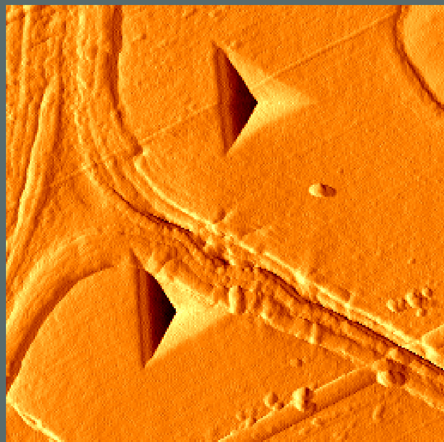
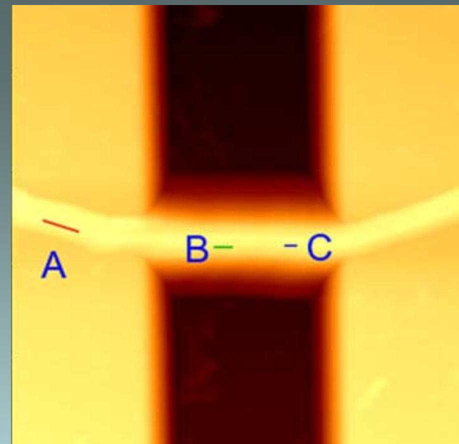




Young's Modulus of Cellulose Fibrils Measured Using Atomic Force Microscopy

2008 International Conference on Nanotechnology for the Forest Products Industry, June 25-27, 2008, St. Louis, Missouri

Siqun Wang, Qingzheng Cheng

University of Tennessee

Composites and nanocomposites

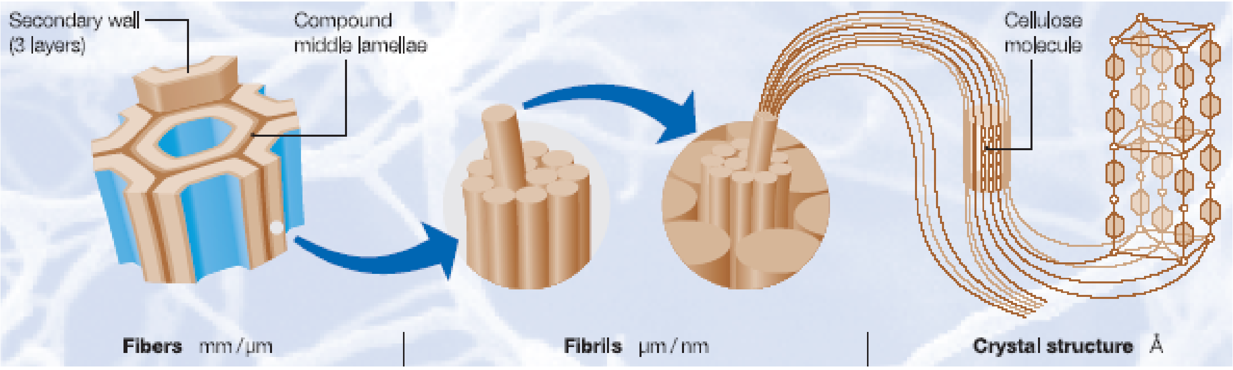
- **The Boeing airplane 787 Dreamliner is a milestone for advanced carbon fiber composites that make up over 50 percent of the materials of the airplane.**

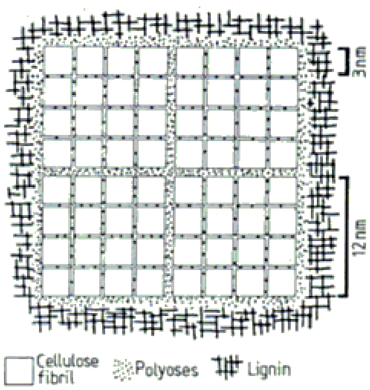
Composites and nanocomposites

To design fiber reinforced polymer composites, we need to know

- Matrix
- Fiber
- Interphase

Cellulose fibrils and nanocrystal



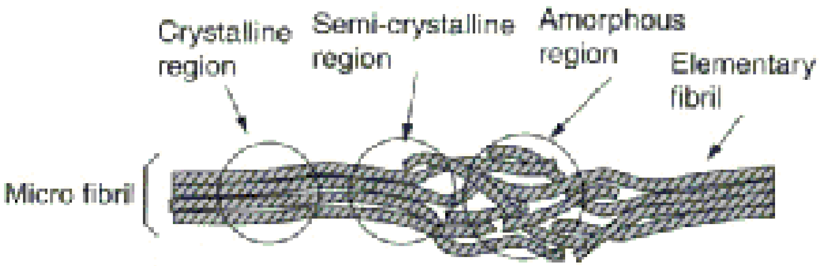


Fiber: One of the thin thread-like parts that from many plant growths, µm-mm;

Fibril: A small, slender parts of fibers, bundles of microfibrils, nm-µm;

Microfibril: Cellulose molecule bundles surrounded with hemicellulose.

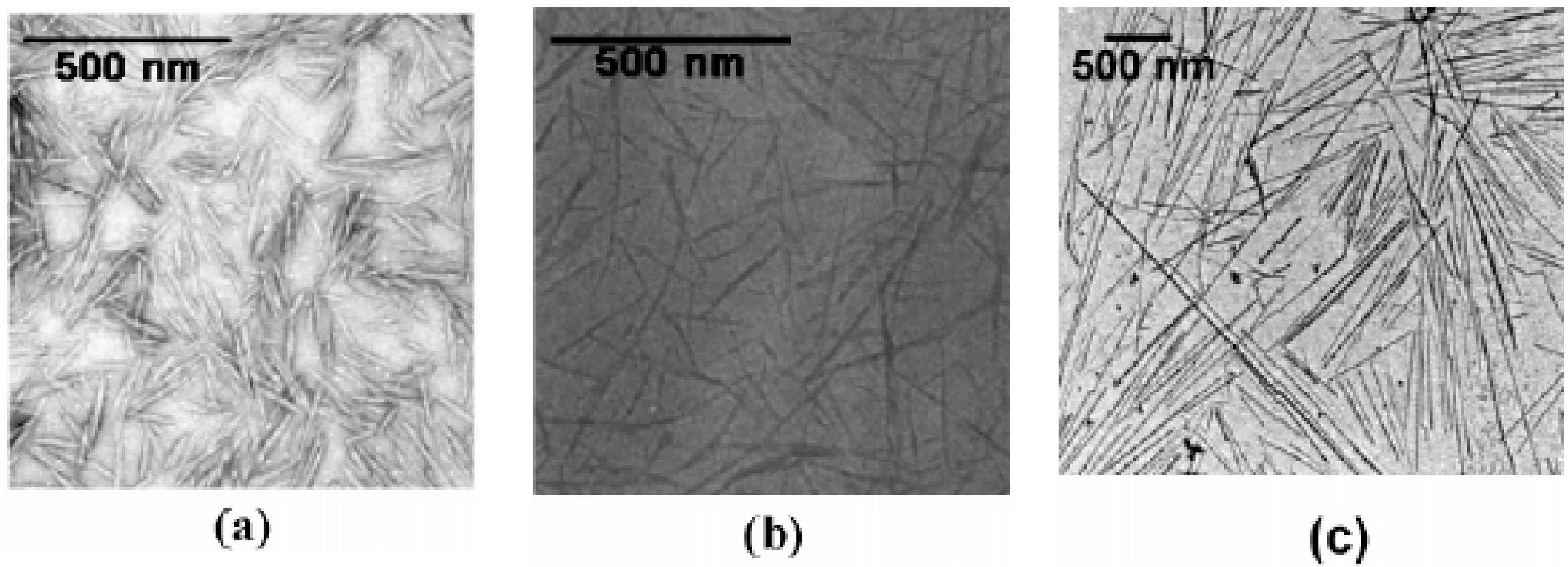
Elementary microfibril: smallest fibril or unit in cell wall, 3-5 nm diameter for wood, also called nanocrystal or whisker.



Source: www.empa.ch/; Fengel, D., and G. Wegener. 1984

Cellulose fibrils and nanocrystal

Chemical treatments: acid hydrolysis to remove the amorphous regions, obtain nanocrystal or whisker.

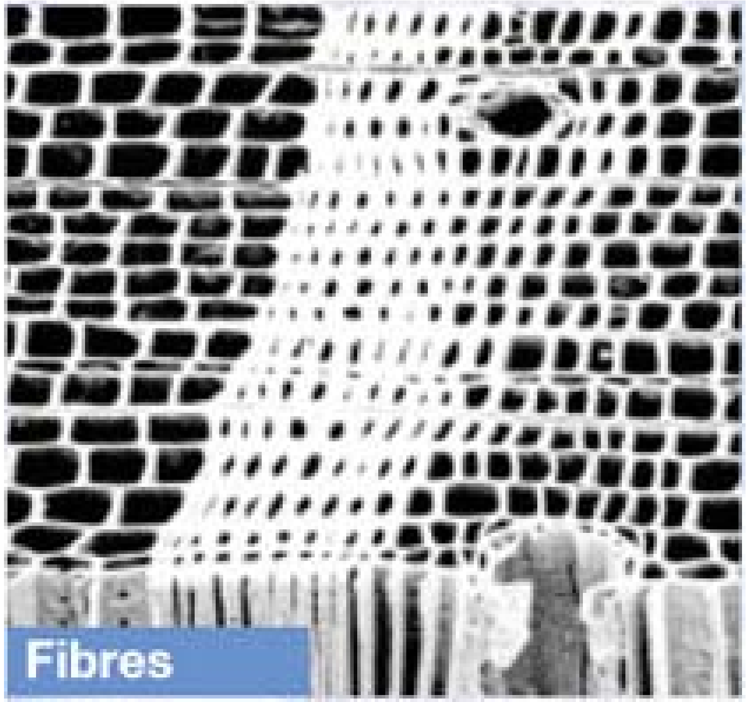


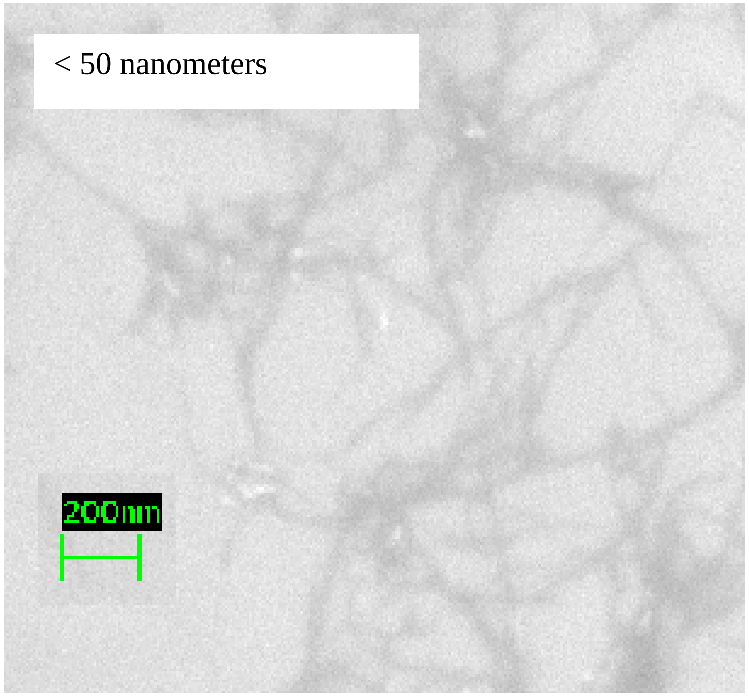
Transmission electron micrograph from a dilute suspension of hydrolyzed (a) cotton, (b) sugar-beet pulp and (c) tunicin.

Source: Samir, Biomacromolecules **2005,** 6, *612-626*

Cellulose fibrils and nanocrystal

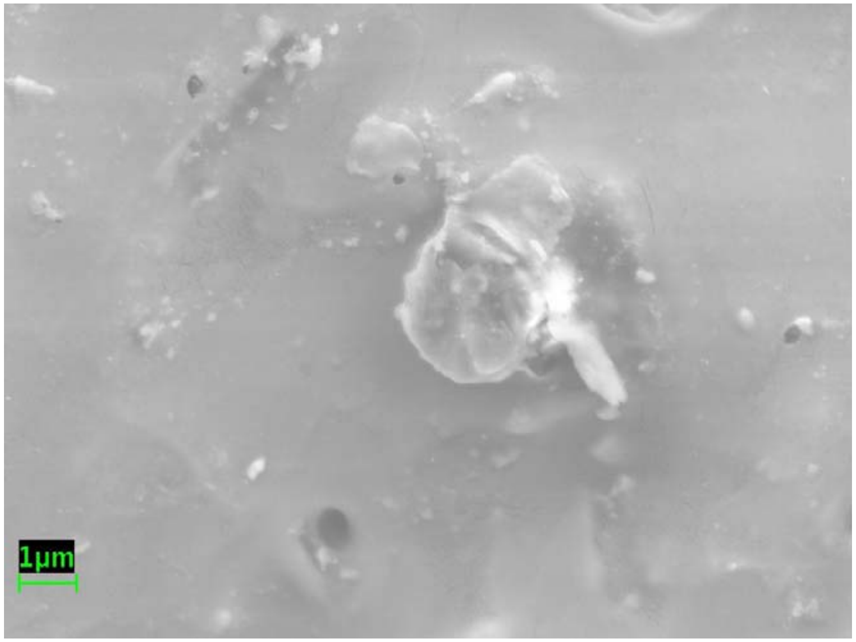
Mechanical treatment: high pressure homogenizer, grinder treatment, fibrils in nano and micro scales.





Cellulose based nanocomposites

- **Fibers:**
  - **Cellulose whisker**
  - **Bacterial cellulose**
  - **Cellulose microfibrils**
  - **Microfibrillated cellulose**
- **Matrix:**
  - **Polypropylene (PP)**
  - **PE**
  - **Poly(lactic acid) (PLA)**
  - **Polyvinyl alcohol (PVA)**

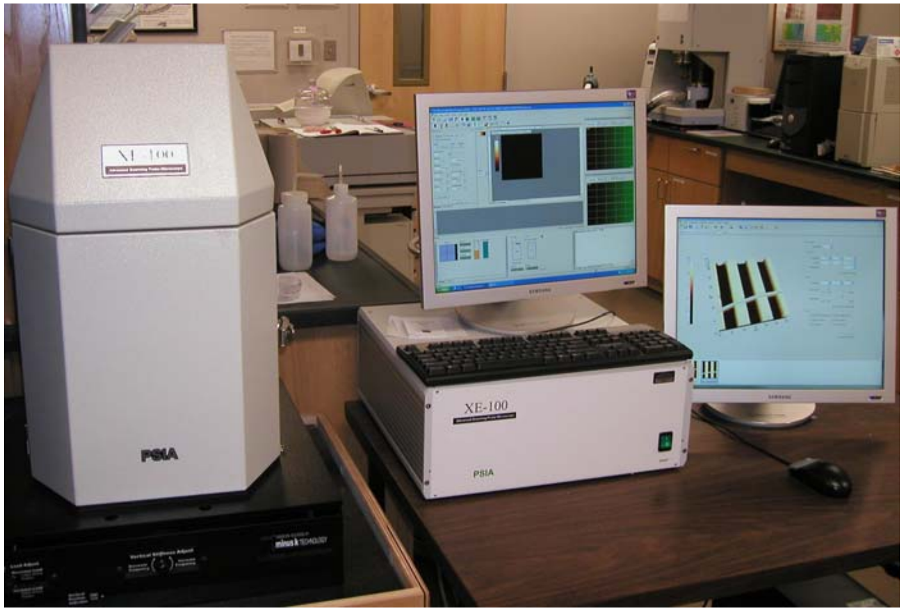


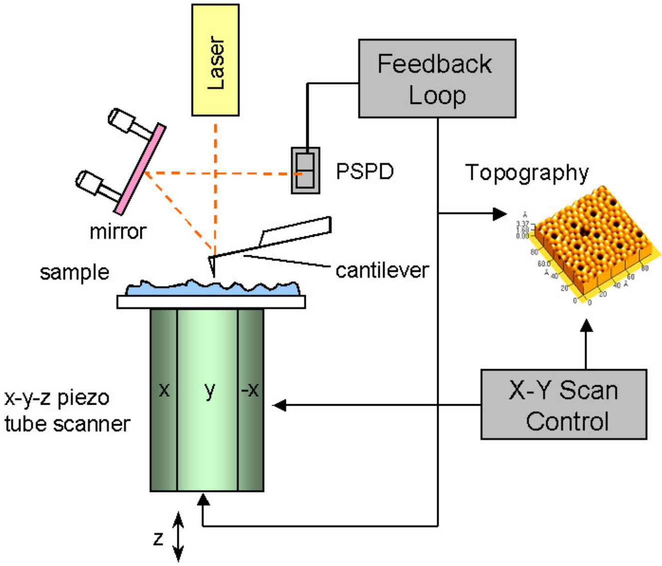
Objectives

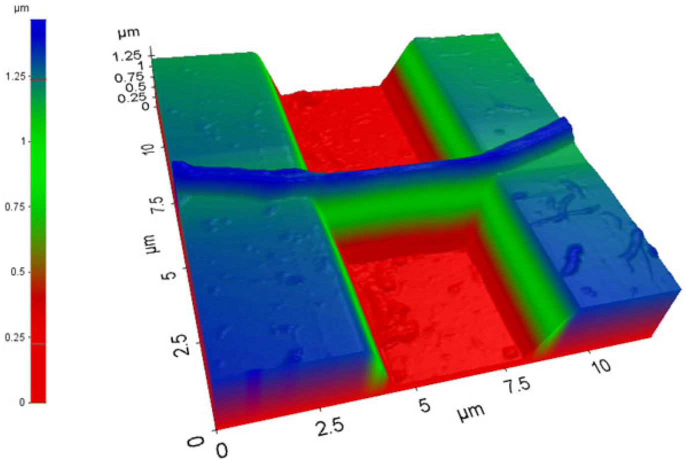
- **Conduct the nano-scale three-point bending testing to measure elastic moduli of individual cellulose fibril**
- **Study some factor that may affect the determination of bending deflections**

Materials and Method

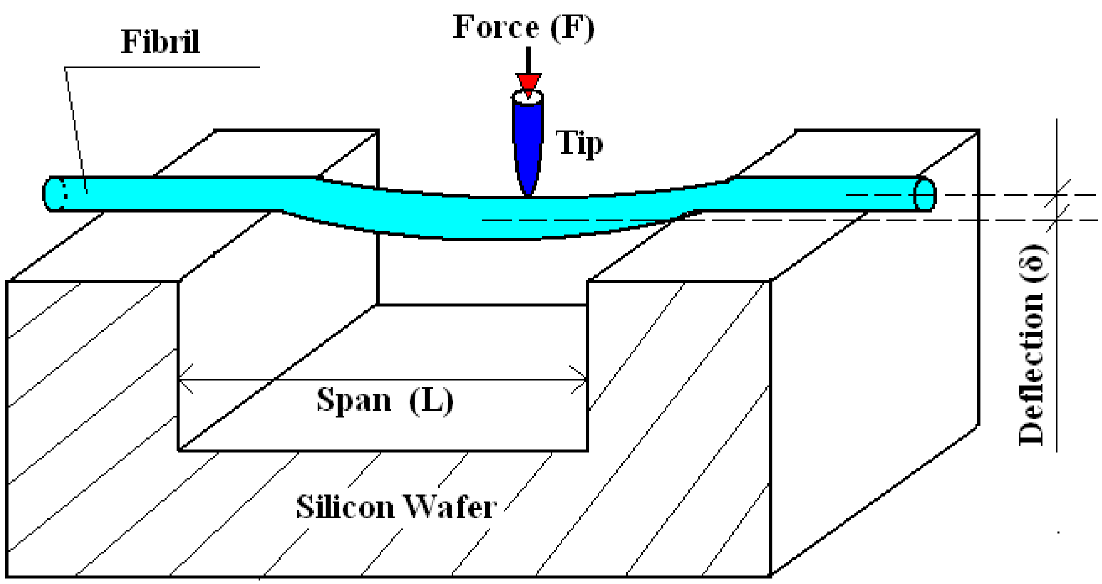
Nano-scale three-point bending in AFM.



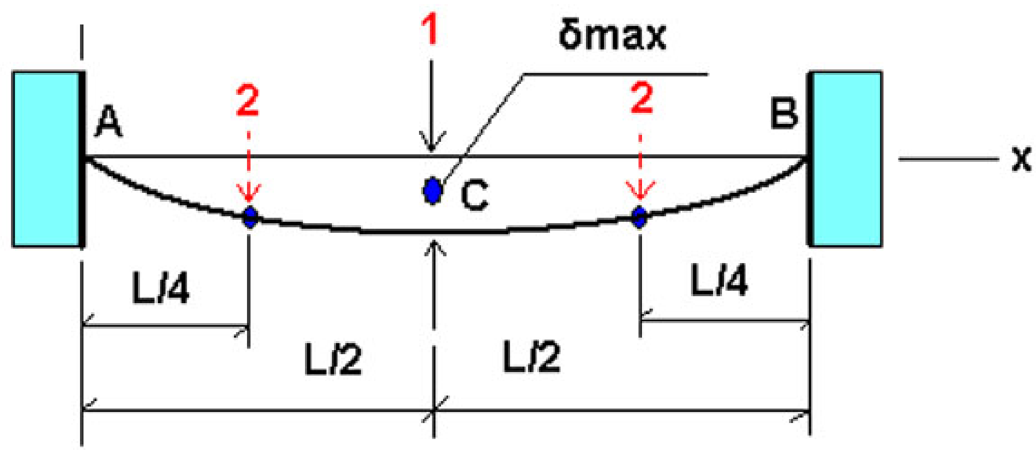




Nano-Scale Three-Point Bending Test in AFM

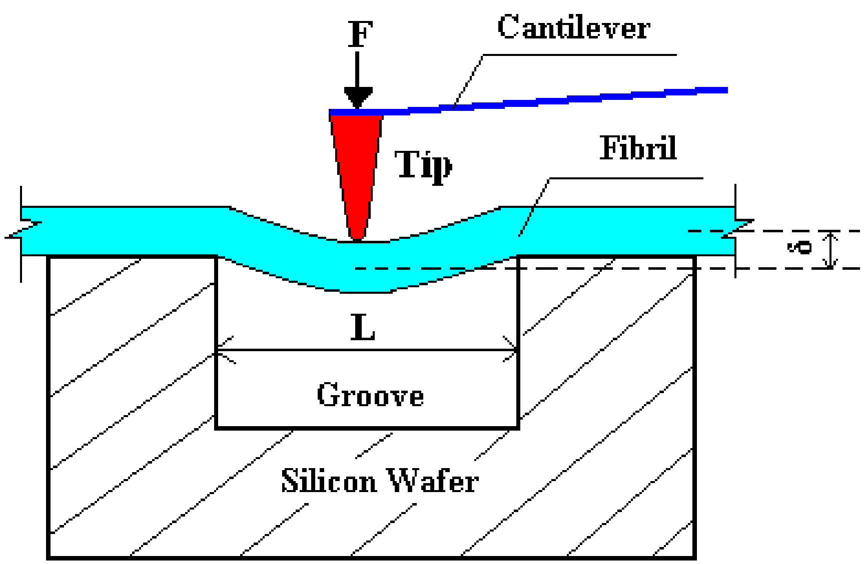


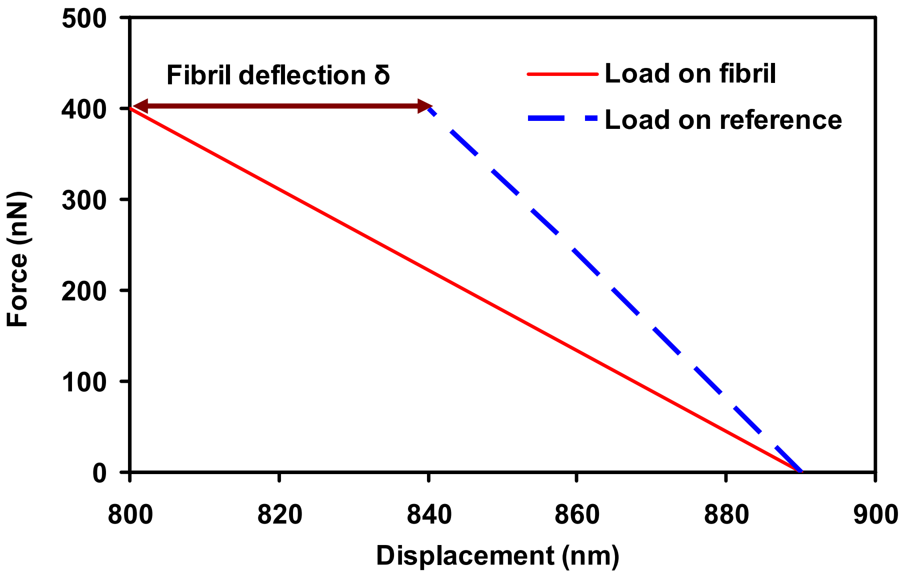
$$E = \frac{FL^3}{192\delta I},$$

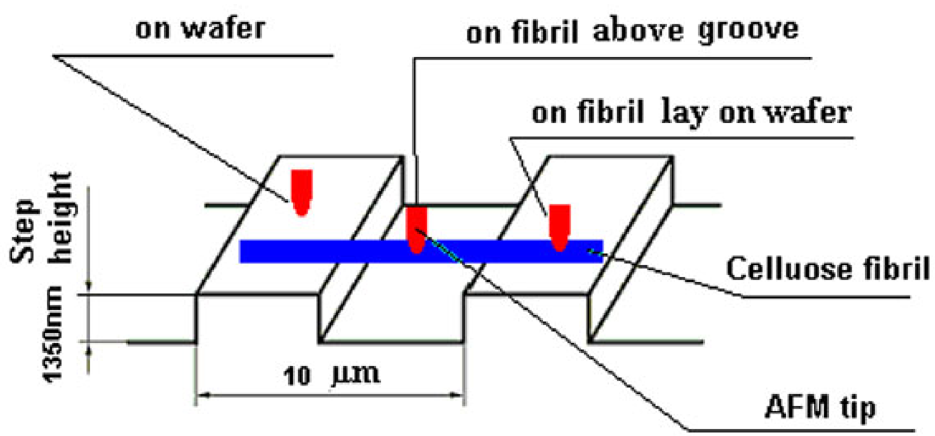


where F is the maximum f
length, δis the deflection o
second moment of area of
the beam diameter.

Fibril Deflection Determination







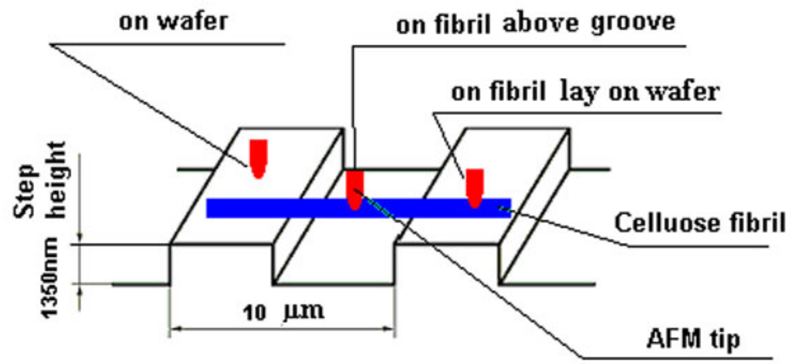
Many factors affect the determination of deflection δ.

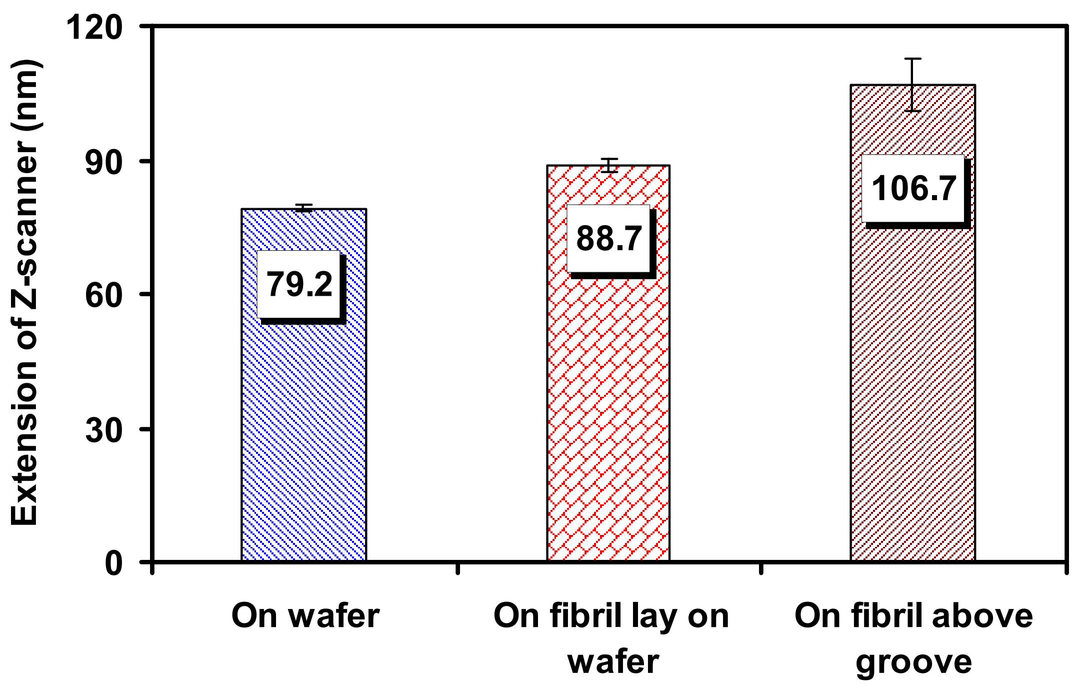
Reference: on wafer, on fibril above wafer, or clean wafer

Fibril deflection determination

To determine δ,

Reference: on wafer, on fibril lay on wafer





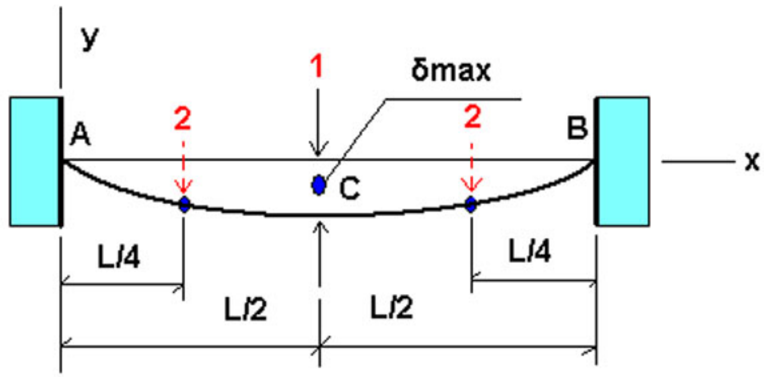
Cantilever spring constant: 2.3 N/m,

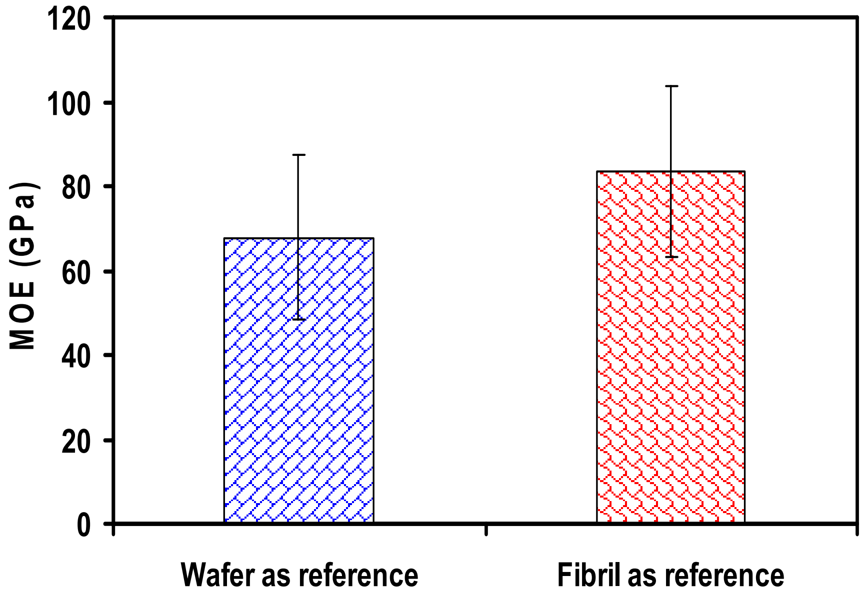
max force: 400nN

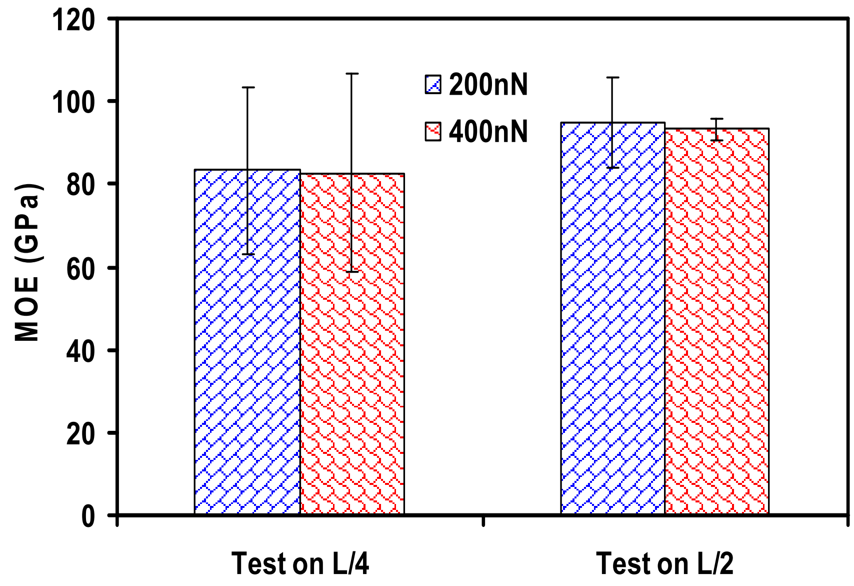
Elastic modulus determination

Affected by "Reference"

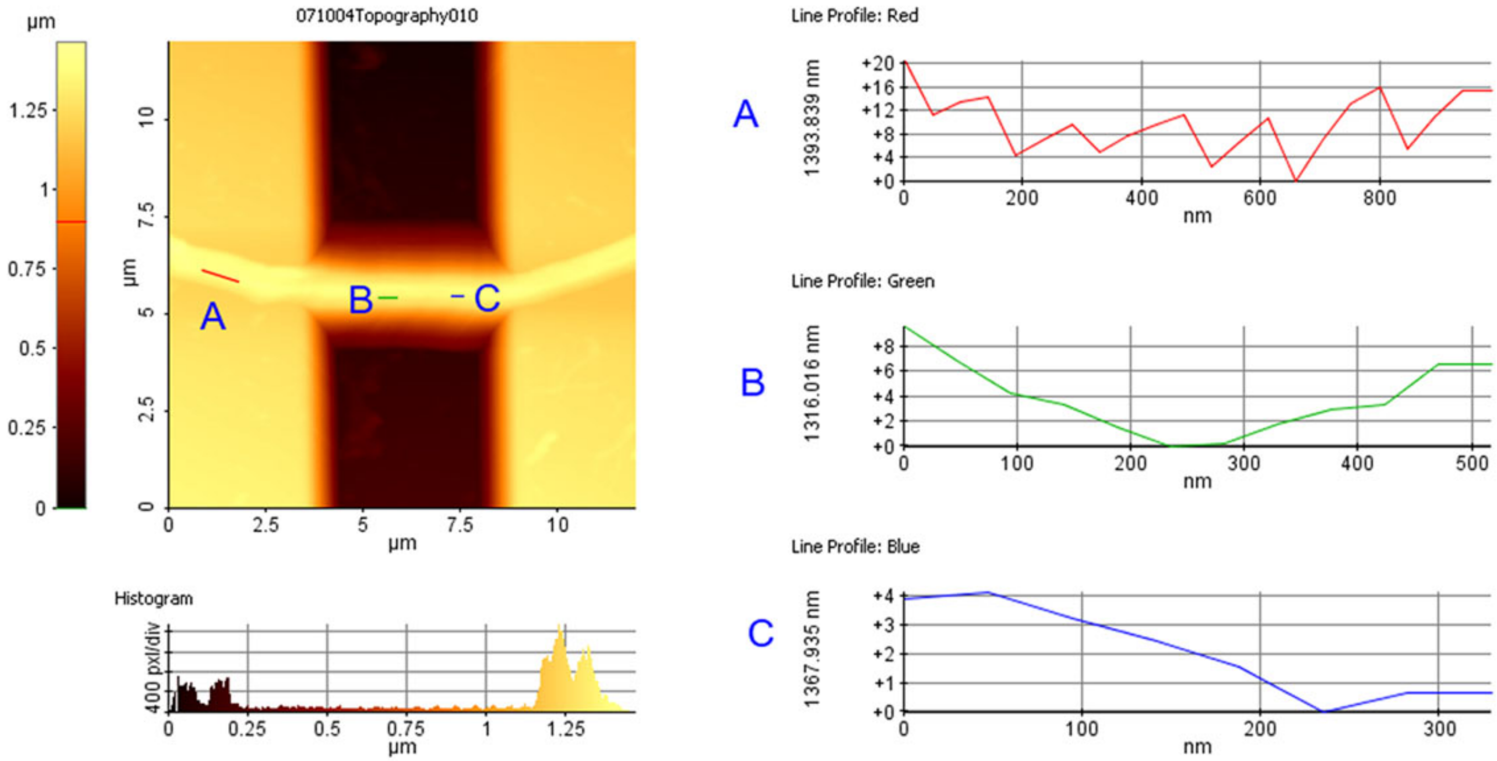
Not much between 200nN & 400nN, as well on L/4 & L/2.







Fibril surface roughness

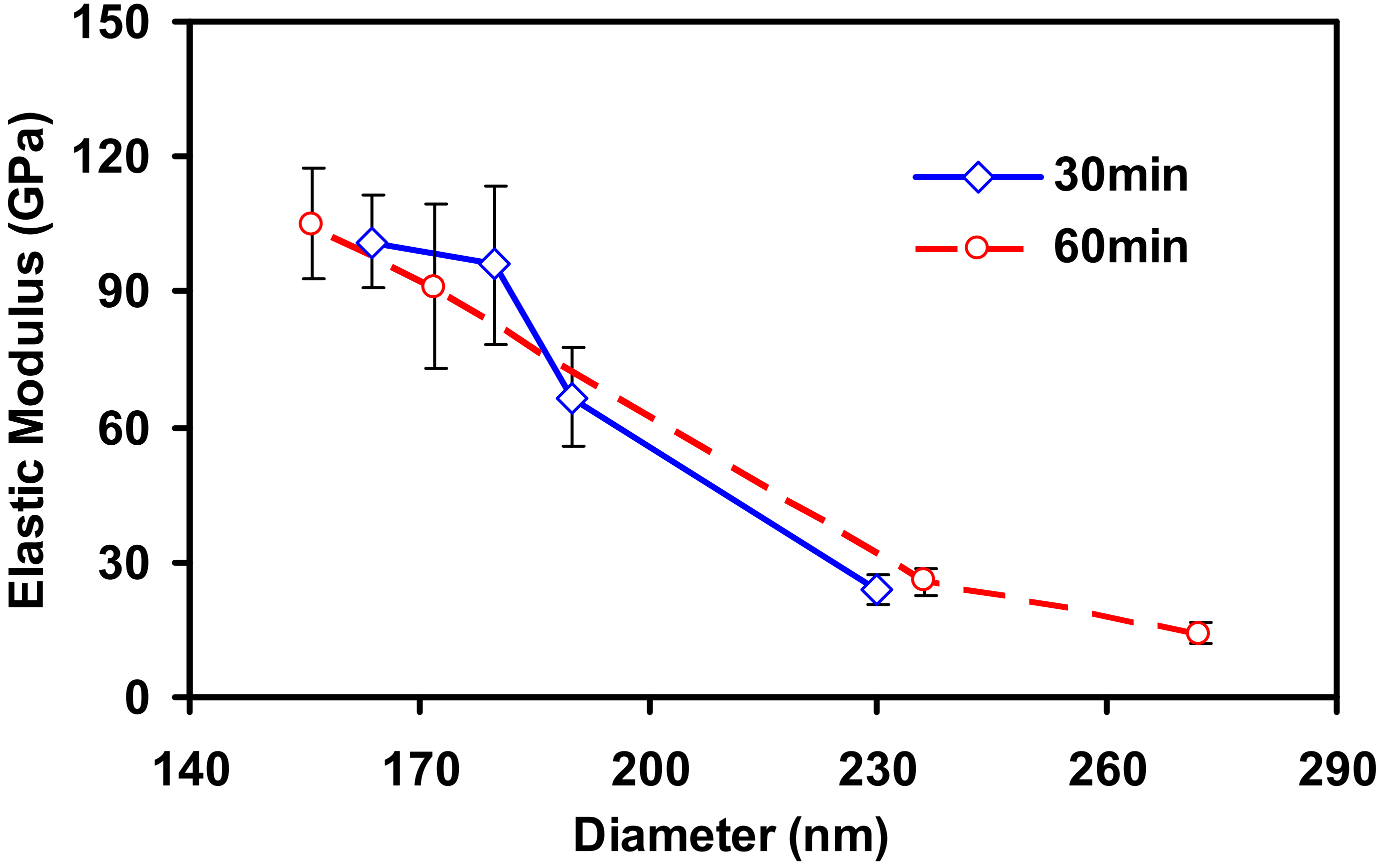


Statistics

Line	Min(µm)	Max(µm)	Mid(µm)	Mean(µm)	Rpv(µm)	Rq(µm)	Ra(µm)	Rz(µm)	Rsk	Rku
Red	1.394	1.414	1.404	1.403	0.020	0.005	0.004	0.000	-0.140	2.696
Green	1.316	1.326	1.321	1.320	0.010	0.003	0.002	0.000	-0.649	2.526
Blue	1.368	1.372	1.370	1.370	0.004	0.001	0.001	0.000	0.216	1.598

Elastic modulus of Lyocell fibrils

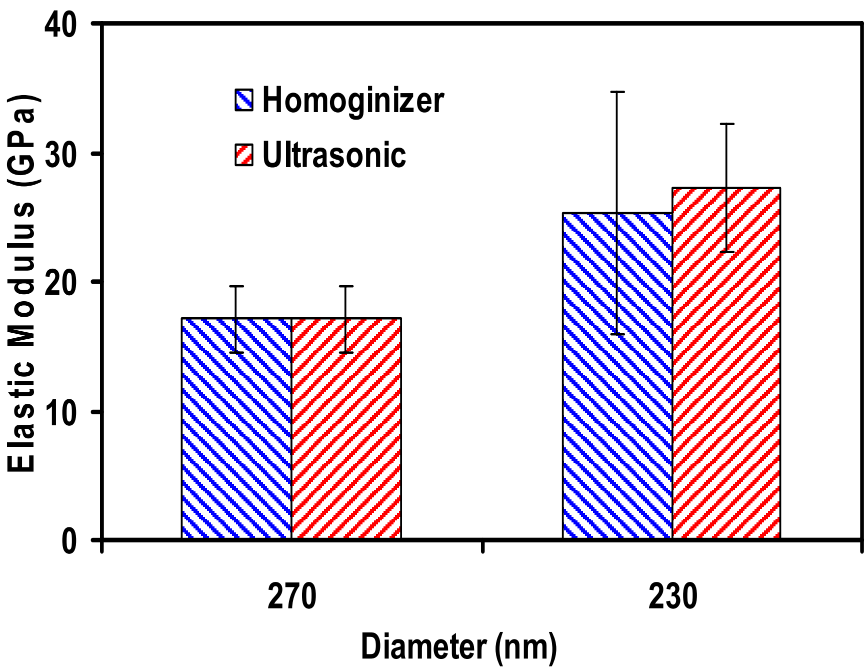
HIUS treated 30 vs 60 min, d>180nm, E dramatically decreased.

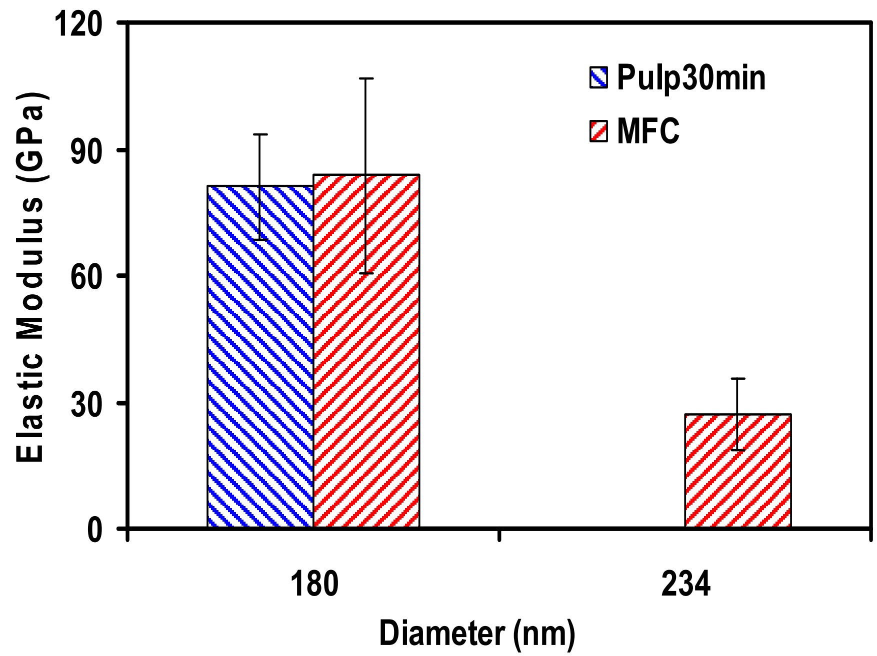


Elastic modulus of different fibrils

HIUS vs Homoginizer: no big difference;

Pulp vs MFC: no big difference.





Summary

- Nano-scale three-point bending test using AFM can be used to measure the elastic modulus of single cellulose fibrils;

- The penetration of AFM tips to the cellulose fibril surfaces need to be considered; the elastic modulus of Lyocell fibrils with d from 150 to 180 nm was evaluated to be 100 GPa, but it decreased dramatically when d > 180 nm.

Acknowledgements

- USDA Wood Utilization Research Grant
- Tennessee Agricultural Experiment Station MS#96 and #83