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

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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 nanocrystalline fibers

**Fiber:** One of the thin thread-like parts that form 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](http); 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

where $F$ is the maximum force applied, $L$ is the suspended length, $\delta$ is the deflection of the beam at midspan, and $I$ is the second moment of area of the beam, where $I = \frac{\pi D^4}{64}$ and $D$ is the beam diameter.

$$E = \frac{FL^3}{192\delta I}.$$
Many factors affect the determination of deflection $\delta$.

Reference: on wafer, on fibril above wafer, or clean wafer
Fibril deflection determination

To determine $\delta$,

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
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.
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.
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