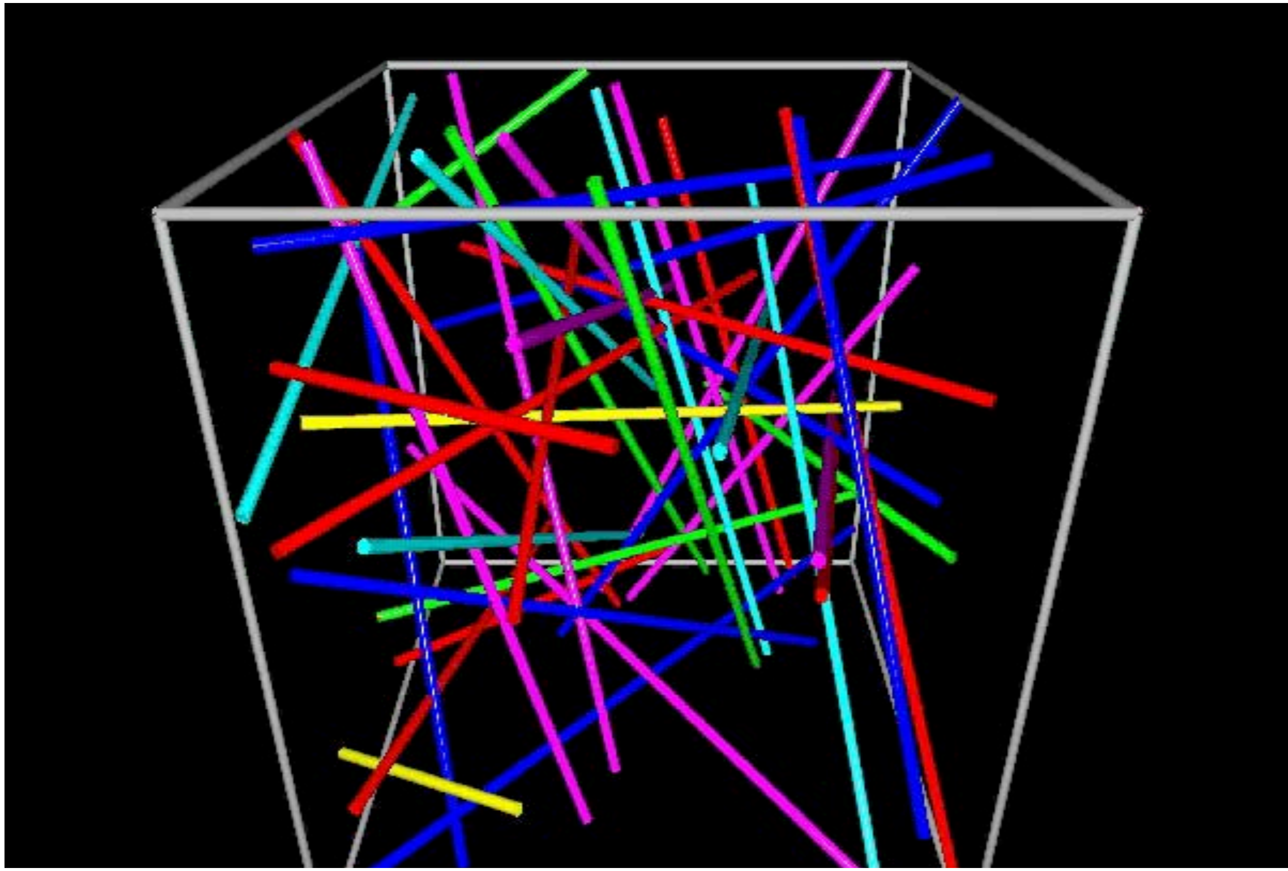
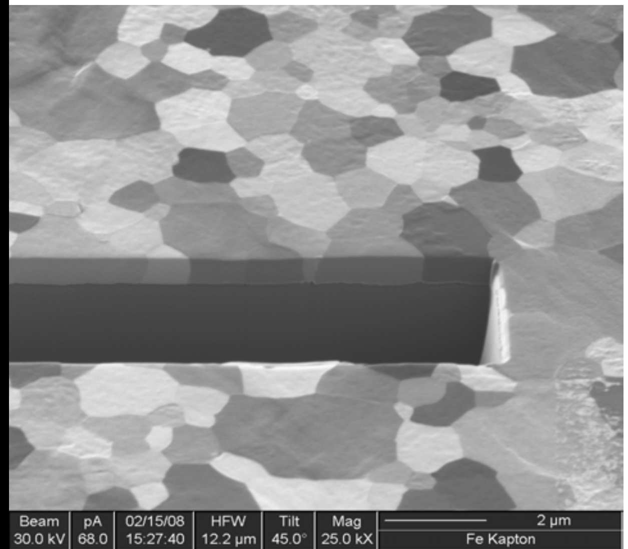


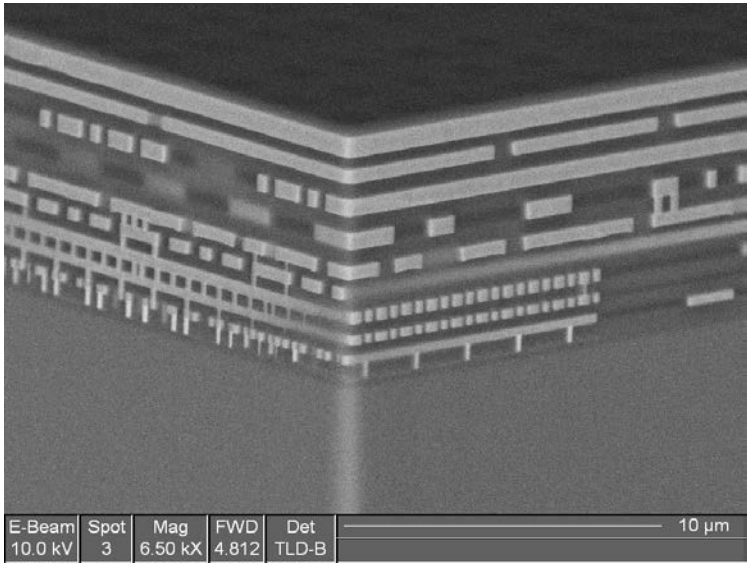


NANO/BIO-MATERIALIEN: MIKRO/NANOMETALLE

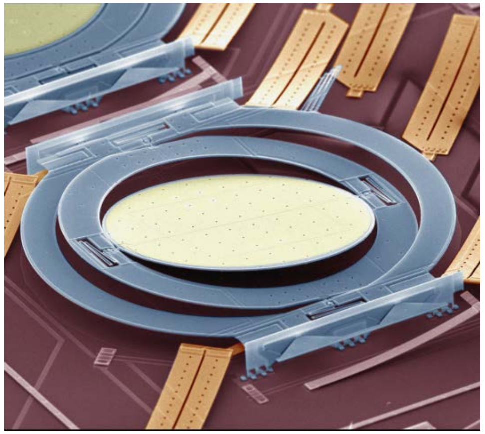
Dr. Karsten Moh und Prof. Eduard Arzt

WS 2018

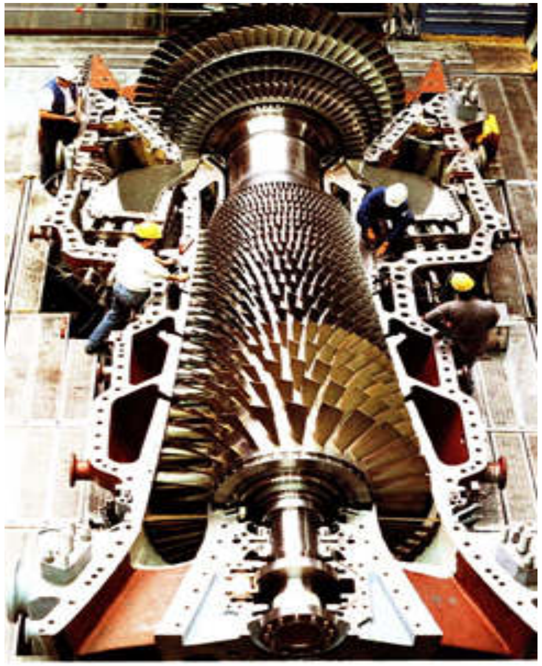
METALLIC THIN FILMS



integrated circuits, data storage



micro-electro-mechanical systems (MEMS)



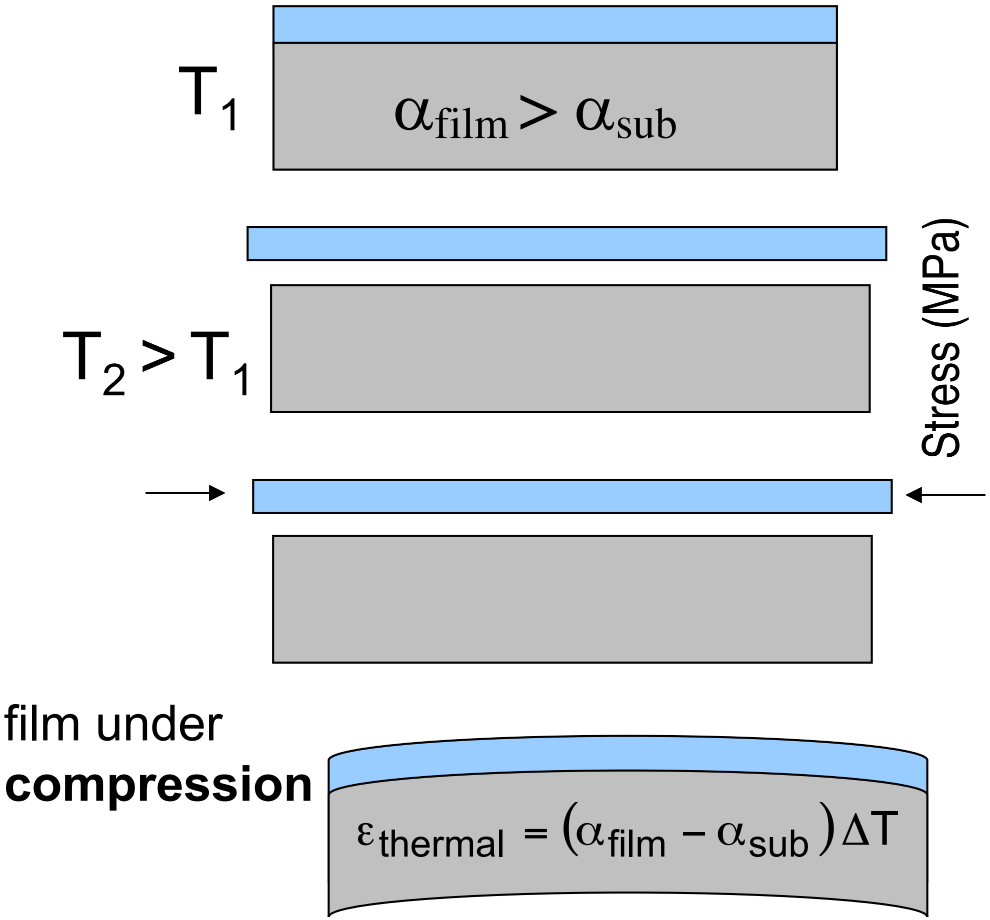
wear or corrosion protection,
thermal barriers

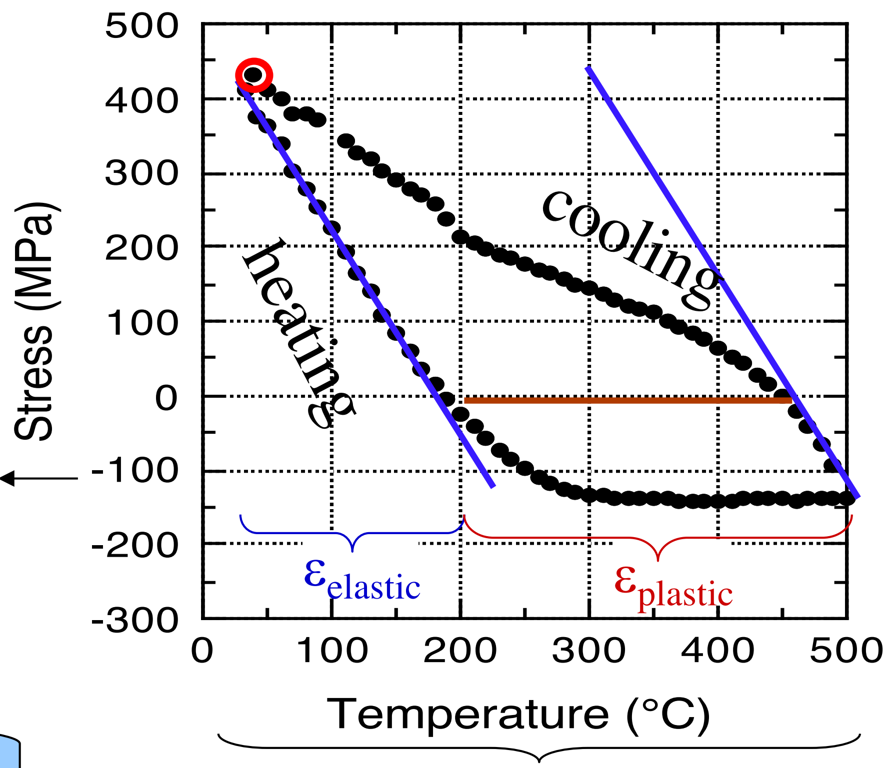
- in SAW filters (cellular phones)
- metallization in semiconductor devices
  and on polymer substrates
- delamination and AC induced
  damage by Joule heating

→ micromechanics important!

STRESSES IN THIN FILMS

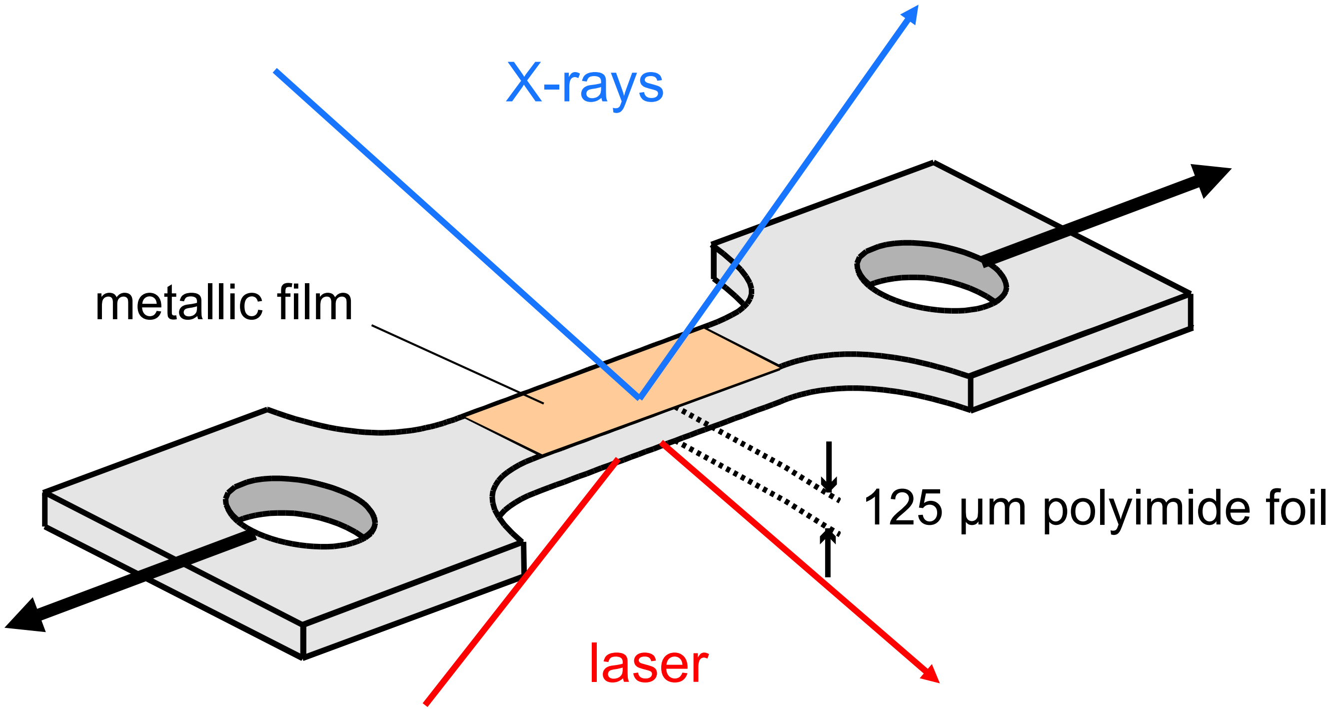
Stresses are always present in thin films. They can limit the reliability of systems.
Stresses can be measured by optical, electrical or X-ray techniques.





$$\varepsilon_{thermal} = \varepsilon_{elastic} + \varepsilon_{plastic}$$

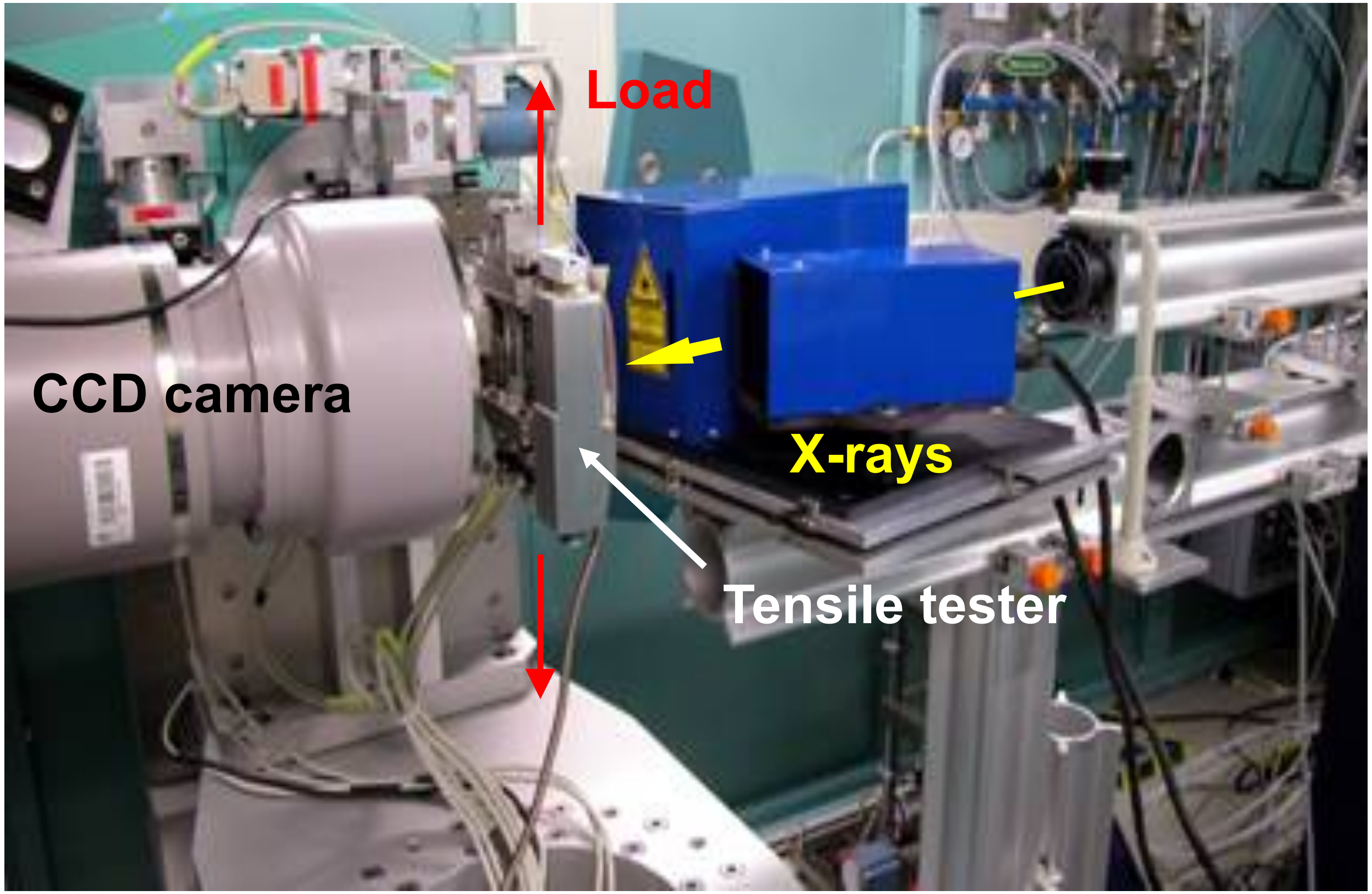
THIN FILMS: STUTTGART MICROTENSILE TEST



Elastic strain in a crystalline metallic film is determined by X-ray diffraction and compared to the total strain of the sample measured by a laser extensometer. Note that stress state is biaxial (Poisson contraction).

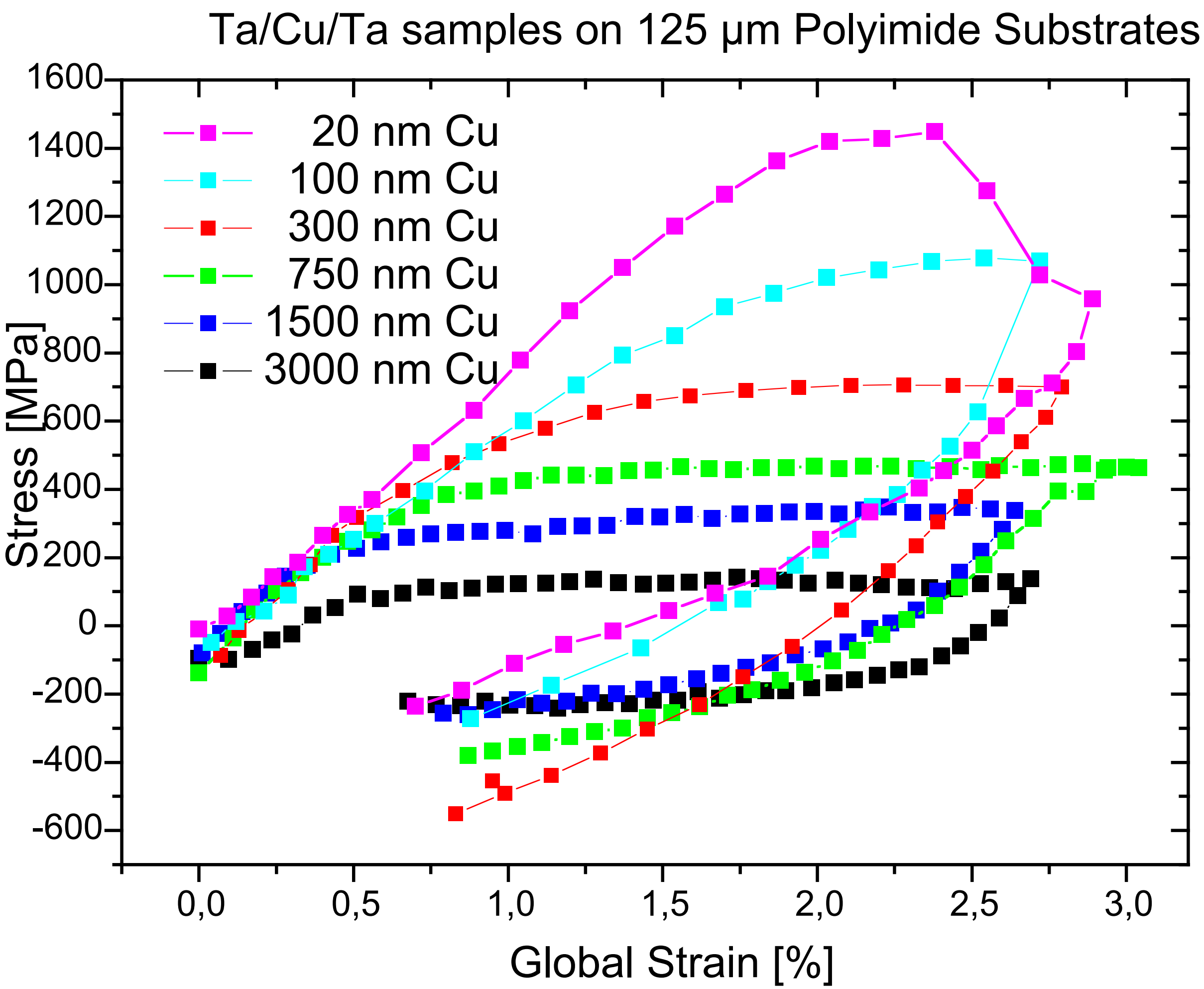
Baker, Kretschmann, Hommel, Kraft, MPI Metallforschung

SYNCHROTRON-BASED TENSILE TESTING TECHNIQUE



J Boehm et al, Rev. Sci. Instr. **75,** 1110 (2004)
PA Gruber et al, Acta Mater **56,** 2318 (2008)

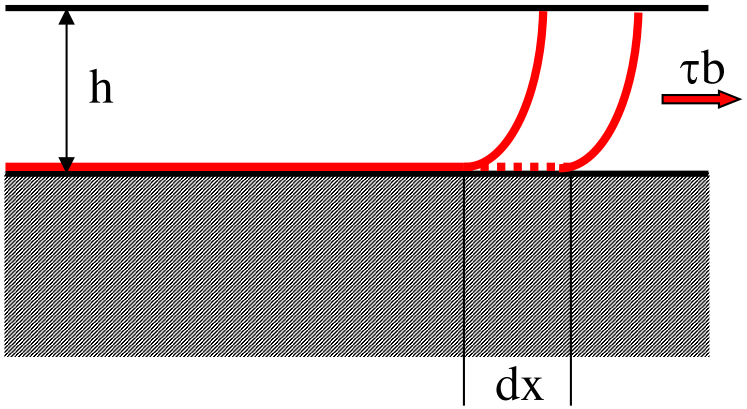
STRESS-STRAIN CURVES OF THIN CU FILMS



The yield stress increases strongly with decreasing film thickness.

MODELLING THIN FILM STRENGTH: NIX/THOMPSON

Thin film strength is modeled by considering dislocation advance constrained by the film.
As a result, the yield stress should scale inversely with film thickness.



$$\sigma_y \propto \frac{G_{eff} b}{4\pi(1-\nu)\, \mathbf{h}}$$

$$\text{with } G_{eff} = \frac{2GG_s}{G+G_s} \ln\left(\frac{\beta_s h}{b}\right)$$

W.D. Nix, Metall. Trans. A20, 2217, 1989

energy balance

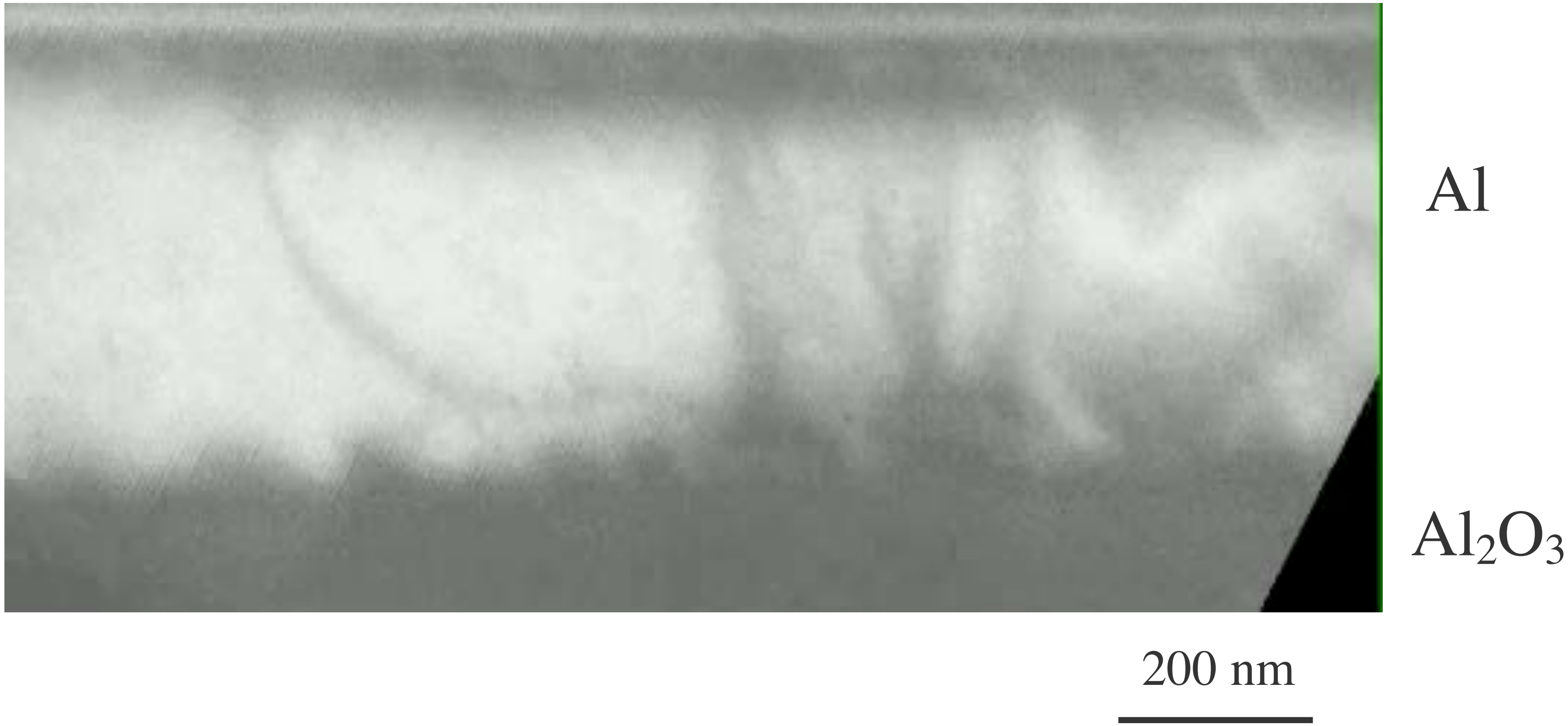
$$dW = \boxed{h}\,\tau b\, dx$$

$$dE = \frac{Gb^2}{4\pi} \ln\frac{R}{r}\, dx$$

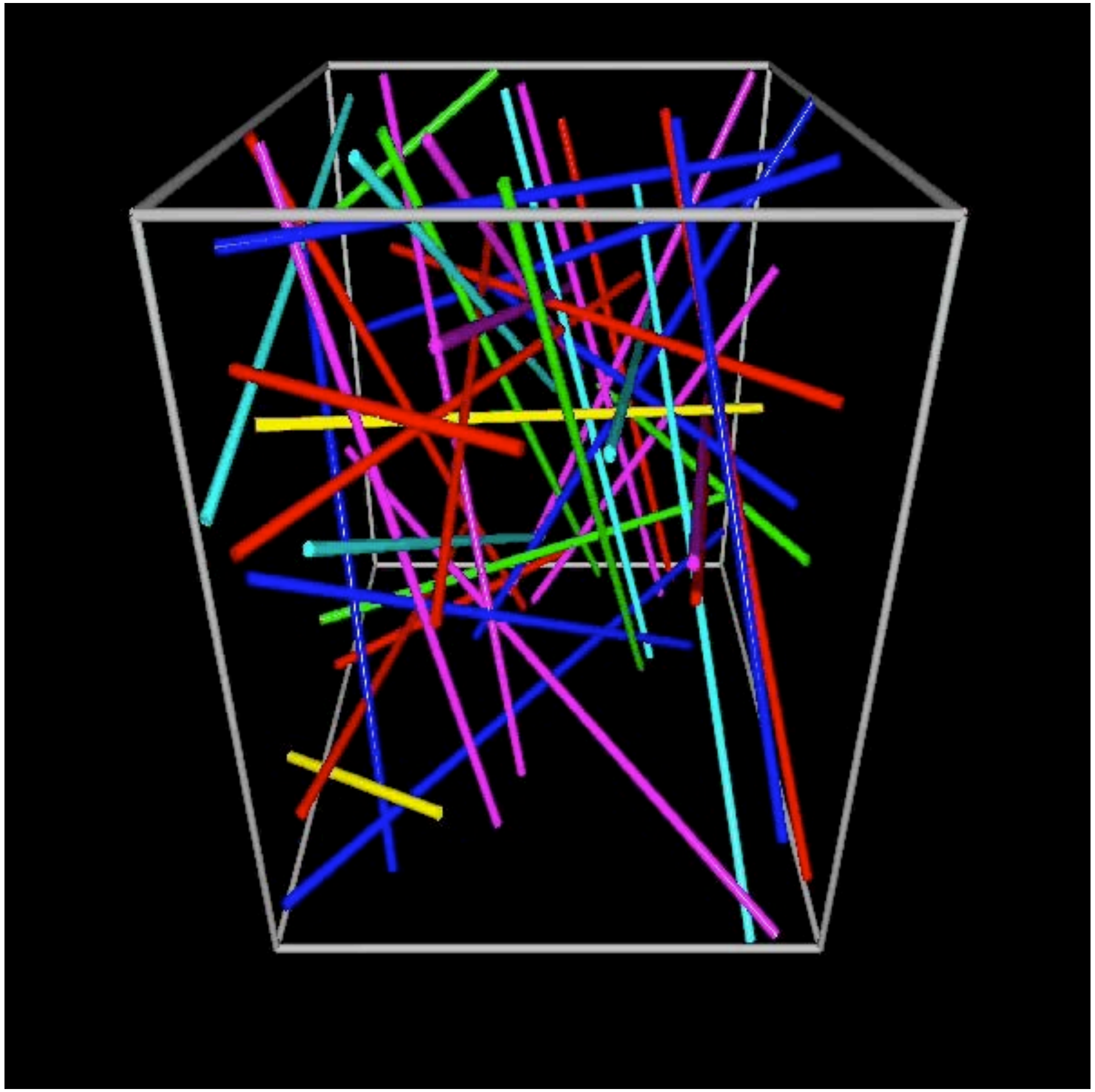
τ…shear stress
b…Burgers' vector
G…shear modulus
E…line energy

IN SITU TEM EXPERIMENT

Epitaxial Al film on (0001) α-Al_2O_3

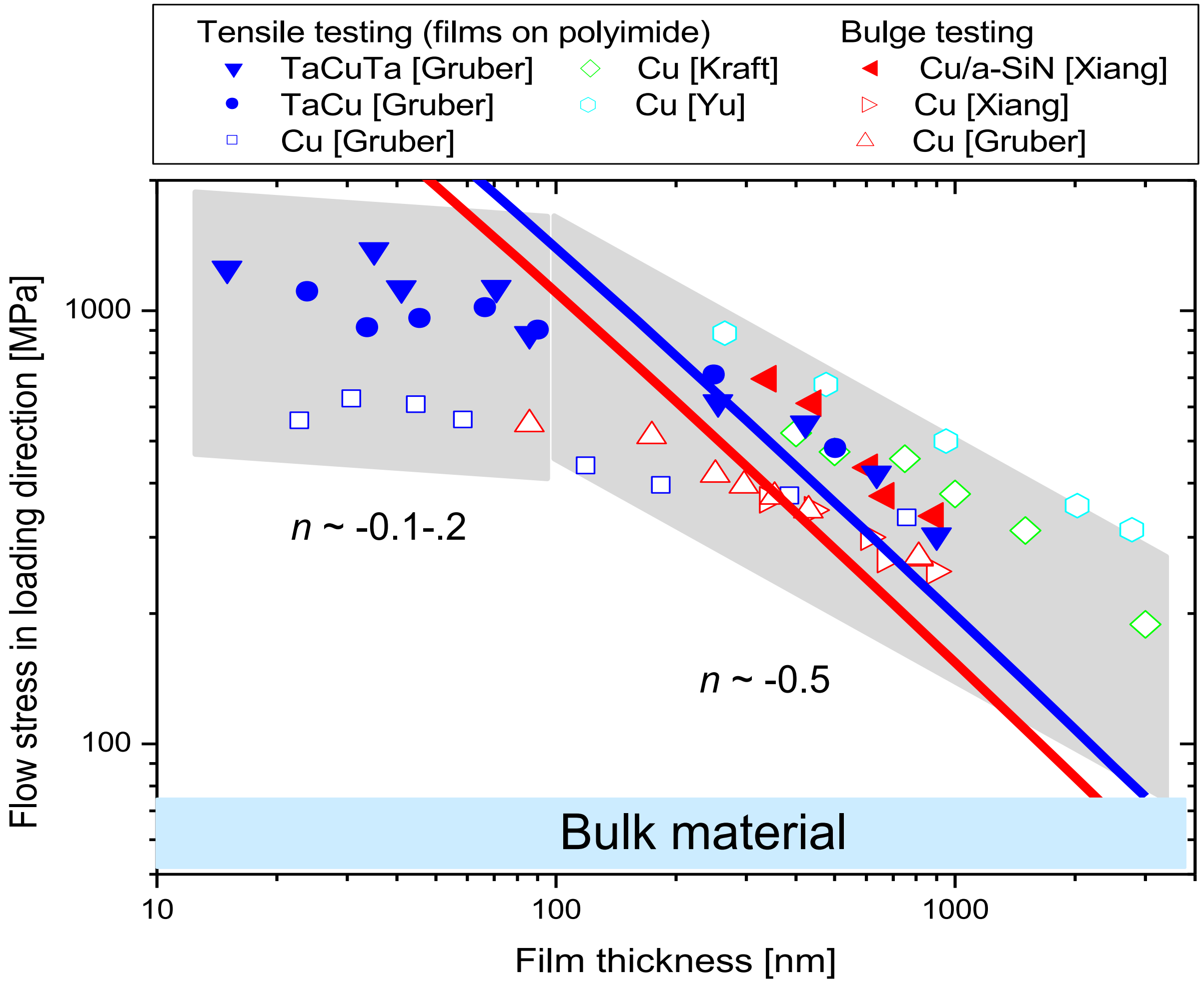


Dehm et al., MPI Metallforschung



Numerical dislocation modeling inside a cube by v. Blanckenhagen et al.

DATA COMPILATION FOR COPPER



tensile testing
bulge testing

Yu & Spaepen (2004)

Kraft et al. (2000)

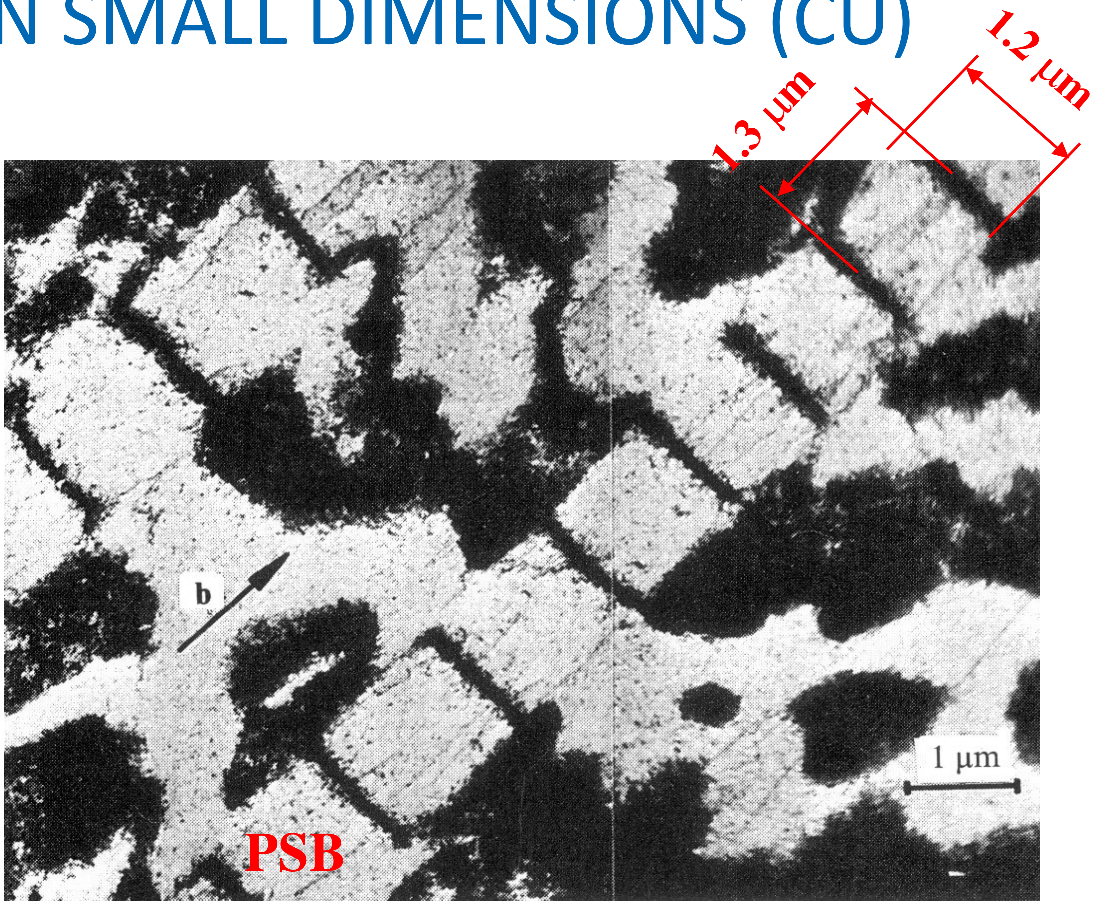
Xiang & Vlassak (2006)

open symbols: free surface
filled symbols: passivated

Nix/Thompson model:
underestimate
for h > 100 nm

O. Kraft et al., Ann. Rev. Mat. Sci. 40, 293 (2010)

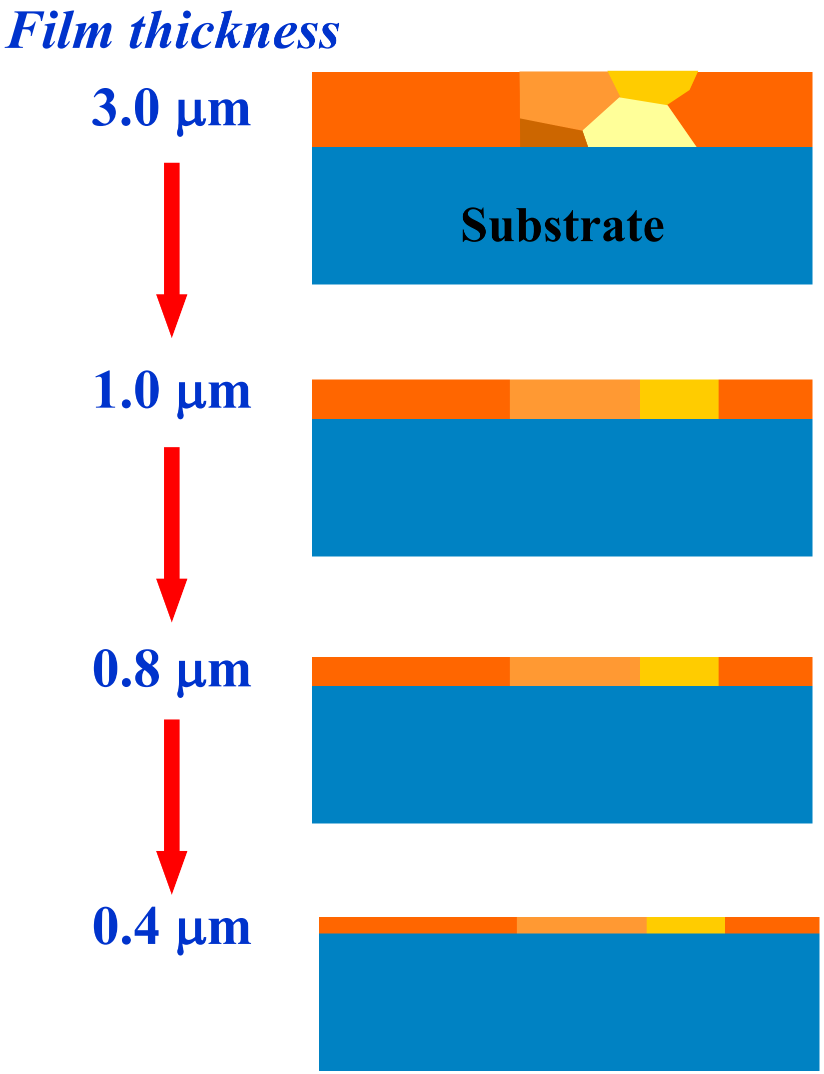
FATIGUE IN SMALL DIMENSIONS (CU)

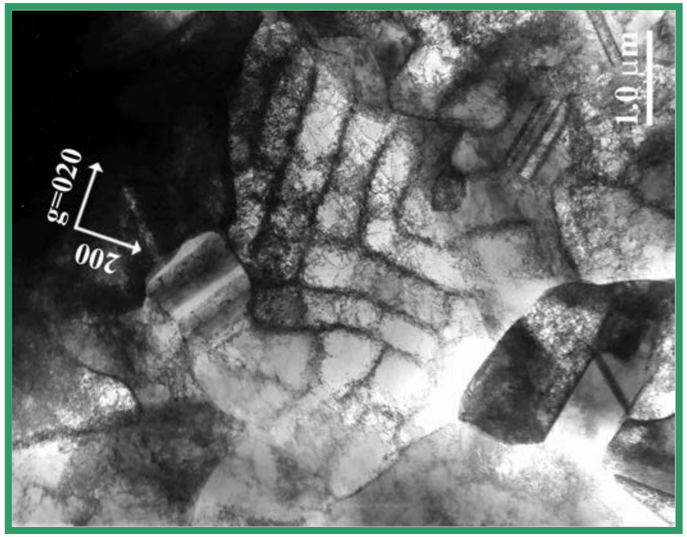


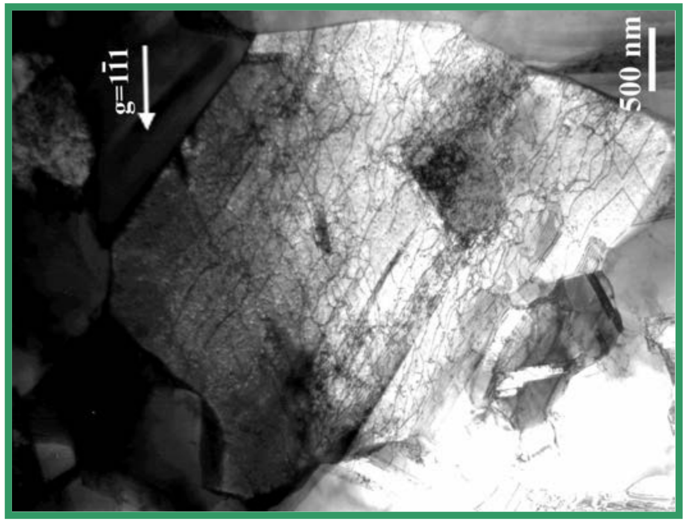
Mughrabi et al. 1979

During repeated loading, dislocations rearrange into soft regions (**PSB=persistent slip bands**). These regions may act as crack starters and lead to fatigue damage.

FATIGUE OF THIN CU FILMS: SIZE EFFECTS

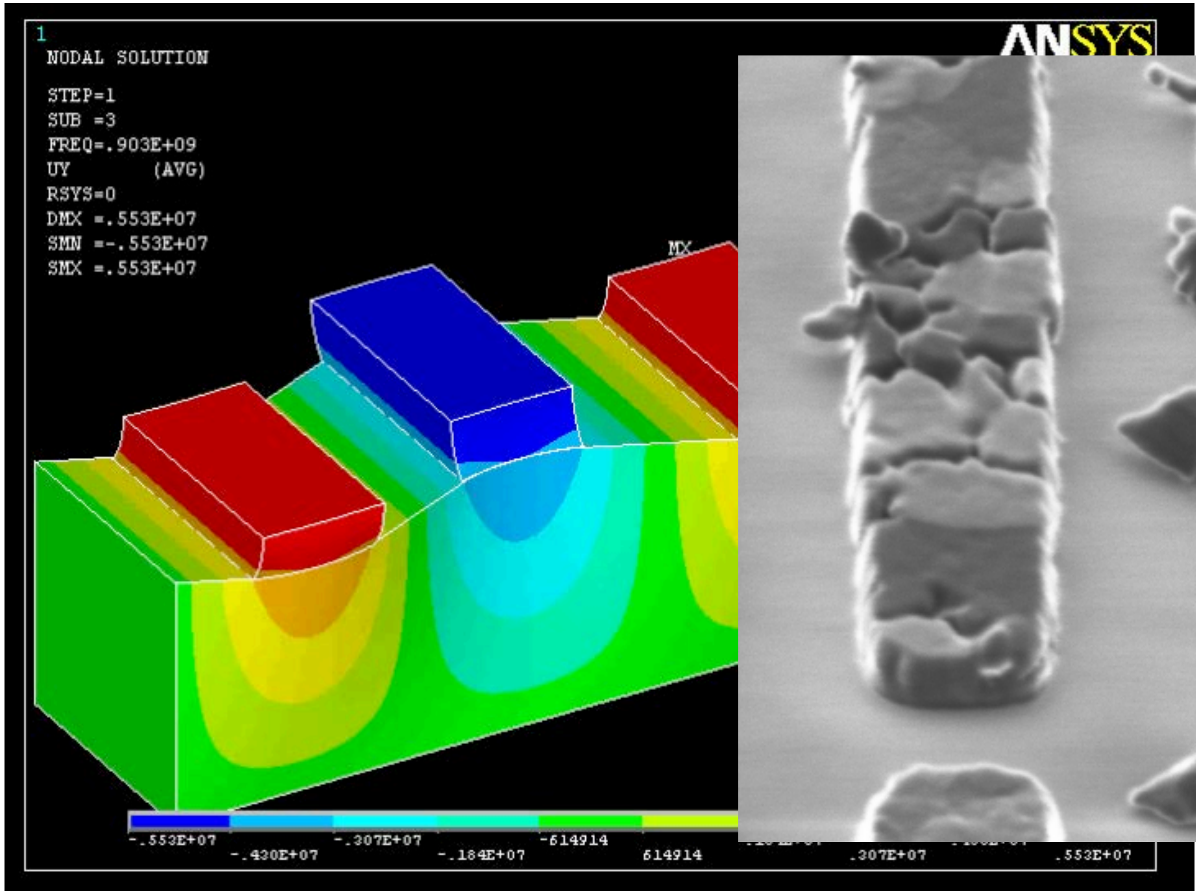


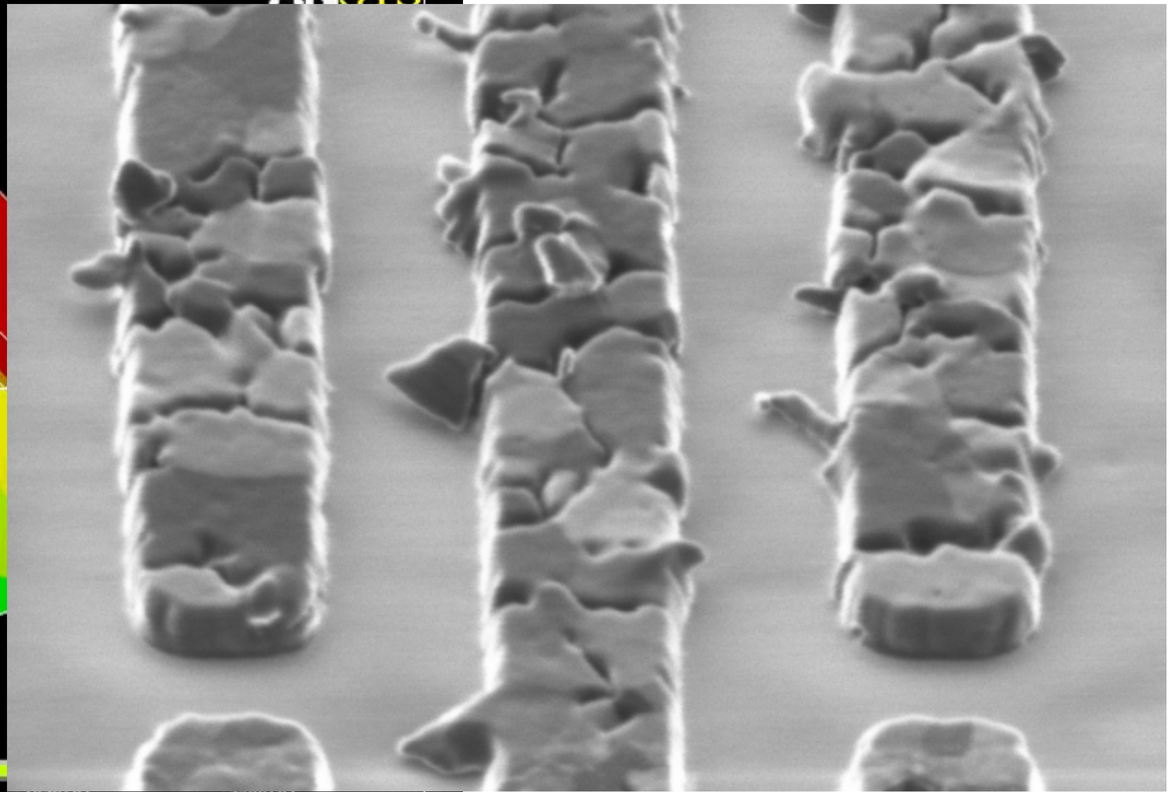




Zhang, Kraft, Volkert, Arzt

FATIGUE AT ULTRAHIGH FREQUENCIES

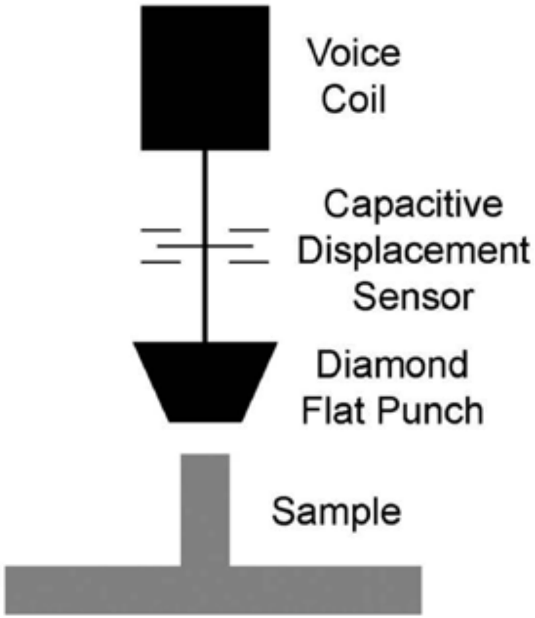


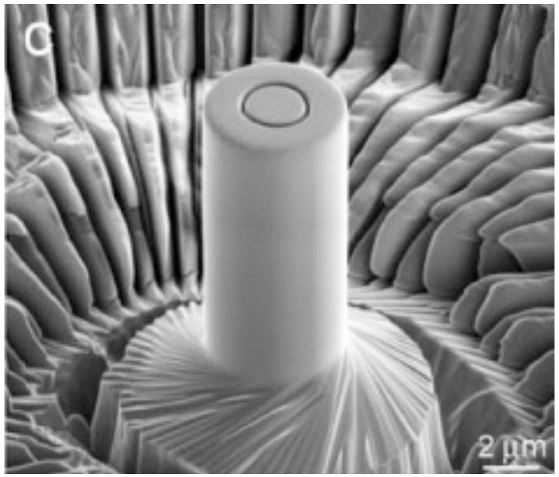


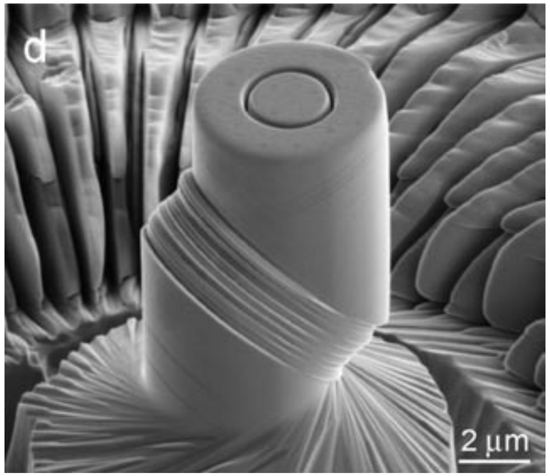
Eigenfrequency analysis of an electrode/substrate model

C. Eberl et al., MPI Metallforschung

“1D”: Microcompression test on pillars



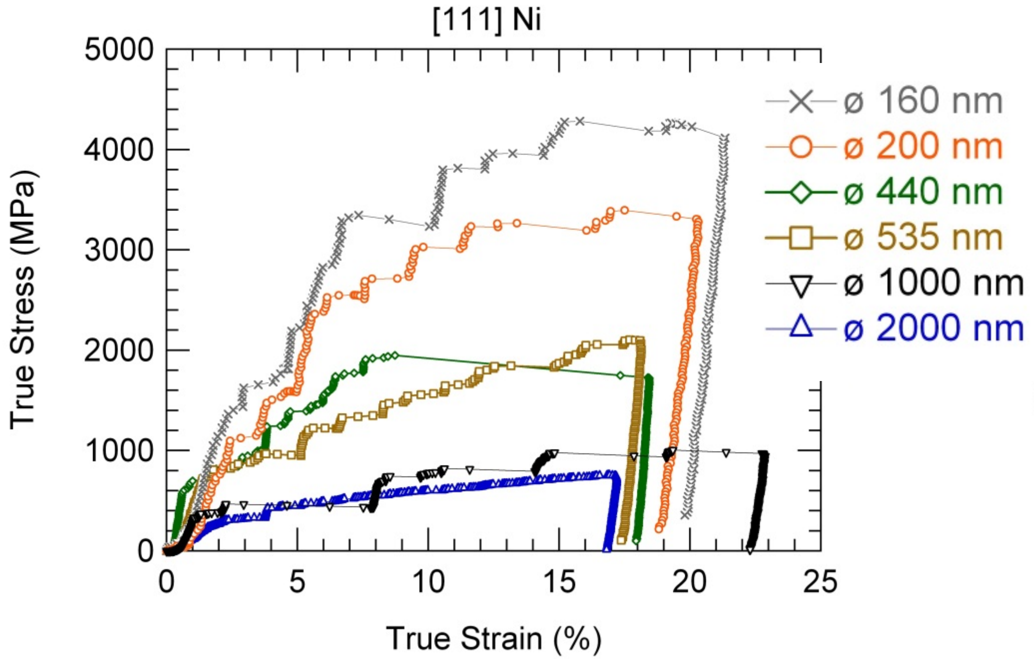


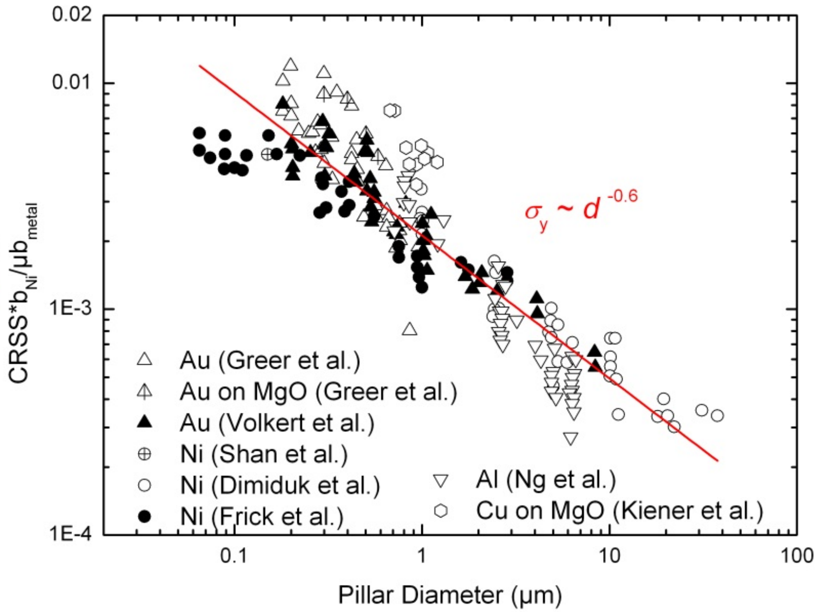


Micropillars (FIB-machined): compressed with a nanoindenter equipped with a flat punch
Advantages: uniaxial stress state, constant test temperature, easy sample handling……

Uchic et al., Science (2004); Uchic et al., Annu. Rev. (2009)

OVERVIEW OF FCC METALS RESULTS





Compression tests on FIB machined, single crystalline pillars show:

- intermittent plastic flow (burst behavior)
- a rising flow stress with decreasing sample size
- power law scaling of yield stress and pillar diameter

Frick and Schneider, MSEA (2008)

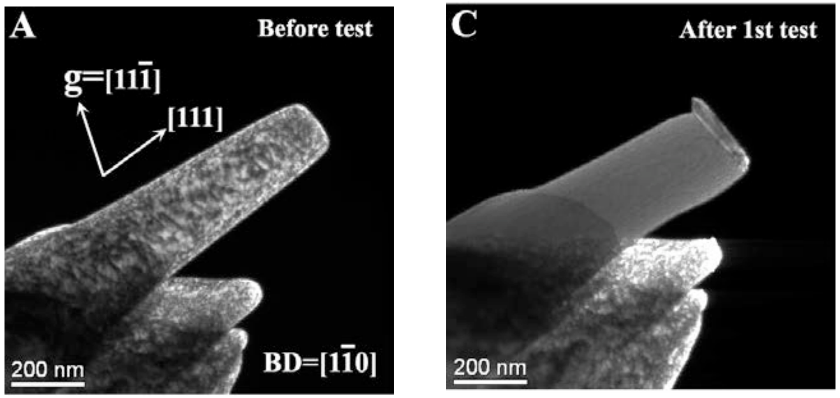
Micropillar – Size effect: explanations

$$\sigma_y \propto \frac{1}{d^n}$$

Starvation theory

Dislocations leave the pillar before multiplication processes can occur

→Dislocation nucleation required



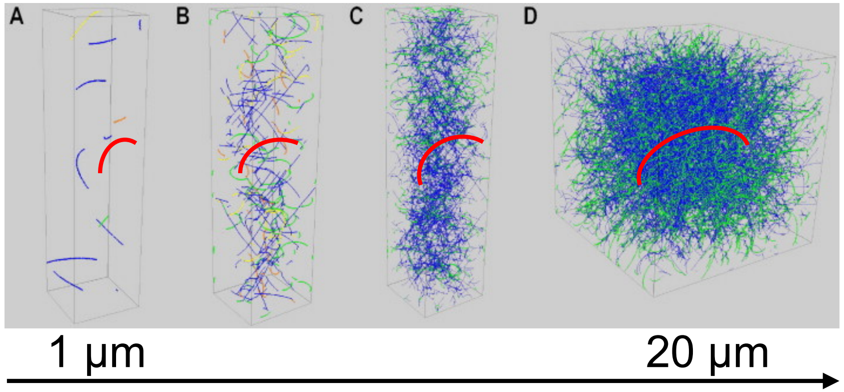
Shan et al. (2008)

Source exhaustion

Limited number of active dislocation sources in small dimensions

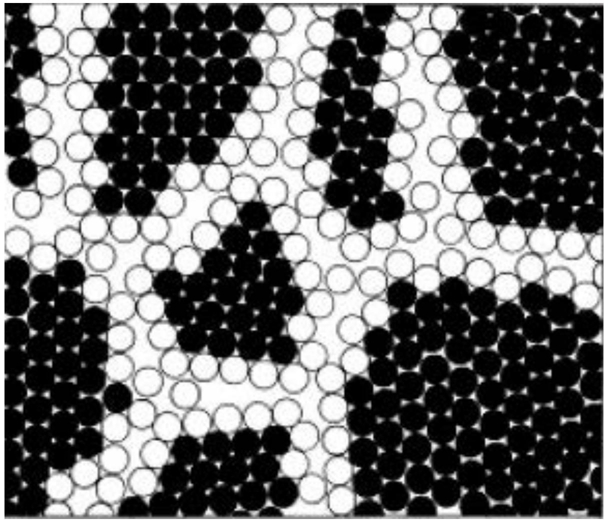
→Source exhaustion leads to the activation of less favorable sources

Source controlled deformation



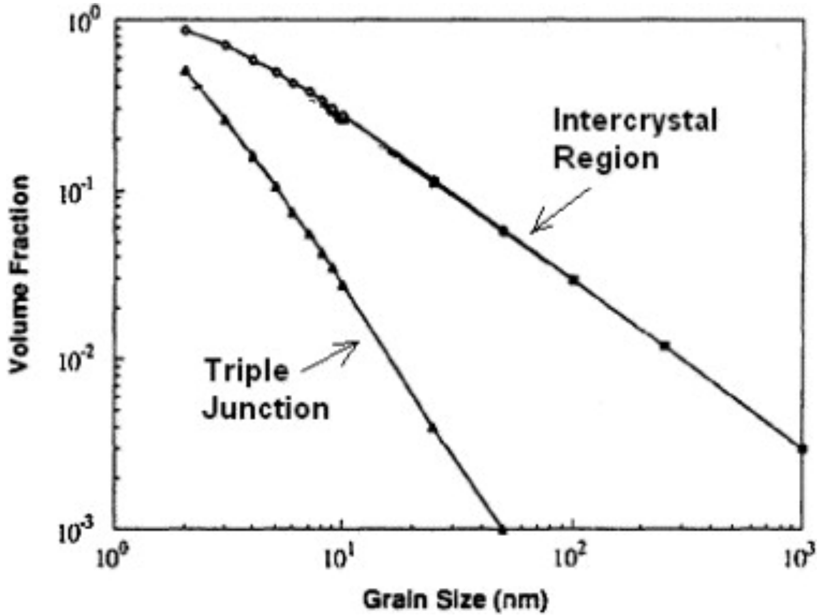
Rao et al. (2008)

NANOCRYSTALLINE MATERIALS (3D)



Disordered grain boundary atoms

Crystalline lattice atoms

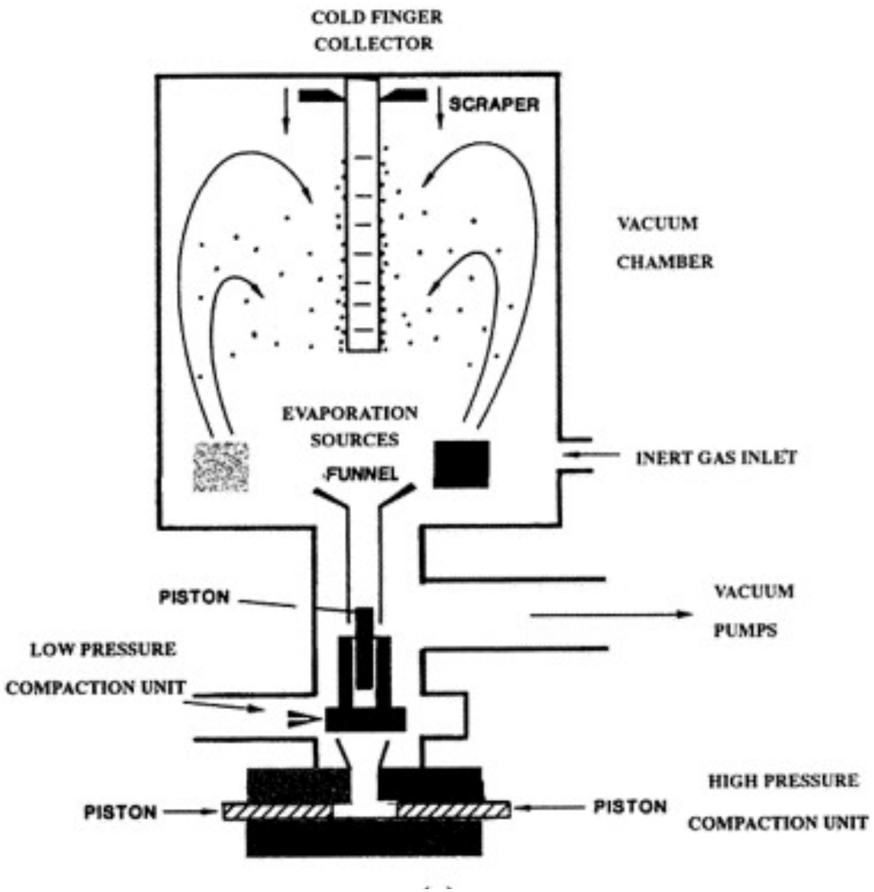


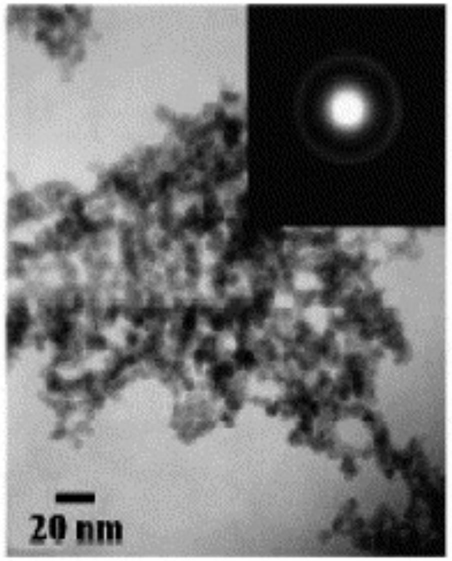
As the grain size is decreased, an increasing fraction of atoms can be ascribed to the grain boundaries (and triple junctions)

Gleiter (2000); Palumbo et al. (1990)

Nanocrystalline materials – Synthesis

Inert gas condensation



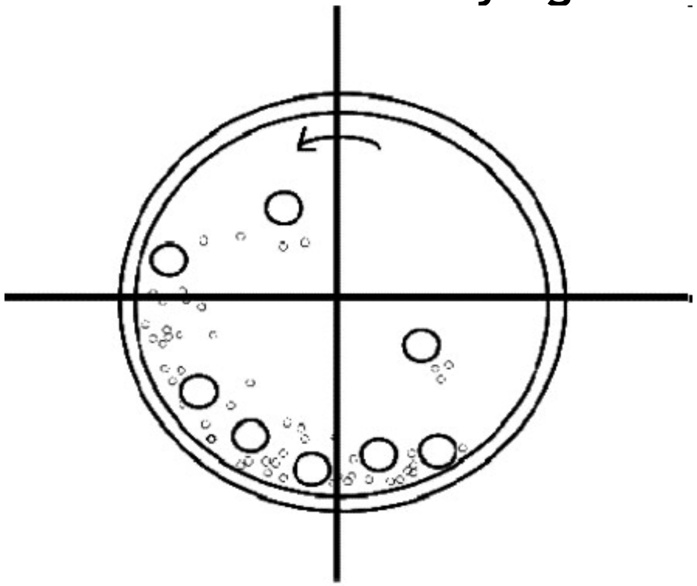


Bright field image of TiO_2 nanoparticles prepared by inert gas condensation

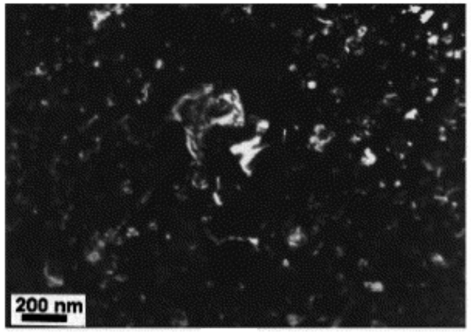
Morris (1998); Lee et al. (2004)

Nanocrystalline materials – Synthesis

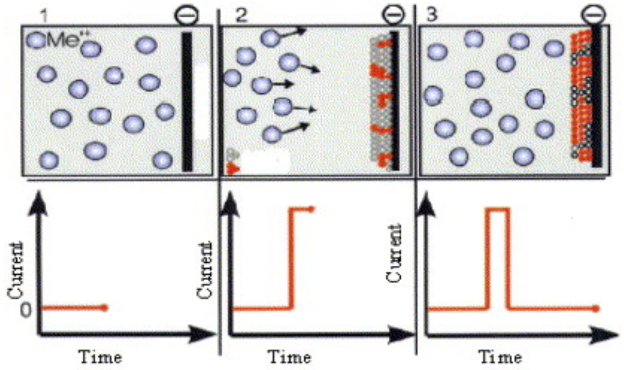
Mechanical alloying

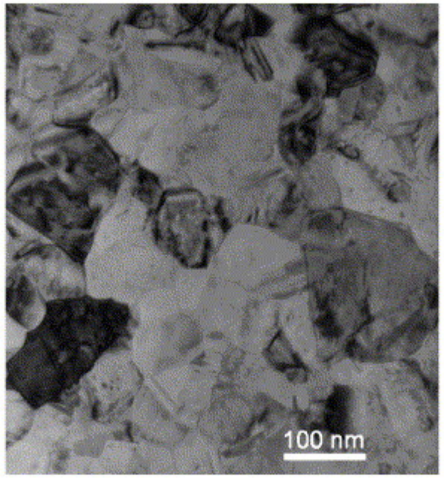


Dark field image of nano-crystalline Al-Mg alloy synthesized by cryogenic ball milling



Electrodeposition





Pulsed electro-deposited Ni

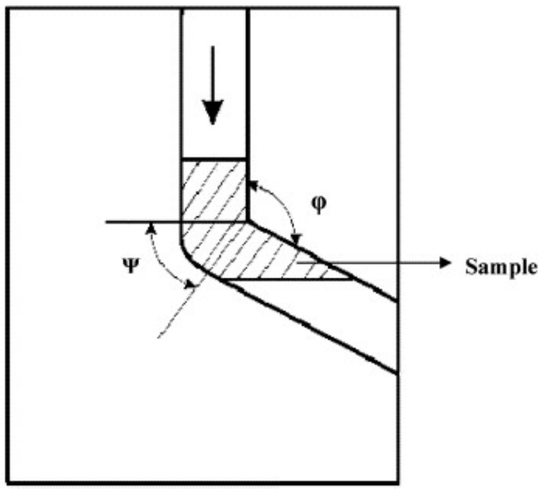
Zhou et al. (2000); Courtesy of M. Göken

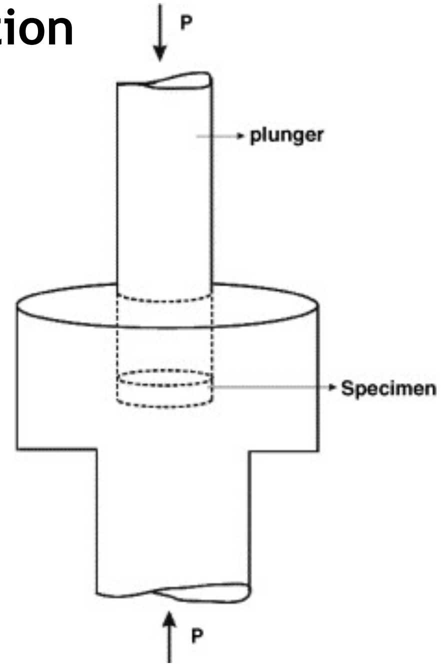
Nanocrystalline materials – Synthesis

Severe plastic deformation

ECAP

Equal-Channel Angular Pressing

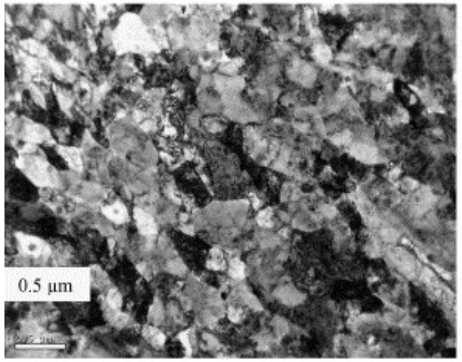


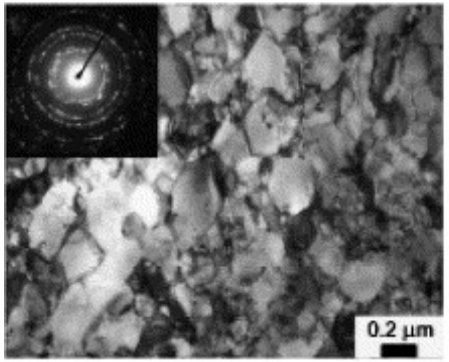


HPT

High-Pressure Torsion

Bright field image of Cu processes by 8 ECAP passes





TEM micro-structure of pure Ni produced by HPT

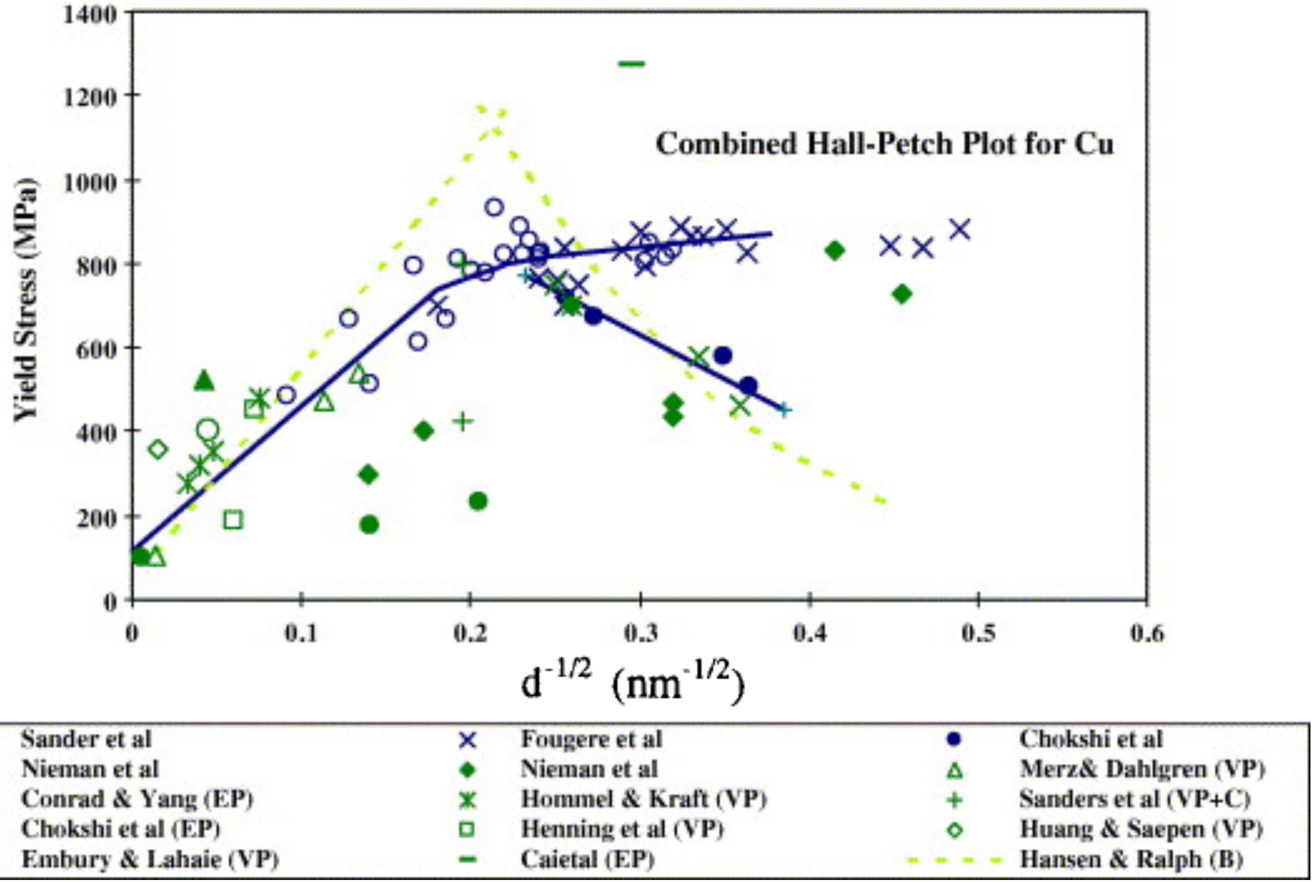
Meyers et al., (2005); Zhilyaev et al. (2001)

Nanocrystalline materials – size effect

$$\boxed{\tau_a \geq \tau_0 + kd^{-1/2}}$$

d.. grain size

Inverse Hall-Petch effect



Meyers et al., (2005)

Nanocrystalline materials – Hall-Petch effect 1

Stress acting at the head of the pile-up

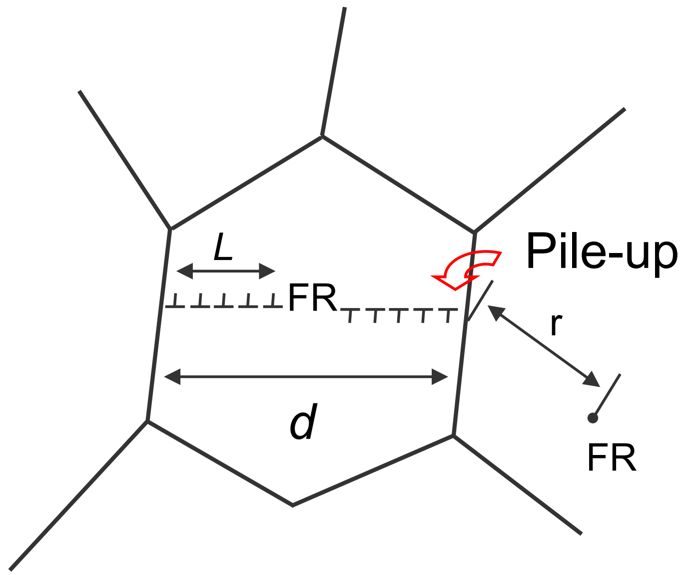
$$\tau = n\tau_a$$

The number of dislocations in a pile-up depends on the length of the pile-up

$$L = \frac{\alpha nGb}{\pi\tau_a}$$

If the source of the pile-up is located at the center of the grain, then

$$L = \frac{d}{2}$$



τ_a: resolved shear stress
n: number of dislocations in the pile-up
d: grain size
L: length of the pile-up
a: geometrical constant

Nanocrystalline materials – Hall-Petch effect 2

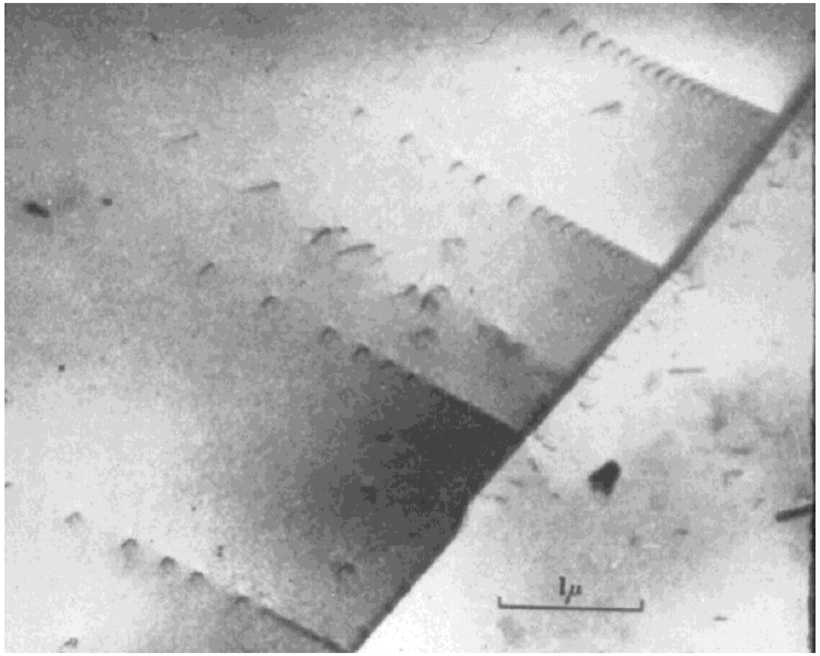
The dislocations will be able to overcome the grain boundary if

$$n\tau_a \geq \tau_c$$

$$\frac{\alpha d \tau_a}{2Gb/\pi}\tau_a \geq \tau_c \quad \textbf{or} \quad \frac{\alpha d \tau_a^2}{2Gb/\pi} \geq \tau_c$$

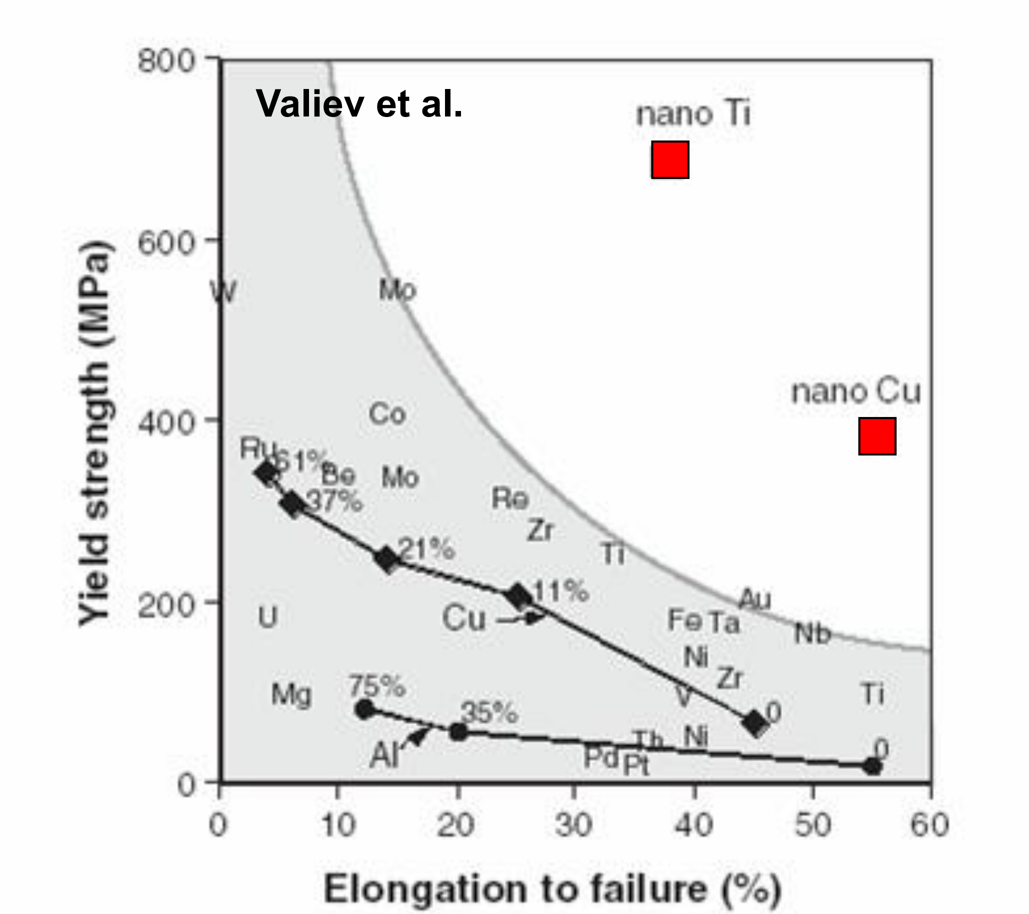
Taking into account the friction stress τ_0

$$\boxed{\tau_a \geq \tau_0 + kd^{-1/2}}$$



τ_c: critical stress to overcome obstacle
τ_0: friction stress

Nanocrystalline materials - strength + ductility



Usually strengthening decreases ductility and toughness. By contrast, grain refinement can improve both **yield stress** and **ductility**. New studies (Valiev et al.) indicate that this particularly significant in **nanocrystalline materials**.

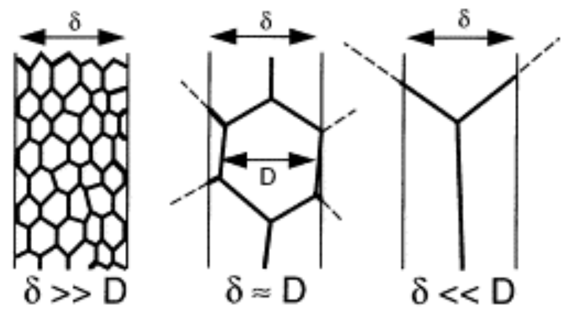
Nanocrystalline materials – Applications

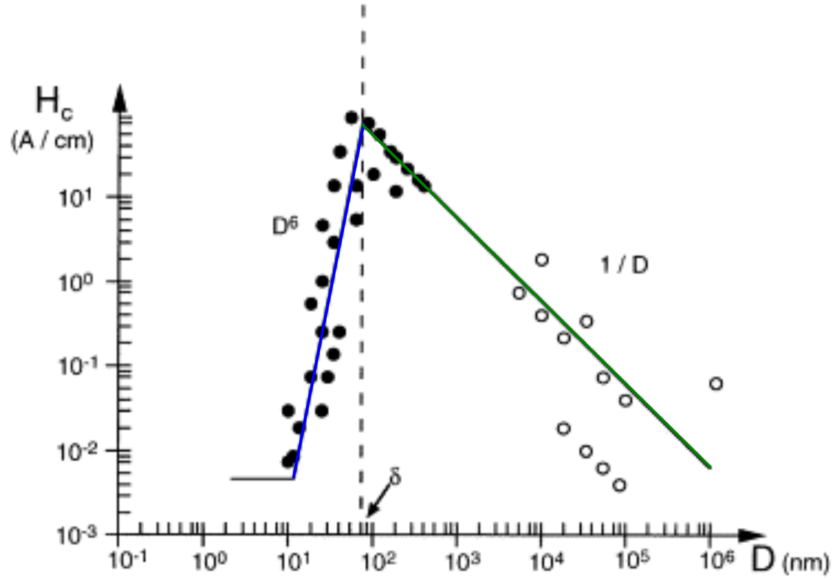
Grain size effects in magnetism

$$\boxed{H_C = H_{C0} + \frac{k_m}{d}} \qquad \delta = \pi\left(\frac{A}{K_1}\right)^{1/2}$$

dreak down for

$$\delta \approx d \quad \Rightarrow \quad \boxed{H_c \propto d^6}$$





Nanocrystalline alloys, where the grains are smaller than the domain wall width, provide low coercivity H_C
→ soft magnets

Herzer (1993); Arzt (1998)

SUMMARY

Mechanical effects of miniaturization:

yield strength	d^{-1}	(2D: thin films)
	$d^{-0.5...-0.8}$	(1D: small pillars)
	$d^{-0.5}$	(3D: nanocrystalline)

Nix-Freund model for single-crystal films
Polycrystals: additional strengthening, constrained diffusion

Fatigue strength increases for smaller systems (Cu: ca. 3 µm)

SMALL = STRONG but: stress relaxation necessary!

Pillars (1D): fundamental studies possible

Nanocrystalline metals: high strength + high ductility
better biocompatibility (?)
application: e.g. soft magnets