REMISOL

GT NanoMécanique – 4/12/2019 Grenoble

(Rappel de) Mécanique du contact et (petite) analyse critique des modèles utilisés en AFM (ou pas)

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LABORATOIRE DE MÉCANIQUE ET GÉNIE CIVIL - UM/CNRS

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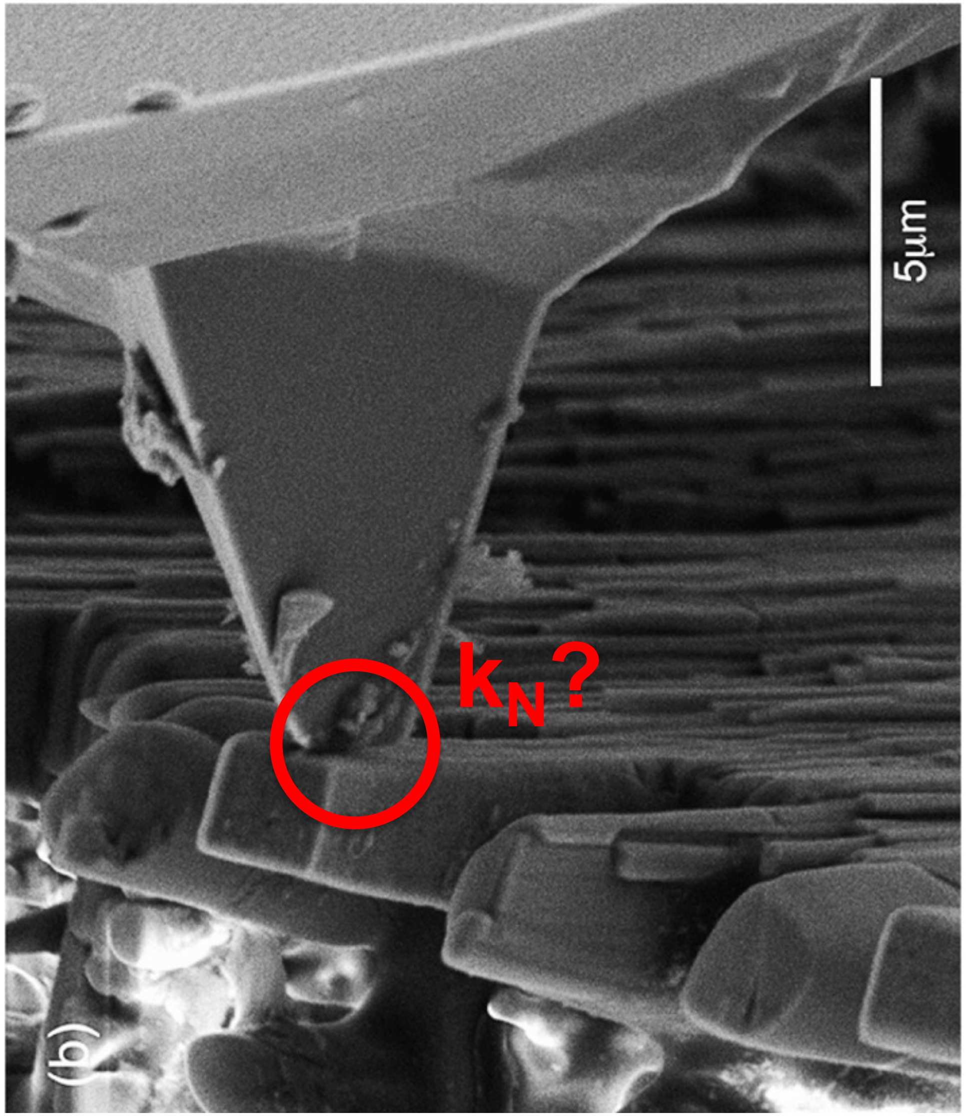
Bone micromechanics using in situ AFM in SEM



- Théorie de Hertz
- Adhésion (DMT-JKR)
- Autres cas et comparatif
- Anisotropie et viscosité
- Plasticité > NanoIndentation

[Jimenez-Palomar *et al.*, J. Mech. Beh. Bio. Mat., 2011]

Reminder

- Requirements:
  - Cantilever stiffness

$$\frac{d}{z}=\frac{1}{1+\dfrac{k_C}{k_N}}$$

$$k_N \gg k_C \rightarrow d = z$$

$$k_N \ll k_C \rightarrow d = 0$$

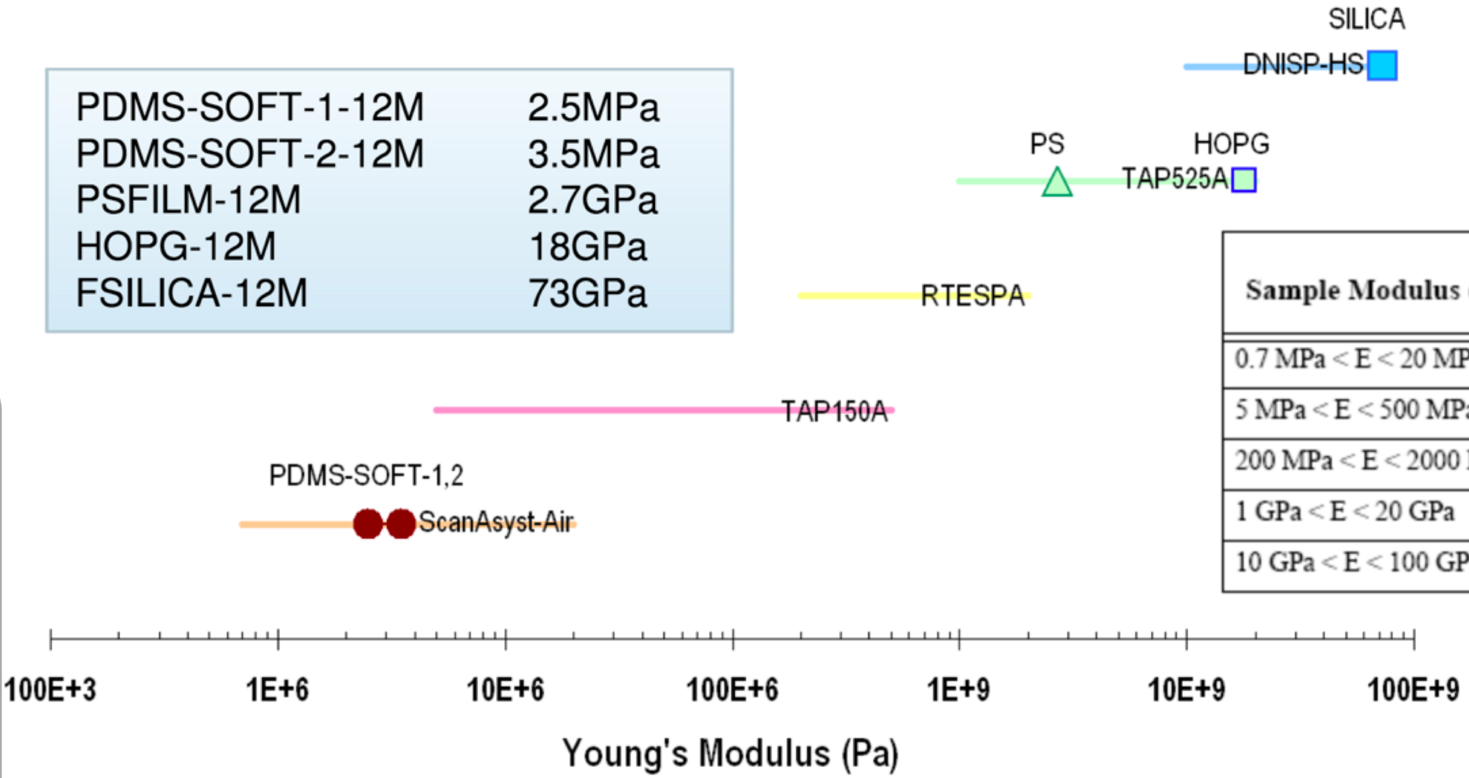
$$k_N \sim k_C$$

$$k_{N_{DMT}} = \left(6M^2R\left(F+F_{ad_{DMT}}\right)\right)^{1/3}$$

with

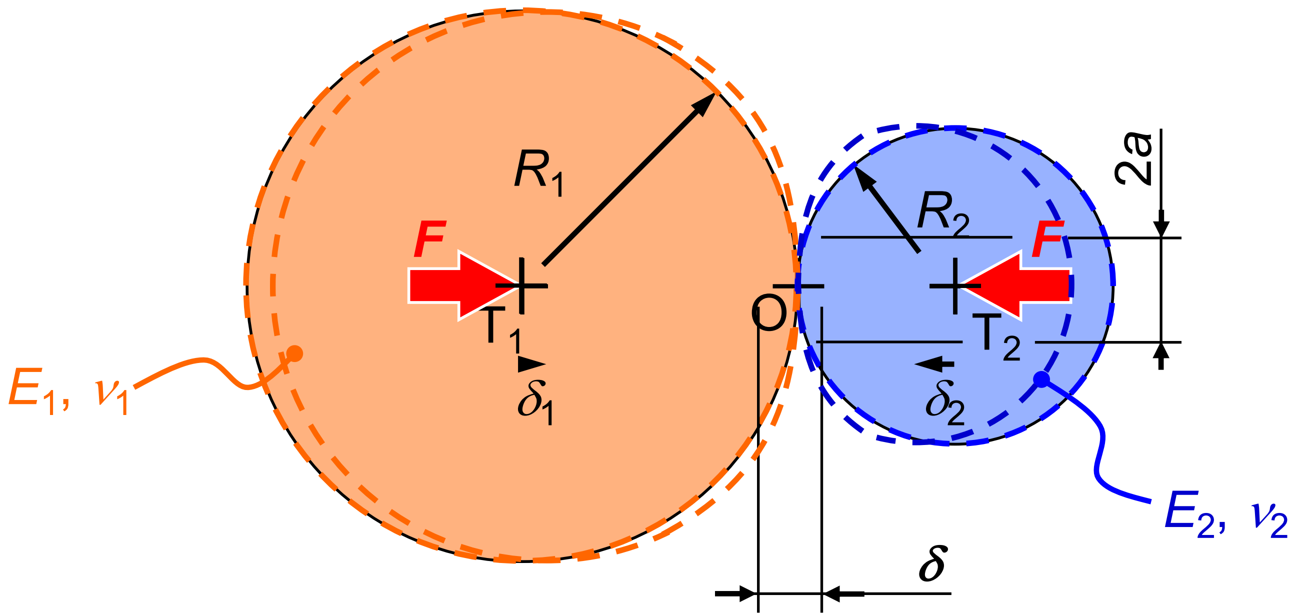
$$F = k_c d = k_c \chi V_{A-B}$$

PDMS-SOFT-1-12M	2.5MPa
PDMS-SOFT-2-12M	3.5MPa
PSFILM-12M	2.7GPa
HOPG-12M	18GPa
FSILICA-12M	73GPa



Sample Modulus (E)	Probe	Nominal Spring Constant (k)
0.7 MPa < E < 20 MPa	SNL-A	0.5 N/m
5 MPa < E < 500 MPa	Tap150A	5 N/m
200 MPa < E < 2000 MPa	RTESPA	40 N/m
1 GPa < E < 20 GPa	Tap525A	200 N/m
10 GPa < E < 100 GPa	DNISP-HS	350 N/m

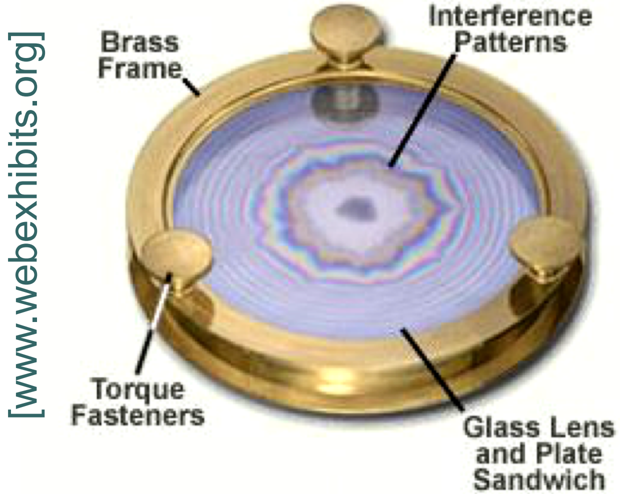
Contact mechanics



- Aims at finding the relationship between:
  - the load F and the relative displacement δ of the two bodies (far away from the contact)
  - the load F and the shape and size a of the contact area
  - the load F and the stress fields in each bodies

Contact mechanics

- Hertz's theory (1882 Ger. → 1896 Eng.)
  - Elastic deformation of two glass lenses in contact



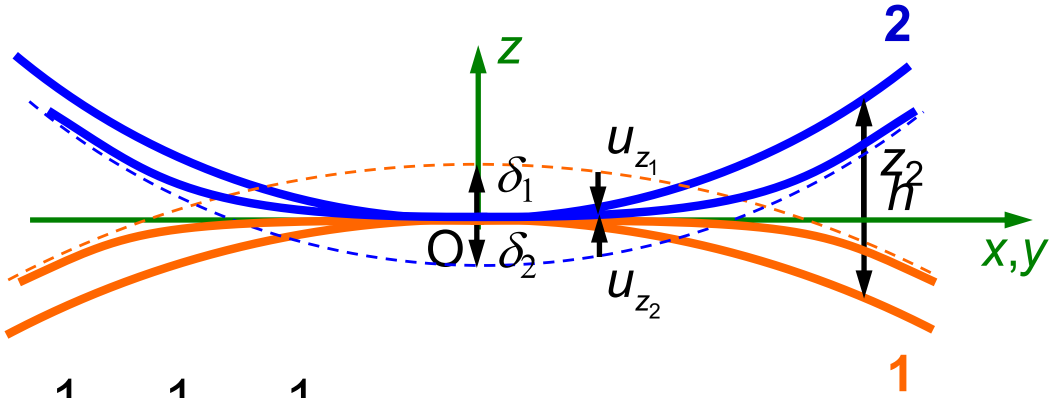
  - Assumptions:
    - Surfaces are continuous and **non-conforming** (i.e., $R_1 \neq -R_2$) → $a << R$
    - Strains are small: $a << R$
    - Linear elastic isotropic and homogeneous materials
    - Friction is neglected
  - Each solid is considered as an elastic half-space (plane) loaded over a small *elliptical* region of its plane surface

[K.L. Johnson, Contact Mechanics, Cambridge University Press, 2001]

Contact mechanics

- Hertz's theory (1882)
  - Each solid is locally described as paraboloid of revolution, for a sphere:

$$z_2 = \frac{1}{2R_2}\left(x^2 + y^2\right)$$



  - Distance separation between the two solids

$$h = z_1 + z_2 = \frac{1}{2R}\left(x^2 + y^2\right) \text{ with } \frac{1}{R} = \frac{1}{R_1} + \frac{1}{R_2}$$

  - Under load

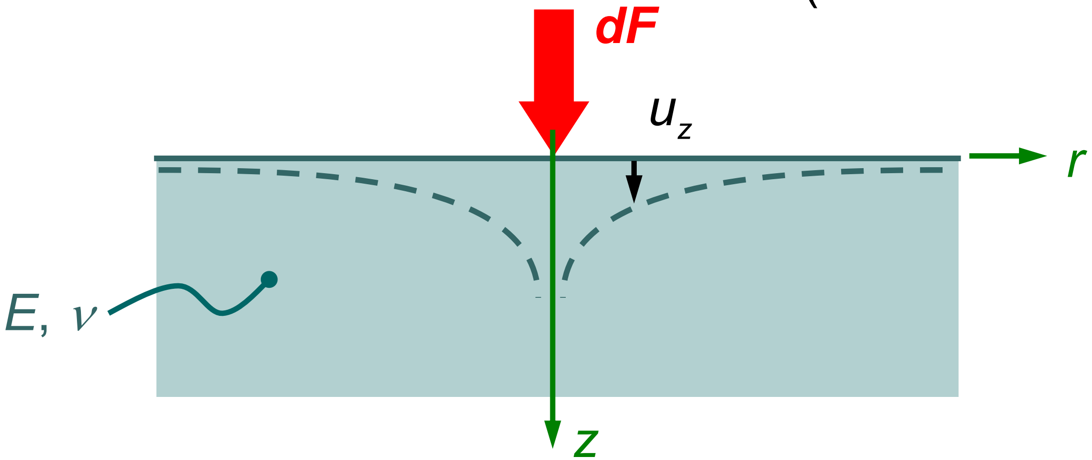
$$h - \left(\delta_1 + \delta_2\right) + \left(u_{z_1} + u_{z_2}\right) \begin{cases} = 0 \text{ in the contact area} \\ > 0 \text{ outside } (\underline{\underline{\sigma}} \cdot \underline{z} = \underline{0}) \end{cases}$$

  - What's the pressure distribution at the contact surface?

[K.L. Johnson, Contact Mechanics, Cambridge University Press, 2001]

Contact mechanics

- Hertz's theory (1882)
  - ➲ Concentrated normal force (Green's function method):



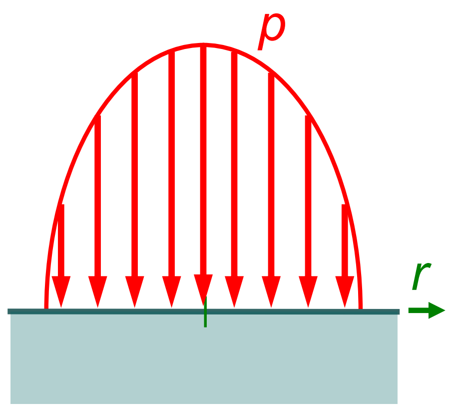
$$u_z(r) = \frac{\left(1-\nu^2\right)}{E}\frac{dF}{\pi r}$$

+ $u_r(r)$ and stress field (r,z)

→ linear elasticity → superposition for a distributed normal load $f(x,y)$!

$$u_z(x,y) = \frac{1-\nu^2}{E}\iint_S \frac{dF(x',y')}{\pi\sqrt{(x-x')^2+(y-y')^2}}$$

and $\dfrac{1}{2R}(x^2+y^2)-\left(\delta_1+\delta_2\right)+\left(u_{z_1}(x,y)+u_{z_2}(x,y)\right)=0$



- ➲ Hertz's pressure distribution: $p(r) = p_0\sqrt{1-\left(r/a\right)^2}$

[K.L. Johnson, Contact Mechanics, Cambridge University Press, 2001]

Contact mechanics

- Hertz's theory (1882)
  - ➲ Results: macroscopic relationship

$$a = \left(\frac{3RF}{4E^*}\right)^{1/3} \qquad p_0 = \left(\frac{6FE^{*2}}{\pi^3 R^2}\right)^{1/3}$$

$$\delta = \delta_1 + \delta_2 = \left(\frac{9F^2}{16RE^{*2}}\right)^{1/3} = \frac{a^2}{R} \quad \Bigg| \quad \boxed{F = \frac{4}{3}E^*\sqrt{R}\delta^{3/2}} \qquad F = \frac{4}{3}E^*\frac{a^3}{R}$$

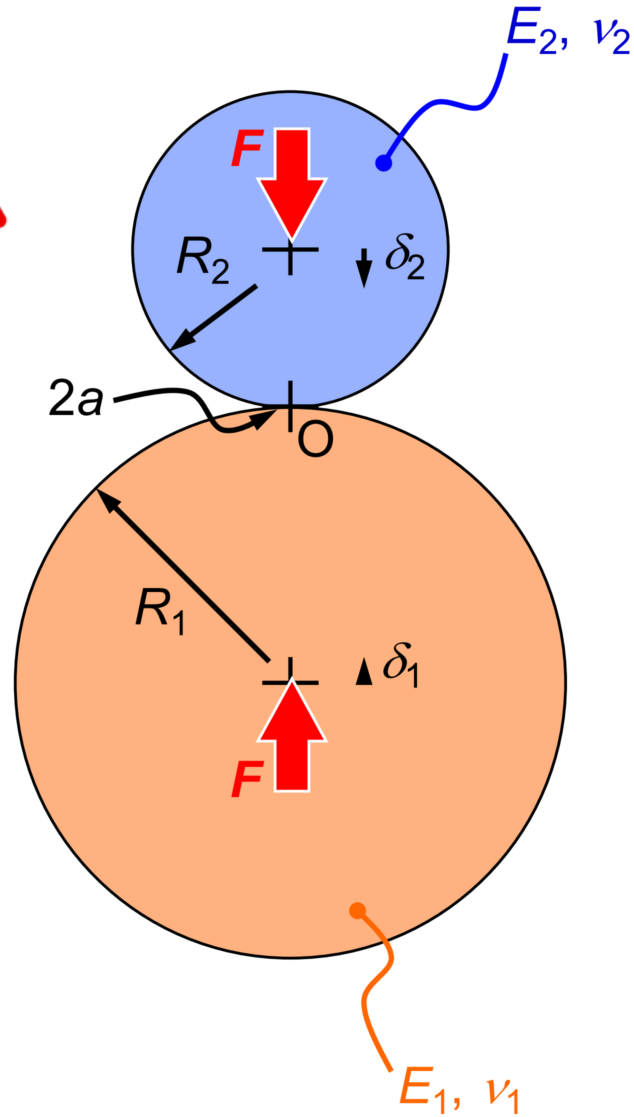
$$\boxed{k_N = \frac{\partial F}{\partial \delta} = \left(6E^{*2}RF\right)^{1/3}}$$

$$\mathbf{0} \leq \boldsymbol{k_T} \leq \frac{\mathbf{2}-\boldsymbol{\nu}}{\mathbf{2}(\mathbf{1}-\boldsymbol{\nu})}$$

$$\frac{1}{E^*} = \frac{1-\nu_1^2}{E_1} + \frac{1-\nu_2^2}{E_2} \qquad \frac{1}{R} = \frac{1}{R_1} + \frac{1}{R_2}$$

Contact modulus

Plane-stress modulus



Contact mechanics

- Hertz's theory (1882)
  - ➲ Examples in the case of Si-AFM probe ($R_2 = 10$ nm, $E_2 \approx 150$ GPa) in contact with the planar surface ($R_1 \rightarrow \infty$) of different materials [www.ntmdt.com]

Material and its Young's modulus	Contact area radius a, nm		Penetration due to deformation h, nm		Contact pressure P, GPa	
Elastomer, $E = 0.65\,\mathrm{GPa}$	3.74	8.04	1.04	6.46	0.11	0.25
PS, $E = 1\,\mathrm{GPa}$	3.24	6.98	1.05	4.87	0.15	0.33
Copper, $E = 120\,\mathrm{GPa}$	0.79	1.7	0.062	0.29	2.55	5.51
Tungsten, $E = 400\,\mathrm{GPa}$	0.68	1.46	0.046	0.21	3.44	7.47
Diamond, $E = 1000\,\mathrm{GPa}$	0.64	1.38	0.041	0.19	3.88	8.36
at loading force F, nN						
	5	50	5	50	5	50

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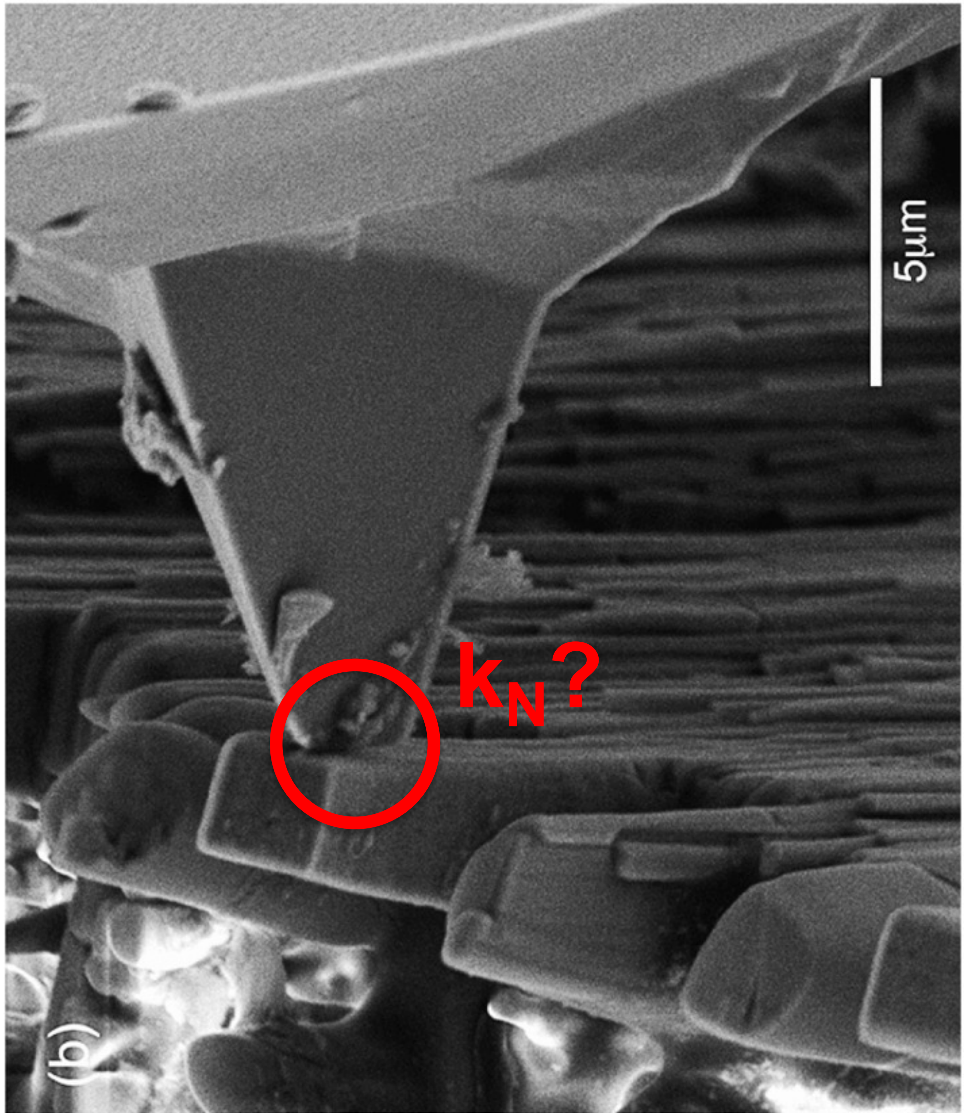

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[Jimenez-Palomar *et al*., J. Mech. Beh. Bio. Mat., 2011]

Contact mechanics

- **Adhesion** (~contact at the nano-scale: capillarity, van der Waals, etc.)
  - ➲ Derjaguin, Muller and Toporov (DMT) model [J. Coll. Int. Sci., 1975]
    - For stiff material (glassy polymers, crystal), small radius, low adhesive and long range interaction forces outside the contact area
    - Simple “translation” in load of Hertz’s equations

$$F \to F + F_{adh} \quad \text{with} \quad F_{\text{adh}} = 2\pi R w_{\text{adh}}$$

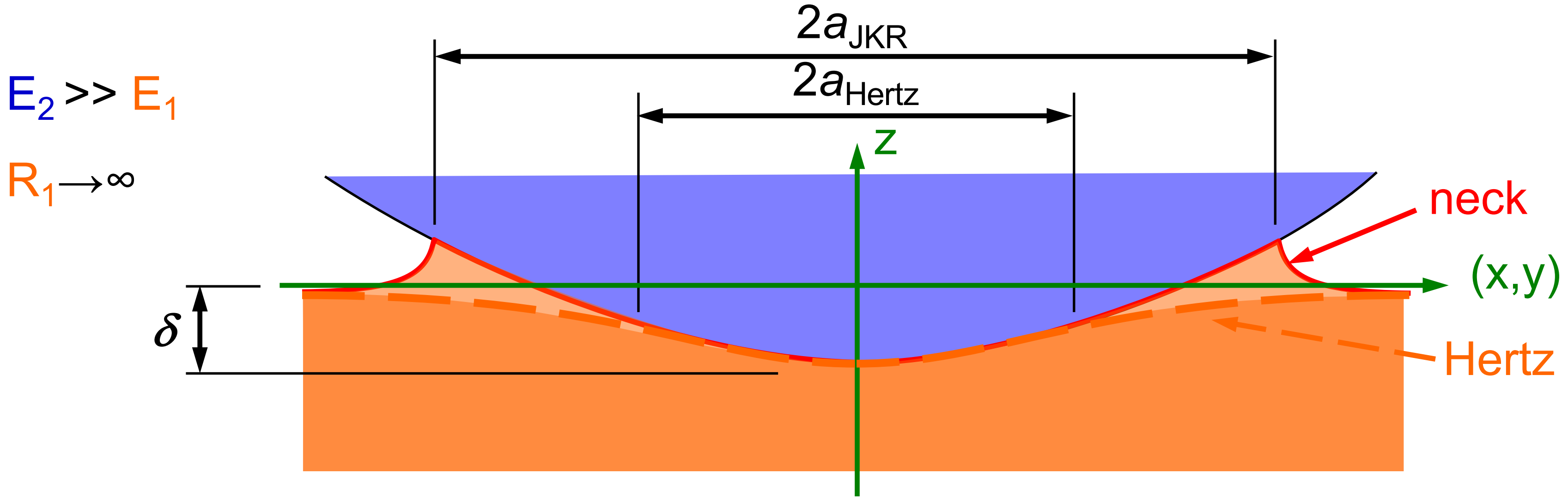
and Dupré’s work of adhesion $w_{\text{adh}} = \gamma_1 + \gamma_2 - \gamma_{12}$

$$a = \left[\frac{3R\left(F + F_{\text{adh}}\right)}{4E^*} \right]^{1/3} \qquad k_N = \left[6E^{*2} R \left(F + F_{\text{adh}} \right) \right]^{1/3}$$

- At pull-off δ=0 and a =0
- Applicable in the case of capillarity forces with $w_{\text{adh}} \approx 2\gamma_{\text{LV}}$

Contact mechanics

- **Adhesion** (~contact at the nano-scale: capillarity, van der Waals, etc.)
  - ➲ Johnson, Kendall and Roberts (JKR) model [Proc. R. Soc. London A, 1971]
    - For soft sample (elastomers), large radius, strong adhesive and short range interaction forces inside the contact area



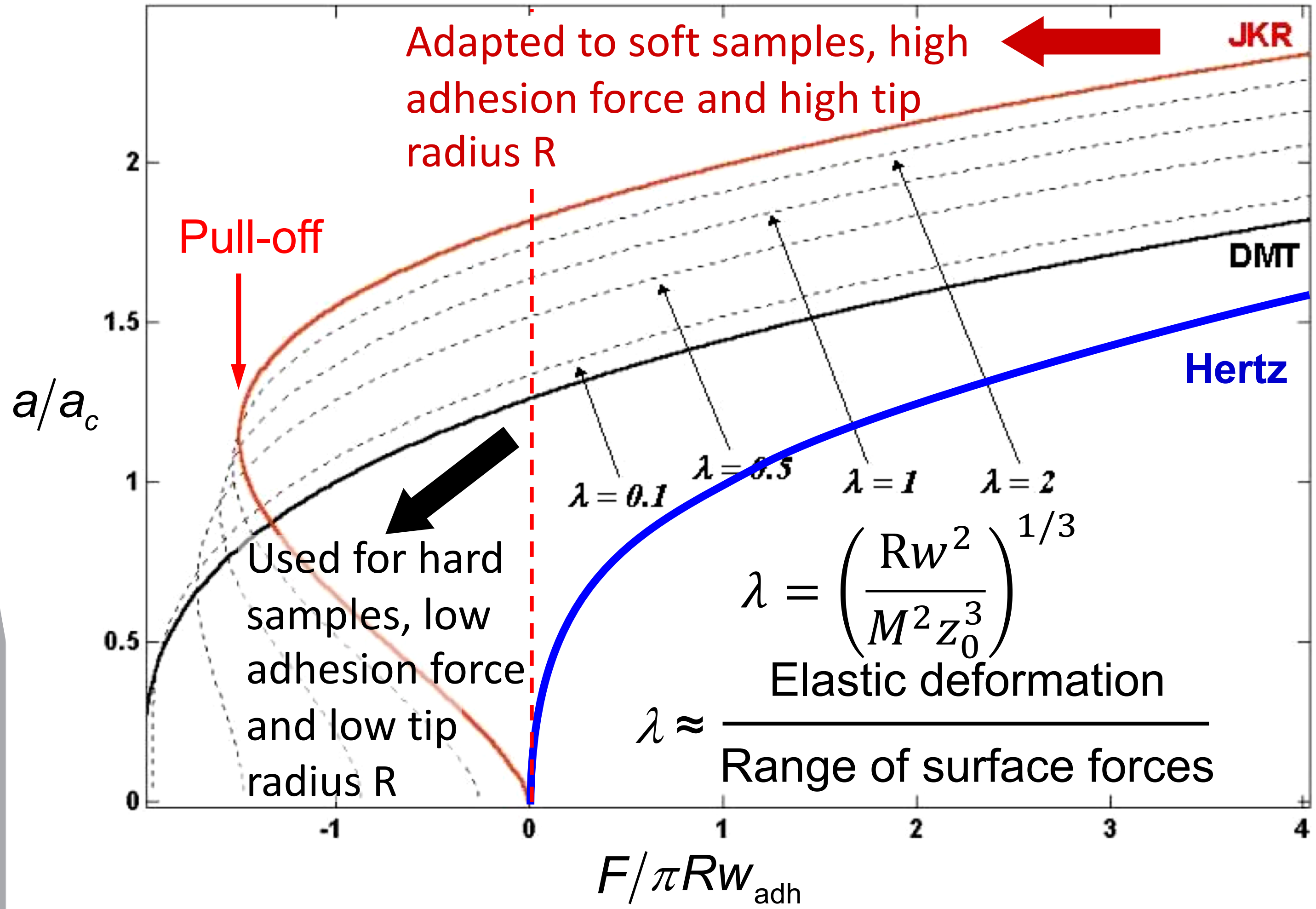
- Hertz's theory is no longer valid: no direct relation between F and δ

$$F = \frac{4}{3} E^* \frac{a^3}{R} - \sqrt{8\pi E^* w_{adh} a^3} \qquad \delta = \frac{a^2}{R} - \sqrt{\frac{2\pi w_{adh} a}{E^*}}$$

- At pull-off $a \neq 0$ and $F = -F_{adh} = -\frac{3}{2}\pi R w_{adh}$

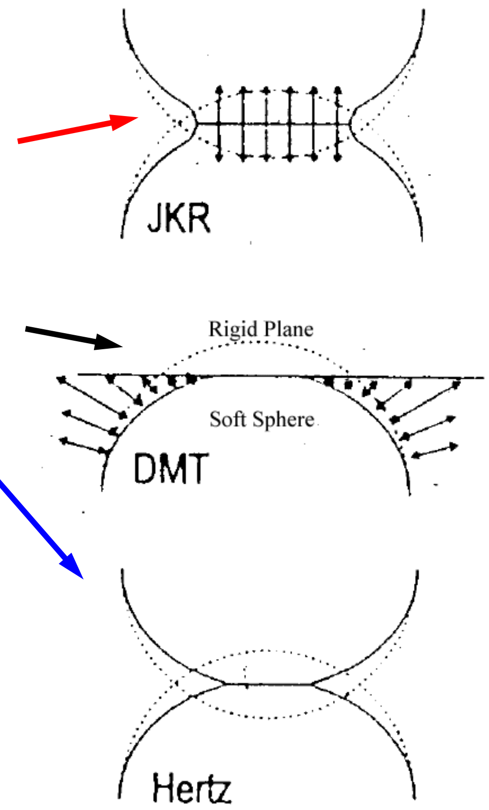
Contact mechanics

- Adhesion (~contact at the nano-scale: capillarity, van der Waals, etc.)
  - ➲ Intermediate cases: Maugis-Dugdale
    [J. Colloid Int. Sci., 1992; E. Barthel, *J. Phys. D*, 2008]



$$\lambda = \left(\frac{Rw^2}{M^2 z_0^3}\right)^{1/3}$$

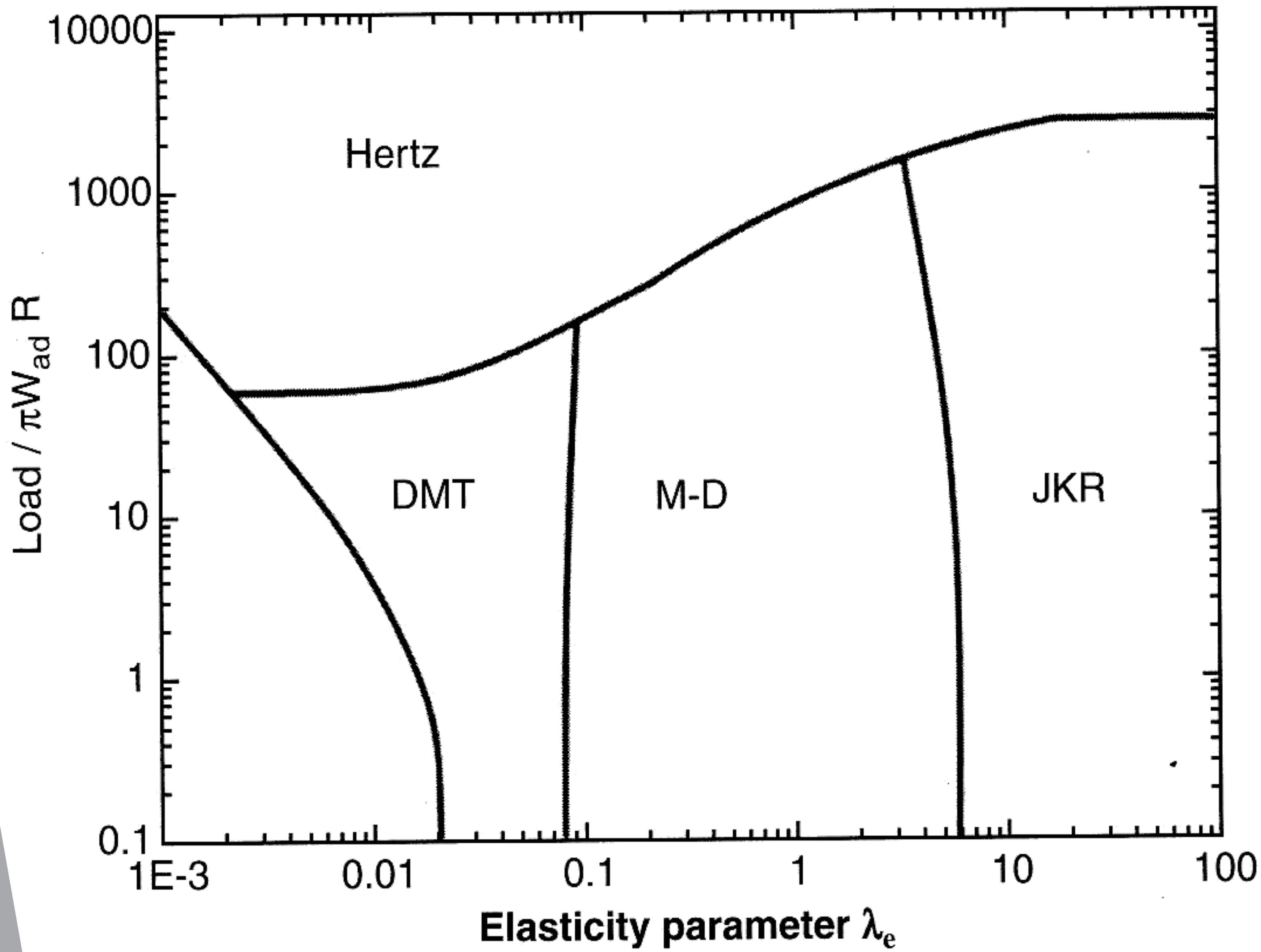
$$\lambda \approx \frac{\text{Elastic deformation}}{\text{Range of surface forces}}$$

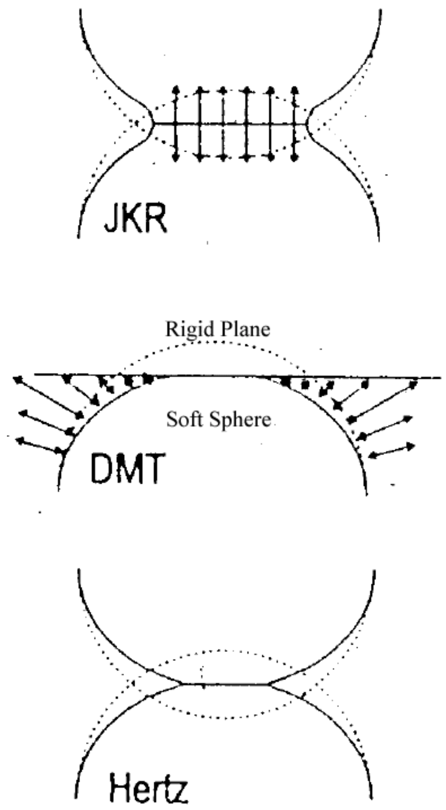


[R.M. Overney]

Contact mechanics

- Adhesion (~contact at the nano-scale: capillarity, van der Waals, etc.)





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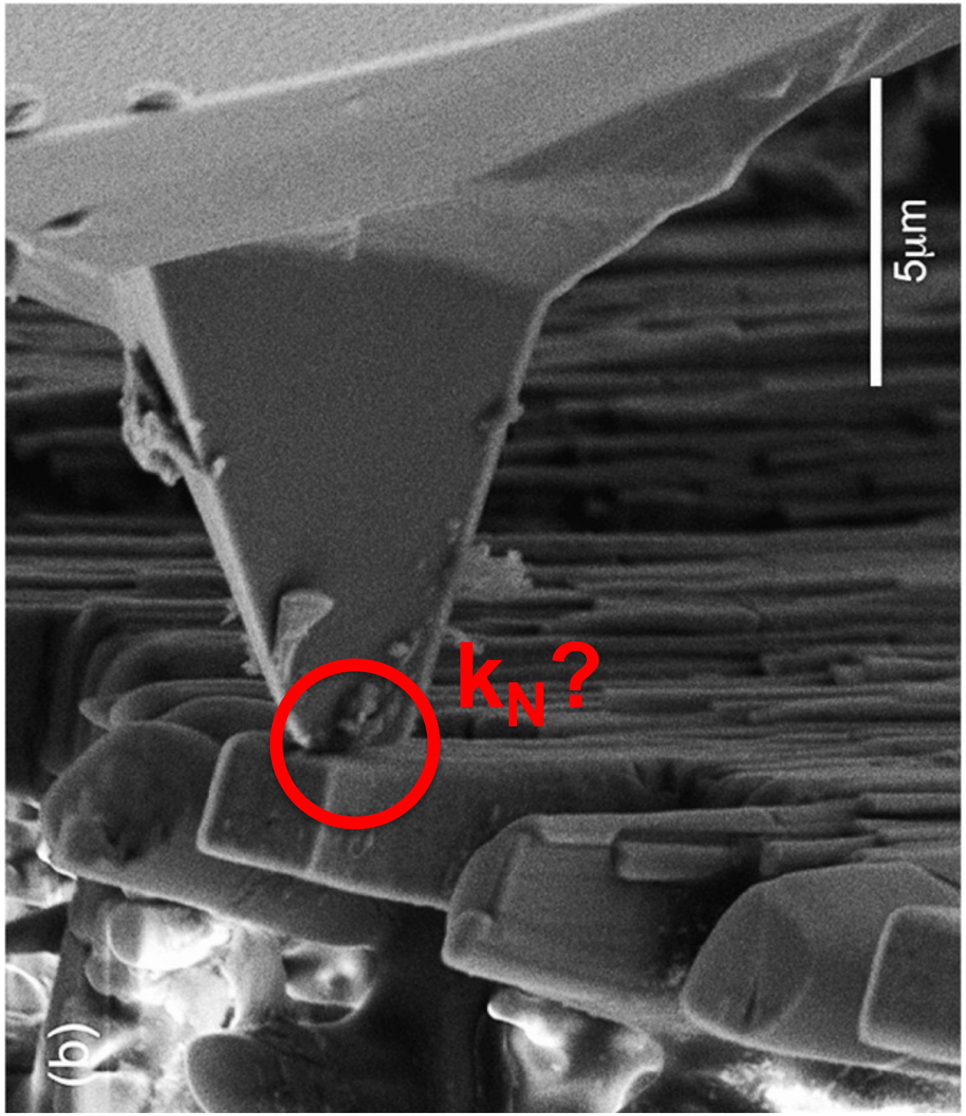

[Jimenez-Palomar *et al*., J. Mech. Beh. Bio. Mat., 2011]

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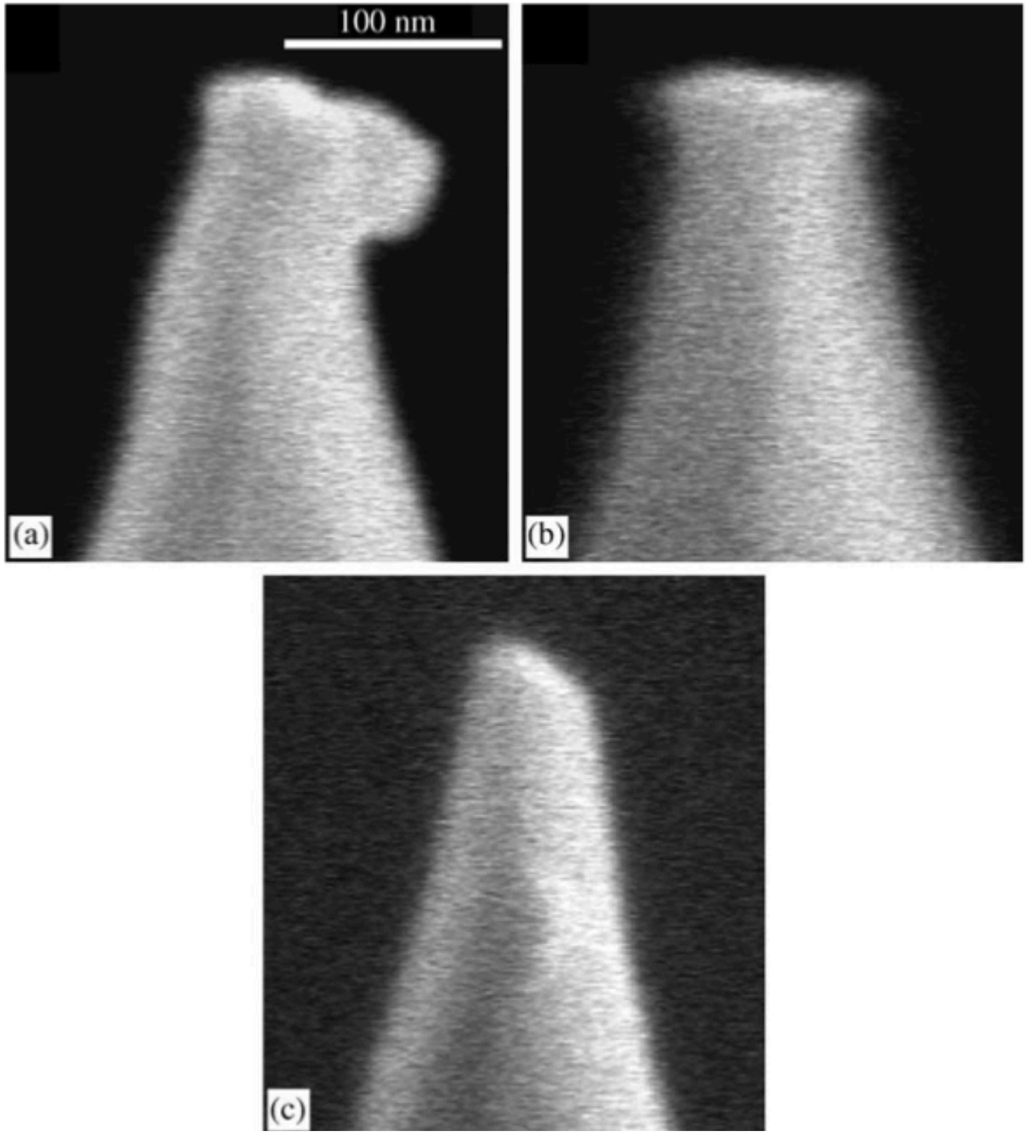
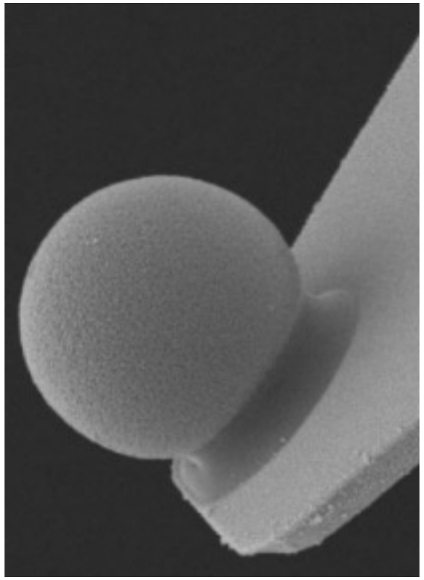
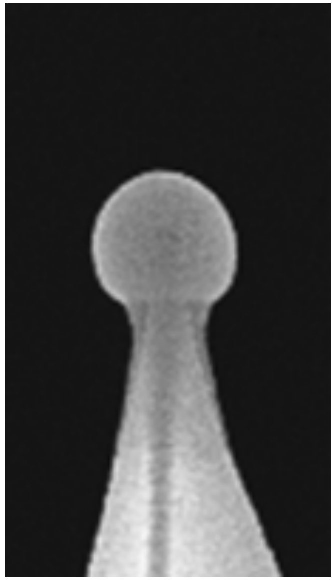
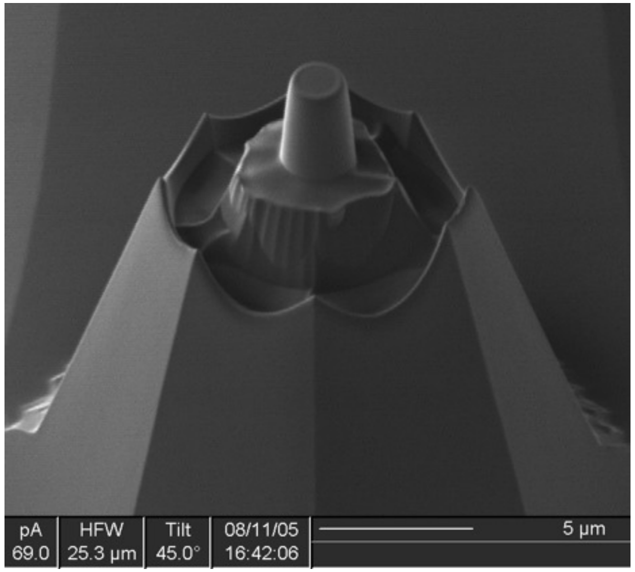
Contact mechanics

- Tip shapes





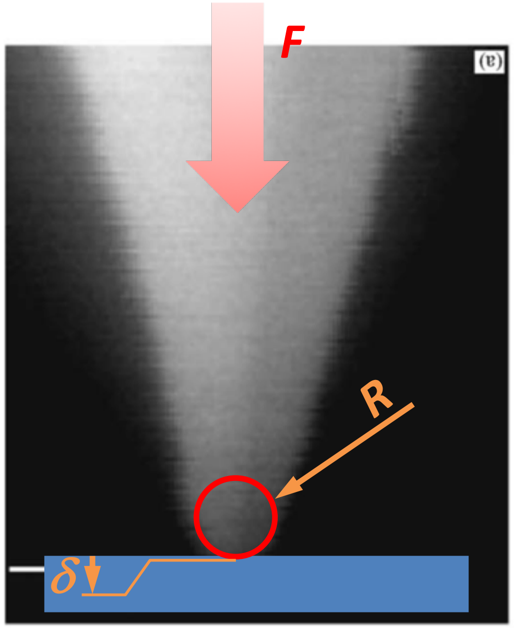


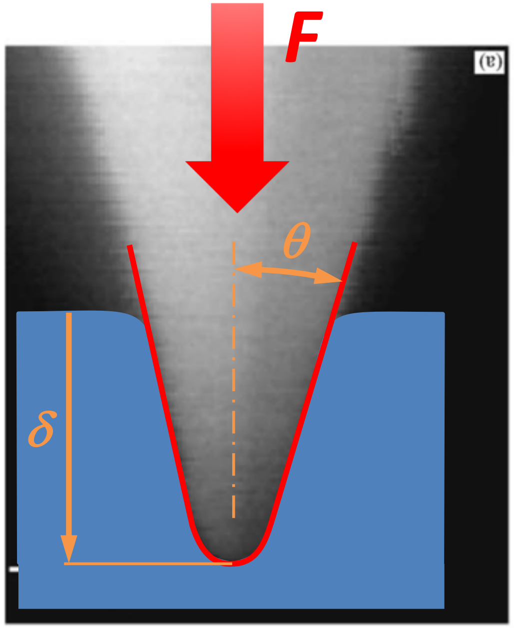


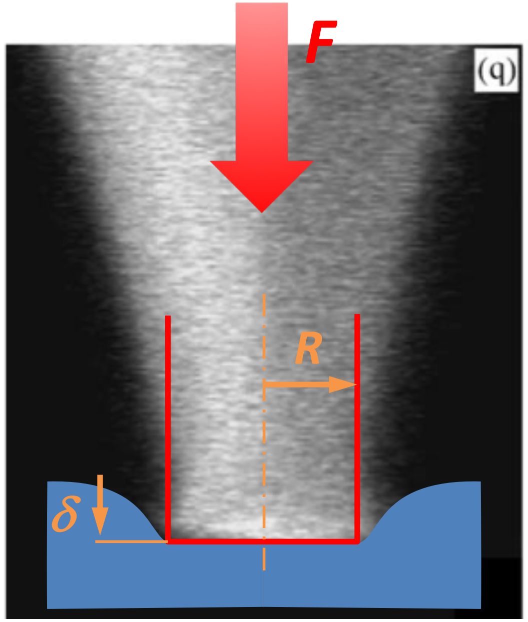
[www.nanoandmore.com]

[Kopycinska-Müller et al, Ultramicroscopy, 2006; Nanotechnology, 2016]

Contact mechanics







✓ Hertz (1885)

$$F = \frac{4}{3} M R_{eq}^{1/2}\, \delta^{3/2}$$

$$k_N = \left(6\, M^2 R_{eq} F\right)^{1/3}$$

δ << R

✓ Sneddon (1965)

$$F = \frac{2}{\pi} M \tan\theta\, \delta^2$$

$$k_N = \left(\frac{32}{\pi^3} M \tan\theta\, F\right)^{1/2}$$

δ ≈ R
[Sneddon1965]

δ >> R

✓ Flat punch (1965)

$$F = 2MR\,\delta$$

$$k_N = 2MR$$

M: Reduced/contact modulus

$$\frac{1}{M} = \frac{1-\nu_{Tip}^2}{E_{Tip}} + \frac{1-\nu_{Sample}^2}{E_{Sample}}$$

Non conforming

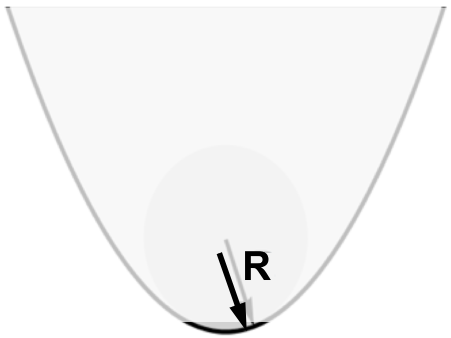
E^*_{sample} << E^*_{tip}

LINEAR ELASTIC HOMOGENEOUS and *ISOTROPIC* materials

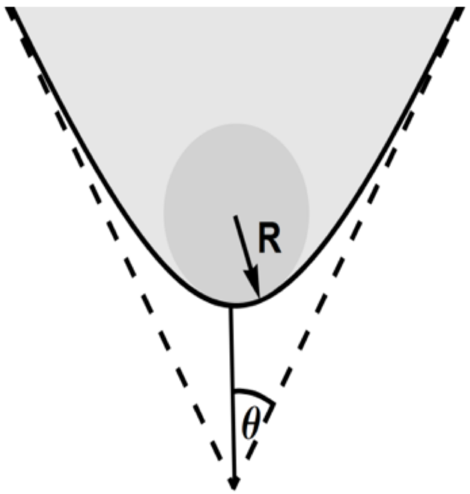
[Kopycinska-Müller et al, Ultramicroscopy, 2006]

Contact mechanics

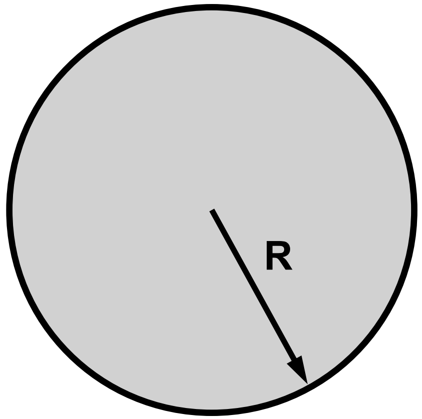
- Different cases implemented in *AtomicJ*



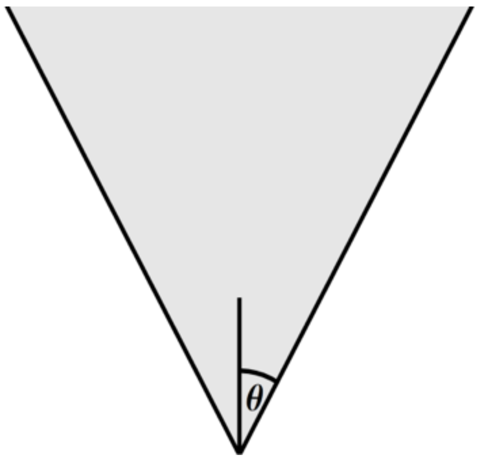
Paraboloïd (Hertz)
δ **<< R**
+ adhesion (DMT, JKR)



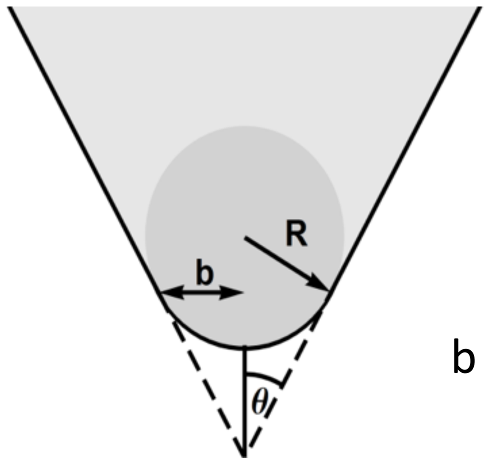
Hyperboloïd **+ adhesion**



Exact sphere (Sneddon) **+ adhesion (JKR)**

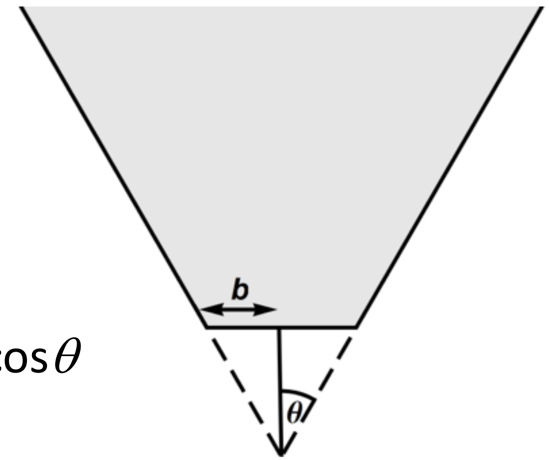


Cone (Sneddon)



Blunt cone
(**Hertz -** δ **< b²/R !** +cone)

b = R cosθ



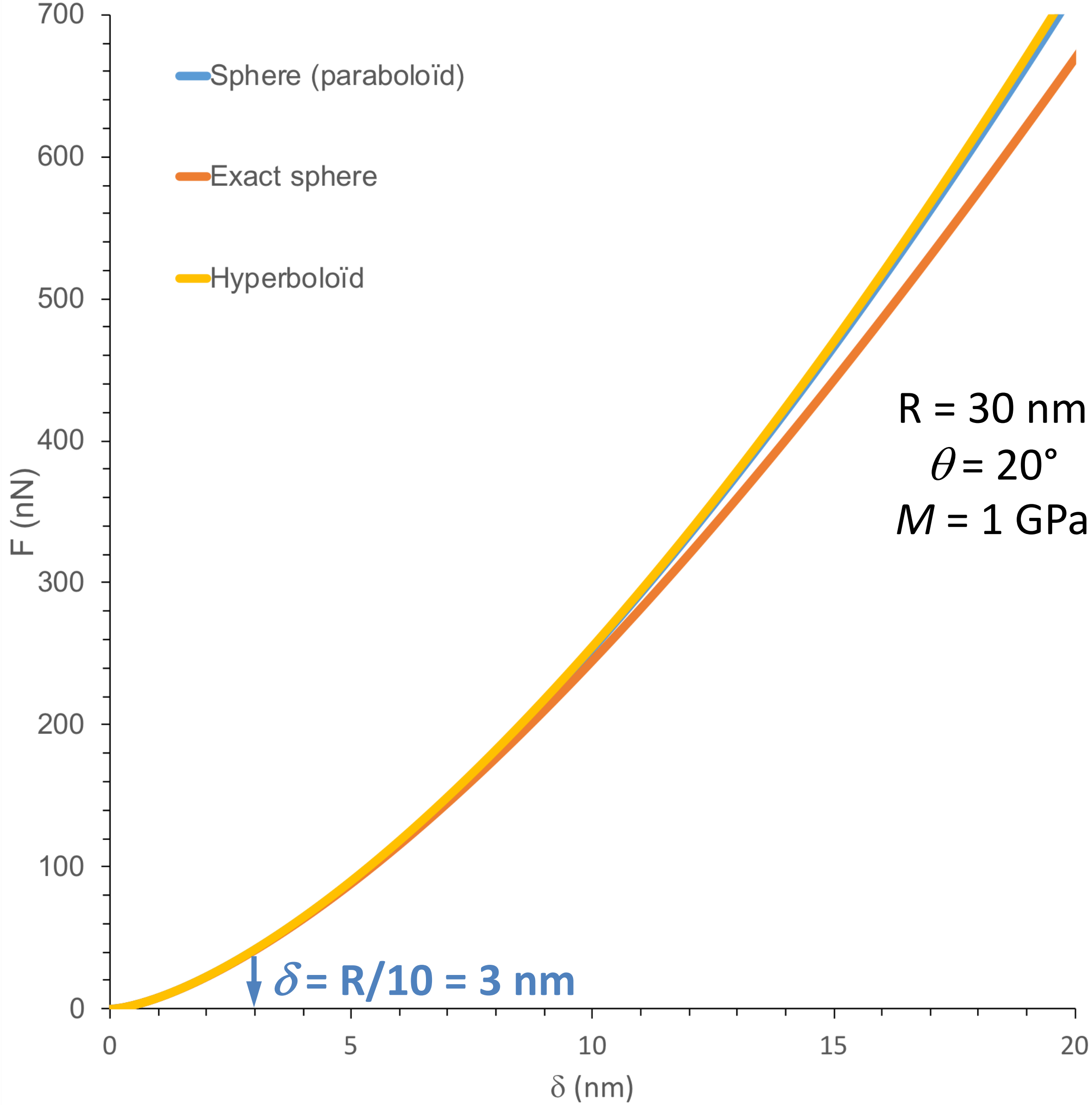
Truncated cone
(≈flat punch)

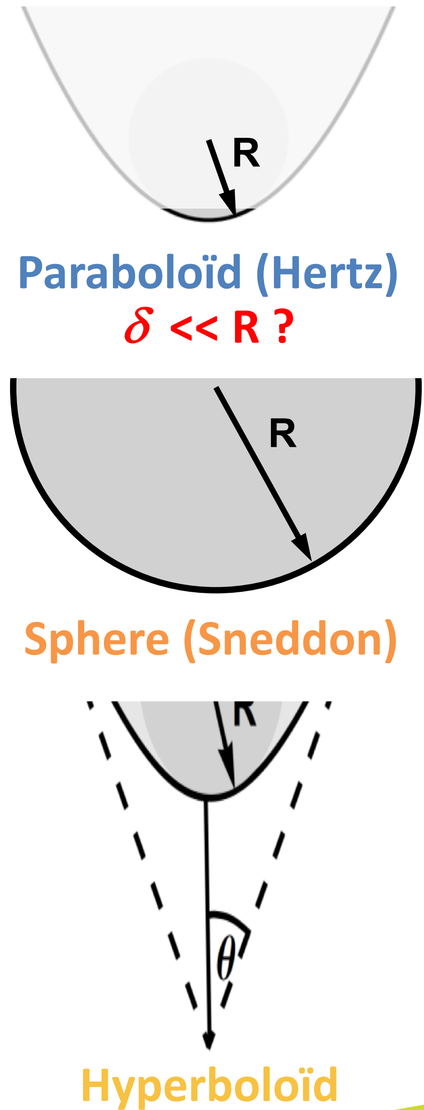
Suppl. Mat. [Hermanowicz et al, Rev. Sci. Inst., 2014]

and many others (hyperelastic, thin, pyramid)…

Contact mechanics

- Different cases implemented in *AtomicJ*





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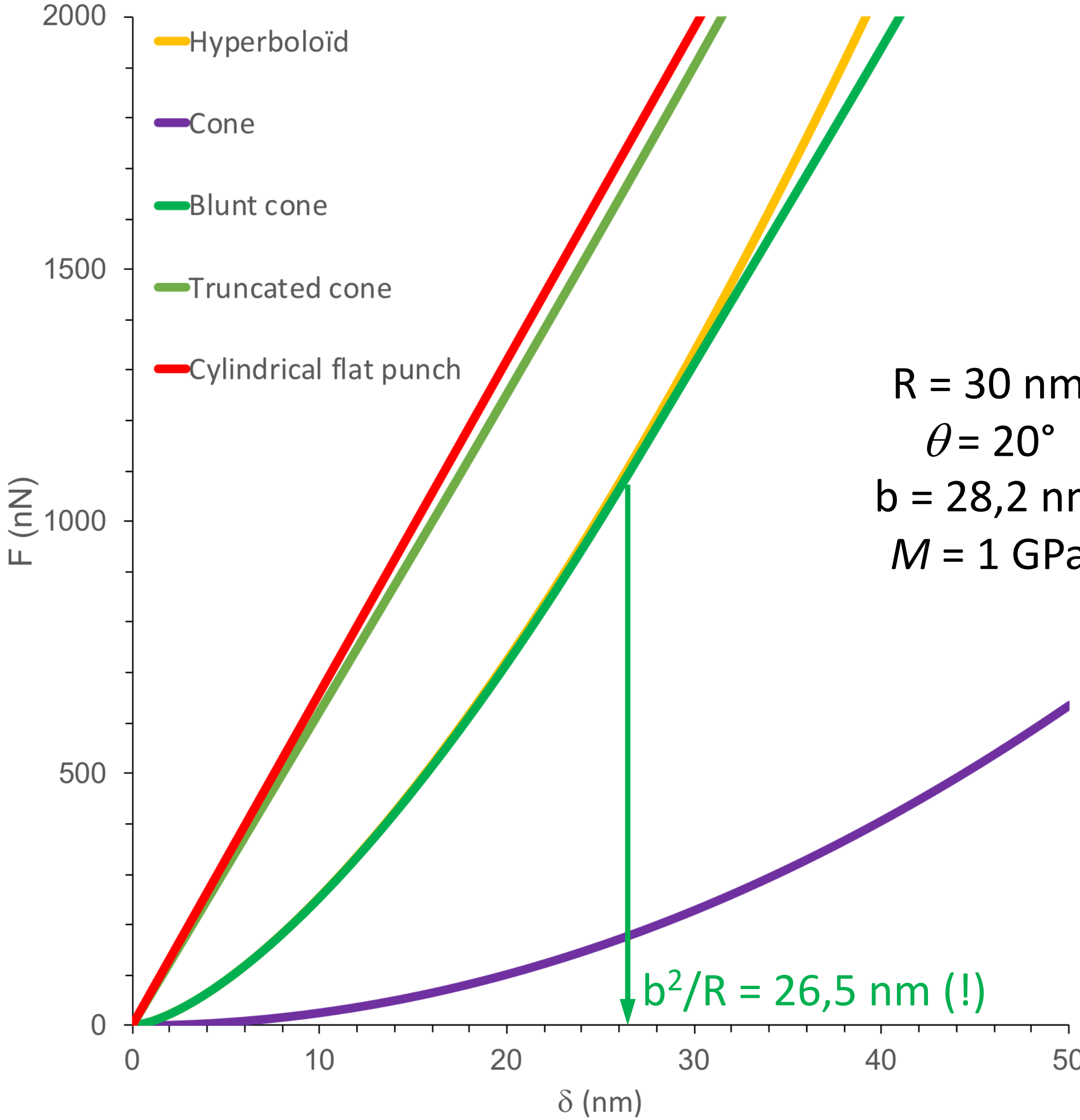

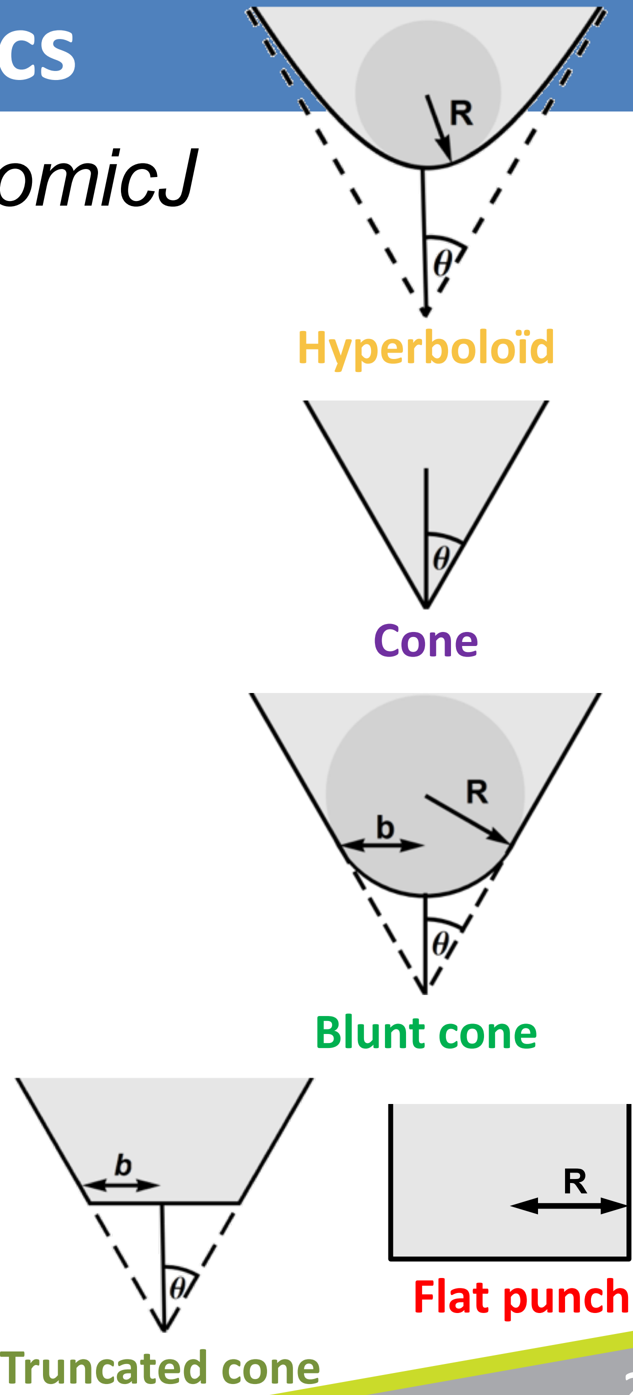

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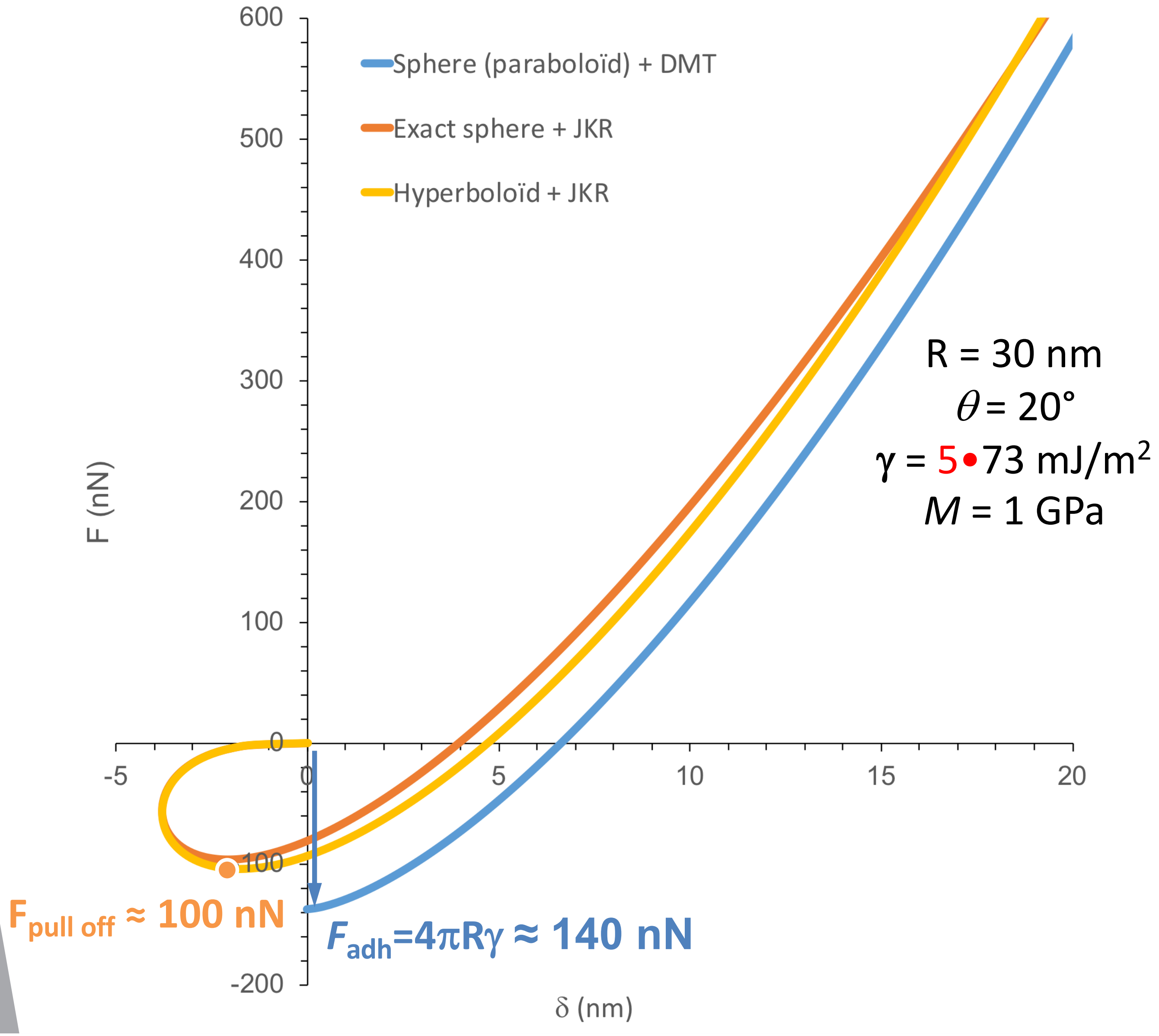

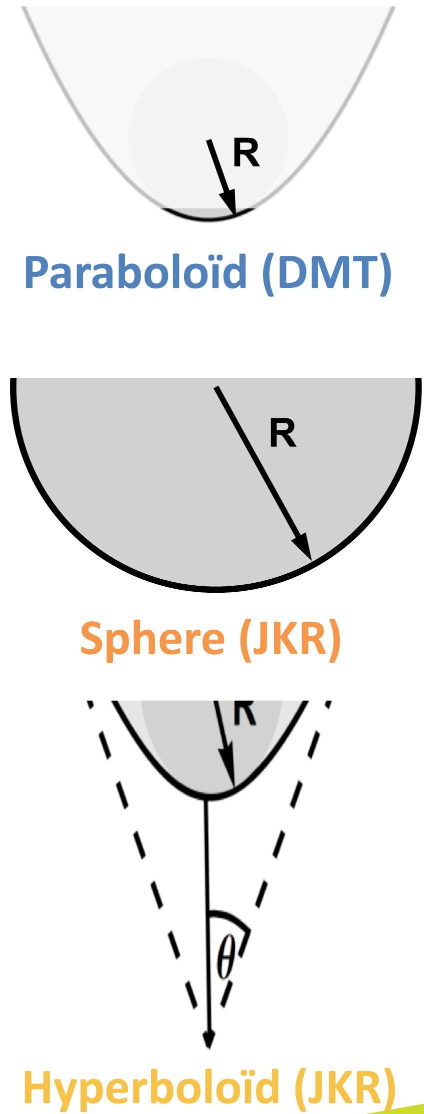

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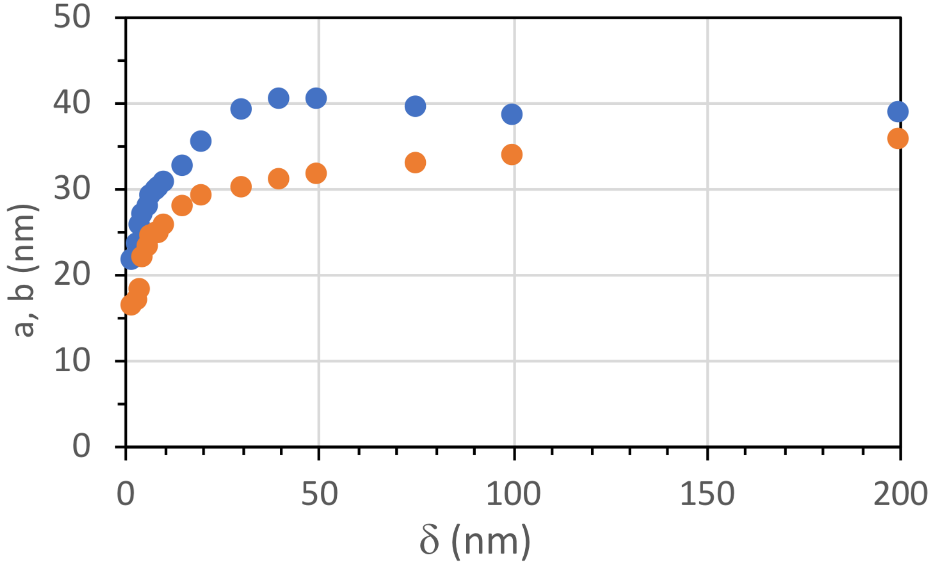
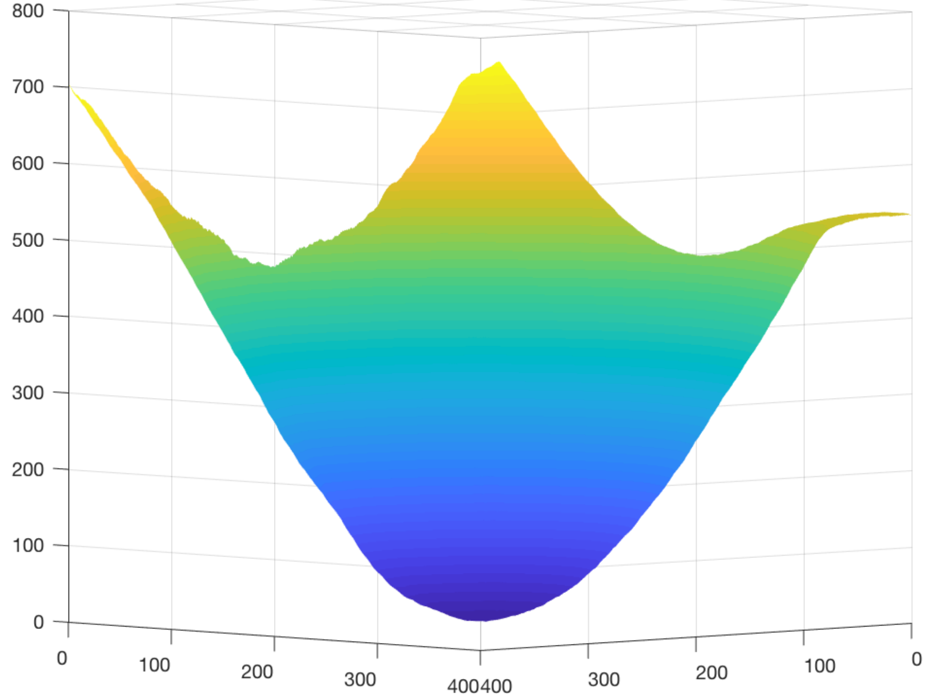
Contact mechanics

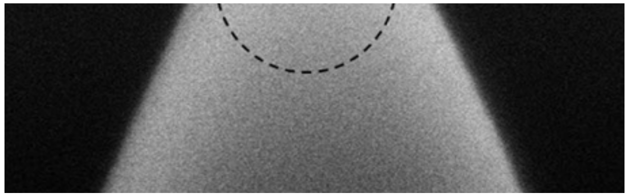
- Examples of tip shape

Bruker RTESPA 525(-30)

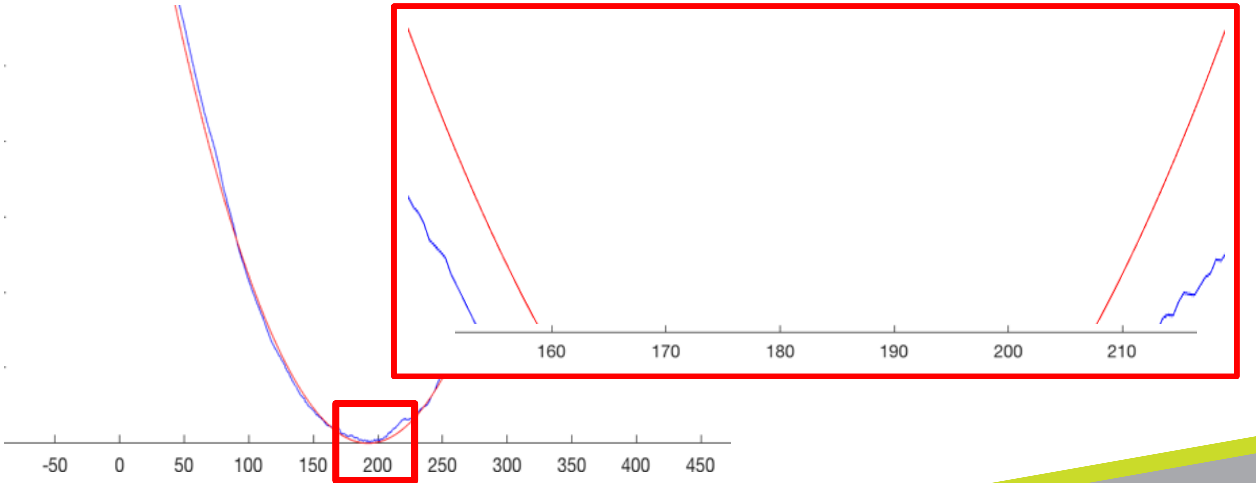


Tip shape (nm) measured using TGT (NT-MDT)





Geometry:	Rotated (Symmetric)
Tip Height (h):	10 - 15μm
Front Angle (FA):	15 ± 2º
Back Angle (BA):	25 ± 2 º
Side Angle (SA):	17.5 ± 2 º
Tip Radius (Nom):	30 nm
Tip Radius (Max):	36 nm



Adjustment with a paraboloïd

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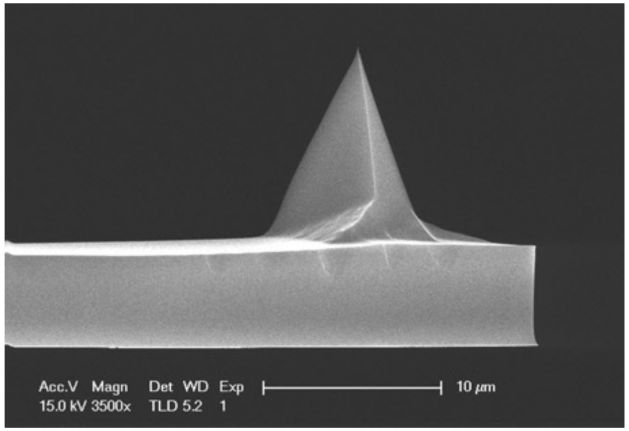

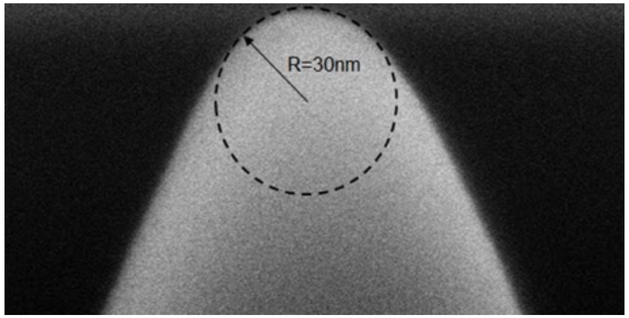

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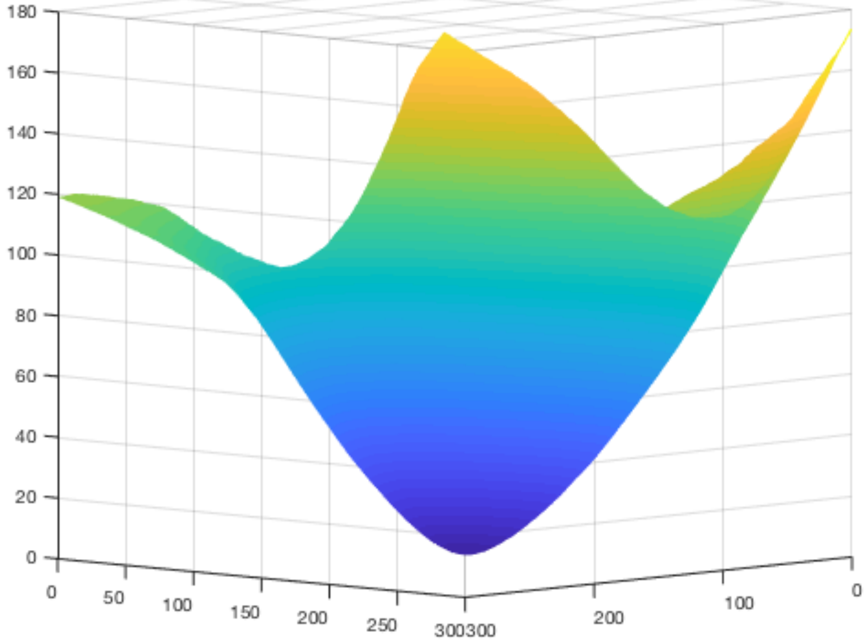

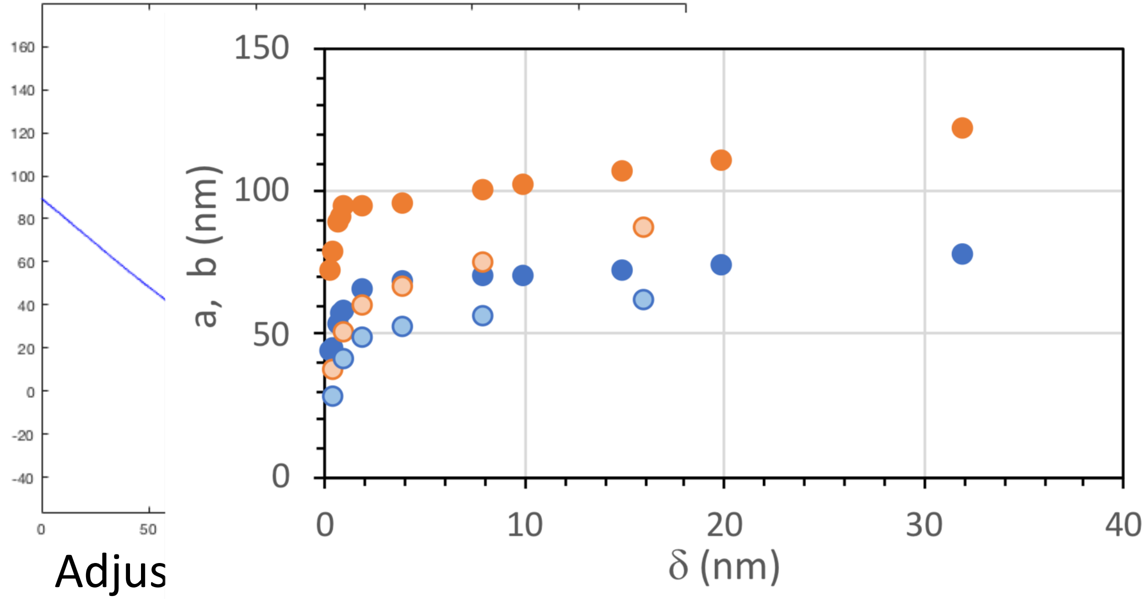

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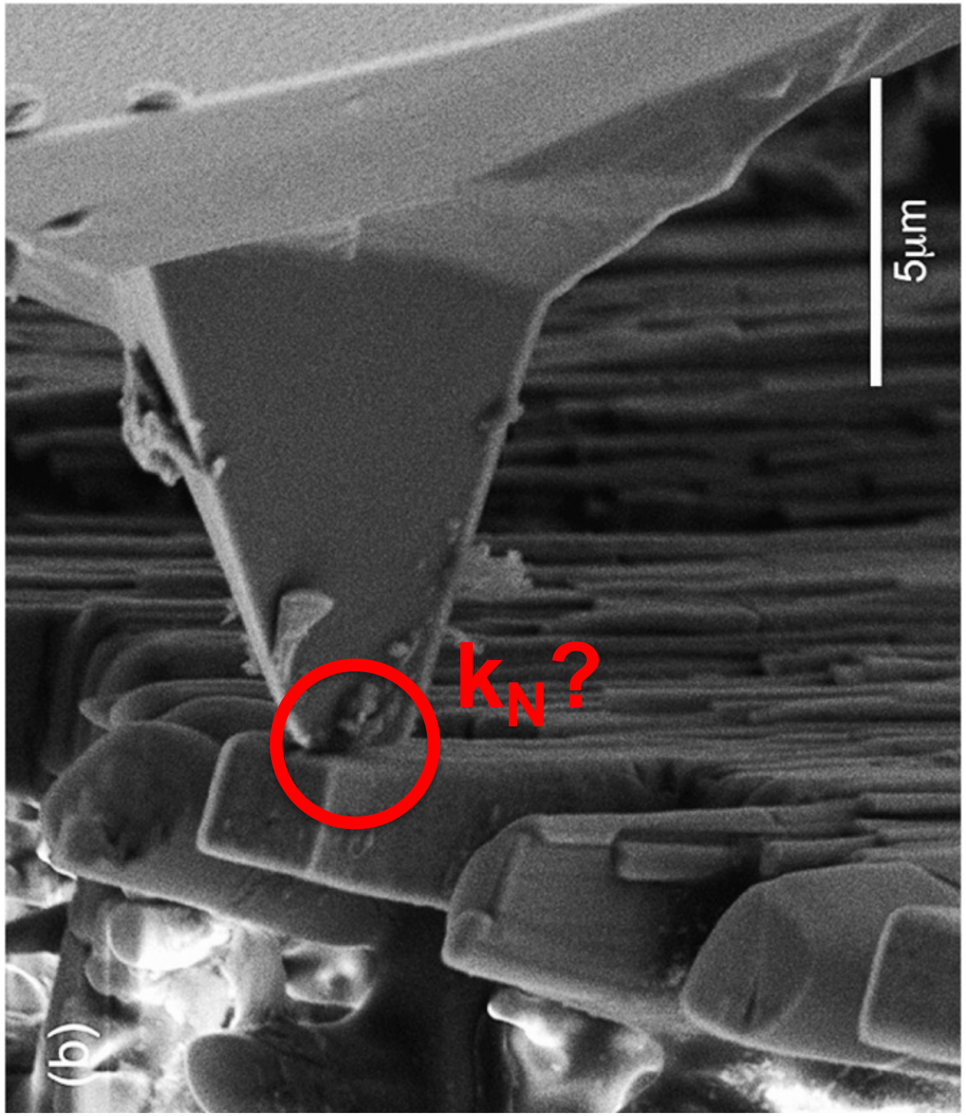
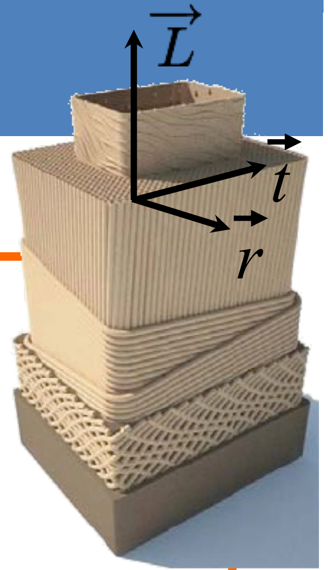
Bone micromechanics using in situ AFM in SEM



- Théorie de Hertz
- Adhésion (DMT-JKR)
- Autres cas et comparatif
- Anisotropie et viscosité
- Plasticité > NanoIndentation

[Jimenez-Palomar *et al*., J. Mech. Beh. Bio. Mat., 2011]

Contact mechanics



- Anisotropy

For a typical S2-layer with MFA~0° [Jäger et al, 2011]

$M_L \approx 19$ GPa whereas $E_L \approx 45$ GPa

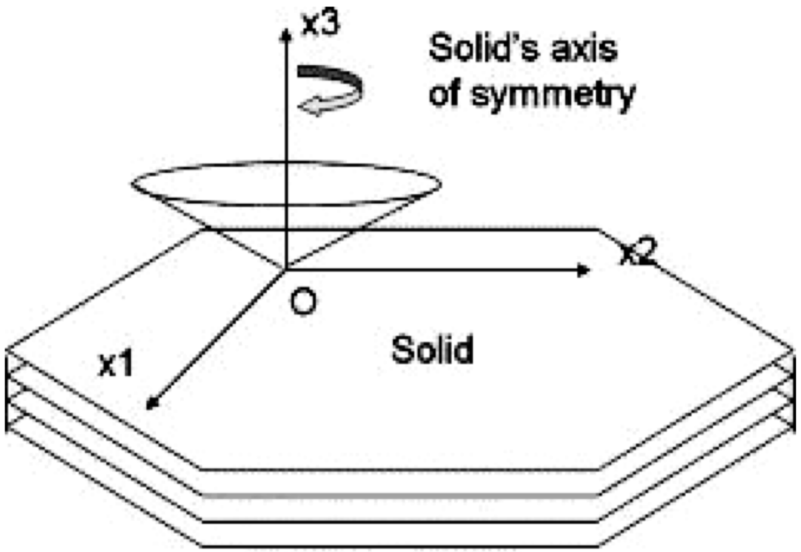
($E_t \approx E_r \approx 12$ GPa, $\nu_{tL} \approx \nu_{rL} \approx 0.028$, $\nu_{rt} \approx 0.28$
$G_{tL} \approx G_{rL} \approx 2.5$ GPa, $G_{rt} \approx 2$ GPa)

Kevlar fibre: M// ≈ 15-20 / E// ≈ 80 GPa [Arnould et al, 2017]

in particular case…
→ transversely isotropic // contact surface
[Hanson, J. Appl. Mech., 1992]

$$M_3 = 2\sqrt{\frac{C_{11}C_{33}-C_{13}^2}{C_{11}}\left(\frac{1}{C_{44}}+\frac{2}{\sqrt{C_{11}C_{33}}+C_{13}}\right)^{-1}}$$

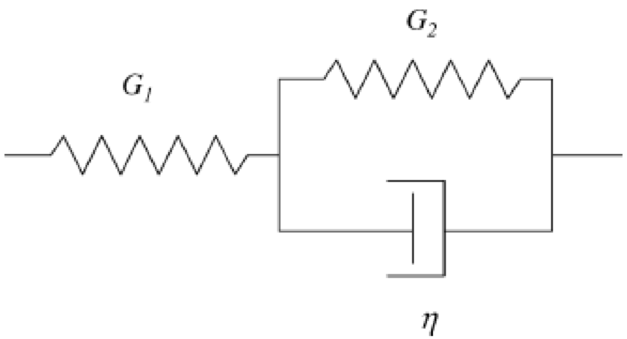
1=2: C_{22} C_{23} $G_{13}=G_{23}$



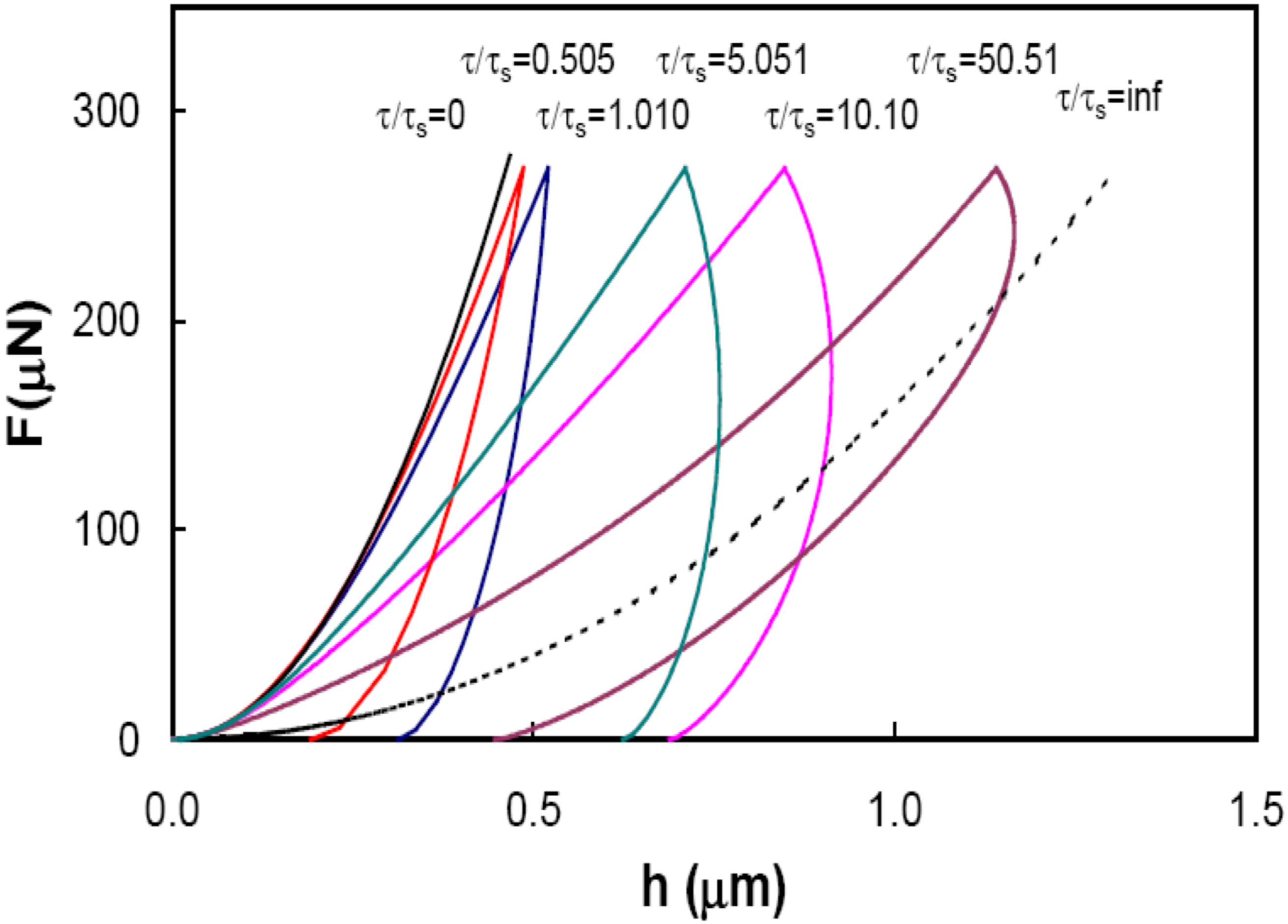
[Delafargue *et al*., Int. J. Sol. Struct., 2004]

k_T = ? > *work in progress*

- Viscoelasticity
  - ➲ Loading-unloading curve of a pure viscoelastic material [Cheng and Cheng, *Mat. Sci. Eng. R*, 2004]



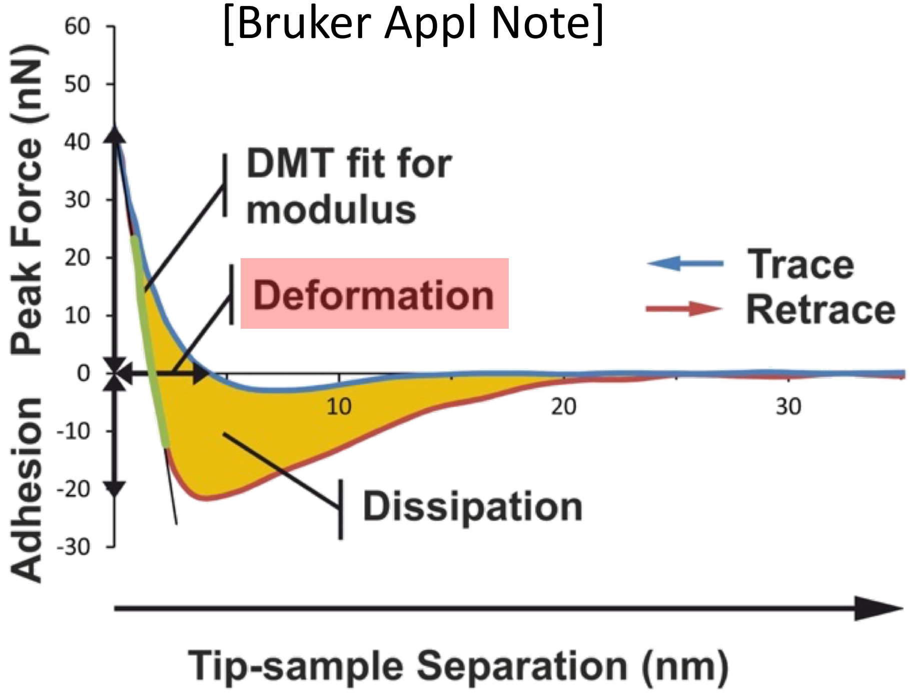
G_1 = 234 MPa G_2 = 26 MPa
τ_s = 1 s
K = 688 MPa



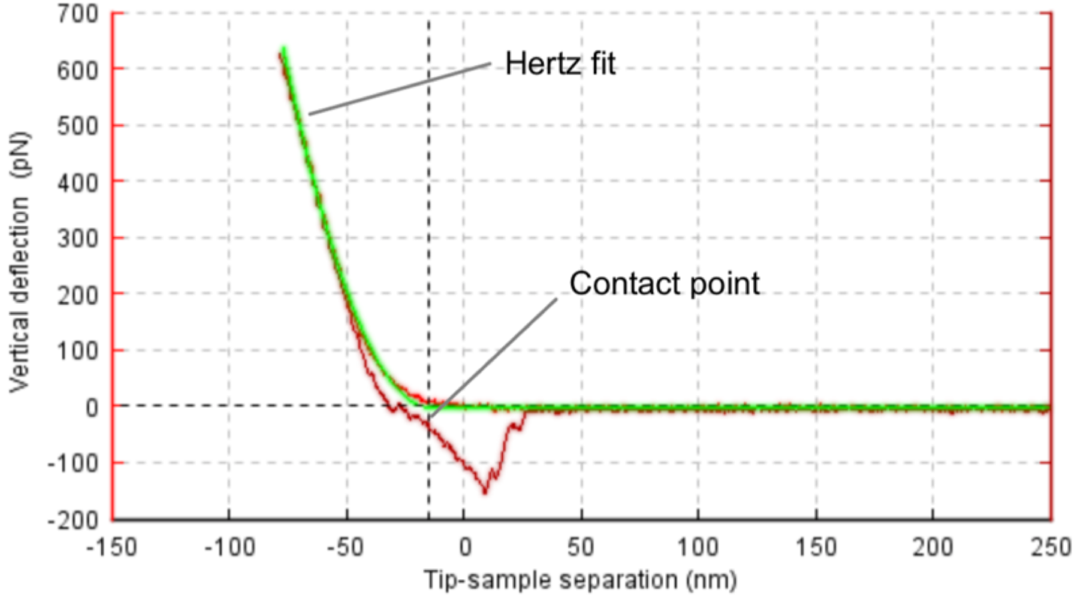
Contact mechanics

- Peak Force QNM (Bruker) vs. QI (JPK)

[Bruker Appl Note]



[JPK Appl Note]



"Typically unloading curves are preferably taken for analysis to avoid the plastic deformation that is present during loading. [...] because of a compliance of the cantilever, [...] leads to a more stable contact during unloading."

[Cohen et al, Beilstein J Nanotech, 2013]

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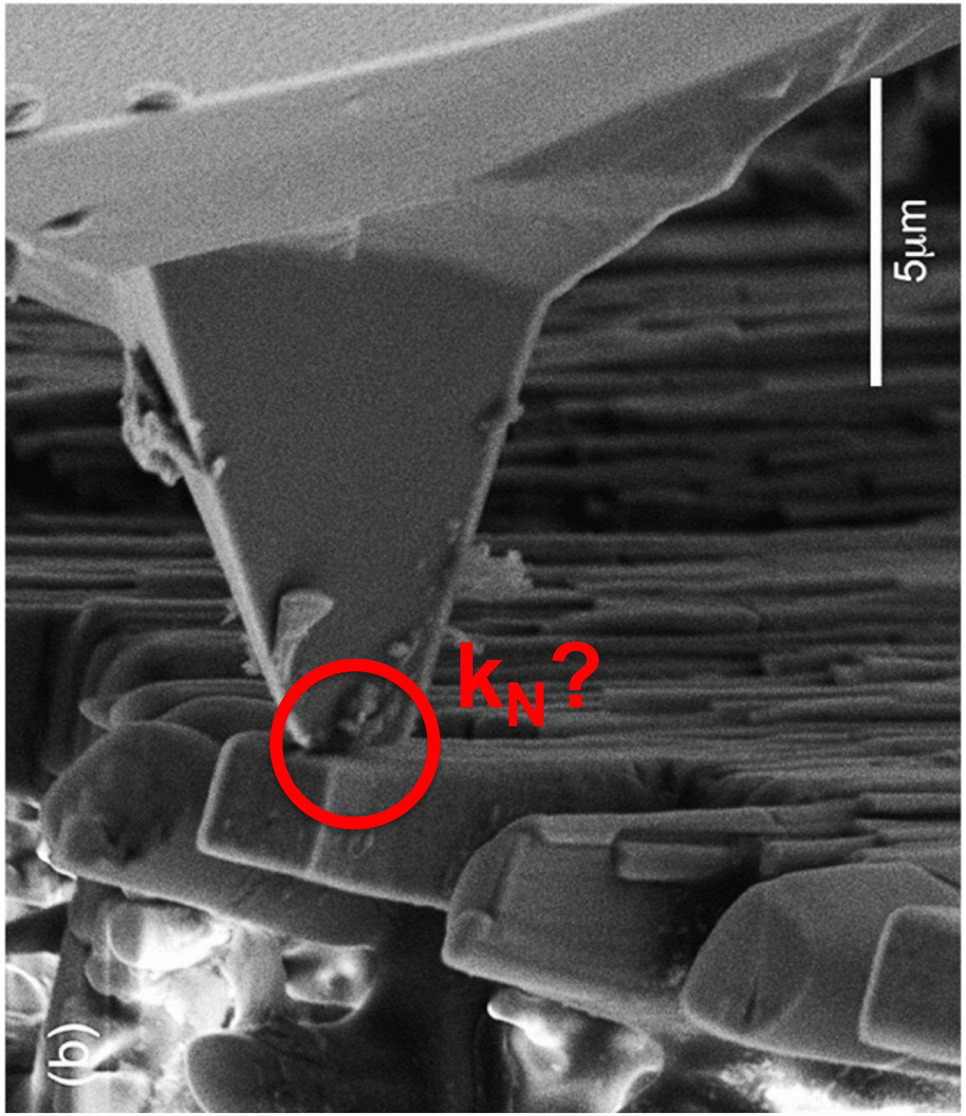

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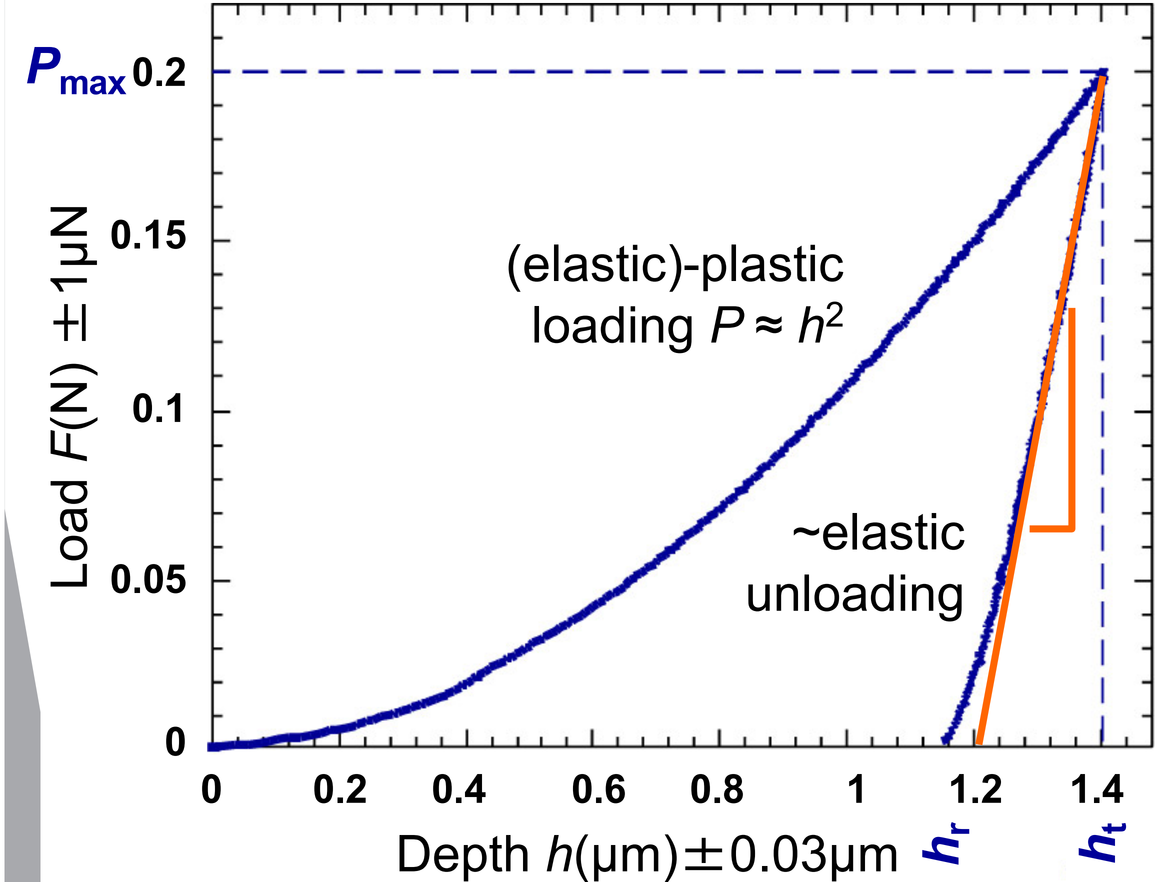
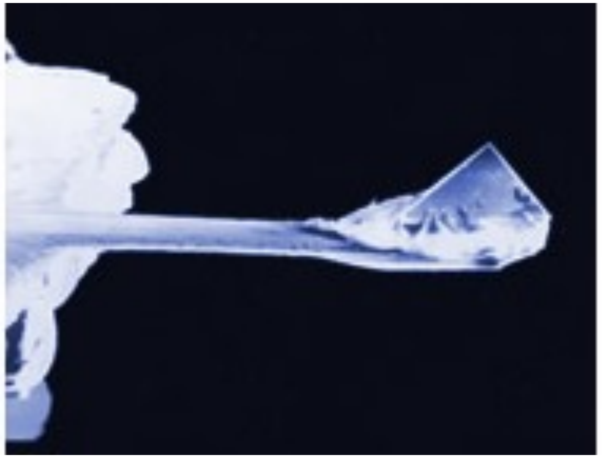
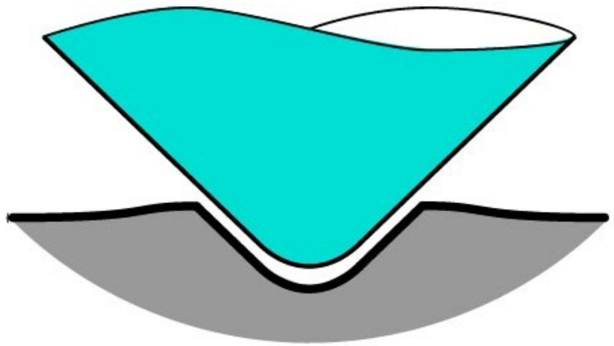
- Plasticity : NanoIndentation
  - ➲ The depth of penetration h (δ!) is measured during load application mainly with a Berkovich indenter (3-sided pyramid)



Nanoindentation curve on electrodeposited Ni





[www.brukerafmprobes.com]

Contact mechanics

- Plasticity : NanoIndentation

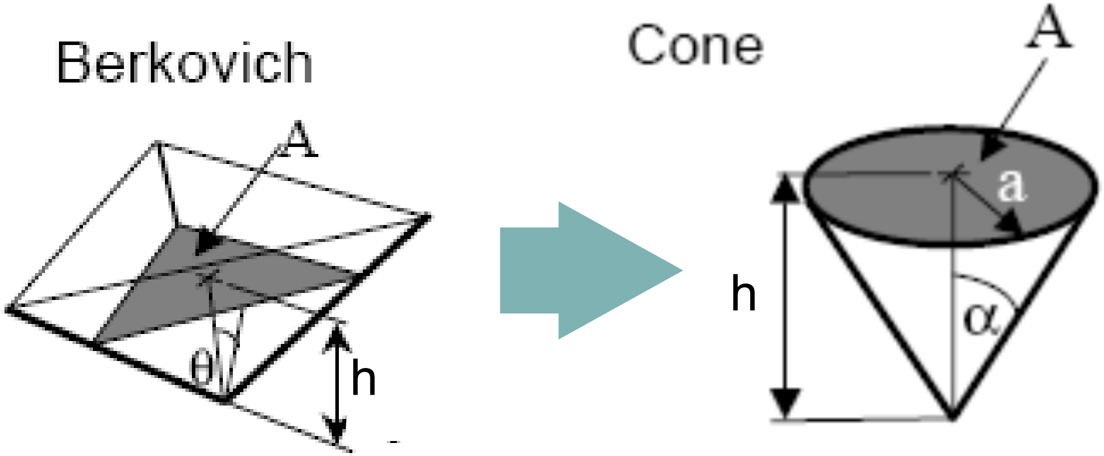
$$\boxed{E^* = \frac{\sqrt{\pi}}{2}\frac{dF}{dh}\frac{1}{\sqrt{A_c}}}$$

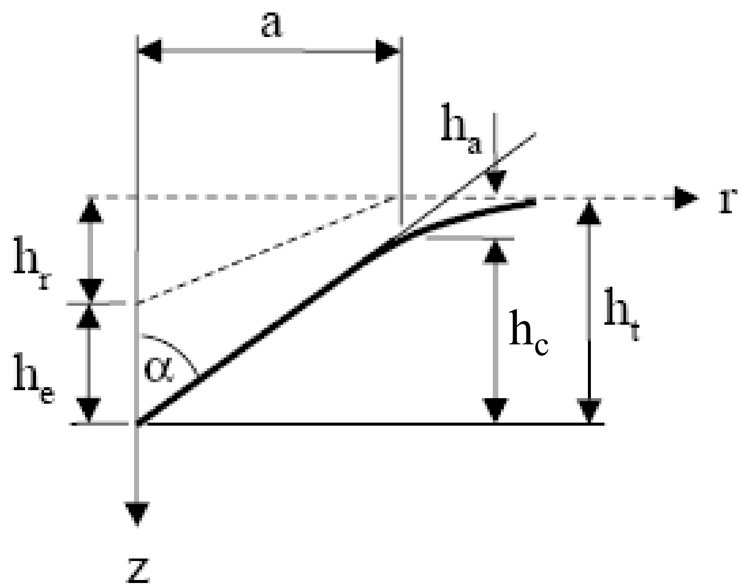
With E* or M reduced/contact/indentation modulus

$$\frac{1}{E^*} = \frac{1-\nu_i^2}{E_i} + \frac{1-\nu^2}{E}$$ for linear ***ISOTROPIC*** material

- Case of the Berkovich indenter

[A.C. Fischer-Cripps, *Vacuum*, 2000]



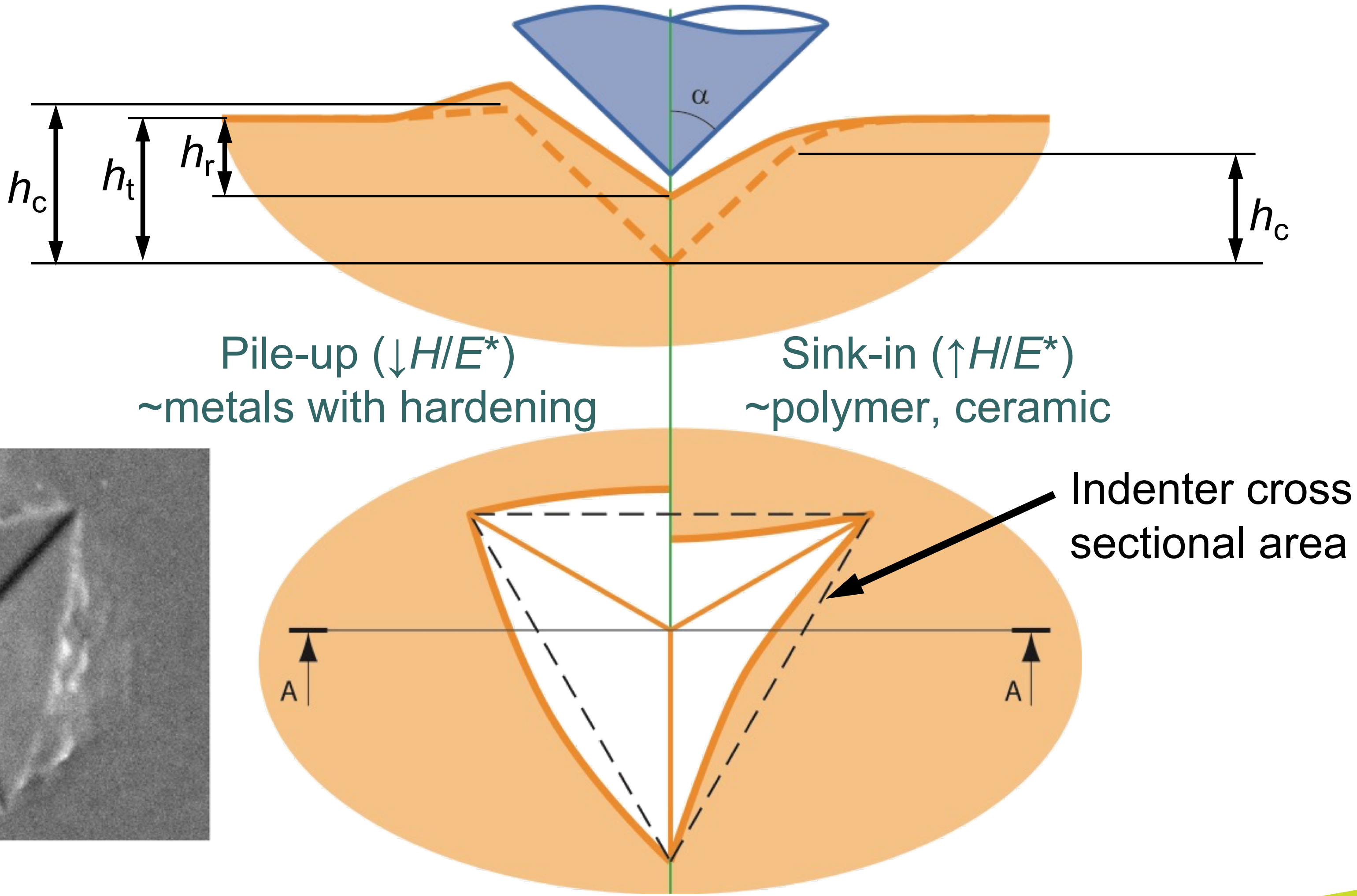


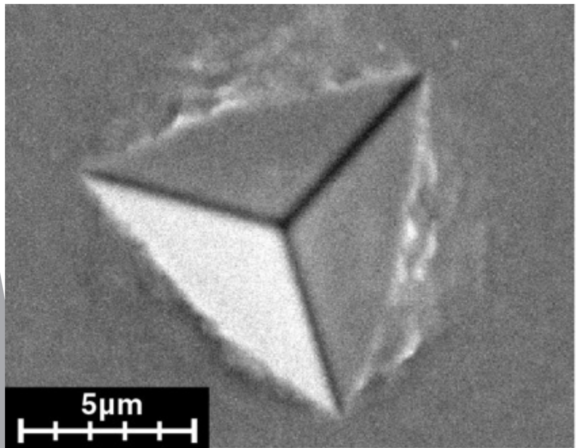
$A = \boxed{24.5} h^2$ $\qquad \alpha = 70.3°$ $\qquad A = \boxed{\pi \tan^2 \alpha} h^2$

$$h_c = h_t - \varepsilon P \left(\frac{dP}{dh}\right)^{-1} \quad \text{with } \varepsilon = \begin{cases} \left(\dfrac{2(\pi-2)}{\pi}\right) & \text{cone} \\ 0.75 & \text{Berkovich} \end{cases}$$

Contact mechanics

- Plasticity : NanoIndentation





Conclusions – Questions ouvertes

- Respecter/vérifier les domaines de validité des modèles de contact suivant les conditions expérimentales (profondeur indentation, adhesion, rayon de pointe, rigidité du matériau testé, …)
- Choix, et mesure de la forme, de la pointe + dimension optimale de la pointe / rugosité ou hétérogénéité de la surface (+ tenue à l'usure)
- Effet de la topographie et de la profondeur de mesure par rapport aux effets de surface (liés à la préparation des échantillons + comportement matériaux)
- Calibration des échantillons de référence en terme d'échelle, de fréquence (TTSP ?) et de mode de sollicitation ? Idem pour le matériau mesuré…
- Attention au choix de la raideur (et facteur de qualité) du levier / matériau à tester
- Attention au comportement anisotrope ($E \neq M$) surtout aux échelles nano
- Indentation non normale… raideur tangentiel en anisotrope ? Comment la mesurer ?
- Utilisation de la courbe d'approche ou de retrait ou … ? Et effet de la viscosité ?
- Problème de la vitesse de sollicitation / comportement viscoélastique