Mechanical characterization of wood viscoelasticity at sub - microscale

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Introduction

		Dry conditions (12% moisture, 20 °C)	Moist conditions (20 °C)
Cellulose	E_l	134 GPa	134 GPa
	E_t	27.2 GPa	27.2 GPa
	[illegible]	[illegible]	[illegible]

Extracted/regenerated constituents or theoretical/numerical estimations

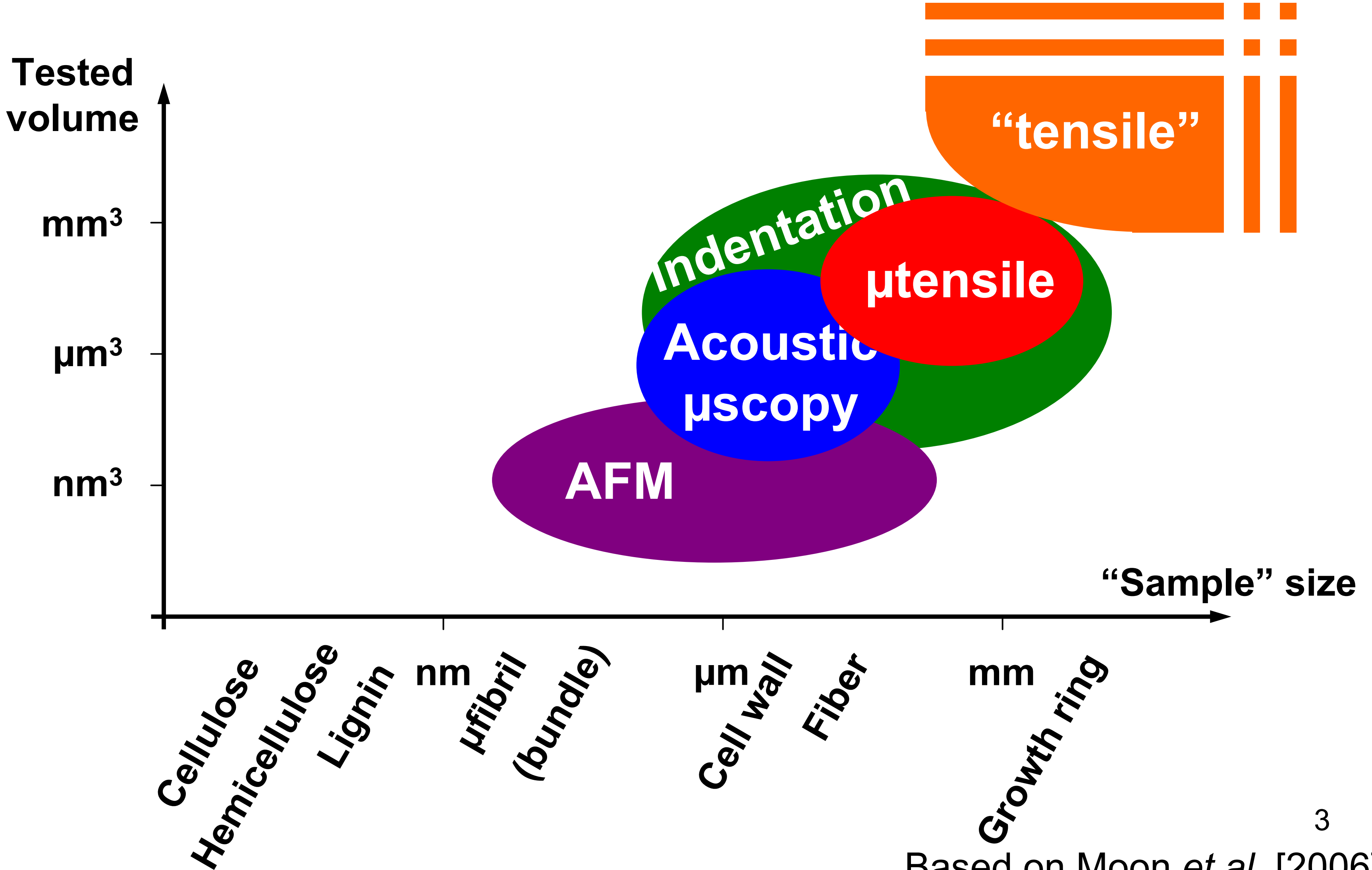
+

lack of data about the rheological behavior (viscosity) at this scale !

Lignin	E_l	[illegible]	[illegible]
	E_t	1.0 GPa	1.0 GPa
	$G_{lt\ ?}$	0.6 GPa	0.6 GPa
	$\nu_{lt\ ?}$	0.3	0.3

Compilation from Salmén [2004]

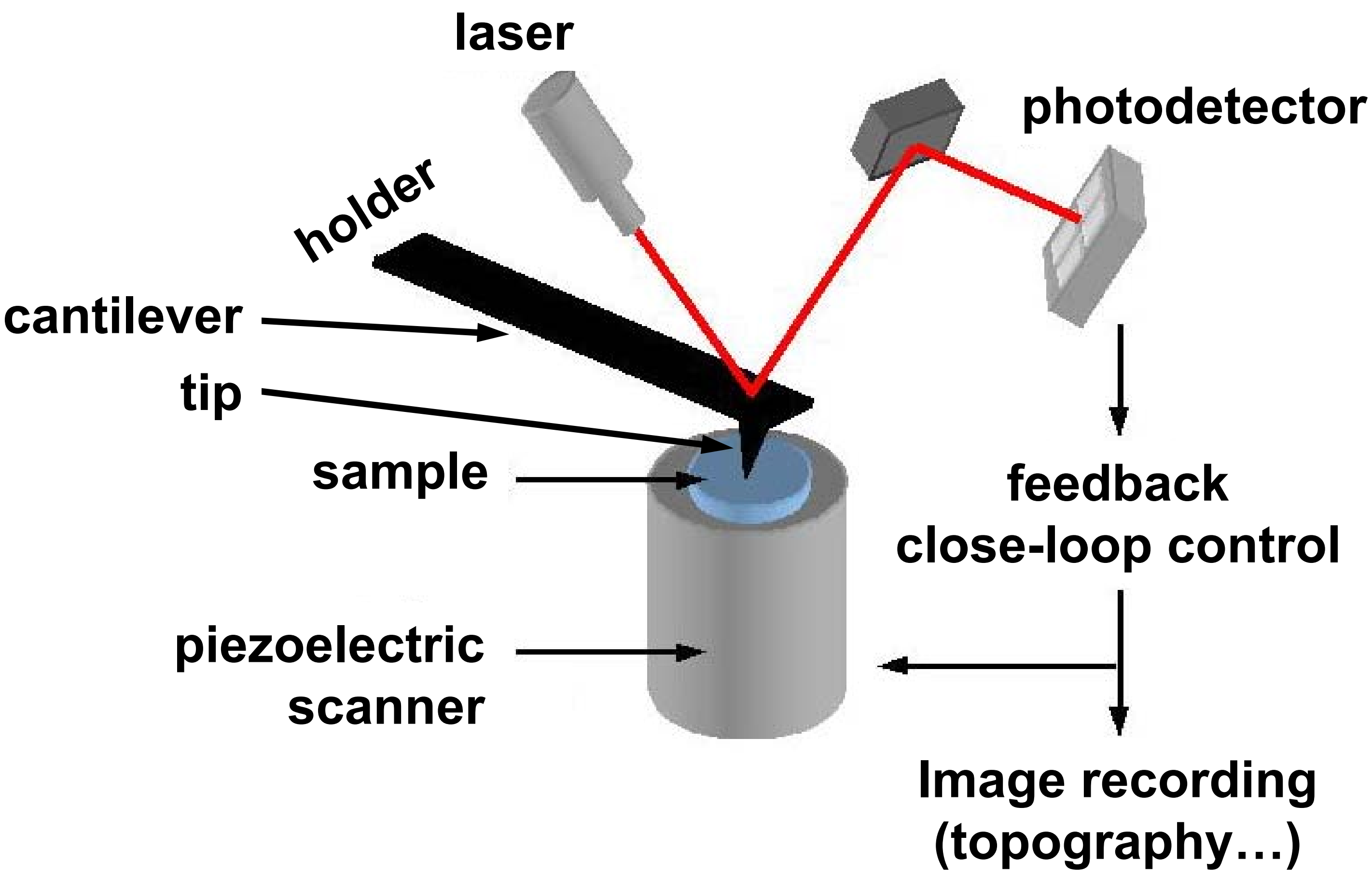
Introduction



Based on Moon *et al.* [2006]

Ultrasonic Contact - Atomic Force Microscopy

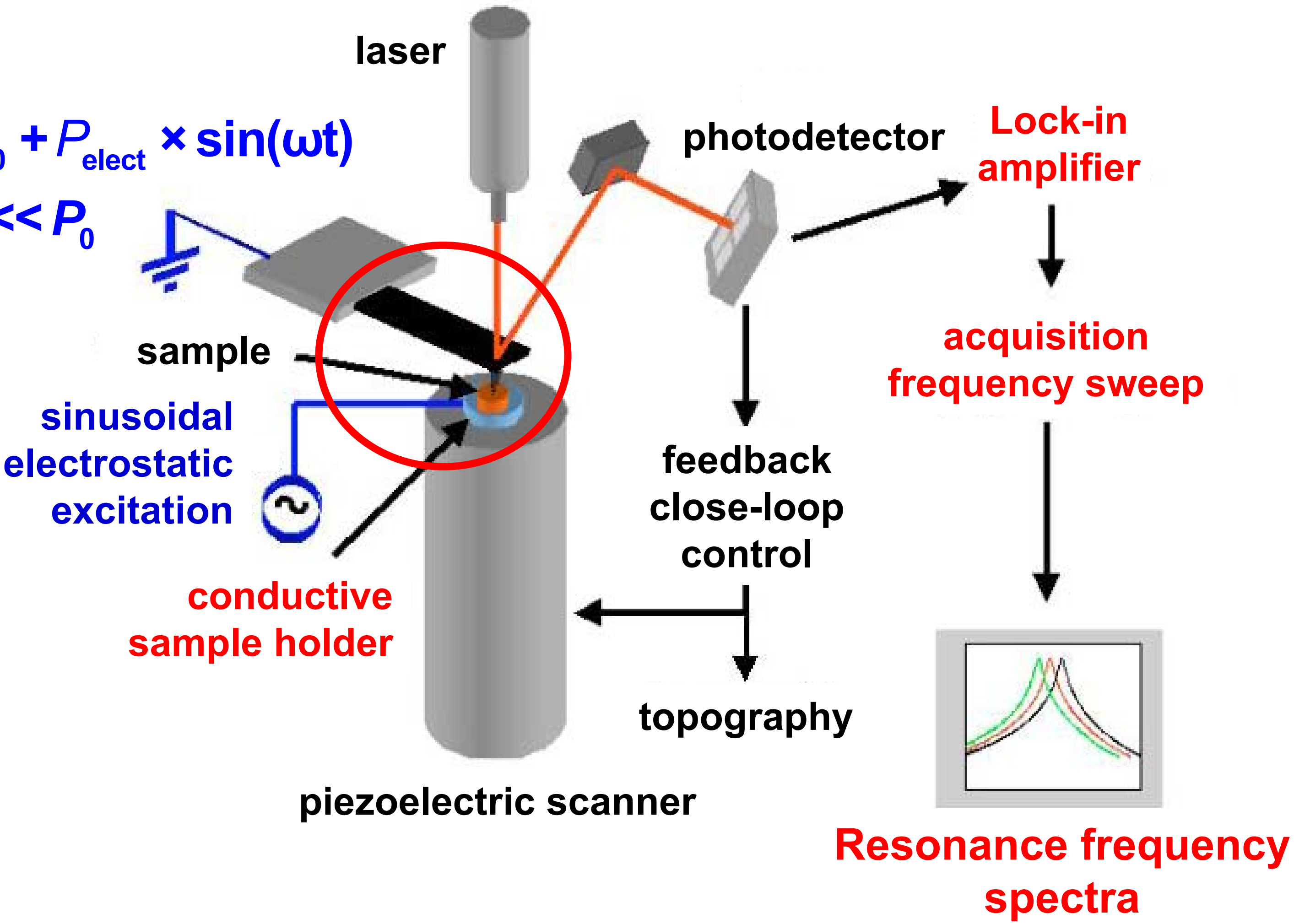
Atomic Force Microscopy – basic configuration



Atomic Force Microscopy – Ultrasonic Contact Mode

$$P \approx P_0 + P_{elect} \times \sin(\omega t)$$

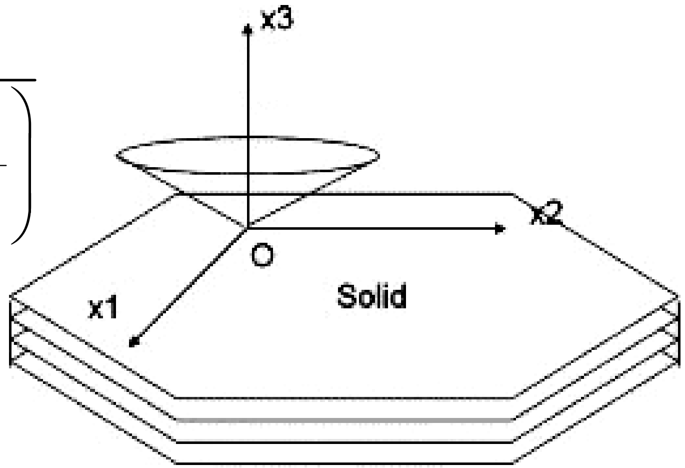
$$P_{elect} << P_0$$



Atomic Force Microscopy – Elastic anisotropy

Closed form solution of *M* only available in particular case…
→ along the principal axes of an orthotropic solid [Delafargue *et al.*, 2004]

$$M_3^2 = 4\sqrt{\frac{\dfrac{C_{11}C_{33}-C_{13}^2}{C_{11}}\cdot\dfrac{C_{22}C_{33}-C_{23}^2}{C_{22}}}{\left(\dfrac{1}{C_{44}}+\dfrac{2}{\sqrt{C_{11}C_{33}}+C_{13}}\right)\cdot\left(\dfrac{1}{C_{55}}+\dfrac{2}{\sqrt{C_{22}C_{33}}+C_{23}}\right)}}$$

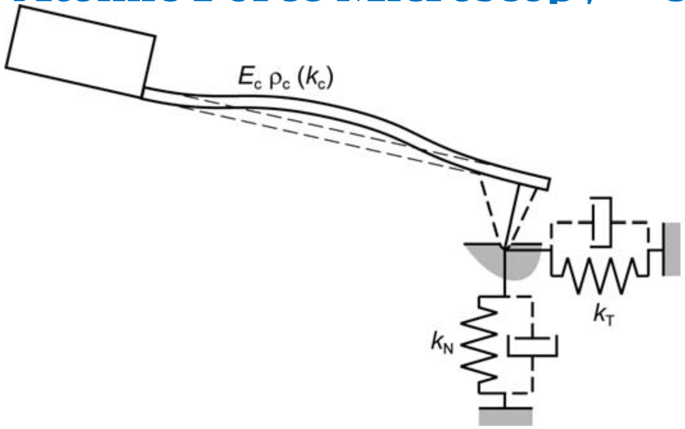


Typical dry poplar S2-layer (30% crystalline cellulose + 70% matrix, MFA~0°)

$E_L \approx 45$GPa, $E_t \approx E_r \approx 12$GPa, $\nu_{tL} \approx \nu_{rL} \approx 0.028$, $\nu_{rt} \approx 0.28$
$G_{tL} \approx G_{rL} \approx 2.5$GPa, $G_{rt} \approx 2$GPa

→ $M_L \approx 19$GPa and $M_{r\ or\ t} \approx 9.5$GPa

Atomic Force Microscopy – Ultrasonic Contact Mode



Vibration equation if k_T ~ 0 (slip) & negligible viscosity [Rabe, 2002]

$$1+\cos(cL)\cdot \mathrm{ch}(cL)=\frac{3k_{\mathrm{N}}}{k_{\mathrm{C}}c^{3}}\left[\cos(cL)\cdot \mathrm{sh}(cL)-\sin(cL)\cdot \mathrm{ch}(cL)\right]$$

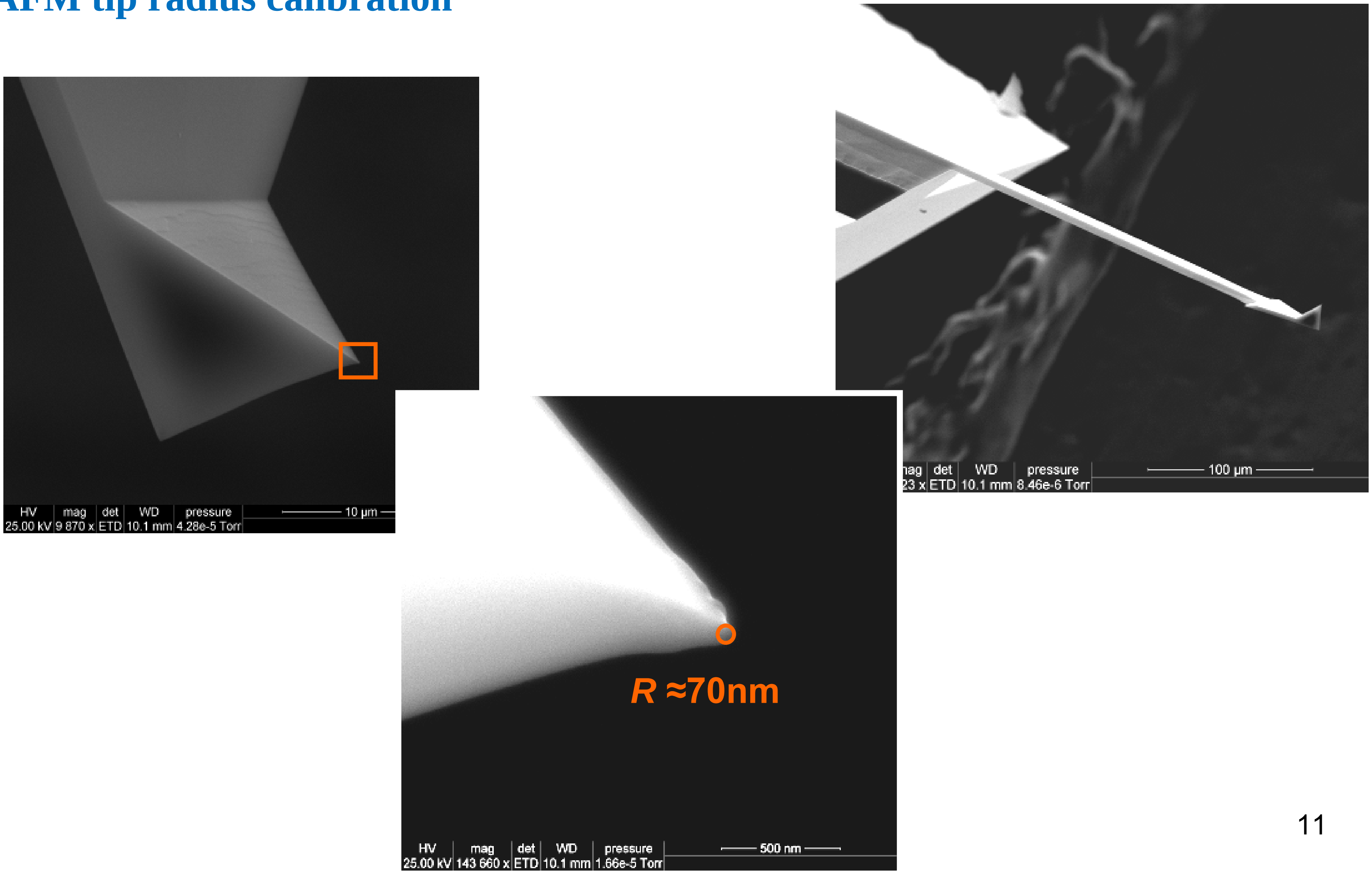
$$\text{with} \quad c_n=\left(\frac{48\pi^2\rho_c}{E_c e_c^{\,2}}\right)^{1/4} f_n^{\;1/2}$$

Hertz contact equation with DMT adhesion model

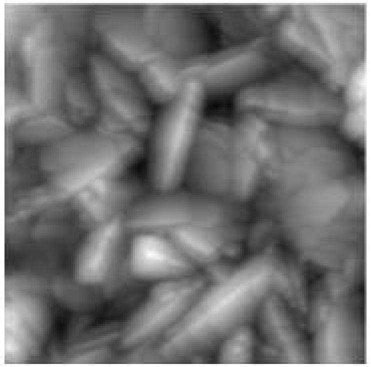
$$k_N=\left[6E^{*2}R(P+P_{\mathrm{adh}})\right]^{1/3} \qquad \frac{1}{E^*}=\frac{1-\nu_t^2}{E_t}+\frac{1}{M}\approx\frac{1}{M}$$

Cantilever calibrations

AFM tip radius calibration



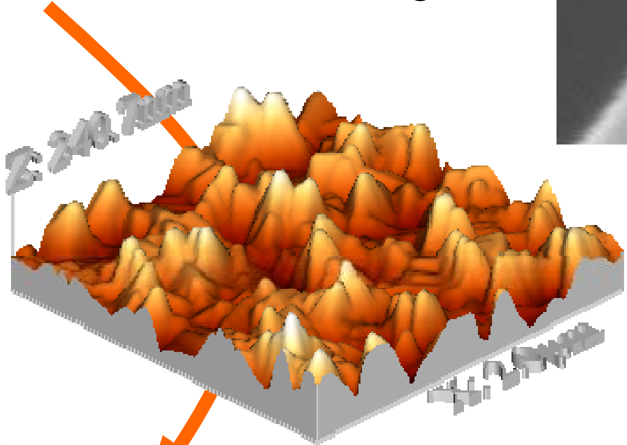
AFM tip radius calibration

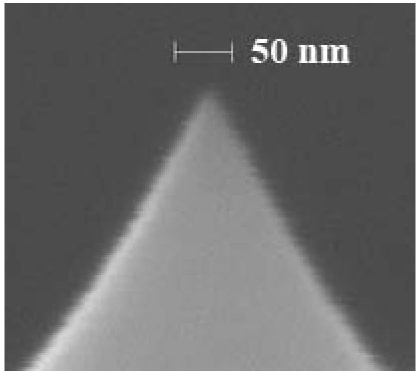


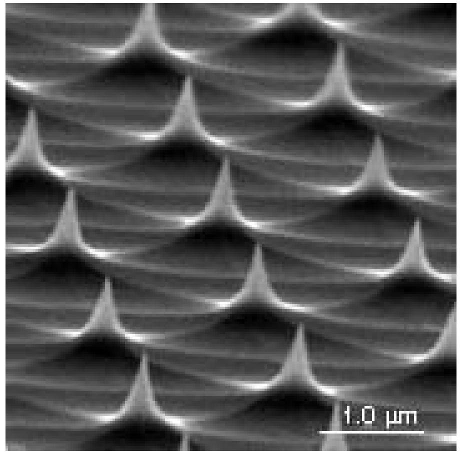
2 µm

TipCheck
[www.budgetsensor.com]

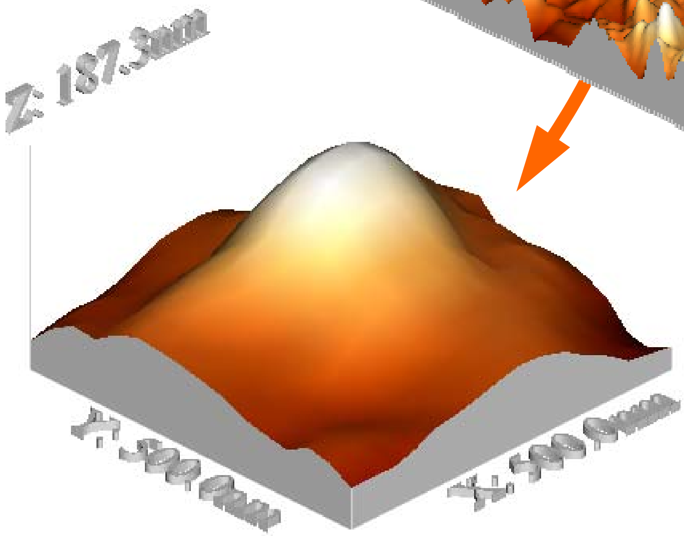
titanium film coating



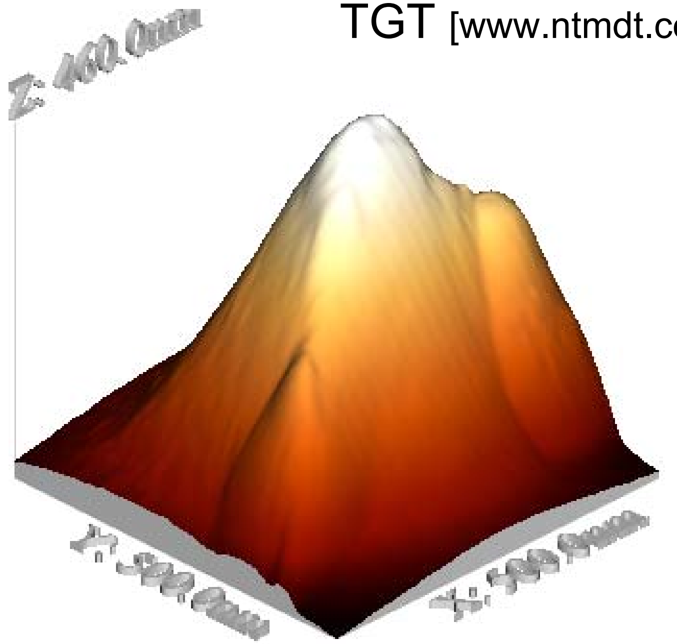




TGT [www.ntmdt.com]



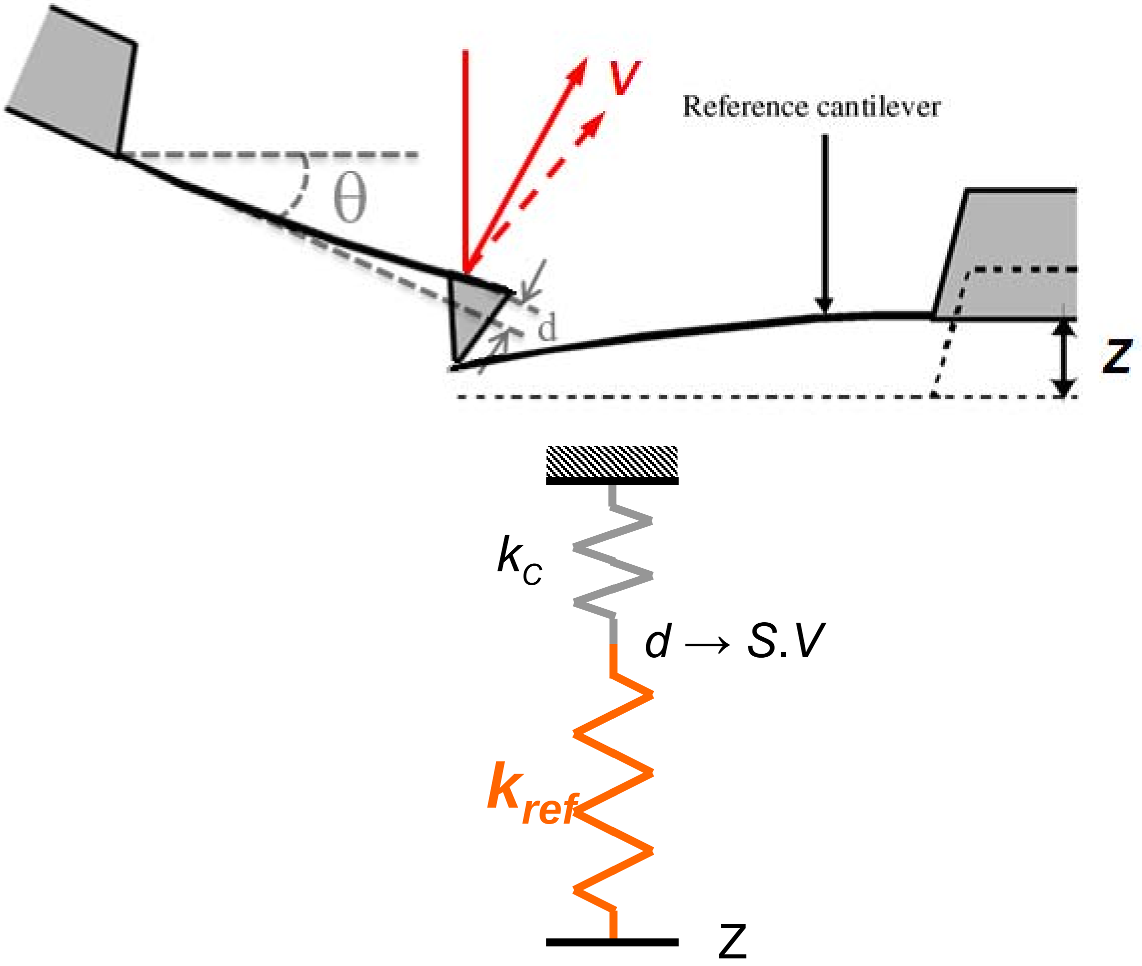
$R_1 \approx 60$nm - $R_2 \approx 20$nm

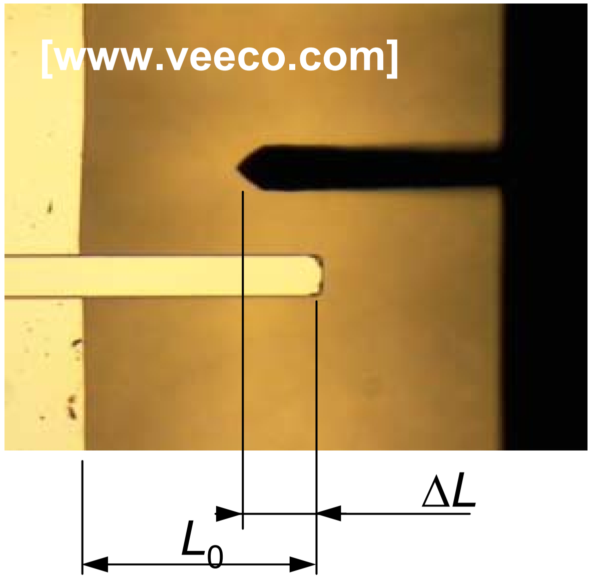


$R_1 \approx 65$nm - $R_2 \approx 20$nm

AFM cantilever stiffness calibration

[Butt *et al.*, 2005; Gibson *et al.*, 1996]



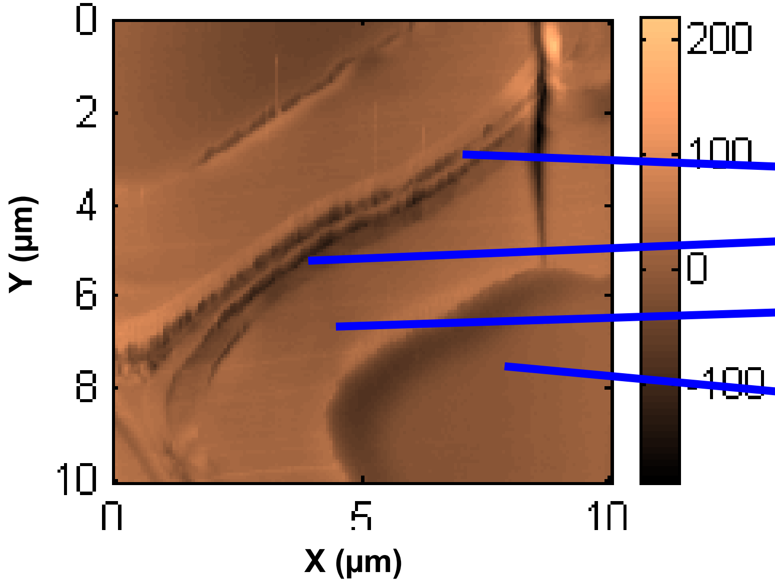


Results on wood

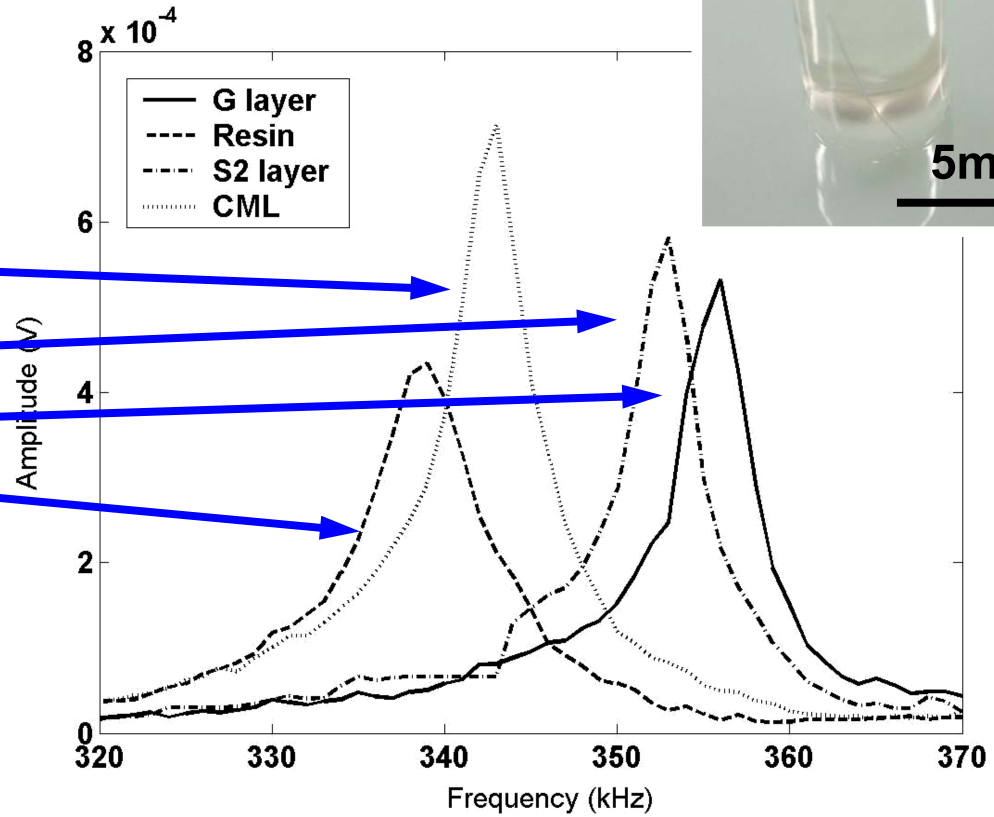
Results on chestnut TW

Frequency spectra

R **~ 50nm,** $P_0 + P_{adh}$ **~ 265nN,** k_C **~ 3.7N/m**



Topography (nm)



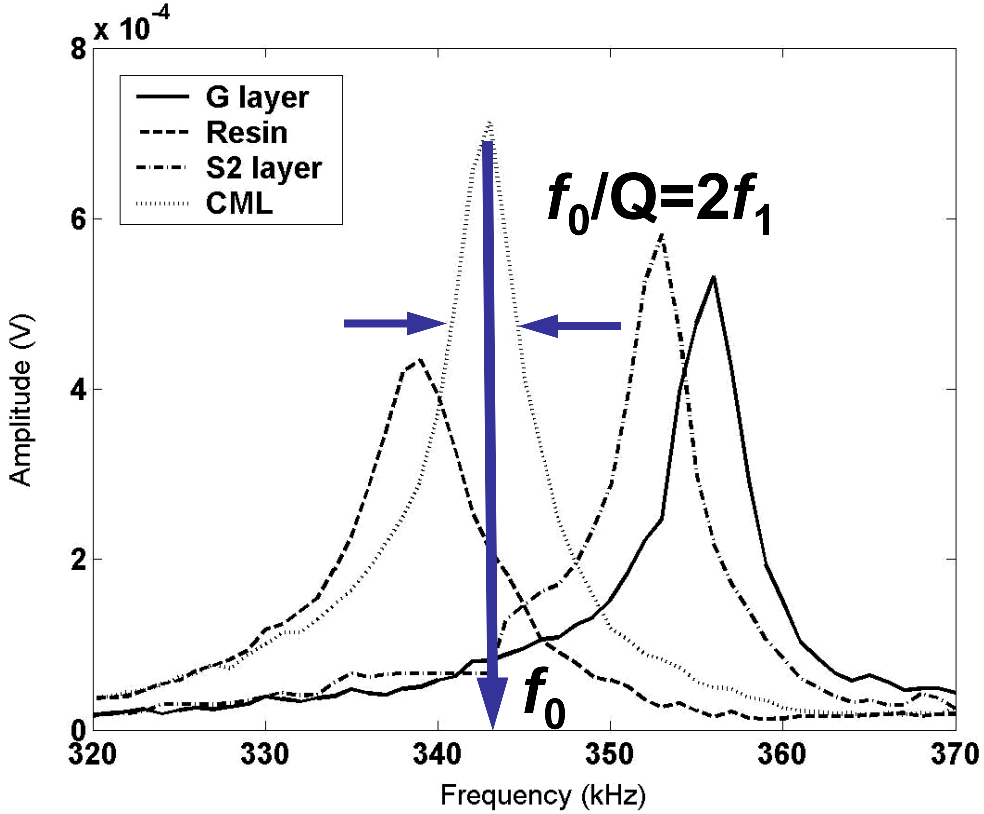
Results on chestnut TW

Frequency spectra

***R* ~ 50nm, $P_0 + P_{adh}$ ~ 265nN, k_C ~ 3.7N/m**

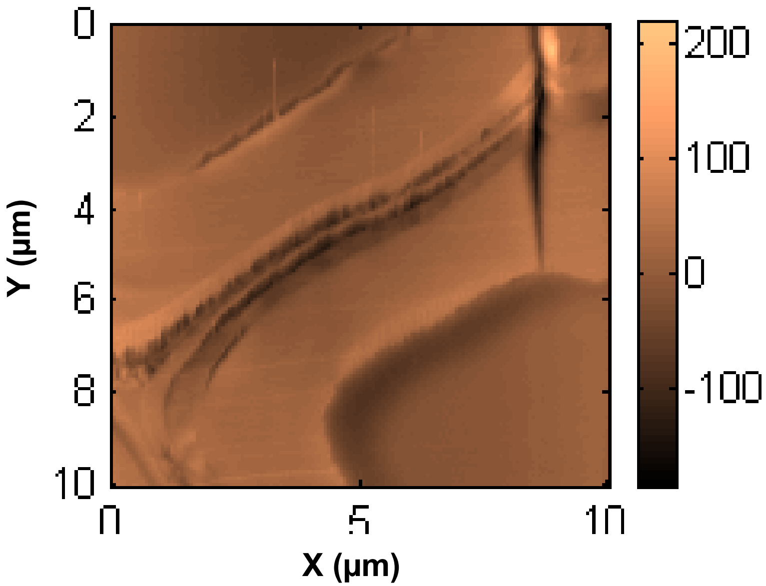
	M_L (GPa)	1/Q
Resin	4.1	0.015
CML	5.5	0.014
S2	12.6	0.009
G	15.9	0.009

$$Q \sim \frac{1}{\tan\delta}$$

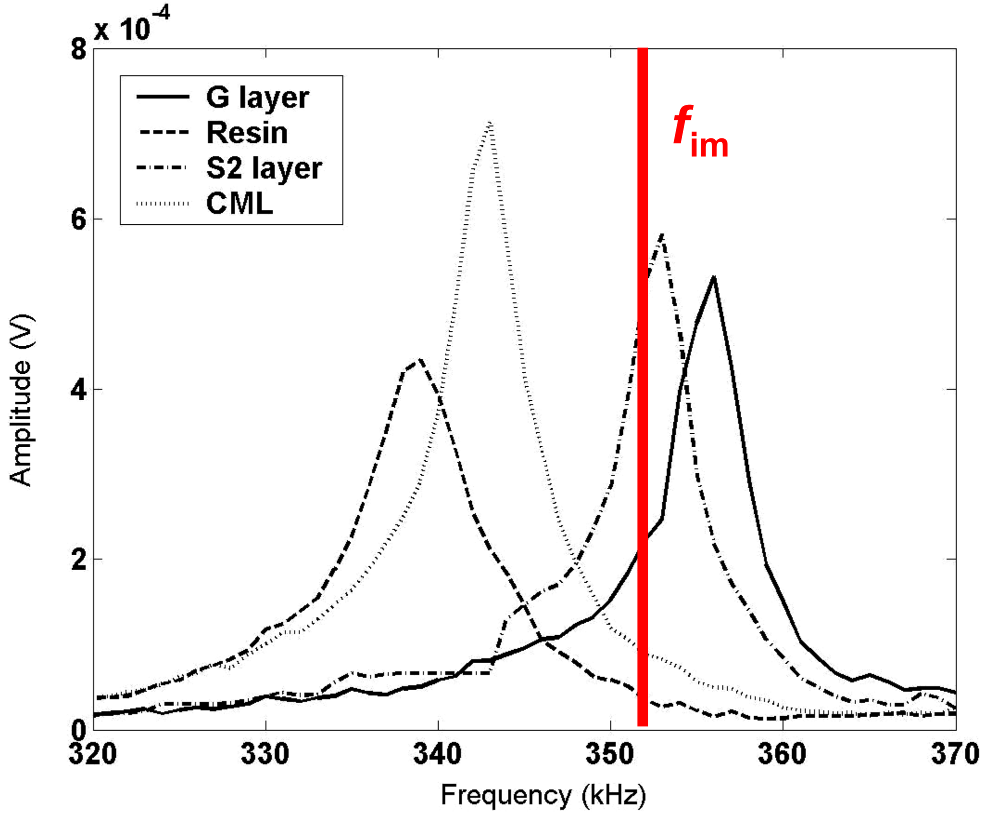


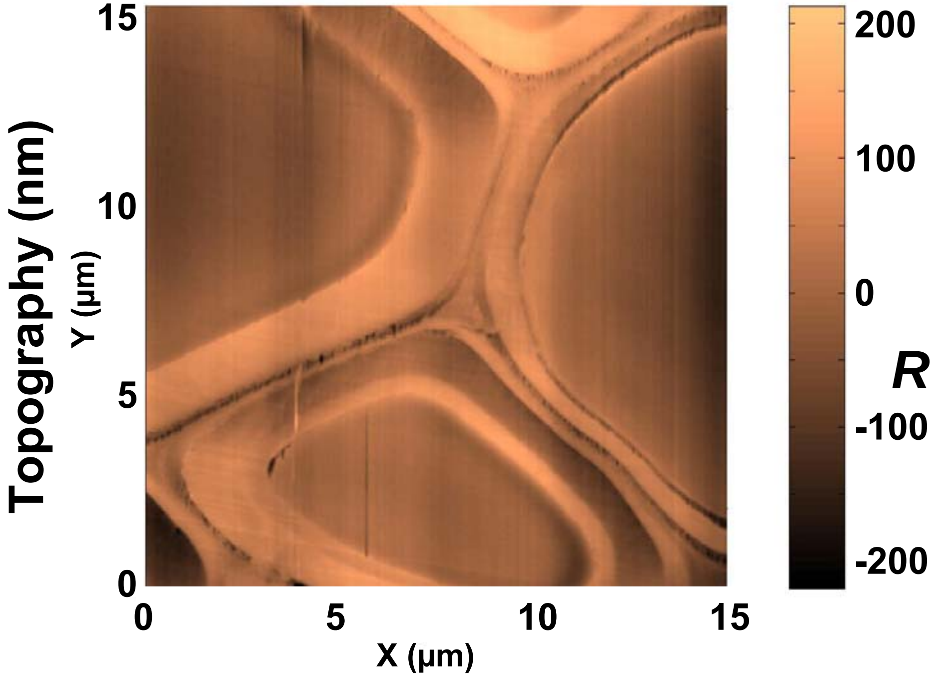
Results on chestnut TW

Mapping



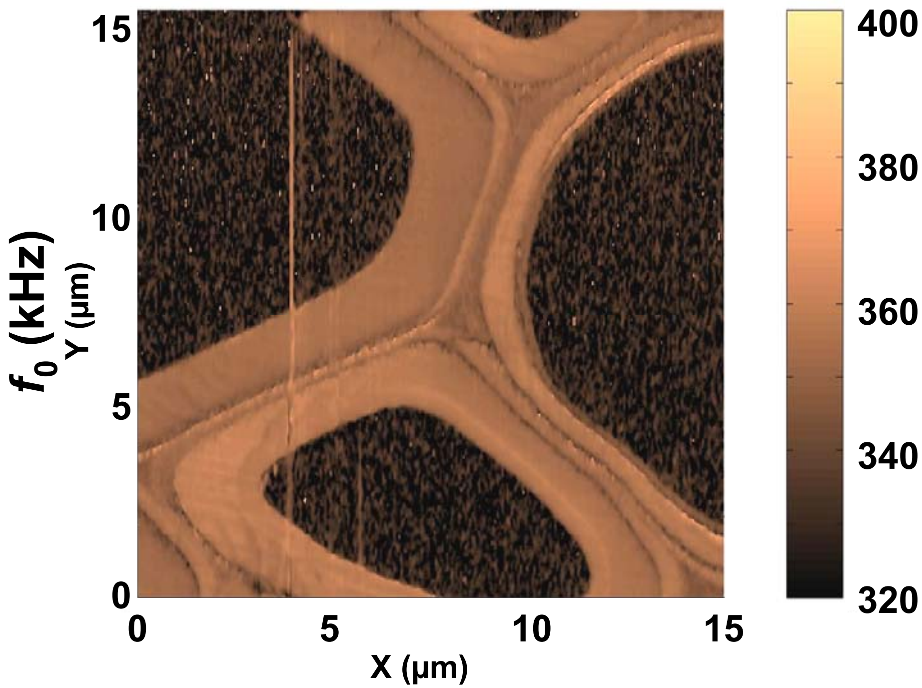
Topography (nm)

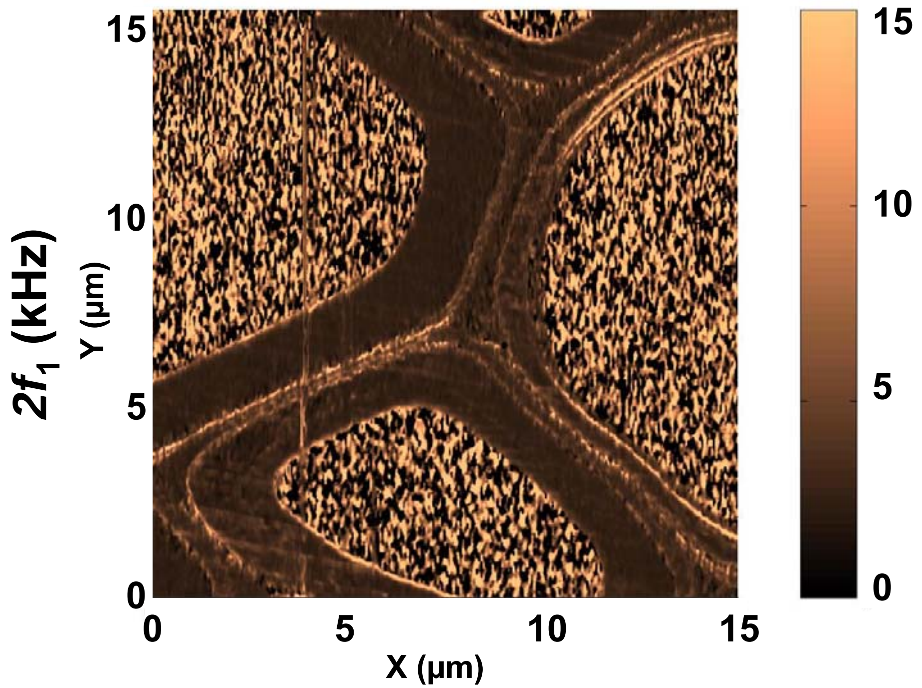


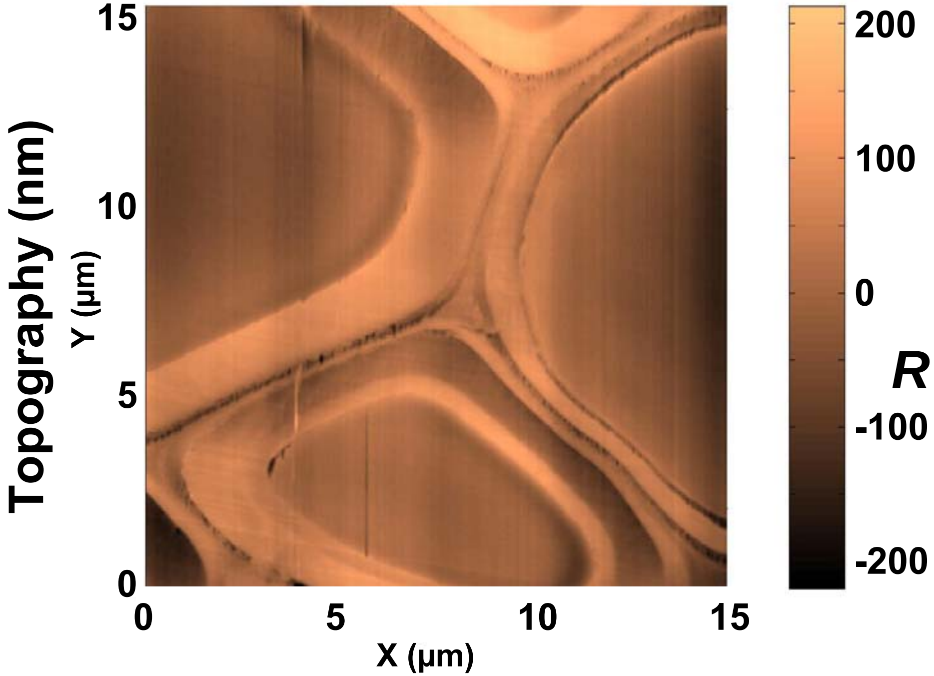


Mapping at 352kHz

R ~ 50nm, P_0 +P_{adh} ~ 265nN, k_C ~ 3.7N/m



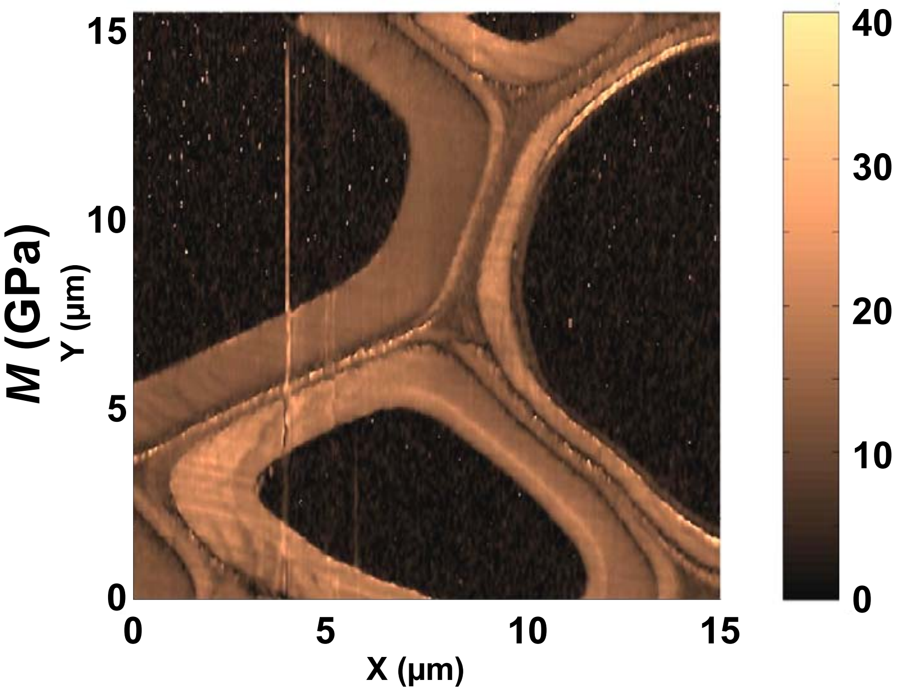


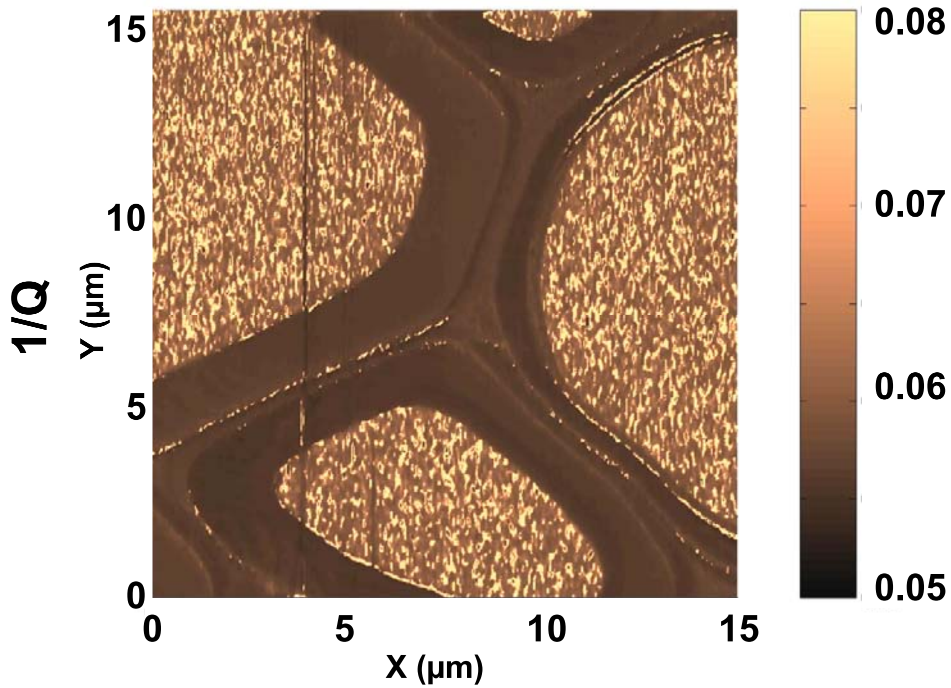


Mapping at 352kHz

R ~ 50nm, $P_0 + P_{adh}$ ~ 265nN, k_C ~ 3.7N/m

$2a$ ~ 15nm





Conclusions and future developments

- Ability of UC-AFM to measure (visco)elastic properties of the cell wall has been demonstrated…

- Tests to be done to find all the stiffness constants of a cell wall?
→ measurement in many orientations: example on G-layer of chestnut TW
($k_C \approx$ 11N/m, $R \approx$ 10nm)

Angle / MF direction	0°	45°	90°
f_0 (kHz)	535	522	480

- Needs of calibration on reference materials, especially for tip slip (or not!) estimation and real content of Q... currently in progress!

- Topography effect/correction and optimal tip shape / cell-wall ultrastructure…

- Spatial resolution of UC-AFM may allow us to measure *in situ* viscoelastic properties of the cell wall *constituents... Not yet!*