Mécanique aux petites échelles

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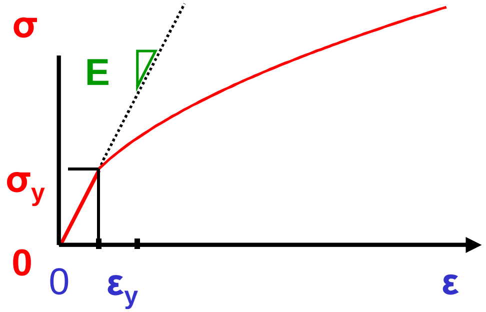
L [µm - nm] ⟺

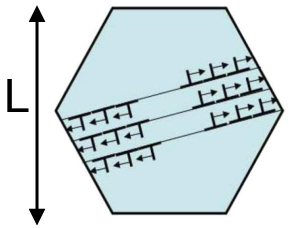


$$d\varepsilon/dt = F(\sigma, T, \mathbf{S})$$

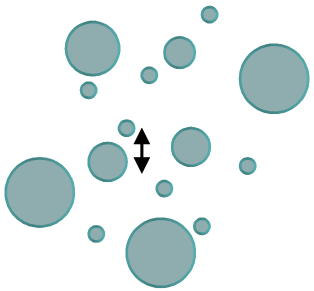
$$E, \sigma_y, n$$

Microstructure:

Distance entre interfaces

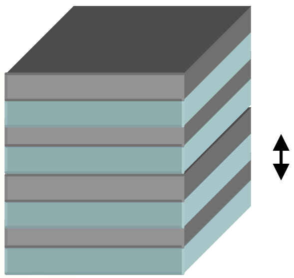


Taille de grains, dislocations



Pores / précipités

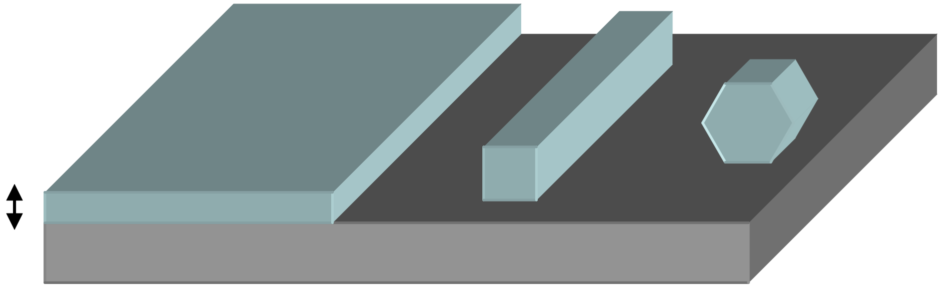
Multicouches



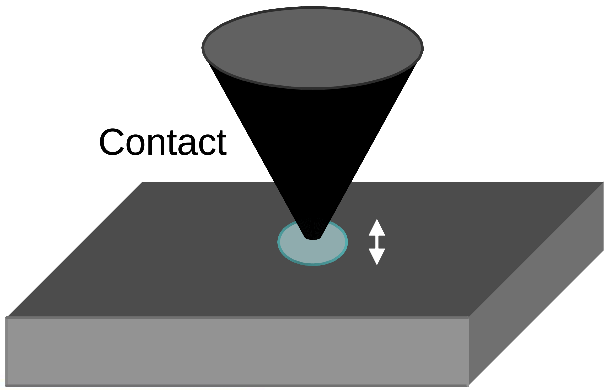
Dimensions:

Taille de l'objet / sollicitation

Film, fil, plot

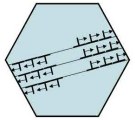


Contact



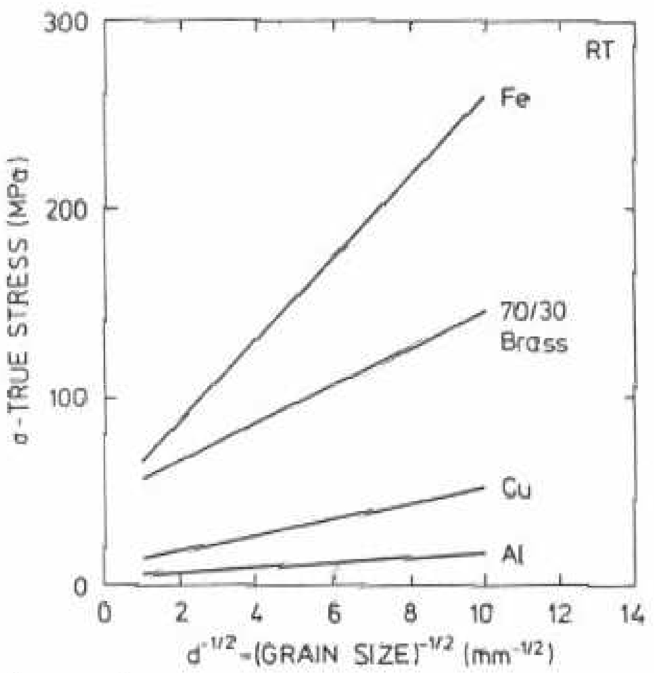
Microstructure:

Grain size

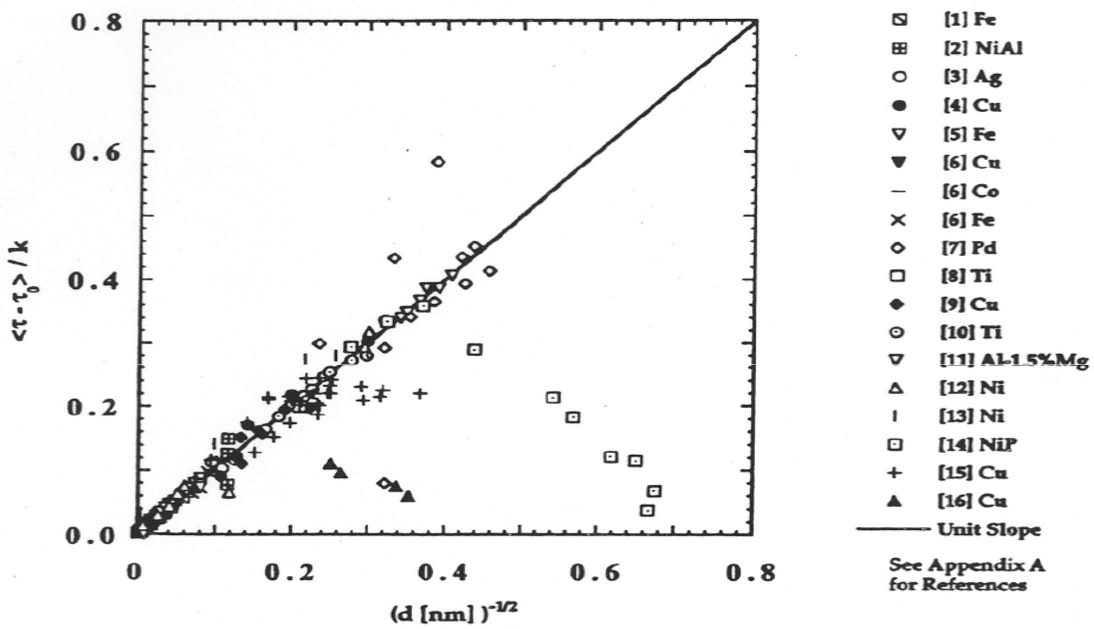


Motivation: strengthening

What about submicron scale ?



[N.Hansen, *Met. Trans* 1985]

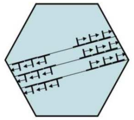


$$\sigma_y = \sigma_o + \frac{K_{HP}}{\sqrt{d}}$$

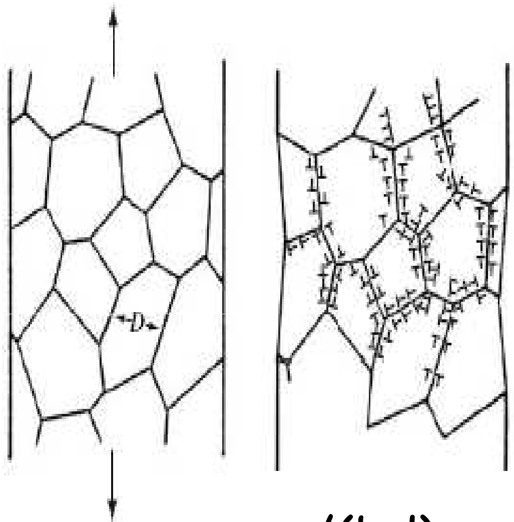
[Hall *Proc.Roy.Soc.* 1951; Petch *Jal Iron Steel* 1953]

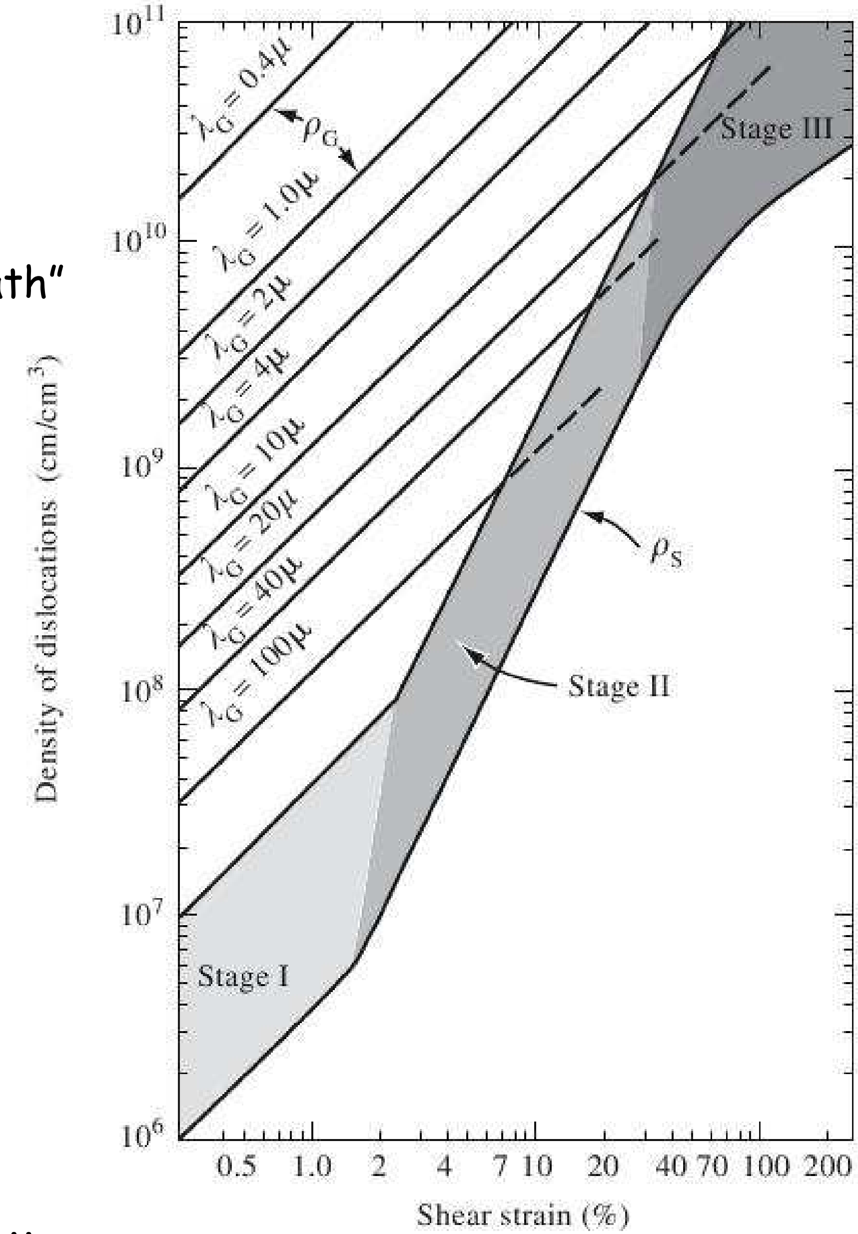
Microstructure:

Grain size



λ "mean free path"





$\rho_{GND} \propto \varepsilon/(bd)$

[Ashby. 1971]

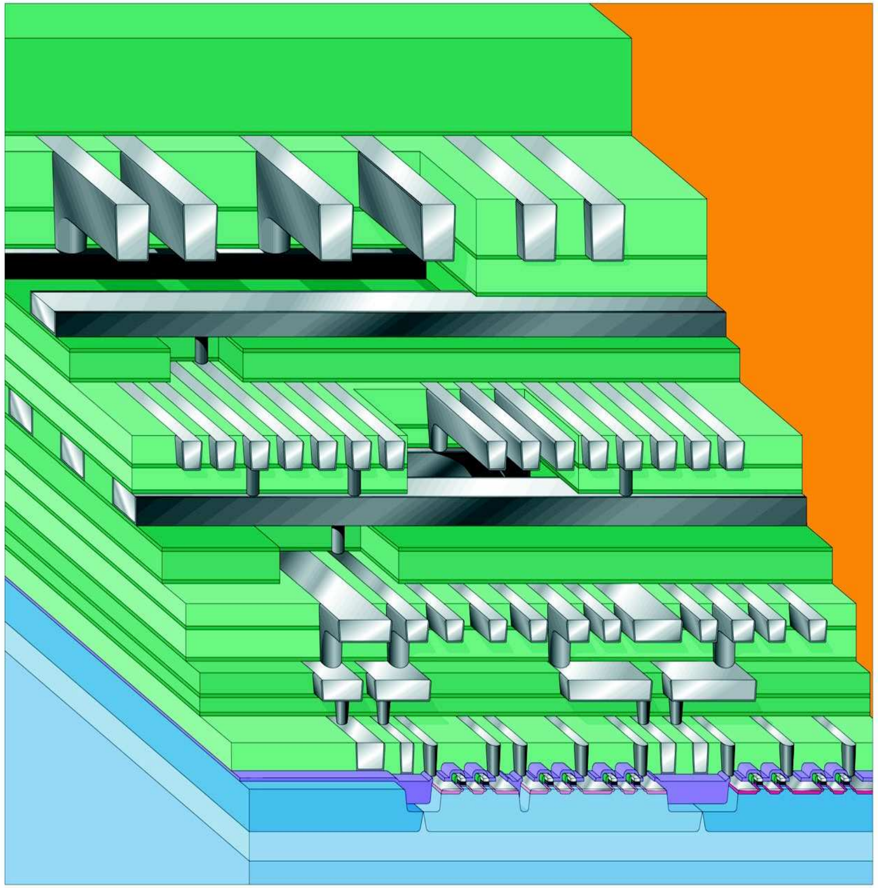
$\sigma \propto \mu b/\lambda, \quad \lambda = 1/\sqrt{\rho}$

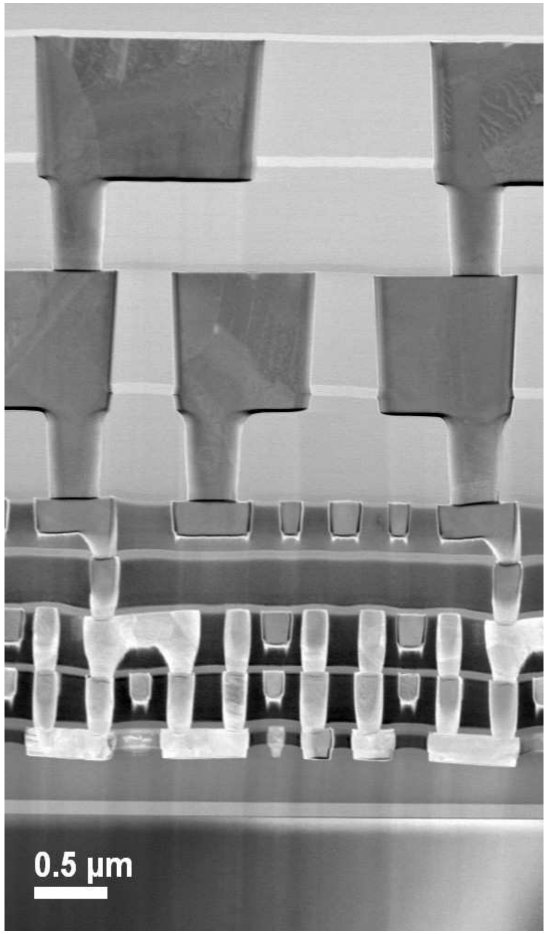
$$(\sigma_y - \sigma_o)^2 / (\alpha\mu b) = \rho_{stat} + \beta\, \varepsilon/d$$

=> Strong effect at small strain

Functionnal materials

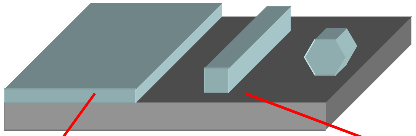
Motivation: scale reduction





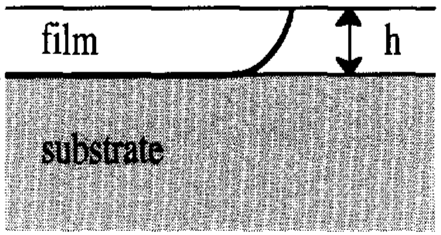
65nm design rules on 300mm wafer:
Metal levels interconnects using Cu and porous α-SiOCH

Dimensions:
Object size



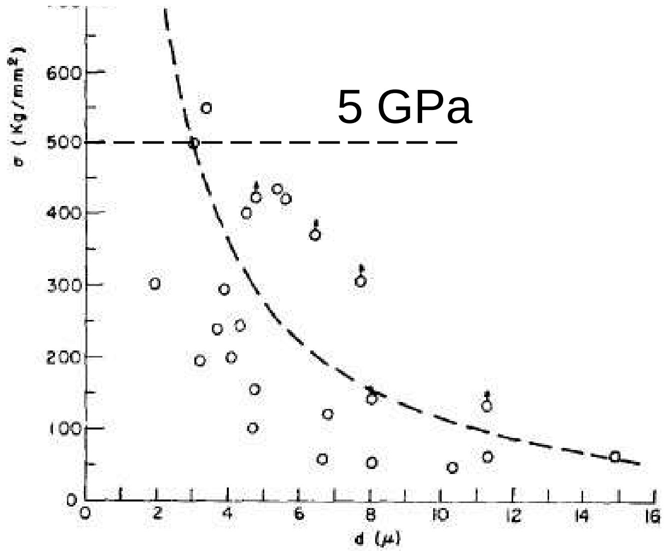
Whiskers

Films



$$\tau_o = \frac{\mu b}{4\pi h} Log\left(\frac{h}{b}\right)$$

Coherent/semicoherent epitaxy (Orowan/Matthews…)



[S.S.Brenner, JAP 1954; Acta 1956]

$\tau_{th} \sim \mu/10$	Theoretical strength
$\tau_{act} = \frac{\mu b}{4\pi h} Log\left(\frac{h}{b}\right)$	Expansion of a dislocation

Questions on scale effect (submicron range) as compared with "bulk"

- Is "smaller harder" ?
  - Strength evolution with scale ?
  - Strain hardening ?

- Mechanisms of deformation ?

- Is there a limit in ductility ?

OUTLINE

Introduction

-Motivations

Mechanical loading

- Uniaxial: tensile, compression
- Flexion
- Curvature
- Indentation
- Large strain
- Field measurements

Principle, limits and artefacts

Single crystal / Polycrystal – Interfaces(multilayers)

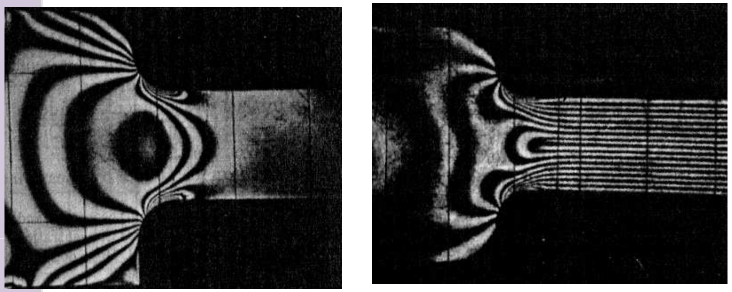
Challenges

Traction: l'idéal

$\sigma_{11}= E.\varepsilon_{11}$;

$\nu= \varepsilon_{22}/\varepsilon_{11}$

+ contrainte homogène



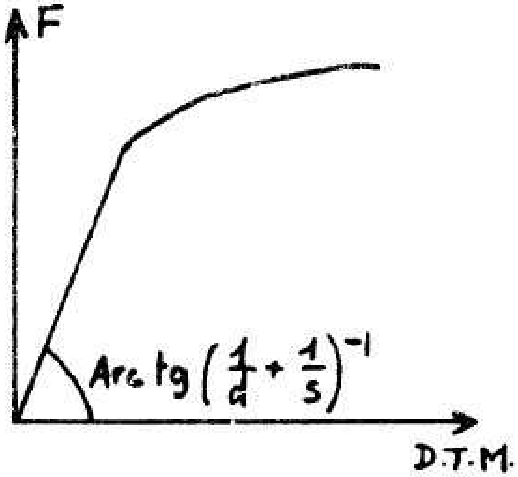
[Timoshenko]

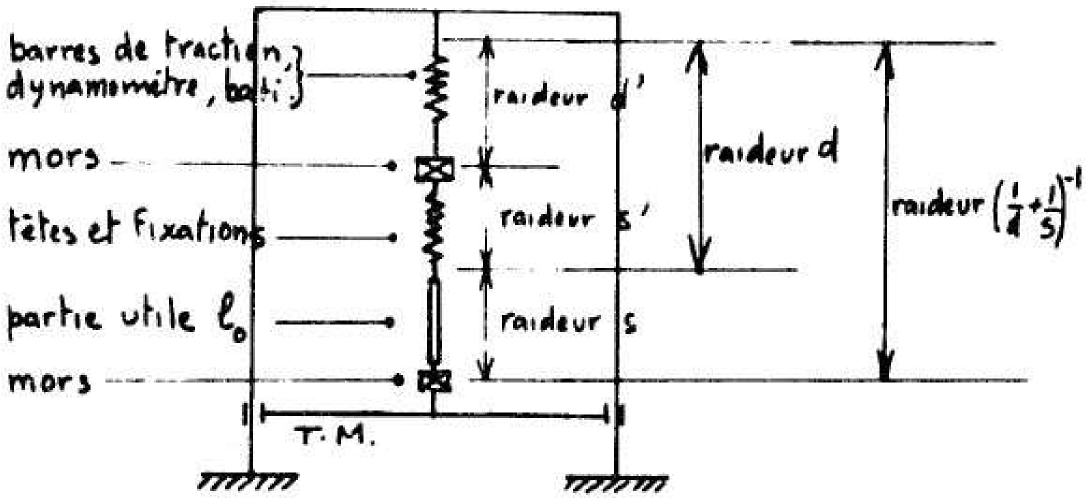
Rigidité cinématique (F),
Déformation mesurée sur l ' éch.

Alignement 3D sinon cintrage superposé !

Conditions limites aux mors (rotation)

Limite en déformation par striction,
dépend de:
-l'écrouissage (Considère)
-La sensibilité vitesse déformation

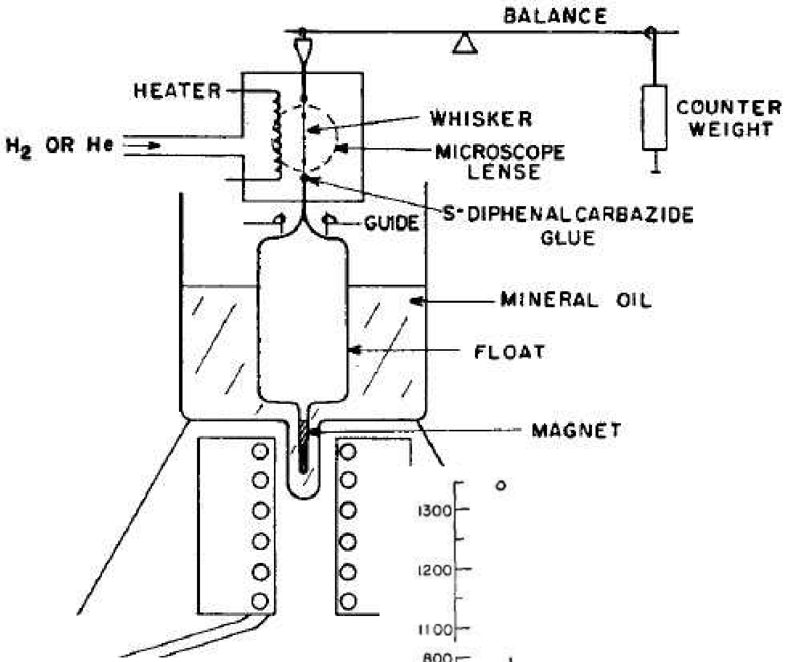


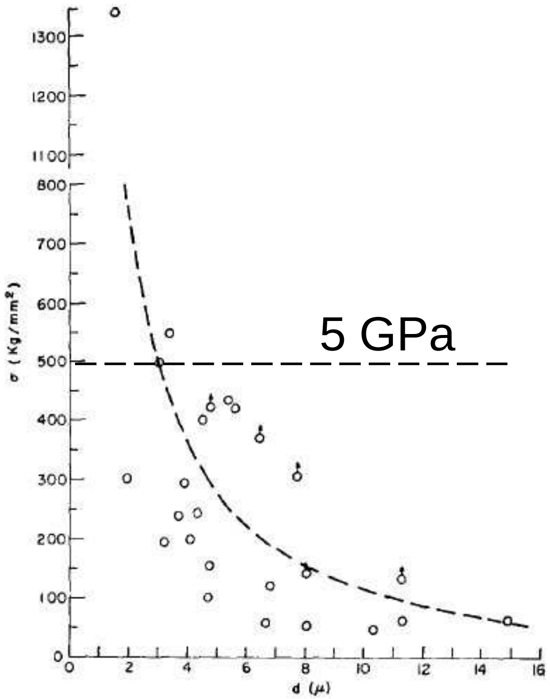


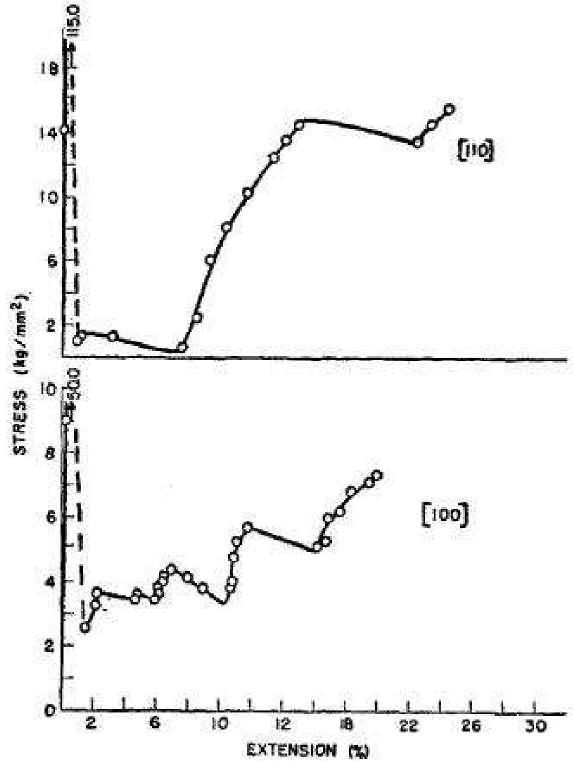
[JP/Cottu-Pratique de l'essai de traction- Yravals 1979]

Whiskers

[S.S.Brenner, JAP 1954; Acta 1956]







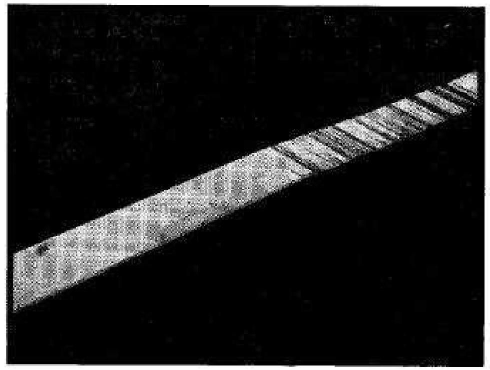
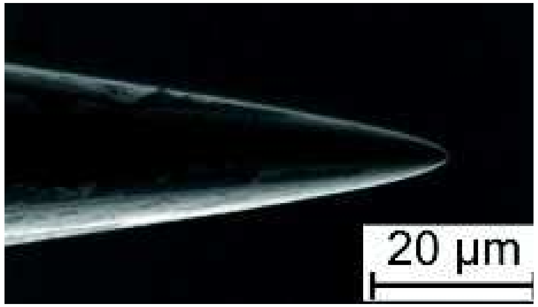
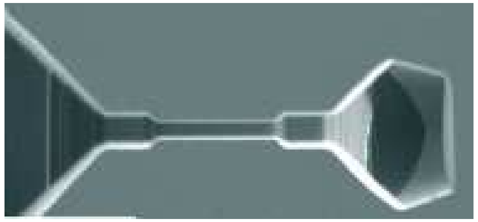


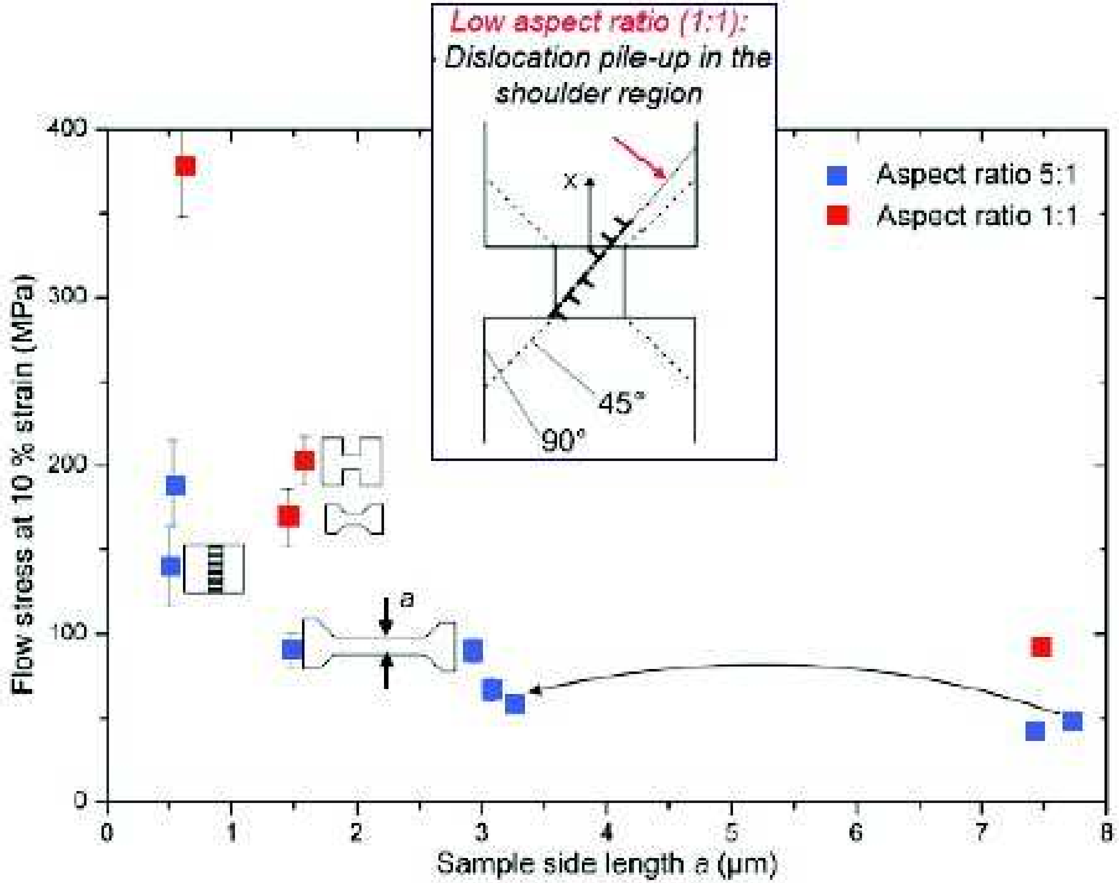
FIG. 4. Propagation of a Lüders band in a copper whisker, mag. 80×.

FIB shaped single crystal (Cu) in-situSEM -single slip orientation



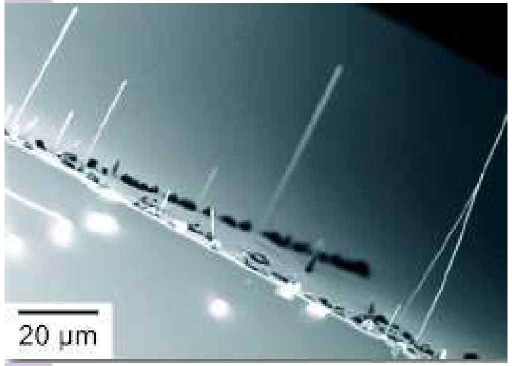
D.Kiener et al Acta Mat.56(2008)

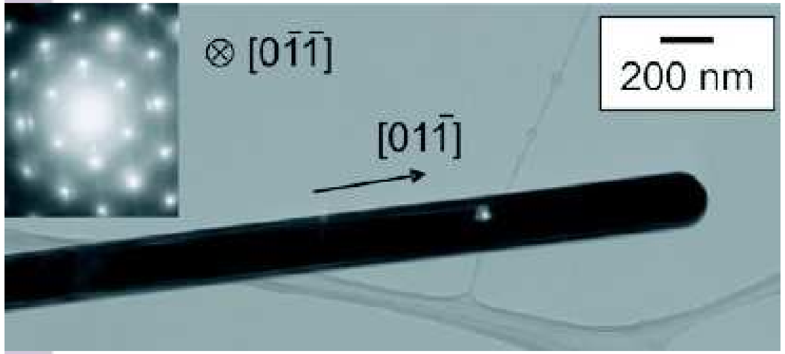


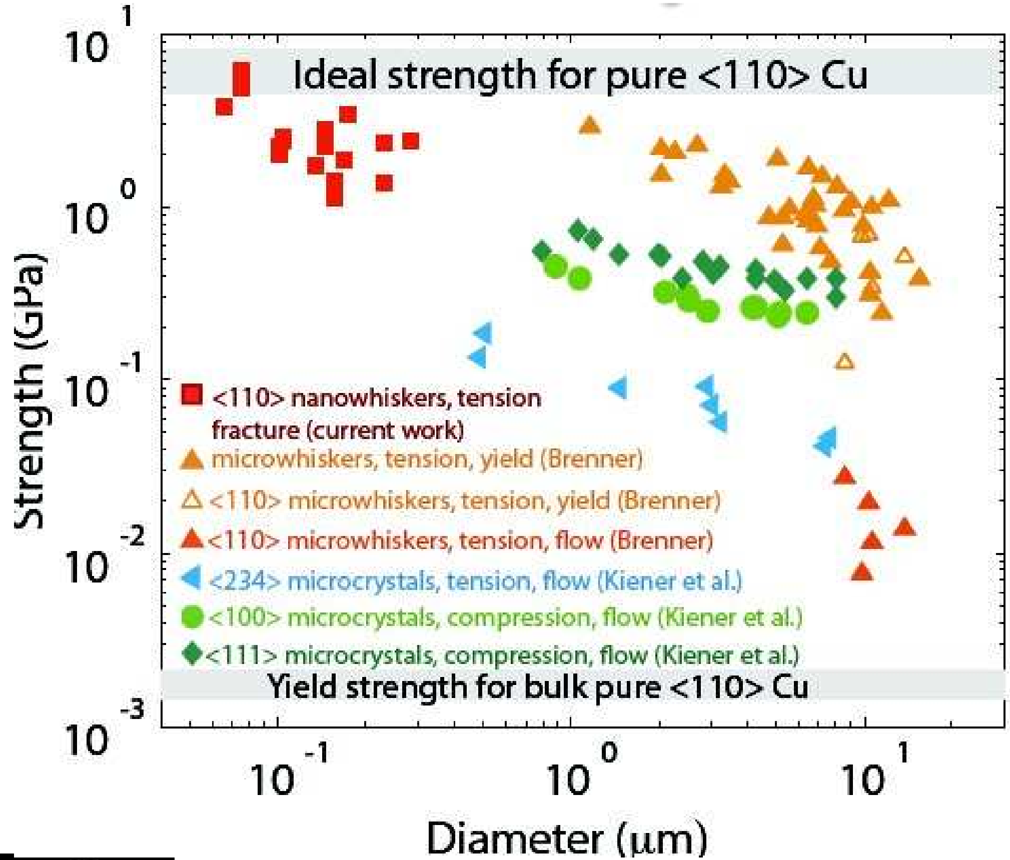


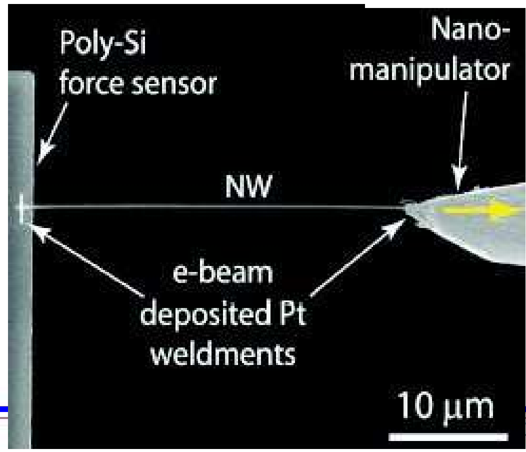
=> Small stress increase, artefacts increase it (aspect ratio; grips effect..)

Cu growth [Richter MPI]





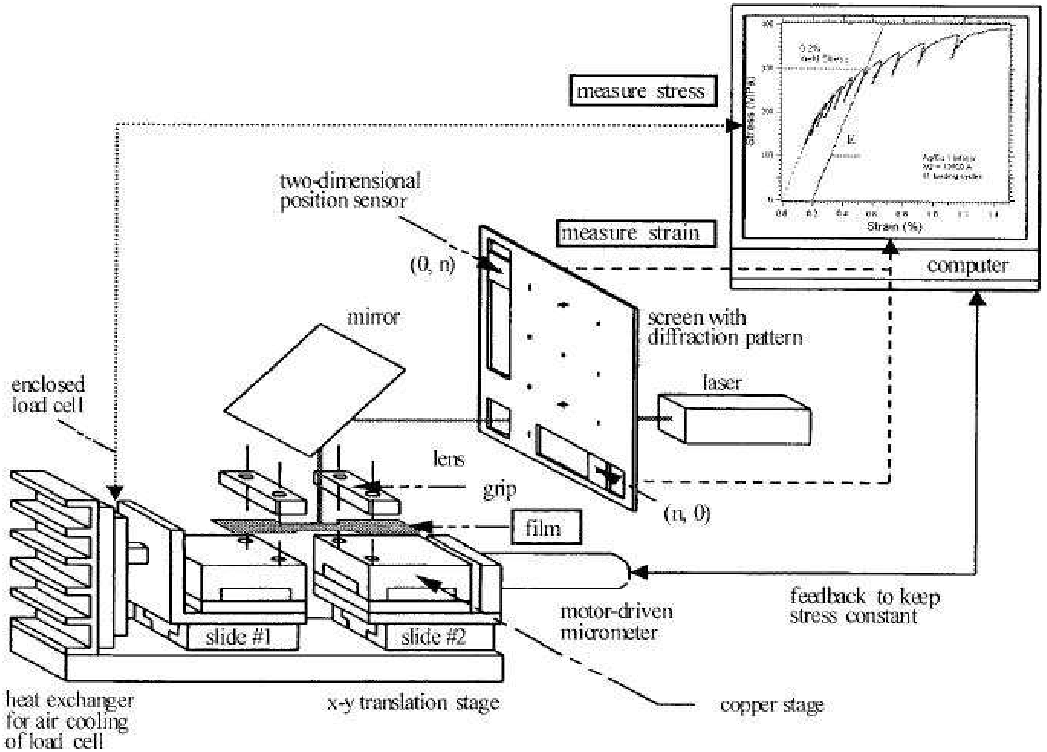


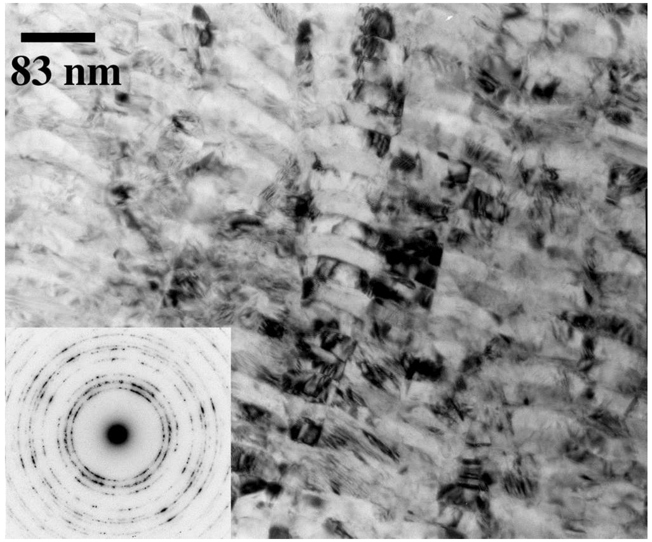


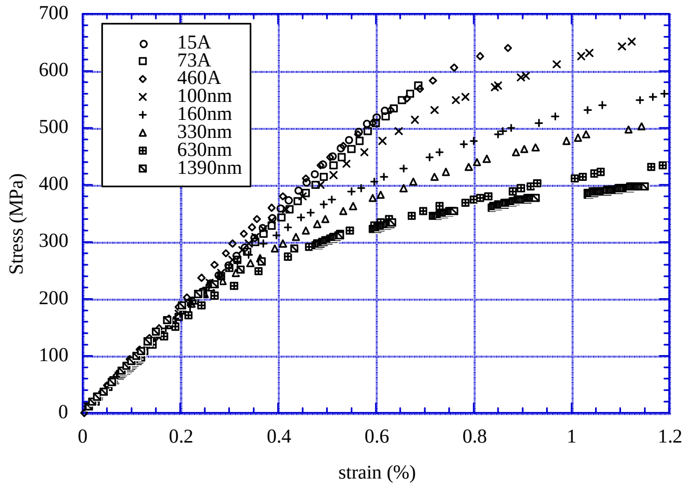
[D.Gianola et al. Penn State]

Example: free standing films Cu, Ag, Cu/Ag

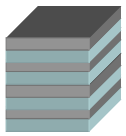
2 µm thick films



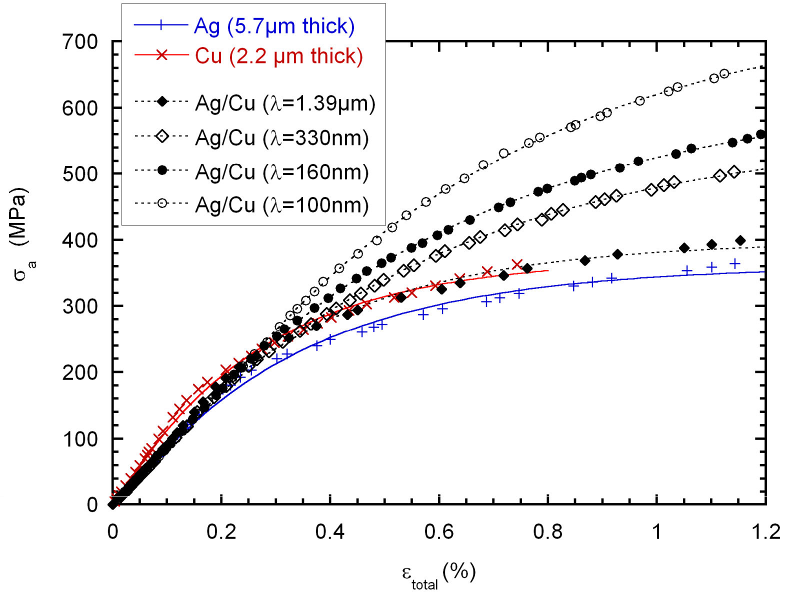




[Huang et al, Acta Mat. (2002)]



Transition élasto-plastique / écrouissage



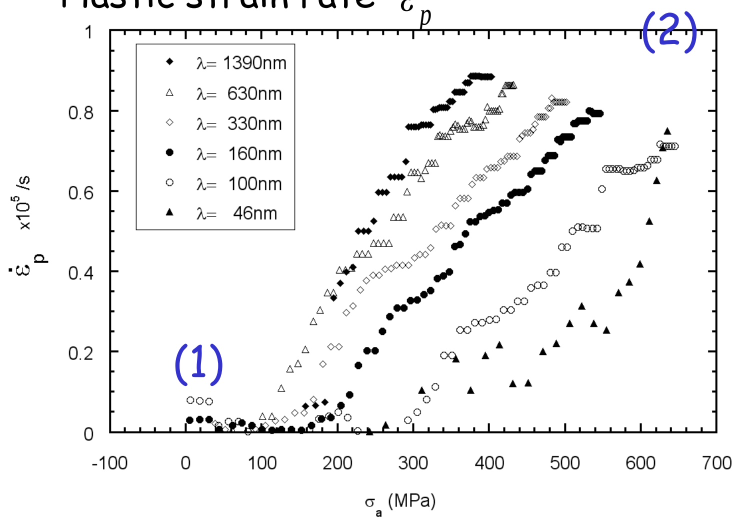
-> Hall-Petch like behavior, $\sigma_{0.2\%} \propto h^{-0.44}$

-> Increasing initial strong work hardening

- $\theta(0.2\%) > \theta_{II}$ and ↑

[M.Verdier et al, Phil Mag 86,5009 (2006)]

Plastic strain rate $\dot{\varepsilon}_p$



$$\sigma_a = \sigma_m + \sigma_r \left[1 - \exp\left(C \frac{\varepsilon_m - \varepsilon_a}{\sigma_r}\right)\right]$$

(1) Microplast. stress $\sigma_m \propto 1/h$

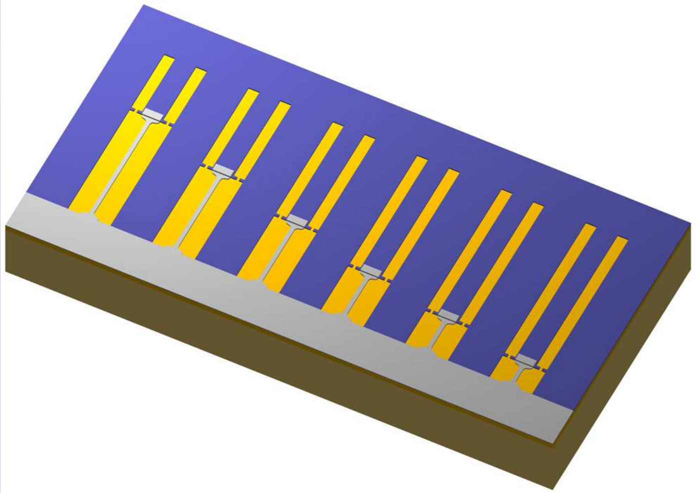
$$\sigma_m \approx 0.4 \frac{\mu b}{\lambda} \ln \frac{\lambda}{b}$$

(2) Saturation stress $\sigma_{sat} \propto 1/(h)^{0.5}$

$$\sigma_{sat} = 278 + 9570 \sqrt{\frac{b}{\lambda}}$$

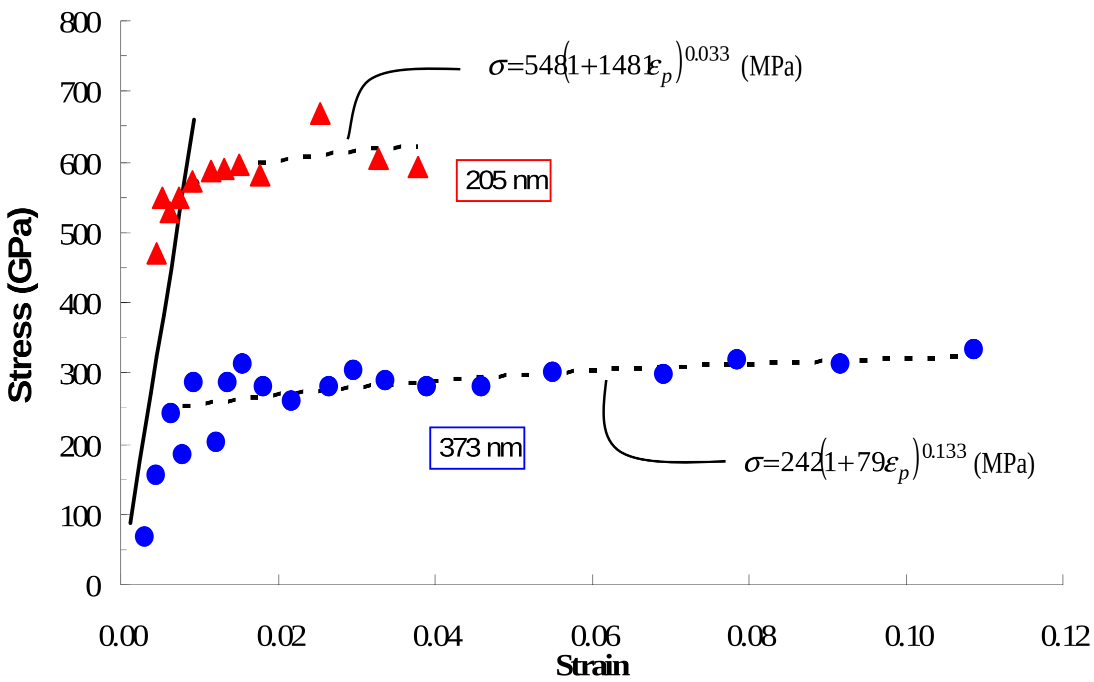
[G.Saada et al Phil. Mag.,87,4875 (2007)]

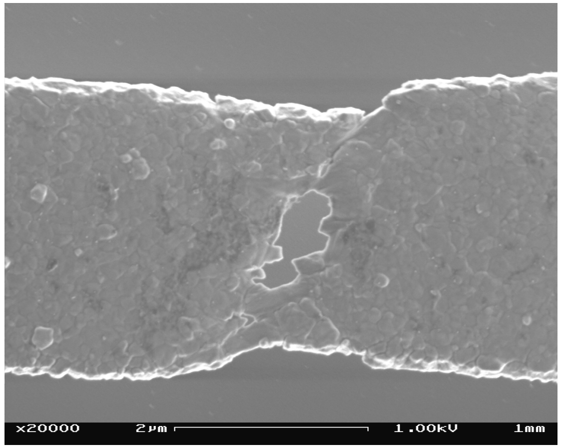
Ex: Elegant on chip simple system (films Al pχ), [T.Pardoen et al. UCL]



- SiN actuator (blue): release of residual stress
- One sample / strain, under load observations !
- No alignment problem (integrated meas.)

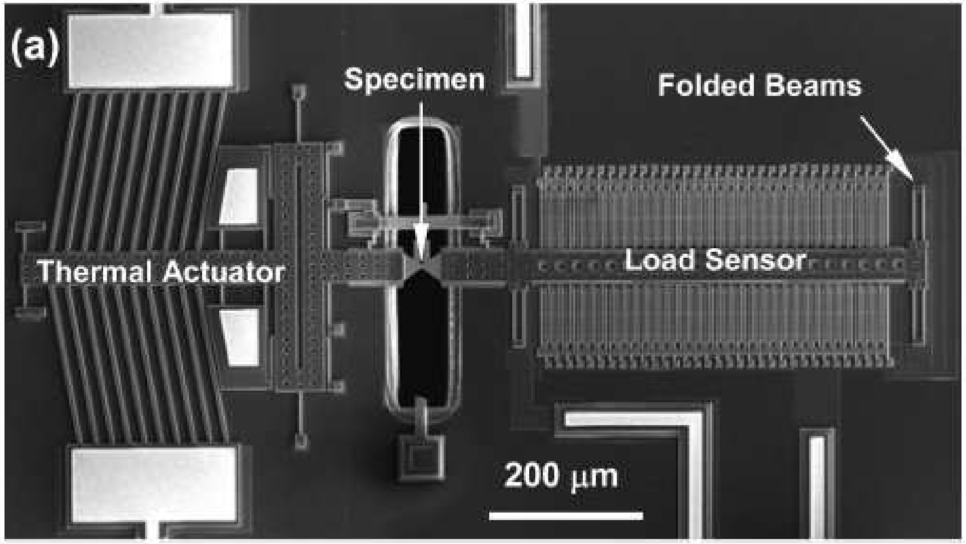
=> Large strain (4% for 200nm thick films)

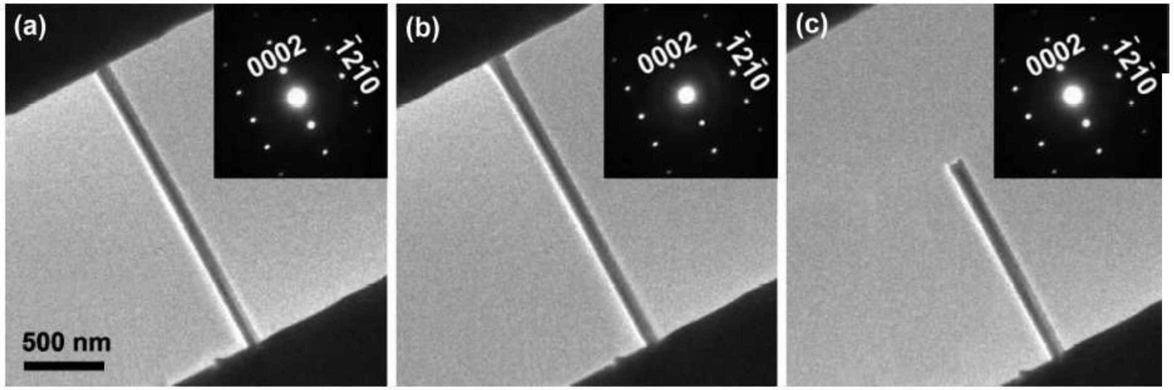


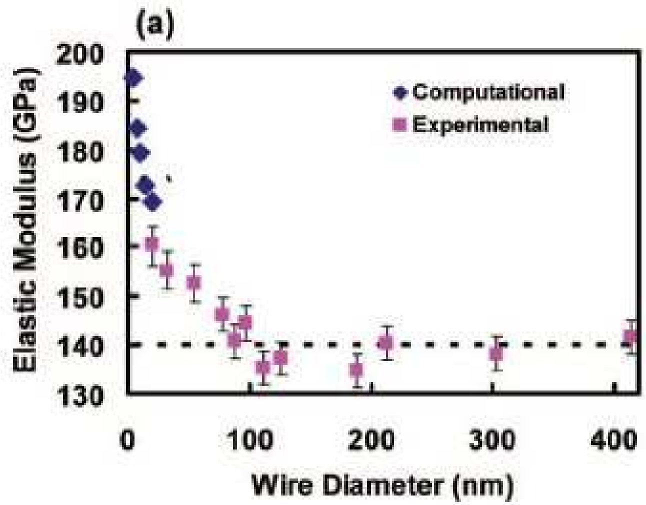


Full on-chip (not cheap) test

Nanowhiskers: ex. ZnO ,nMTS

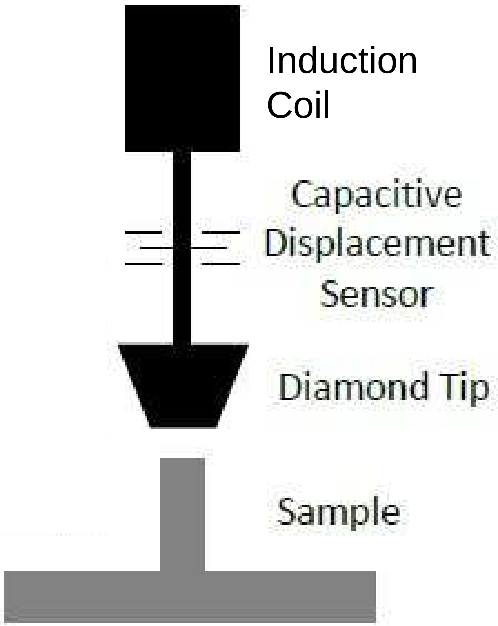


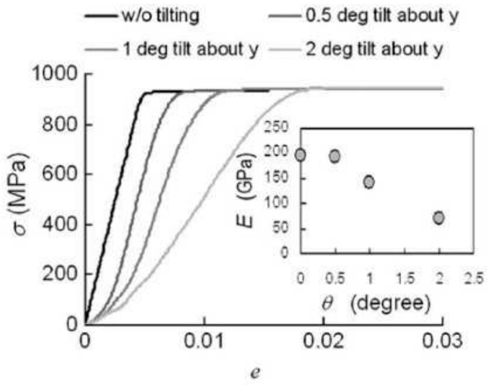




Espinosa et al. Nano Letters,8 (2008)

Compression: « Micropillar »





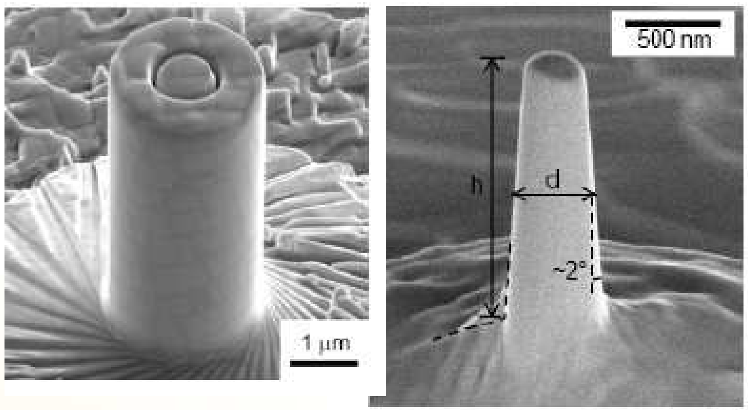
[Y.S.Choi et al. Scripta Mat.(2007)]

Stiffness (base mat.)

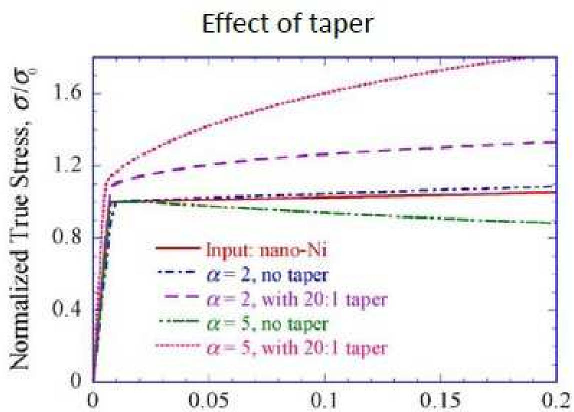
Alignment

Geometrical limit:
Buckling (flambement)
Barreling (Tonneau)

=> Boundary condition
(in theoory: no friction)

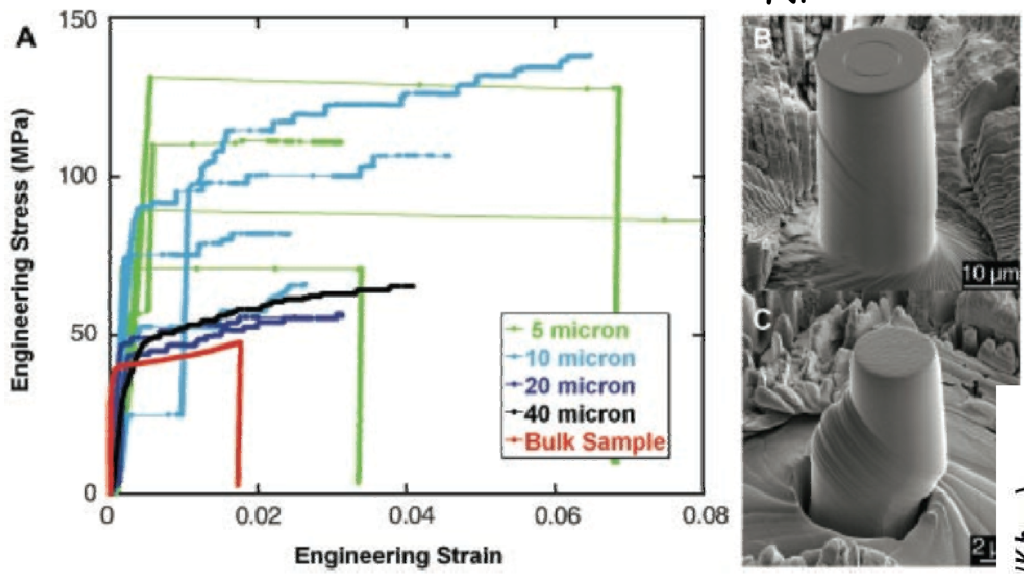


440 nm Dia. Au Pillar
Volkert & Lilleodden 2006

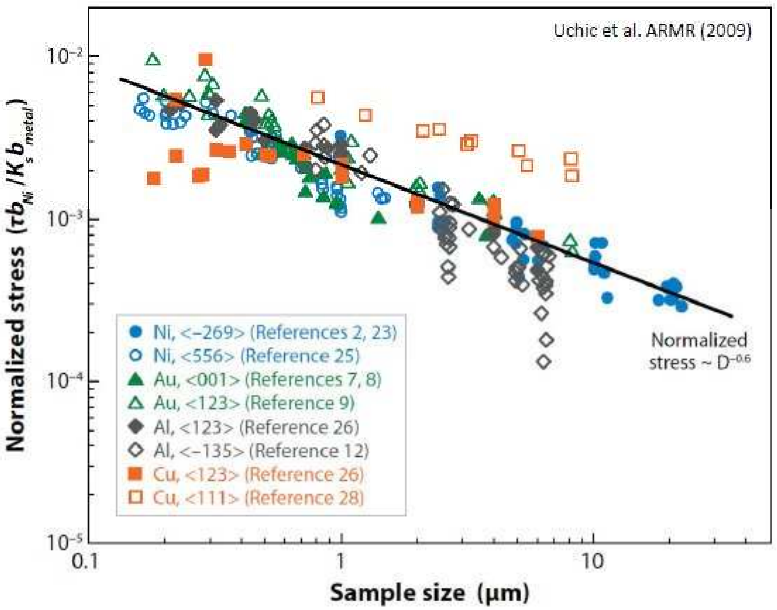


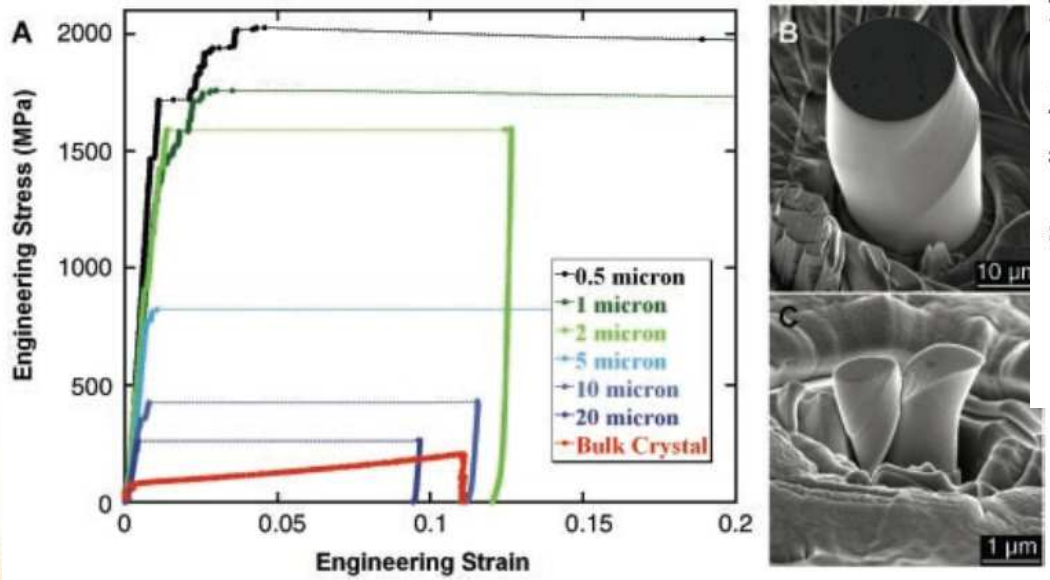
H.Zhang et al. Scripta Mat.(2006)

Ni



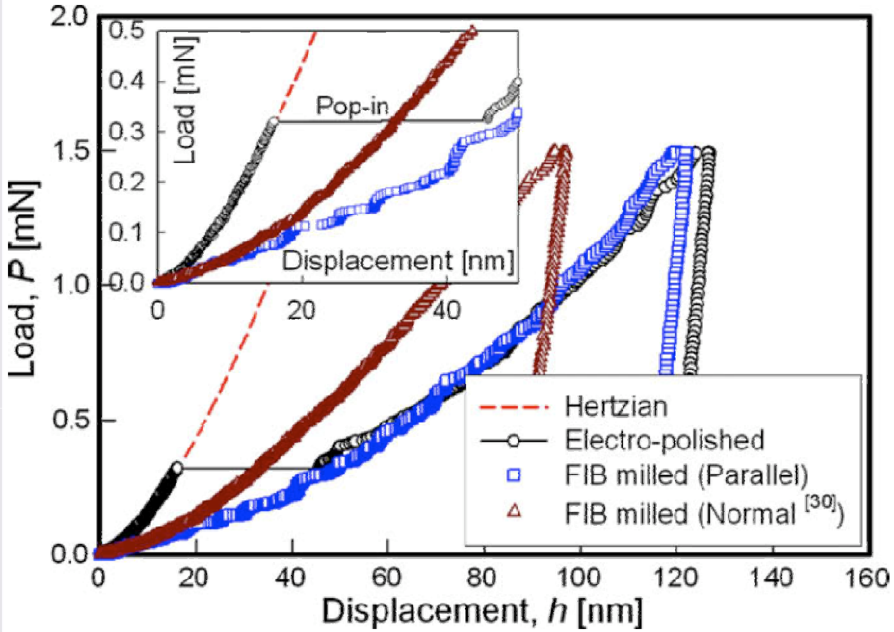
Huge effect !!



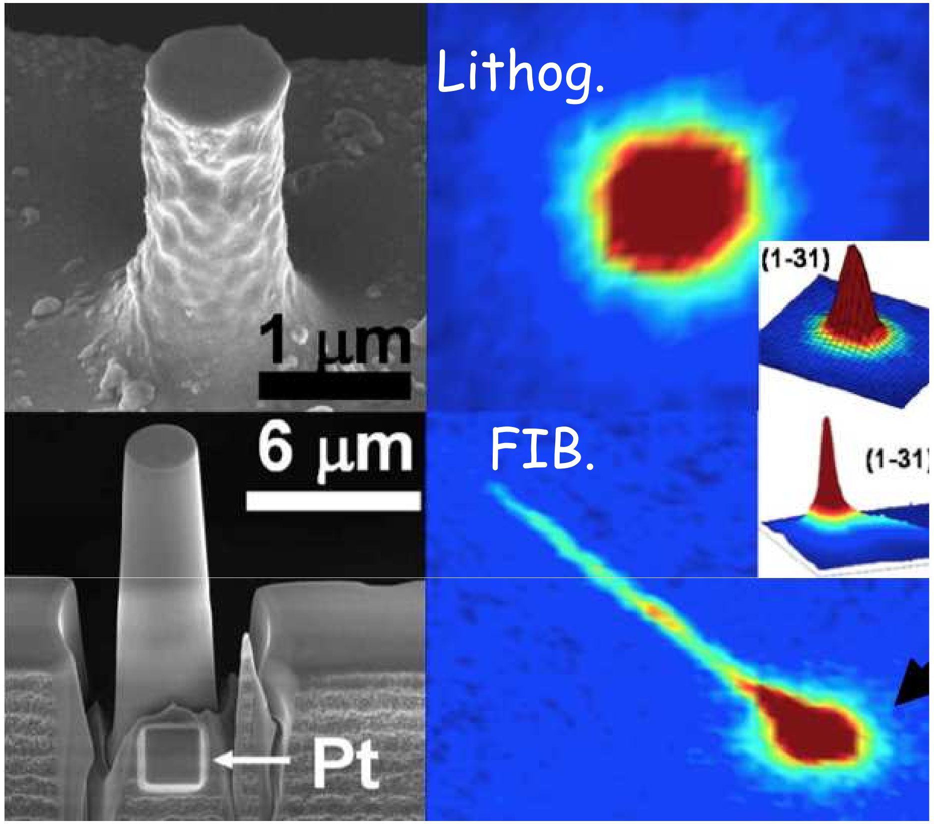


Uchic, Florando, Dimiduk, Nix, Science 2004

Problem : FIB damage



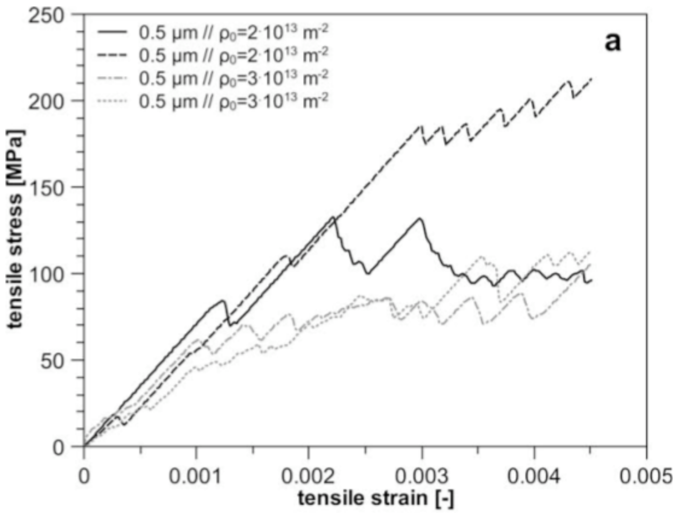
Shim, Bei, George, Pharr, Acta Materialia 2009.



Van Swygenhoven,
Volkert et al

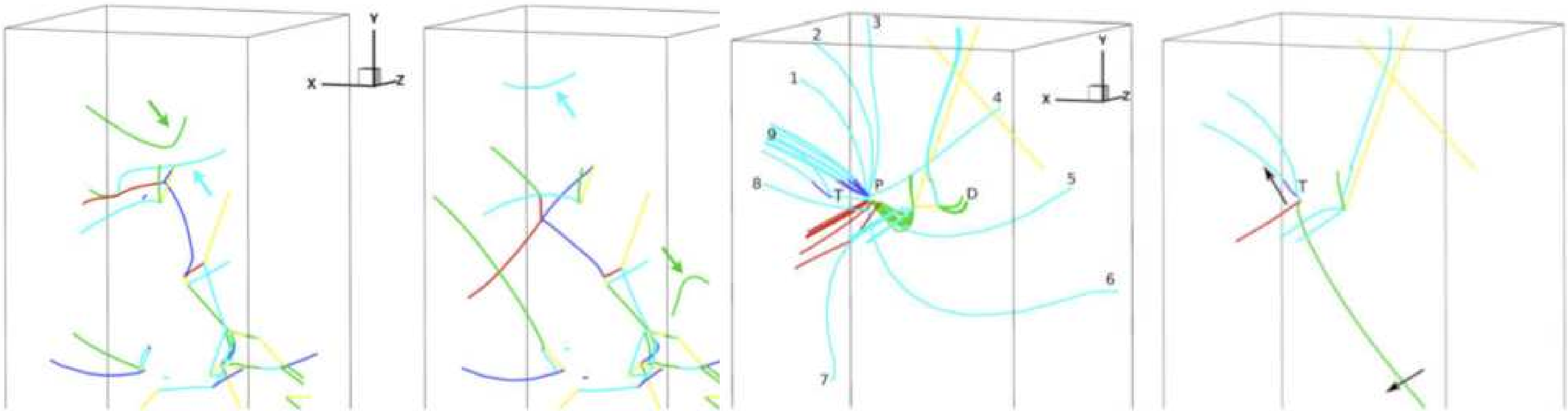
Swiss Light Source /PSI
In situ Laue X-ray

Dislocation Dynamics of micro-pillar



DDD: D.Weygand (KIT)

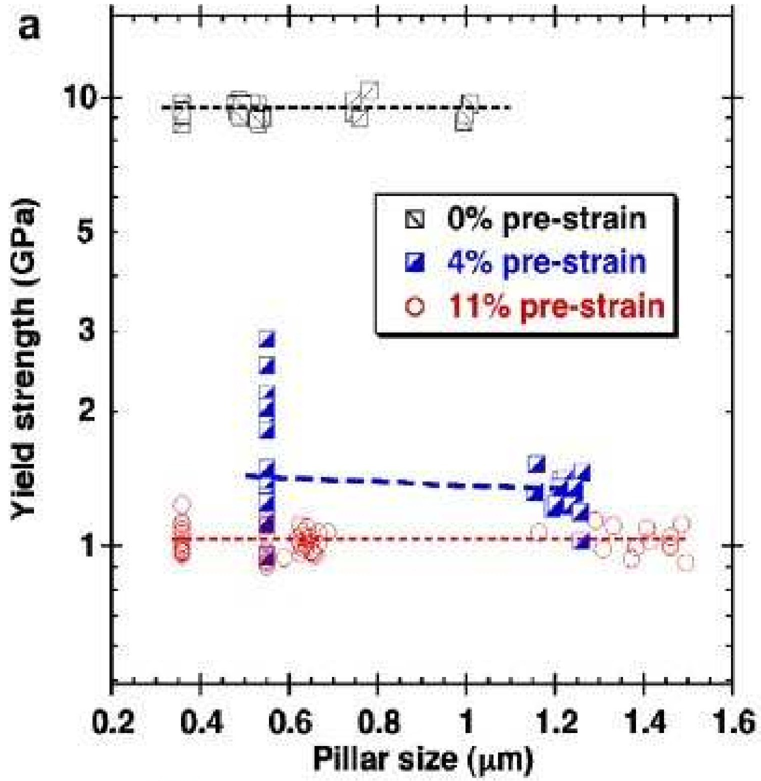
Main effect: initial density of source, distribution of spiral source

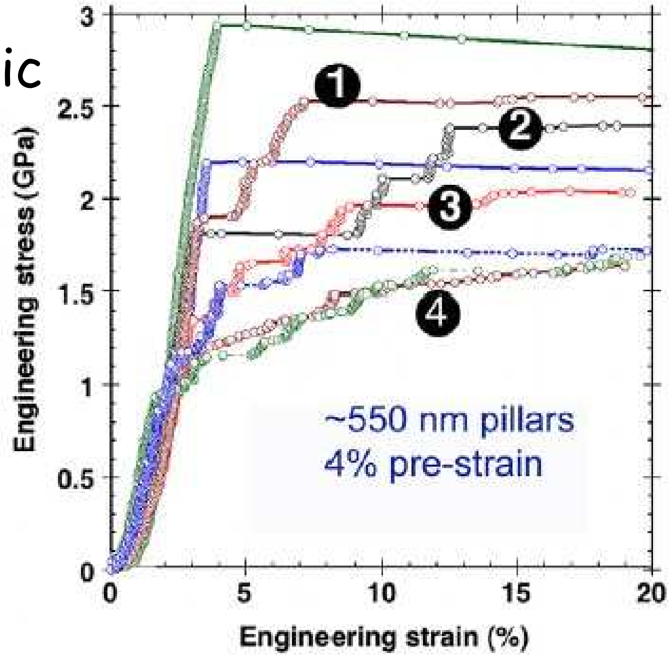


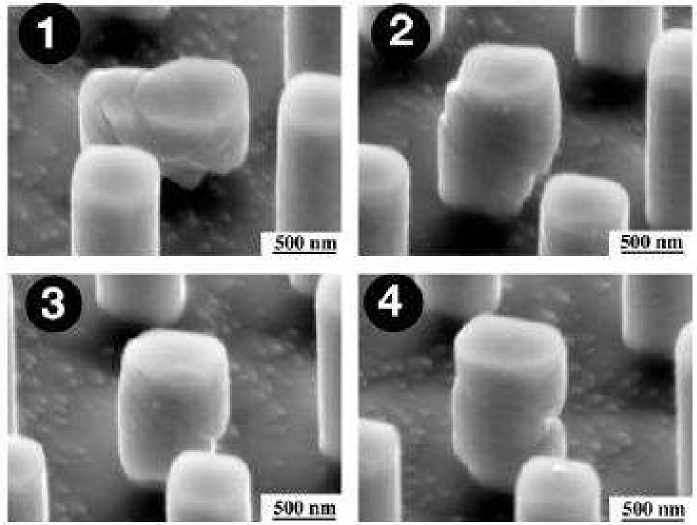
Motz, Weygand, Gumbsch et al, Acta Mat 2009

=> Main effect: initial density of defects…

Mo pillars: chemical etching of NiAl-Mo eutectic





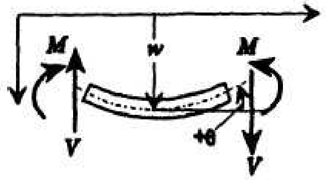


[Bei et al. Acta 56 (2008)]

Flexion:

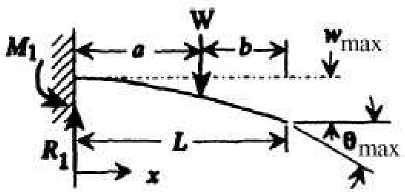
Grande dépendance géométrique: Module (L^3), σ en L^4,
Plasticité: gradient de déformation, σ(épaisseur),
Déformation localisée aux CL, rigidité des bords
3 points (Moment linéaire), 4 points (Moment constant)

Notation



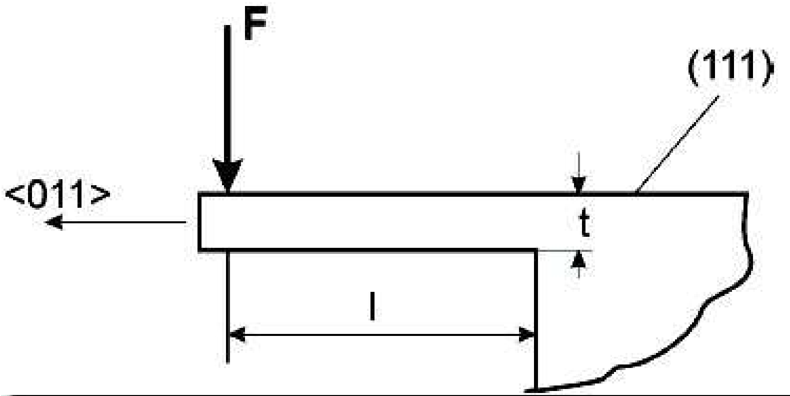
L = length of beam
E = modulus of elasticity
I = moment of inertia

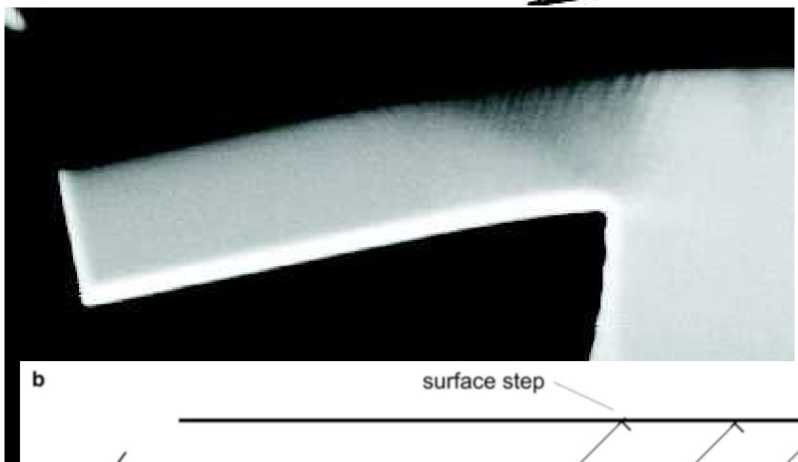
The positive directions of the reactions (R_1, R_2, M_1, M_2) are shown in the figures for each case. Coordinate x is measured from the left-hand end for all entries in this table.

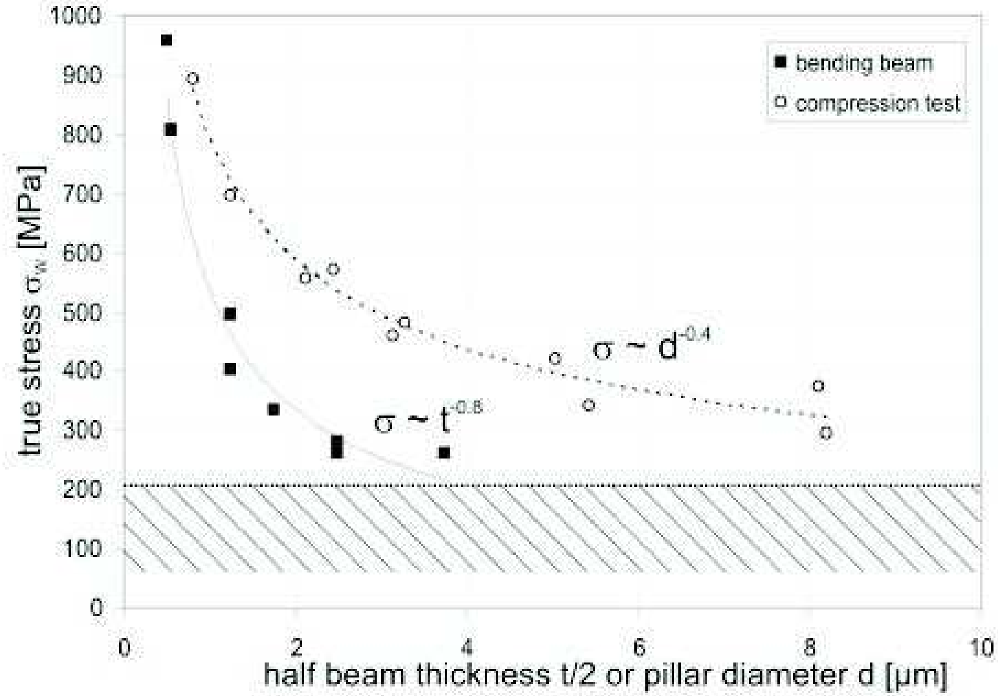
Type of Beam	Reactions	Deflection at Any Point x
1. 	$R_1 = W$ $M_1 = Wa$	For $x < a$, $\frac{W}{6EI}(-x^3 + 3x^2 a)$ For $x \geq a$, $\frac{W}{6EI}(3a^2 x - a^3)$

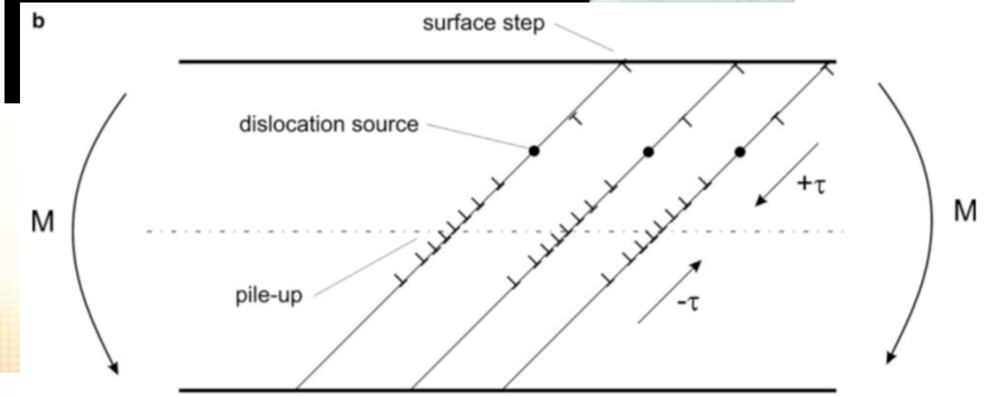
Flexion:

D.Kiener et al. Adv.Eng.Mat. **8** (2006)







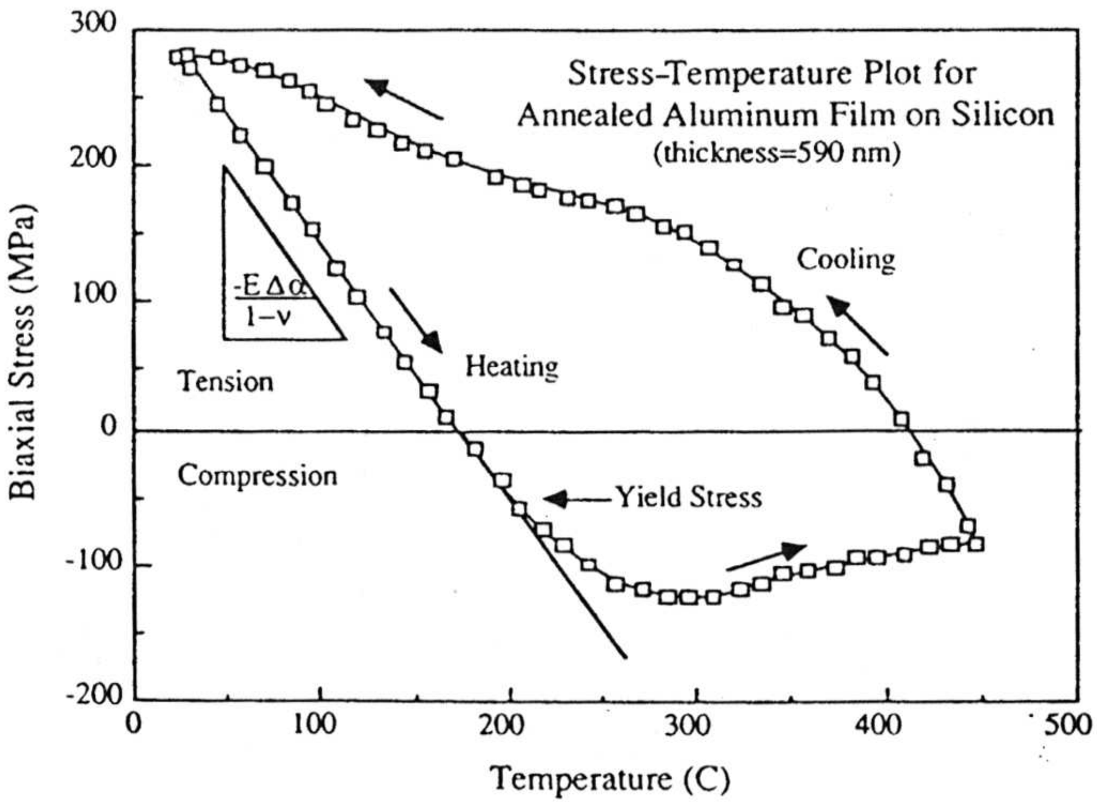


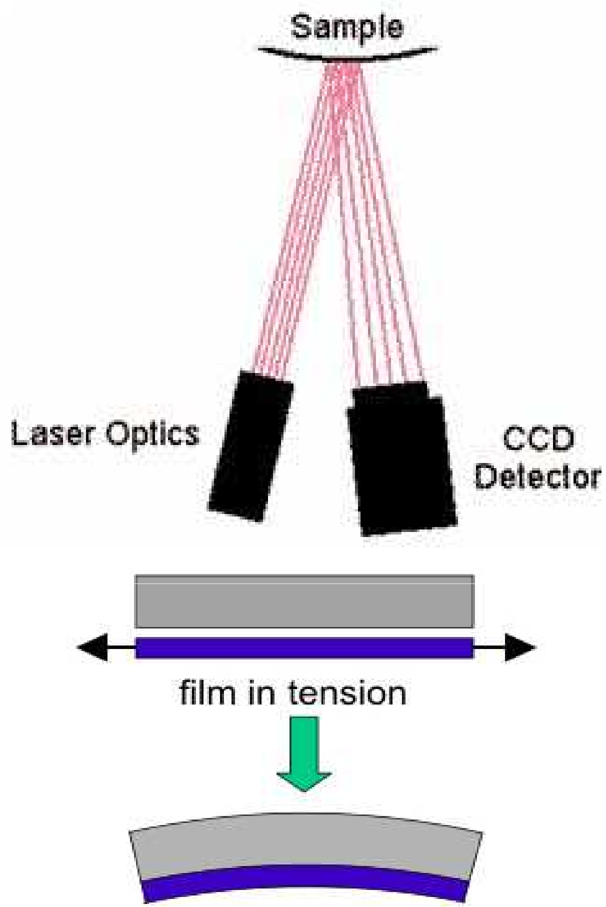
Strong effect of strain gradient
Exponent n of t^{-n} > micropillar

Curvature measurement

Imposed strain: thermal mismatch $\Delta\alpha\Delta T$: < 0.5%

- Substrate is the strain gauge (elastic)
- Plasticity on heating and cooling



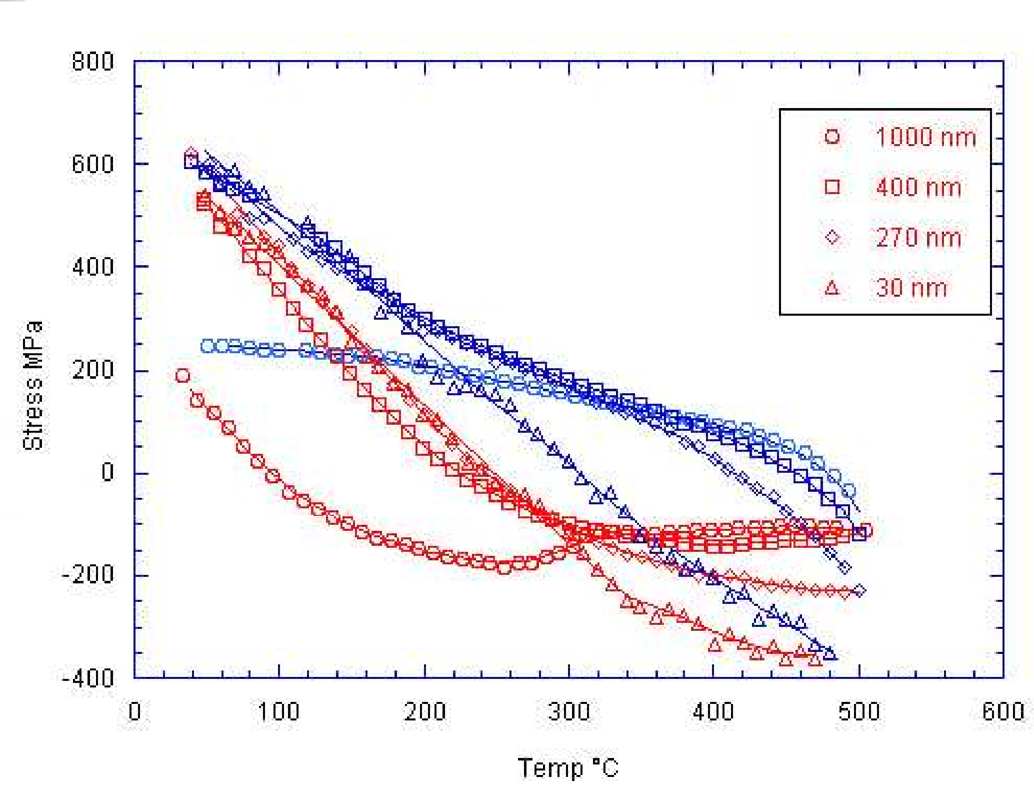


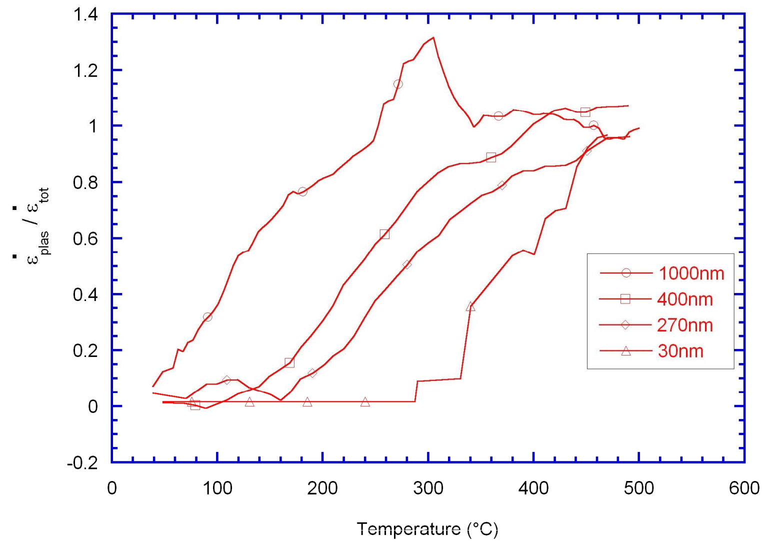
Stoney's Equation:

$$\sigma_{film} \cdot h_{film} = M_{sub} \frac{h_{sub}^2}{6R}$$

Thin film on substrates: stress temperature measurements

Data from E.Arzt group: T.Balk et al (2003),G.Dehm et al (2003), Weiss et al (2001)



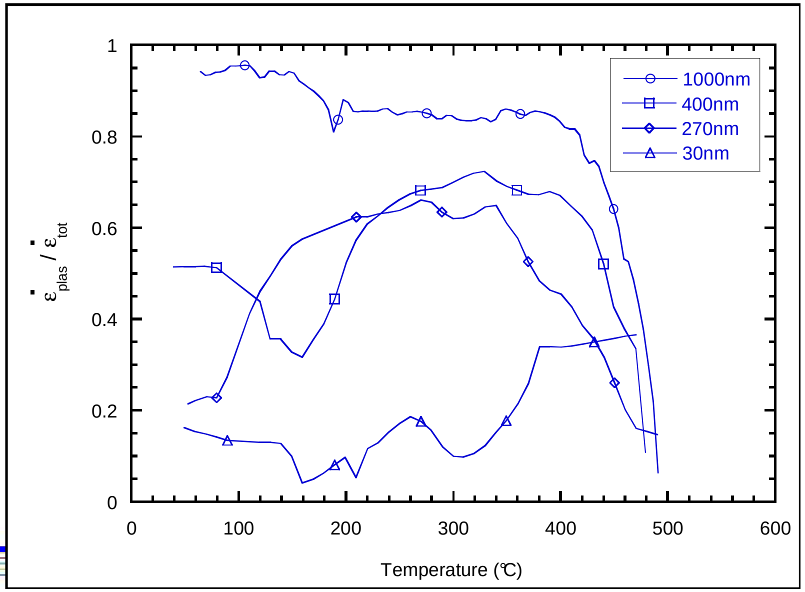


$$\dot{\sigma}_a = C(\dot{\varepsilon}_a - \dot{\varepsilon}_p)$$

$$\theta \equiv \frac{d\sigma_a}{d\varepsilon_a} = C\left(1 - \frac{\dot{\varepsilon}_p}{\dot{\varepsilon}_a}\right)$$

$$\longrightarrow \quad \dot{\varepsilon}_p = \dot{T}\left(\alpha - \frac{d\sigma}{CdT}\right)$$

[Saada et al. 2007]



Thin film on substrates: stress temperature measurements

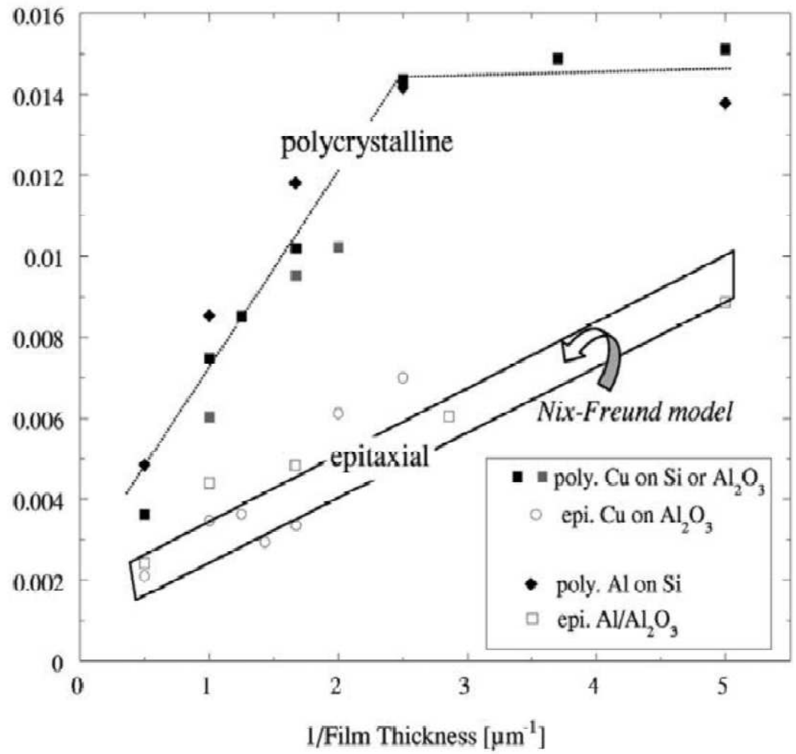
On cooling, deformation in polycrystalline films is in the elasto-plastic transition (h<1µm)

Only thick film deforms in the plastic regime,

Applied stress more or less sufficient

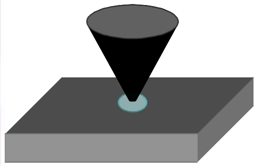
⇒Residual stress is NOT the elastic limit

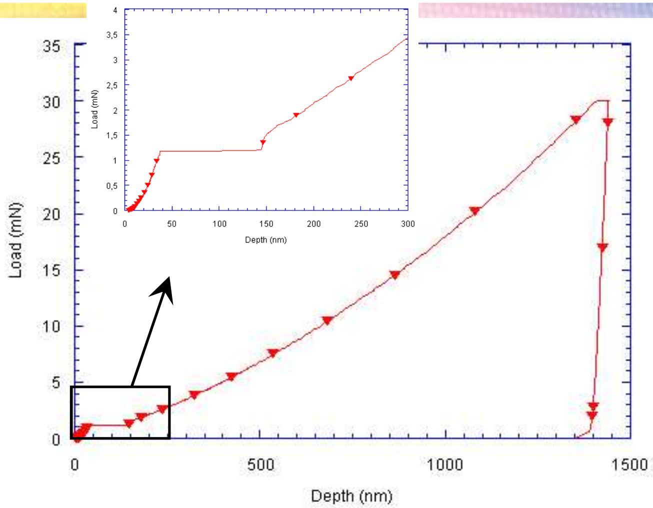
Not the case for epitaxial films



G.Dehm et al , Jal Microelect. Eng.(2003)

Nanoindentation





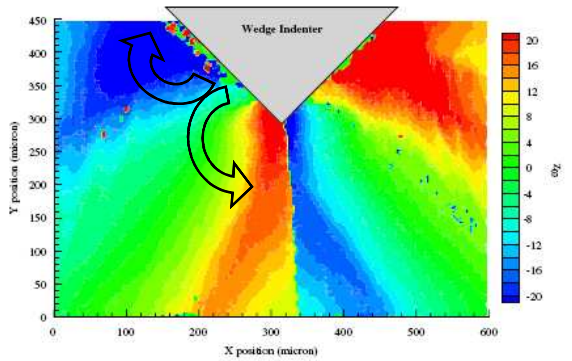


Fig. 7. In-plane lattice rotation (in degrees) map of Cu single crystal under wedge indentation.

J. W. Kysar et al. / J. Mech. Phys. Solids 55 (2007)

Module :E/(1-ν^2),

Conditions limites mixtes, chemin déformation complexe…

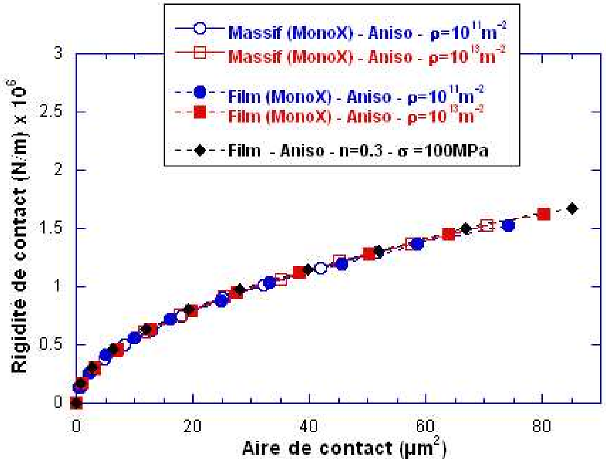
Effet géométrie pointe: type sollicitation : ε cst –cone autosimilaire; ε croissant -sphere,

-Gradient de déformation

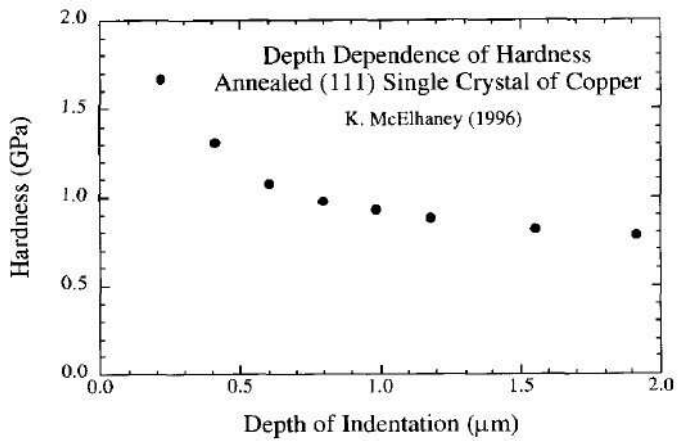
$\Rightarrow$Unique relation(Sneddon): rigidité du contact: **S= 2.E*.r$_c$** toujours valide (élast./plastique…) !

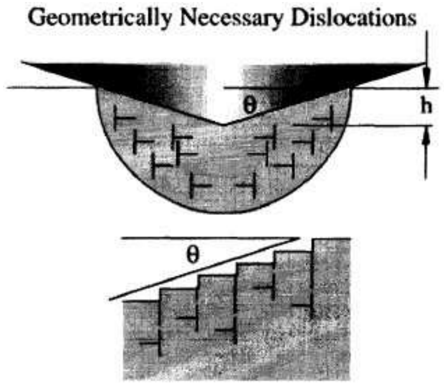
(Elastic)

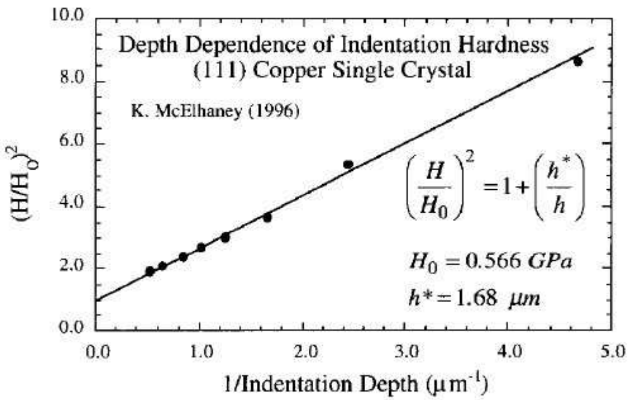
Indenteur	Cône	Sphère	Poinçon plat
$P(h)$	$\frac{2}{\pi}E_{eq}\tan(\psi)h^2$	$\frac{4}{3}E_{eq}\sqrt{R}h^{\frac{3}{2}}$	$2E_{eq}Rh$
$S(h)=\frac{dP}{dh}$	$\frac{4}{\pi}E_{eq}\tan(\psi)h$	$2E_{eq}\sqrt{Rh}$	$2E_{eq}R$
$r_c(h)$	$\frac{4}{\pi}E_{eq}\tan(\psi)h$	$\sqrt{Rh}$	R
$S(r_c)$	$2E_{eq}r_c$		



Size effect







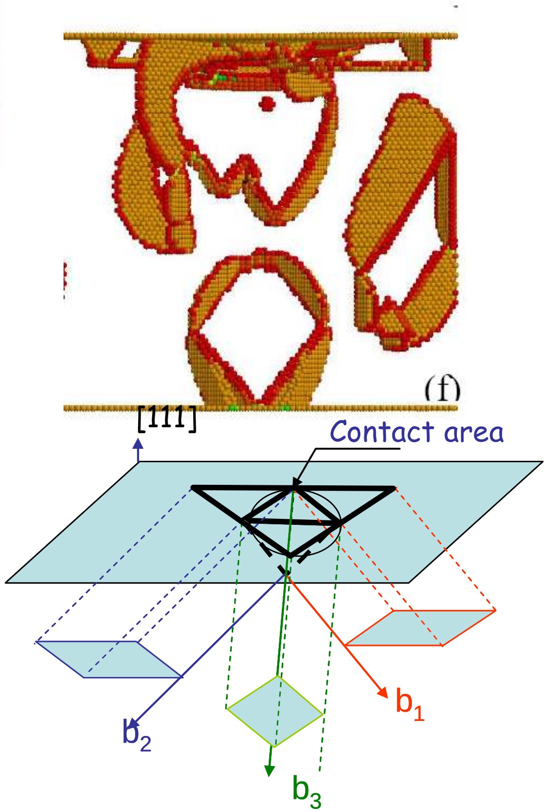
$$\rho_G = \frac{3}{16bh(\cot\theta)^2} \xrightarrow{\tau = \alpha\mu b\sqrt{\rho_G + \rho_0}} \left(\frac{H}{H_0}\right)^2 = 1 + \frac{h^*}{h}$$

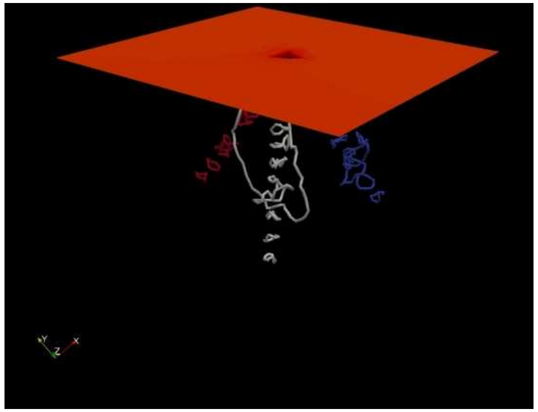
!! Surface preparation / crystal prep. (UHV) not mech. Polishing…

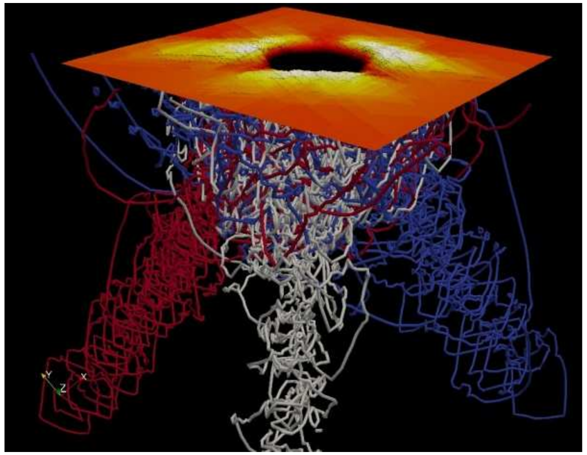
-More an elastic-plastic transition (nucleation/expansion: see DDD)

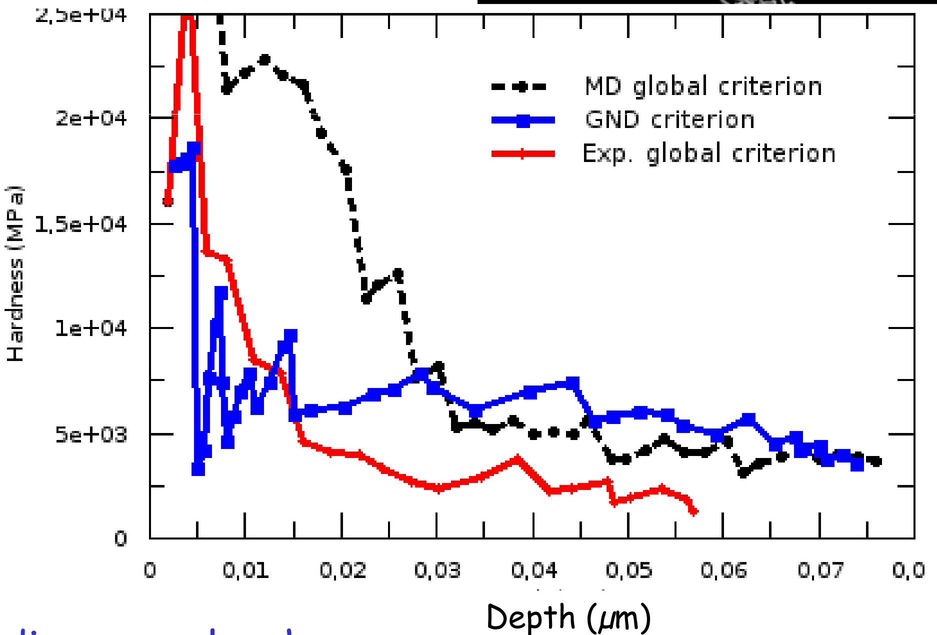
- Tip shape is not ideal on the whole length scale

Dislocation dynamics (TRIDIS)









Nucleation criteria: to be investigated

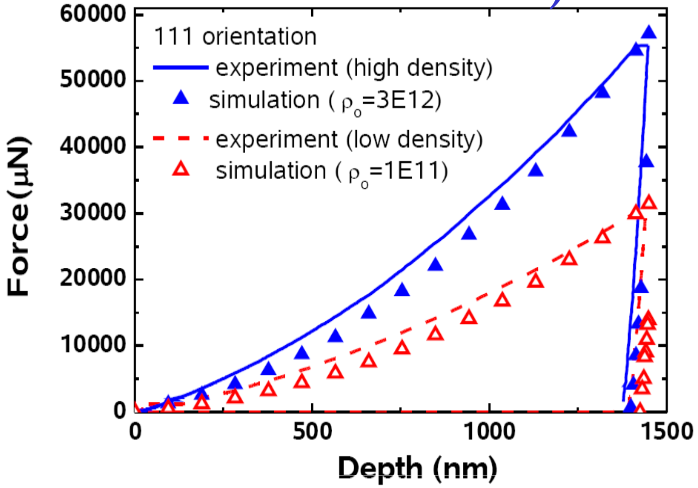
Size effect here with prismatic loop radius -> avalanche

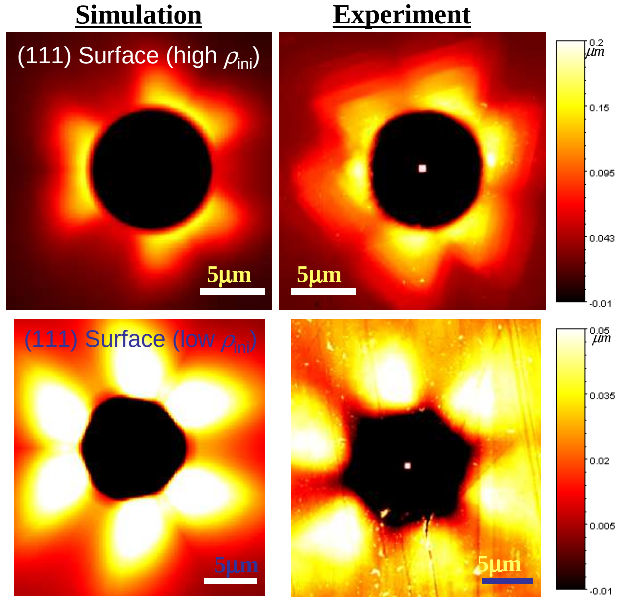
[Huang et al. CRAS 2011]

Crystal plasticity FEM

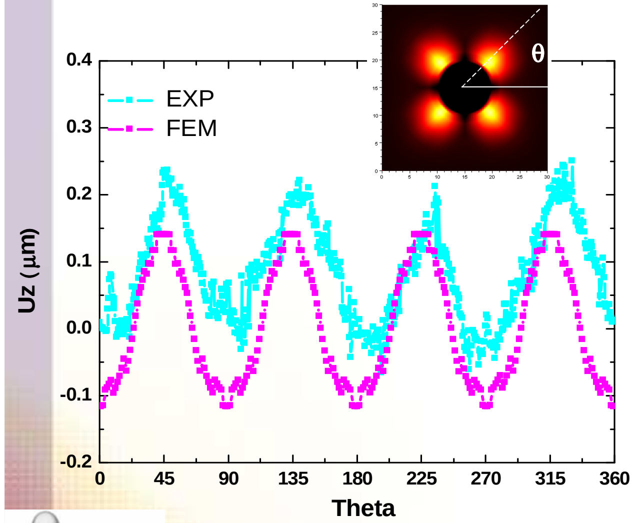
Unique ajustement: densité de dislocation initiale sur la courbe de force:

1) bon ordre de grandeur (/RX)
2) déformée de surface aussi !!

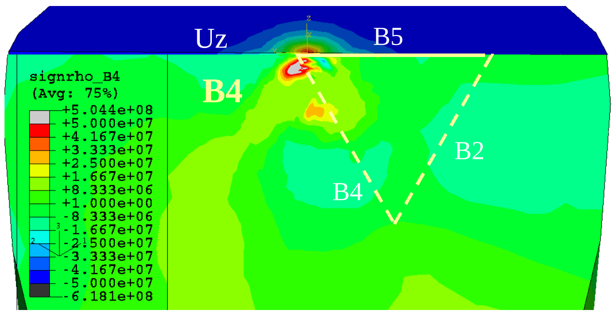


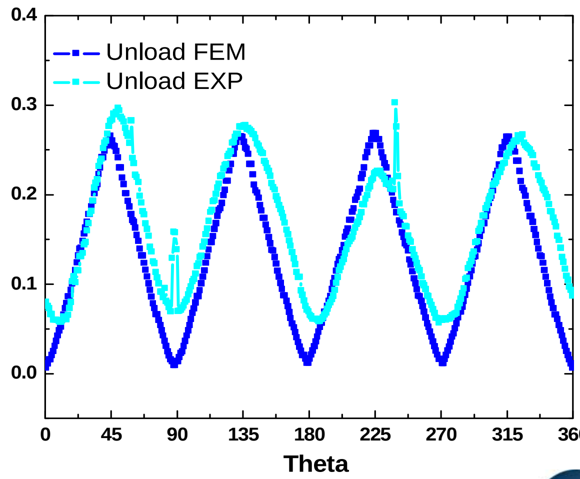


Ex: 2 monocristaux Cu [111]



Ex: monocristal Cu [001]

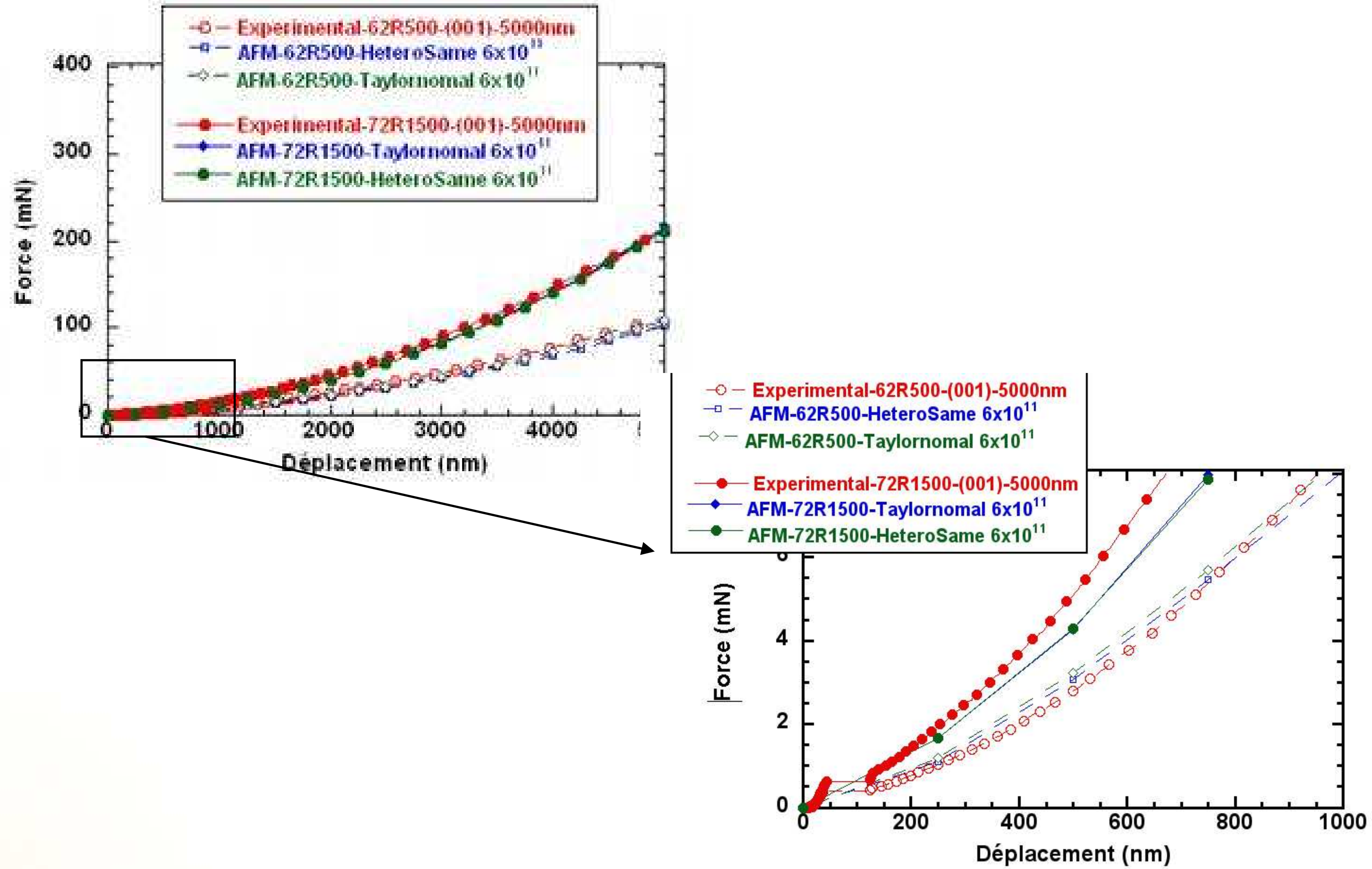




[HJ Chang, Thèse 2009]

Robust modelling: change of indenter tip angle

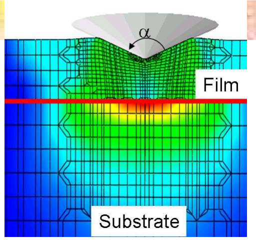
Same single crystal[001], 2 different tip angle (62, 72°)

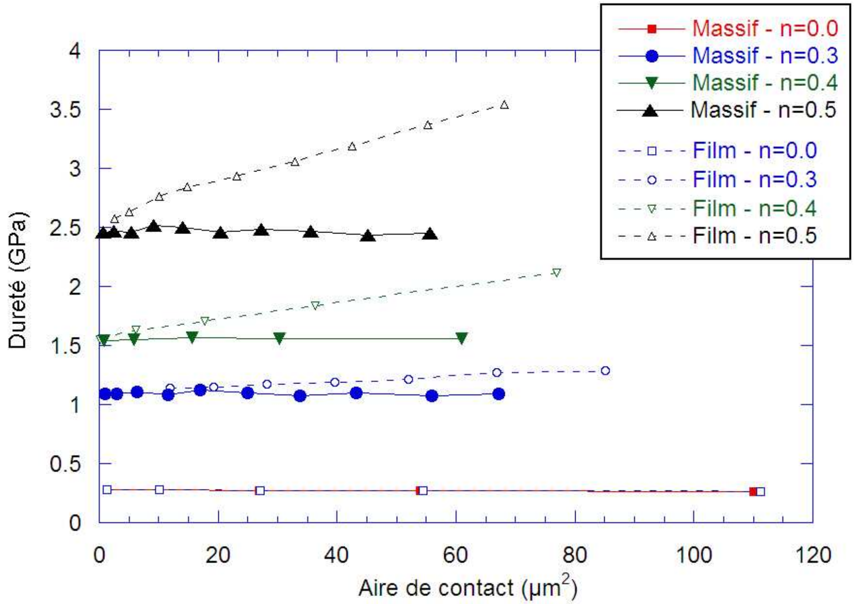


[Thèse S.VuHoang]

Film on substrate

[Thèse S.VuHoang]

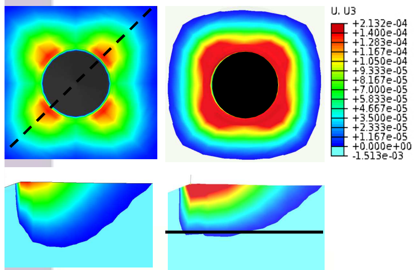


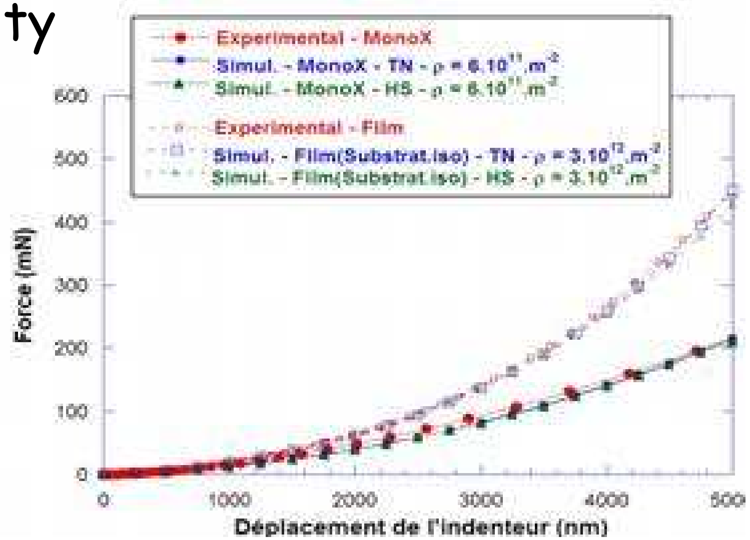


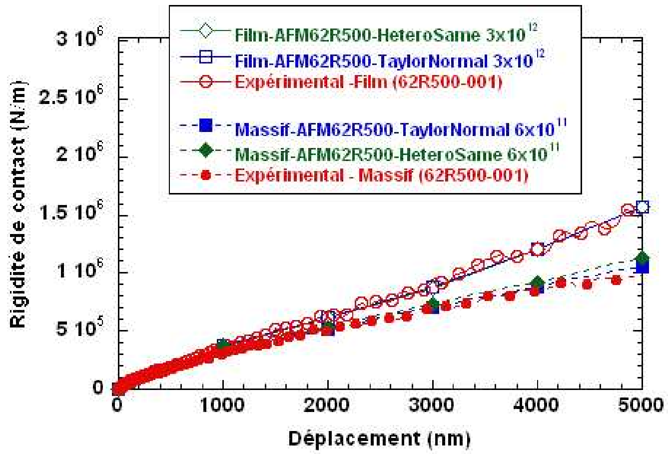
Introduction of length scale (film thickness):

Hardness(depth) : a good probe for strain hardening !

Crystal plasticity



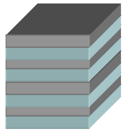




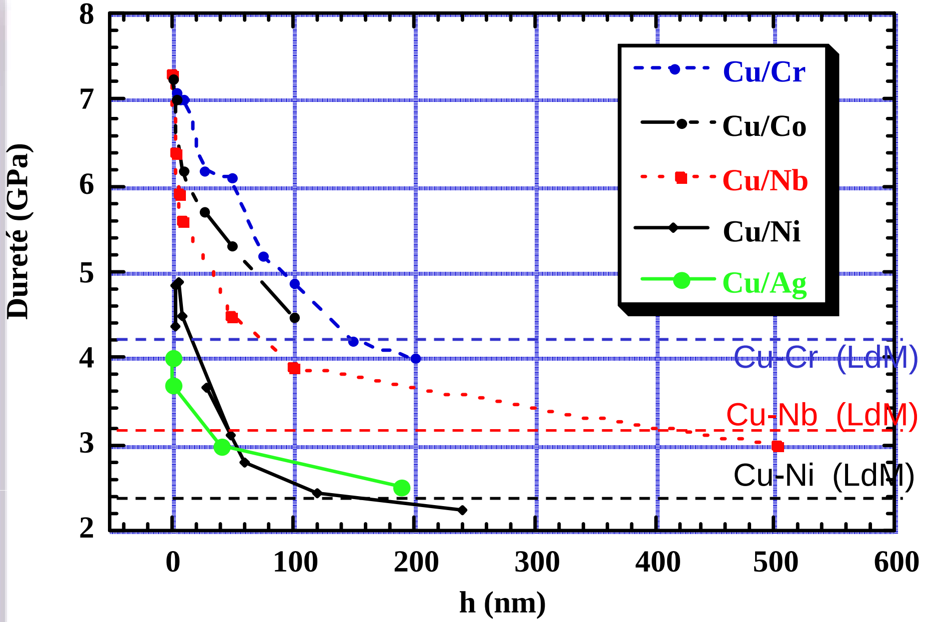
⇒Good quantitative aggreement with experiments (tested up to 50% film thickness),

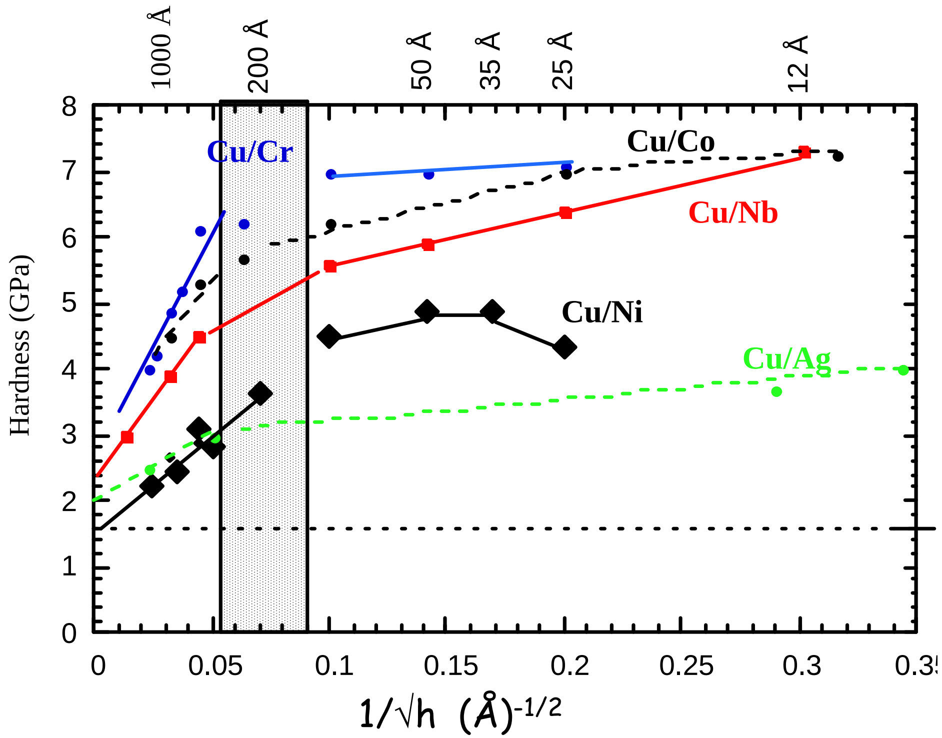
No strain gradient formulation, Load(displacement) AND S(displacement) $2.E^*.r_c$

Microstructure:
Multilayers



Mechanical strength



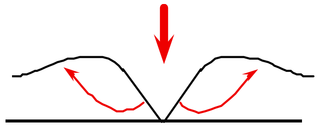


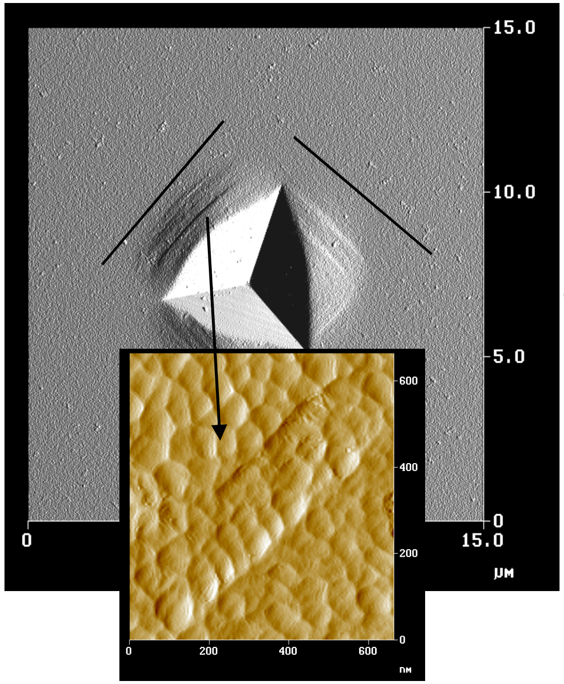
Système :	Structure cristall.	Misfit / relation d'orientation ou texture mesurée	μ_X / μ_{Cu}
Cu / Cr	cfc / cc	2.3 % {111}cfc // {110}cc	2.4
Cu / Co	cfc / hc	2.5 % <111> texture	1.5
Cu / Nb	cfc / cc	10.5 % {111}cfc // {110}cc et <110>cfc//<111>cc	0.8
Cu / Ni	cfc / cfc	2.5 % Epitaxy Cube/Cube	1.5
Cu / Ag	cfc / cfc	10.2 % <111> texture	0.56

[Misra et al. Scripta Mat. 1998; Verdier et al. Advanced Mat. 2001]

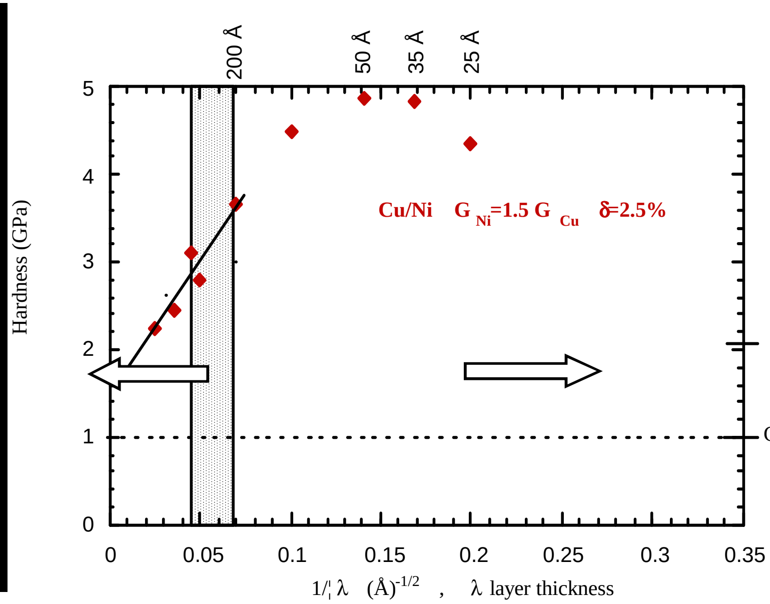
Deformation mechanism

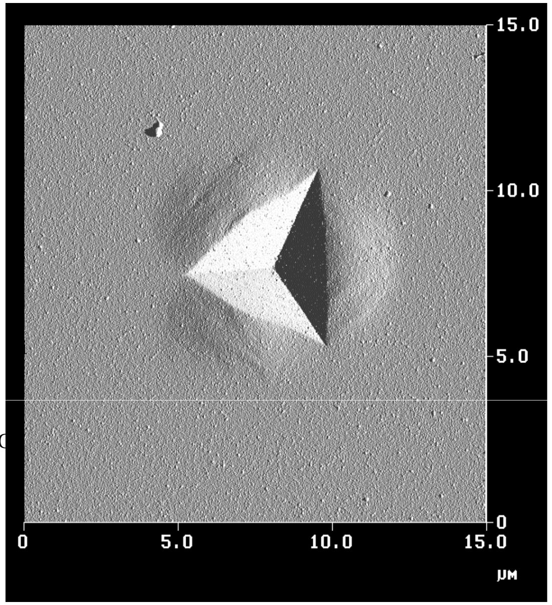
Cu/Ni [001]





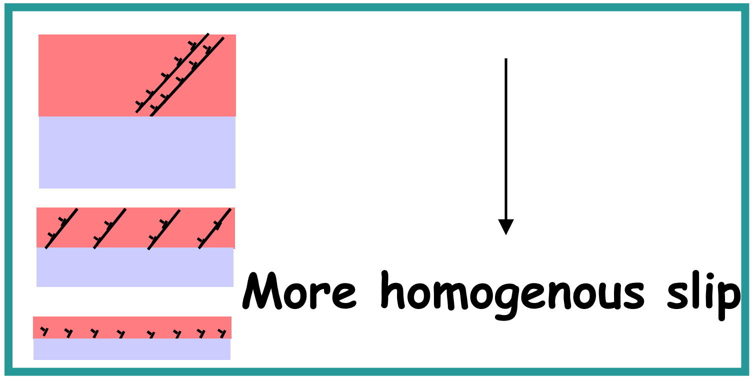
h= 50 nm





h= 2.5 nm

[M.Verdier et al, MRS v522, 1998]



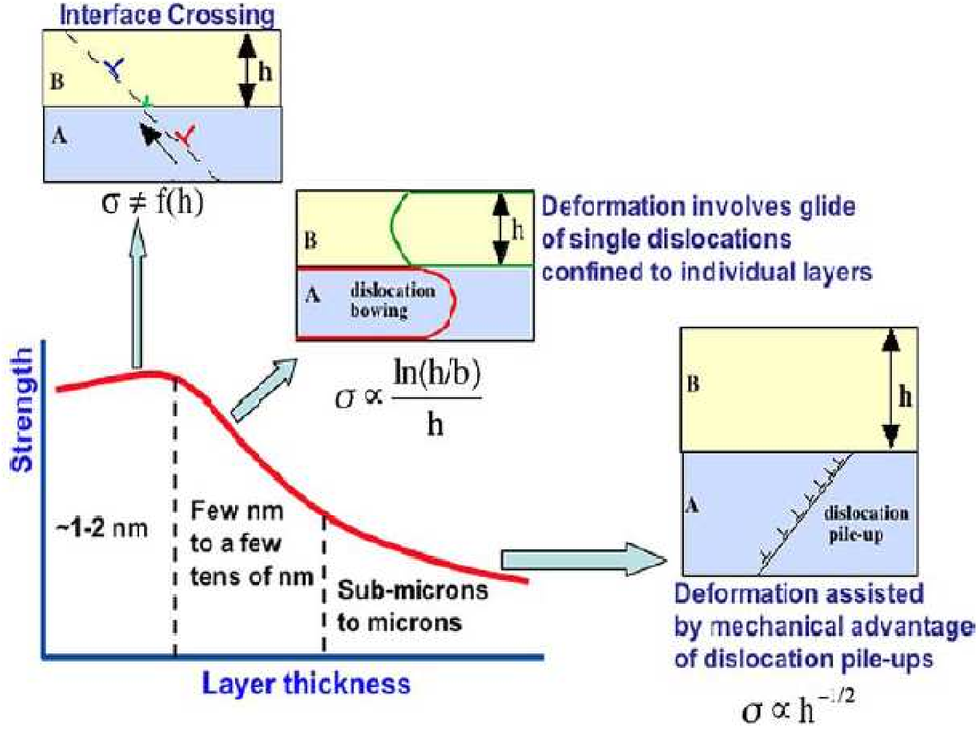
Current interpretation

'Bulk like'

phenomenological Hall-Petch slope k <-> interface strength τ^*

$\tau^* \approx (4\mu b/\pi(1-\nu))^{0.5}$

-Order of mag. of k relates to the max. strength obtained : not quantitatively

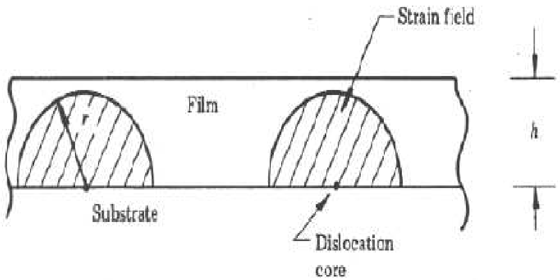


[Embury,Hirth, Acta Met Mat 42 (1994)] [Misra et al, Scripta 41,973 (1999)]

Single dislocation mech. / nature of interface

Coherent-semicoherent interface

Cu/Ni　　　　Cu/Ag



-coherency stress :

provides confinement

cancels image force (non lin. elast. in Cu/Ni).

-semi-coherent : planes between misfit core is still a strong barrier (coherent)

-if small misfit spacing: core spreading lowers interface barrier (Cu/Ag)

Incoherent interface

Cu/Nb

-Orowan stress : $\mu b/h \, Ln(\alpha h/b)$

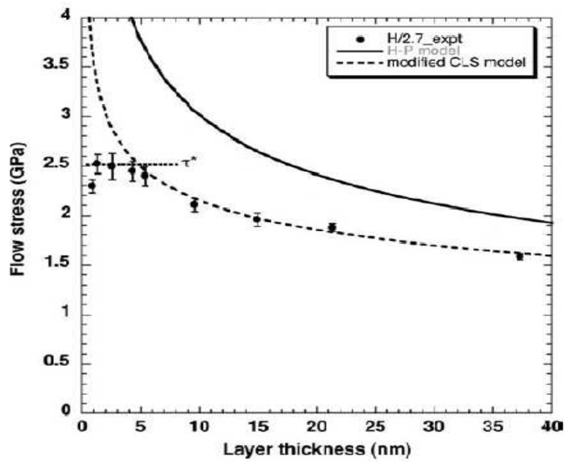
-> α: core spreading at interface (0.2) (from MD)

-Interface stress : **- f/h**

$f=2.9 \, J/m^2$

-Misfit / dislocation-dislocation interaction,

Spacing of loops: λ $+C/\lambda$



From simple modelling and extensive EAM studies:

[Embury,Hirth, Acta Met Mat 42 (1994)] [Misra et al, Scripta 41, 973 (1999)] [Misra et al, Acta Mat. 53,4817 (2005

[Rao,Hazzledine, Phil.Mag 80,2011 (2000)], [Hoagland et al, Phil.Mag 82,643 (2002)]

Large strain

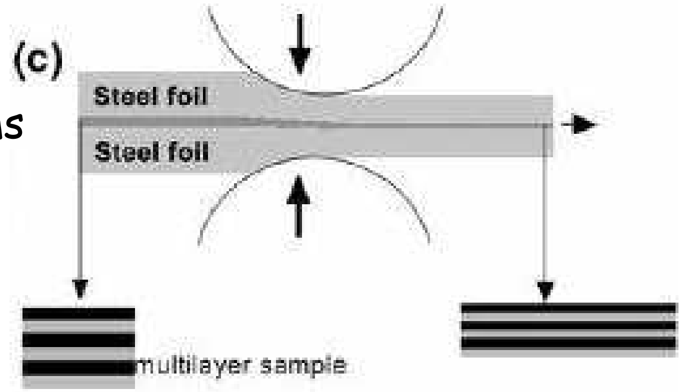
- Evaluation of work hardening at 30 nm length scale (Cu/Nb)

15μm films

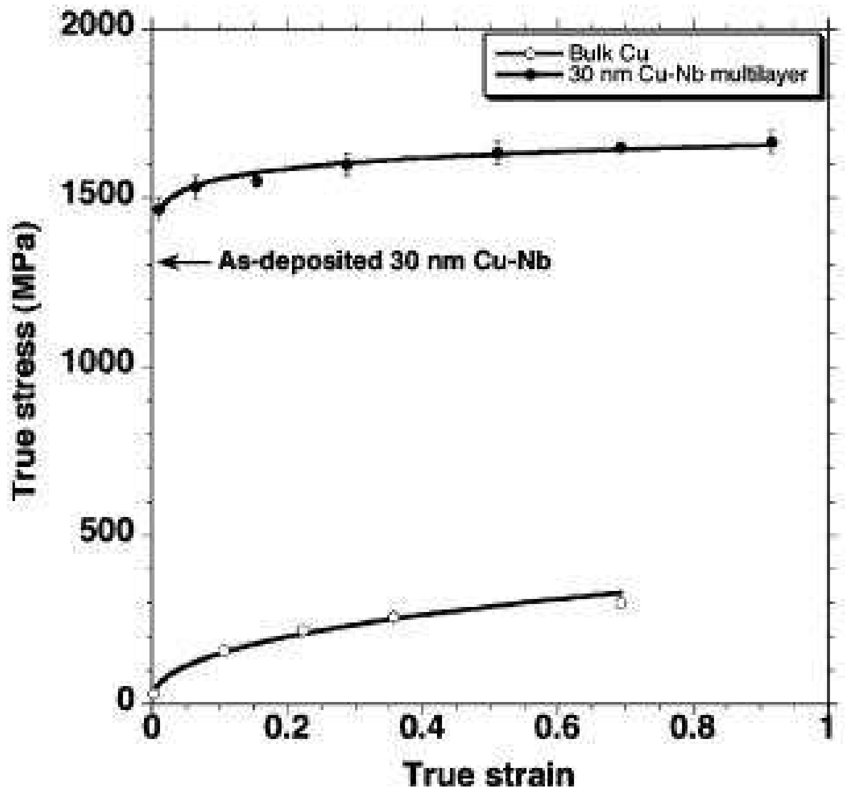
32nm

...

75nm



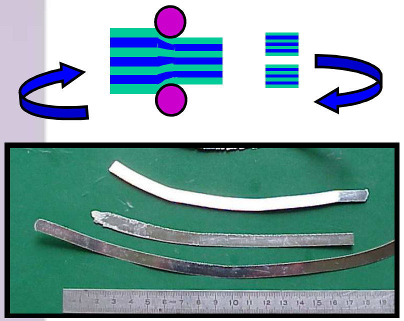
30nm

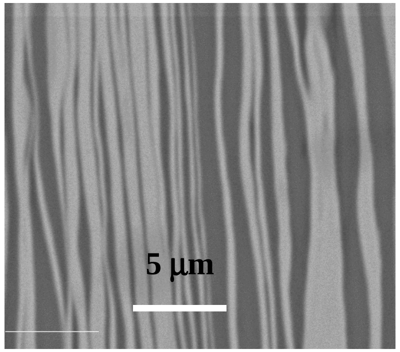


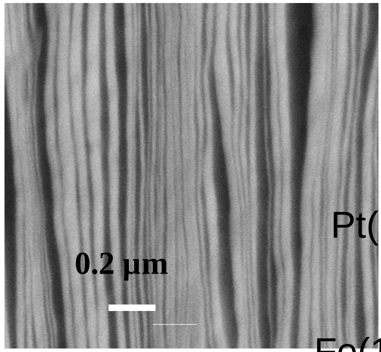
[Misra et al Acta 53 (2005)]

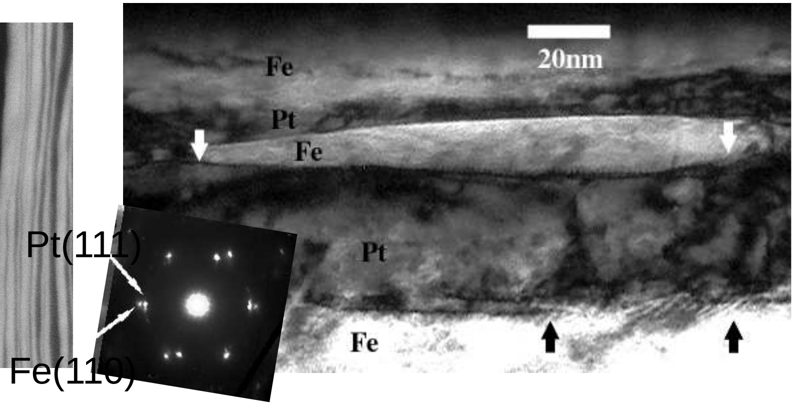
Large strain ex: Fe/Pt

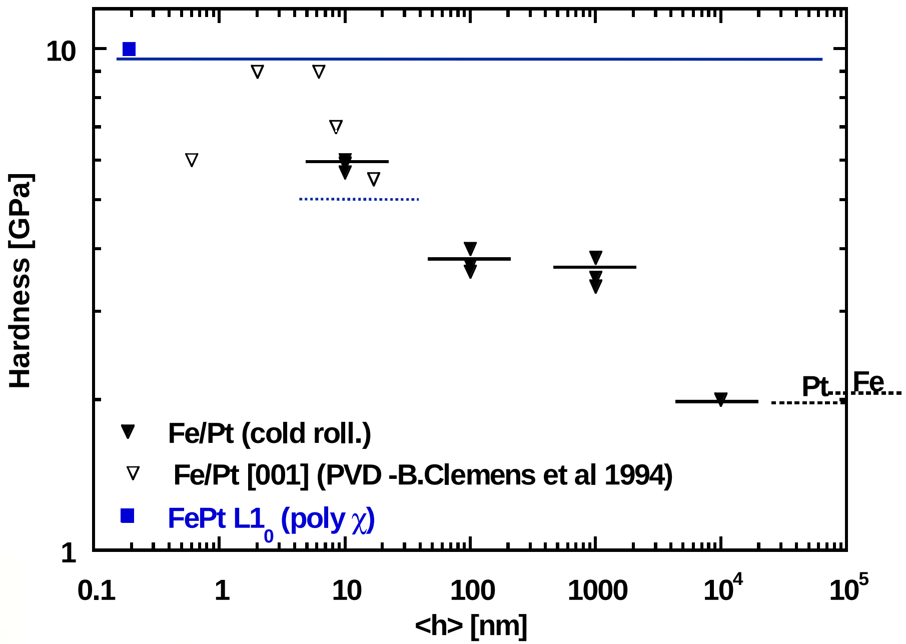
Plastic codeformation –sheath cold rolling : layer reduction down to a few nm





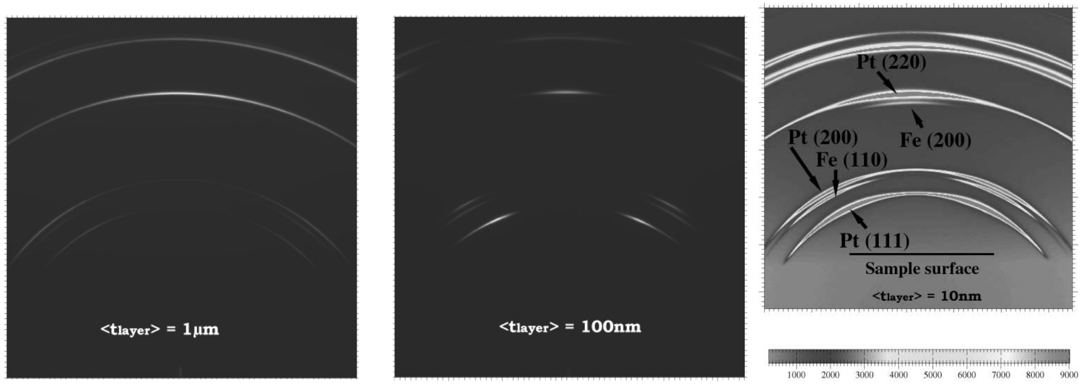






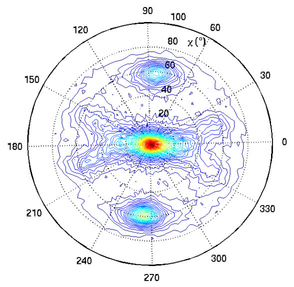
Signature of interface plasticity

(<1µm) : plasticity confined at interfaces

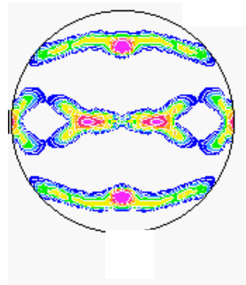


1µm 100nm Log(I)-100nm

Pole figure (011)
Pt – experimental

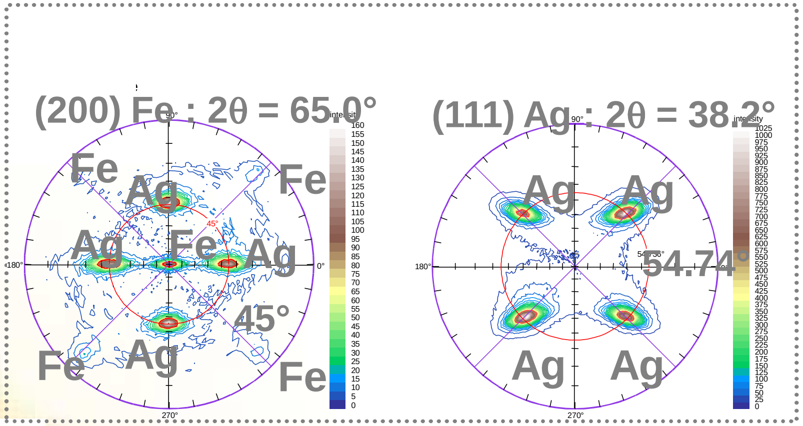


cold rolled fcc

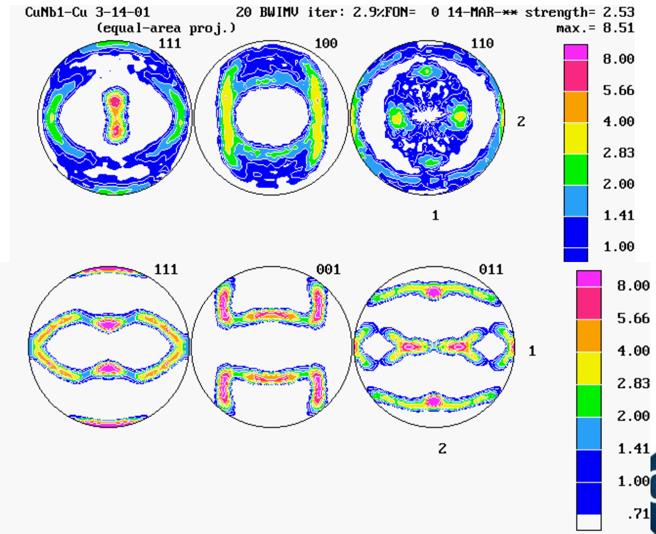


[Hai et al. JMMA (2003), Verdier et al PhilMag (2005)]

Fe(100)[011]//Ag(100)[001] (Cube/Cube)



Cu/Nb: (KS)



In summary

Small strain measurements:

- Large extent of micro-deformation with decreasing size, elasto-plastic behavior
- Microplasticity obviously depends on #source, stress activation is still (1/h), boundary cond
- Conventional 0.2% has no physical meaning, 1 dislocation @200nm =0.1% => b/h
- Hall Petch-like behavior stems from very large initial strain hardening

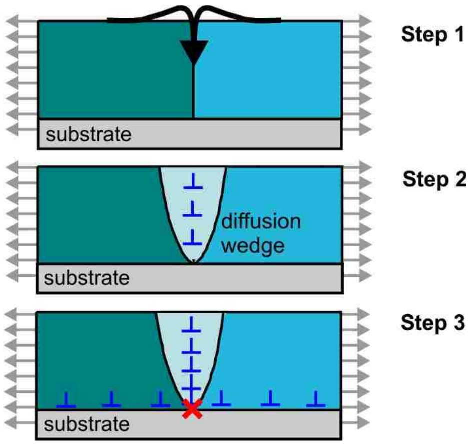
Large strain characteristics:

- small hardening (as compared to bulk)
- Texture refinement for confinment <1µm ,
- unsual/unpredicted orientations (case/case)

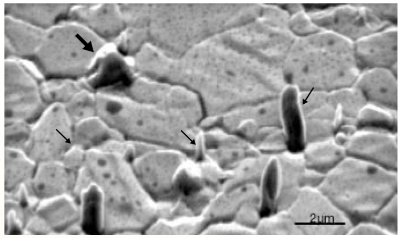
Left over : **in situ techniques**

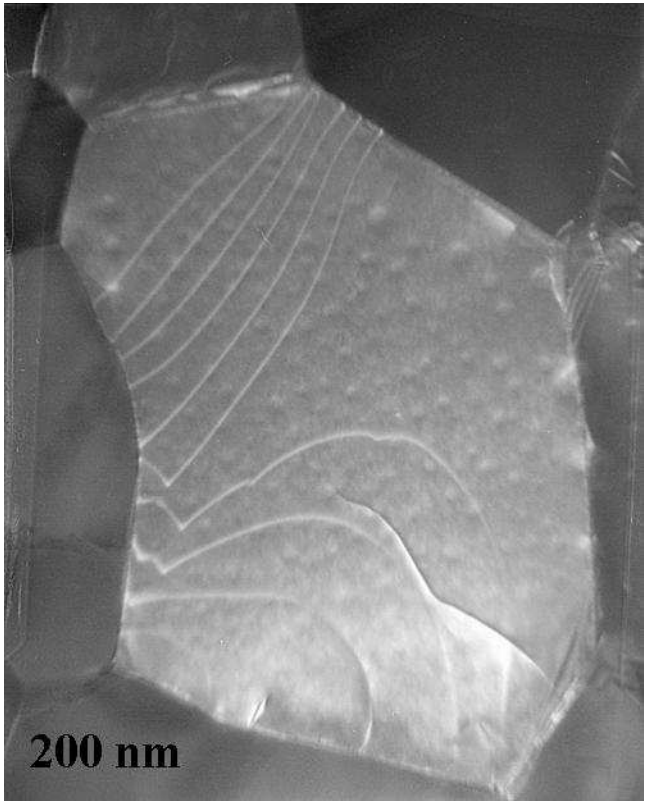
TEM:

- A.Minor/Berkeley : nanopillars
- M.Legros (CEMES): polycristalline thin films, compressive stress relaxation by diffusion at g.b., // dislocation = transport



F.Gao model

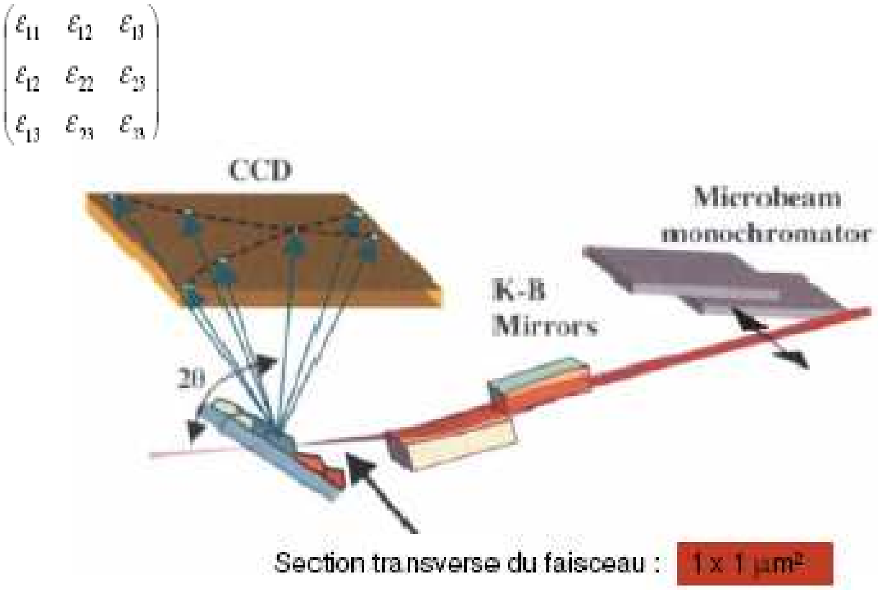




200 nm Cu films

Balk et al Acta Mat 2003

Synchrotron facilities:



- µLaue (white beam): H. Van Swygenhoven (SLS PSI): tensile/ micropillar

- 3D full strain tensor determination: (N.Tamura ALS) µLaue-Monochromatic beam

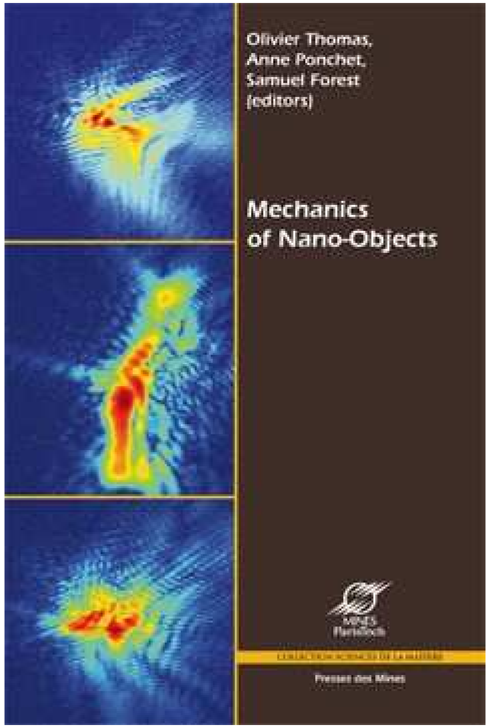
ESRF:

- O.Robach (interconnect Cu lines)
- S.Labat (IM2NP) fcc polycrystalline films / thermal stress

Related references:

-GDR-3180(2009-2012) Mécano -> GDRi MECANO

-Réunion 30-31/10/2012, Mines de Paris



-**Thin films materials science: stress, defect formation and surface evolution**, B.Freund, S.Suresh, Cambridge Univ. Press

-W.Nix Stanford lectures 'Thin film mechanical properties'

Challenges

-Plasticity at interfaces : a case by case problem

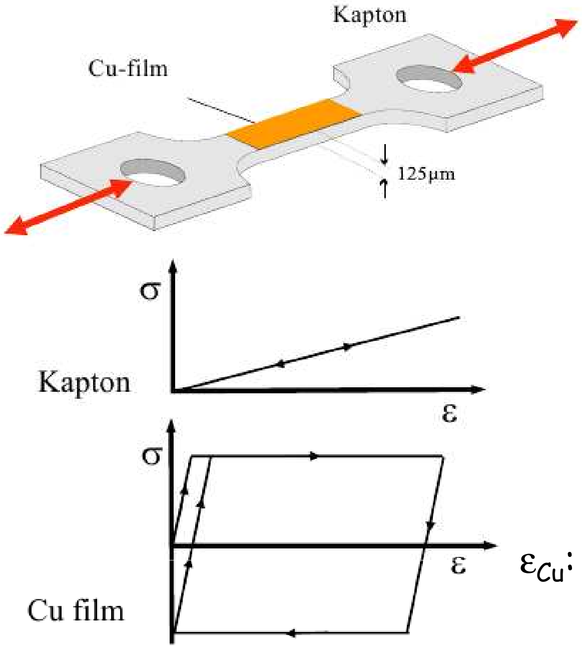
- Measurements in T: creep, strain rate sensitivity, …

-Initial microstructure: defects ? => SYNCHROTRON (non destructive)

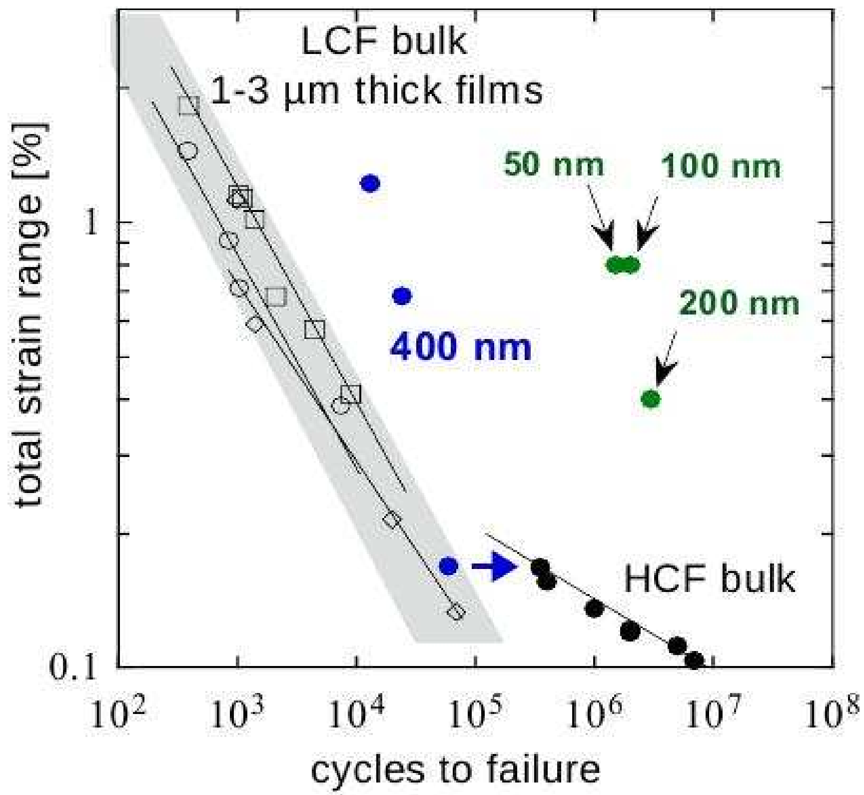
- Fatigue ?

- Non-crystalline (amorphous) metallic systems ?

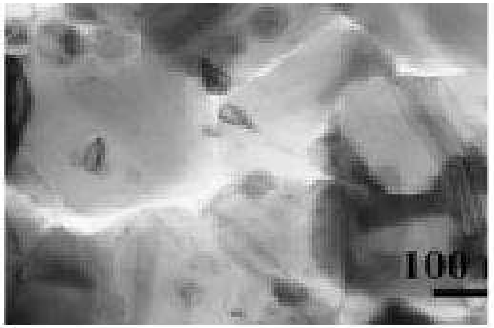
Fatigue [O.Kraft KIT]

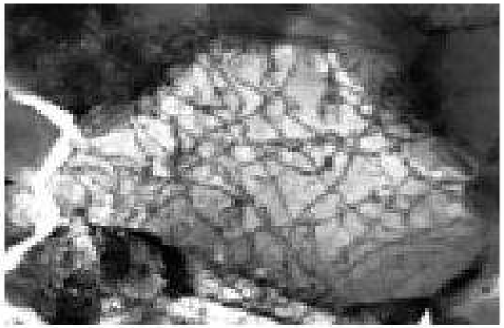


ε_{Cu}: X-Ray



Zhang, Volkert, Schwaiger, Arzt, Kraft, JMR 2005

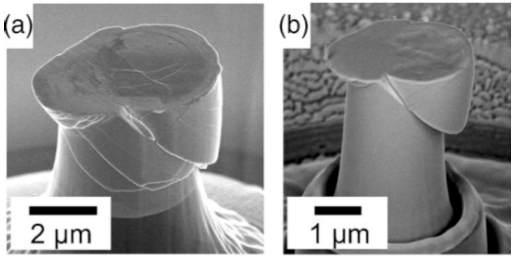




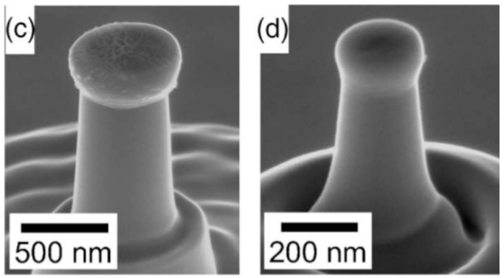
Voids condens. 100 nm individual 1 µm collective

Bulk metallic glass

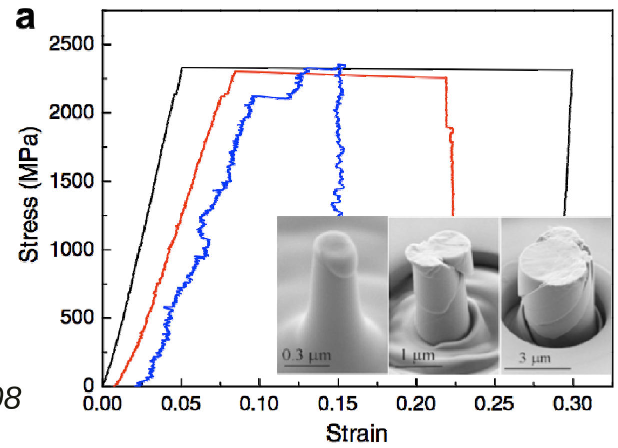
$Pd_{77}Si_{23}$ sputtering

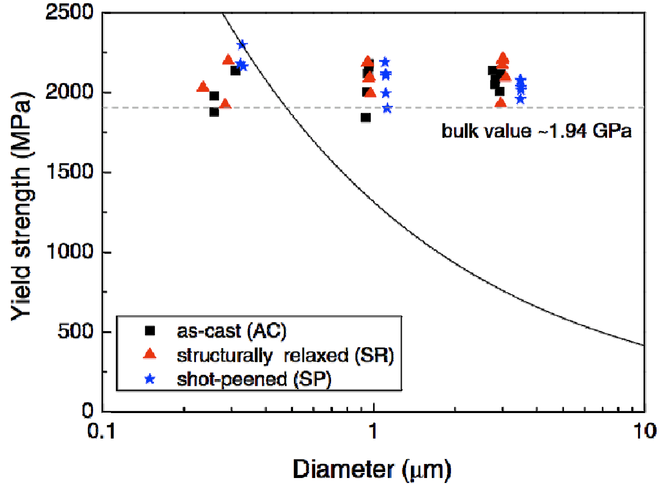


Volkert Donohue Spaepen, J. Appl. Phys., 2008



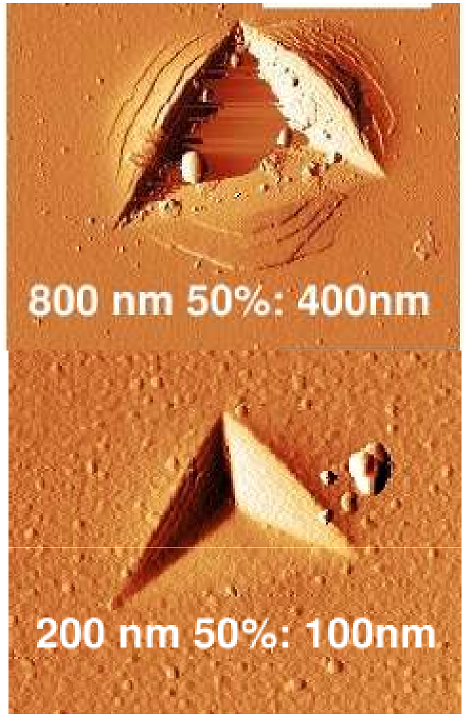
$Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$

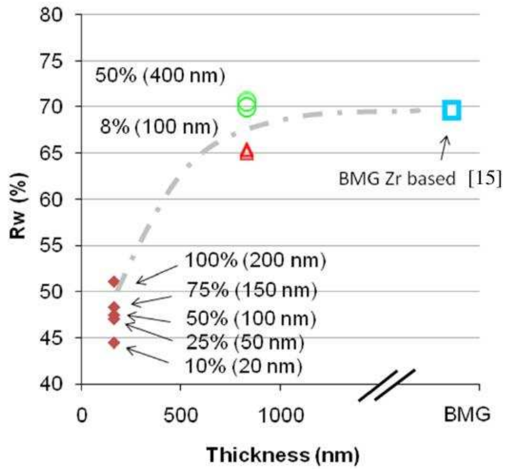




Dubacha, Raghavan, Löffler, Michler, Ramamurty, Scripta Materialia 2009

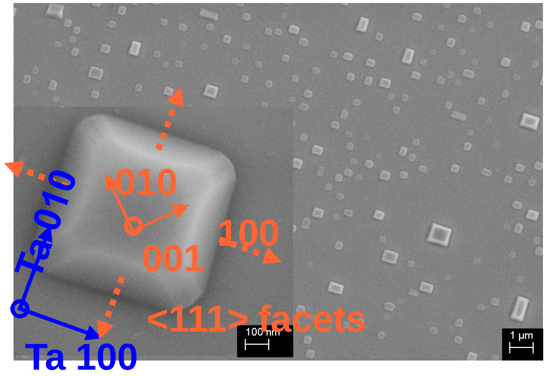
ZrNi/Si thin films





[Phd A.Volland]

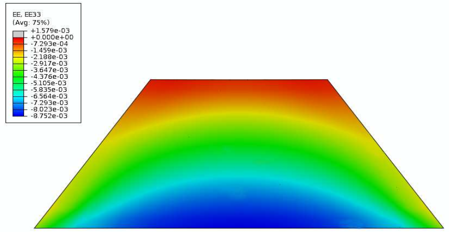
Diffraction of coherent XRay (ANR Blanche Mécanix)



Ability to recover u(r) from phase and amplitude of diffraction

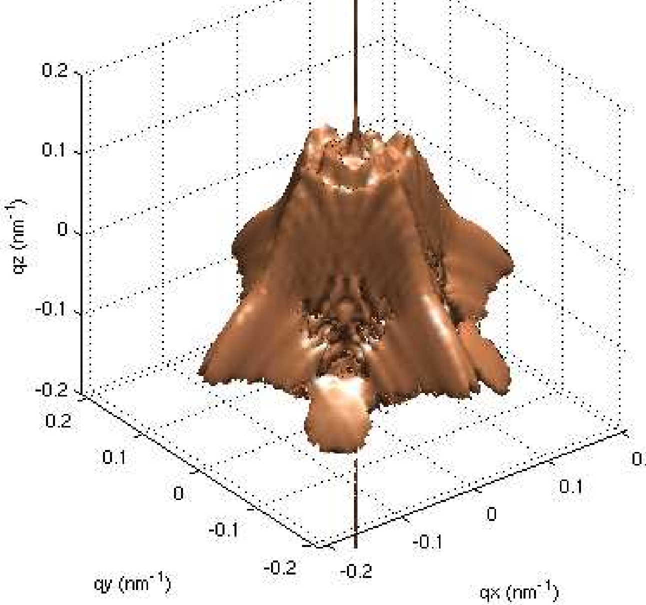
Signature of dislocations : phase shift => speckle !

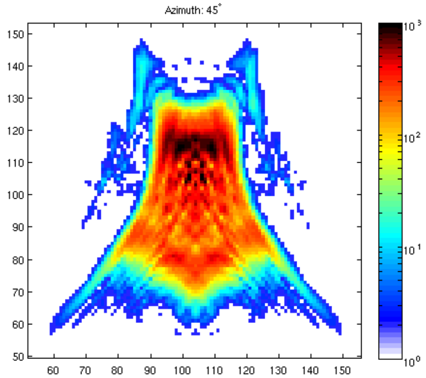
Simulation élastique anisotrope Cu/Ta :
déformation 0.6% du cristallite



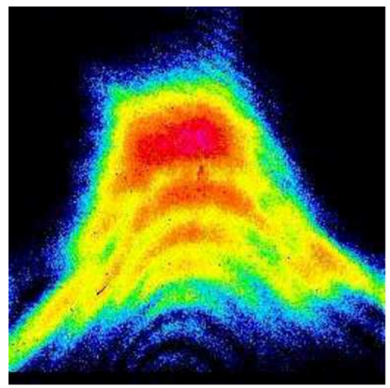
Coupe ε_{33}

Bragg simulé (Pynx GPU [V.Favre-Nicolin])





Coupe simulée

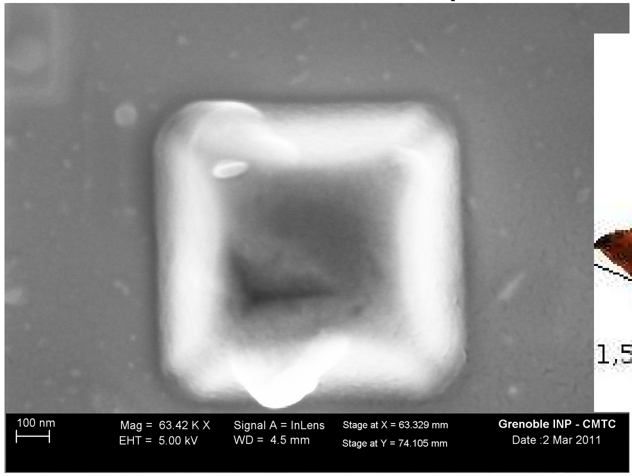


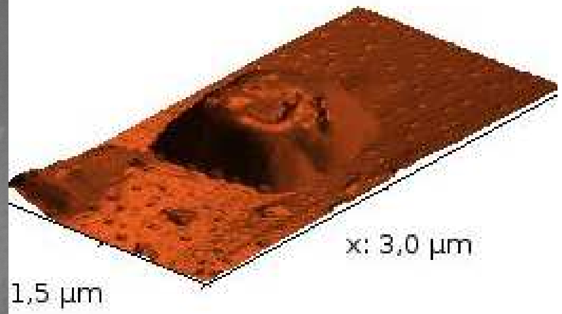
Coupe expérimentale

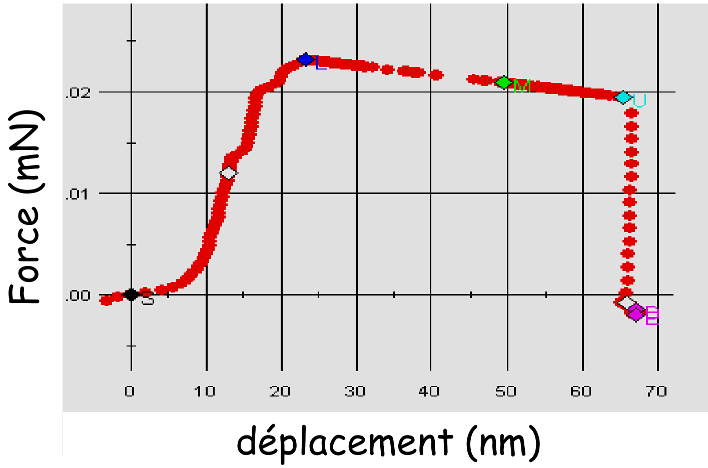
[Beutier et al. Thin Sol. Films 2012]

Exemple de déformation plastique d'un cristallite Cu [001]

(990x910x325 nm) pré- indenté (30nm)

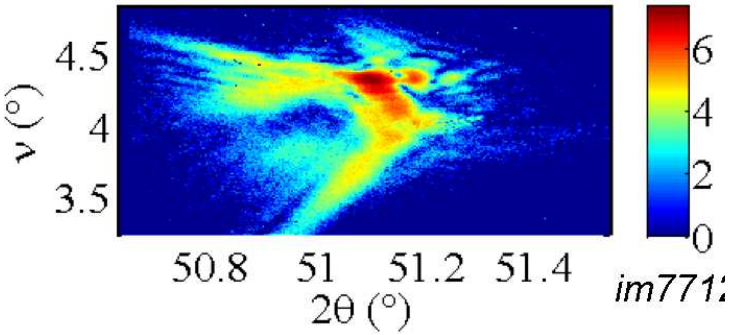




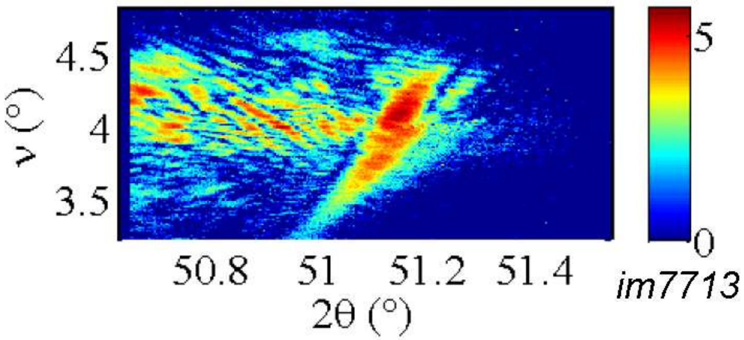


nanoindentation in-situ d'un plot

Coupe CCD avant



après



[Beutier et al. Jal of Physics: Conf. series 2012]