Friction Measurements on Cotton Fiber Bundles and Single Fibers

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Introduction

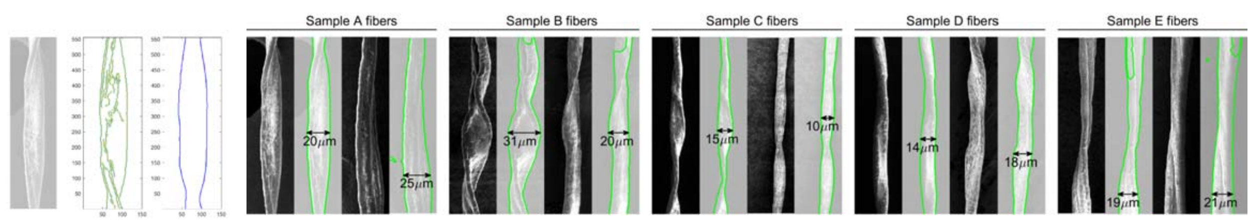
- Motivations for studying mechanical and frictional properties of plant cells at the nanoscale:
  Cell morphogenesis – biomimicry – bioproducts storage/ handling [1][2]

- Atomic Force Microscope (SFM) has been previously employed to study the nanomechanical properties of plant cells cuticular membranes. [3][4][5]
- **The objective of this research was to measure and compare different surface attributes of cotton fibers using the AFM.**
- Those attributes of the surface include surface nanoscale roughness, friction, adhesion, deformation and estimated contact area as obtained from the JKR, the Hertz, and DMT models.
- Our hypothesis was that the surface characteristics of cotton fibers vary between different cotton varieties since the macroscale frictional properties and the cohesion of bulk of cotton fibers vary significantly across different varieties.

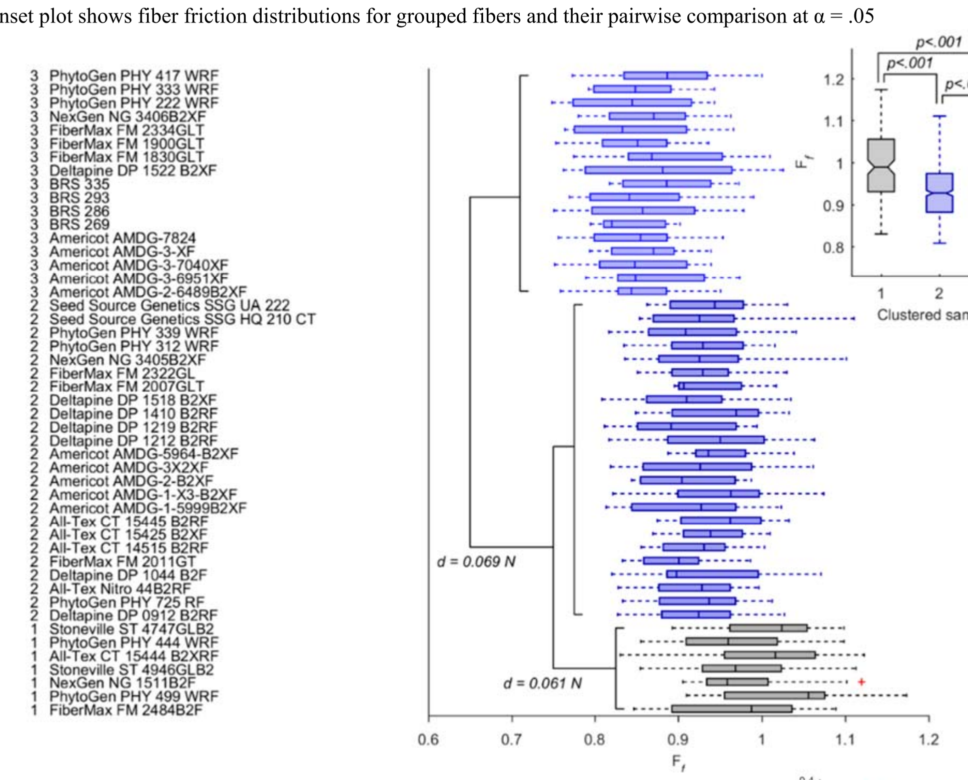
Summary of Our Previous Study on Macroscale Fiber Friction

- **Summary of Experimental**:
- Macroscale friction of 48 cotton varieties were measured using friction apparatus fixture
- Four different normal loads were applied: 5.3, 7.3, 10.2, and 12.2 N

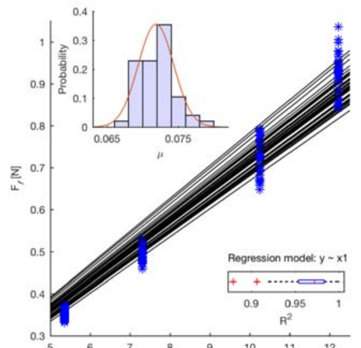
- **Summary of Results**:
- Feature extraction from 2D SEM images with Canny operator (edge detection followed by series of dilutions and erosions)



- Next plot shows distribution of fiber friction–under 12.2 N normal load–for different cotton samples, sorted by mean friction force.
- In this plot, cotton varieties are classified into three groups using arbitrary class boundaries
- The inset plot shows fiber friction distributions for grouped fibers and their pairwise comparison at α = .05



- The following plot shows the mean friction force as a function of normal load
- Thee friction coefficient was measured by fitting the data to the $F_f = \bar{\mu}L$ model.
- The inset plot shows the distribution of friction coefficient



- **Conclusions on macroscale friction:**
- Frictional characteristics of cotton fibers varied significantly across varieties.
- Fiber μ is related to fiber orientation, dimensions, elongation, and yellowness.

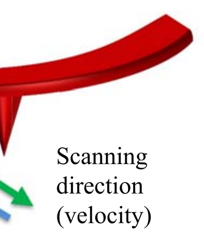
Experimental Procedure

- Different AFM operation modes used in this study are briefly reviewed here.

1) Contact mode:
   - lateral deflection of cantilever indicates the friction between tip and surface
   - Normal load can be controlled by adjusting the vertical deflection of the cantilever

In theory: $F_f = \bar{\tau} K L^{\frac{2}{3}}$

How it is measured: $F_f = \Delta V \times S \times K_t$



   - Measuring friction in newton requires S and k_t to be known:
     S – the cantilever torsional sensitivity in volts/meter k_t – the cantilever torsional spring constant in newton/meter
   - The main disadvantage is the possibility of tip damage

2) Force volume mode:
   - Multiple force-distance curves (FDC) is obtained across the sample surface.
   - FDC can be defined as Plot of net forces acting on AFM tip as a function of z-position of the piezo

3) Force tapping mode (Bruker's PeakForce Tapping® technology):
   - It is a modified version of force-volume mode. In this mode, z-piezo is driven by a sinusoidal wave (instead of a triangular one)
   - The main advantages of this mode are:
     - Indentation force is applied more precisely (possible to test soft samples)
     - Frictional forces have been eliminated (reduces tip damage)
   - Extracting modulus of soft surfaces is challenging (surface roughness and viscoelasticity)
   - The main working principle is as follows (Figure 1):
     - force-time plot is obtained ➔ it will be converted to FDC ➔ Modulus, deformation adhesion, and dissipated energy are extracted from the FDC in real-time

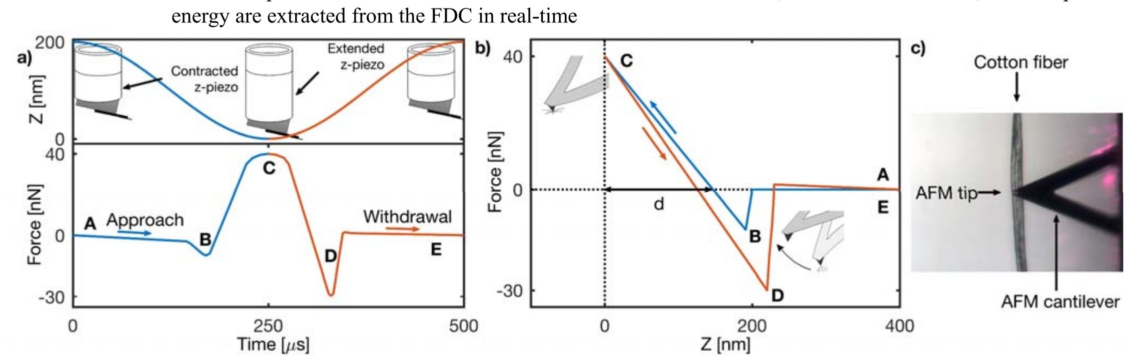


Figure 1. a) Vertical deflection of tip vs. time in a typical Peak Force Tapping mode cycle; b) vertical deflection of tip vs. piezo displacement; A – probe tip above sample surface, B – tip jumps into contact with surface, C – ultimate contact is made and tip indents the surface at a predefined PeakForce setpoint, D – tip-surface separation (adhesion force), E – z-piezo contracts and probe springs back to starting point; c) probe in contact with cotton fiber during scanning (view from built-in camera)

- **Nanoscale friction experiments:**
  - Eight fibers were randomly drawn from bulk of two different samples (Table 1)
  - They were quite distinct in terms of their macroscale friction.
  - They were fixed on glass slide with glue
  - Probe characteristics: R = 40 nm | C = 0.06 N/m | silicon nitride tip
  - Scanning parameters (Figure 2): Scan size = 2 μm × 2 μm | scan speed = 4 μm/s | normal force = 10 - 100 nN
- **Nanomechanical properties experiments:**
  - Deflection sensitivity and spring constant were determined
  - All measurements were carried out in air.
  - Probe characteristics: R = 40 nm | C = 0.06 N/m | silicon nitride tip
  - Scanning parameters: Scan size = 5 μm × 5 μm | Scan rate = 0.5 Hz (tip velocity of 5μm/s) | Peak Force setpoint = 10 nN | Peak Force amplitude = 300 nm | z-piezo frequency = 1 kHz
  - Topography, deformation, and adhesion images were collected.

Figure 2. Changes in fiber surface topography (wear process), before and after friction experiments

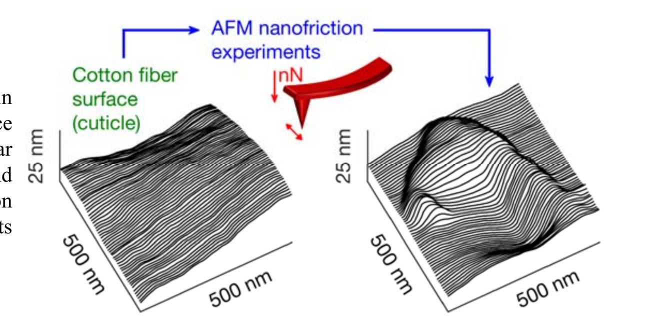


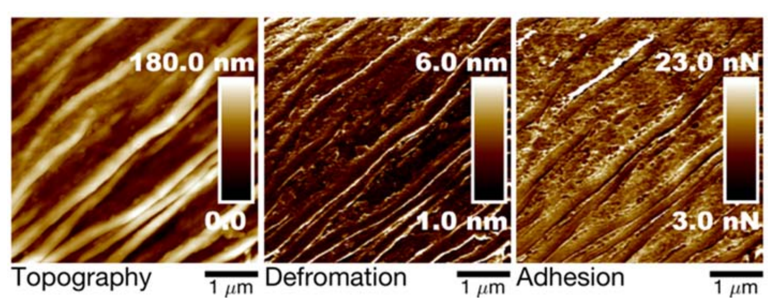
Table 1. Physical and Dimensional Properties of Cotton Fiber Samples.

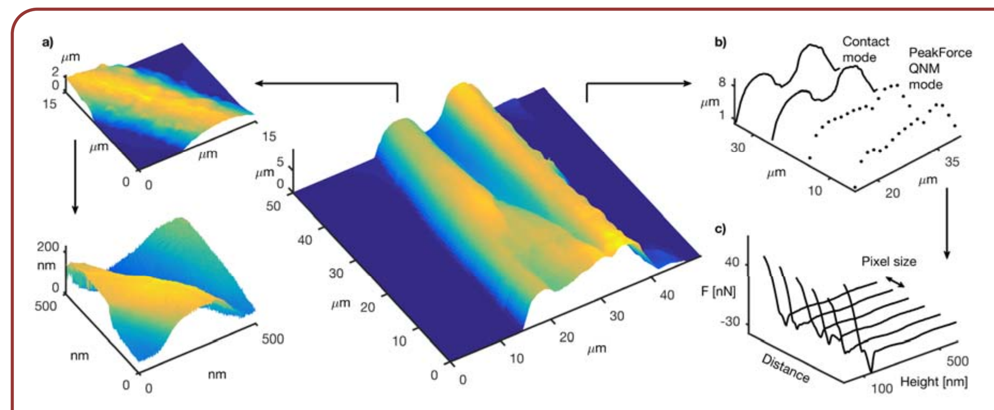
Samples	Macroscale friction coefficient	Micronaire	Length [cm]	Strength [g/tex]	Elongation [%]	+b
A (BRS 293)	0.080	4.7	2.8	32.1	7.4	8.4
B (ST 4747GLB2)	0.066	4.1	2.8	33.1	8.6	9.6

Results and Discussion

1. Typical AFM images of fibers:

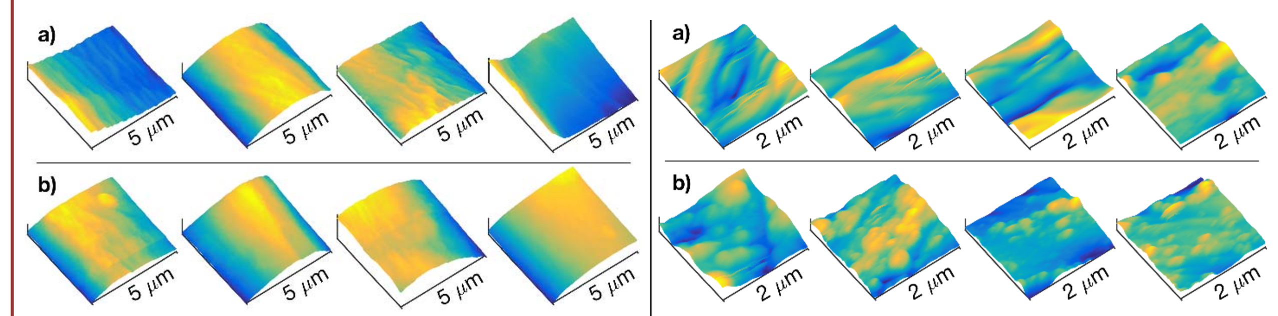
Typical false-color AFM images of cotton fiber surface obtained in force tapping mode





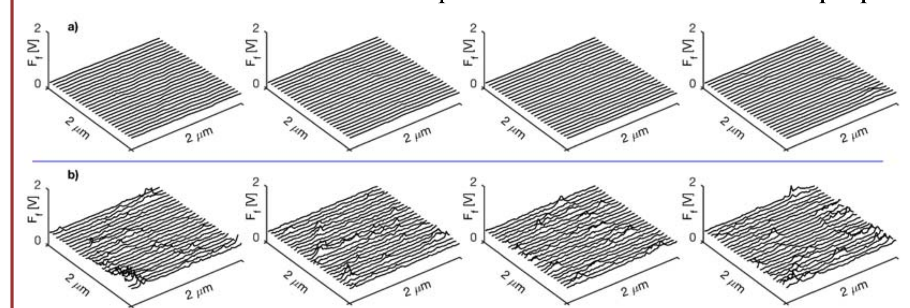
a) Surface topography images of a cotton fiber obtained at different scan size; as can be noted cotton fiber has a bean-shape cross-section.
b) Different scan lines produced from different AFM modes.
c) An array of force-distance curves produced along a scan line in PeakForce tapping mode.

2. Comparison of surface topography:



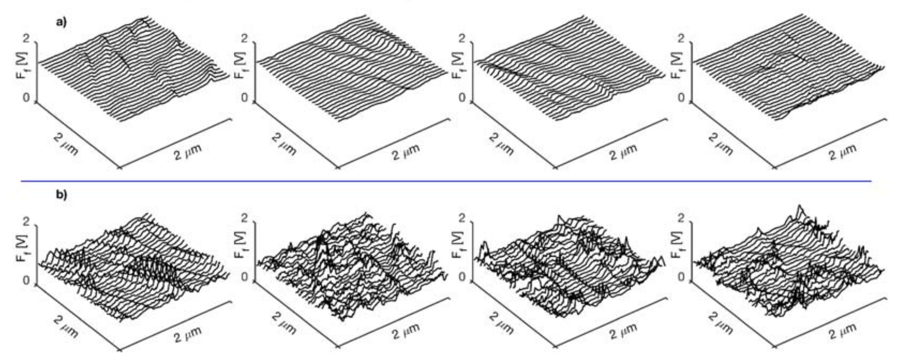
- Force tapping mode:
  - Fiber has a bean-shaped cross-section.
  - Scanned areas can be concave, convex, or relatively flat.
  - The furrows and ridges on fibers surface are attributed to the wrinkles in the fibers primary cell wall.
  - They are formed during fiber shrinkage.
  - They are characteristics of dried, untreated cotton fiber.
  - Difference are not clear at this scale
- Contact mode:
  - In addition to the furrows and ridges, irregular-shaped, particulate/plaque-like surface aggregates is evident at this length scale.
  - They more abundant on sample B fibers.
  - Their exact nature is unclear
  - Can be due to the self-assembly and growth of wax crystals.
  - Crystalline microstructures: tubules, platelets, and rodlets

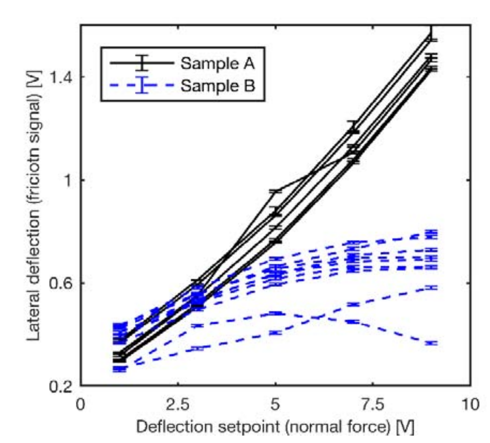
3. Comparison of nanoscale frictional properties:

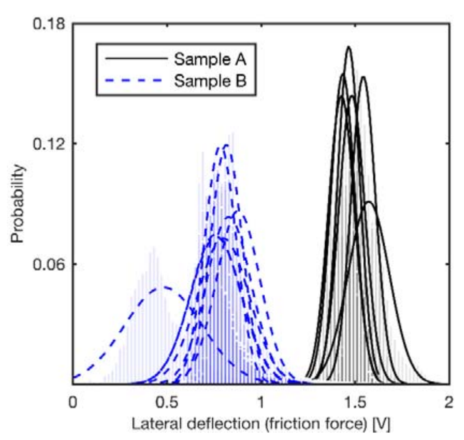


- Friction images of the sample A (upper panel) and sample B (lower panel) fibers obtained under the 10 nN normal load.
- Four scans per each sample are shown here.
- Under this normal load, differences in friction signal between two samples were not significant.

- Friction images of the same fibers, obtained under L = 50 nN.
  - Fibers from sample A (higher macroscale friction) were characterized with significantly higher nanoscale friction. (1.5 V compared to 0.75 V)
  - Local variations in nanofriction signal – more evident on sample B fibers – are due to topographical effects (the ratchet mechanism component of friction)

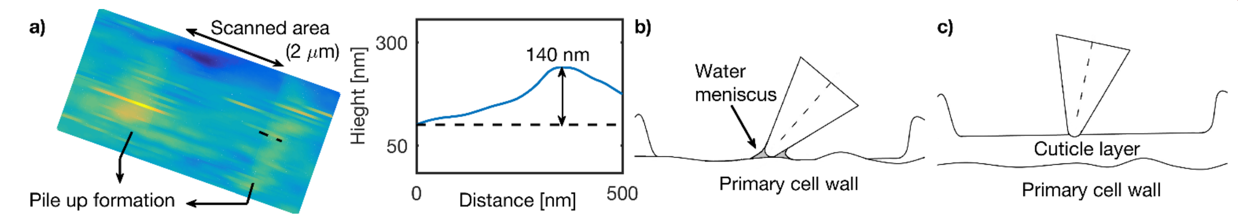






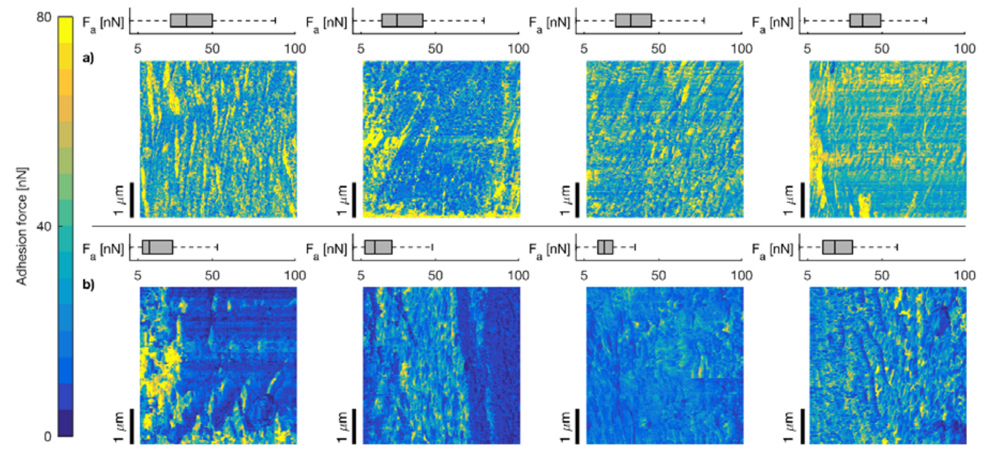
- **Friction vs. normal load**
- No significant difference at low normal loads
- The relatively blunt probe with low spring constant could not penetrate into cuticle.
- It slid on their waxy layer
- Long chain hydrocarbons acted as a lubricant layer

- **Friction at higher** L**:**
- Abrasive friction led to wear in sample B.
- The cuticular molecules have been displaced.
- The layer underneath (cellulose from primary cell wall) is more hydrophilic.
- Formation of water meniscus has increased friction.



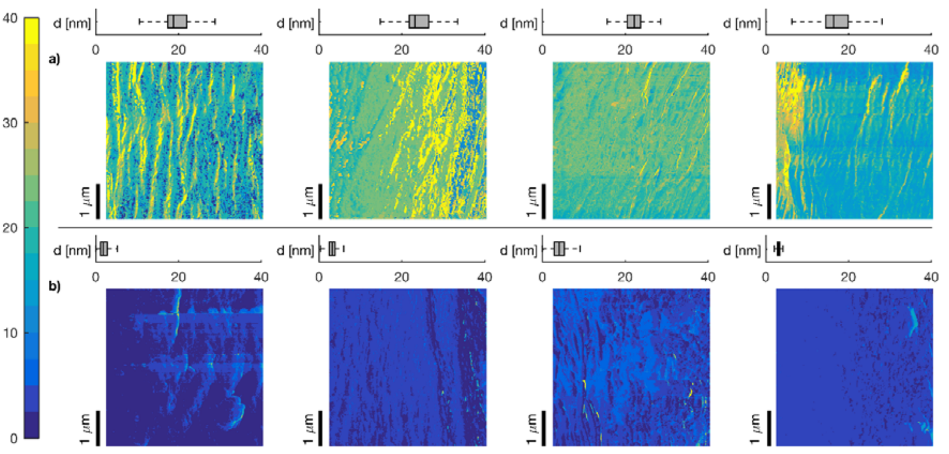
- Figure (a) shows a typical wear mark after a set of friction experiments; plot shows pile-up formation
  - Single-asperity friction should hold a nonlinear relationship with L (such as sample B fibers)
  - Deviation from nonlinearity for sample A proves that the cuticle layer was very thin and new substrate has been exposed to friction experiments
- Schematic of tip-surface interaction for fibers with thin (a) and thick (b) cuticular layer

4. Comparison of nanomechanical properties:



- Adhesion images of the sample A (upper panel) and sample B (lower panel) fibers
- Mean pull-off force for sample A was 17.2 ± 6.2 nN, compared to 45.5 ± 10 nN for sample B.

- Deformation images of the sample A (upper panel) and sample B (lower panel) fibers
- Mean surface deformation for sample A is 6.3 ± 1.2 nm, compared to 22.5 ± 3 nm for B.



- Estimated contact radius a as a function of normal force for silicon nitride tip with R of 40 nm
- The values obtained from three famous models: Hertz, DMT, and JKR
- According to the JKR model

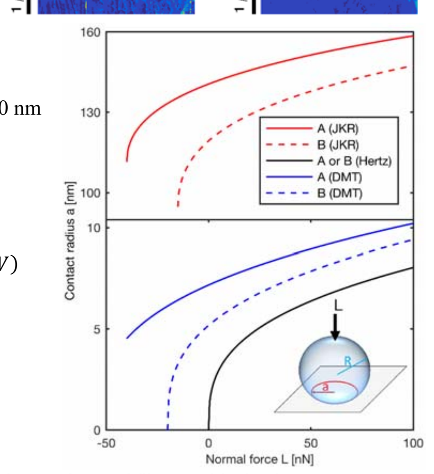
$$F_{ad} = \left(\frac{3}{2}\right)\pi R W \Rightarrow W = 2F_{ad}/3\pi R$$

- The contact area between the AFM tip and fibers surface.

$$a = \sqrt[3]{\frac{R_1 L}{E_{tot}}} \qquad a^3 = \frac{R_1}{E_{tot}}\left(\sqrt{\frac{3}{2}\pi R_1 W} + \sqrt{L + \frac{3}{2}\pi R_1 W}\right)^2 \qquad a^3 = \frac{R_1}{E_{tot}}(L + 2\pi R_1 W)$$

- The reduced modulus:

$$\frac{1}{E_{tot}} = \frac{3}{4}\left(\frac{1-v_s^2}{E_s} + \frac{1-v_a^2}{E_a}\right)$$



Conclusions

- Surface of both fibers were characterized with series of furrows and ridges while the surface of fibers with the lower macroscale friction (sample B) were also characterized with certain granular-like surface deposits.
- Differences in fibers nanofriction were more apparent under higher normal forces.
- Fibers from sample B with lower macroscale friction were also characterized with lower nanoscale friction.
- It was discussed that differences in nanofrictional properties under higher normal forces can be due to changes in fibers surface hydrophilicity because of the cuticle layer removal.
- Since the increase in nanofriction as a function of normal force was not as significant for sample B fibers, it was concluded that theses fiber are covered with thicker layer of the cuticular materials.
- Fibers from sample B were characterized with both lower average nanoscale adhesion and deformation
- While different contact mechanics models estimated different values for the real contact area, it was shown that, under any model, fibers from sample B are always characterized with smaller contact radius.

References

1. Sui, R.; Thomasson, J. A. et al. Trans. ASABE 2008, 51 (6), 2201–2208
2. Sui, R.; Thomasson, J. A. et al. J. Cotton Sci. 2010, 153, 145–153
3. J. D. Batteas et al., "Surface and interfacial studies of plant biopolymers," in *Molecular Interfacial Phenomena of Polymers and Biopolymers*, Elsevier, 2005, pp. 580–608.
4. A. N. Round, et al. "The Influence of Water on the Nanomechanical Behavior of the Plant Biopolyester Cutin as Studied by AFM and Solid-State NMR," *Biophys. J.*, vol. 79, no. 5, pp. 2761–2767, 2000.
5. Mirabet, V. et. al; The Role of Mechanical Forces in Plant Morphogenesis. Annu. Rev. Plant Biol. 2011, 62 (1), 365–385.