Short Term Scientific Mission

Adhesion characteristics of modified wood

in the framework of COST Action FP1006:

Bringing new functions to wood through surface modification

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Report

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1 Introduction

Fundamental mechanisms of wood bonding on different types of modified solid wood are in focus of this work. Bonding mechanisms via secondary interactions or mechanical entanglement are of major interest. It is necessary to define those bonding characteristics in dependence on both the adherent material as well as the adhesive system, because material properties of modified and unmodified wood are differentiating distinctively, for example.

Studying bonding features wood modification systems are utilized to identify properties influencing the bonding strength specifically. Thermal, wax, melamine or furfuryl modifications cause both physical and chemical alterations of material properties. In combination with a dielectric barrier discharge at atmospheric pressure surface characteristics are examined being able to estimate adhesion behavior.

In the framework of the Short Term Scientific Mission those changes of nanoscale-interactions are investigated by means of Nanoindentation of which Prof. Konnerth and his working group have longstanding experiences. The effect of plasma treatments on aged and freshly cut surfaces is studied varying wood species and modification system. All adhesion characteristics are quantified separately in dependence on wooden tissue (early wood / late wood), cell-layer (bondline / adjacent) as well as cell wall (S2 / S3).

2 Materials and Methods

2.1 Object of study of Nanoindentation measurements

Nanoindentation measurements are used to determine adhesion characteristics between modified wood and various adhesive systems. Five variables are examined (Table 2-1).

Table 2-1: Variables, which are object of study for Nanoindentation measurements.

No.	Variables	Characteristics		
1	Wood Species	beech	spruce	
2	Modification	untreated	thermal	furfuryl
3	Surface treatment	freshly cut	aged	plasma
4	Cell wall layer	S2	S3	

5	Adhesive system	MUF	PUR

The wood species of interest are beech (*Fagus sylvatica* L.) and spruce (*Picea abies* Karst.).

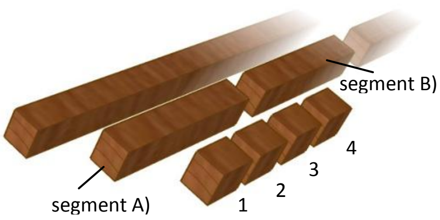
2.2 Sample preparation

Nanoindentation measurements are extremely sensitive to both various physical as well as chemical characteristics of the wooden tissue (Konnerth and Gindl 2006). As a result, great care is taken during sample preparation. It is implemented in accordance to examinations (Konnerth, Harper et al. 2008).

2.2.1 Preparation of adherents for bonding

Bonding of specimens is implemented in Göttingen. Previous to the bonding process the wooden material is treated specifically (Table 2-2).

Table 2-2: First preparation steps of adherent production.

1/4	Göttingen 	**Precutting** - Board: cutting, dressing, planing - Pillars are formed (10 x 10 x 400mm³) - Parallel to fiber orientation
2/4	Göttingen  	**Sectioning** - Cutting of pillars into segments (10 x 10 x 50mm³) - Cutting of segments into four blocks (10 x 10 x 10mm³)
3/4	Göttingen 	**Finishing** - Cutting of the radial surface by a microtome

4/4 Göttingen

Surface treatment & bonding

- Surface treatment according to the following steps:

Surface	Fresh	Aged (24h)
untreated	A1+A2	B1+B2
Plasma	A3+A4	B3+B4

The first two blocks of a segment are bonded without any plasma pre-treatment, whereas the third and fourth blocks are plasma treated prior to bonding. Plasma modification is implemented by the working group of our cooperation partner Prof. Dr. Viöl at the University of Applied Sciences and Arts (HAWK), Faculty of Natural Sciences and Technology in Göttingen, Germany. For plasma activation a dielectric barrier discharge (DBD) at atmospheric pressure is used to increase hydrophilic surface properties (Table 2-3).

Table 2-3: Parameters of plasma treatment.

Gas	Air		Frequenz	70	[kHz]
Pressure	1013	[hPa]	Object distance	2	[mm]
Plasma power	90	[%]	Treatment time	10	[s]

For the treatment wood blocks are placed between two isolated electrodes, of which the discharging gap is about 2mm (Figure 2-1).

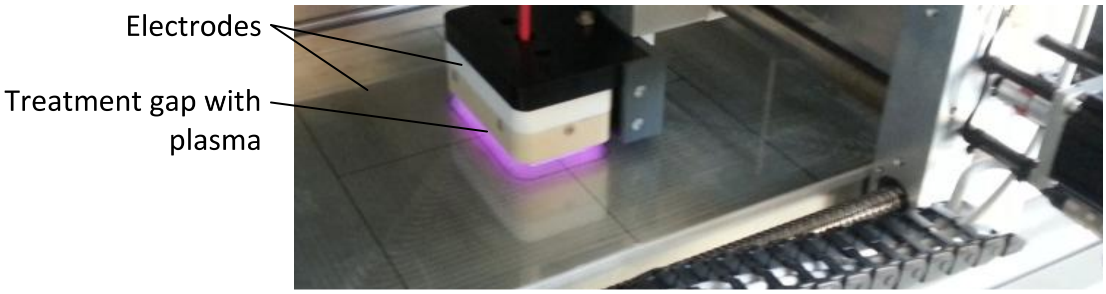


Figure 2-1: Equipment for plasma treatment.

2.2.2 Preparation of specimens for Nanoindentation measurement

Preparation of specimens for Nanoindentation measurements is conducted in Vienna (Table 2-4).

Table 2-4: Second preparation steps of specimen production.

1/4 Vienna	**Bondline focussing**
- Cutting of specimens centering the bondline	

		- Rectangular fiber alignment towards the grain
2/4	Vienna	**Specimen fixation**
		- Preparation of small specimens (2 x 2 x 2mm³)
		- Fixation of samples on AFM specimen discs
3/4	Vienna	**Surface treatment**
		- Reduction of surface area using a razor blade
		- Flatting the surface utilizing an ultramicrotome
4/4	Vienna	**Bondline mapping**
		- Microscopic mapping of the bondline (Figure 2-2)

Surfaces are cut by an ultramicrotome from Leica, (Ultracur R), which is equipped with a diamond knife from Diatome (Histo, ultra35). Bondline mapping is based on magnifications of 100x and 500x, respectively (Figure 2-2).

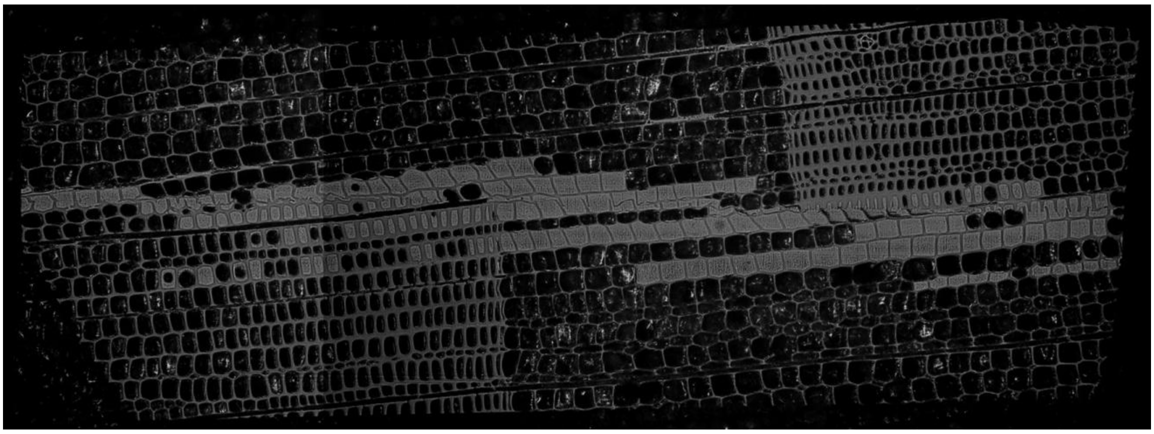


Figure 2-2: Microscopic mapping of the bondline, magnification 100x.

The overall procedure of sample preparation is implemented in accordance to earlier work (Konnerth, Harper et al. 2008).

2.3 Measurements

For all Nanoindentation experiments a Triboindenter system (Hysitron, TI-900, Minneapolis) is utilized. A Berkovich-type diamond tip with an opening angle of 143.2° is used to determine the mechanical properties of the cell wall and adhesive, respectively. The load ramp is force controlled and consists of three segments and has a peak load of 250µN. Maximum load is reached within 3s, is held constant for 10s and eventually is unloaded within 3s. Only cell wall areas, which are adjacent to adhesive on all sides, are tested right in the middle of the cross section (Figure 2-3). Adhesive characteristics are measured in the middle of the filled lumen diameter (Figure 2-4).

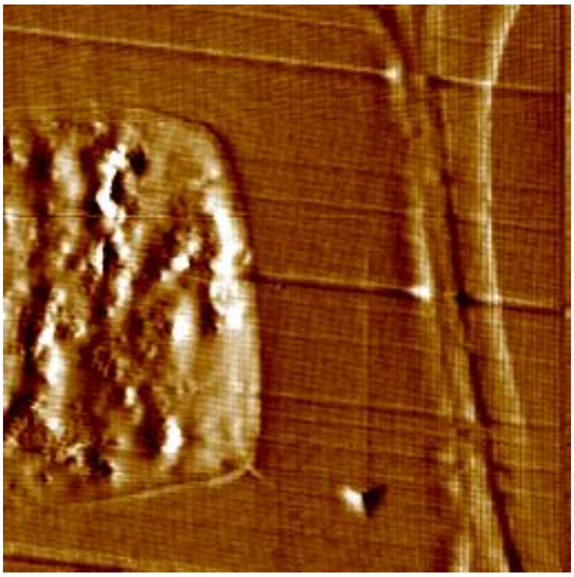


Figure 2-3: Example of an indent for cell wall characterization.

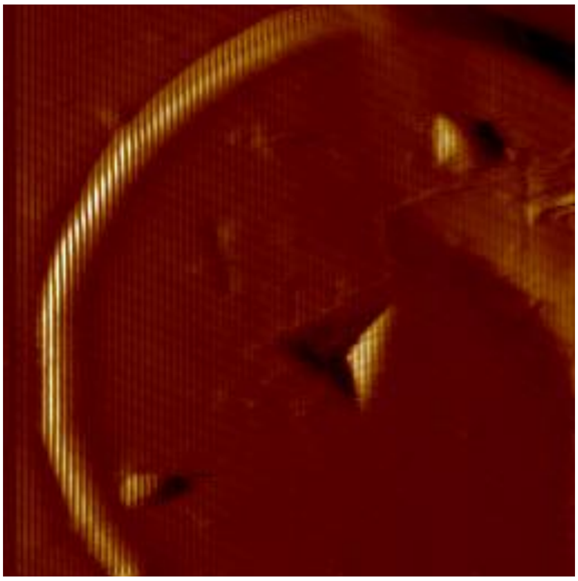


Figure 2-4: Example of an indent for adhesive characterization.

Adhesion is determined by placing the indenter tip directly into the interface between adhesive and wood cell wall (Figure 2-6). The procedure of indentation is implemented in accordance to former work (Obersriebnig, Veigel et al. 2012).

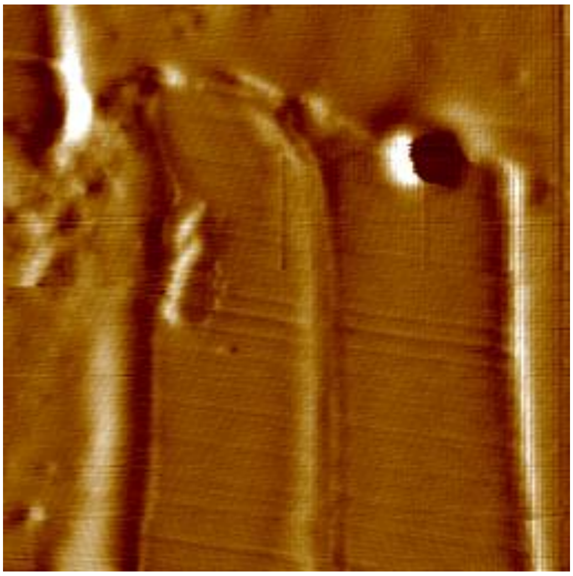


Figure 2-5: Location of nanoindents for quantification of adhesion of S2 and adhesive.

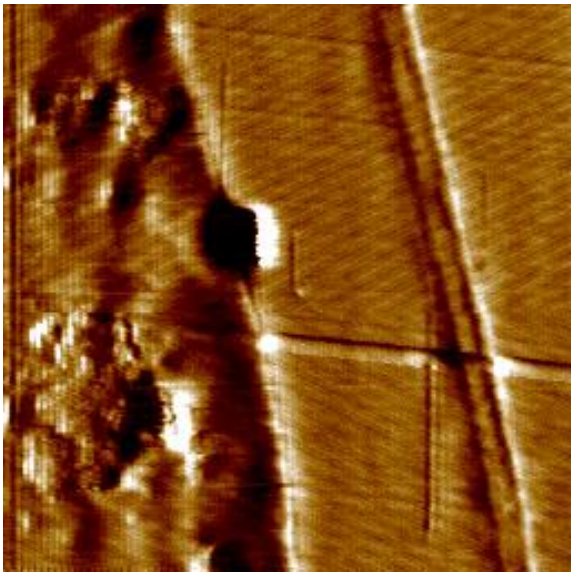


Figure 2-6: Location of nanoindents for quantification of adhesion S3 and adhesive.

A cone-shaped diamond tip with an opening angle of 60° and a tip radius of approximately 150 nm is utilized. In contrast to material characterization measurements the indents for adhesion evaluation are performed in a displacement controlled mode. A four-cycle load function with maximum penetration depths of approximately 64, 230, 490 and 850nm is implemented. The displacement level is reached within 1s. A partial unloading of 50 % of the maximum penetration depth of the corresponding step at the end of the first three cycles is defined (1s). Moreover, the maximum displacement level of the first three cycles is held constant for 5s each. On the contrary, the last displacement level is held steady for 10s. Subsequently, it is unloaded completely (Figure 2-7, Table 2-5).

Figure 2-7: Displacement curve for Nanoindentation measurements of adhesion.

Table 2-5: Displacement cycles of Nanoindentation measurements of adhesion.

Phase		Cycle			
		1	2	3	4
Loading	Time in [s]	1	1	1	1
Holding	Time in [s]	5	5	5	10
	Displacement in [+nm]	64	230	490	850
Unloading	Time in [s]	1	1	1	1
	Displacement in [-%]	50	50	50	100

Before testing scanning probe microscopy is utilized to position the indents, of which images are generated by means of the indenter tip. The accuracy of positioning of all individual indents is ensured by an additional image being scanned subsequent to the indentation measurement.

Generally due the fact that stiffness and compression strength of early wood cell walls are usually relatively small, latewood tissue is in focus of investigation (Niemz 1993). Indentation hardness and reduced modulus (indentation modulus) in accordance to the Oliver and Pharr method are going to be used for comparing cell wall characteristics (Oliver and Pharr 1992). Calculation of adhesion energy is based on the total indentation work. Therefore, the area underneath the load-depth curve is going to be integrated. For each combination of variables a certain number of repetitions is implemented (Table 2-6).

Table 2-6: Distribution of indents in dependence on variables implemented on each specimen; *cell layer in contact with plasma/aging; **LW: late wood, EW: early wood.

Focus	Adherent	Cell layer*	Cell wall	Tissue*	No. of indents
Cell wall	Top	--	--	LW	10
	Bottom	--	--	LW	10
Adhesive	--	--	--	--	14
Adhesion	Top	1	S2	LW	7
			S3	LW	7
				EW	7
		2	S3	LW	7
				EW	7
	Bottom	1	S2	LW	6
			S3	LW	6
				EW	6
		2	S3	LW	6
				EW	6
				Total	99

Three replicates are produced of every specimen of specific factor combination.

3 Gain of knowledge

Due to the intensive preparation of specimens as well as time consuming measurements it is not possible to communicate first results. However, initial evaluations of load functions and displacement behavior of examined materials show promising results. Some of the findings seem to be in accordance to former work (Obersriebnig, Konnerth et al. 2013). Based on the results of Nanoindentation and Atomic Force Microscopic measurements potential effects of aging in combination with a plasma-induced regeneration of surface polarity seem to be able to be investigated on a nanoscale.

Personally I already got a detailed view into preparation techniques of samples dedicated for surface evaluation using ultramicrotomes during the stay in Vienna. In addition, I had the chance to learn much about material mechanics since all conclusions from Nanoindentation measurements are based on micromechanical behavior of both wood and adhesive, respectively.

Up to this point there are still a lot of experiments, which have to be accomplished, focusing on both Nanoindentation and Atomic Force Microscopic measurements. It is the aim to be able to identify suitable combinations of adhesive systems and adherent materials. On the one hand, it is intended to realize further collaboration intensifying characterizations of bonding mechanisms of surface modified materials on microscopic scales. On the other hand, in the framework of future investigations those findings are going to be verified by implementing adapted bonding processes with subsequent macroscopic testing in accordance to DIN EN 302-1:2011 (Adhesives for load-bearing timber structures - Test methods – Part 1: Determination of longitudinal tensile shear strength).

All in all the time of the STSM at the University of Natural Resources and Life Sciences in Vienna was greatly beneficial giving me the opportunity to gather knowledge and get in contact with other researchers dealing with similar topics.

4 References

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