Mesures des propriétés viscoélastiques des matériaux : des mesures macroscopiques à la cartographie quantitative à la nanoéchelle

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Plan

La viscoélasticité, qu'est-ce que c'est ?

La spectroscopie mécanique ou DMA

L'équivalence temps-température

Limites et perspectives : vers les mesures locales

Extension à la nanoéchelle

- nanoDMA
- CR-AFM
- Intermodulation AFM
- …

Conclusions et Perspectives

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Conclusions et Perspectives

La viscoélasticité

L'expérience de la goutte de poix

Elasticité

Viscosité

Fluide visqueux

?

Solide fragile

La viscoélasticité

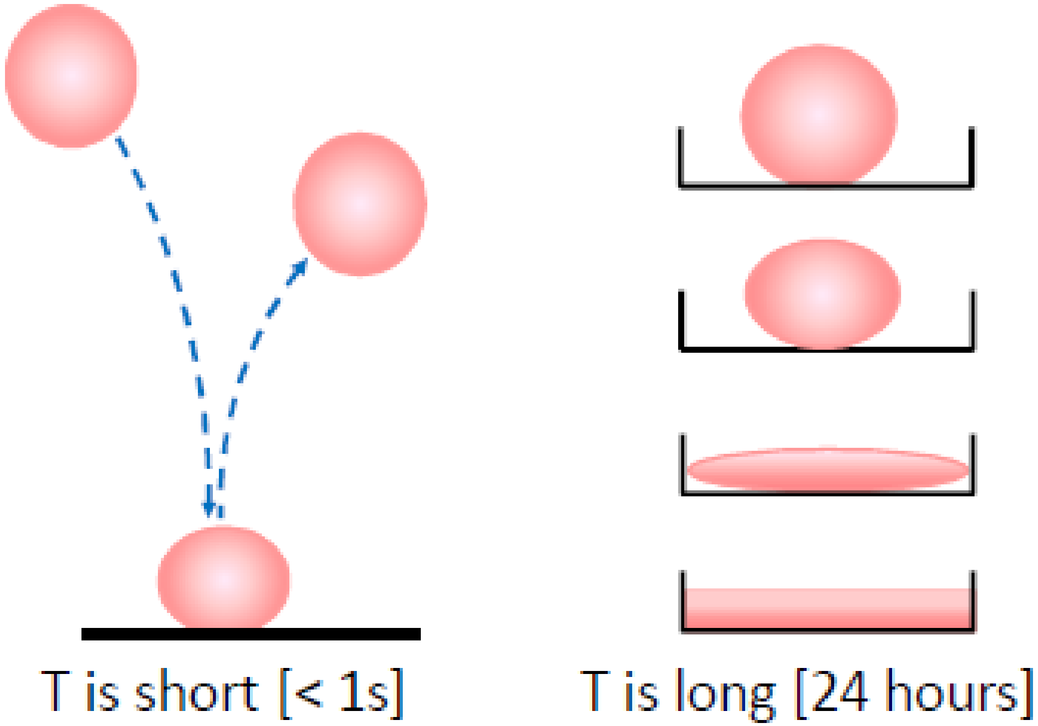
La maïzena dans l’eau

La viscoélasticité

Un matériau peut avoir l'apparence d'un « solide » si :

1- il a un temps de relaxation caractéristique très long

2- le processus de déformation correspondant est très rapide

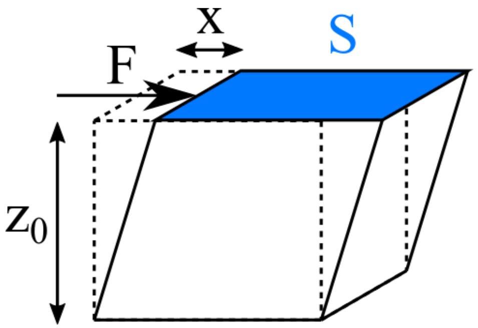


La viscoélasticité

La viscoélasticité

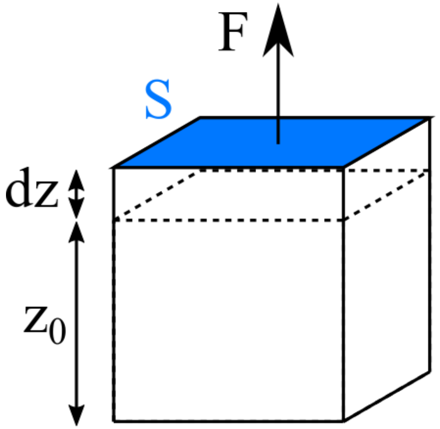
Mesurer les propriétés mécaniques

Contrainte σ, déformation ε



Cisaillement

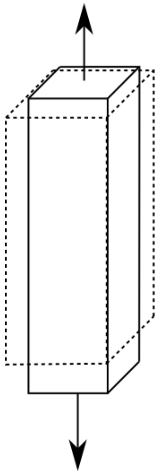
$$\sigma = \frac{F}{S} \qquad \gamma = \frac{x}{z_0} \qquad \dot{\gamma} = \frac{1}{z_0}\frac{dx}{dt}$$



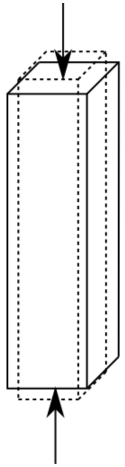
Traction

$$\sigma = \frac{F}{S} \qquad d\varepsilon = \frac{dz}{z_0} \qquad \dot{\varepsilon} = \frac{1}{z_0}\frac{dz}{dt}$$

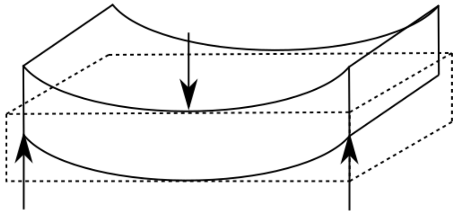
Modes de sollicitation



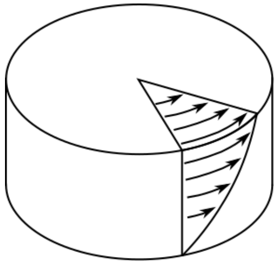
Traction



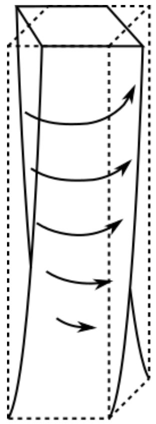
Compression



Flexion



Cisaillement

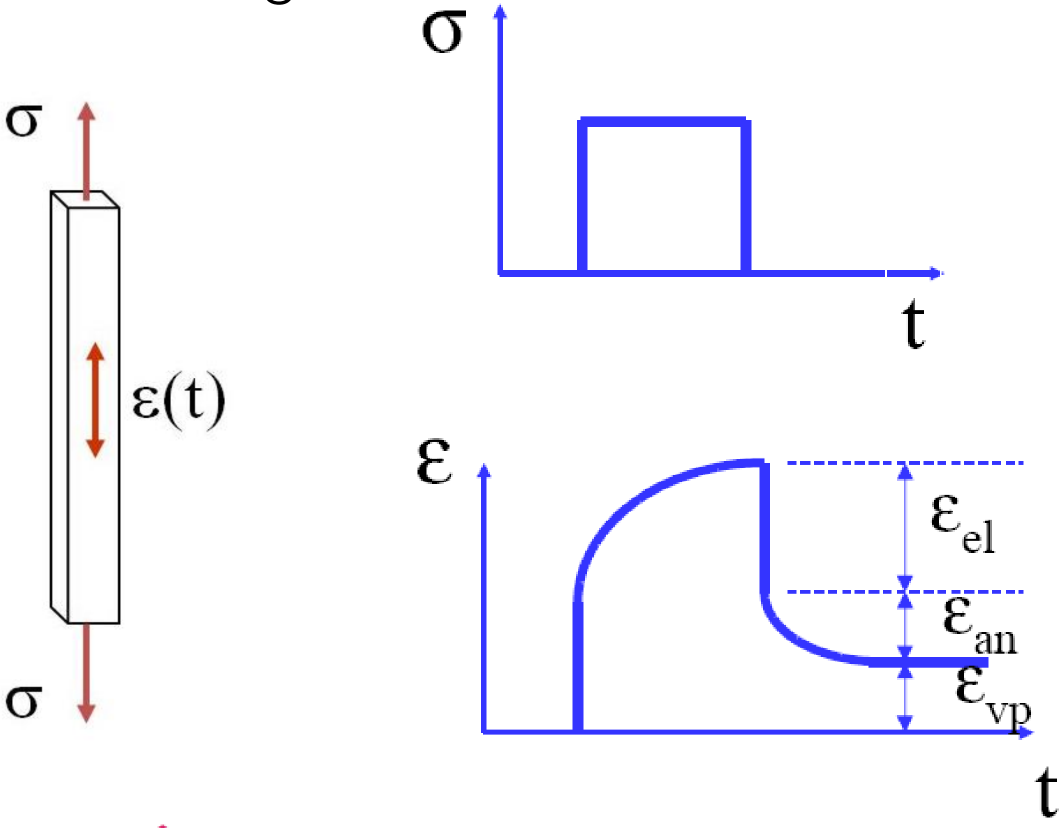


Torsion

La viscoélasticité

Manifestation expérimentale de la viscoélasticité

Essai de fluage



el : élastique; retour réversible, immédiat

an : anélastique ou viscoélastique; retour réversible, différé (f(temps))

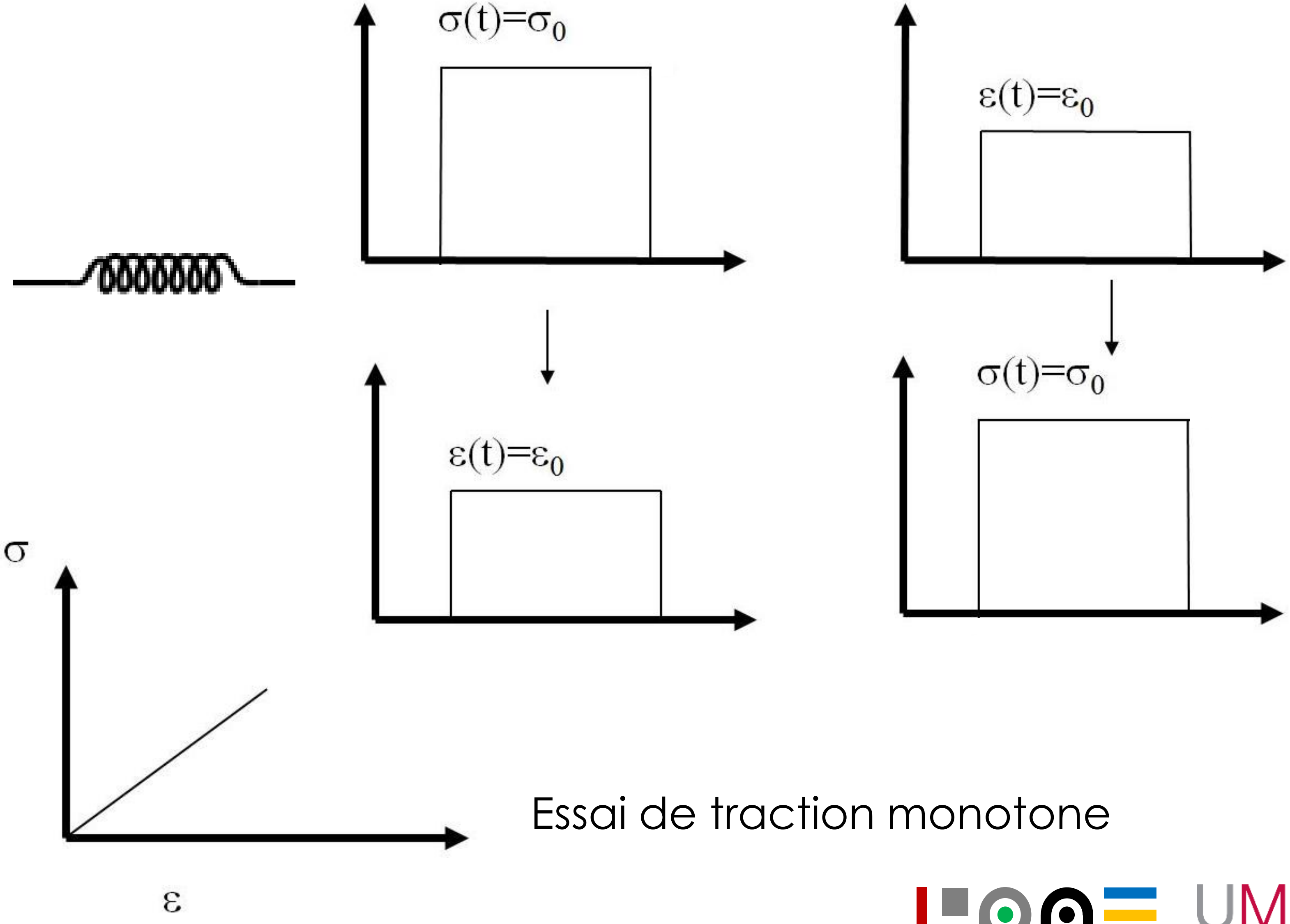
vp : viscoplastique; irréversible, pas de retour (ε_{vp} peut être recouvrée à $T > T_g$)

La viscoélasticité

L'élasticité

Essai de fluage

Essai de relaxation



Essai de traction monotone

Loi de Hook
En traction : $\sigma = E\varepsilon$
En cisaillement : $\sigma = G\gamma$

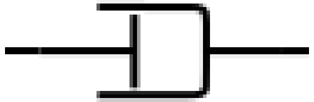
Module d'élasticité :
$\mathbf{E = 2(1 + \nu)G}$

La viscoélasticité

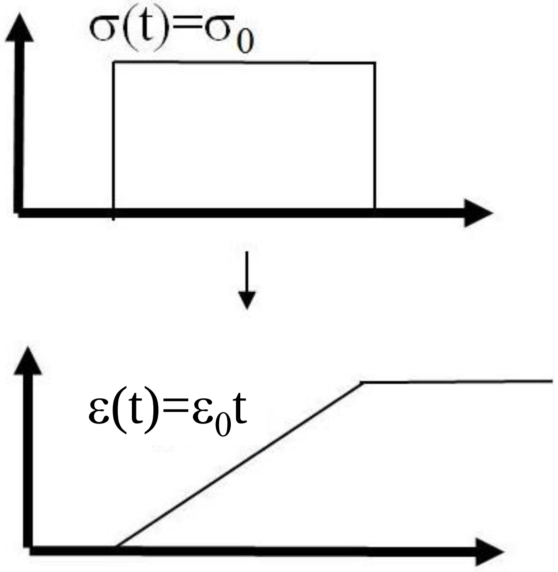
La viscosité

Loi de Newton
En traction : $\boldsymbol{\sigma = \eta\dot{\varepsilon}}$
En cisaillement : $\boldsymbol{\sigma = \eta\dot{\gamma}}$

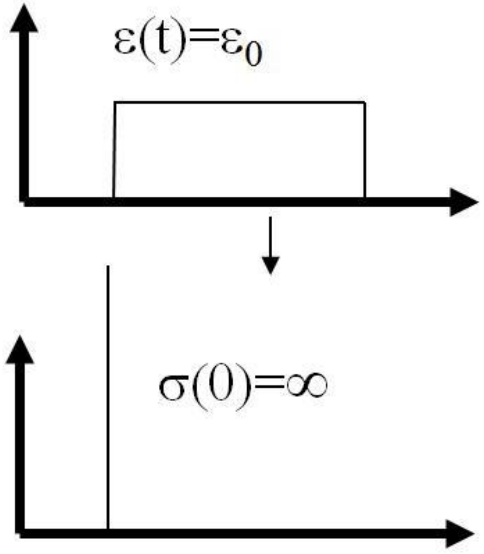
$\boldsymbol{\eta}$: Viscosité

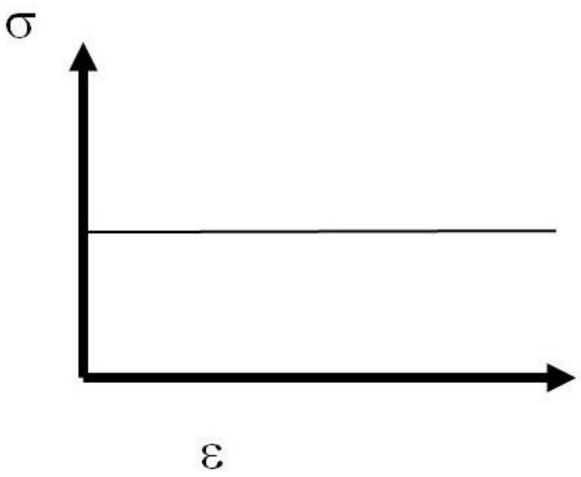


Essai de fluage



Essai de relaxation

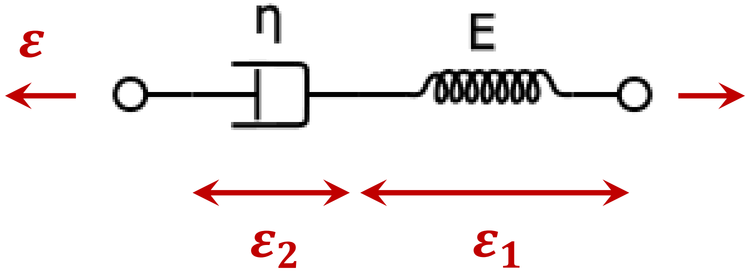




Essai de traction monotone

La viscoélasticité

Et la viscoélasticité alors ?



$$\sigma = \sigma_1 = \sigma_2 \qquad \sigma_1 = E\varepsilon_1$$

$$\varepsilon = \varepsilon_1 + \varepsilon_2 \qquad \sigma_2 = \eta\dot{\varepsilon}_2$$

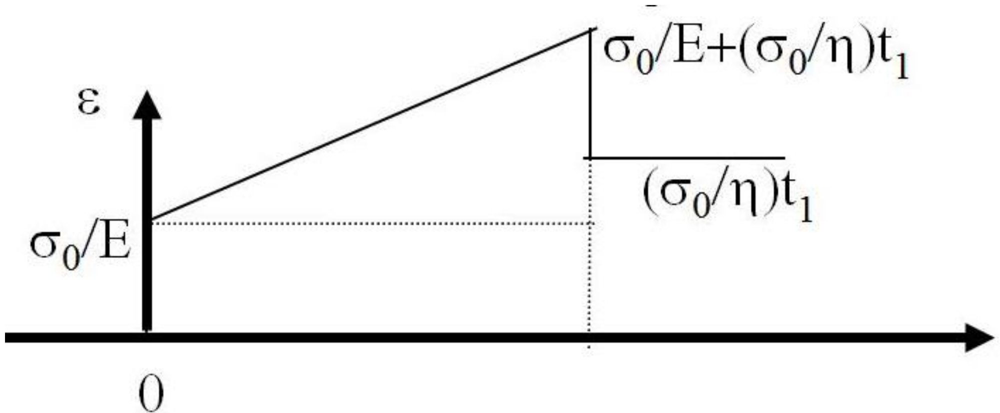
$$\dot{\varepsilon} = \frac{d\varepsilon}{dt} = \frac{1}{E}\frac{d\sigma}{dt} + \frac{1}{\eta}\sigma$$

Essai de Fluage

À $t = 0, \sigma = \sigma_0$ et $\varepsilon = \frac{\sigma_0}{E}$

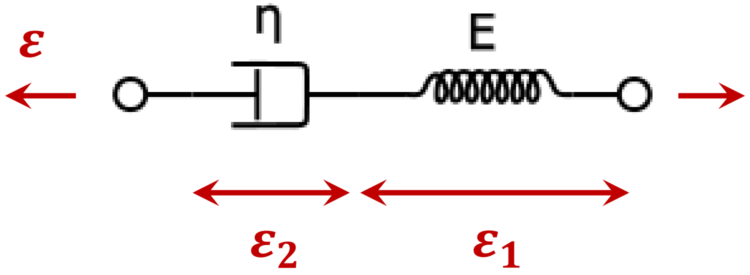
À $0 < t < t_1$ $\quad \varepsilon(t) = \frac{\sigma_0}{\eta}t + \frac{\sigma_0}{E}$

À $t > t_1$ $\quad \varepsilon_1 = 0 \quad \varepsilon_2 = \frac{\sigma_0}{\eta}t_1$



La viscoélasticité

Et la viscoélasticité alors ?



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$$\varepsilon = \varepsilon_1 + \varepsilon_2 \qquad \sigma_2 = \eta\dot{\varepsilon_2}$$

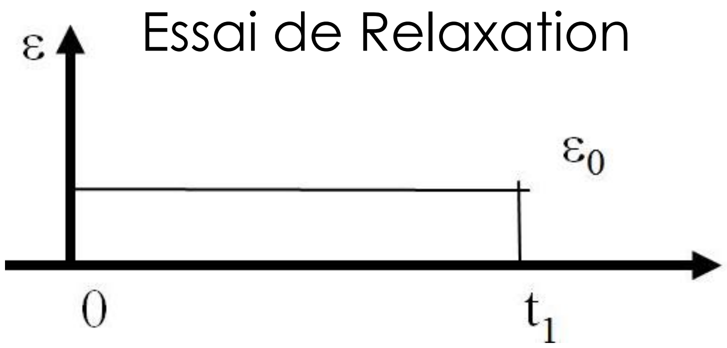
$$\dot{\varepsilon} = \frac{d\varepsilon}{dt} = \frac{1}{E}\frac{d\sigma}{dt} + \frac{1}{\eta}\sigma$$

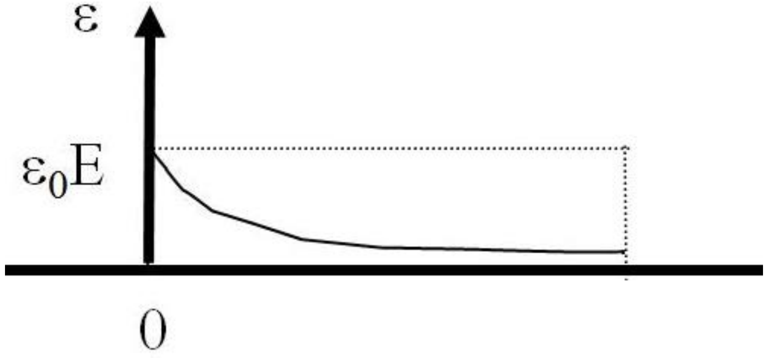
Avec $\frac{d\varepsilon}{dt} = 0 \rightarrow \sigma(t) = Ae^{-\frac{t}{\tau}}$

À $t = 0, \sigma = \varepsilon_0 E \rightarrow A = \varepsilon_0 E$

$$E_{eq}(t) = \frac{\sigma_0}{\varepsilon} = Ee^{-\frac{t}{\tau}}$$

$\tau = \frac{\eta}{E}$: temps de relaxation


Essai de Relaxation



La viscoélasticité

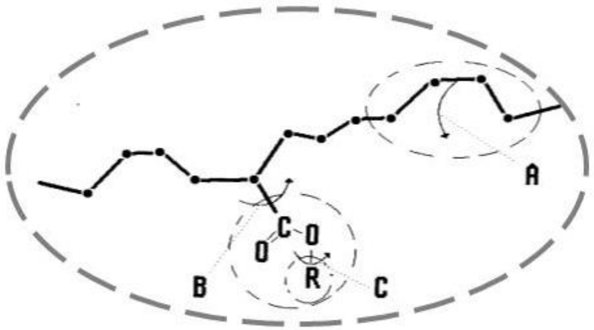
$$E(t) = E_0 e^{-\frac{t}{\tau}}$$

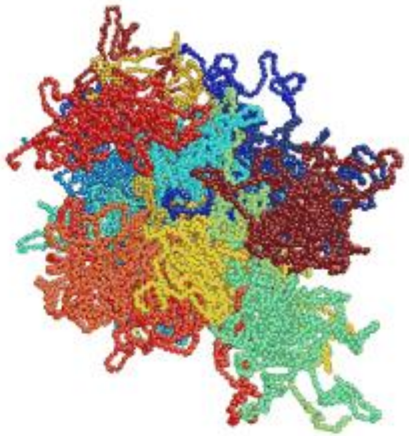
Et la viscoélasticité alors ?

Temps de relaxation ➔ « mécanisme relaxationnel » ou relaxation

Lorsque le matériau est soumis à une sollicitation mécanique, les phénomènes intervenant au sein du matériau ont besoin d'un certain temps pour accommoder la contrainte.

Pour quoi du temps ?



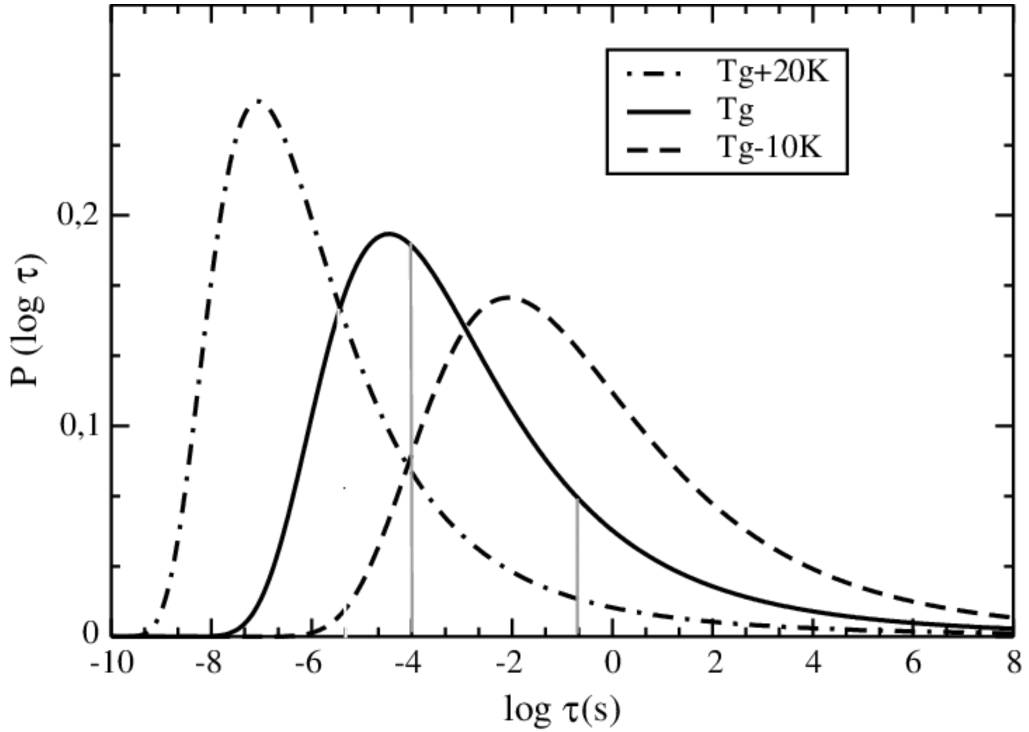


Le concept de relaxation est rencontré dès qu'un temps est nécessaire avant le retour à un équilibre.

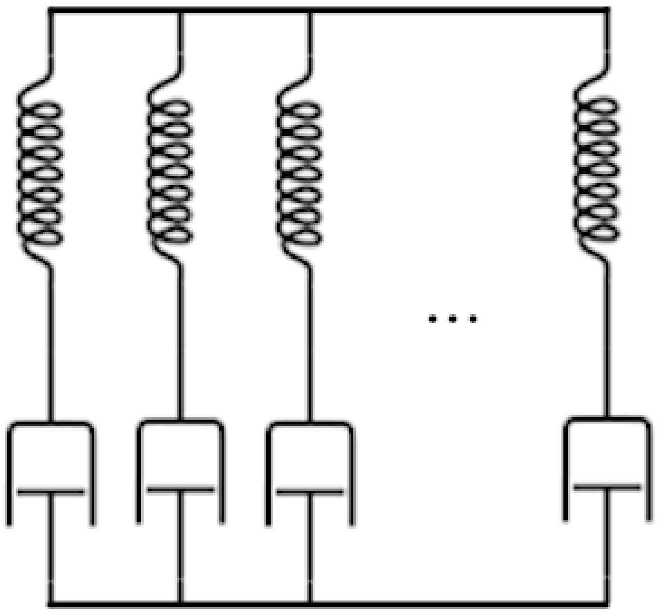
La viscoélasticité

Comment décrire ce plat de serpent viscoélastique ?

Spectre de relaxation d'un polystyrène



Modèle de Maxwell généralisé



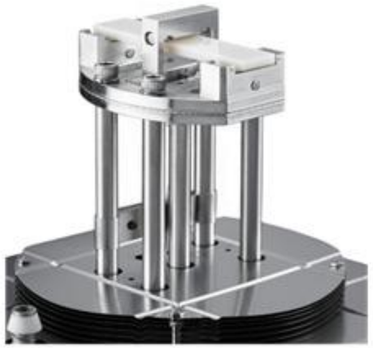
La spectrométrie mécanique ou DMA

Comment mesurer les propriétés viscoélastiques ?

S/D Cantilever

Film/Fiber Tension

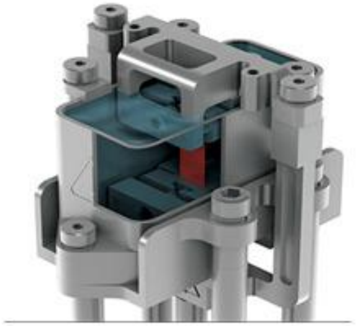
3-Point Bending



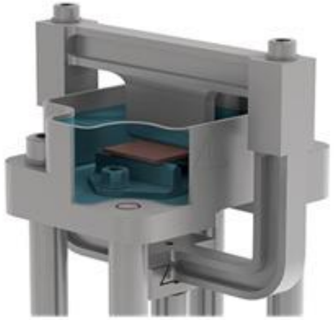
Compression

Shear Sandwich

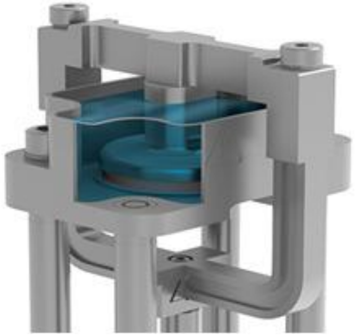
Submersible Tension



Submersible Bending



Submersible Compression



Analyse
Mécanique
Dynamique
(DMA en anglais)

La spectrométrie mécanique ou DMA

f : 10^{-2}Hz → 10^{2}Hz

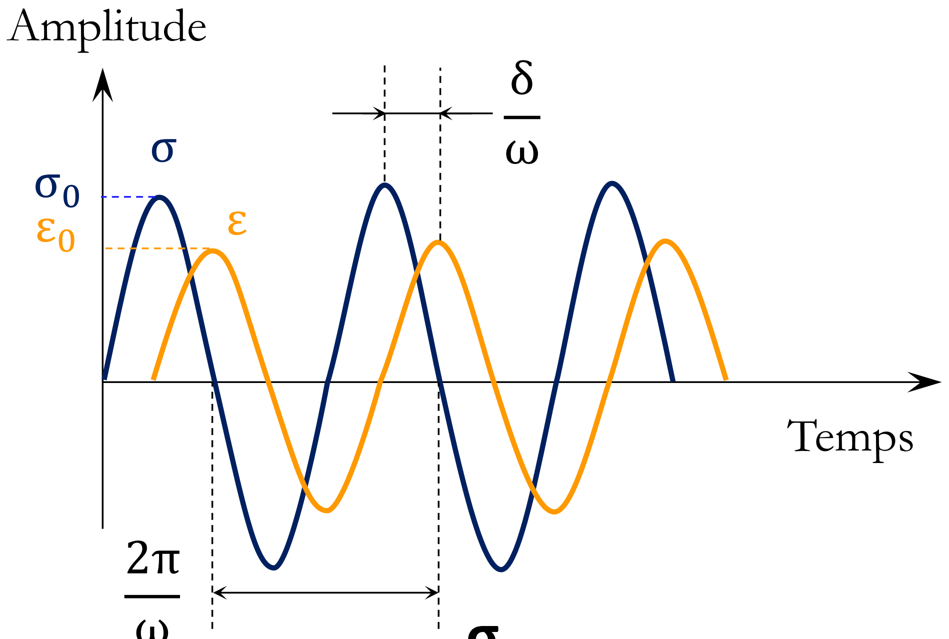
- Déformation sinusoïdale imposée : $\varepsilon^* = \varepsilon_0 e^{i\omega t}$
- Contrainte déphasée : $\sigma^* = \sigma_0 e^{i(\omega t+\delta)}$

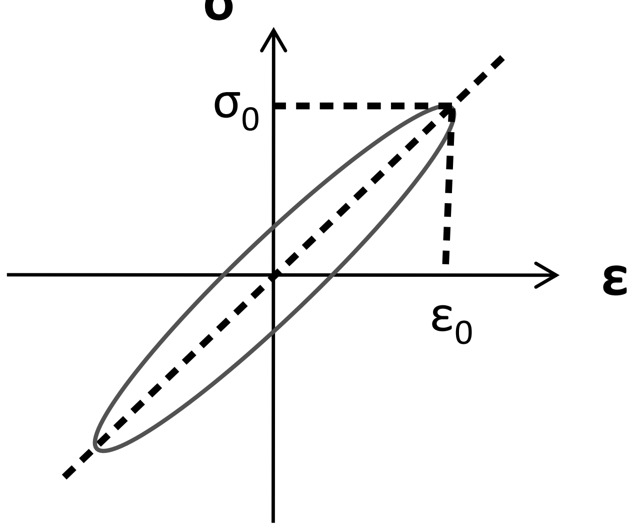
⇒ **Module d'Young complexe :** $E^* = \dfrac{\sigma^*}{\varepsilon^*} = E' + iE''$

E ' = Module de conservation (élasticité)
E '' = Module de perte (anélasticité)

⇒ **Facteur de perte :** $tan(\delta) = \dfrac{E''}{E'}$

Module complexe en fonction de la **température** (isochrone) ou de la **fréquence** (isotherme) de sollicitation

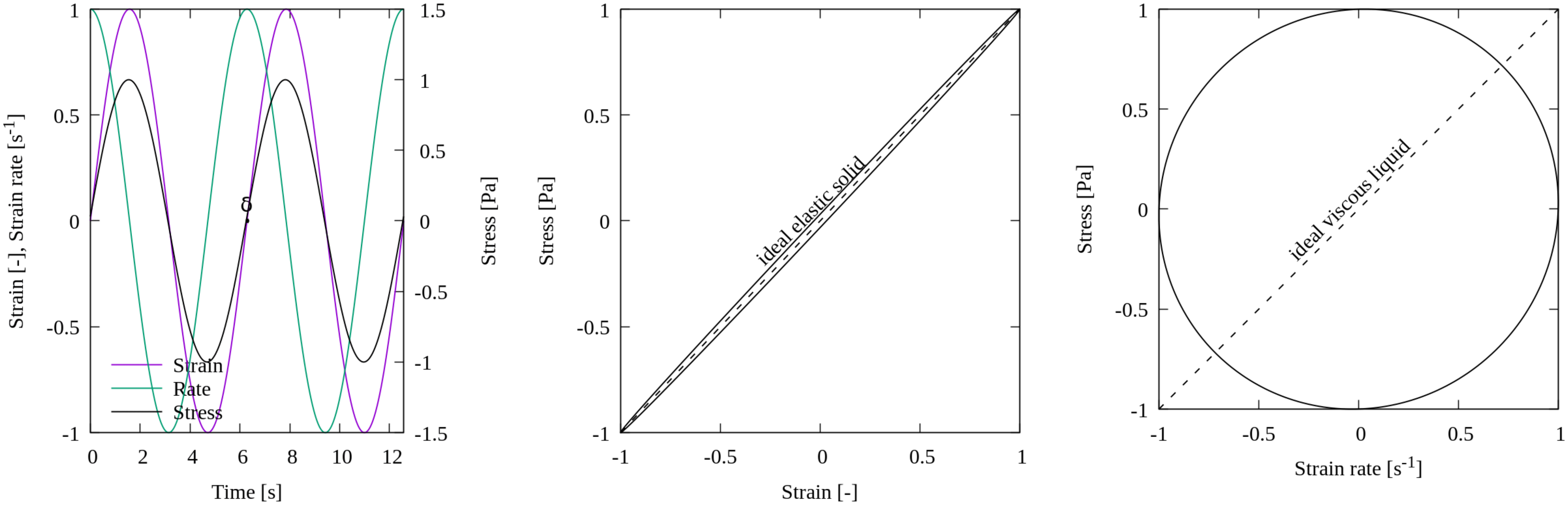




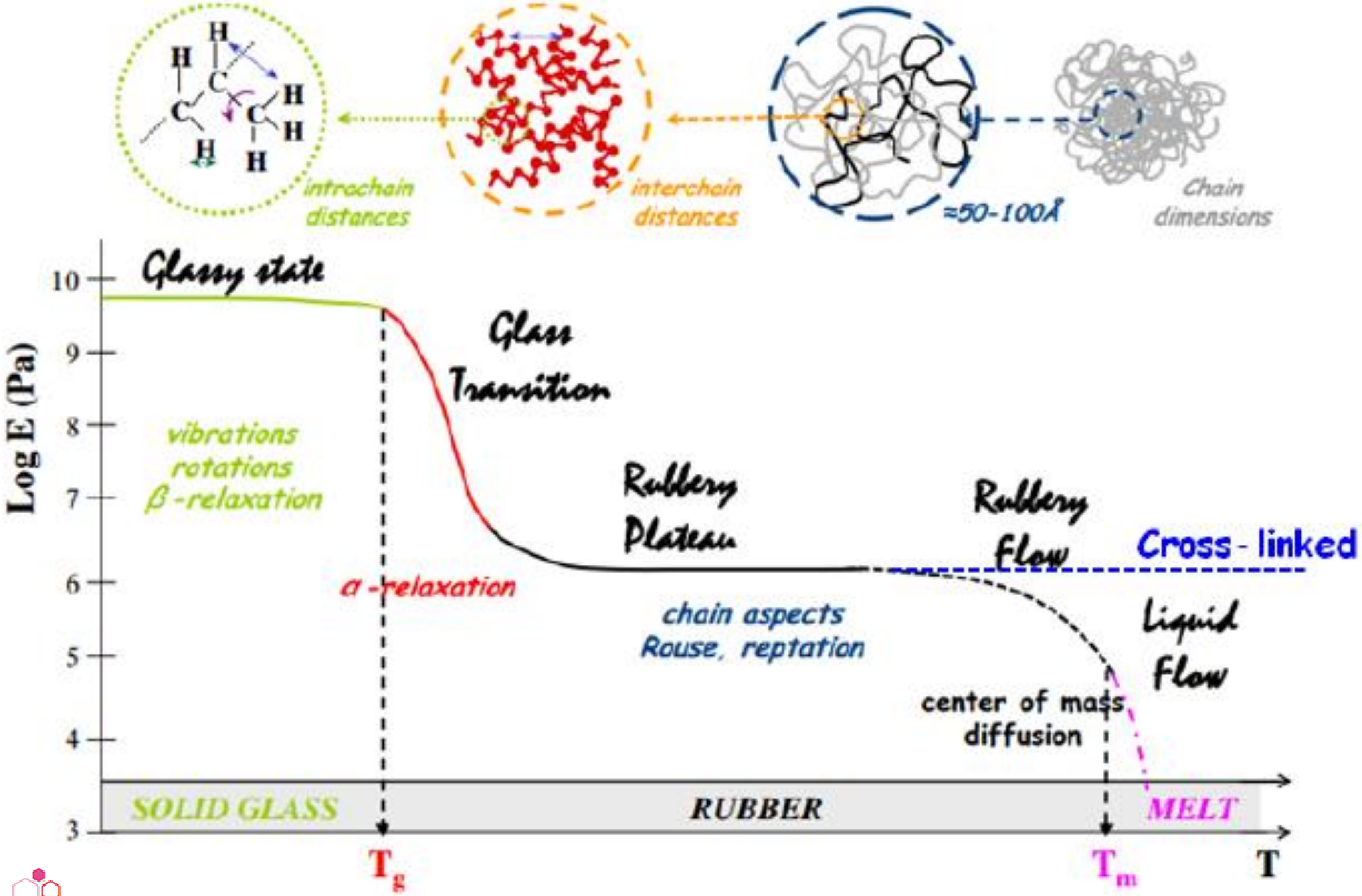
La spectrométrie mécanique ou DMA

$\tan \delta = G''/G' = 0.03$

more elastic

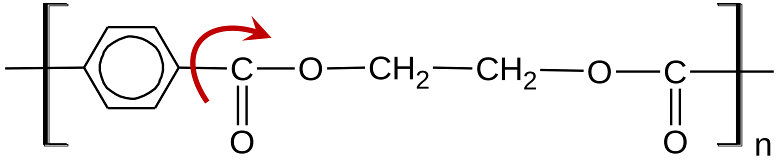


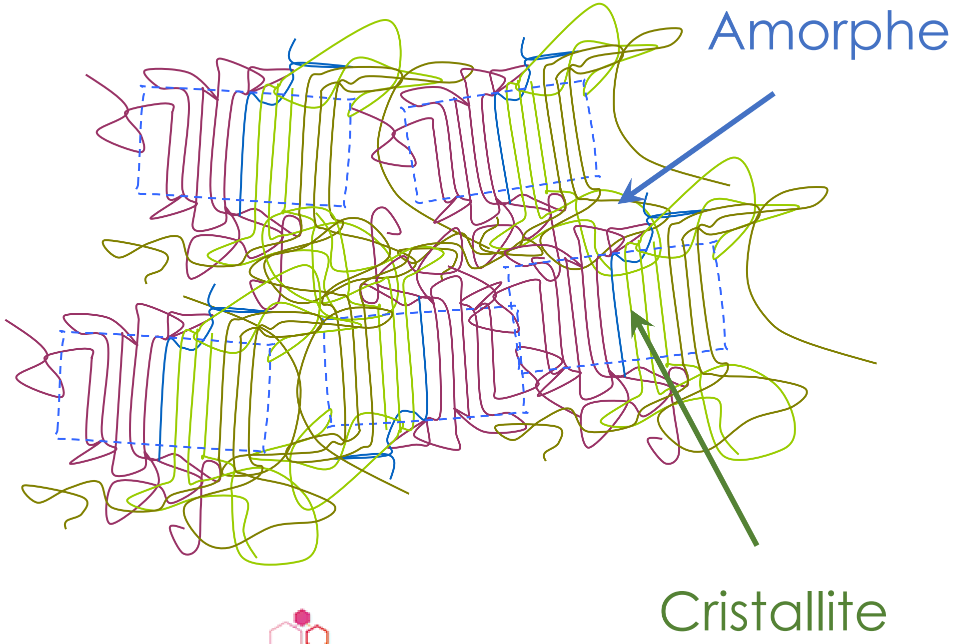
La spectrométrie mécanique ou DMA

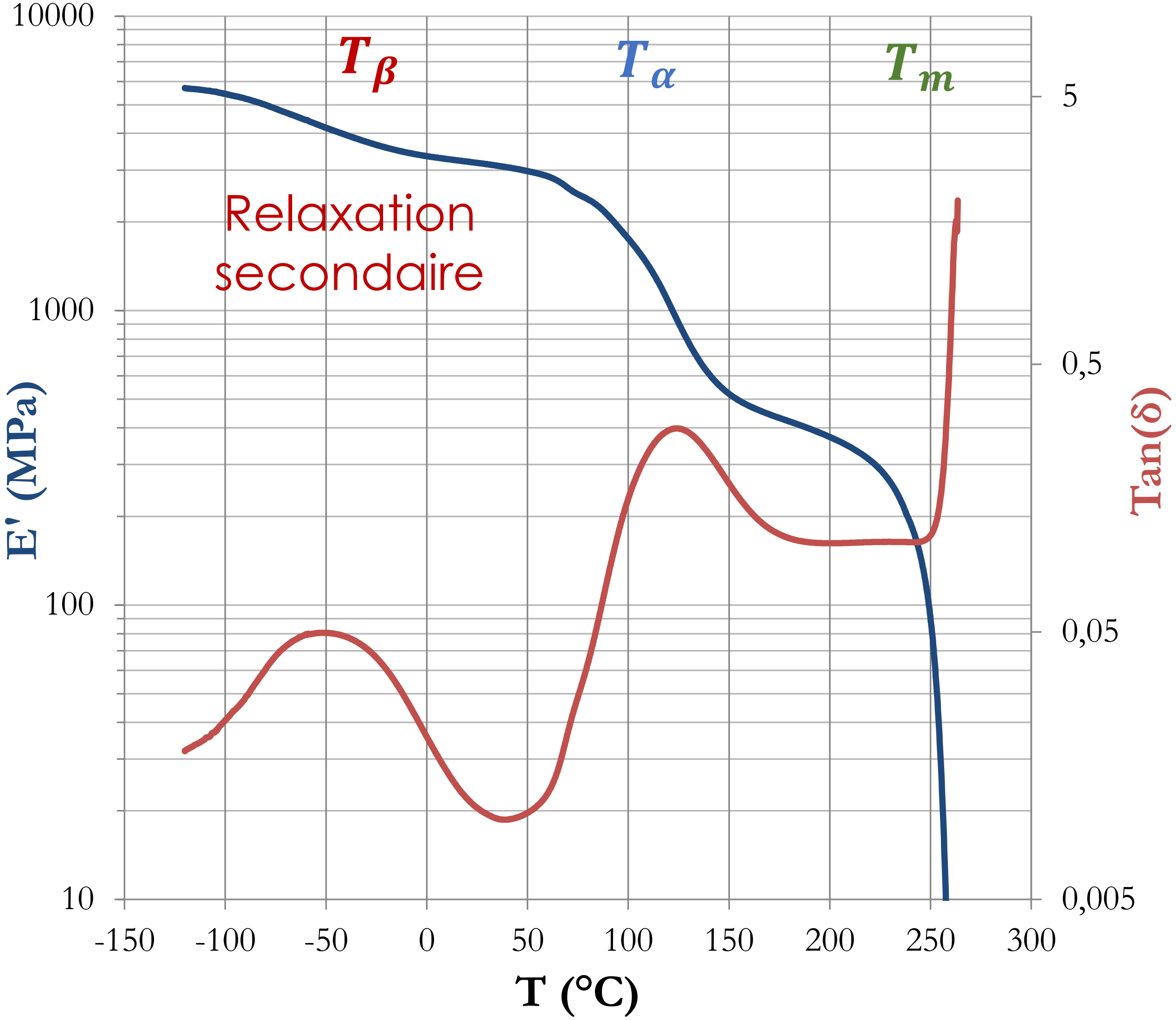


La spectrométrie mécanique ou DMA

Relaxations dans le polyéthylène téréphtalate (PET)



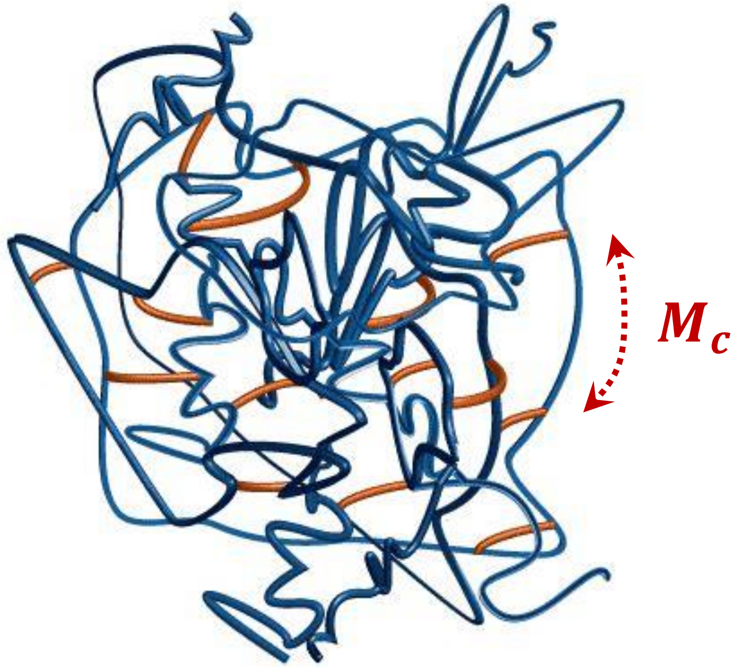


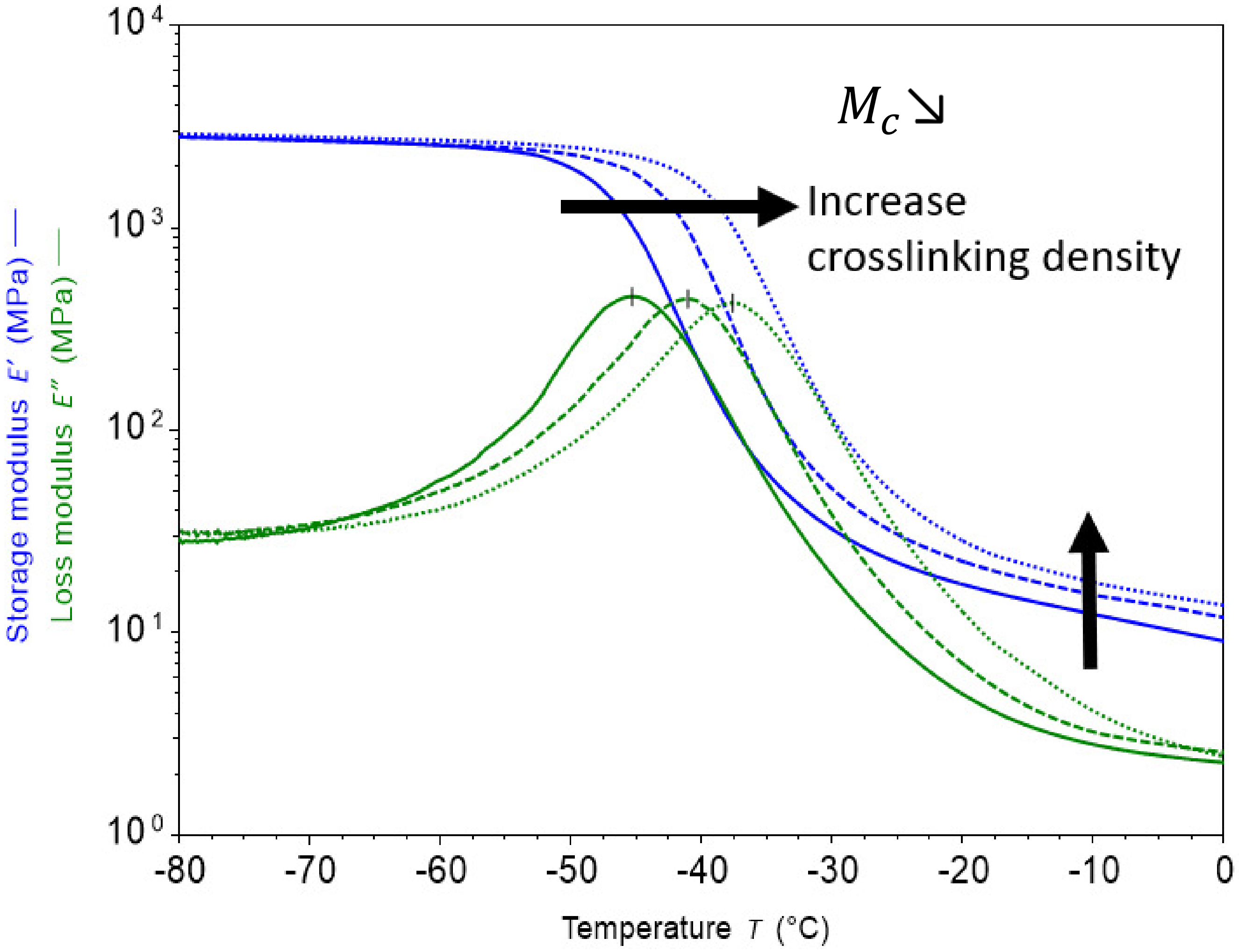


La spectrométrie mécanique ou DMA

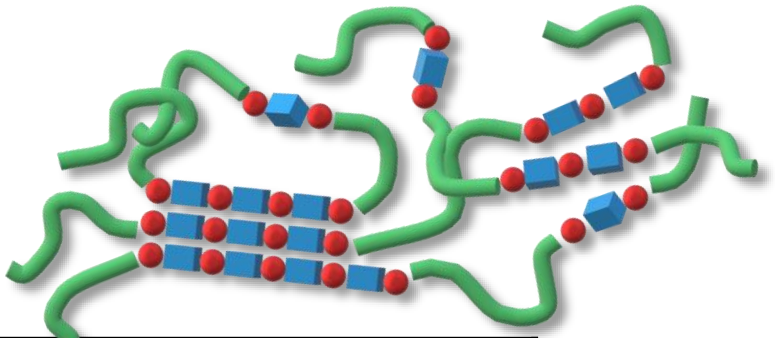
Quelques exemples

Elastomère réticulé : effet de la densité de réticulation

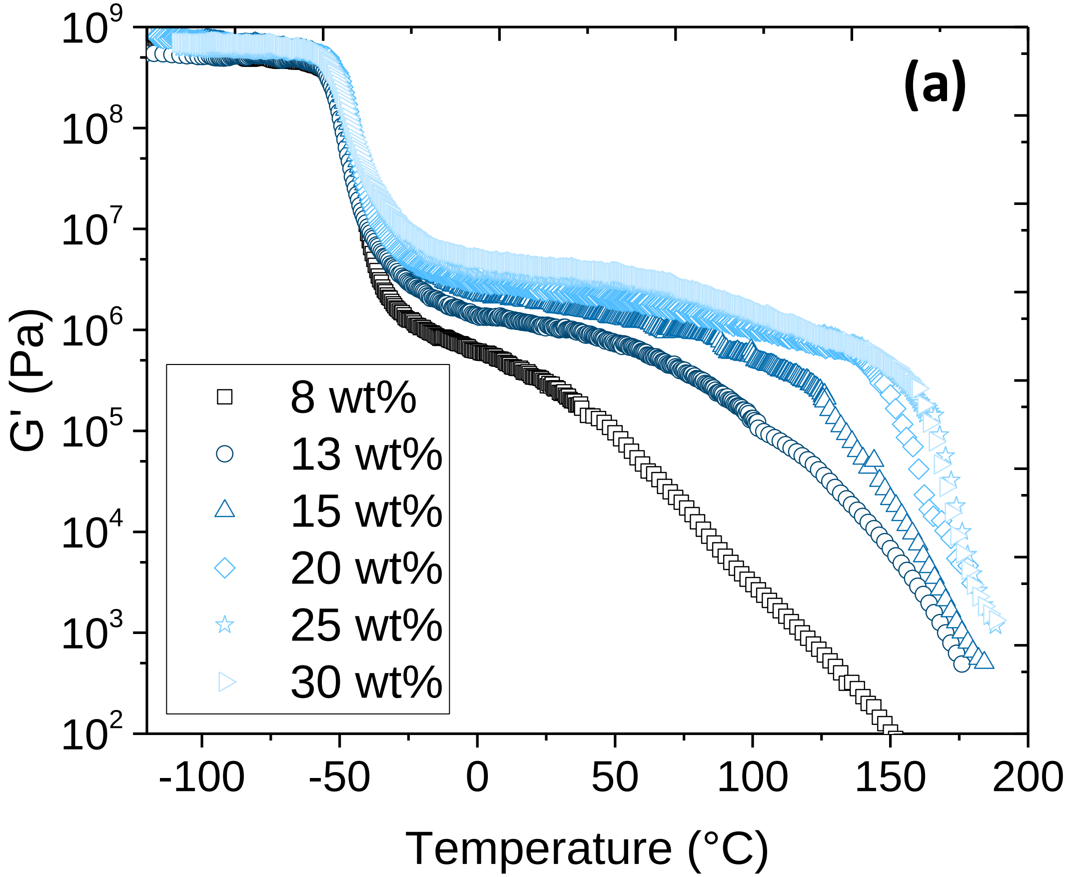


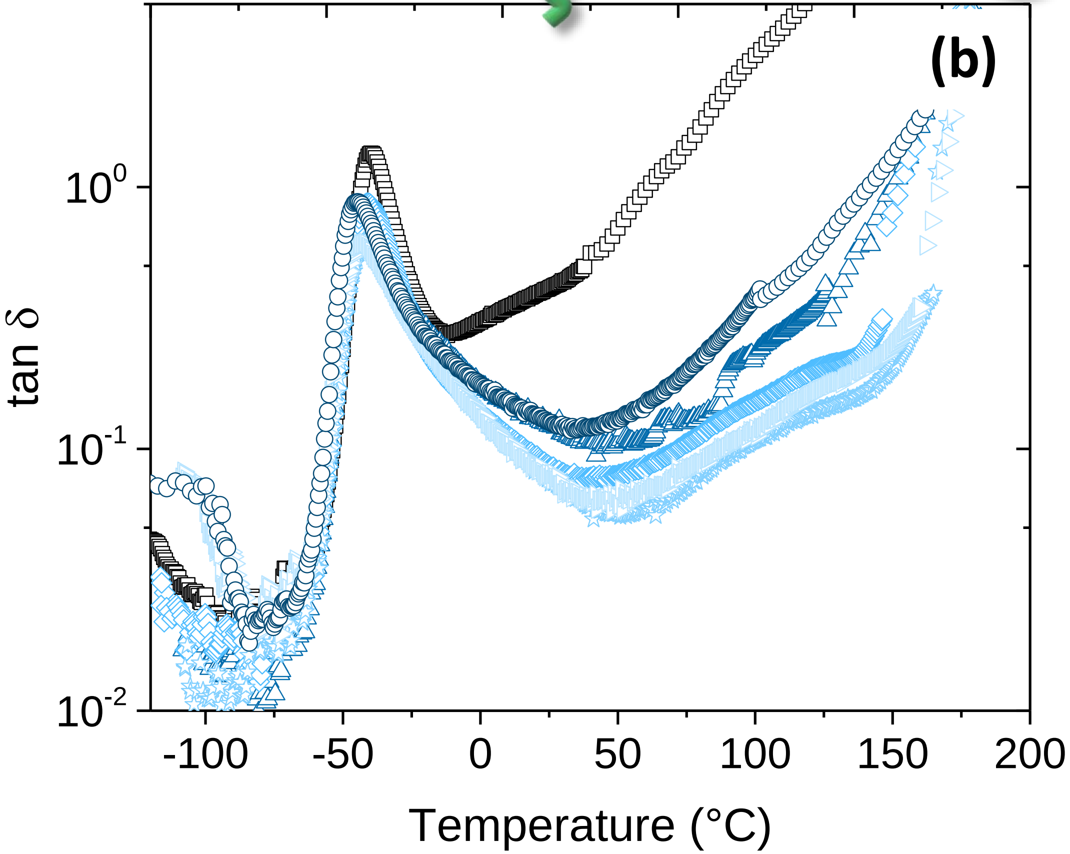


La spectrométrie mécanique ou DMA



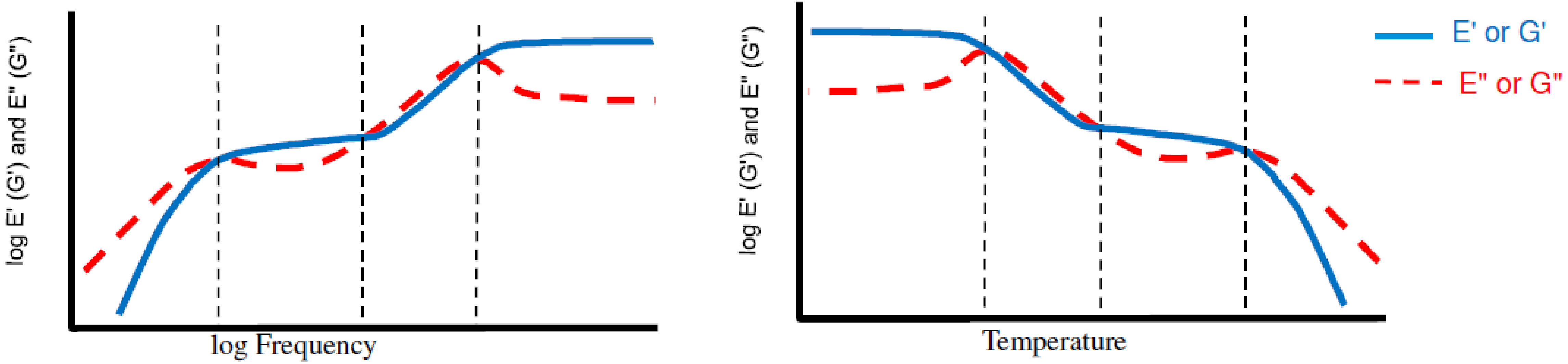
Polyuréthane thermoplastique : effet du taux de segments rigides





R. Gallu et al., *Polymer*, 207, 122882 (2020).

Et l’équivalence temps-température ?

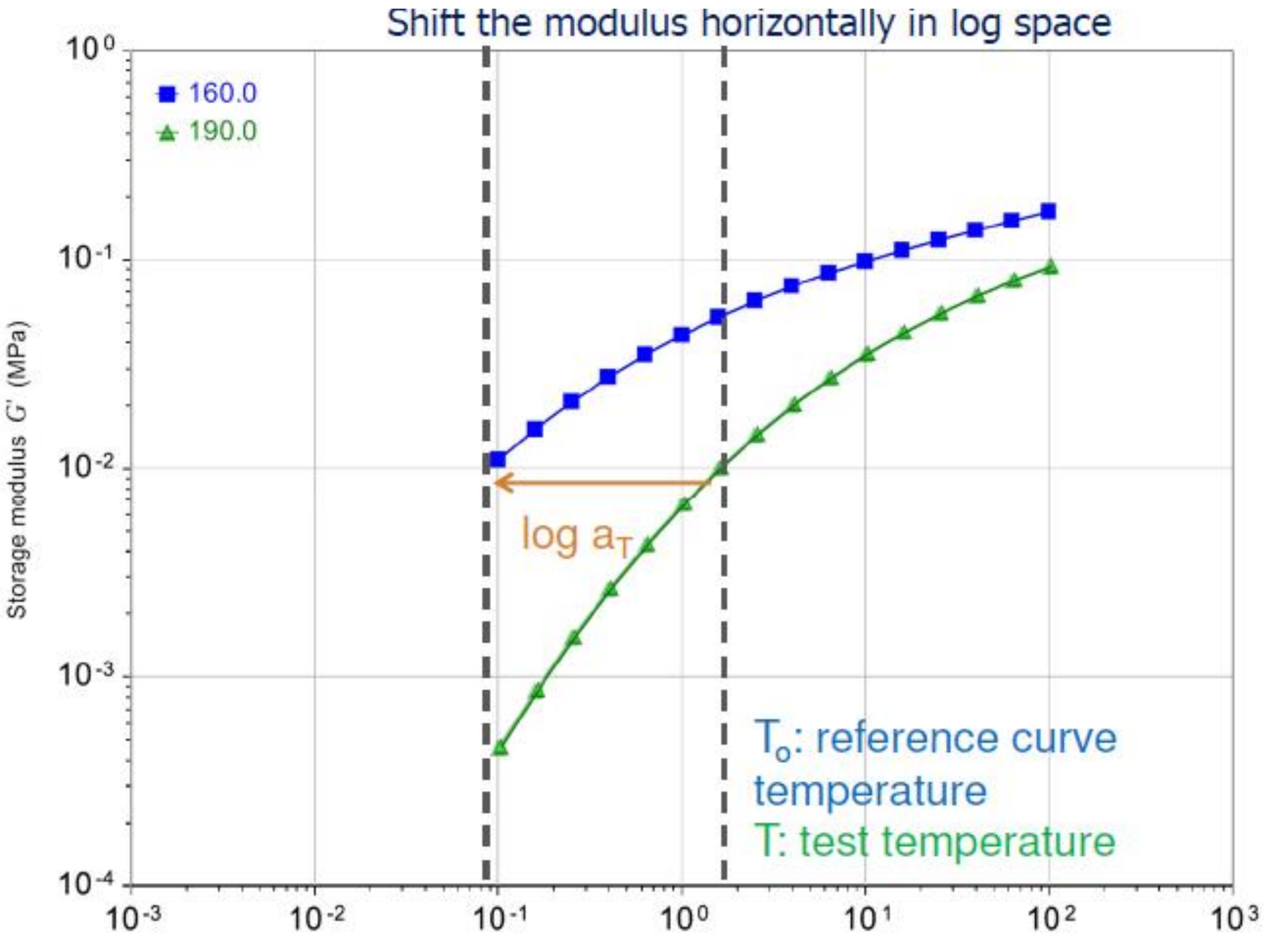


Certains matériaux présentent une dépendance temporelle proportionnelle à la dépendance thermique.

- ➔ Diminuer la température ≡ Augmenter la fréquence et vice versa
- ➔ Pour ces matériaux, les changements de température peuvent être utilisés pour « rééchelonner » le temps et prédire le comportement sur des échelles de temps qui ne sont pas facilement mesurables.

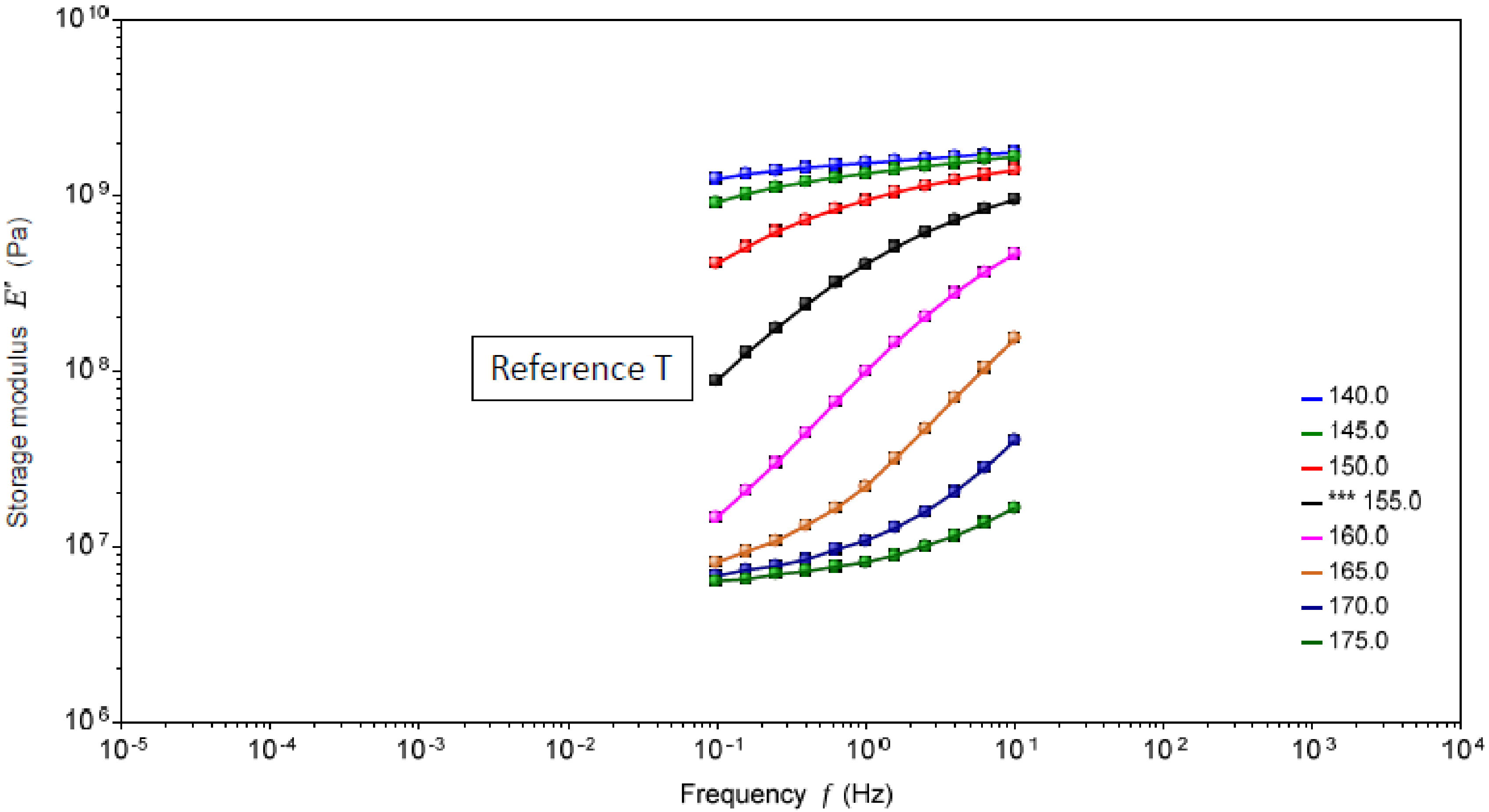
Et l’équivalence temps-température ?

a_T = facteur de glissement



Dealy, J., Plazek, D., *Time-Temperature Superposition – A Users Guide*, Rheology Bulletin, **78**(20) 16 (2009)

Et l’équivalence temps-température ?



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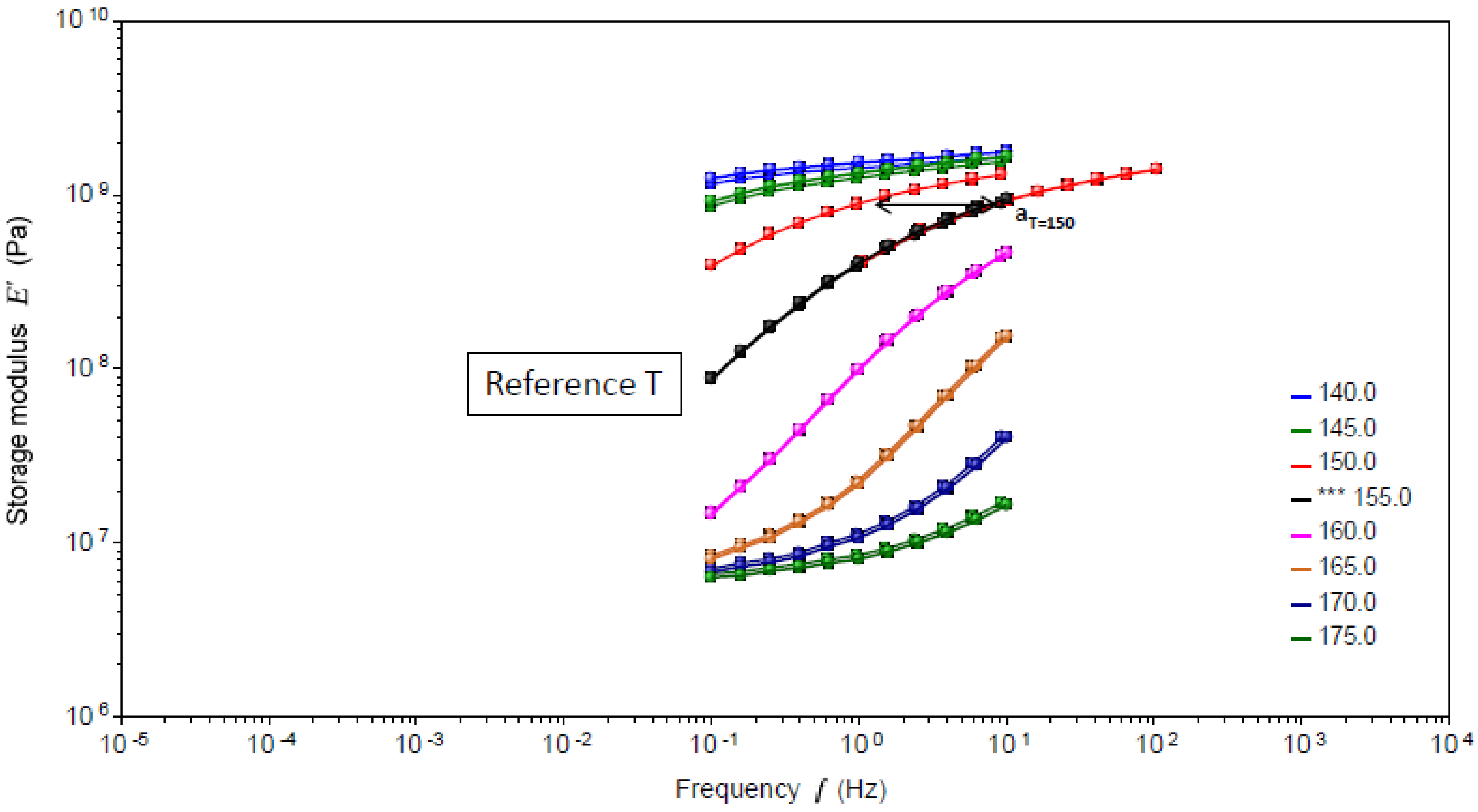

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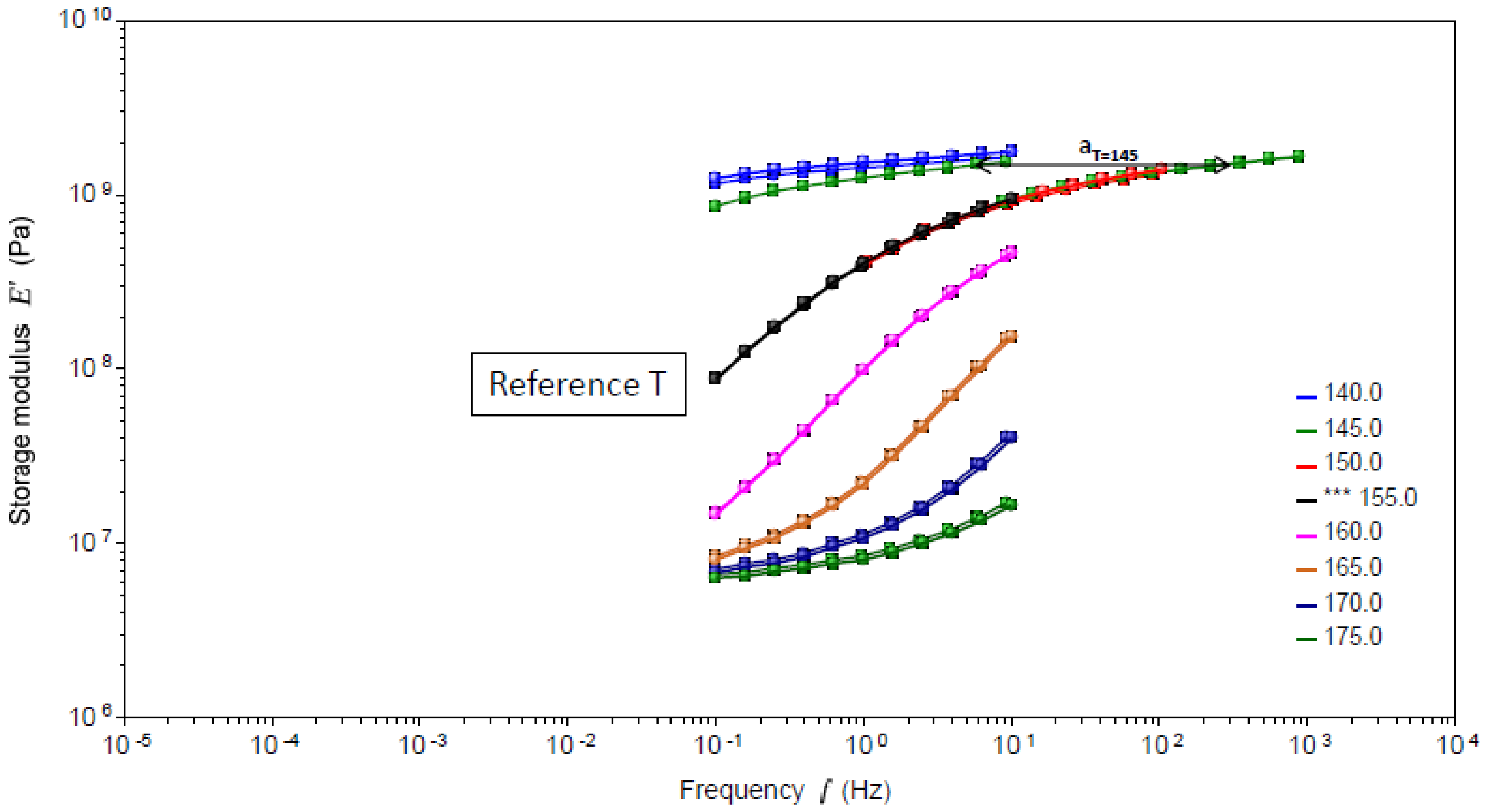

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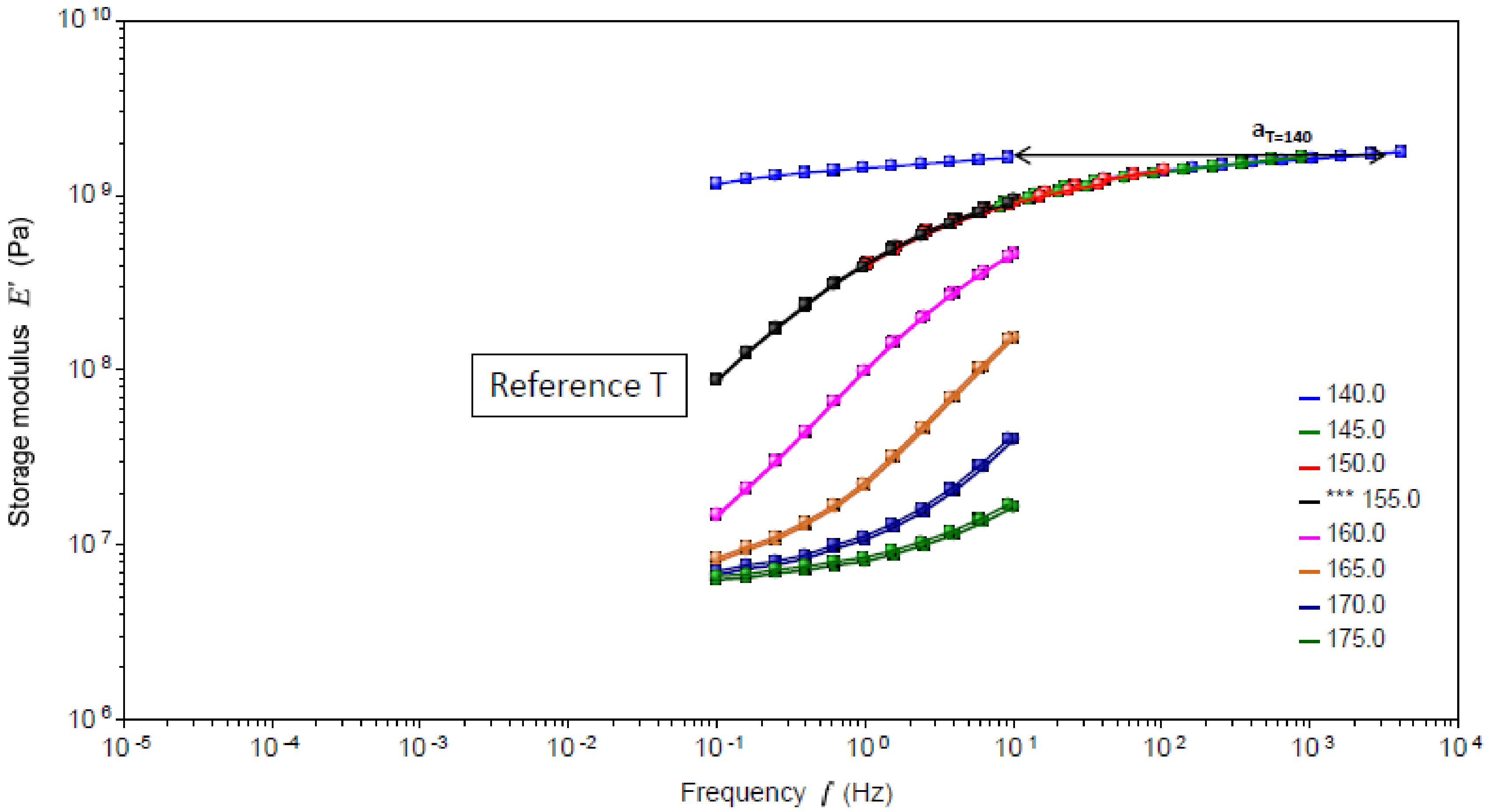

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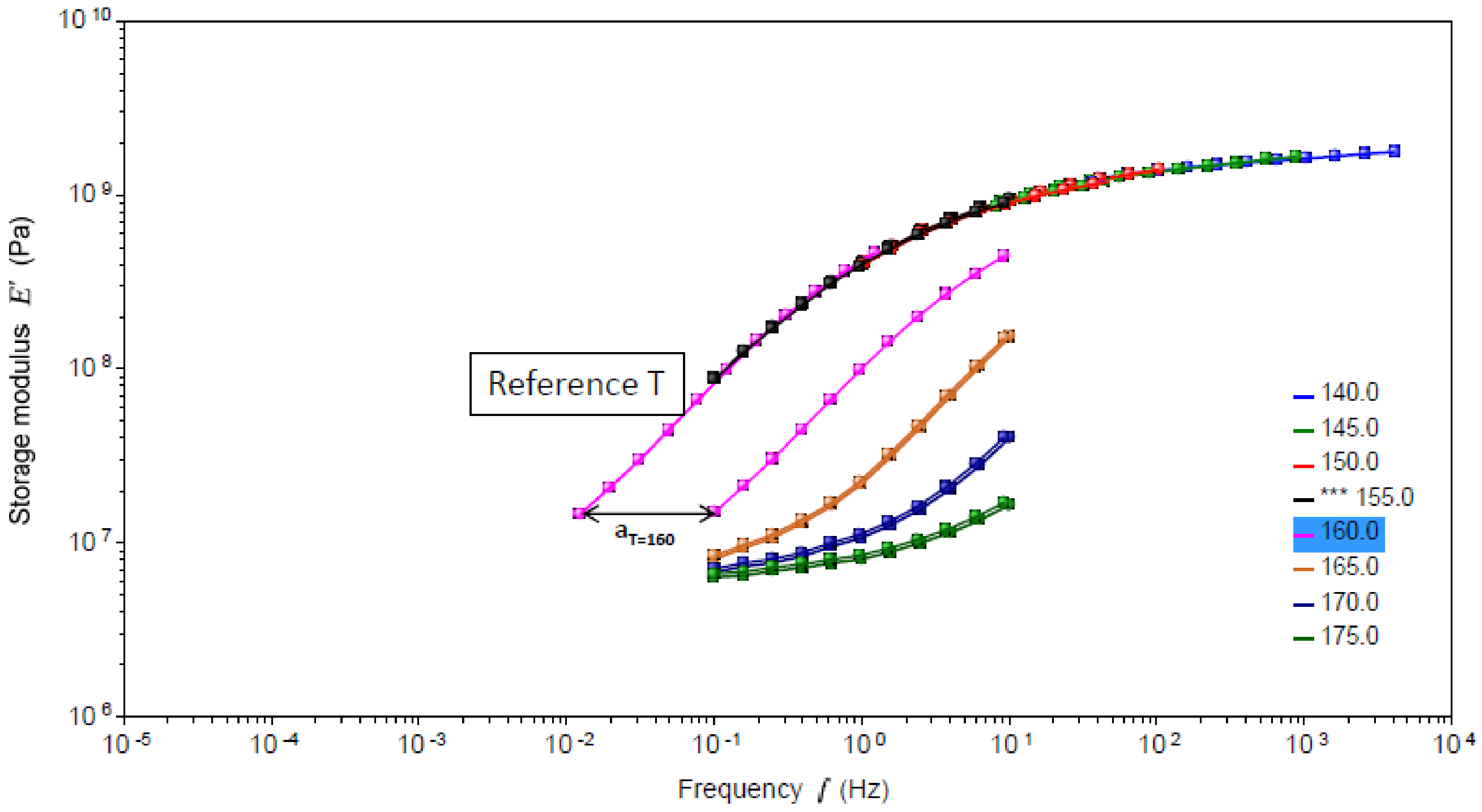

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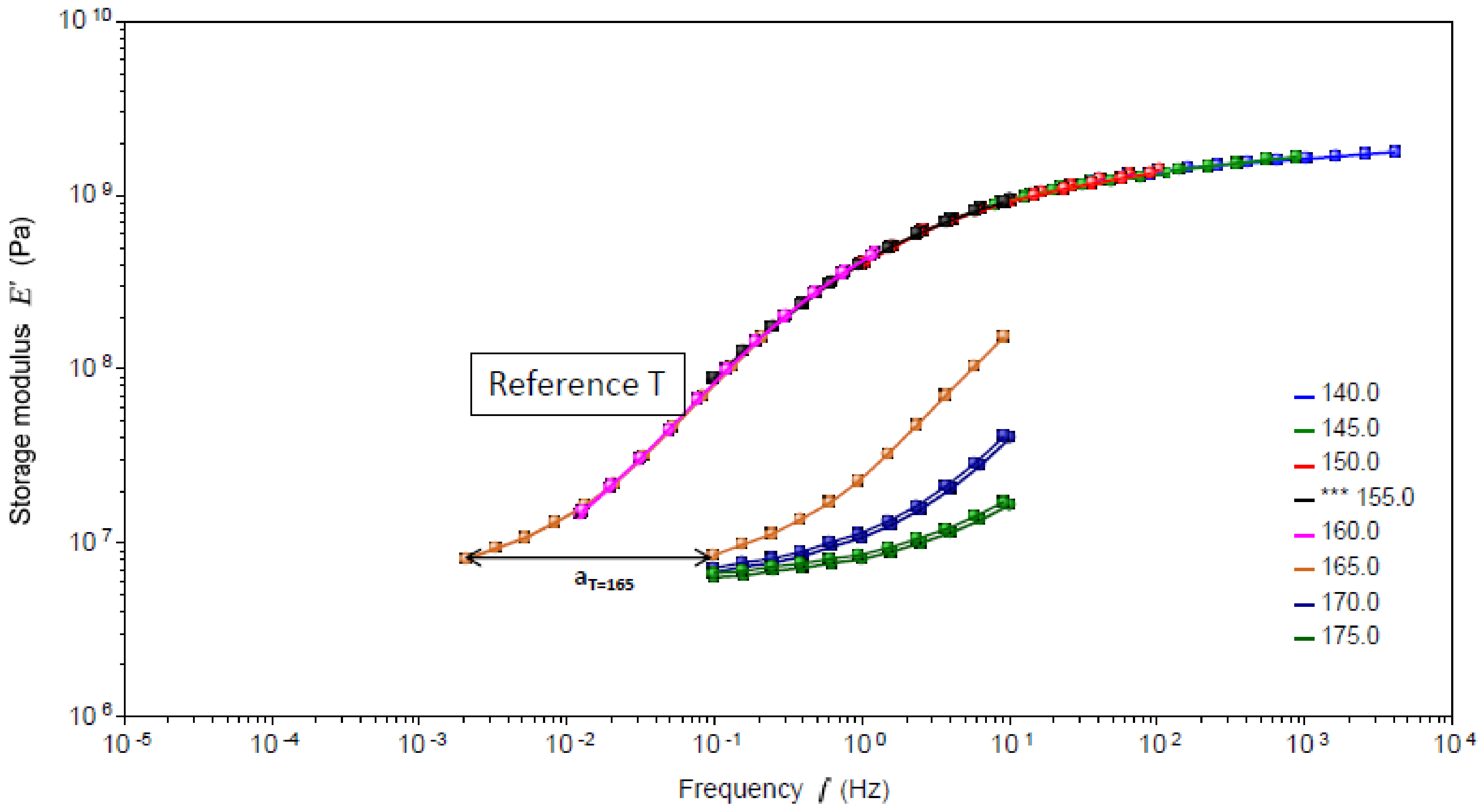

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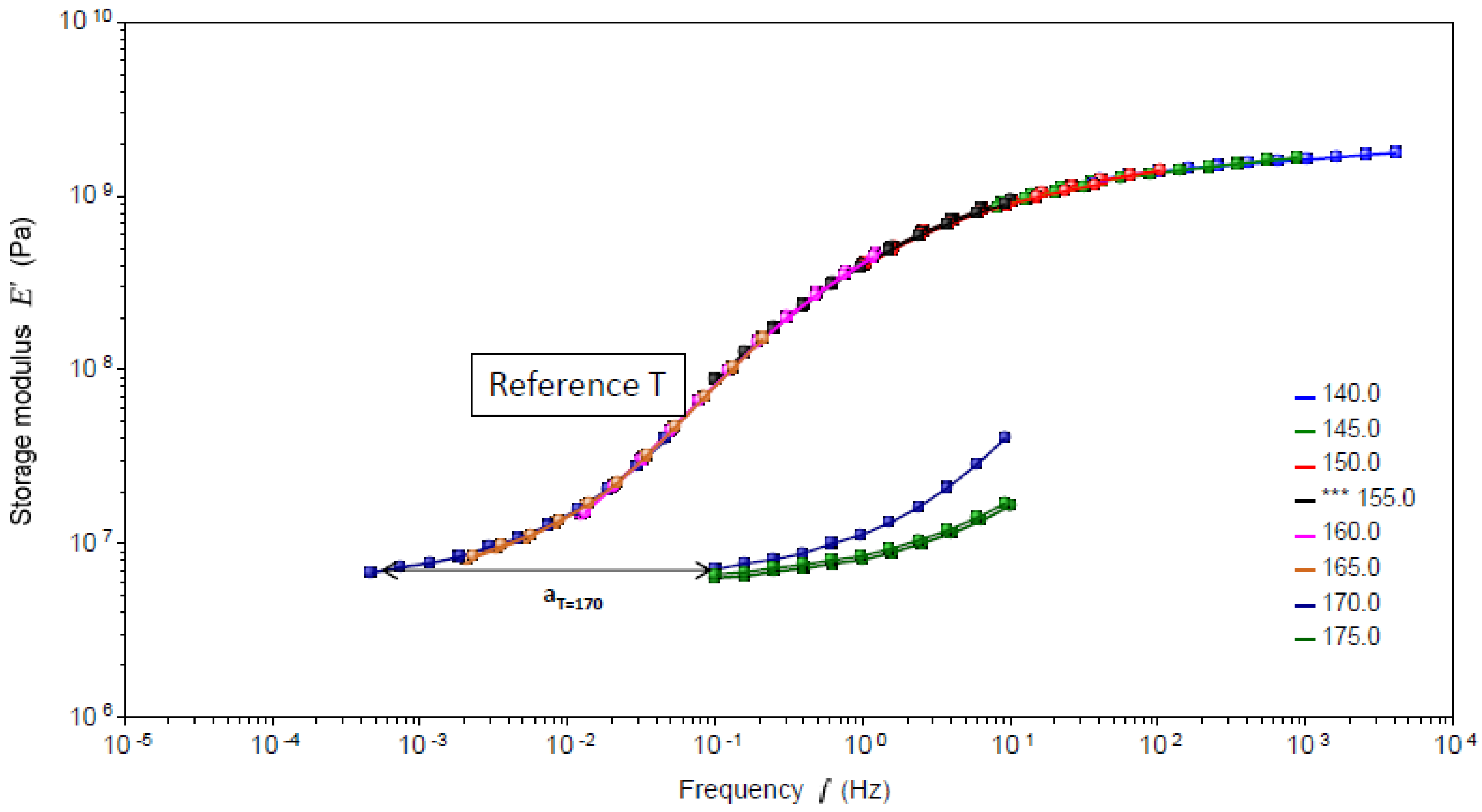

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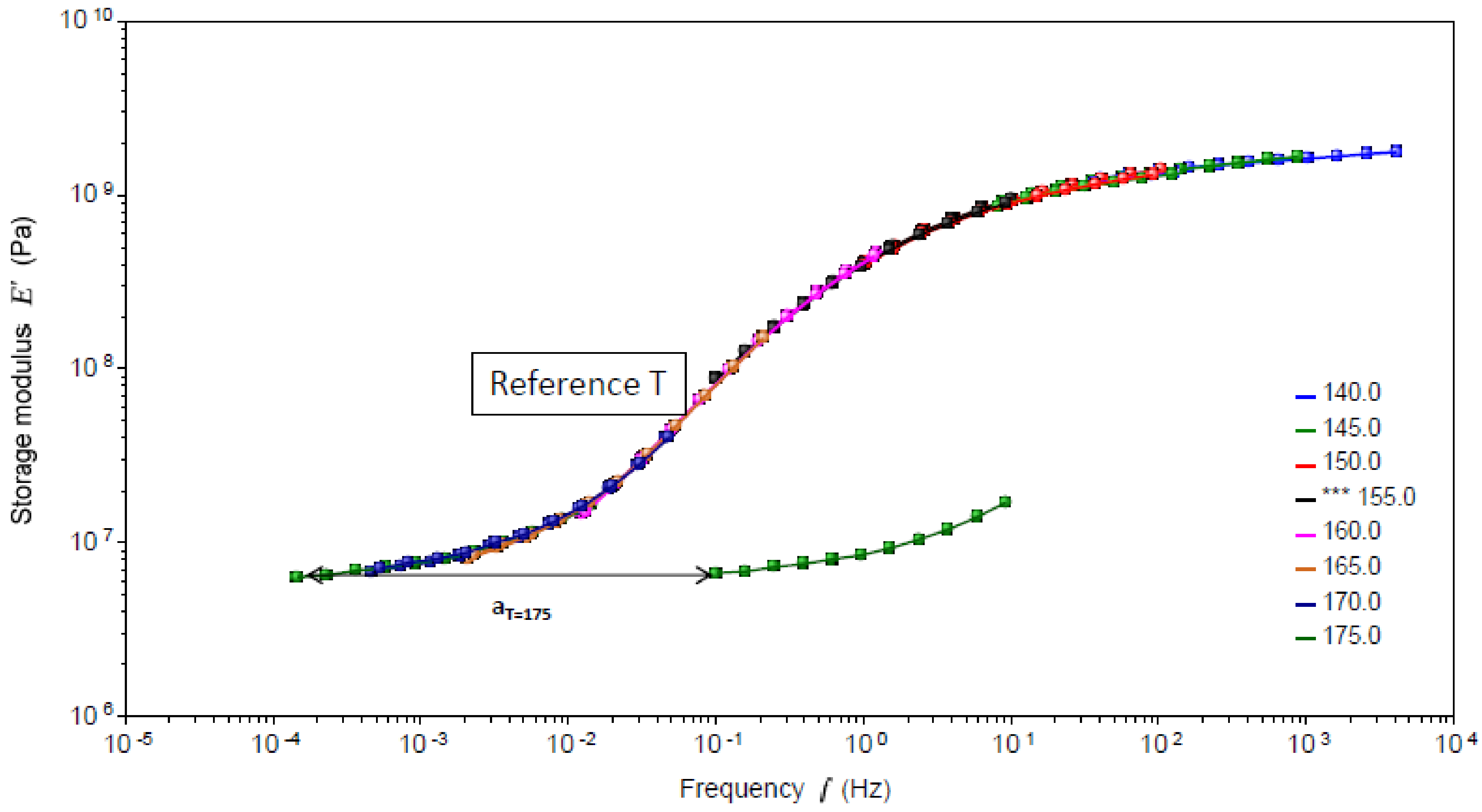

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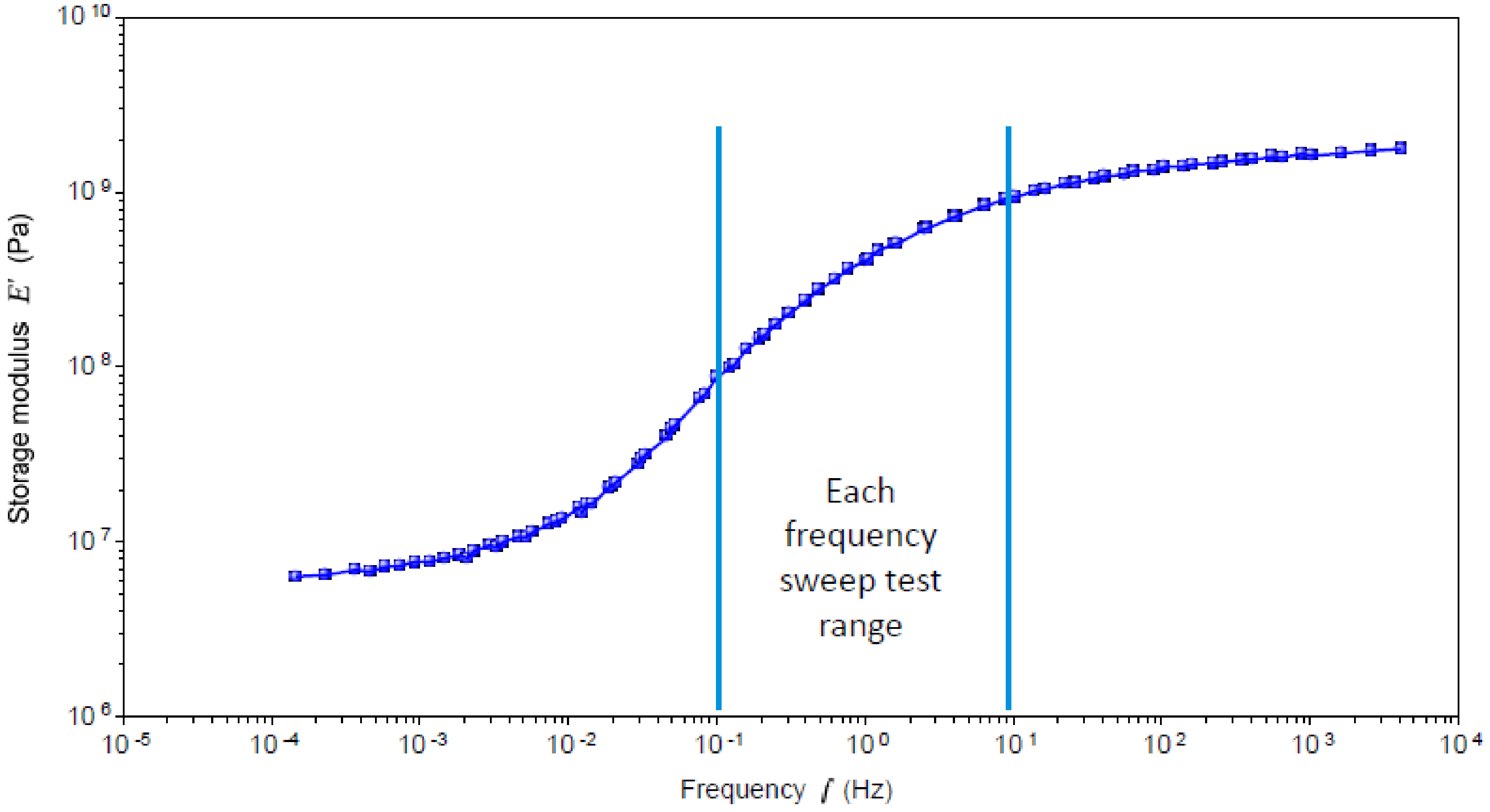

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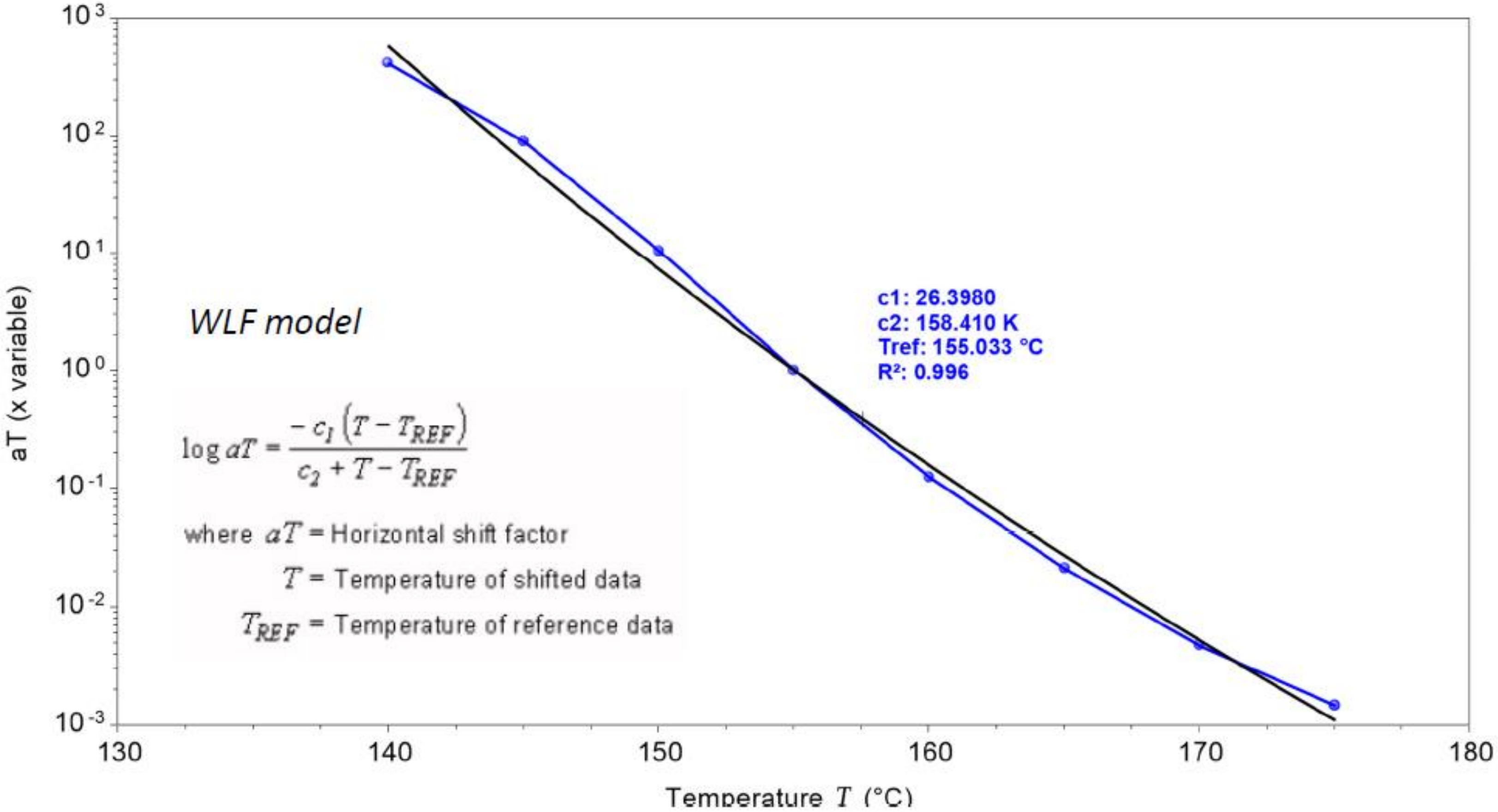
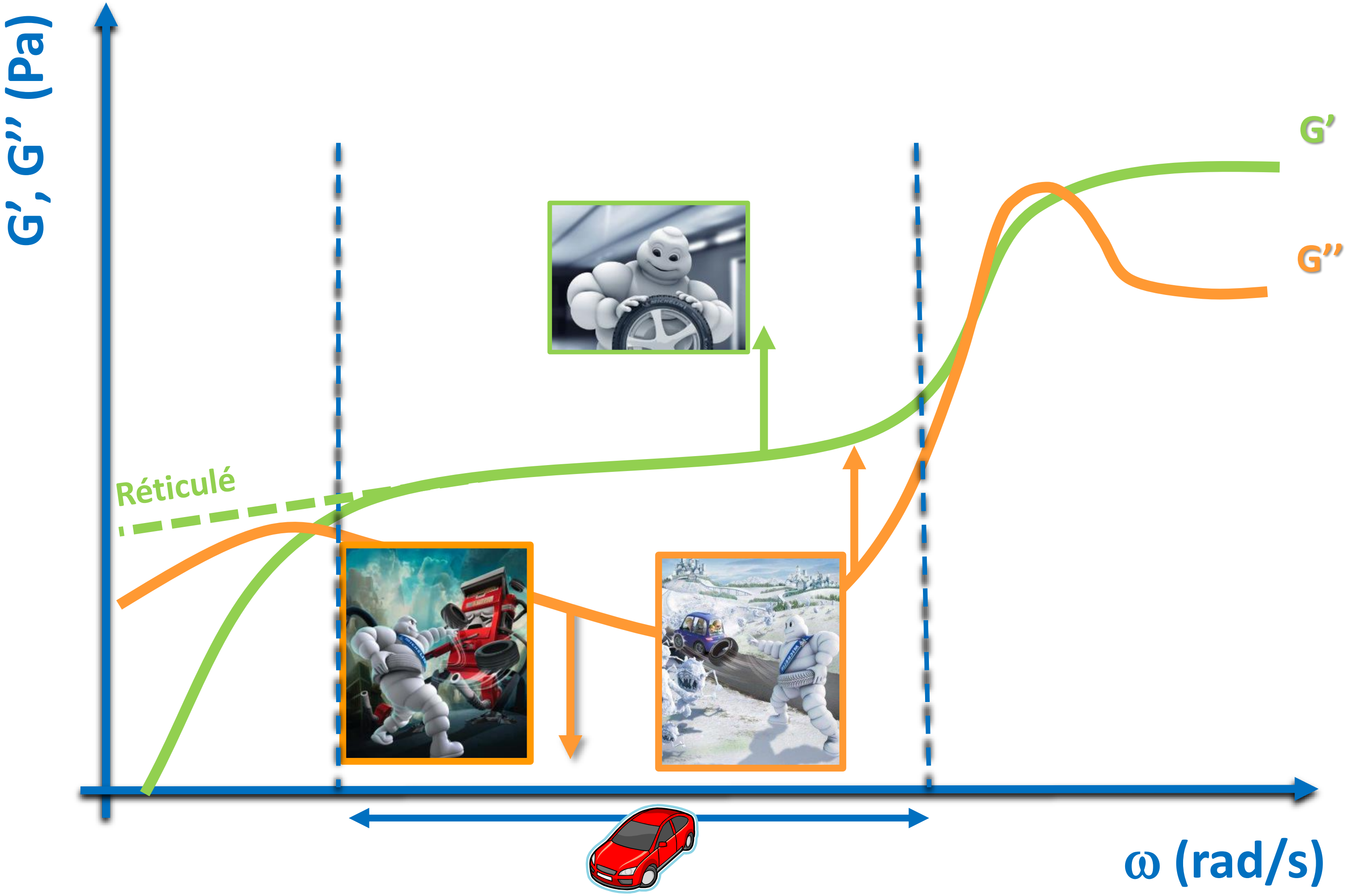
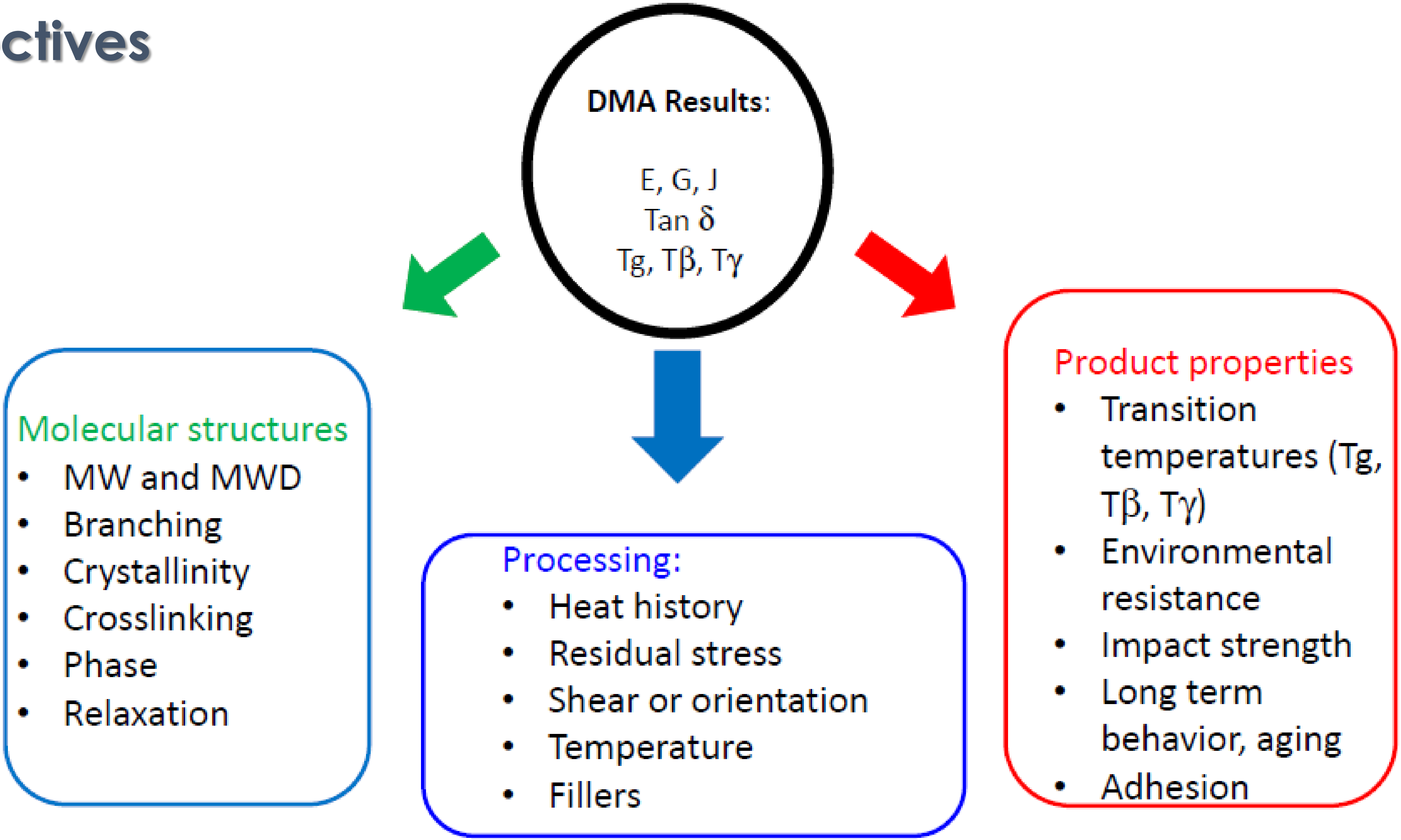


L’évolution du facteur de glissement a_T avec la température permet de remonter à des énergies d’activations des phénomènes de relaxation via des lois de type Arrhenius ou WLF (Williams Landel Ferry).

Et l'équivalence temps-température ?



Perspectives



DMA Results:

E, G, J
Tan δ
Tg, Tβ, Tγ

Molecular structures
- MW and MWD
- Branching
- Crystallinity
- Crosslinking
- Phase
- Relaxation

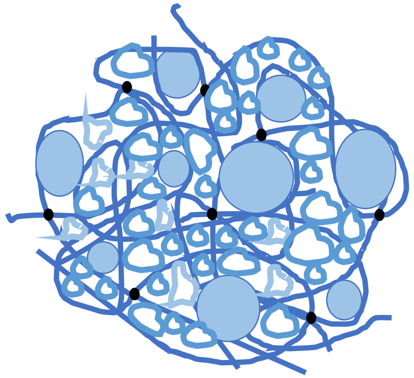
Processing:
- Heat history
- Residual stress
- Shear or orientation
- Temperature
- Fillers

Product properties
- Transition temperatures (Tg, Tβ, Tγ)
- Environmental resistance
- Impact strength
- Long term behavior, aging
- Adhesion

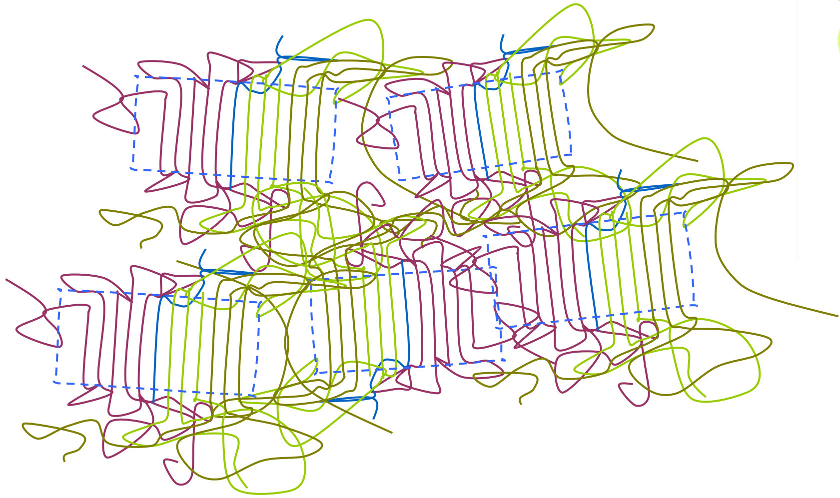
Perspectives

Problématique des polymères hétérogènes : présence d'interfaces et/ou d'interphases + l'équivalence temps-température ne s'applique pas.

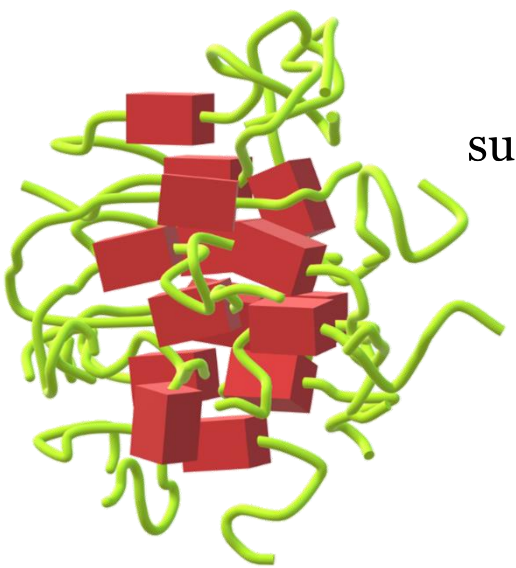
- ➔ Localement, modification de la mobilité moléculaire et des propriétés viscoélastiques
- ➔ Présence d'hétérogénéités de mobilité.



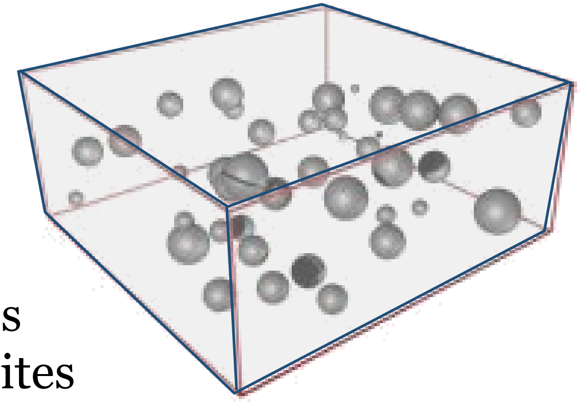
Les mélanges physiques



Polymères semi-cristallins



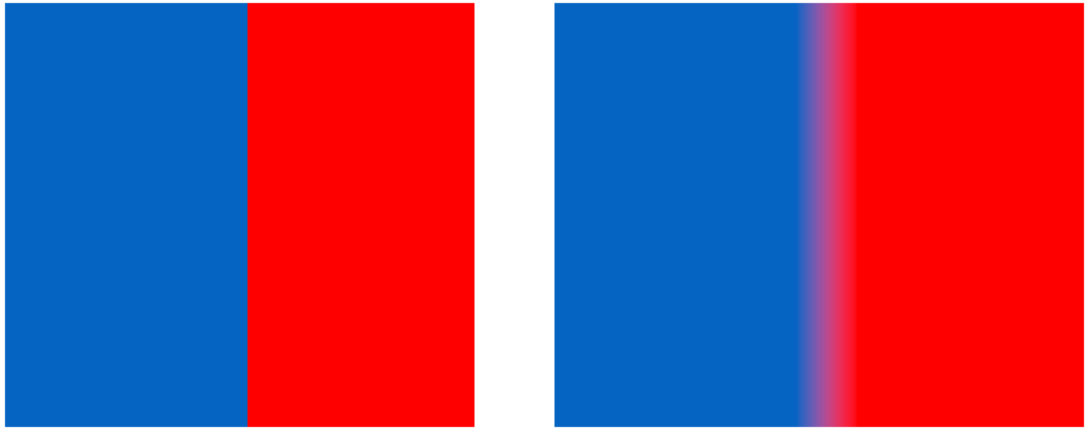
Assemblages supramoléculaires
Copolymères
TPE



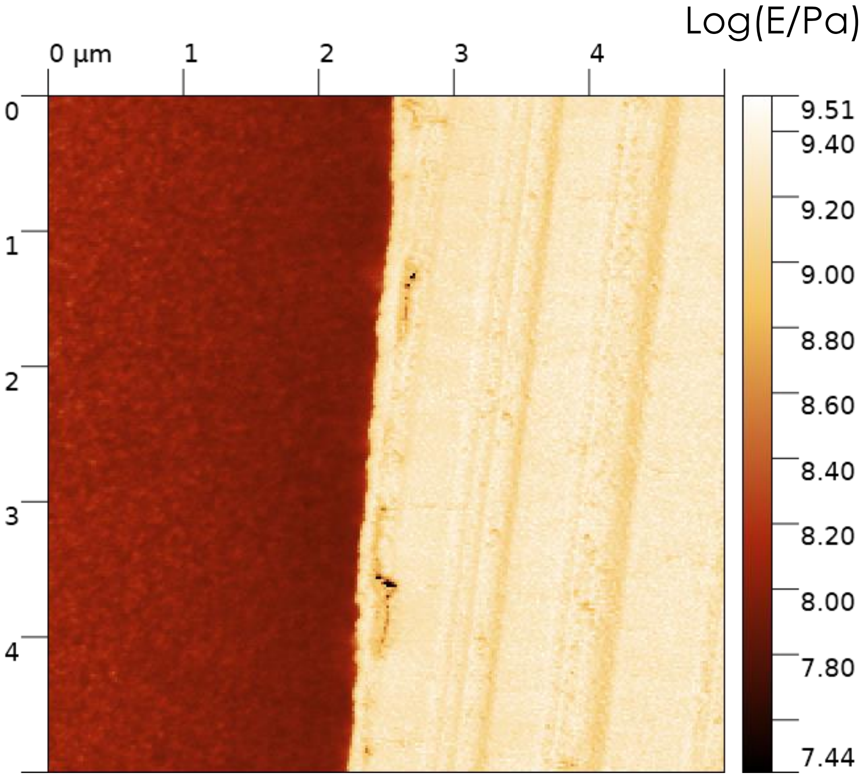
Composites
Nanocomposites

Perspectives

Exemple : Interface dans un mélange de polymère



Interface ? Interphase



- ➔ Compréhension des propriétés viscoélastiques locales pour mieux appréhender le comportement macroscopique du matériau
- ➔ Paramètres d'entrée pour des approches de modélisation

Vers la nano-DMA !

Plan

La viscoélasticité, qu'est-ce que c'est ?

La spectroscopie mécanique ou DMA

L'équivalence temps-température

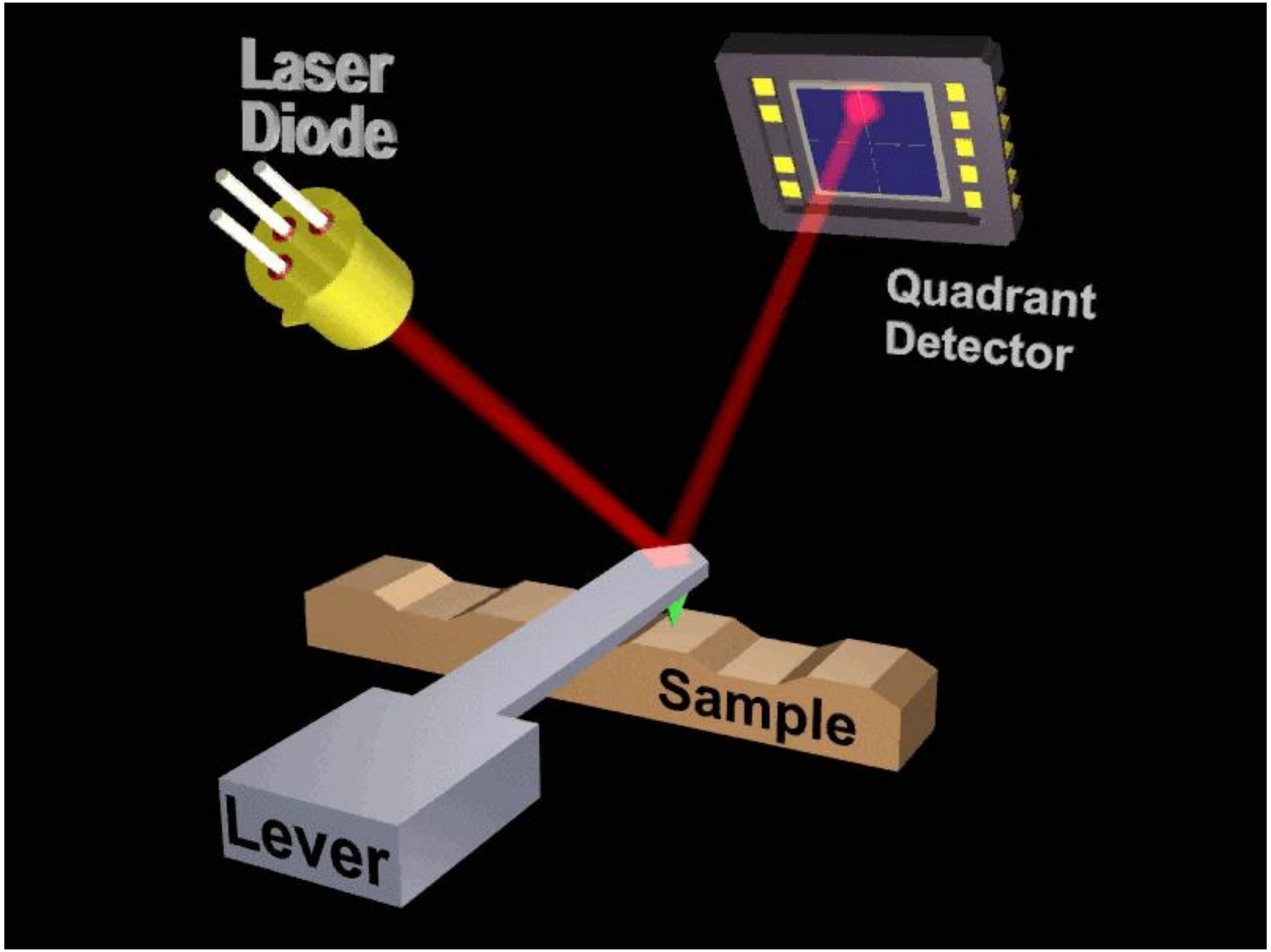
Limites et perspectives : vers les mesures locales

Extension à la nanoéchelle

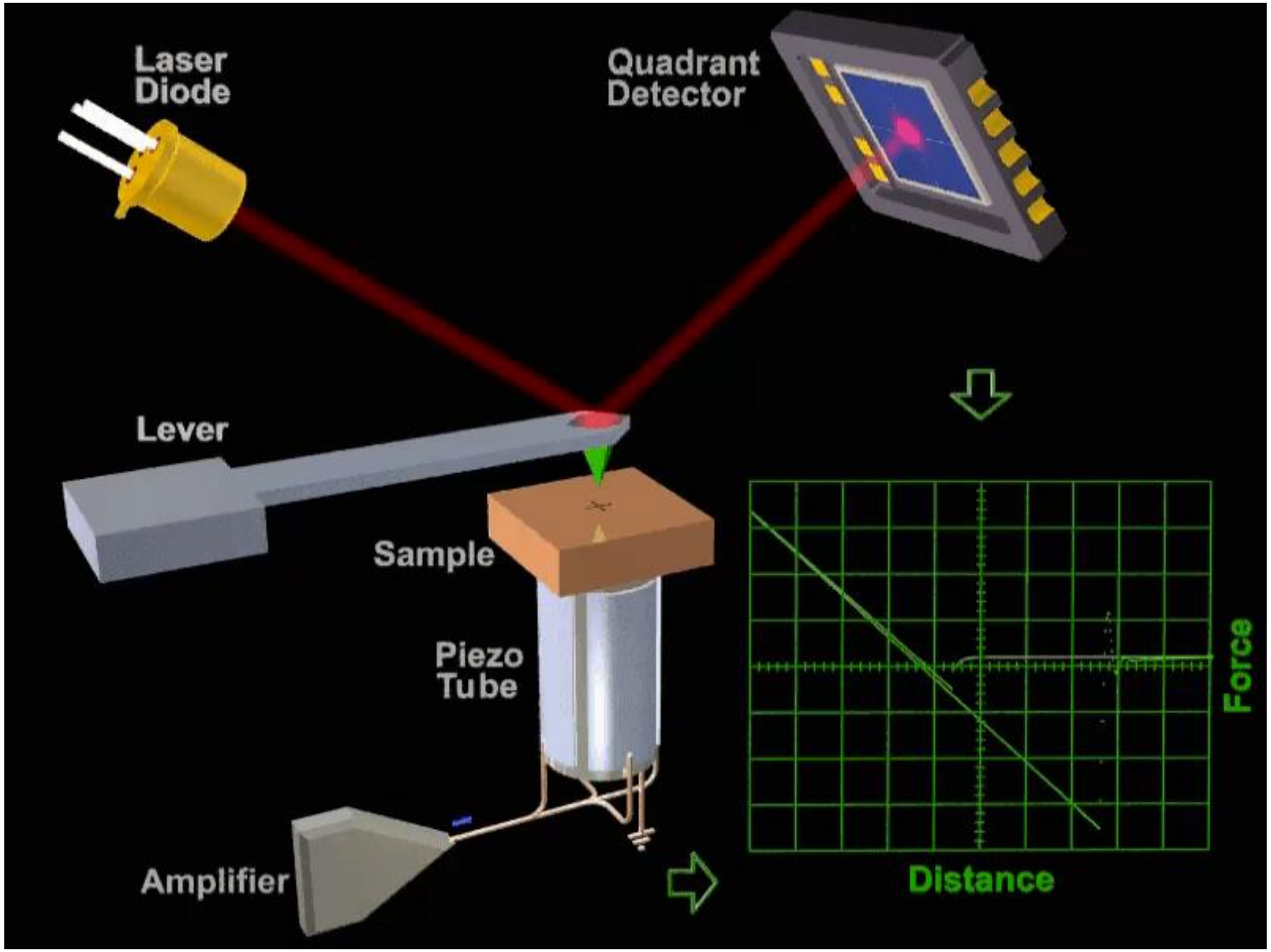
- nanoDMA
- CR-AFM
- Intermodulation AFM
- …

Conclusions et Perspectives

Scanning Probe Microscopy



Scanning Probe Spectroscopy

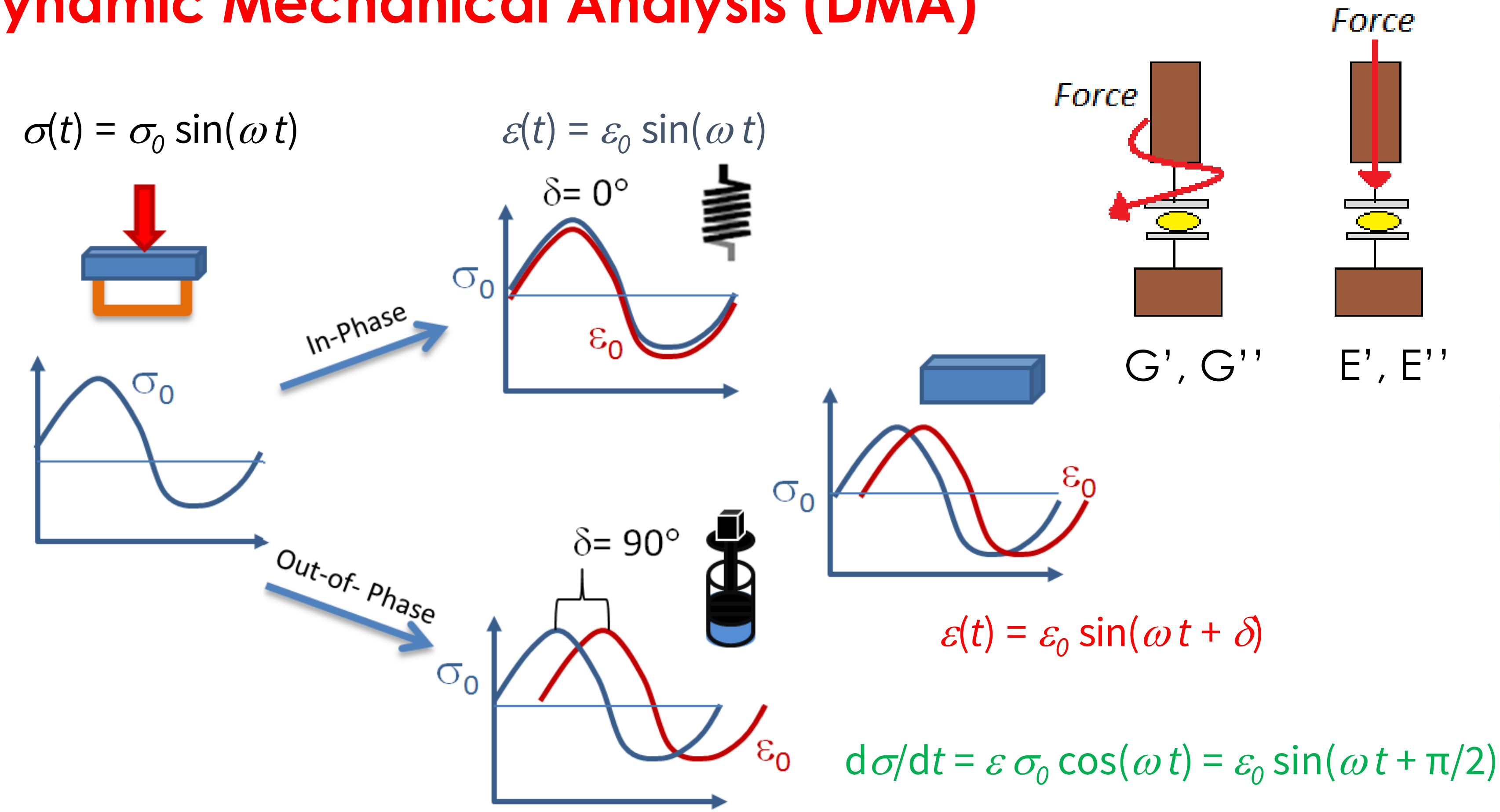


Viscoelastic properties at the nanoscale

Dynamic Mechanical Analysis (DMA)

$\sigma(t) = \sigma_0 \sin(\omega t)$

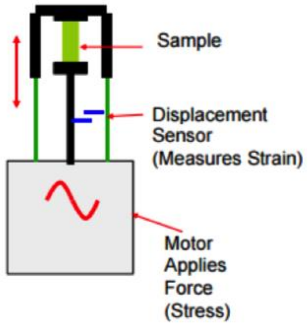
$\varepsilon(t) = \varepsilon_0 \sin(\omega t)$

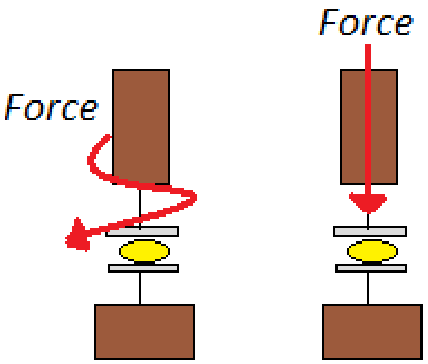


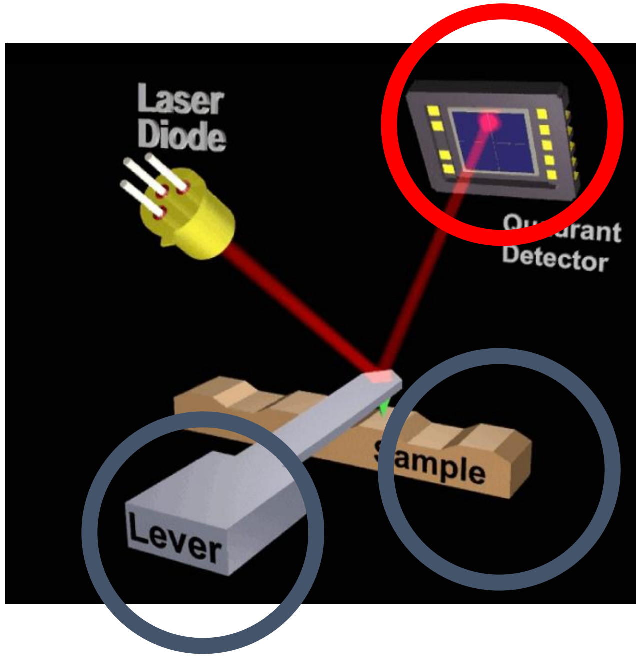
$\varepsilon(t) = \varepsilon_0 \sin(\omega t + \delta)$

$d\sigma/dt = \varepsilon\, \sigma_0 \cos(\omega t) = \varepsilon_0 \sin(\omega t + \pi/2)$

From DMA to nDMA …

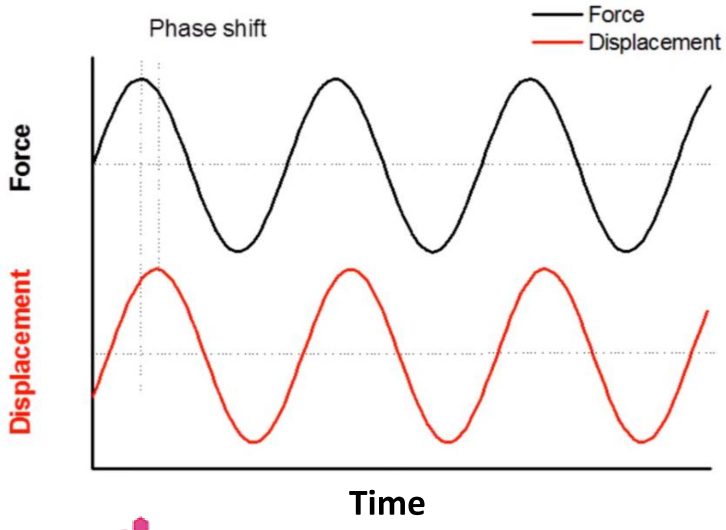


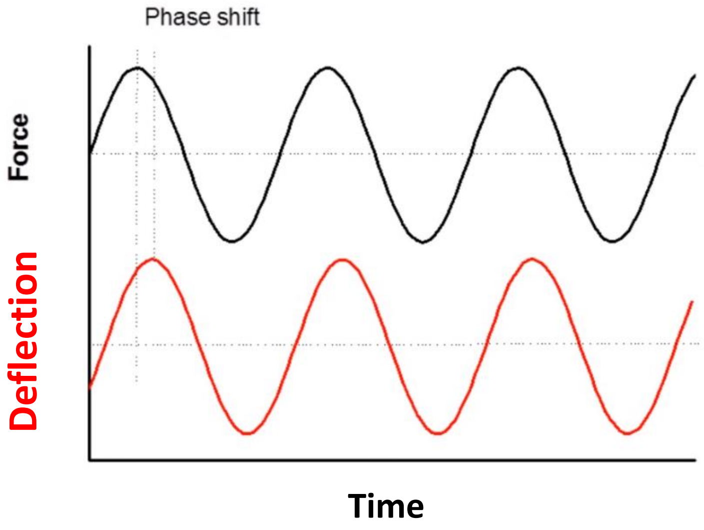


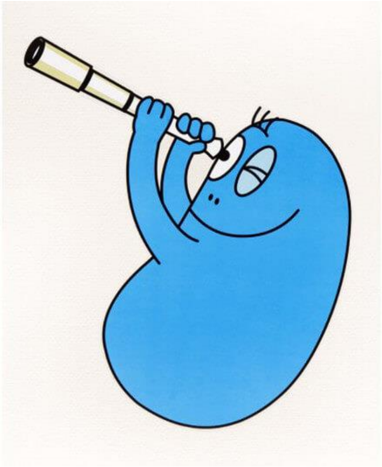


$$D(t)= D_1\, e^{i(\omega t+\varphi)} + D_0$$

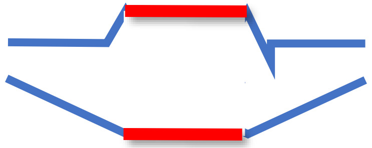
$$Z(t)=Z_1 e^{i(\omega t+\psi)} + Z_0$$







Notation



- One common notation set will describe all three regimes introduced above, with Z modulation via either Z scanner or sample actuator
- Harmonic signals:
- $z(t) = Z_1 \sin(\omega t + \psi) + Z_0$
  - Z displacement, probe or sample actuator
  - Z_1, ψ - amplitude and phase at frequency $\omega = 2\pi f$

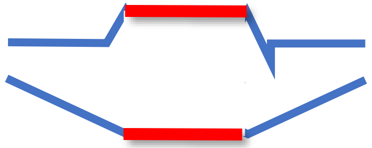
    (from Z-sensor lock-in or deflection-calibrated on a hard reference sample)
  - Measured or calibrated on a hard reference sample
- $d(t) = D_1 \sin(\omega t + \varphi) + D_0$
  - Measured deflection (Vertical deflection signal)
  - D_1, φ - amplitude and phase (from lock-in) at frequency $\omega = 2\pi f$
- K_c - cantilever spring constant (calibrated, known)

Derivation of General Equations for nDMA

- Key idea: Using definition of Dynamic Stiffness
- Stiffness [*N*/*m*] = Force [*n*N] / Deformation [*nm*] : $S^* = F^* / L^*$
  - Force: measured by deflection (channel 1)
  - Deformation: in the displacement measurement (channel 2)
  - Deformation L = Z displacement minus Deflection

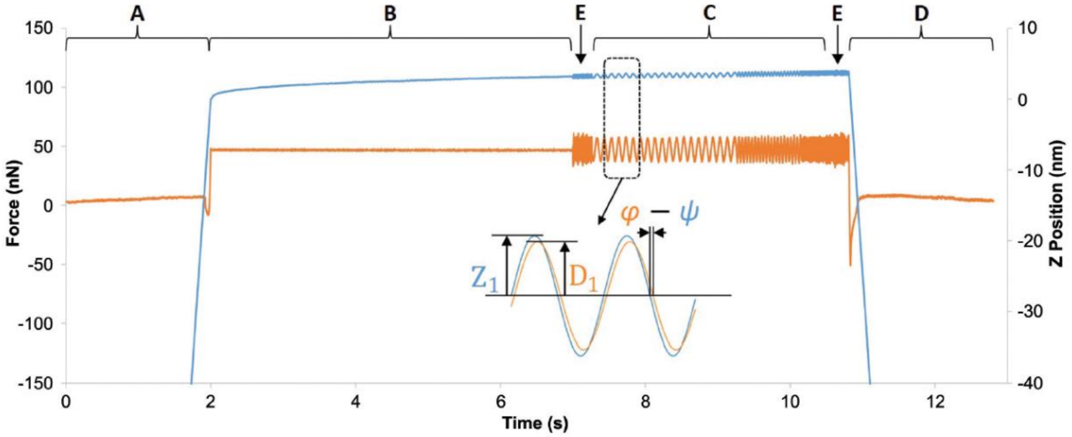
- For dynamic stiffness and harmonic excitation: complex values
- Equations for complex values:
  - $F^* = K_c D_1 e^{i(\omega t+\varphi)}$
  - $L^* = Z_1 e^{i(\omega t+\psi)} - D_1 e^{i(\omega t+\varphi)}$
  - $S^* = S' + iS" = K_c D_1 e^{i(\omega t+\varphi)} / [Z_1 e^{i(\omega t+\psi)} - D_1 e^{i(\omega t+\varphi)}]$

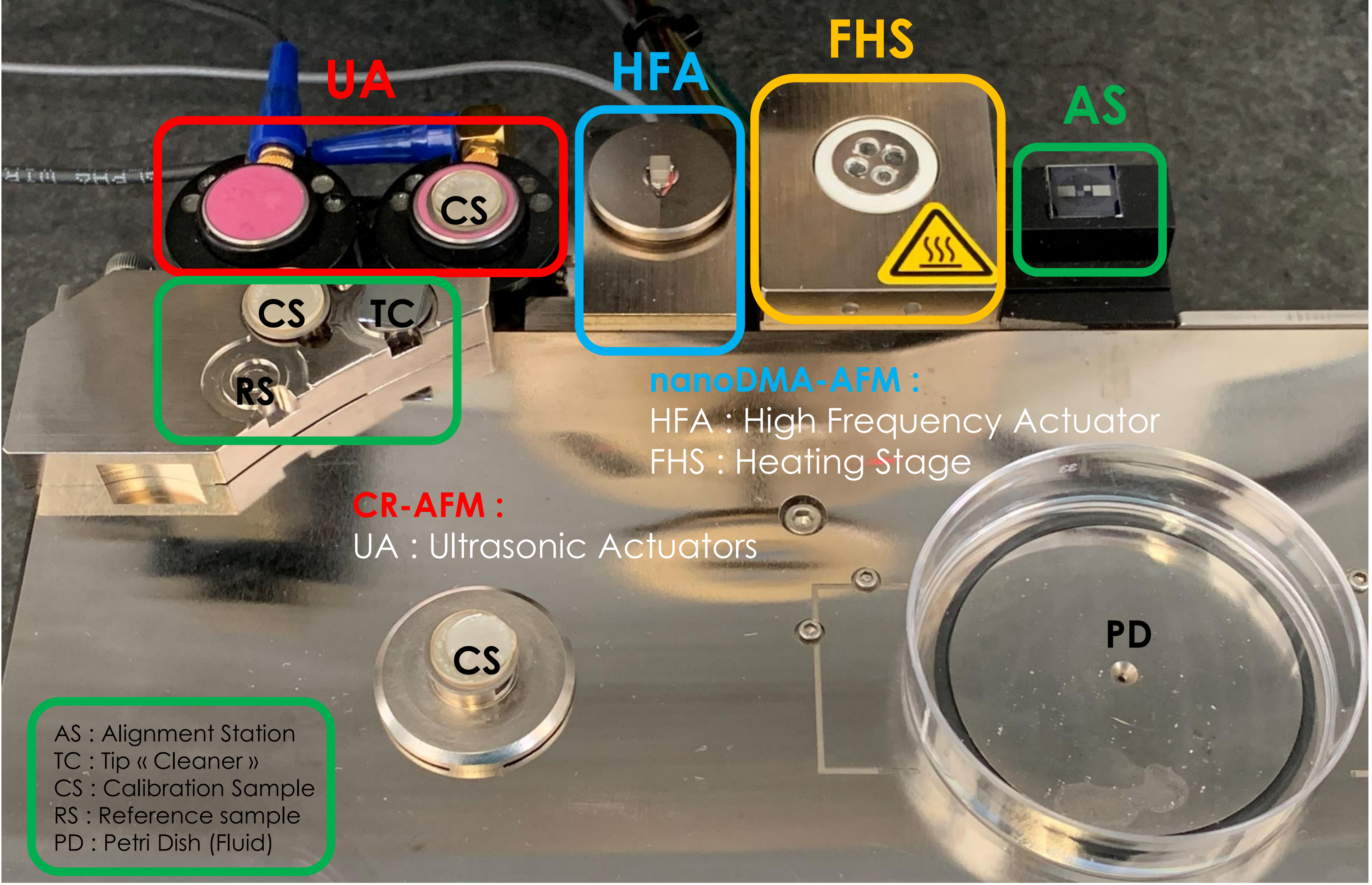
General formulation



Equations relating tan-Delta and Storage/Loss Modulus or Stiffness to measured lock-in amplitudes and phases

- $S^* = S' + iS" = K_c D_1 e^{i\varphi} / [Z_1 e^{i\psi} - D_1 e^{i\varphi}]$
- $S' = \frac{K_c D_1}{Z_1} \frac{\cos(\varphi - \psi) - (D_1/Z_1)}{(D_1/Z_1)^2 - 2(D_1/Z_1)\cos(\varphi - \psi) + 1}$
- $S" = \frac{K_c D_1}{Z_1} \frac{\sin(\varphi - \psi)}{(D_1/Z_1)^2 - 2(D_1/Z_1)\cos(\varphi - \psi) + 1}$
- $\tan\delta = S"/S' = \frac{\sin(\varphi - \psi)}{\cos(\varphi - \psi) - (D_1/Z_1)}$
- $E' = \frac{S'}{2a_c}$; $E" = \frac{S"}{2a_c}$; where a_c is contact radius (e.g., from JKR)



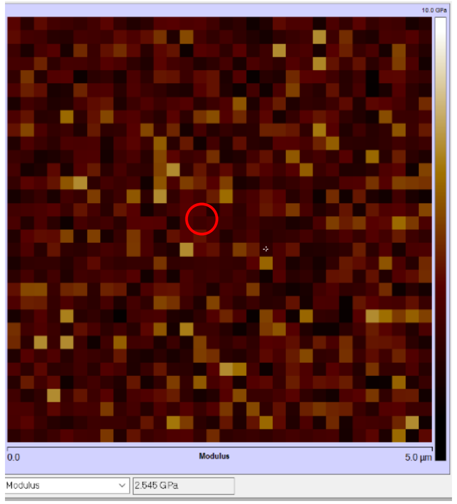


nanoDMA-AFM :
HFA : High Frequency Actuator
FHS : Heating Stage

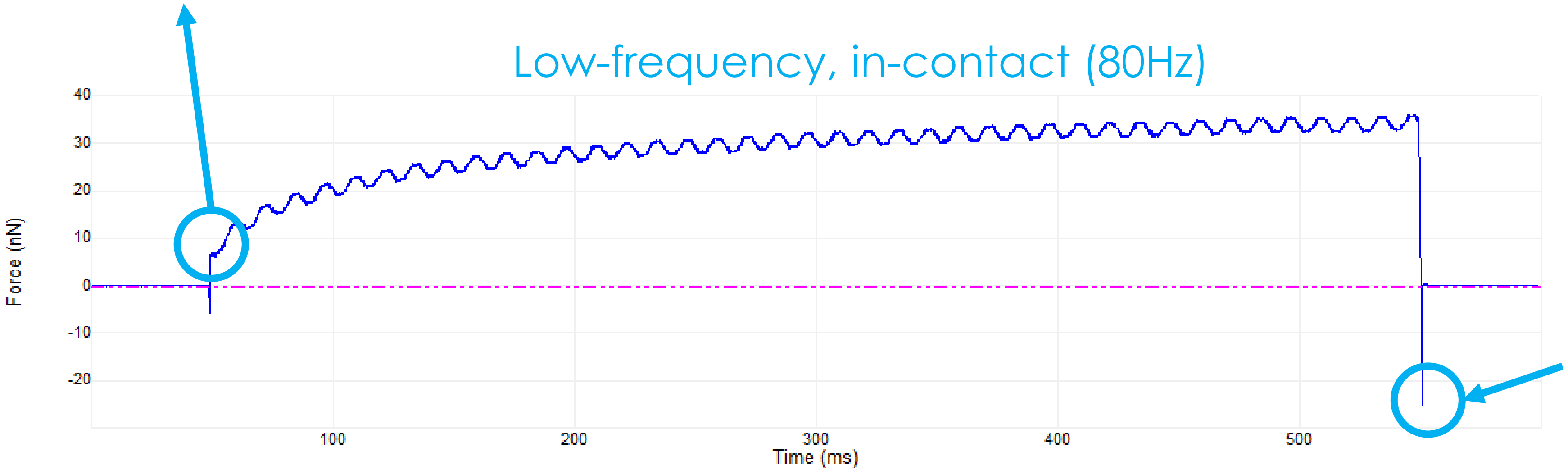
CR-AFM :
UA : Ultrasonic Actuators

AS : Alignment Station
TC : Tip « Cleaner »
CS : Calibration Sample
RS : Reference sample
PD : Petri Dish (Fluid)

nanoDynamic Mechanical Analysis

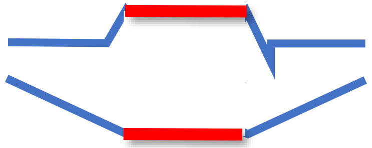


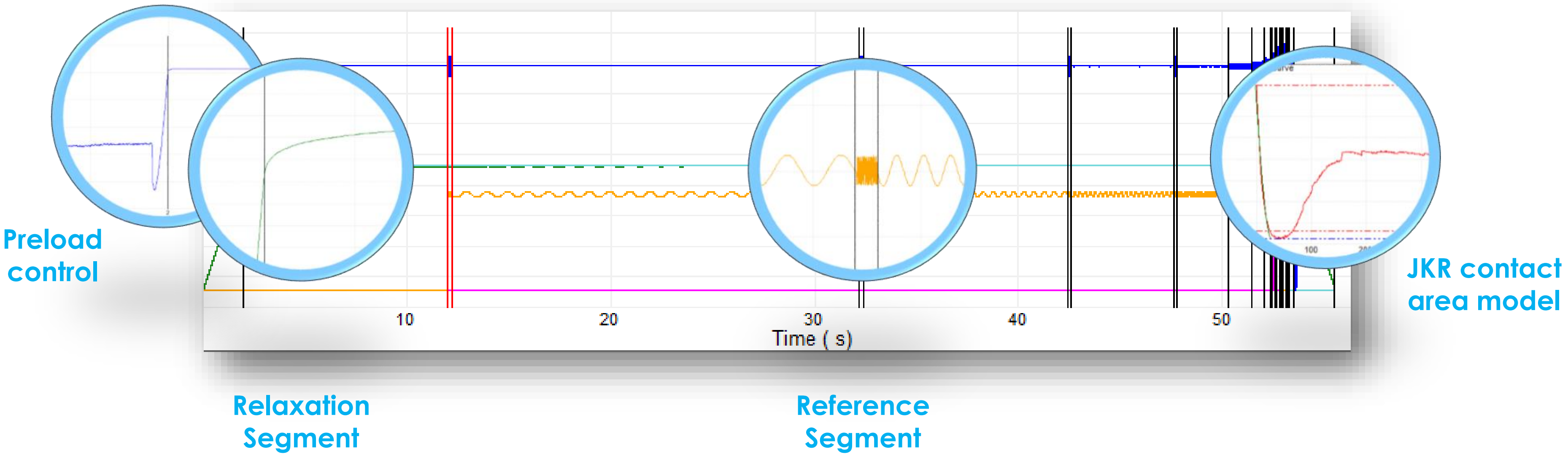
Preload control


Low-frequency, in-contact (80Hz)

Adhesion quantified

nanoDynamic Mechanical Analysis





Modulated force, embedded in force curve

Sub-nm amplitudes, stays in linear regime.

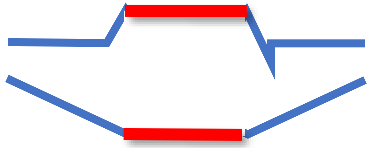
No ripping out of contact during modulation

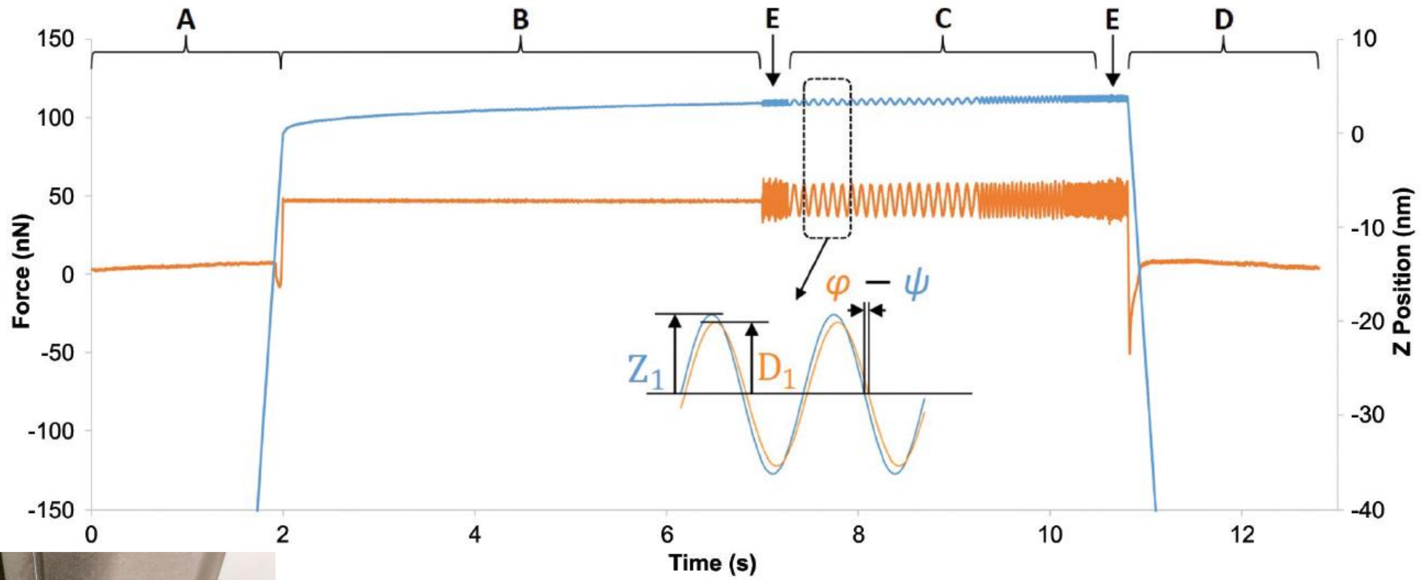
Imaging and point spectroscopy

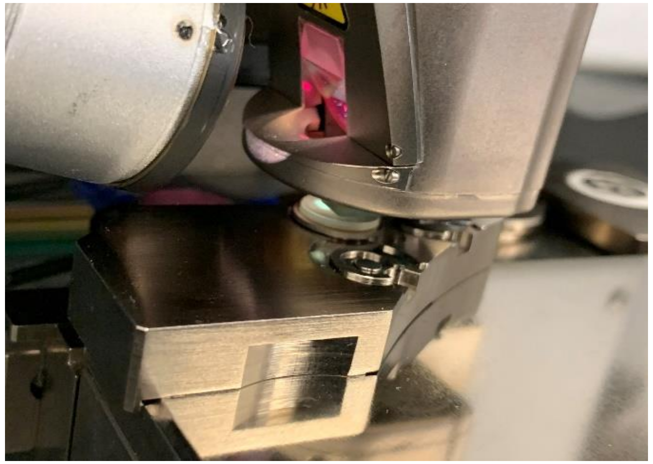
Quantitative data in both

Adapted from Bruker

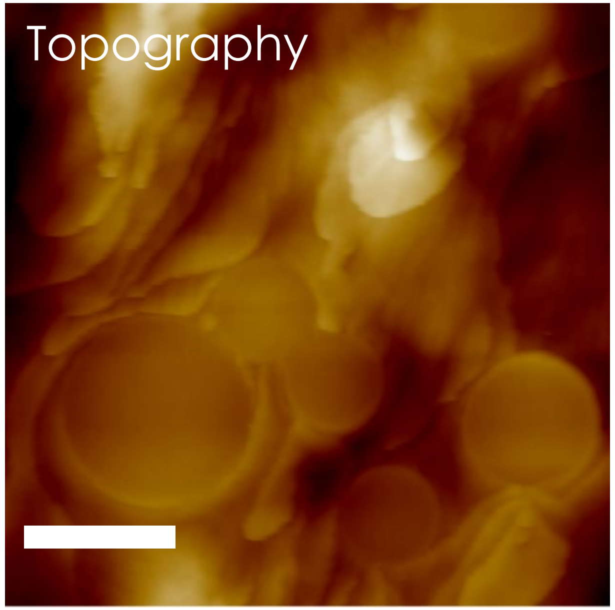
nanoDynamic Mechanical Analysis

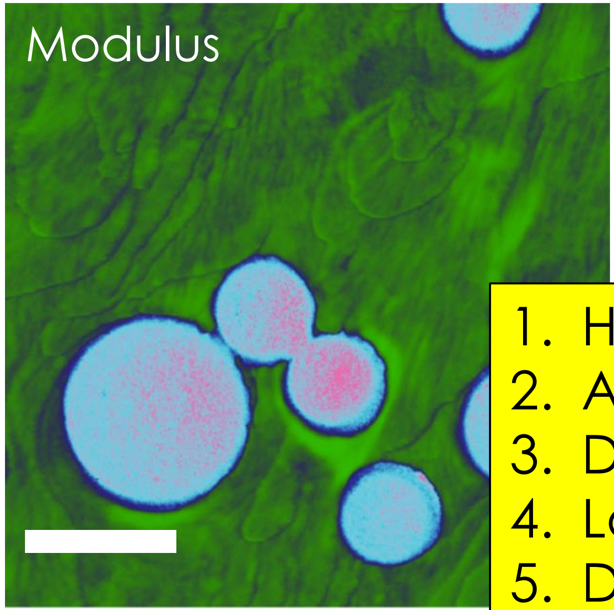


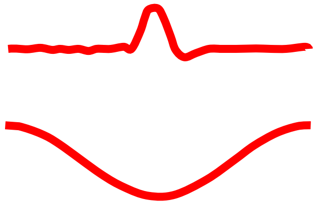




Calibration on Sapphire in the exact same conditions !



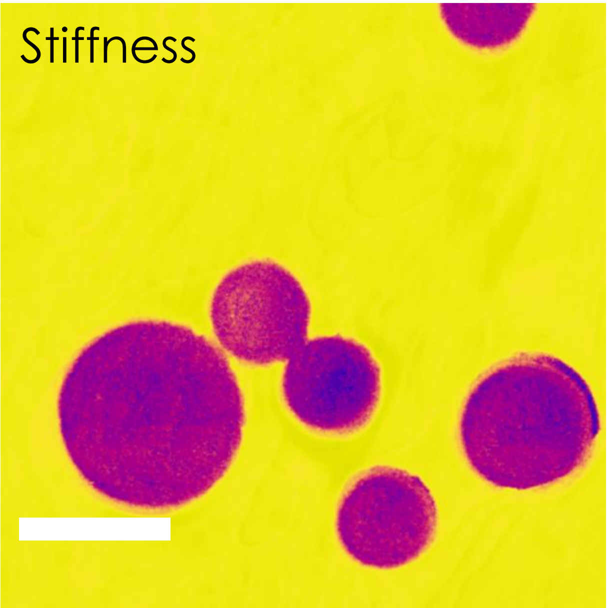


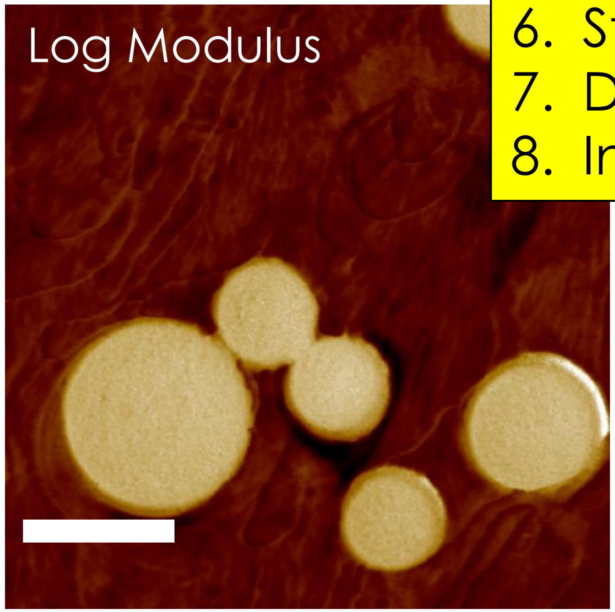


PS – PCL (25:75)

Tip : RTESPA 300 – 125

k = 29.22 N/m
Radius = 125 nm

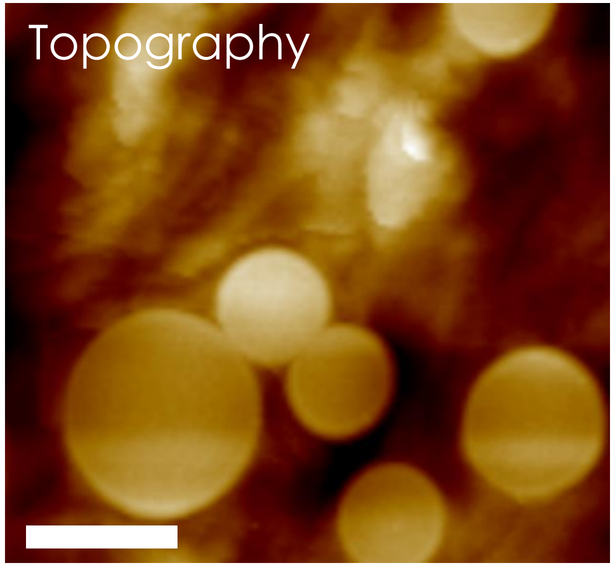


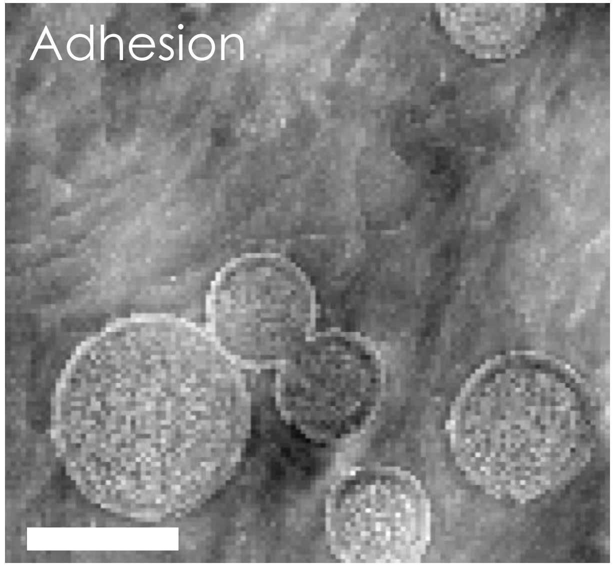


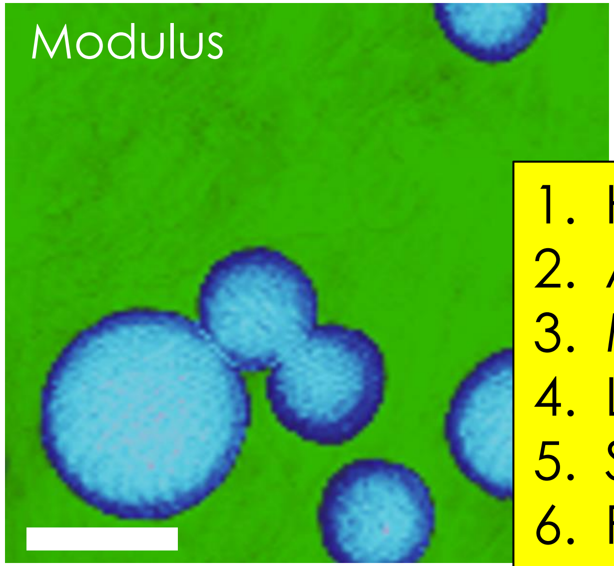
1. Height
2. Adhesion
3. DMT Modulus
4. Log DMT Modulus
5. Dissipation
6. Stiffness
7. Deformation
8. Indentation

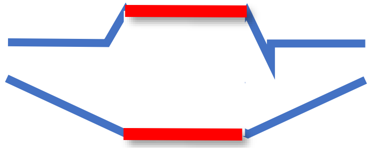
PeakForce Tapping QNM

512 x 512 pixels, PFSP = 15 nN
Scale bar = 2.0 µm







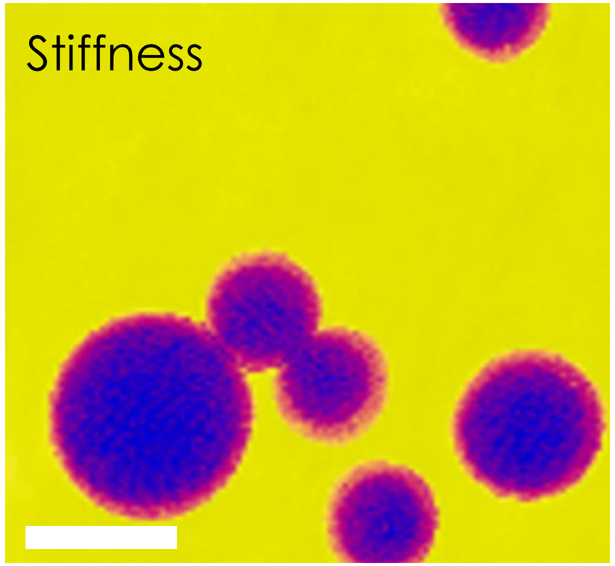


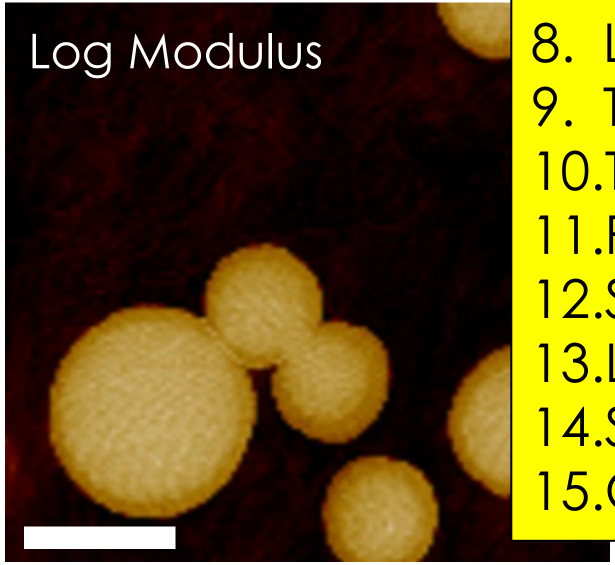
PS – PCL (25:75)

Tip : RTESPA 300 – 125

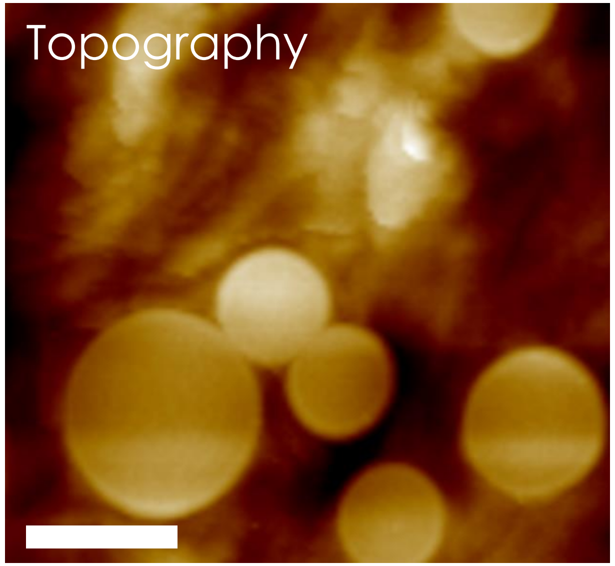
k = 29.22 N/m
Radius = 125 nm

PeakForce Tapping nanoDMA
128 x 128 pixels,
Frequency = 80 Hz
Scale bar = 2.0 µm

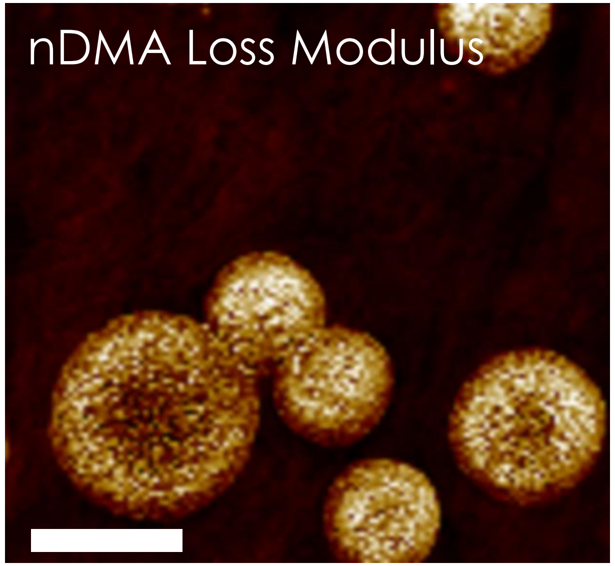


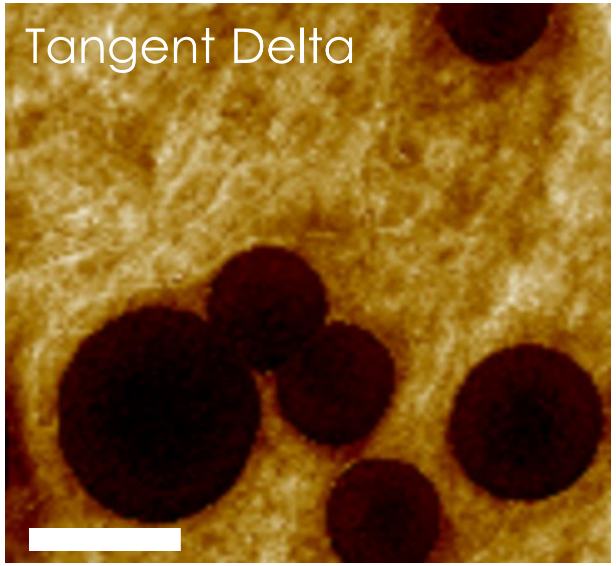


1. Height
2. Adhesion
3. Modulus
4. Log Modulus
5. Stiffness
6. Force Section
7. Storage Modulus
8. Loss Modulus
9. Tan Delta
10. Tan Phase lag
11. Phase lag
12. Storage Stiffness
13. Loss Stiffness
14. Stiffness Mag
15. Contact Radius


Topography

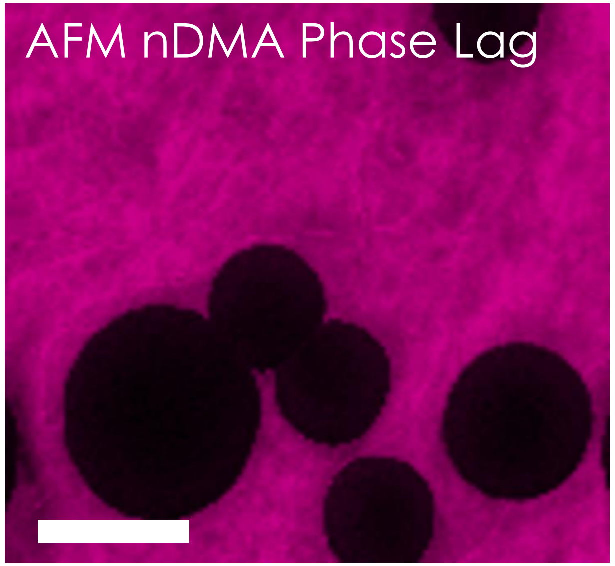

nDMA Storage Modulus

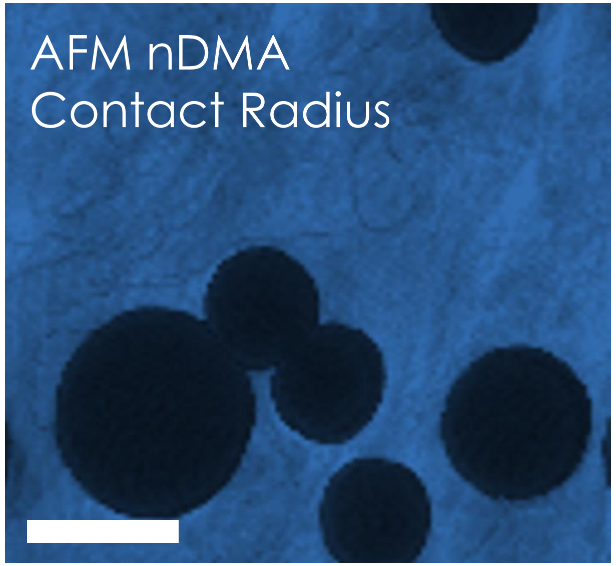

nDMA Loss Modulus


Tangent Delta

+ Temperature
+ Ramp scripting


AFM nDMA
Storage Stiffness


AFM nDMA Phase Lag


AFM nDMA
Contact Radius

pyCAROS

Python Code for Approach and Retract force curve analysis of Organic and hybrid Soft materials

Automatised multidimensional analysis based on Machine Learning algorithms for :

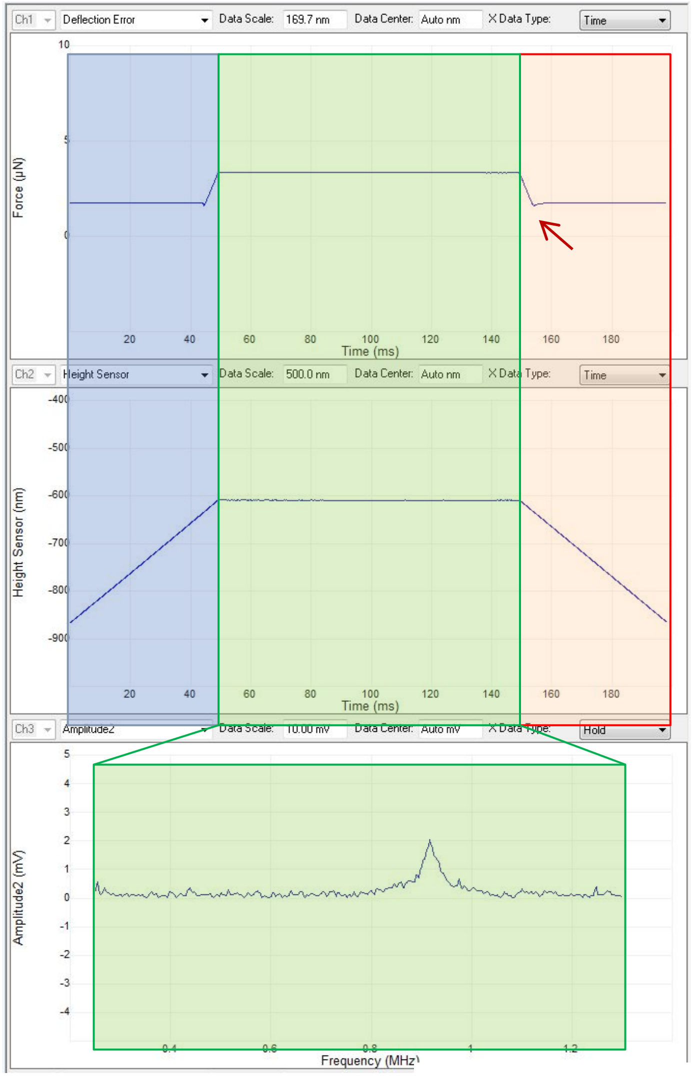
- Clustering of the data (PCA, Kmeans, GMM, …)
- Force curve analysis + «smart» mapping (Tabor, Contact mechanics models, R²)
- Force curve quality analysis

pyCAROS

Contact Resonance

From Frequency and Deflection to modulus

- CR is based on *FAST*Force Volume
  - Provides standard force curve for comparison for each pixel in map
    - Approach
    - Hold Force and sweep frequency
    - Retract
  - **More repeatable**: lateral force on tip is minimized, reducing tip wear
  - **More information**: allows measurement of adhesion force for each pixel better contact mechanics modeling
  - **Real-time maps** of both raw data and mechanical props (E', E'', loss tan)
  - **Whole sweep is saved**, allowing detection of artifact peaks, etc. (unlike frequency tracking methods like DA(F)RT)



Ch1 Deflection Error Data Scale: 169.7 nm Data Center: Auto nm X Data Type: Time

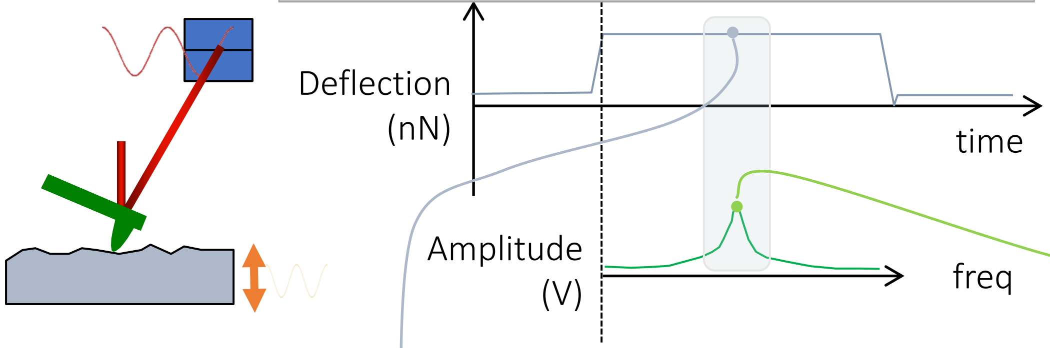
Ch2 Height Sensor Data Scale: 500.0 nm Data Center: Auto nm X Data Type: Time

Ch3 Amplitude2 Data Scale: 10.00 mV Data Center: Auto mV X Data Type: Hold

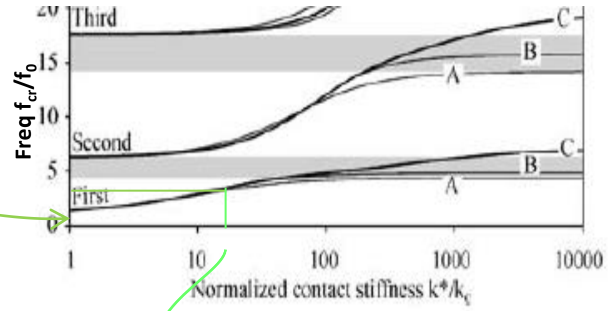
Contact Resonance

From Frequency and Deflection to modulus

1. Measure frequency **(fcr)** & Deflection



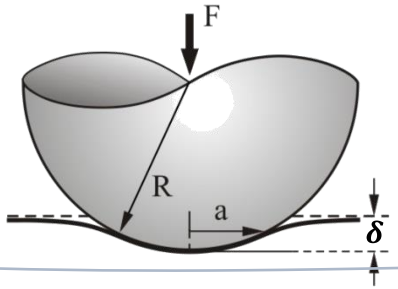
2. Apply cantilever dynamics to calculate k*/kc from fcr/f0



3. Convert Deflection to Force using deflection sensitivity & spring constant (kc)

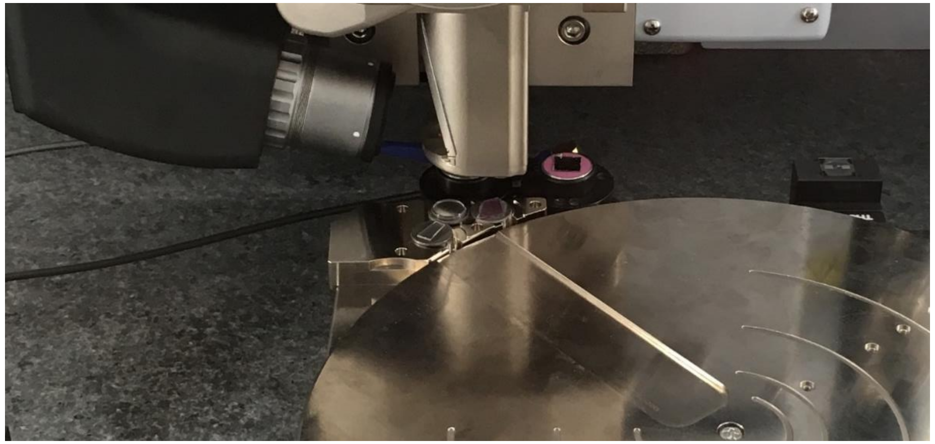
4. Apply Contact Mechanics to calculate E* from Radius (R), Force (F) and contact stiffness (k*)

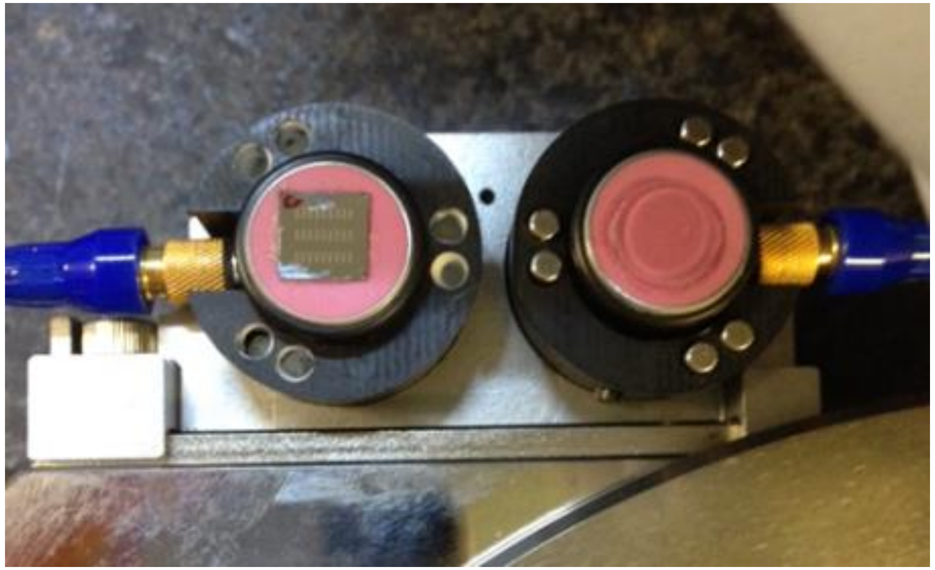
Hertzian Contact



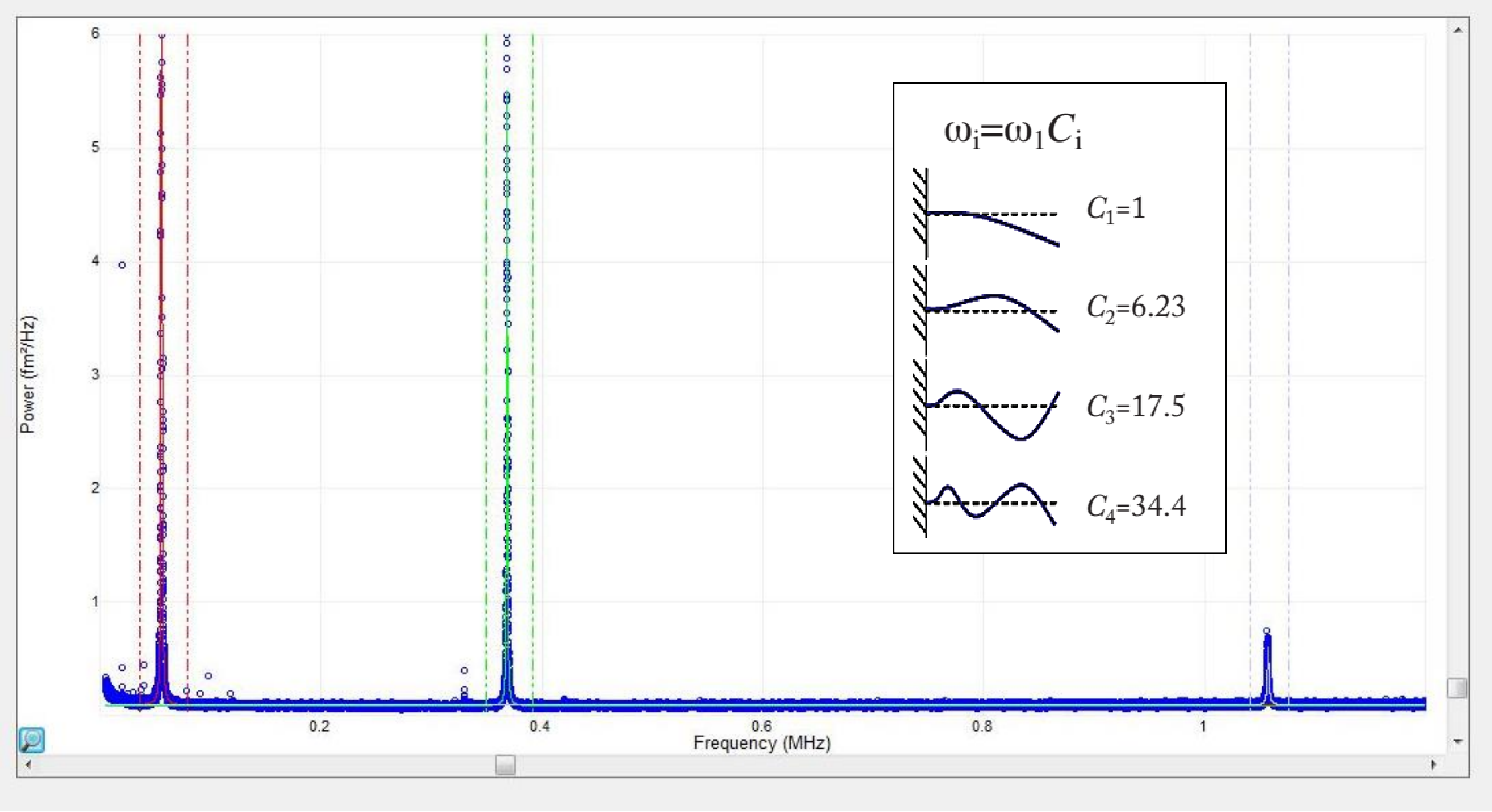
$$E^* = \sqrt{\frac{k^{*3}}{6RF}}$$

Contact Resonance





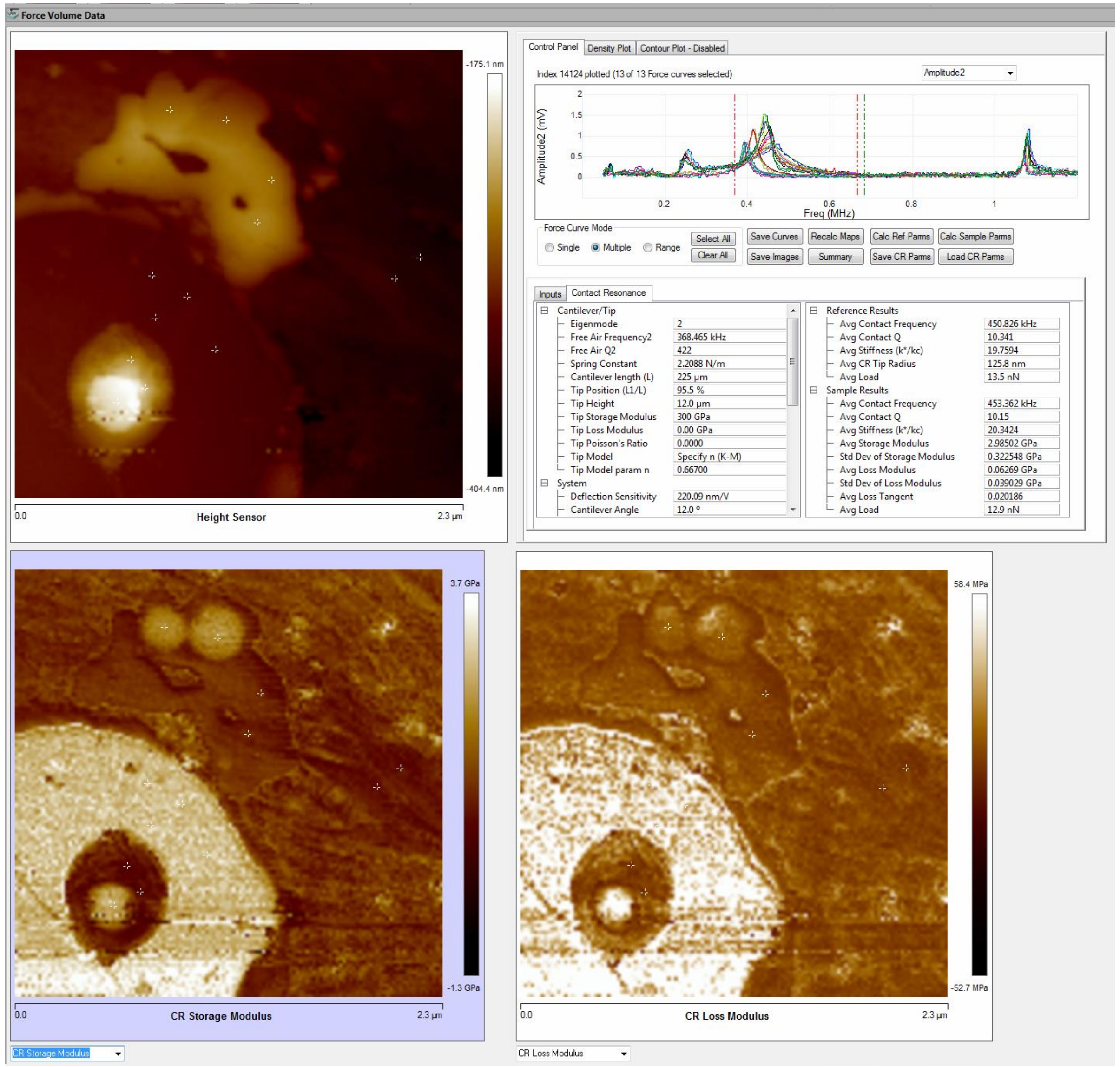
Contact Resonance



Contact Resonance

PS-PCL

2nd peak



Index 14124 plotted (13 of 13 Force curves selected)

Cantilever/Tip
- Eigenmode: 2
- Free Air Frequency2: 368.465 kHz
- Free Air Q2: 422
- Spring Constant: 2.2088 N/m
- Cantilever length (L): 225 µm
- Tip Position (L1/L): 95.5 %
- Tip Height: 12.0 µm
- Tip Storage Modulus: 300 GPa
- Tip Loss Modulus: 0.00 GPa
- Tip Poisson's Ratio: 0.0000
- Tip Model: Specify n (K-M)
- Tip Model param n: 0.66700

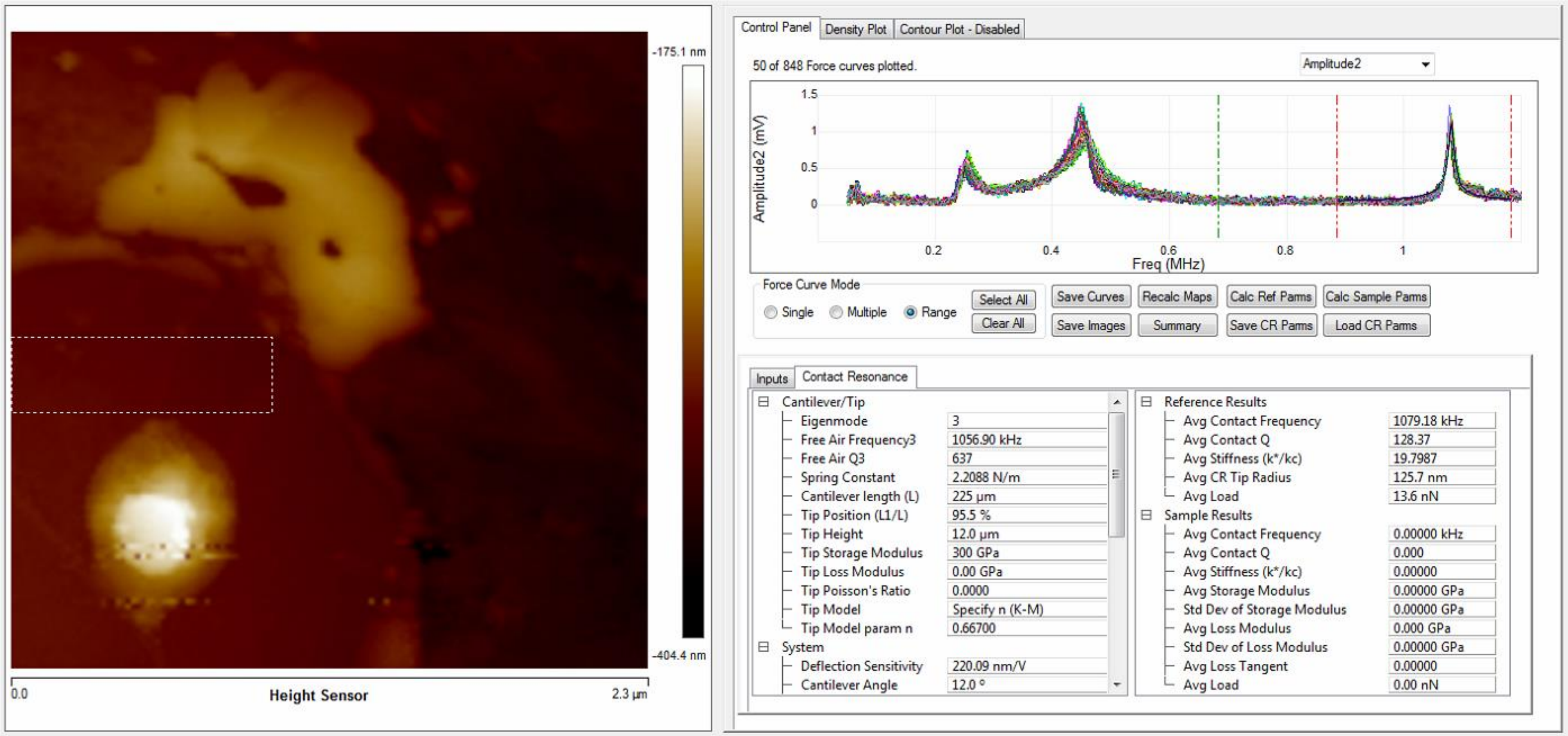
System
- Deflection Sensitivity: 220.09 nm/V
- Cantilever Angle: 12.0 °

Reference Results
- Avg Contact Frequency: 450.826 kHz
- Avg Contact Q: 10.341
- Avg Stiffness (k*/kc): 19.7594
- Avg CR Tip Radius: 125.8 nm
- Avg Load: 13.5 nN

Sample Results
- Avg Contact Frequency: 453.362 kHz
- Avg Contact Q: 10.15
- Avg Stiffness (k*/kc): 20.3424
- Avg Storage Modulus: 2.98502 GPa
- Std Dev of Storage Modulus: 0.322548 GPa
- Avg Loss Modulus: 0.06269 GPa
- Std Dev of Loss Modulus: 0.039029 GPa
- Avg Loss Tangent: 0.020186
- Avg Load: 12.9 nN

Contact Resonance

PS-PCL



50 of 848 Force curves plotted.

Force Curve Mode: ○ Single ○ Multiple ● Range

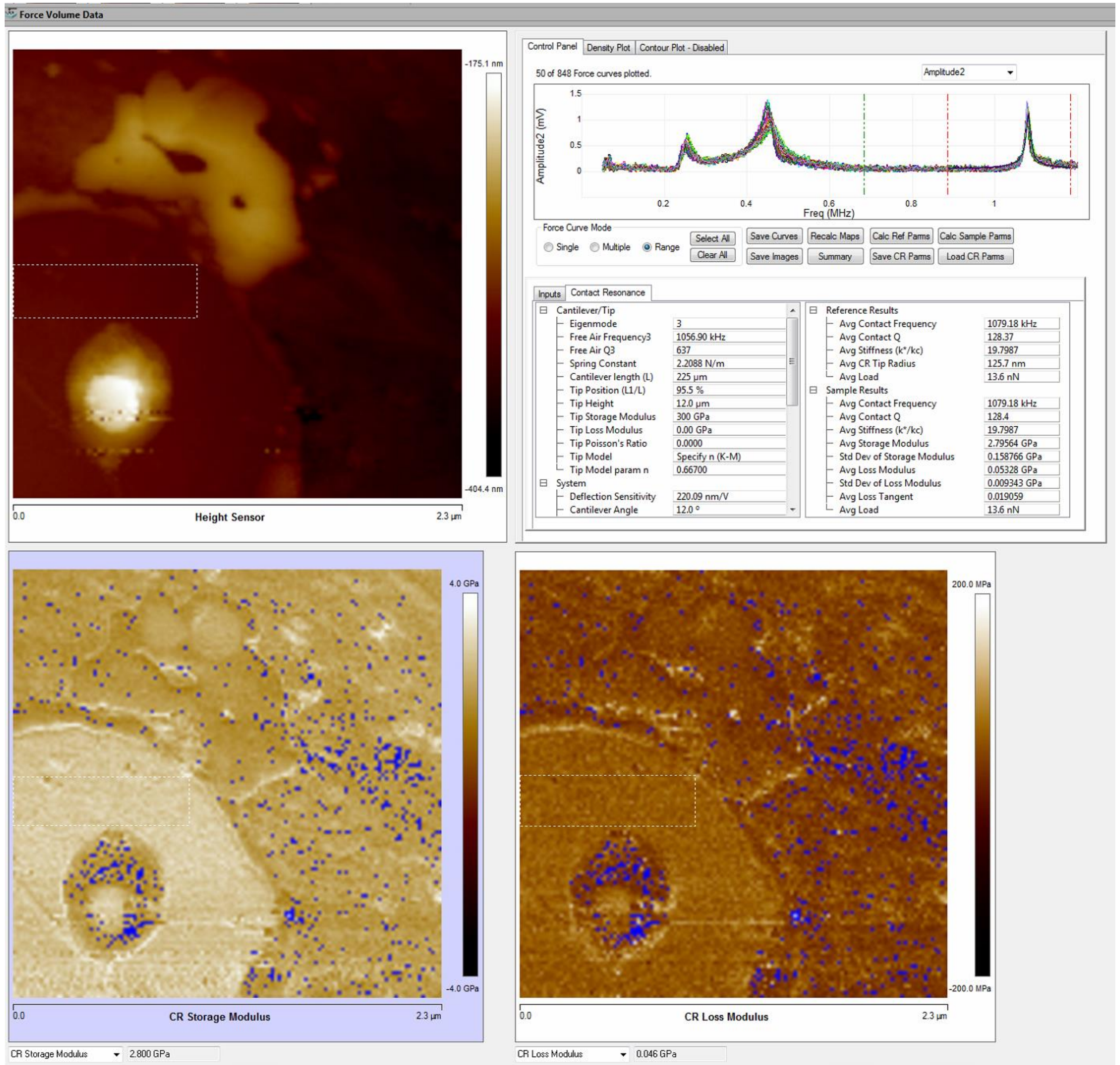
Cantilever/Tip	
Eigenmode	3
Free Air Frequency3	1056.90 kHz
Free Air Q3	637
Spring Constant	2.2088 N/m
Cantilever length (L)	225 µm
Tip Position (L1/L)	95.5 %
Tip Height	12.0 µm
Tip Storage Modulus	300 GPa
Tip Loss Modulus	0.00 GPa
Tip Poisson's Ratio	0.0000
Tip Model	Specify n (K-M)
Tip Model param n	0.66700
System	
Deflection Sensitivity	220.09 nm/V
Cantilever Angle	12.0 °

Reference Results	
Avg Contact Frequency	1079.18 kHz
Avg Contact Q	128.37
Avg Stiffness (k*/kc)	19.7987
Avg CR Tip Radius	125.7 nm
Avg Load	13.6 nN
Sample Results	
Avg Contact Frequency	0.00000 kHz
Avg Contact Q	0.000
Avg Stiffness (k*/kc)	0.00000
Avg Storage Modulus	0.00000 GPa
Std Dev of Storage Modulus	0.00000 GPa
Avg Loss Modulus	0.000 GPa
Std Dev of Loss Modulus	0.00000 GPa
Avg Loss Tangent	0.00000
Avg Load	0.00 nN

Contact Resonance

PS-PCL

3^{rd} peak



50 of 848 Force curves plotted.

Cantilever/Tip
- Eigenmode: 3
- Free Air Frequency3: 1056.90 kHz
- Free Air Q3: 637
- Spring Constant: 2.2088 N/m
- Cantilever length (L): 225 µm
- Tip Position (L1/L): 95.5 %
- Tip Height: 12.0 µm
- Tip Storage Modulus: 300 GPa
- Tip Loss Modulus: 0.00 GPa
- Tip Poisson's Ratio: 0.0000
- Tip Model: Specify n (K-M)
- Tip Model param n: 0.66700

System
- Deflection Sensitivity: 220.09 nm/V
- Cantilever Angle: 12.0 °

Reference Results
- Avg Contact Frequency: 1079.18 kHz
- Avg Contact Q: 128.37
- Avg Stiffness (k*/kc): 19.7987
- Avg CR Tip Radius: 125.7 nm
- Avg Load: 13.6 nN

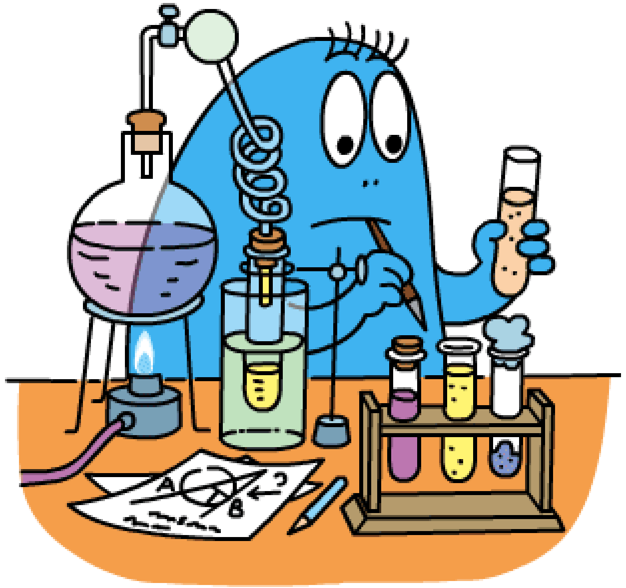
Sample Results
- Avg Contact Frequency: 1079.18 kHz
- Avg Contact Q: 128.4
- Avg Stiffness (k*/kc): 19.7987
- Avg Storage Modulus: 2.79564 GPa
- Std Dev of Storage Modulus: 0.158766 GPa
- Avg Loss Modulus: 0.05328 GPa
- Std Dev of Loss Modulus: 0.009343 GPa
- Avg Loss Tangent: 0.019059
- Avg Load: 13.6 nN

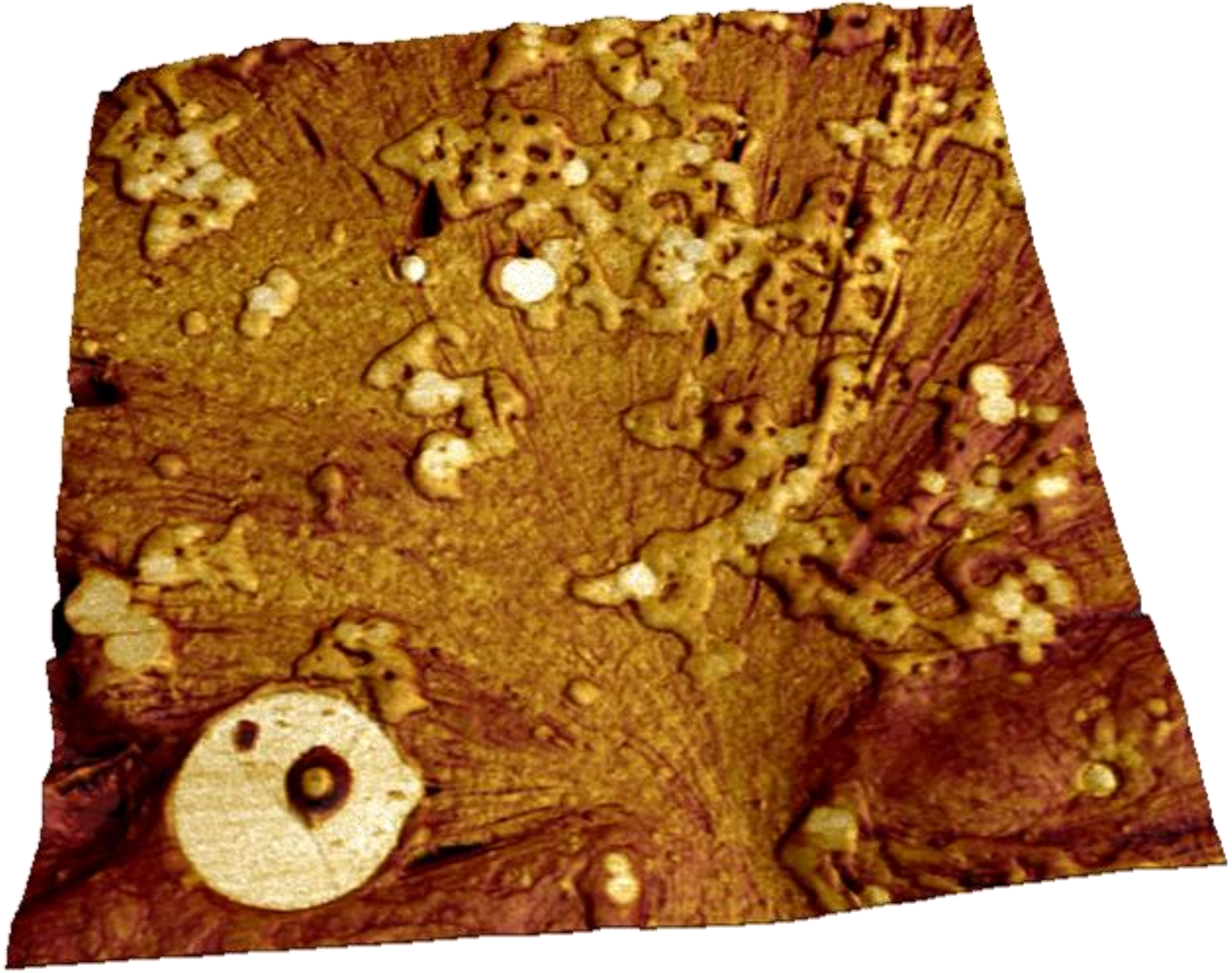
CR Storage Modulus: 2.800 GPa

CR Loss Modulus: 0.046 GPa

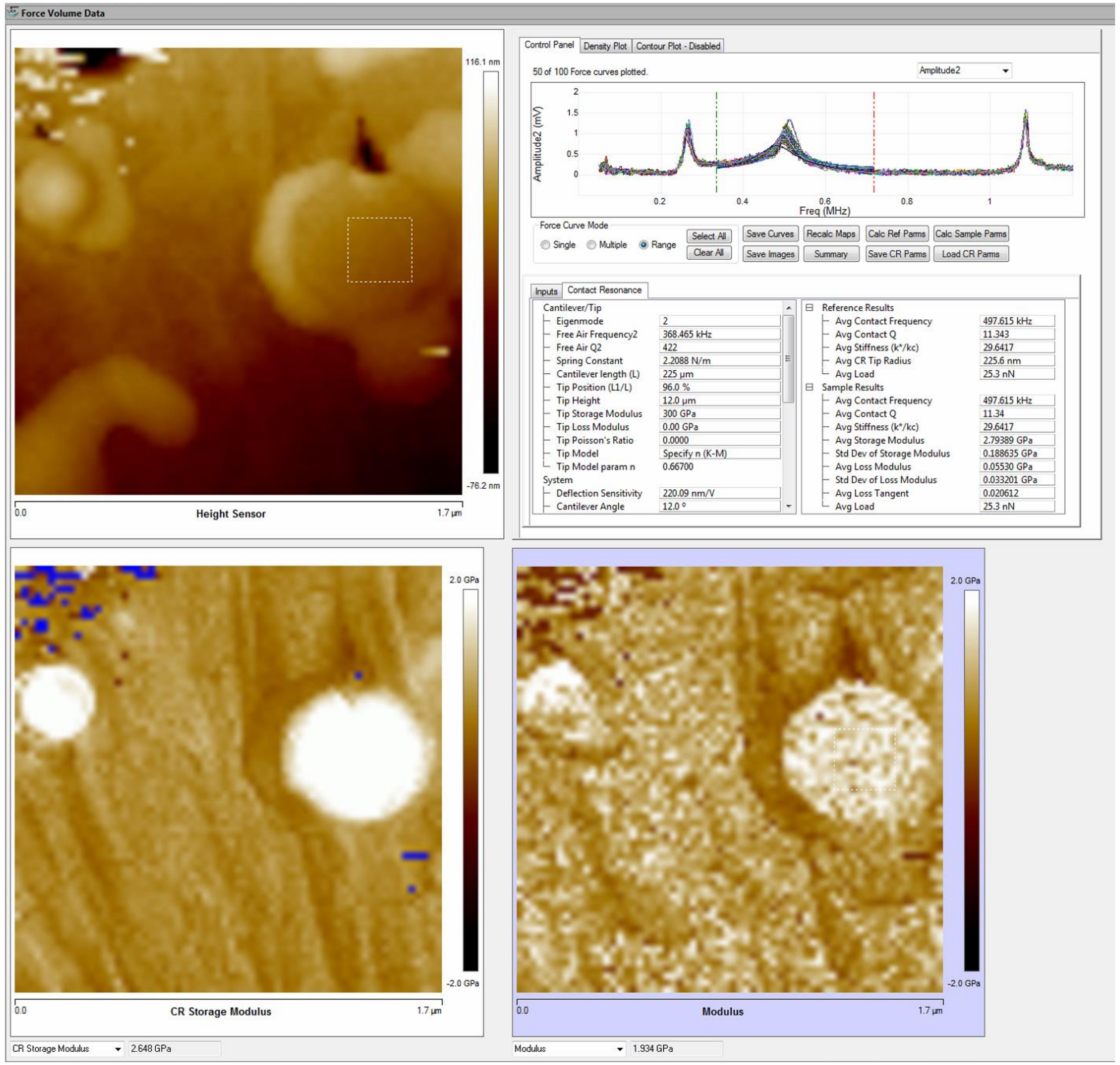
Contact Resonance

PS-PCL





Contact Resonance



50 of 100 Force curves plotted.

Cantilever/Tip

Eigenmode	2
Free Air Frequency2	368.465 kHz
Free Air Q2	422
Spring Constant	2.2088 N/m
Cantilever length (L)	225 µm
Tip Position (L1/L)	96.0 %
Tip Height	12.0 µm
Tip Storage Modulus	300 GPa
Tip Loss Modulus	0.00 GPa
Tip Poisson's Ratio	0.0000
Tip Model	Specify n (K-M)
Tip Model param n	0.66700

System

Deflection Sensitivity	220.09 nm/V
Cantilever Angle	12.0 °

Reference Results

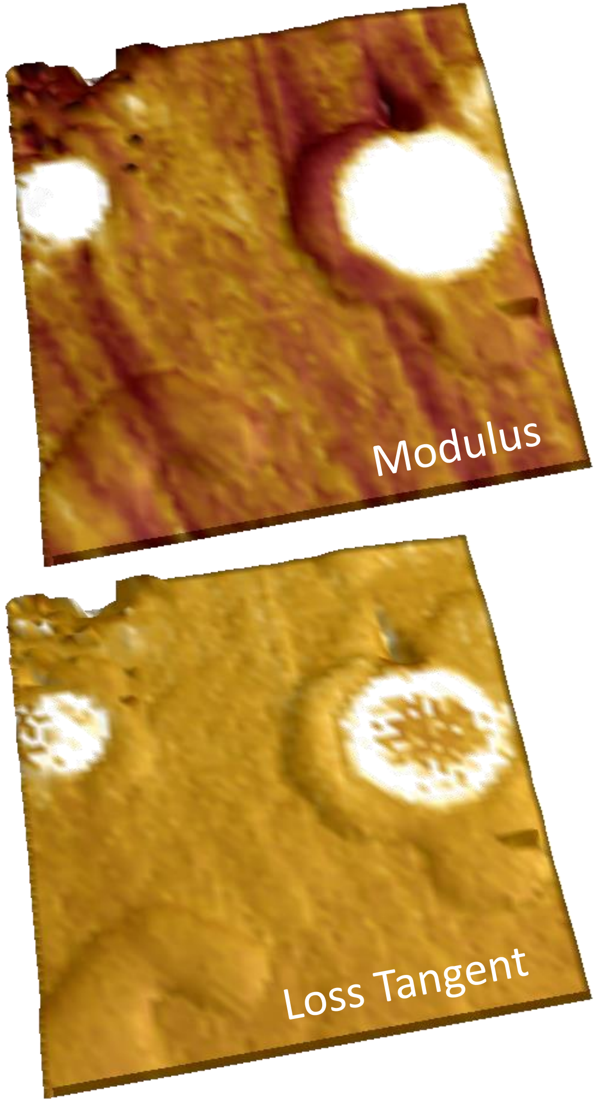
Avg Contact Frequency	497.615 kHz
Avg Contact Q	11.343
Avg Stiffness (k*/kc)	29.6417
Avg CR Tip Radius	225.6 nm
Avg Load	25.3 nN

Sample Results

Avg Contact Frequency	497.615 kHz
Avg Contact Q	11.34
Avg Stiffness (k*/kc)	29.6417
Avg Storage Modulus	2.79389 GPa
Std Dev of Storage Modulus	0.188635 GPa
Avg Loss Modulus	0.05530 GPa
Std Dev of Loss Modulus	0.033201 GPa
Avg Loss Tangent	0.020612
Avg Load	25.3 nN

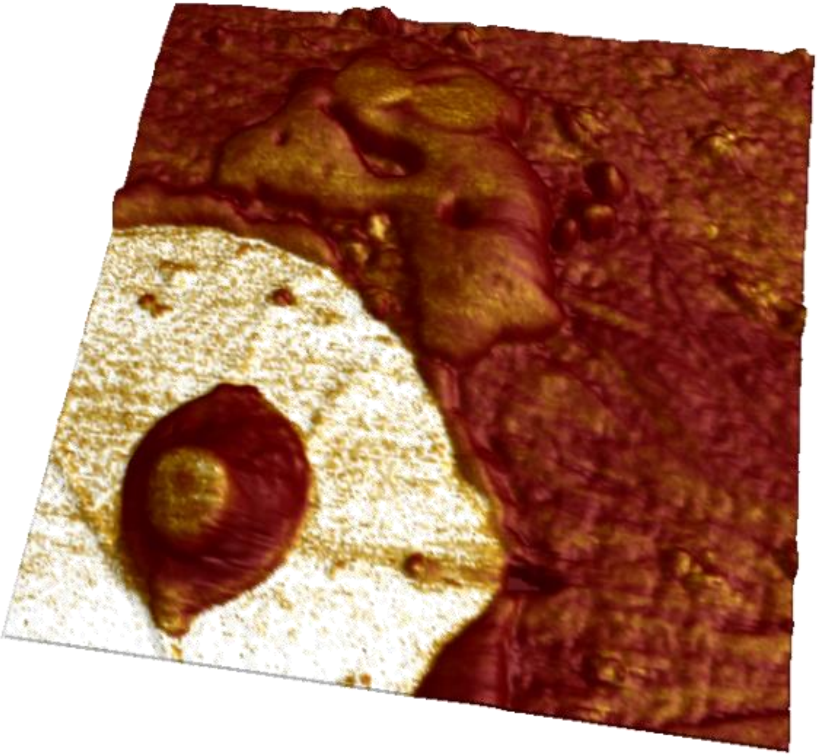
CR Storage Modulus: 2.648 GPa

Modulus: 1.934 GPa

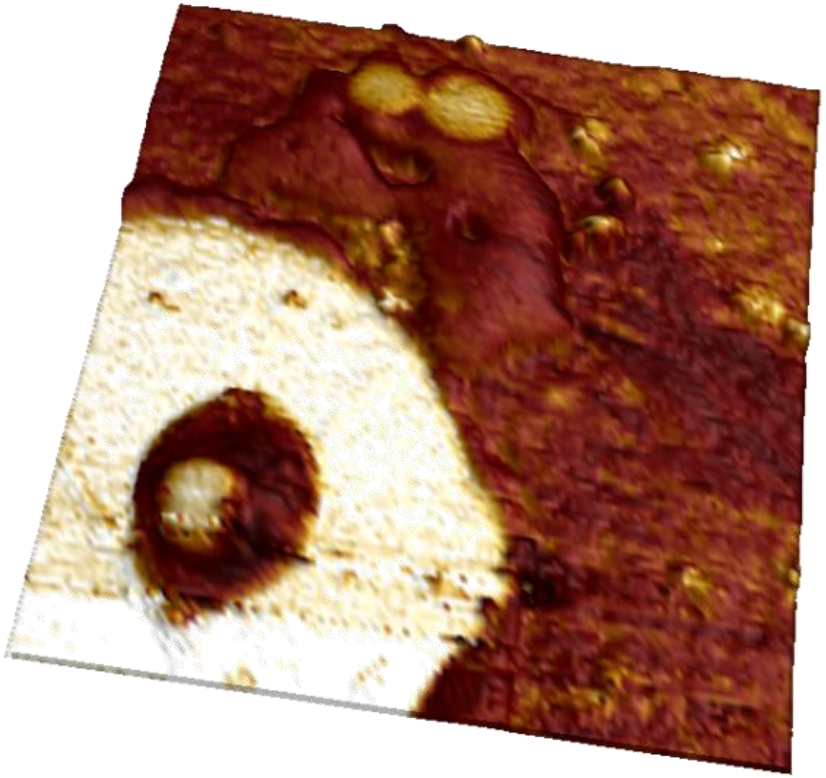


Comparing PFTQNM and CR

- PFTQNM

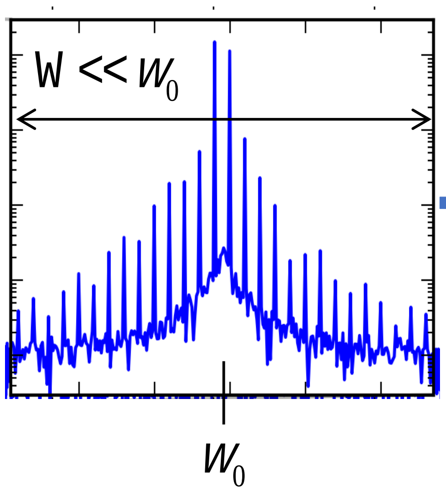


- CR 128x128

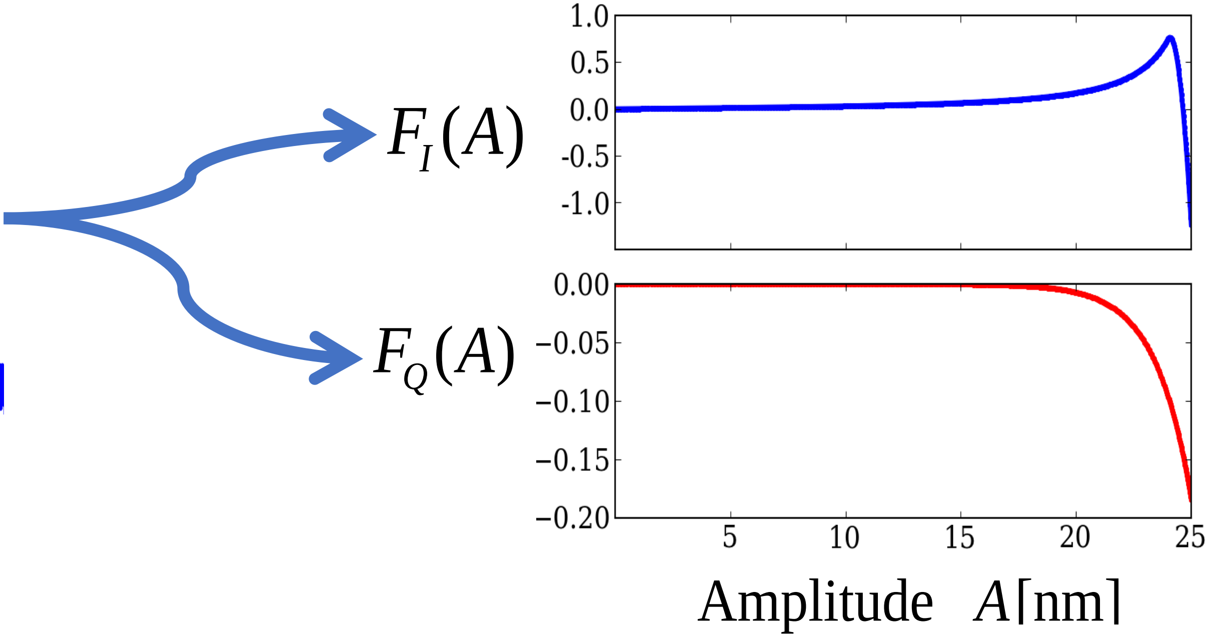


Intermodulation AFM

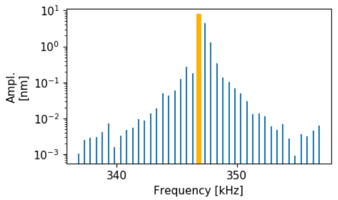
The methodology

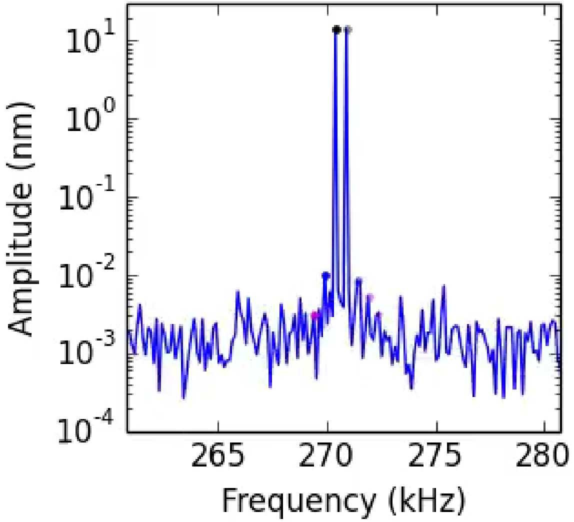


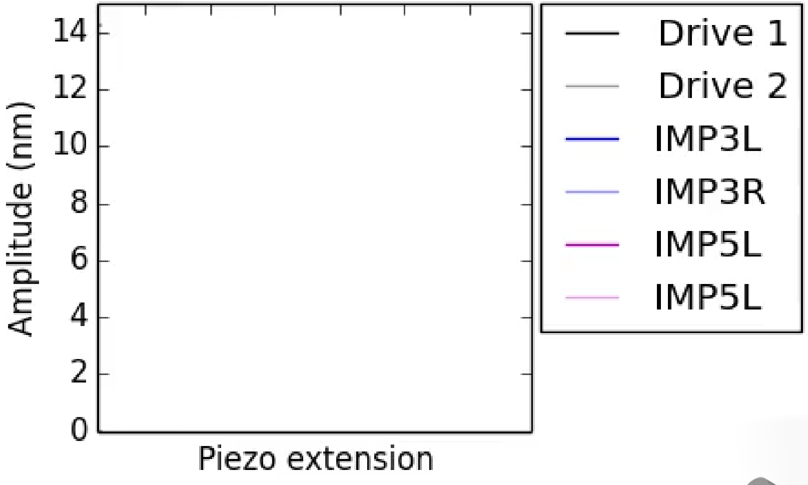
D. Platz *et al.* Nature Comm. (2012)

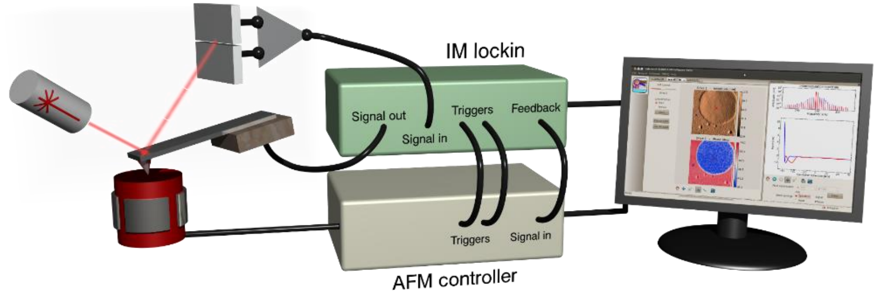


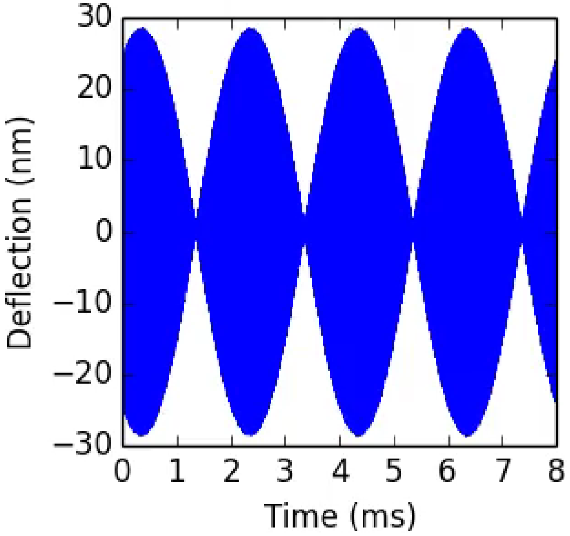
Intermodulation AFM

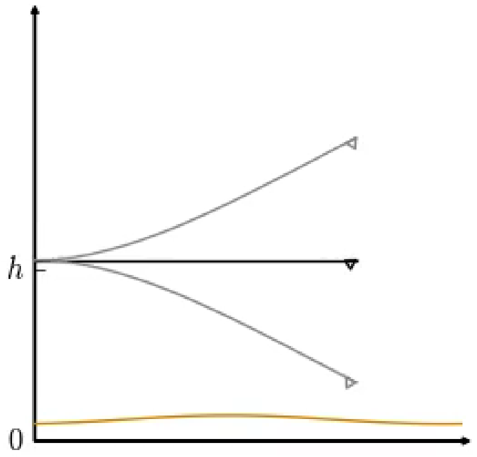




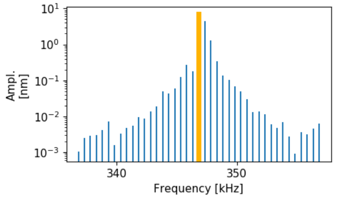


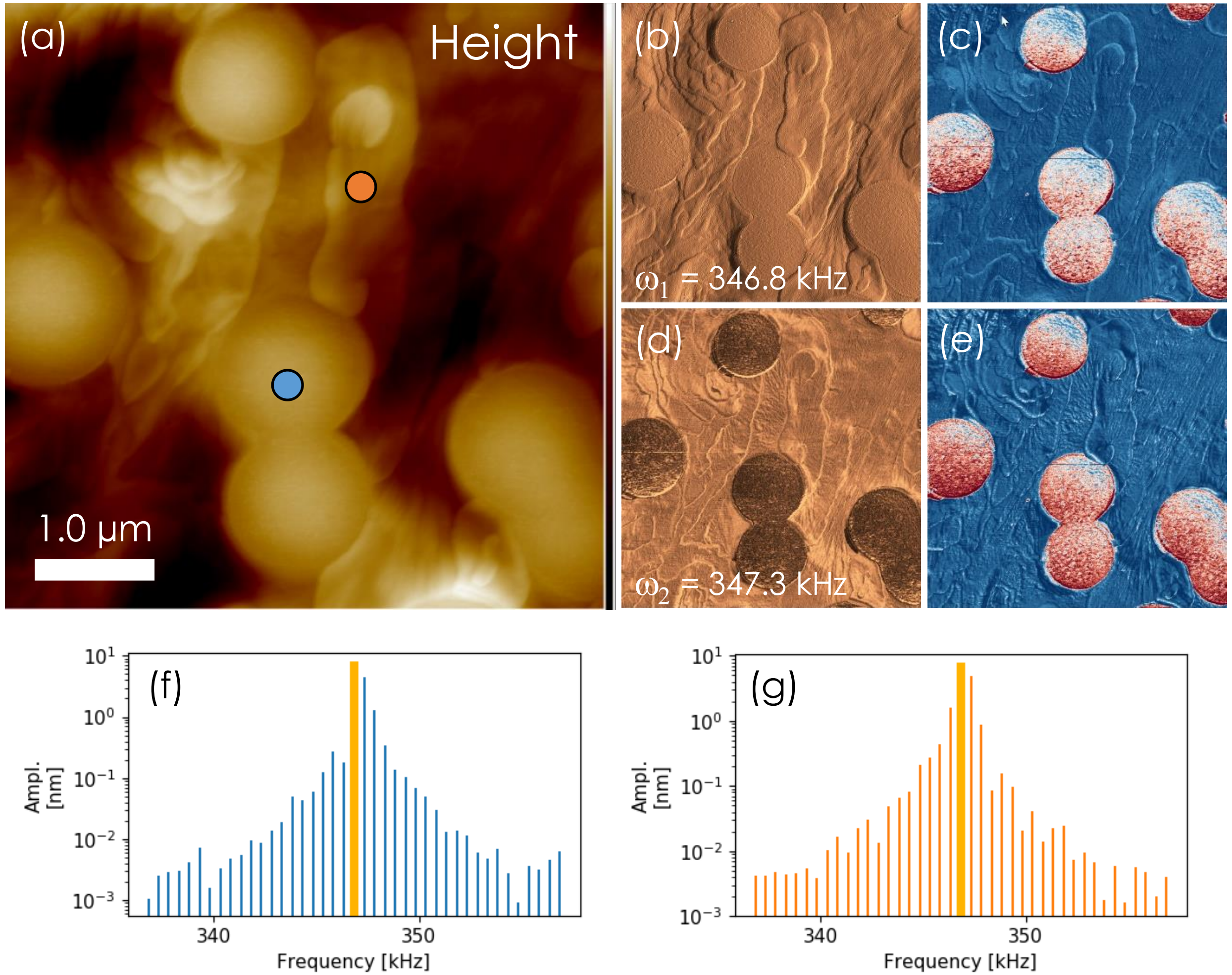






Blend of PS/PCL



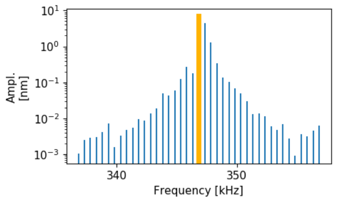


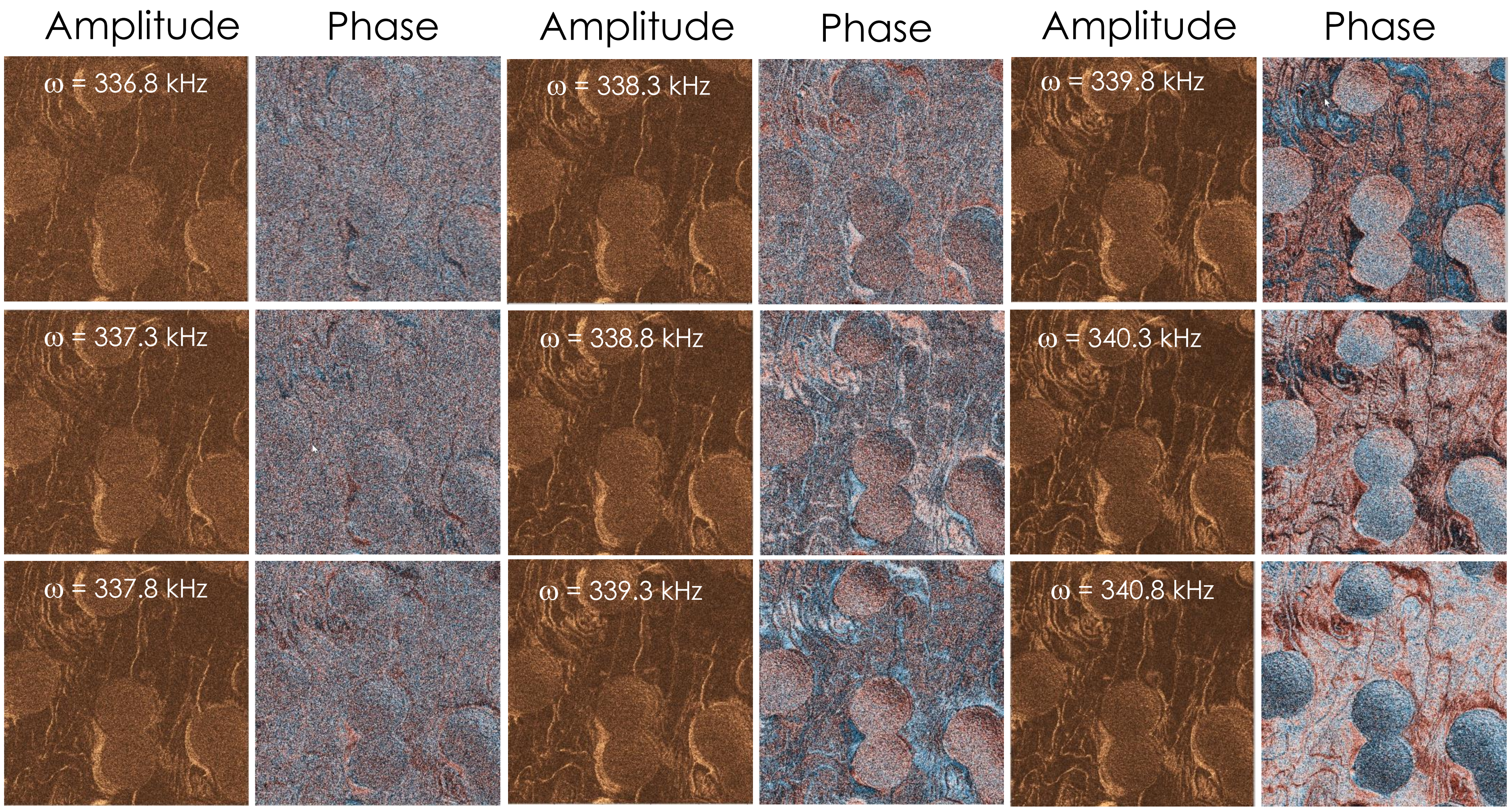
+ 40 others !

Soft Matter (2016), 12, 619.

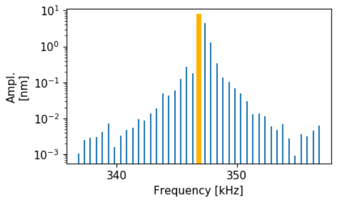
PS:PCL 30:70 blend

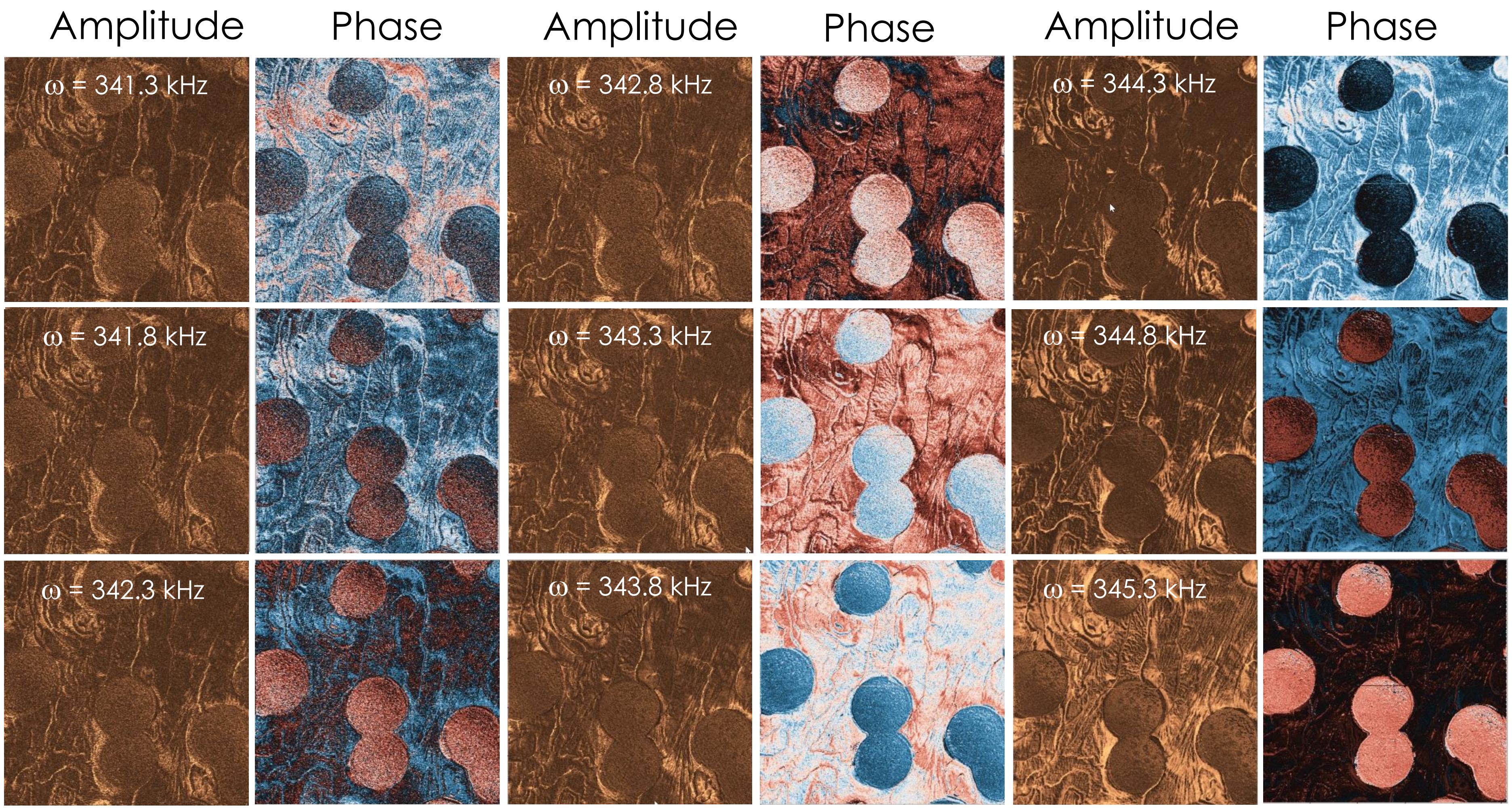
Blend of PS/PCL





Blend of PS/PCL





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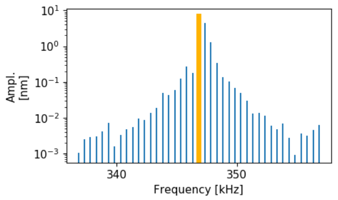

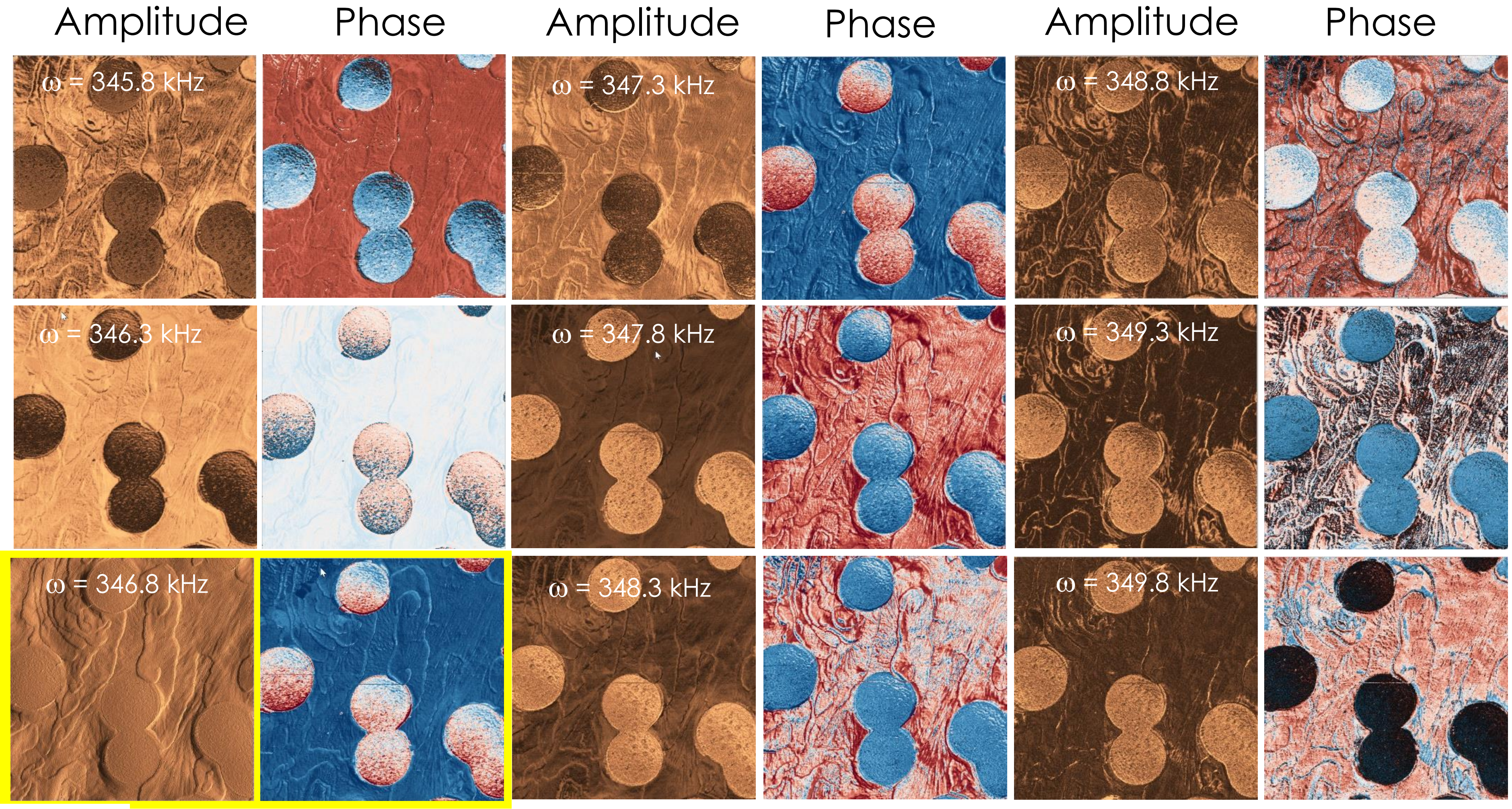

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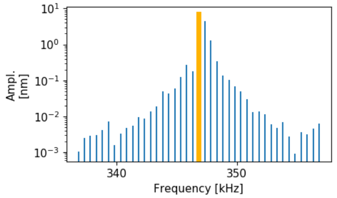

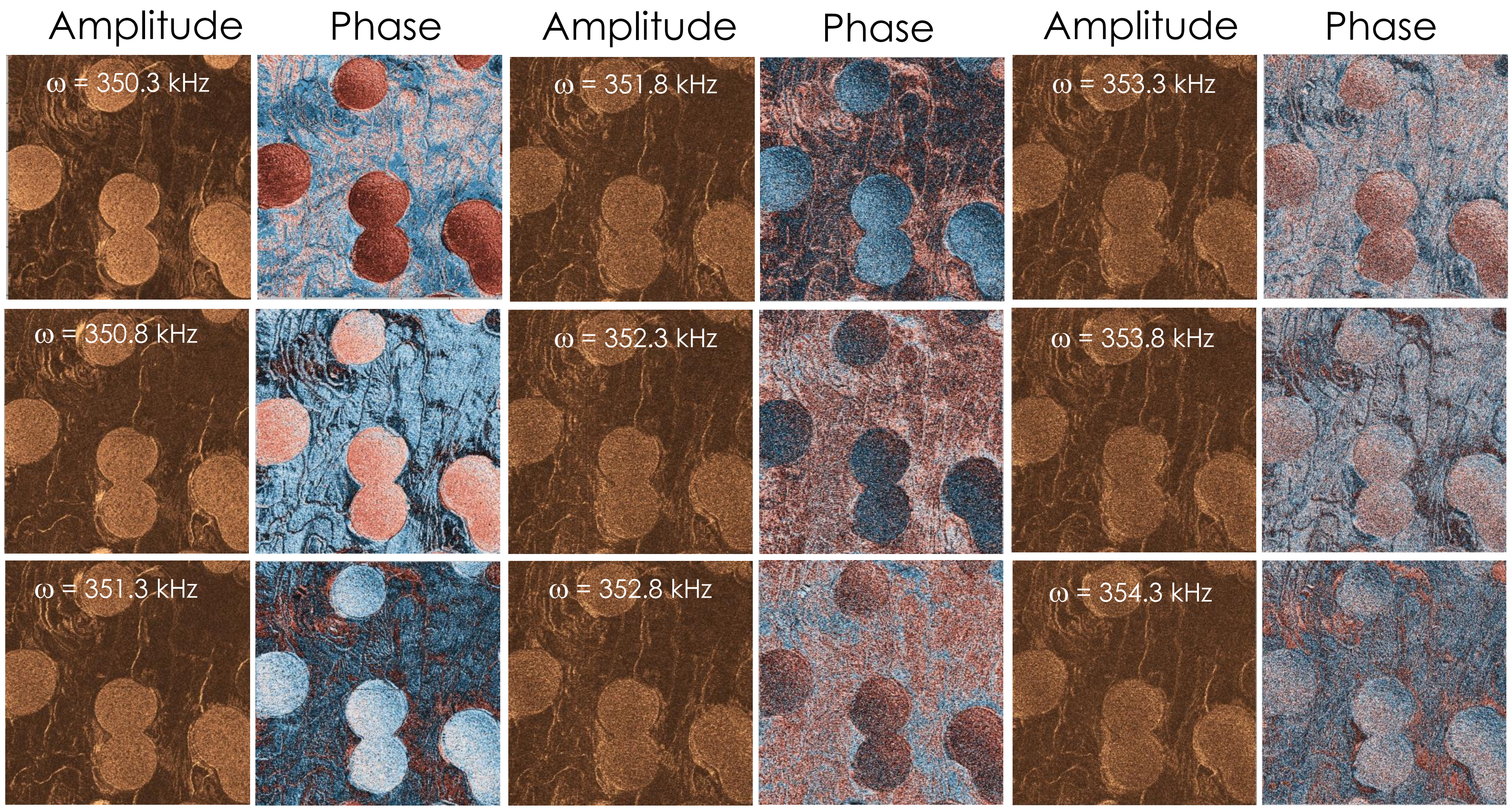

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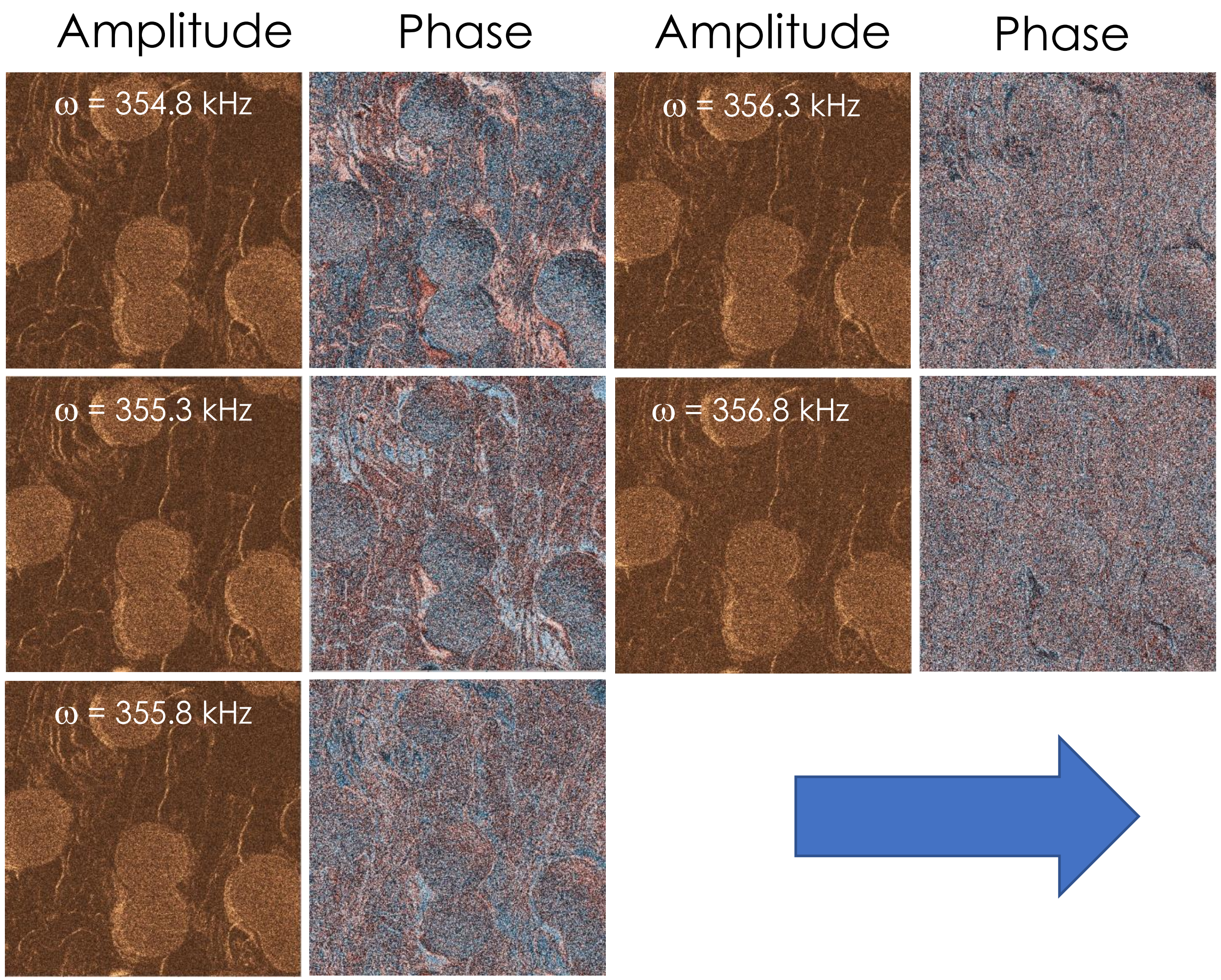
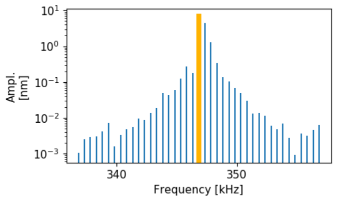
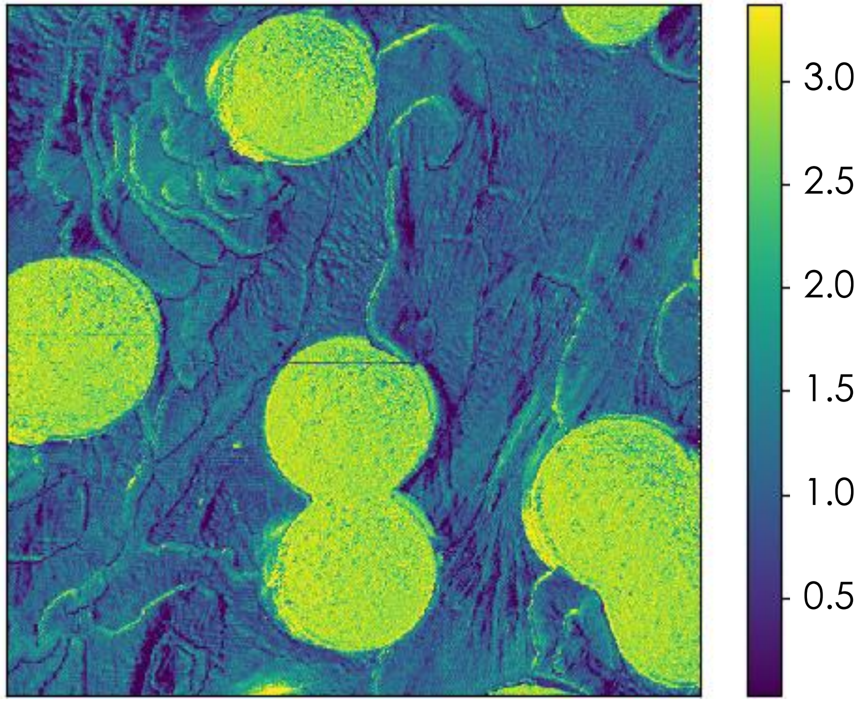
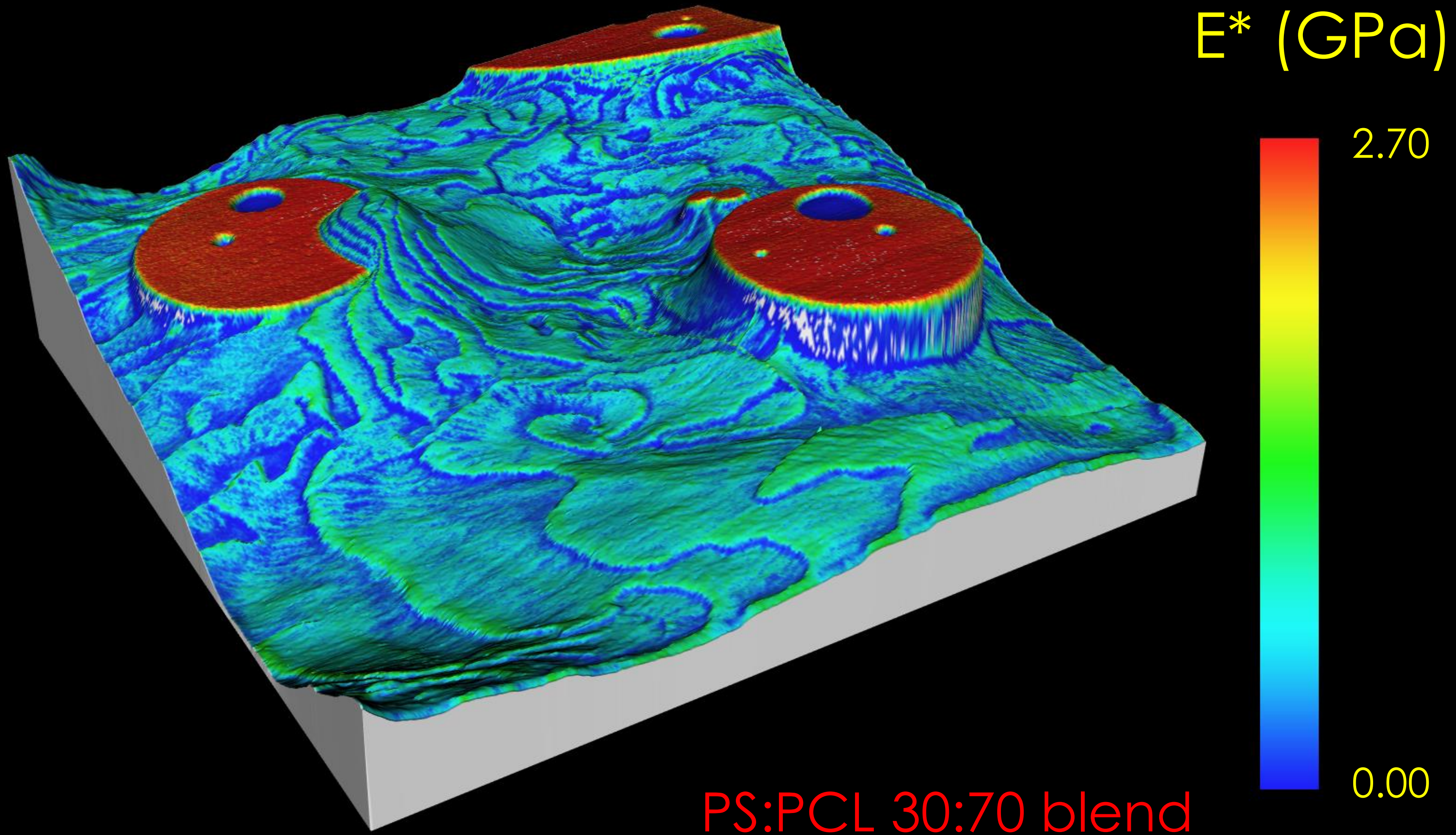




Contact Modulus (GPa)





PS:PCL 30:70 blend

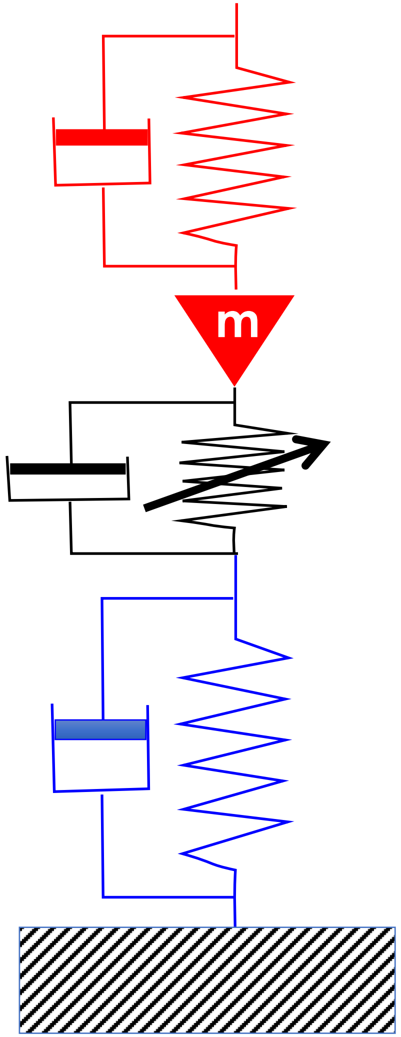
Moving Surface Model

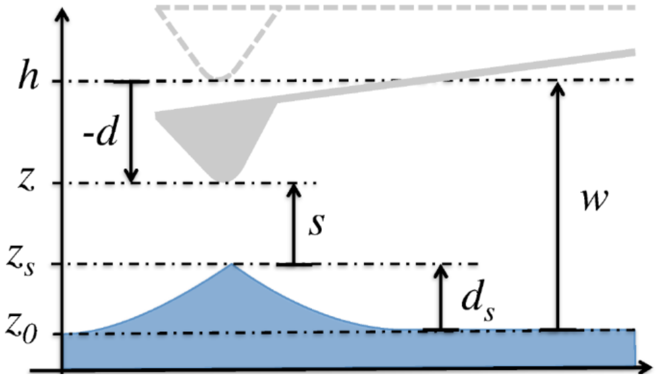
$$\boxed{m\ddot{d} + \gamma m\dot{d} + kd} = F_{TS}(s,\dot{s}) + F_{\text{drive}}$$

$$\boxed{\eta\dot{d}_s + k_s d_s} = -F_{TS}(s,\dot{s})$$

separation $s = (d - d_s) + h - z_0$

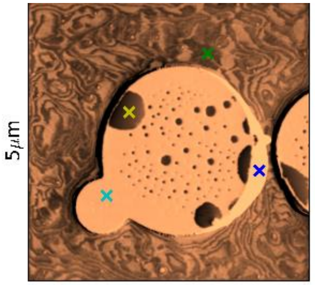
$$F_{\mathrm{TS}}(s,\dot{s}) = \begin{cases} 0 & \text{if } s > 0 \\ -F_{\mathrm{ad}} - k_{\mathrm{v}} s - \eta_{\mathrm{v}}\dot{s} & \text{if } s \leq 0 \end{cases}$$



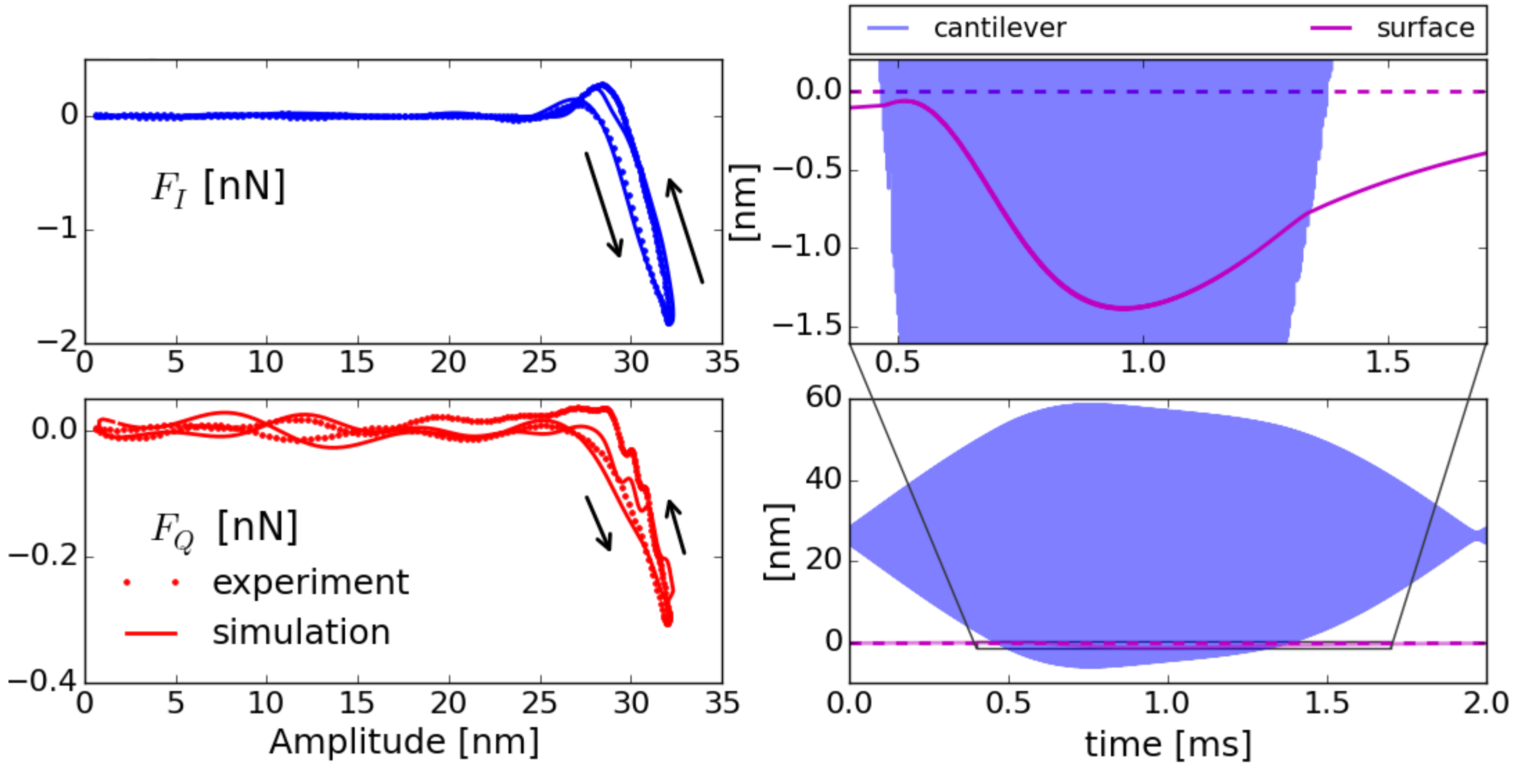


Soft Matter (2016), 12, 619.

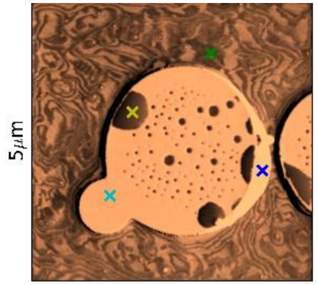
Fit to moving surface model



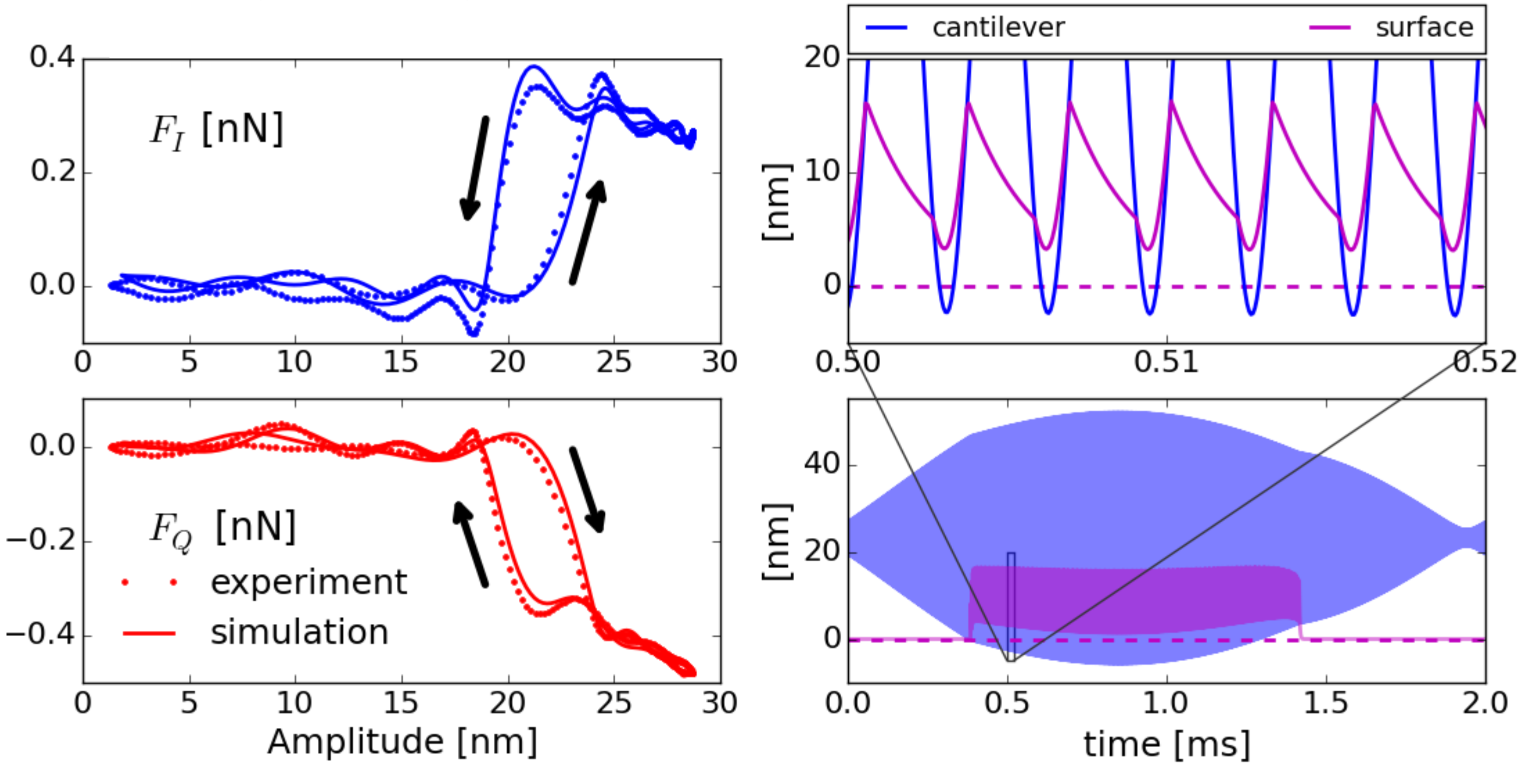
PS phase



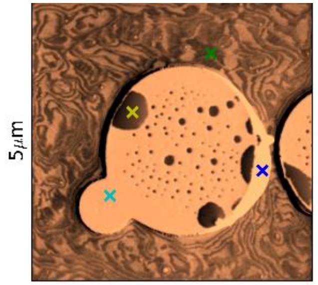
Fit to moving surface model



PCL amorphous phase

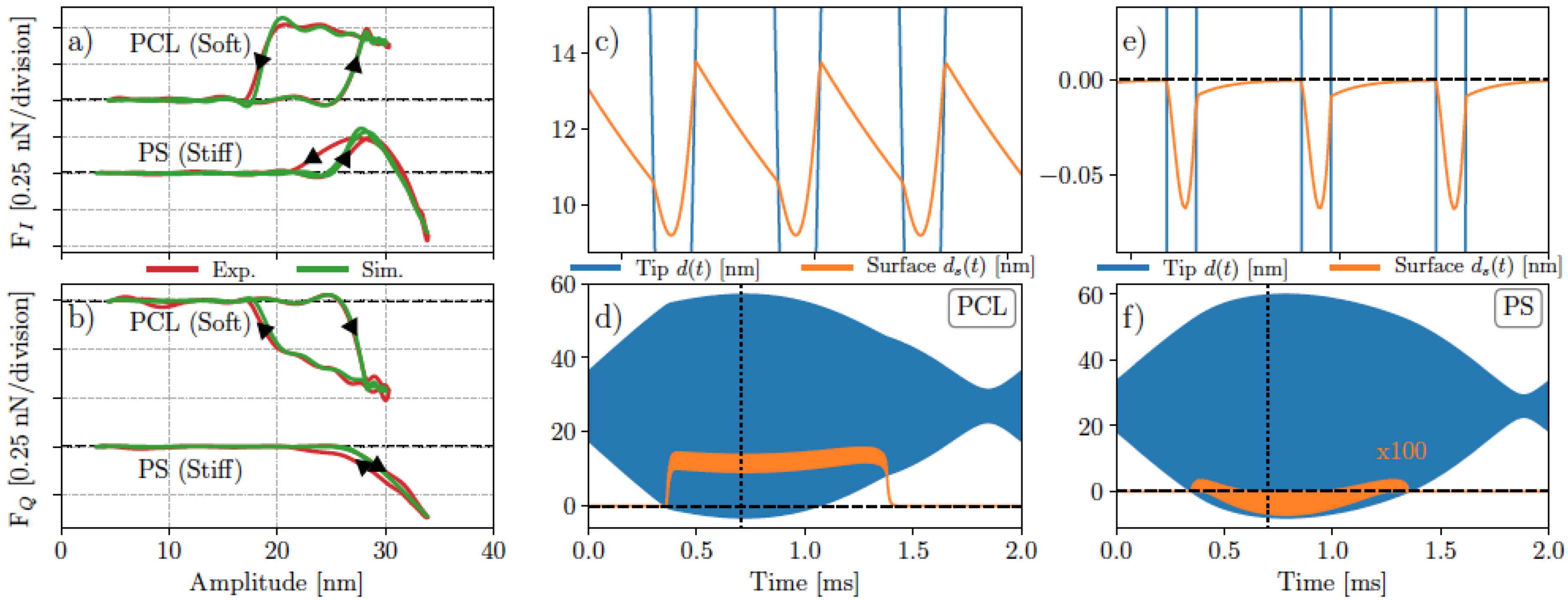


Fit to moving surface model



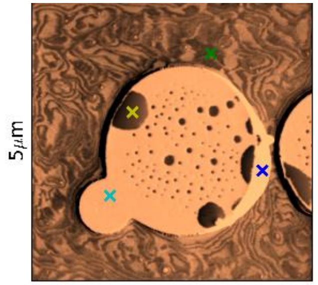
Soft Matter (2016), 12, 619.

Sample: PS-PCL   Cantilever: Tap300 (f_0=310.6 kHz, k=26.03 N/m, Q=465.1)

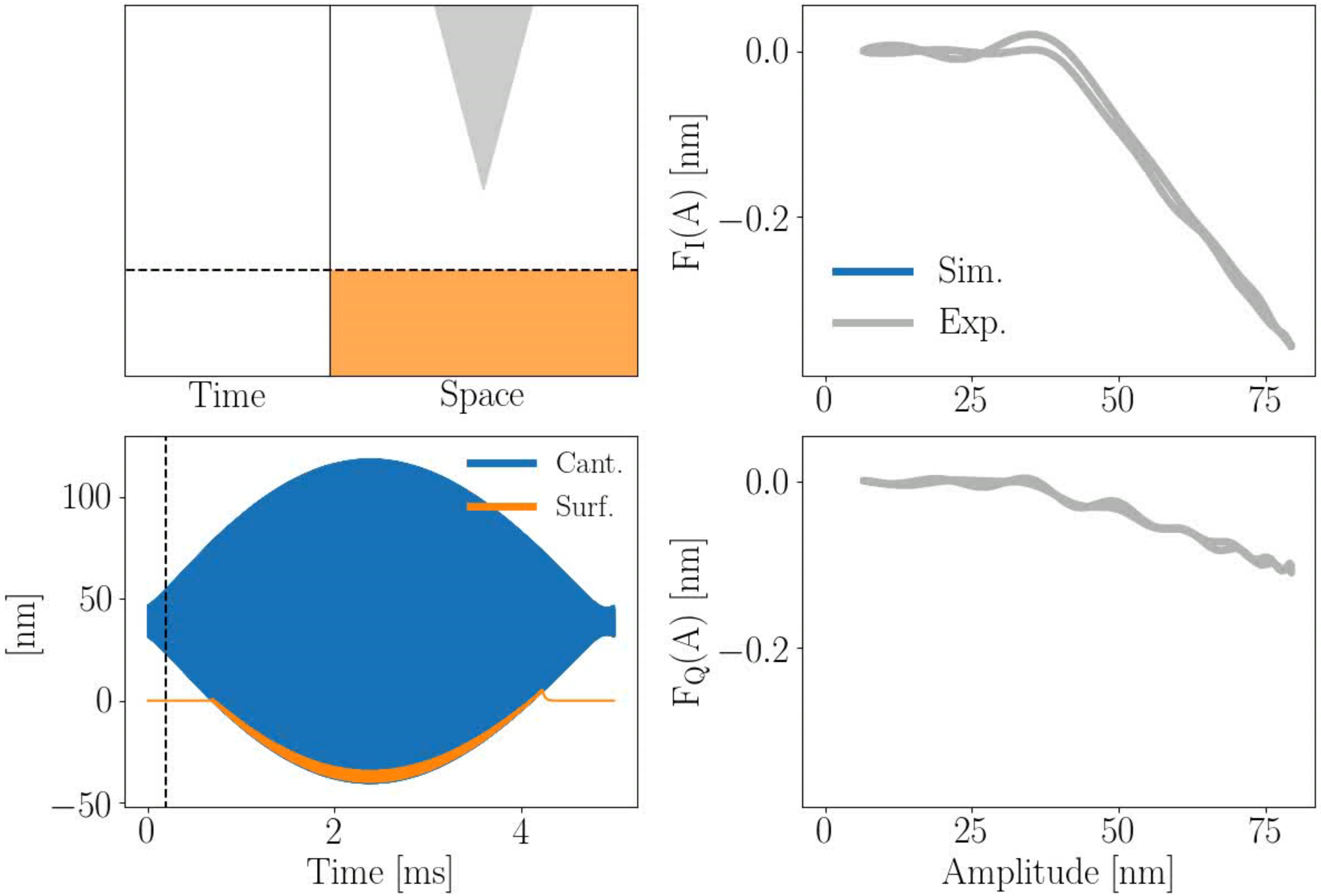


	h [nm]	τ_s [ns]	τ_v [ns]	k_s [N/m]	k_v [N/m]	F_{ad} [nN]	$K=k_s/k_v$	$R=\tau_s/\tau_v$
PCL	26.95	8118	580.3	0.0469	0.1557	2.859	0.3012	13.99
PS	25.98	735.2	152.8	32.53	1.097	3.88	29.65	4.811

Fit to moving surface model



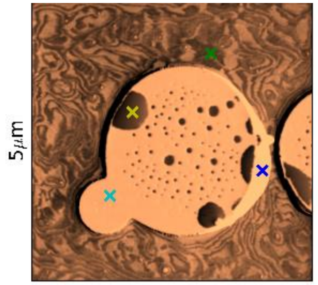
PS phase

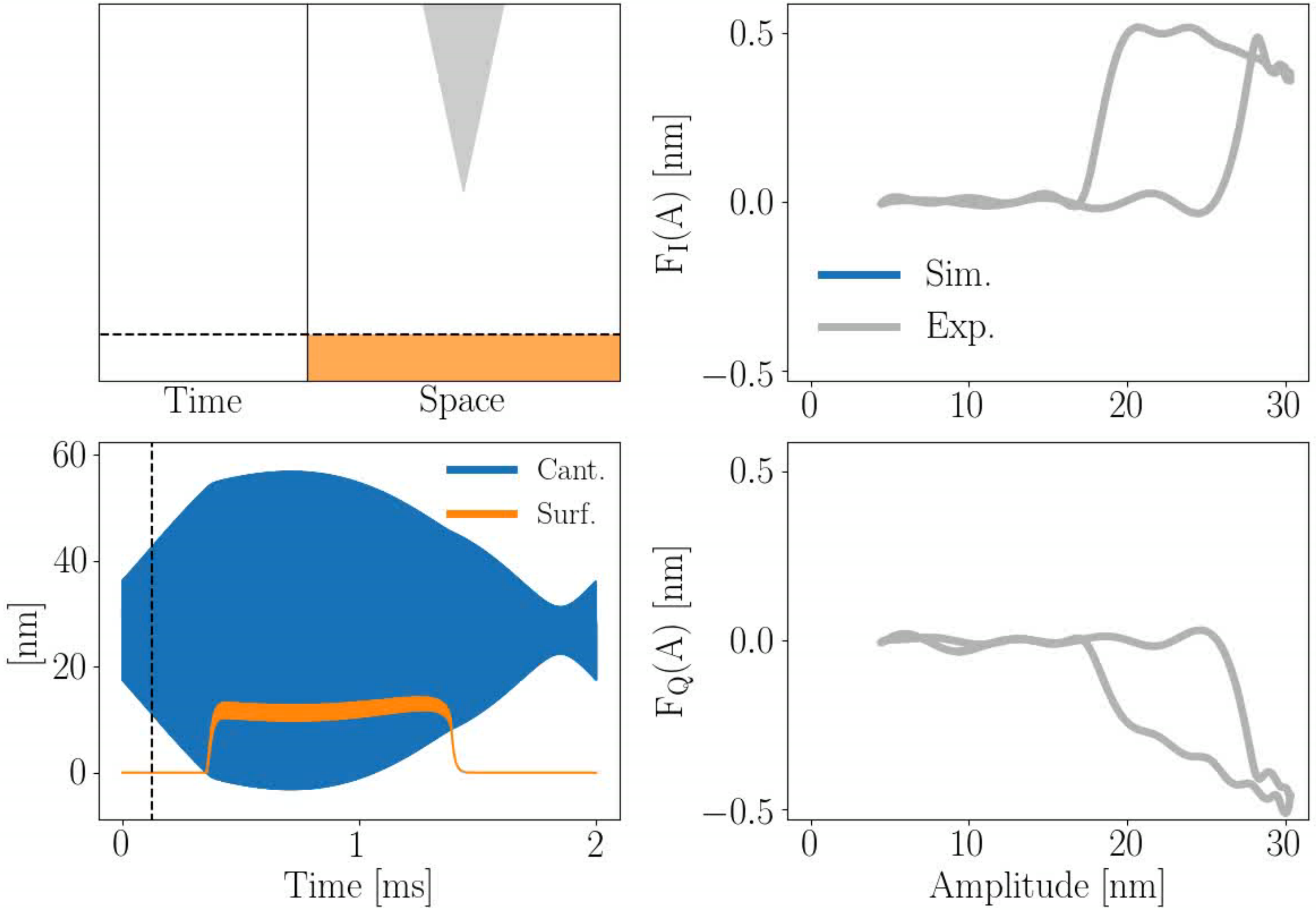


Courtesy of P.A. Thoren

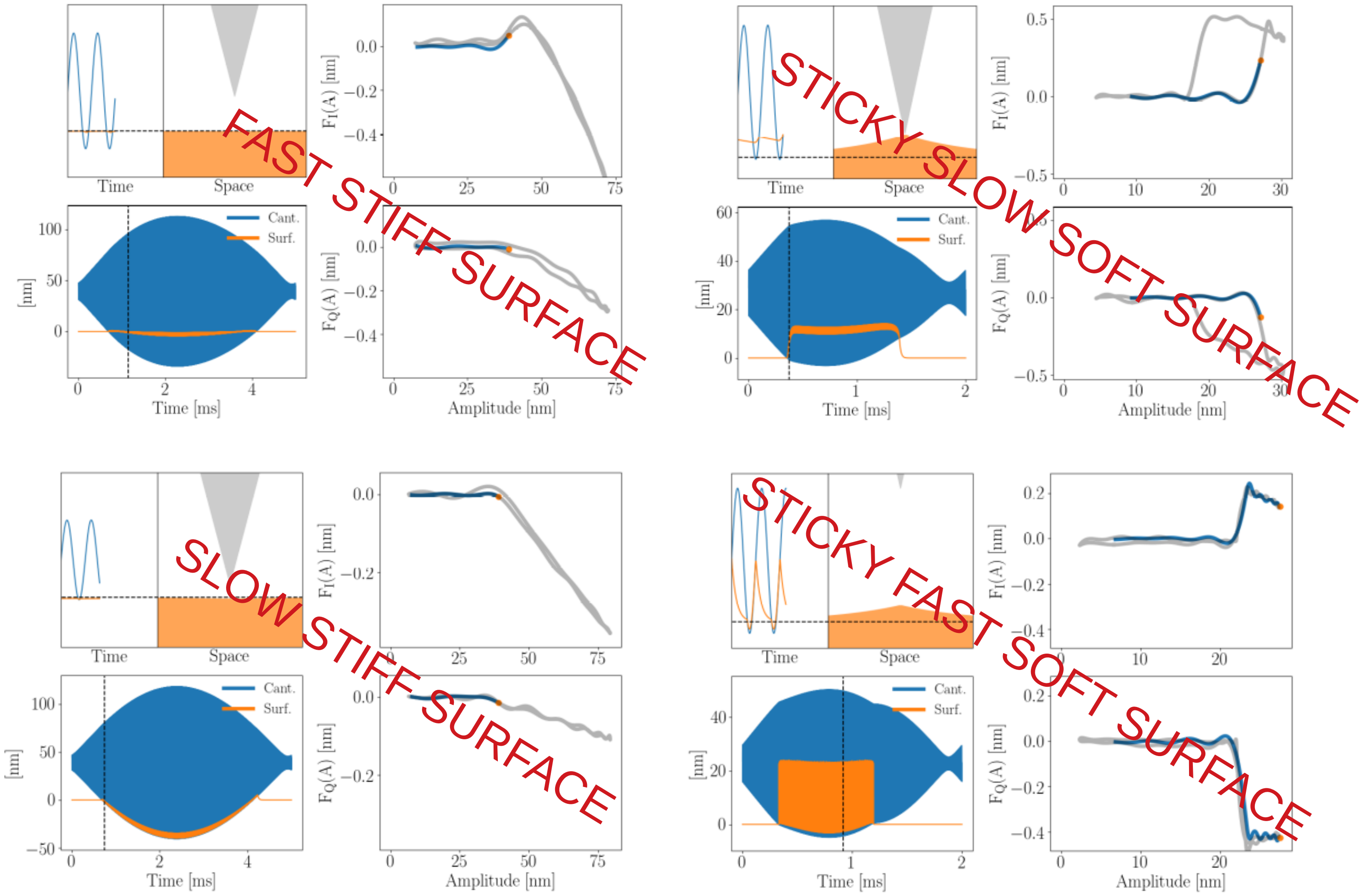
Fit to moving surface model

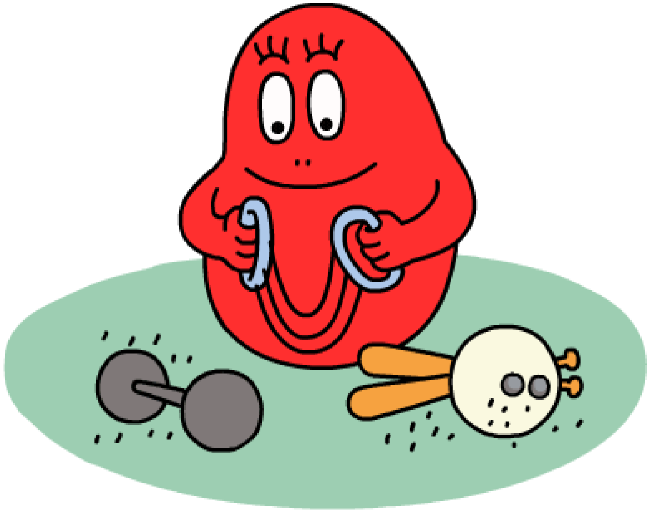
PCL amorphous phase





Courtesy of P.A. Thoren

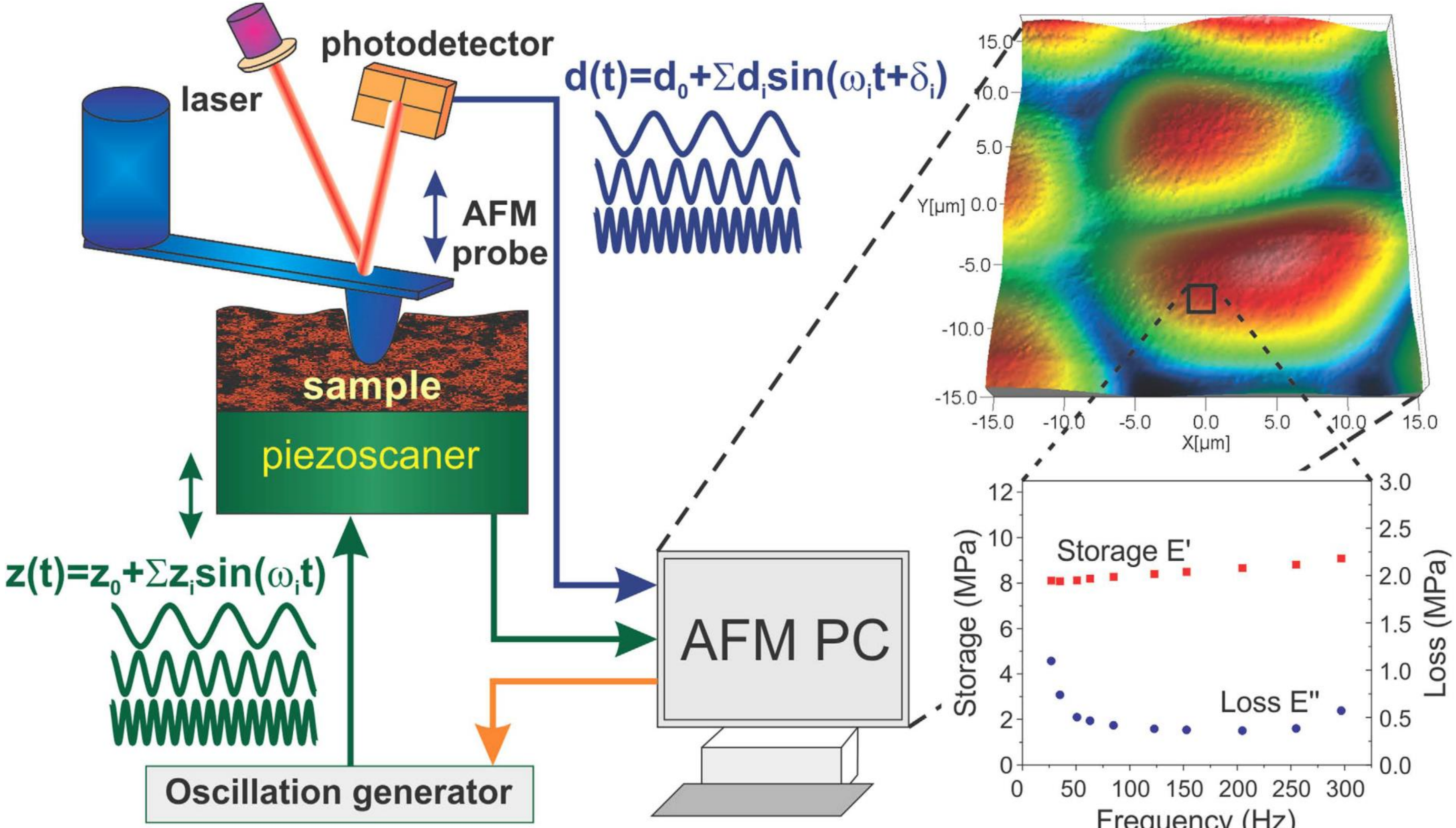




Courtesy of P.A. Thoren

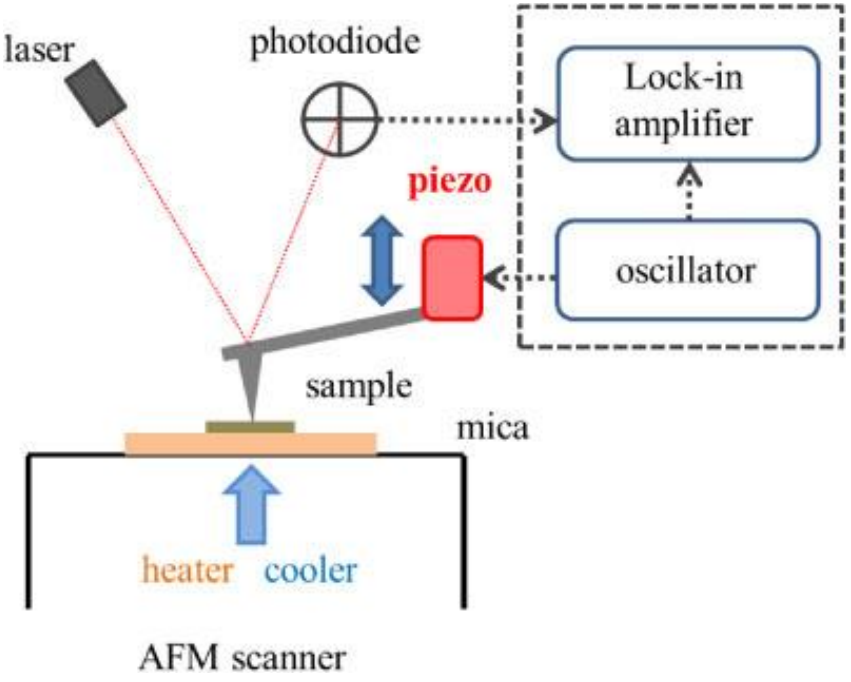
Scientific Reports | 5:12630 | DOI: 10.1038/srep12630

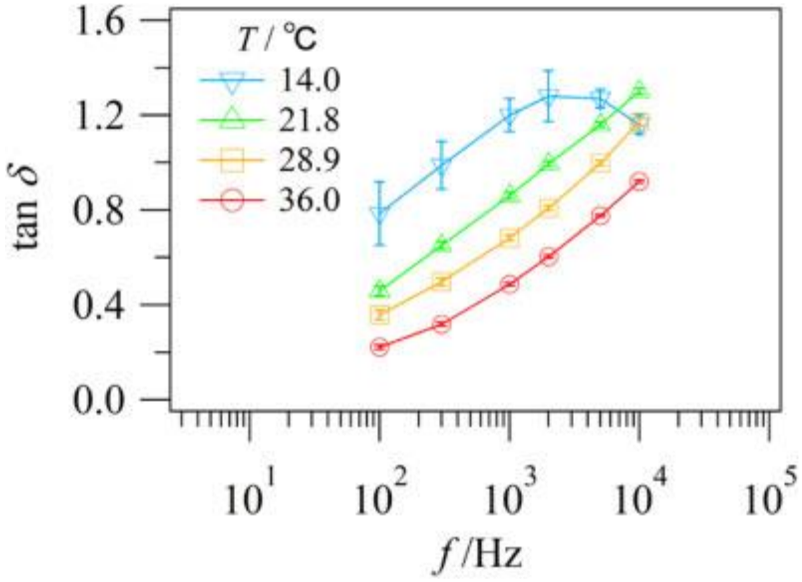
FT-NanoDMA

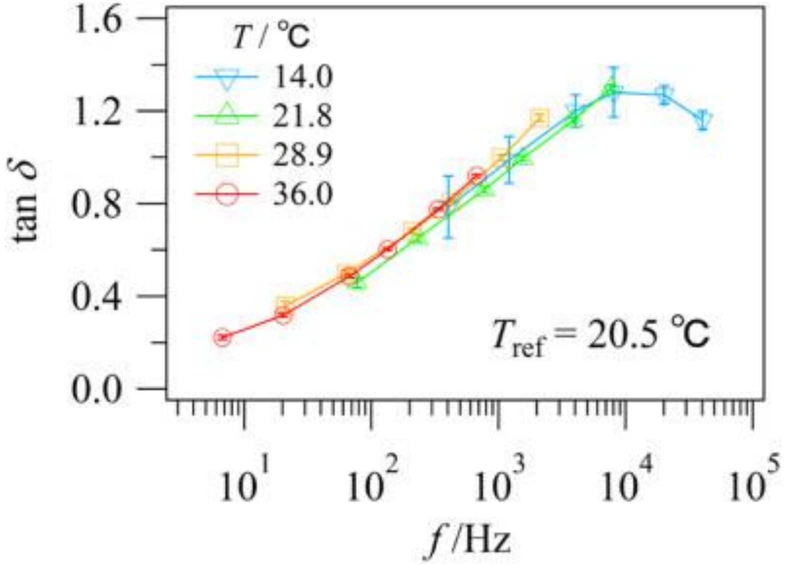


Nanorheological AFM

Vulcanized SBR







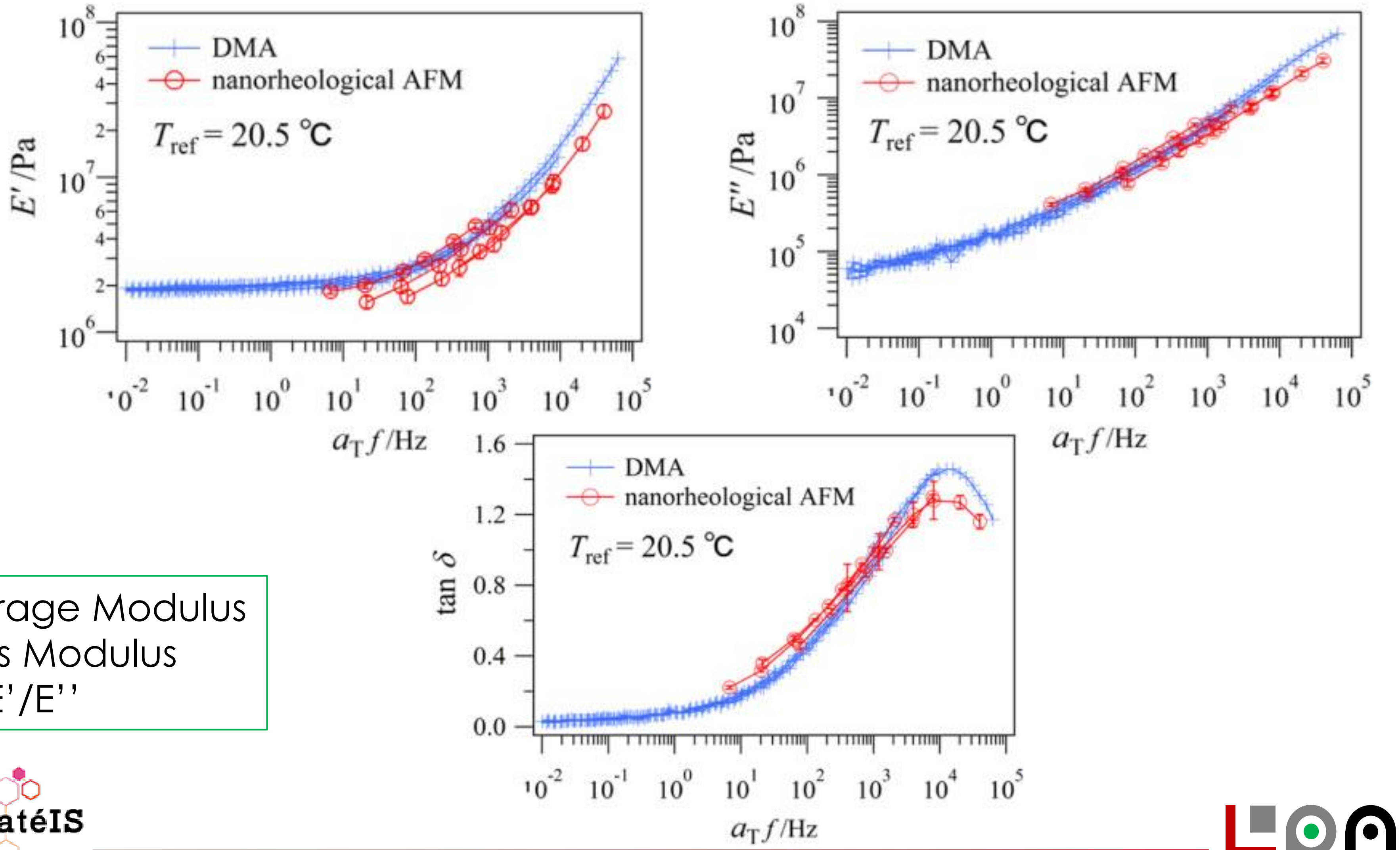
Williams–Landel–Ferry (WLF) equation

Japanese Journal of Applied Physics 57, 08NB08 (2018)

Nanorheological AFM

Vulcanized SBR

Japanese Journal of Applied Physics 57, 08NB08 (2018)



E’ = Storage Modulus
E’’ = Loss Modulus
tan δ = E’/E’’

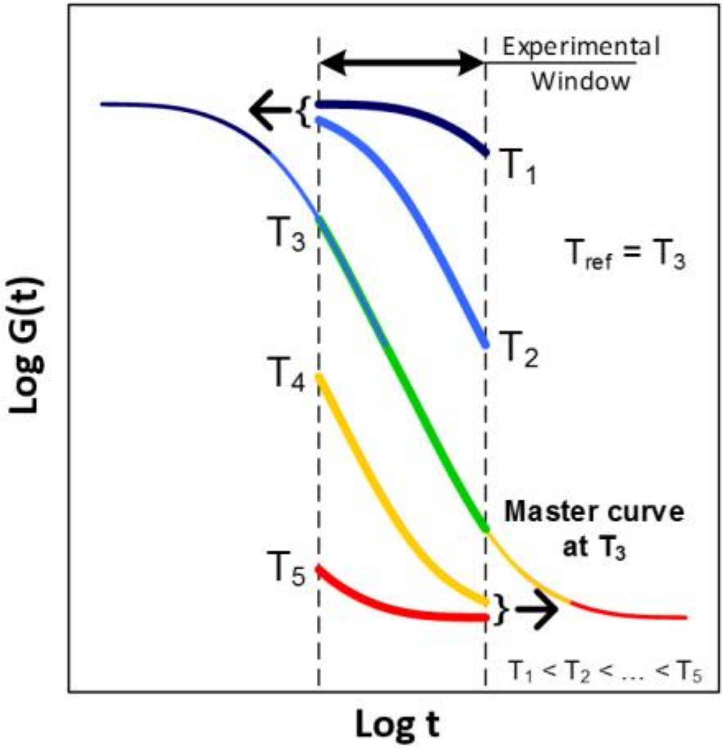
Take home message …

Combined, AFM measurements with non-resonant modes and resonant modes can provide

FV based Contact Resonance for stiff samples at higher frequencies.

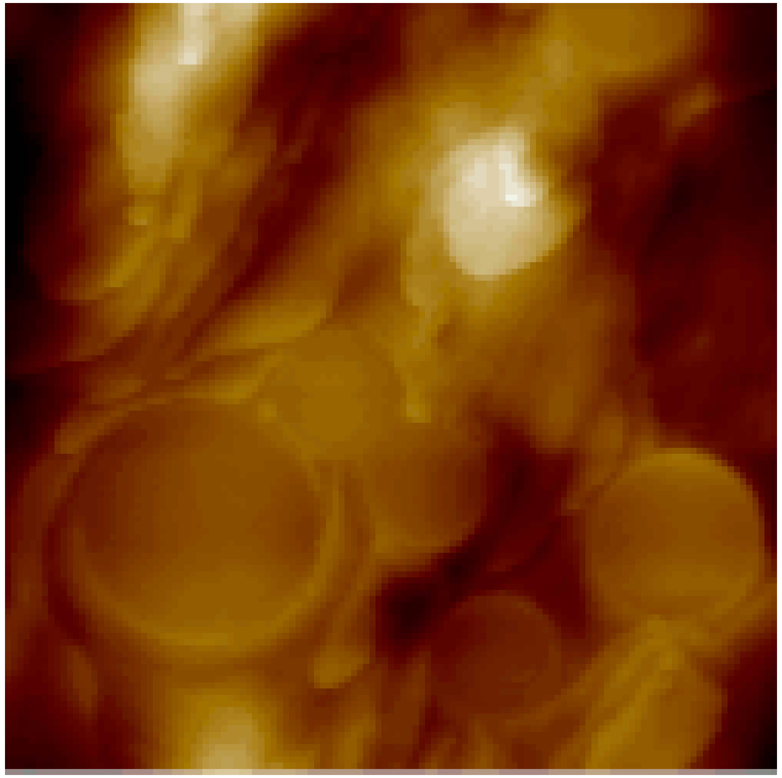
FV force nDMA, and FI/FQ curves (ImAFM) for soft samples at low frequencies.

FV and PFT cover wide range of ramp rates for time-temperature studies.

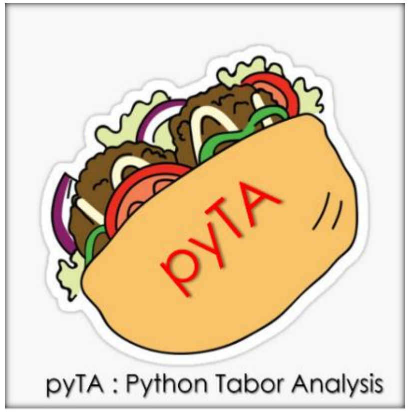


Conclusions

Multifrequency AFM methods are extremely promising but also need some (new) models to provide **quantitative parameters**.
Data-driven materials development and design (machine learning, AI) are most probably the key issue to achieve this goal.



For instance, recording of stiffness, deformation, adhesion and viscoelastic (E', E'', tan δ) property maps in parallel to the topography image is now possible with **quantitative values** and using an appropriate **data clustering approach** and adapted **mechanical model(s)** and **preprocessing** of the force curves (deep learning)..



Questions ?