannes et al. 2007, Trtik et al. 2007).

In our study, we analyzed the vessel network in separated growth rings of beech wood. The porosity and the pore size distribution over the growth ring were investigated, as well as the connectivity and the appearance of the vessel network. This way, it becomes possible to determine the influence of the wood anatomy on the appearance of bondlines in wood.

Material and Methods

The μ -tomography investigations were conducted at the TOMCAT beamline of the Swiss Light Source (SLS, PSI, Villigen, CH) using synchrotron radiation. The principle of the technique is well explained in (Mannes et al. 2009). Bonded beech wood samples were investigated to analyze the penetration behavior of different adhesive systems. In detail the wood was bonded using the three adhesive systems polyurethane (PUR), polyvinyl acetate (PVAC), and urea formaldehyde (UF). To avoid interference during the data acquisition, the samples were turned to a diameter of 3 mm with the bondline in the middle of the sample. After the reconstruction step, the tomography data was available as image stacks of cross-sections with a spatial resolution of 3.7 μm per voxel.

Results

Following the work of (Bosshard and Kucera 1973), the reconstructed images allow the tracing of individual vessels along the longitudinal direction through the wood.

However with modern computer systems, the analysis of the pore network becomes easier and more precise. Using several image-processing steps it is possible to segment the wood from the adhesive and the wood substance from the non-filled vessels (Fig. 2).

The information about the adhesive can be used to investigate its penetration behavior and to quantify the geometry of the bondline in a spatial way. First impressions show distinct differences between the used adhesive types concerning penetration (Fig. 3), but this analysis is still in progress.

To characterize the pore space in the samples, 15 single-growth rings with widths between 768 and 2061 μm were investigated. The porosity and the pore size distribution (resulting from the vessels) were analyzed as a function of the respective position. For comparison, the radial position of the vessels has been normalized by the growth ring width. The growth rings have been binned to obtain the relative porosity, which arises from the ratio of white pixels to black ones as binarized images [Fig. 2 (right)] were used for the analysis. In a next step

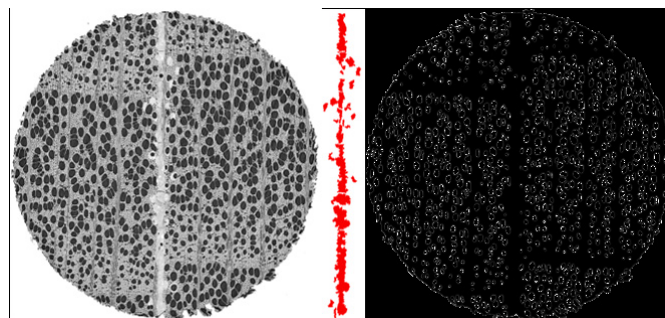


Figure 2. ~ Image processing: Segmentation of adhesive and pore space.

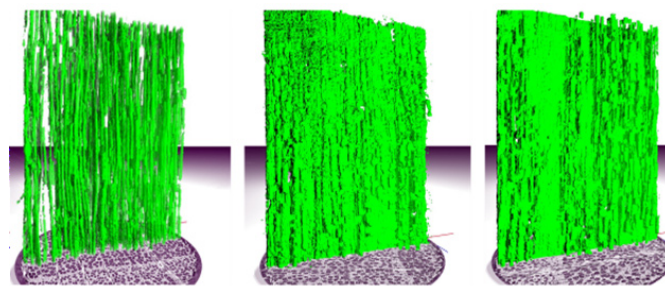


Figure 3. ~ Segmented bondlines in beech wood; right to left: PUR, PVAC, and UF.

the connectivity between the vessels was retraced and the vessel network was reconstructed.

Figure 4 shows the porosity and pore size distribution across the growth ring. Fitting of a third grade polynomial describes the distributions very well, both for narrow and for wide growth rings. This indicates that these two factors are less dependent on the growth ring width, but the position in the growth ring.

The connectivity between the pores in one (reference) layer and a neighboring one is analyzed by projection of the pores into each other. If the projection of a pore meets another pore in the neighboring layer, the vessel

continues. If a projection meets more than one pore, the vessels are joining [**Fig. 5** (left)] and if several projections meet one pore, the vessel is splitting [**Fig. 5** (right)].

Figure 6 (left) shows an example for a reconstructed vessel network. It can be seen clearly that the vessel network is strongly influenced by the wood rays, which force the vessels to “wobble” around them. Moving through the specimen in longitudinal direction, it leaves the impression that the vessels perform an oscillating wave in the LR-plane. The connectivity analysis provides regions in the vessel network, which are interconnected with each other (cluster). Theoretically, in beech

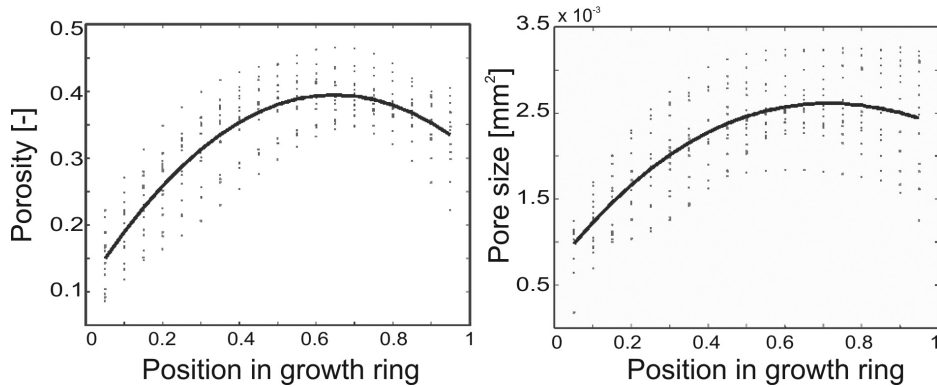


Figure 4. ~ Distribution of porosity and pore size of beech over position in growth ring from latewood (0) to earlywood (1).

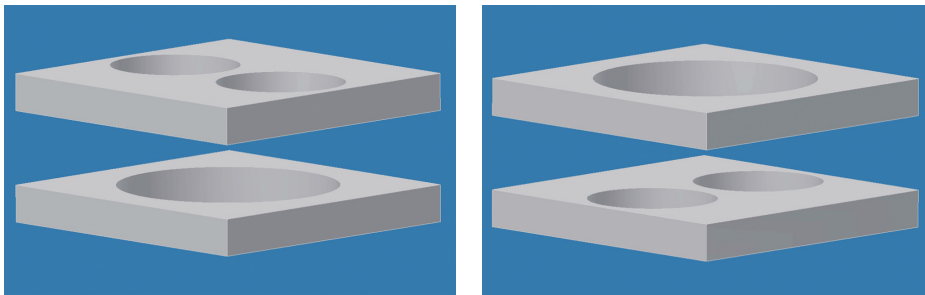


Figure 5. ~ Principle of connectivity analysis; left: splitting of vessels; right: joining of vessels.

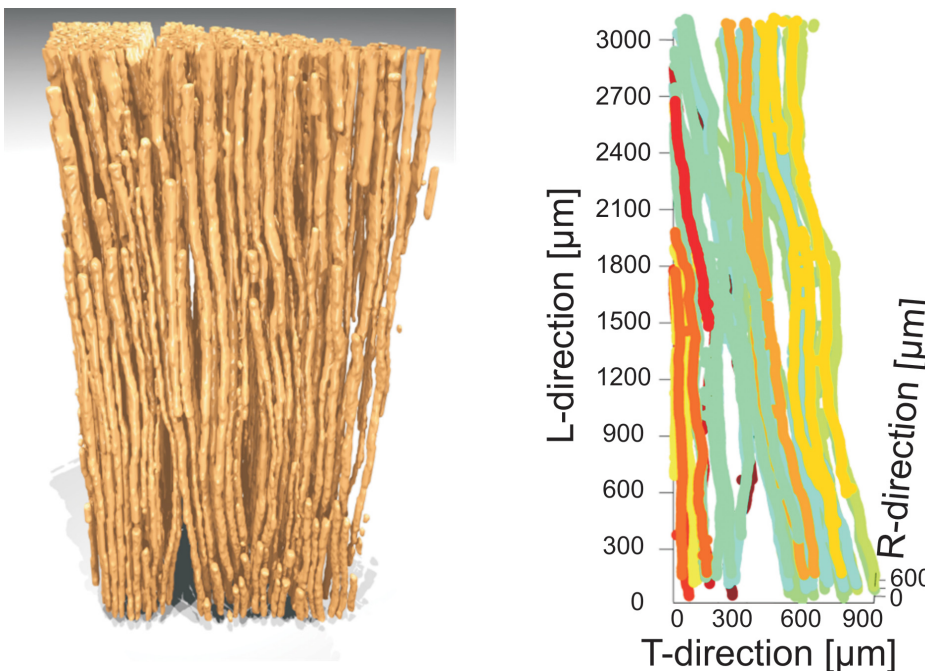


Figure 6. ~ Left: Reconstructed vessel network of a growth ring. Right: Ten biggest clusters.

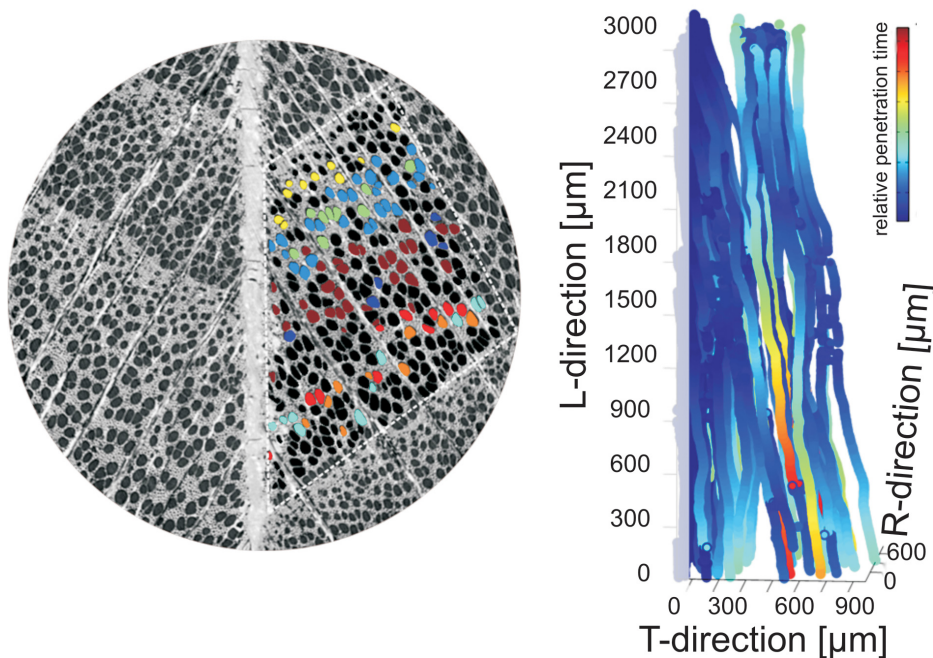


Figure 7. ~ Left: Cross-section with the eight biggest clusters. Right: Penetration face.

wood all the vessels in a growth ring should be interconnected (Wagenführ 1999), but due to the fact that not the whole of the growth ring was analyzed, isolated regions exist and several clusters with different size and expansion are distinguishable [Fig. 6 (right)].

This means that for glued wood parts, even areas which are very close to the bondline do not necessarily have to be filled by adhesive if they are not connected by the network to the bondline. If the information about the clusters is projected into the cross-section of the samples, the tangential orientation of the vessels is confirmed. Figure 7 (left) shows that the clusters are aligned as a tangential band along the tangential direction. This anatomical characteristic of course influences the penetration process of a liquid into the vessel network. A simple penetration simulation along the channels of the vessel network shows that a liquid reaches positions that have a completely different distance from the bondline in the LT-plane at the same time [Fig. 7 (right)].

As a consequence, a single image of a bondline and the measuring of the distance between bondline and adhesive-filled pore does not provide any information about the way the adhesive has traveled to fill this pore and can therefore be insufficient to characterize the penetration behavior of an adhesive. Therefore the idea of the penetration depth as a reference factor for the penetration behavior of adhesives into hardwood should be reconsidered.

Conclusions

The analysis showed that porosity and pore size distribution of beech wood are not connected to the growth ring width, but to the position in the growth ring. The vessel network in beech wood is strongly influenced

by the wood rays, which force the vessels to perform a wave movement in the RL-plane. Therefore the inspection of single cross-sections and the use of the parameter “maximum penetration depth” as the distance of the most distant adhesive-filled pore from the bondline will not provide sufficient information about the penetration behavior of a liquid. The appearance of cured adhesives in cross-sections is predominantly influenced by the connectivity of the vessels and the movement of the vessels around the rays.

Acknowledgments

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