

Nano-Mechanical Properties of the Wood Cell Walls Influenced by Species and Process

2007 IUFRO All Division 5 Conference, Taipei, Taiwan, Oct. 29 –Nov. 2, 2007

Siqun Wang, Yan Wu, Cheng Xing, George M. Pharr

University of Tennessee, United States

Dingguo Zhou, Yang Zhang
Nanjing Forestry University, China



Introductions

Siyan Wang

- The structural performance of any composite composed of discontinuous fibers is based on three variables: physical and mechanical properties of individual wood fibers, fiber-to-fiber stress transfer, and fiber orientation.
- The paper and fiber boards are made of individual wood fiber or fiber bundle.

Introductions

Siyun Wang

- Wood-plastic composite is a very promising material to achieve durability without using toxic chemicals.



Deck and fence



Pool and docks

- In wood plastic composites, wood is even used as a form of cell wall component.

Introductions

- The wood filler used in current WPCs is generally wood flour (obtained by grinding saw dust and industrial wood waste) that requires high energy consumption.
- In order to compete economically with wood flour, alternative approaches for size reduction of solid wood should be considered which require less energy consumption than grinding:
 - Steam explosion
 - Refining



Introductions

Siyun Wang

To design fiber reinforced polymer composites, we need to know

- Matrix
- Fiber
- Interphase



Research Goals and Materials

Siyun Wang

Objectives:

- ❑ To investigate nano mechanical properties of cellulose fibers by nanoindentation;
- ❑ To investigate effects of species on cell wall properties;
- ❑ To investigate effects of refining steam pressure on cell wall properties.

Research Goals and Materials

Materials:

- Ten hardwood species

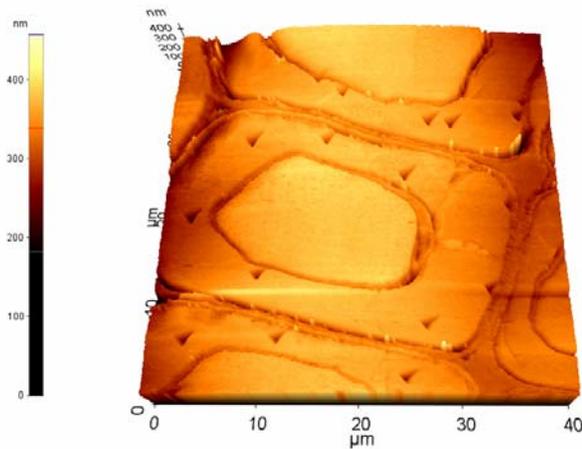
Species	D_o	E_B
	(g/cm ³)	(GPa)
Poplar	0.305	8.1
Manchurian Ash	0.503	12.9
Alder Birch	0.650	12.9
Asian White Birch	0.610	11.2
Red Oak	0.680	12.6
White Oak	0.650	12.3
Mongolian Oak	0.679	13.2
Iroko	0.706	9.4
Kwila	0.839	16.0
KerANJI	1.135	21.1
Maximum	1.135	10
Minimum	0.305	19.2
Mean	0.630	13.02
P (%)	73.1	47.9

E_B : Bending elastic modulus from references (Alden 1995; Cheng et al. 1992)

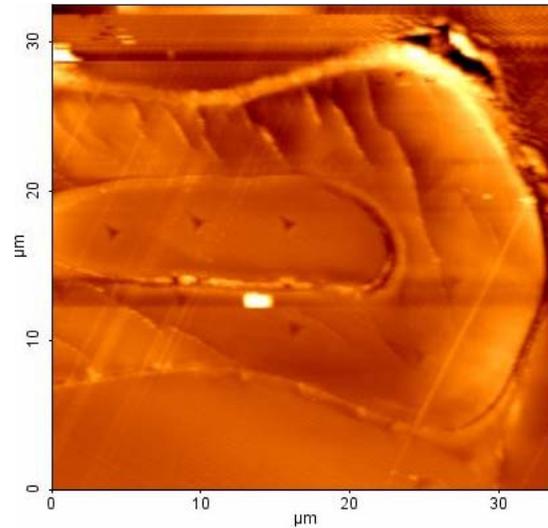
Research Goals and Materials

Materials:

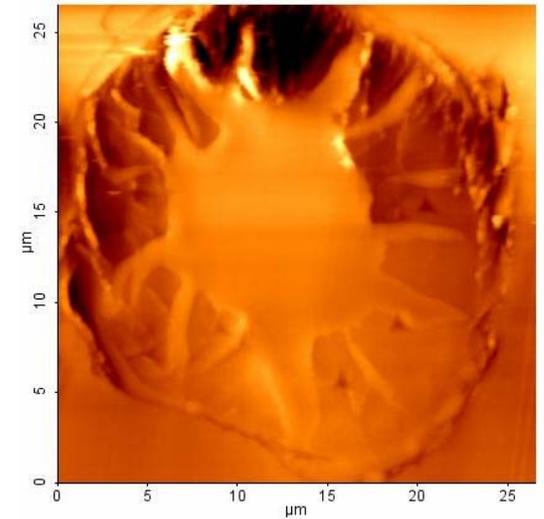
- Refined Loblolly pine wood fibers under different refining steam pressures (2-18 bar)



2 Bars



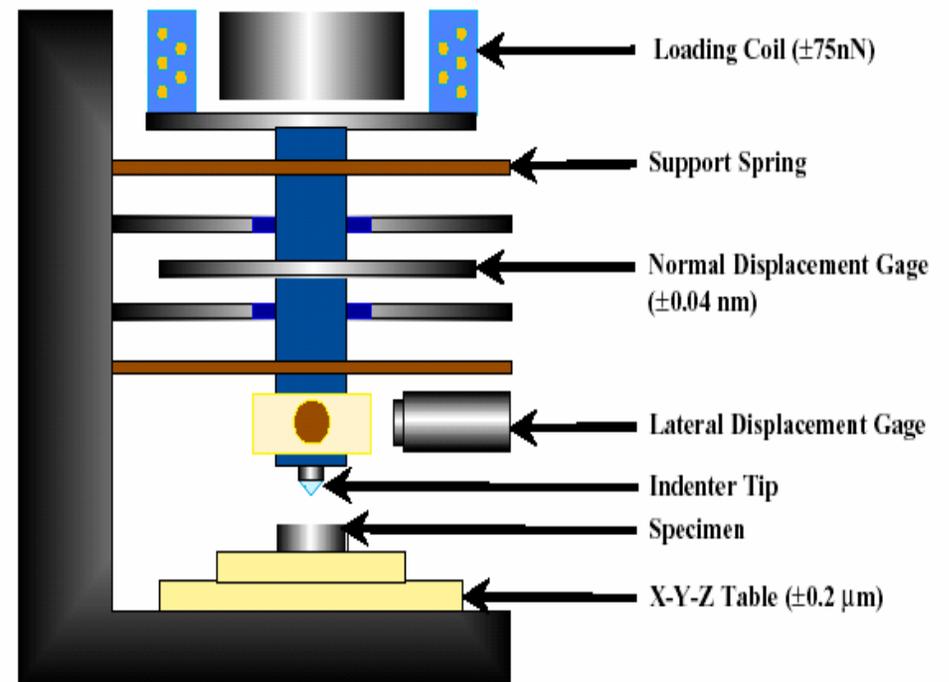
12 Bars



18 Bars

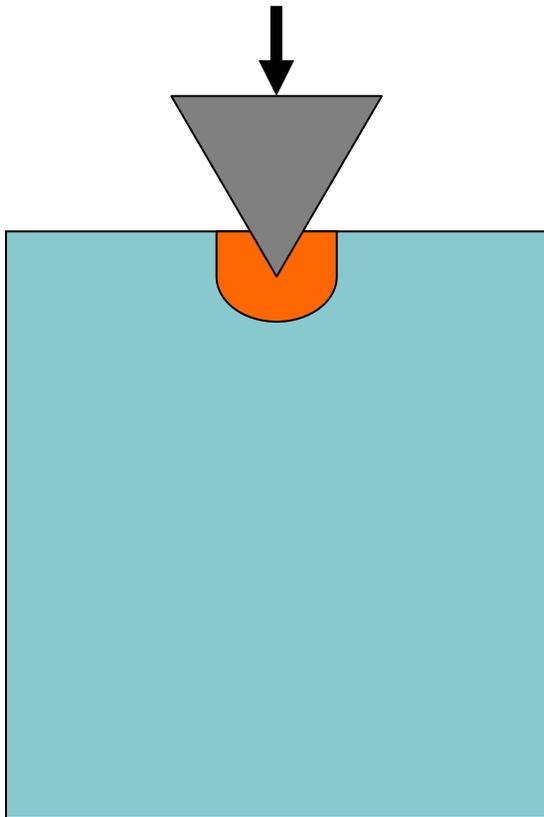
Experimental Method

- Ten hardwoods:
 - Nanoindentation (hardness, elastic modulus)
 - SilviScan (elastic modulus, microfibril angle, density)
 - AFM
- Refined wood fibers under different refining steam pressure:
 - Nanoindentation (hardness, elastic modulus, creep)
 - AFM

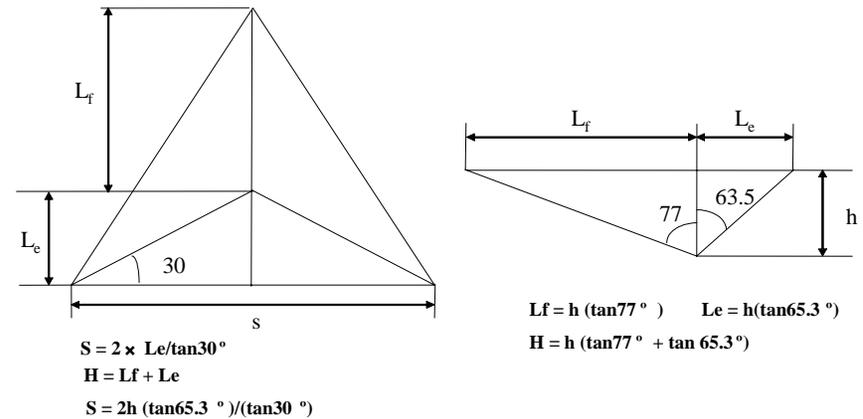


Schematic of the NANO II Indenter

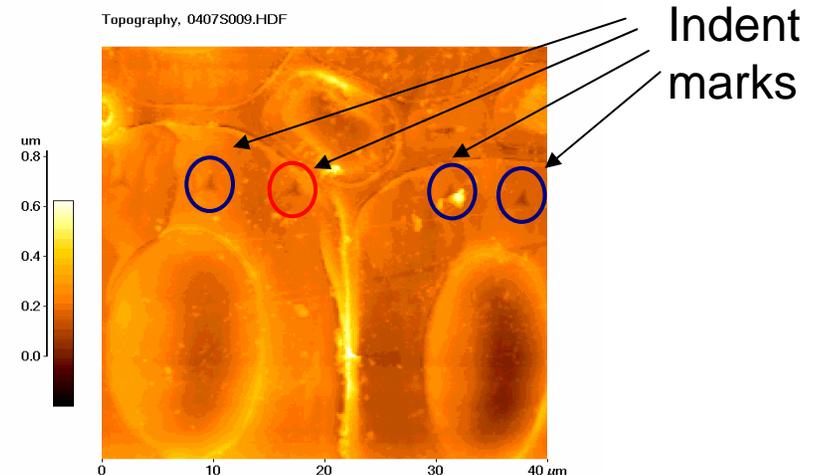
Nanoindentation Instrument and Indentation Procedure



Schematic of the NANO II Indenter



Geometry of nano-indenter (Berkovich diamond tip)



Nanoindentation Instrument and Indentation Procedure

Hardness (H):

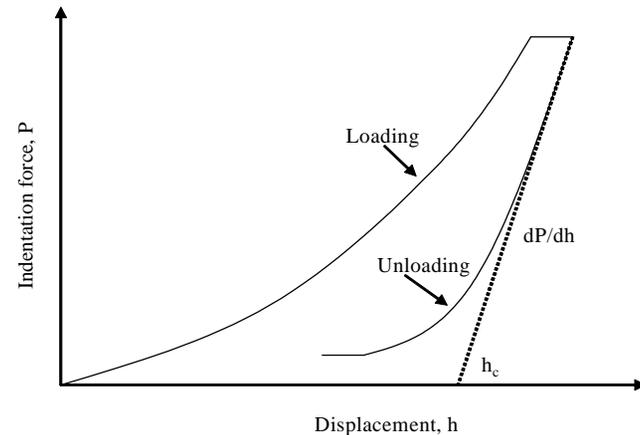
$$H = \frac{P_{\max}}{A} = \frac{P}{24.5h_c^2}$$

Elastic modulus (E_s):

(Oliver and Pharr)

$$E_r = \frac{dP}{dh} \frac{1}{2} \frac{\sqrt{\pi}}{\sqrt{A}}$$

$$E_s = (1 - \nu_s^2) \left(\frac{1}{E_r} - \frac{1 - \nu_i^2}{E_i} \right)^{-1}$$



Typical load-displacement curve

E_r is reduced elastic modulus, which accounts for the fact that elastic deformation occurs in both the sample and the indenter.

ν_s and ν_i (0.07) are the Poisson's ratios of the specimen and indenter, respectively.
 E_i is the modulus of the indenter (1141 GPa).

Results – Ten hardwood species

Siyun Wang

Wood properties of ten hardwoods

Note:

D_O : oven-dry density,

D_S : SilviScan density,

E_N : elastic modulus from nanoindentation,

E_S : elastic modulus from SilviScan,

E_B : Bending elastic modulus from references (Alden 1995;

Cheng et al. 1992),

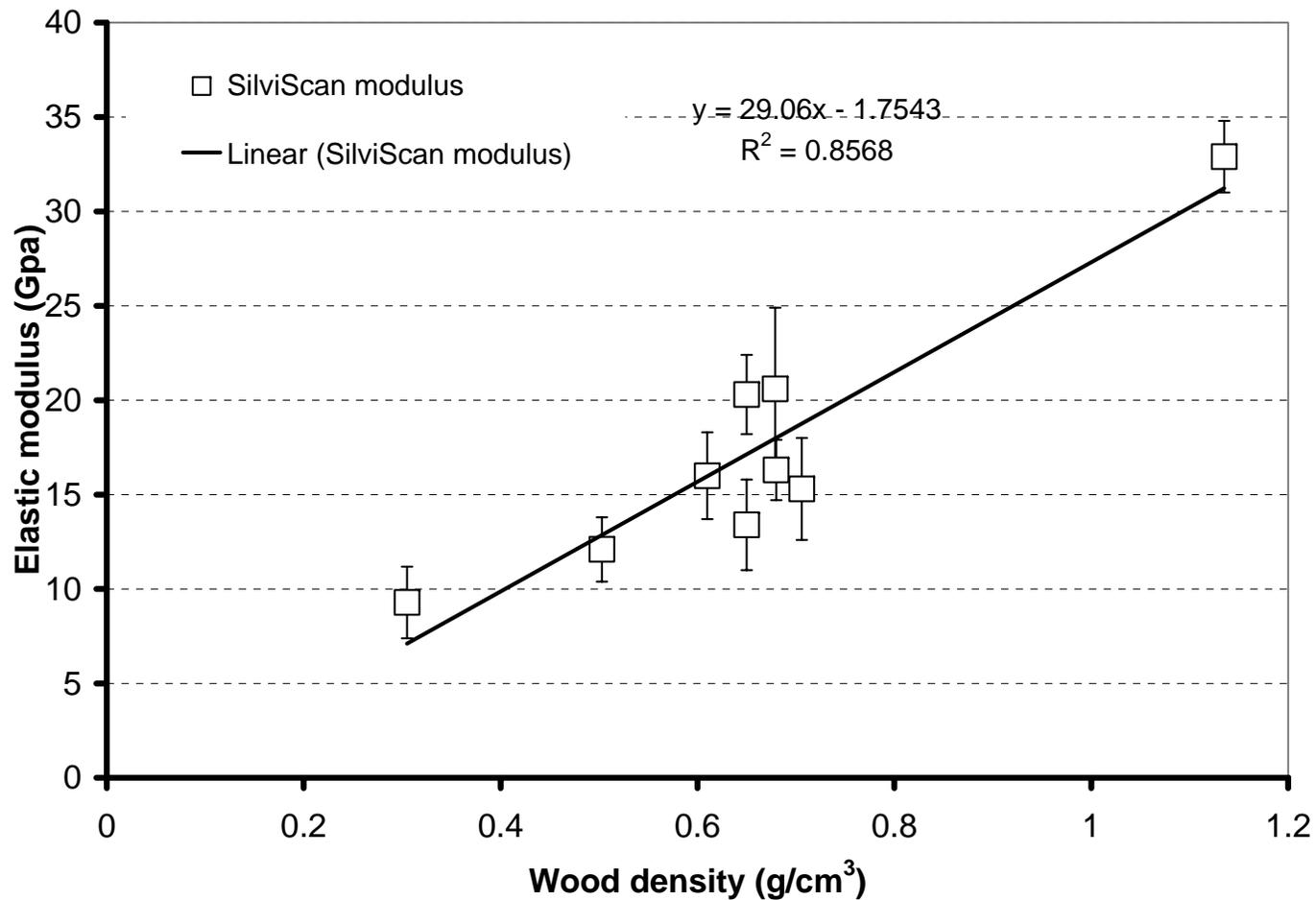
H: hardness,

MFA: microfibril angle.

Species	D_O	D_S	E_N	E_S	E_B	H	MFA
	(g/cm ³)	(g/cm ³)	(GPa)	(GPa)	(GPa)	(GPa)	(degree)
Poplar	0.305	0.409	16.9 (1.9)	9.29 (1.9)	8.1	0.49 (0.047)	18.1 (1.69)
Manchurian Ash	0.503	0.584	18.5 (1.9)	12.1 (1.7)	12.9	0.48 (0.048)	12.7 (0.62)
Alder Birch	0.650	0.760	19.7 (1.1)	20.3 (2.1)	12.9	0.49 (0.032)	12.9 (1.42)
Asian White Birch	0.610	0.700	17.5 (2.1)	16.0 (2.3)	11.2	0.45 (0.033)	13.4 (1.33)
Red Oak	0.680	0.718	22.6 (1.5)	16.3 (1.6)	12.6	0.55 (0.037)	10.8 (1.08)
White Oak	0.650	0.730	19.5 (1.8)	13.4 (2.4)	12.3	0.49 (0.028)	15.0 (4.88)
Mongolian Oak	0.679	0.866	18.4 (2.0)	20.6 (4.3)	13.2	0.44 (0.047)	12.0 (0.94)
Iroko	0.706	0.735	22.9 (2.5)	15.3 (1.9)	9.4	0.51 (0.040)	8.75 (1.31)
Kwila	0.839	0.902	21.2 (1.5)	24.3 (2.7)	16.0	0.56 (0.031)	4.17 (4.36)
Keranji	1.135	1.177	24.6 (2.0)	32.9 (7.4)	21.1	0.54 (0.022)	6.30 (1.48)
Maximum	1.135	1.177	24.6 (2.0)	32.9 (7.4)	10	0.56 (0.031)	18.1 (1.69)
Minimum	0.305	0.409	16.9 (1.9)	9.29 (1.9)	19.2	0.40 (0.028)	4.17 (4.36)
Mean	0.630	0.758	20.2 (1.8)	18.0 (2.8)	13.02	0.49 (0.037)	11.4 (1.91)
P (%)	73.1	65.2	31.3	71.8	47.9	28.6	77

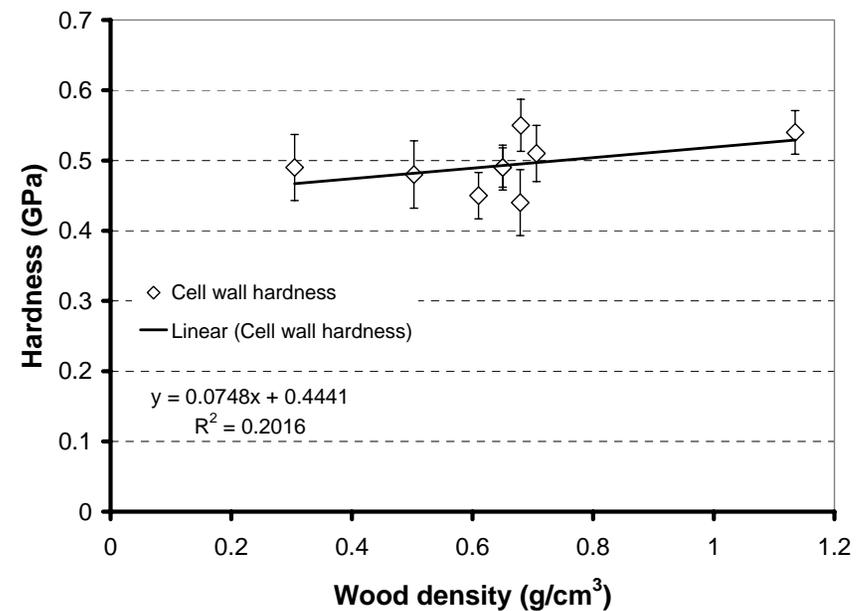
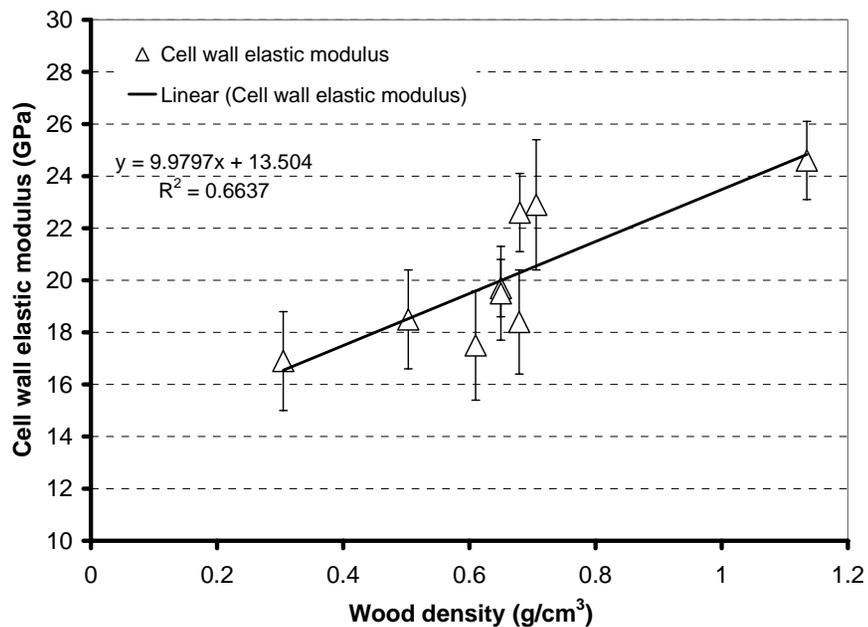
Results – Ten hardwood species

SilviScan elastic modulus as affected by wood density



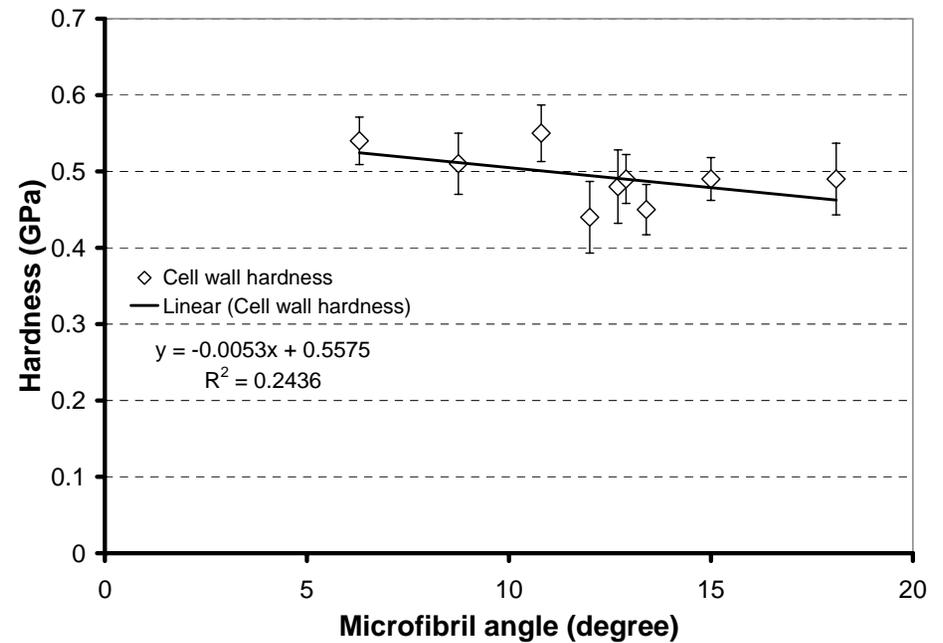
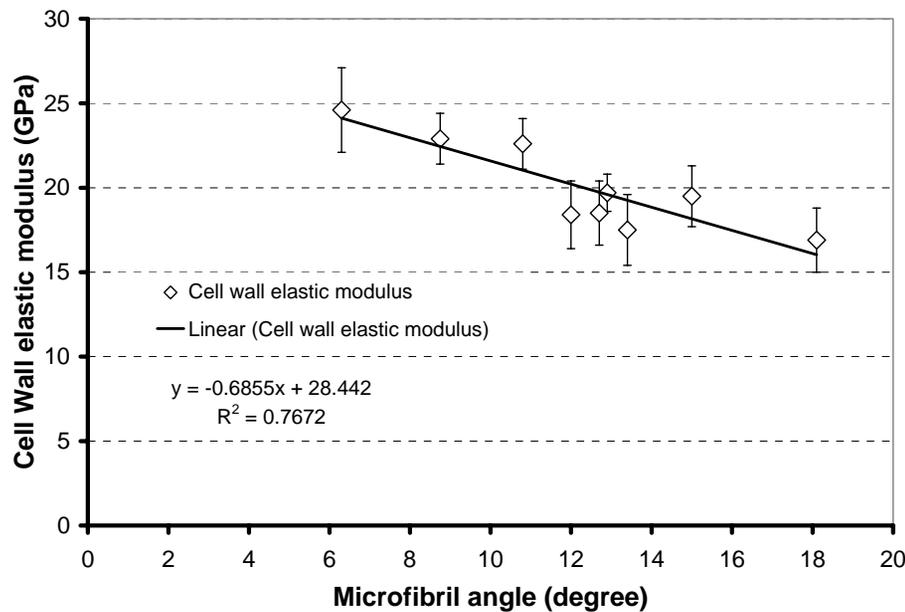
Results – Ten hardwood species

Nanoindentation cell wall elastic modulus and hardness as affected by wood density

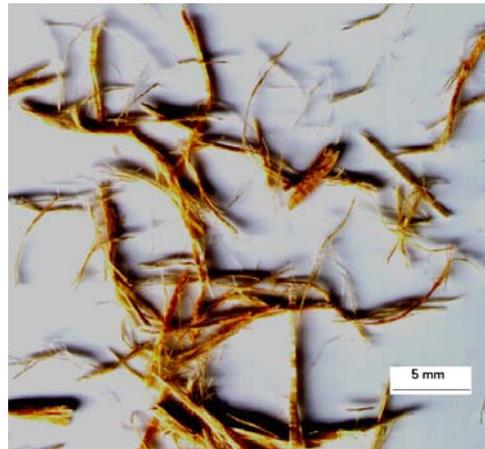


Results – Ten hardwood species

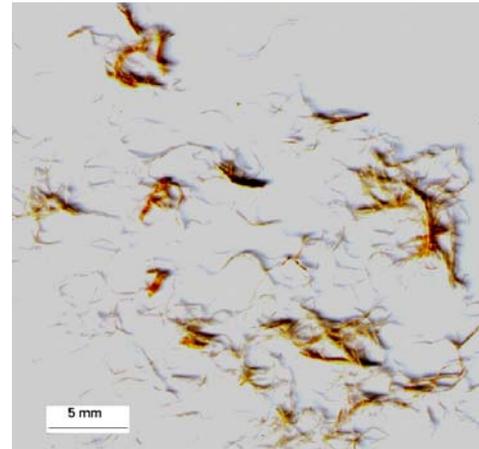
Cell wall elastic modulus and hardness as affected by microfibril angle (MFA)



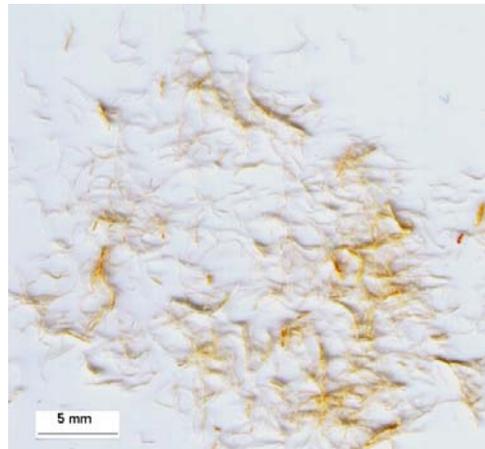
Results – Refined wood fibers



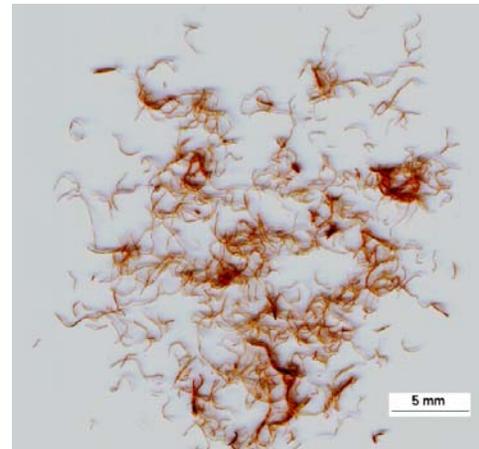
a



b



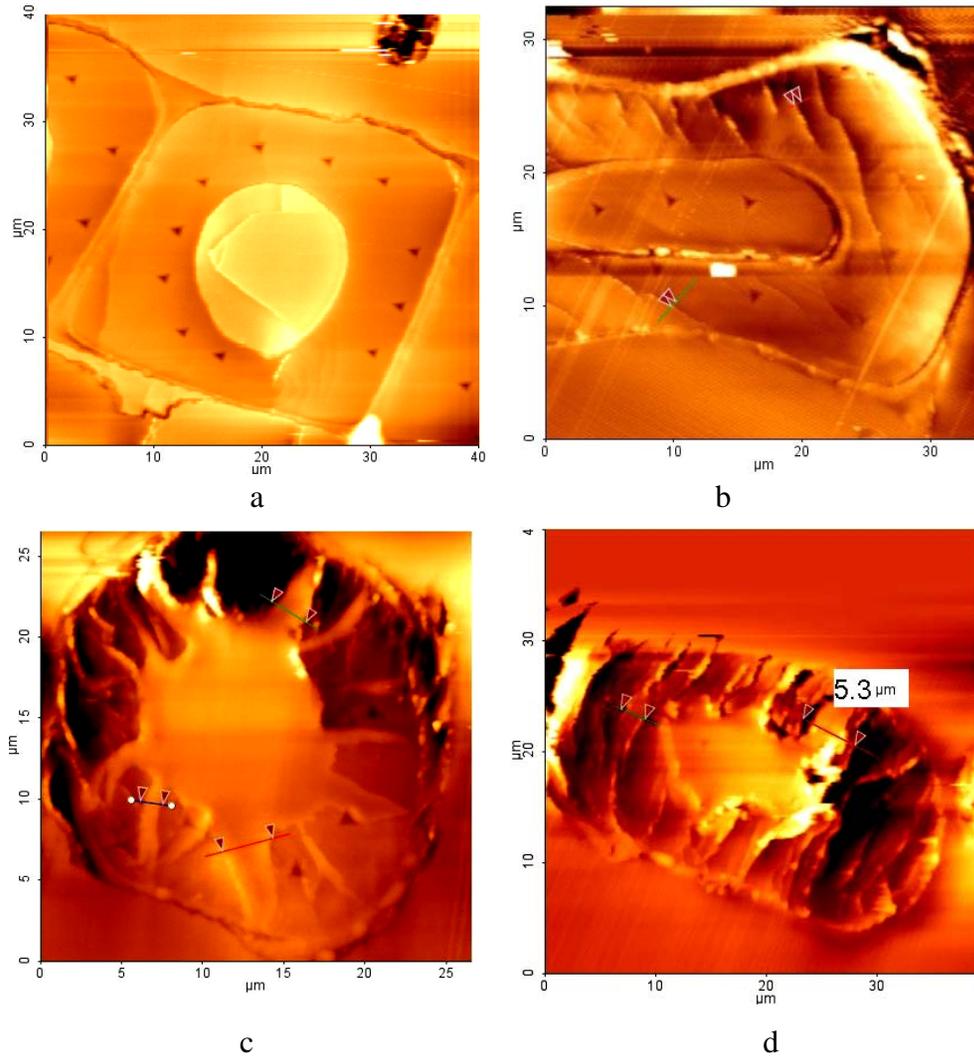
c



d

Refined fibers from different pressure (a: 4 bar, b: 6 bar, c:8 bar, d:14 bar)

Results – Refined wood fibers



Damages in refined fiber cell wall cross sections (a: 2 bar, b: 8 bar, c: 14 bar, d: 18 bar)

Results – Refined wood fibers

Summary of nanoindentation results of refined fiber cell wall

Property/pressure		2 bar	4 bar	6 bar	8 bar	10 bar	12 bar	14 bar	18 bar
Es GPa	Mean	21.35	18.62	15.96	16.83	15.32	14.05	13.09	12.22
	Stdev	2.59	2.97	2.41	2.53	2.51	2.87	3.42	3.29
	CV	12.13	15.95	15.10	15.03	16.38	20.43	26.13	26.92
H GPa	Mean	0.50	0.47	0.47	0.45	0.43	0.43	0.39	0.37
	Stdev	0.04	0.062	0.07	0.05	0.067	0.079	0.078	0.095
	CV	8.00	13.19	14.89	11.11	15.58	18.37	20.00	25.68
Ci %	Mean	7.58	8.72	8.87	8.63	8.24	9.68	12.30	13.08
	Stdev	0.86	1.56	1.25	1.29	1.09	1.79	3.89	3.91
	CV	11.35	17.89	14.09	14.95	13.23	18.49	29.25	29.89
n	Number	31	27	23	28	30	28	14	13

Note: Es: elastic modulus; H: hardness; Stdev: standard deviation; CV: coefficients of variation; Ci: indention creeps; n: the number of indents.

Summary

- ❖ At the cell wall level, the elastic modulus and hardness obtained by nanoindentation were more referable to the properties of natural fibers (cells). The cell wall elastic modulus also increased with wood bulk density and decreased with MFA. Hardness showed an increasing tendency with wood density and a decreasing tendency with MFA, but the trend was not significant as elastic modulus.
- ❖ The physical and mechanical properties of refined wood fiber cross section can successfully be investigated by nanoindentation and AFM techniques.
- ❖ The nano-mechanical properties of refined fibers decreased with refining pressure. The nanoindentation creep in fibers subjected to higher pressures were more obvious than those occurring in fibers subjected to lower pressures.

Acknowledgements

Siyun Wang

- USDA NRI grant number # 2005-02645
- USDA Wood Utilization Research Grant
- Tennessee Agricultural Experiment Station MS#96
- Oak Ridge National Laboratory
- Drs. Les Groom, John Dulap, Maurice Defo

