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Fungal Degradation of Scots Pine (*Pinus sylvestris* L.) Wood: Micromechanical and Microstructural Characterization at the Cell Wall Level

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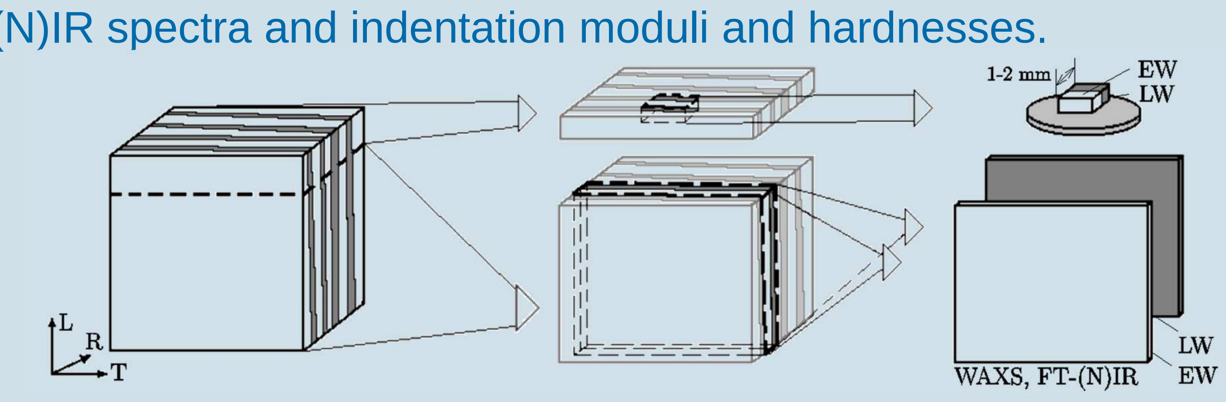
Motivation

Introduction

Fungal activity plays a major role among natural degradation processes, and thus also in relation to the degradation of wood. As a consequence of fungal decay, the inherent heterogeneous microstructure and the composition of wood are modified in different ways, depending on the specific fungal and host species. While white rot fungi degrade all major wood components, brown rot fungi preferably degrade wood polysaccharides, leaving behind a lignin rich microstructure. The mechanical properties of the wood cell wall are controlled by its inherent microstructure, mainly the inclination of the cellulose microfibrils in the S2 layer (microfibril angle, MFA), and its composition. Consequently, changes of microstructure or composition result in altered mechanical properties. To determine how the different degradation strategies of brown rot and white rot fungi affect the mechanical properties of wood cell walls and to establish structure-function relationships for deteriorated wood, sample-specific mechanical properties as well as microstructural and compositional data have to be acquired. Compositional data can be assessed by FT-(N)IR spectroscopic methods and wet chemical analyses, while the MFA can be measured by means of X-Ray diffraction. The typical thickness of a wood cell wall of 2 to 5 µm urges for micromechanical test methods such as nanoindentation.

Material and Sampling

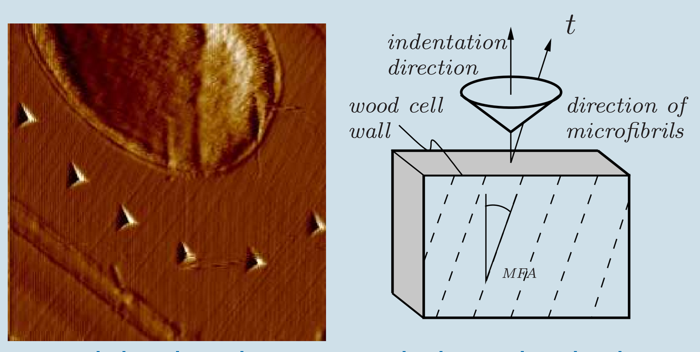
Scots pine (*Pinus sylvestris* L.) sapwood samples degraded by one white-rot and one brown-rot fungus were considered, as well as a set of non-degraded reference samples. Early stages of degradation, characterized by mass losses of 1%, 5% and 10%, were considered only. All experimental procedures were performed on wood from the same annual ring, yielding a set of earlywood/latewood specific MFA, FT-(N)IR spectra and indentation moduli and hardnesses.



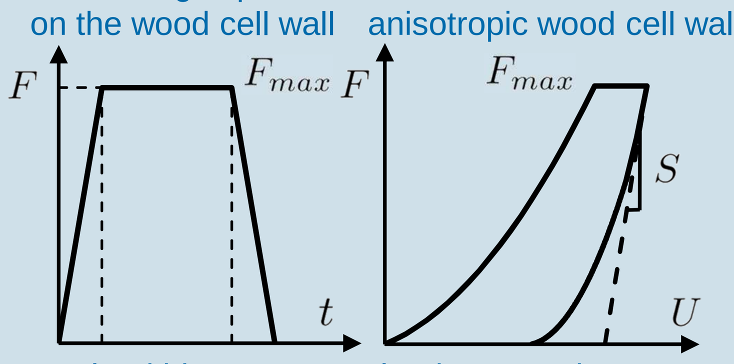
Sampling procedures for experimental program (EW…earlywood/LW…latewood)

Methods: Nanoindentation and FT-(N)IR and WAXS

Nanoindentation Tests [1]



remaining imprints on the wood cell wall — indentation in the anisotropic wood cell wall



load history — load-penetration curve

$$S = \partial F / \partial U \,|_{U=U_{max}}$$

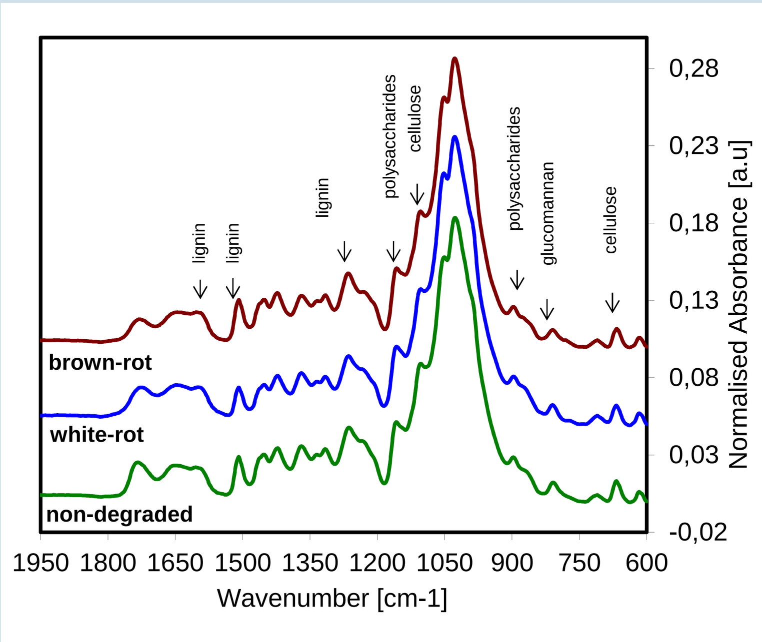
$$M = \frac{\sqrt{\pi}\, S}{2\sqrt{A_C}} \qquad H = \frac{F_{max}}{A_C}$$

S… initial unloading stiffness
A_C… contact area
F… load
U… penetration depth
M… indentation modulus
H… indentation hardness

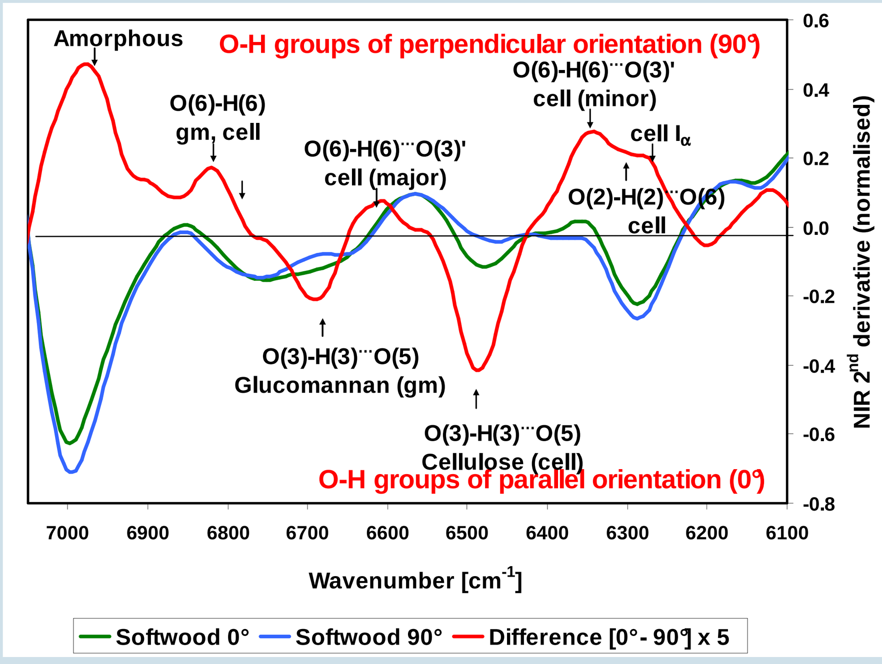
$$M_{ML} = f(C_{middle\ lamella})$$
$$M_{S2} = f(C_{S2\ layer}, MFA)$$
→ **no pure material property**

FT-(N)IR Spectroscopy

FT-(N)IR	Polarized FT-(N)IR	Isotope exchange + FT-(N)IR
↓	↓	↓
Composition, Molecular structure	Molecular orientation	Accessibility

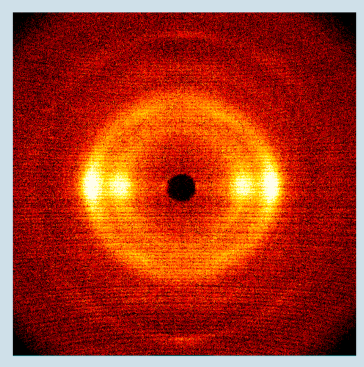


ATR-FT-IR spectroscopy to estimate chemical composition of small sample surfaces within annual rings and for calculation of regression models to predict micromechanical parameters



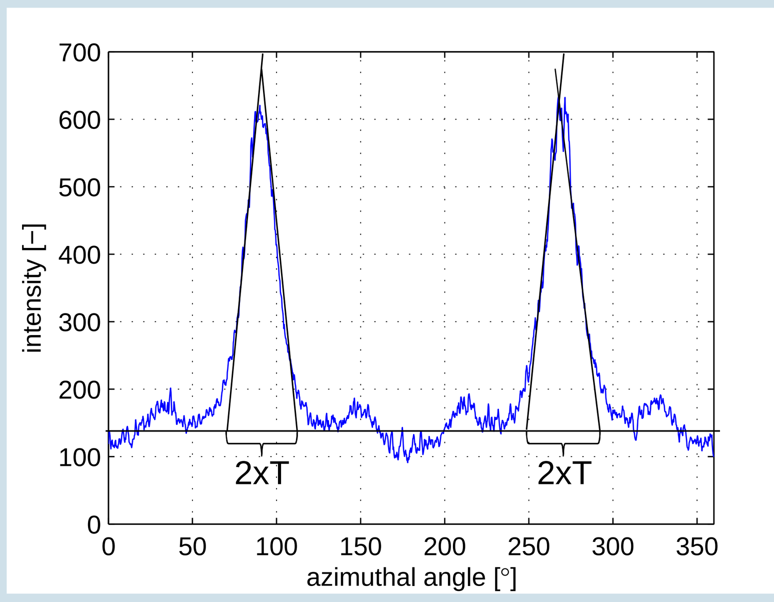
Polarized FT-NIR transmission spectra of thin sections reveal molecular structure and orientation [2]

MFA Determination



WAXS pattern

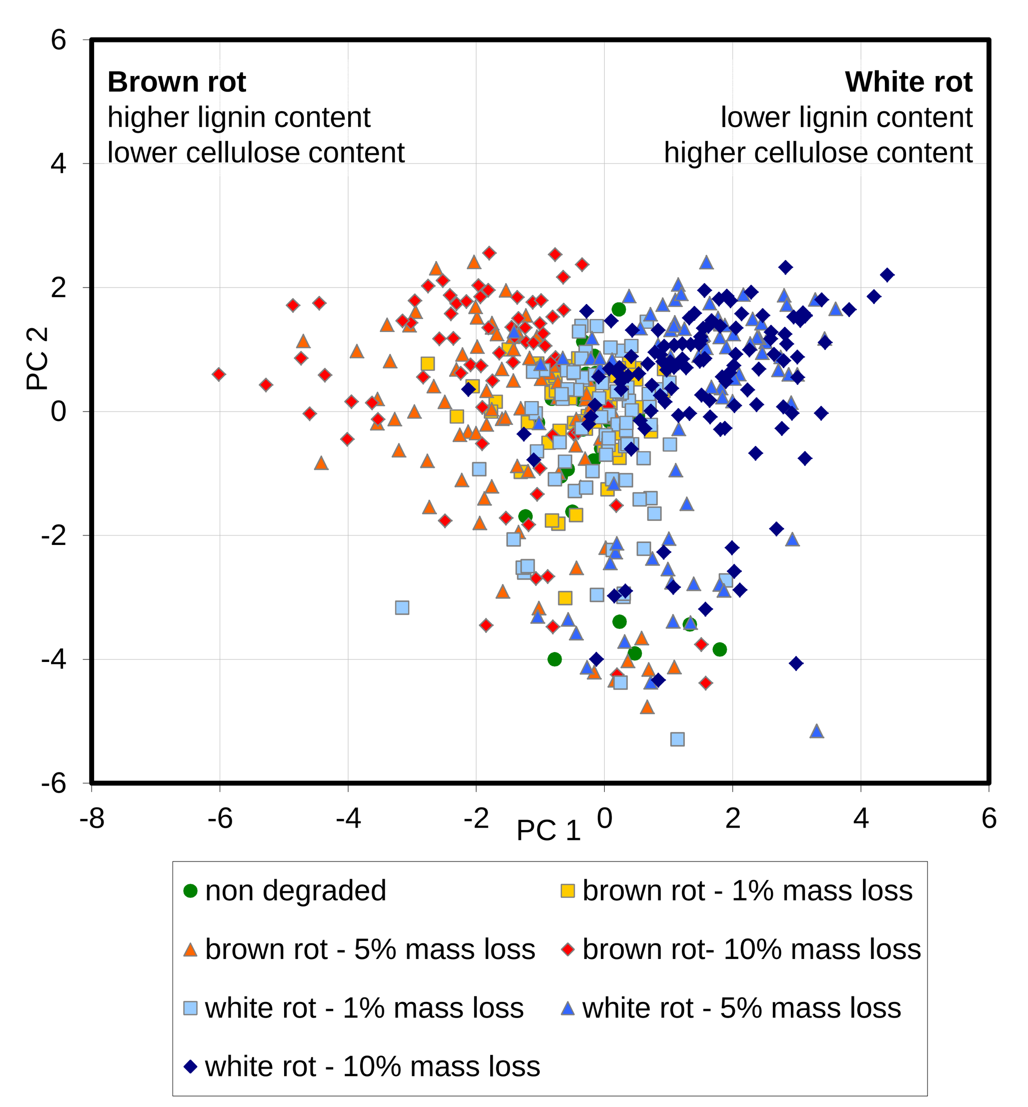
- wide angle X-ray scattering (WAXS) [3]
- integrate intensity of 002 reflection
- plot I(θ) vs. azimuthal angle
- determine "T-angle"



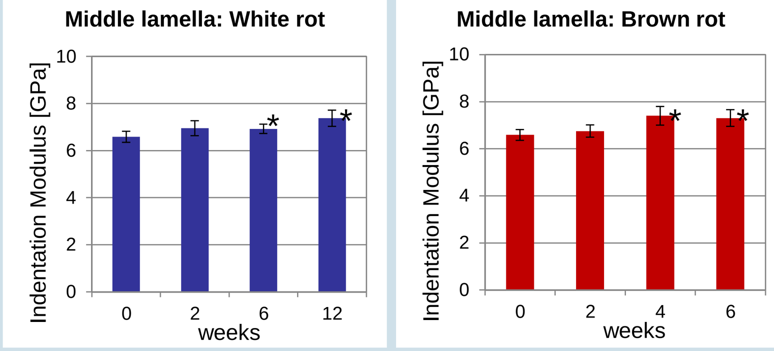
Integrated intensity over azimuthal angle

MFA = 0.6 x T

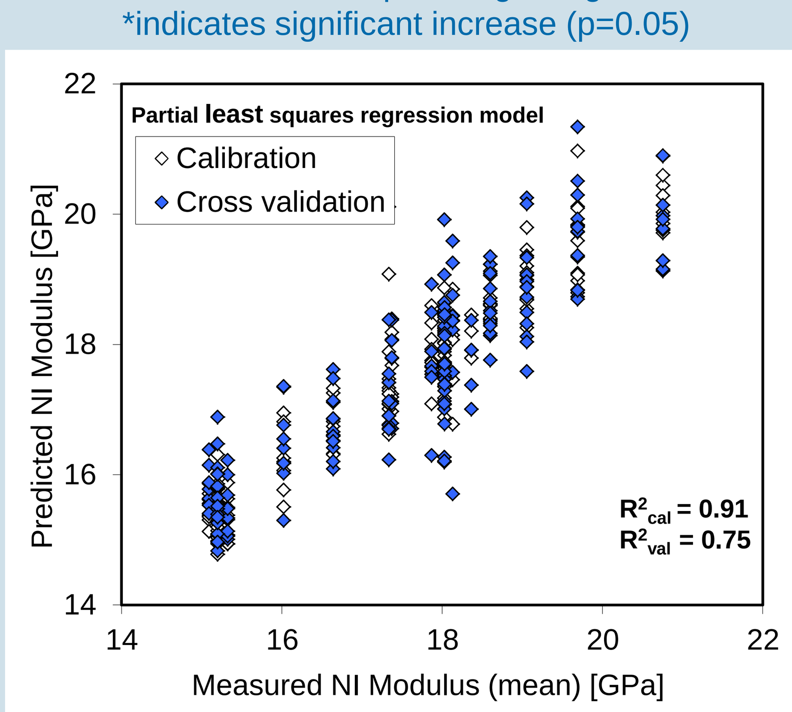
Results & Discussion



Multivariate data analysis (PCA) of FT-IR surface spectra of earlywood thin sections shows still high variability, although brown rot and white rot can be distinguished



Indentation modulus M of the middle lamella (ML) shows an increase upon fungal degradation; *indicates significant increase (p=0.05)



Spectroscopy data reveal high variability within an annual ring of white-rot degraded earlywood and are correlated to corresponding indentation modulus M of the S2 layer

The experimental program led to a data set with a high amount of spectroscopy, microstructural and micromechanical data that were not only EW/LW specific but also site specific within the same annual ring. Spectroscopy data still shows a high variability. In addition to variations in composition, local deviations of the cellulose fibril orientation from the MFA also add some variability to the indentation moduli M of the S2 cell wall layers of all samples, while M of the middle lamella is assumed to be related to the local composition only. An increase of both M and indentation hardness H of the middle lamella with progressing degradation could be observed, whereas no obvious trend for the stiffness of the S2 layer can be identified. However, validated regression analysis, using the highly variable spectroscopy raw data, can be applied to extract correlations between spectroscopy and micromechanics. From this, M of the S2 cell wall layer in EW during white rot can be reasonably predicted.

Further Research Activities:

- regression models to predict the highly variable chemical composition within earlywood and latewood samples from IR data
- use knowledge of altered ultrastructure to predict changes of mechanical properties of the wood cell wall during fungal decay by multiscale-micromechanical modeling
- conventional and multivariate correlation analysis of chemical, spectroscopy and mechanical data
- mapping of chemical composition and mechanical properties on cell wall level by Raman imaging and dynamic mechanical testing (nanoDMA®)

References:
[1] Oliver&Pharr (1992), *J Mat Research* 7(6):1564-1583
[2] Fackler&Schwanninger (2010), *JNIRS* 18(6): 403-416
[3] Cave (1997), *Wood Sci Technol* 31:225-234

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