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NANOINDENTATION-BASED MECHANICAL SPECTROSCOPY OF WOOD CELL WALLS

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Mechanical spectroscopy is the assessment of a mechanical index, such as the viscoelastic Young's modulus or the plastic flow stress, across a broad spectrum of time scale, deformation rate, temperature, or moisture content. In addition to providing thorough mechanical characterization, which is useful to predict material performance over a wide range of conditions, mechanical spectroscopy also provides information about the microphysical processes which are causally linked to the properties. Recently, broadband nanoindentation creep (BNC) has been developed to measure viscoplastic properties across 4-6 decades of strain rate and broadband nanoindentation viscoelasticity (BNV) to measure viscoelastic properties across greater than 4 decades of time scale. The capabilities to perform variable-temperature BNC and BNV at temperatures between 20 and 200°C have also been developed, and the relative humidity (RH) during the test can be controlled between dry air and 95%. In validation studies on polymers, the data generated from BNC, BNV, and the more typical dynamic nanoindentation technique all agreed with conventional viscoplastic and viscoelastic measurements. Furthermore, the nanoindentation results revealed the same information about the microphysical processes causally linked to the polymer properties.

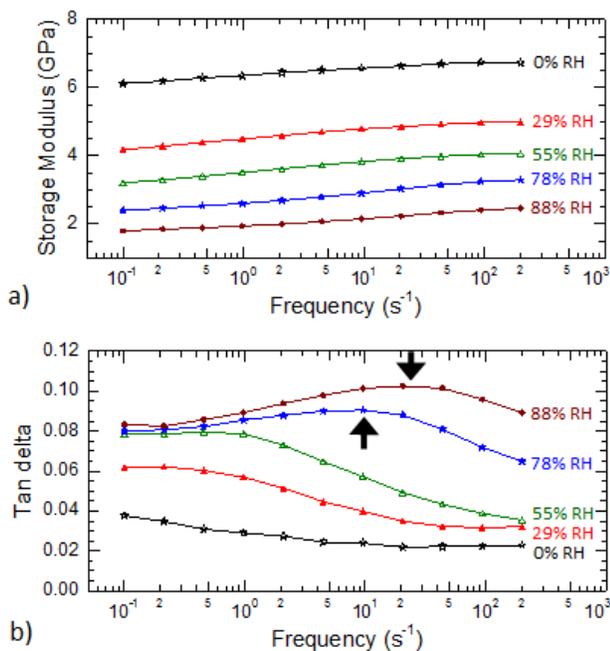


Figure 1 – Nanoindentation-based mechanical spectroscopy of S2 secondary wood cell wall layers in latewood loblolly pine. a) Storage modulus and b) tan delta as a function of frequency and humidity.

Wood possesses hierarchy of structure ranging from individual wood polymers to cells to growth rings. The development of new forest products is hindered by the lack of fundamental understanding of how molecular-scale modifications affect properties of bulk wood and wood composites. Nanoindentation-based mechanical spectroscopy is well suited to probe the individual cell wall layers and provide new information about how modifications of wood polymers affect bulk wood properties. Results will be presented from BNC and dynamic nanoindentation performed on the S2 secondary wood cell walls, which are anisotropic composites consisting of semicrystalline cellulose microfibrils embedded in a matrix of hemicelluloses and lignin. Experiments were performed in multiple orientations to study anisotropic effects and over a wide range of humidity conditions. A sampling of the dynamic nanoindentation results are shown in Figure 1. The experiments were performed in the S2 secondary cell wall layer in latewood loblolly pine on a longitudinal plane, which means the stiff cellulose microfibrils were oriented perpendicular to the indentation direction. Storage modulus (Figure 1a) as a function of frequency and humidity showed over a factor of three decrease from 0% to 88% RH. Interestingly, tan delta (Figure 1b) as a function of frequency and humidity showed a peak (arrows) in value at approximately 10 s⁻¹ in the experiments at 78% RH and 25 s⁻¹ at 88% RH. These peaks were attributed to a moisture-induced glass

transition in the hemicelluloses of the S2 secondary wood cell wall. The moisture-induced glass transition in hemicelluloses' is of particular interest because it is hypothesized that percolated networks of softened hemicelluloses create diffusion channels for ion transport through wood cell walls. Because the fungal decay of wood requires ion transport through wood cell walls, a chemical treatment that prevents the hemicelluloses' glass transition may also be an effective non-toxic wood treatment to protect wood products against decay. Nanoindentation-based mechanical spectroscopy can now be used as a tool in wood science research to study the effects of chemical modifications on the moisture-induced glass transition in hemicelluloses.