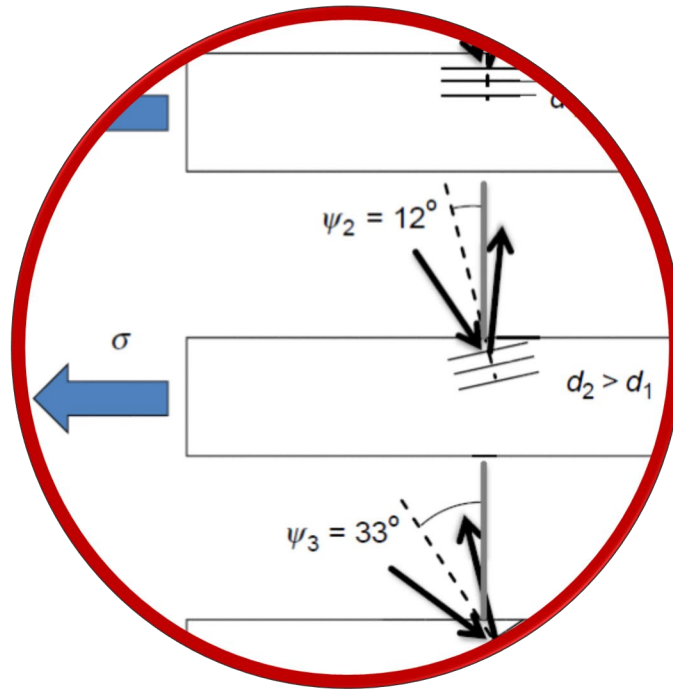
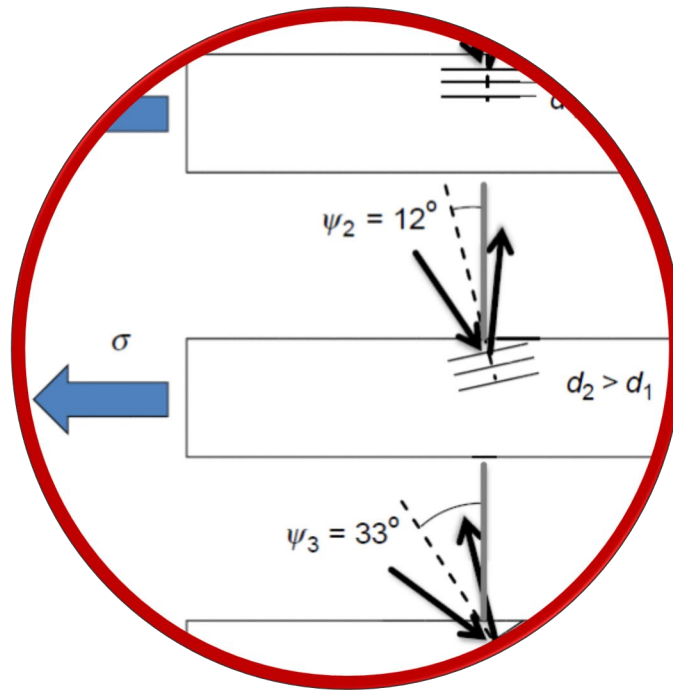


6. Small scale mechanical testing



Sample fabrication techniques



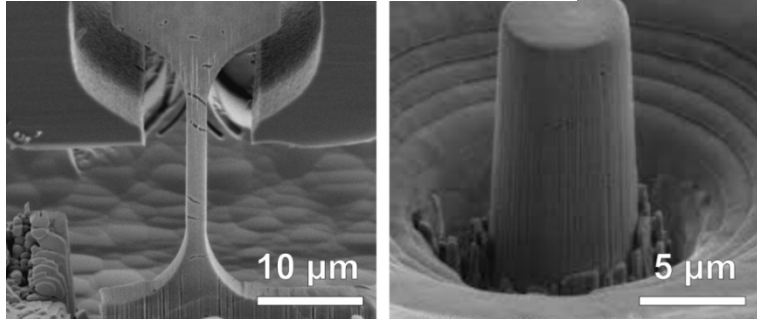
Surface finishing

- Surface roughness needs to be minimized to allow reliable micromechanical testing.
- Rule of thumb: Indentation depth $> 20 \times R_a$
- Polishing with increasing grades of SiC abrasive paper followed by diamond slurry on polishing cloth is an established technique for many materials
- Alternative for softer materials: Ultramilling or microtoming with diamond blades
- For soft metals, beware of strain hardening due to mechanical polishing!
- Electropolishing is a good alternative if strain hardening is a concern
- Ion beam polishing can create even smoother surfaces

Focused ion beam

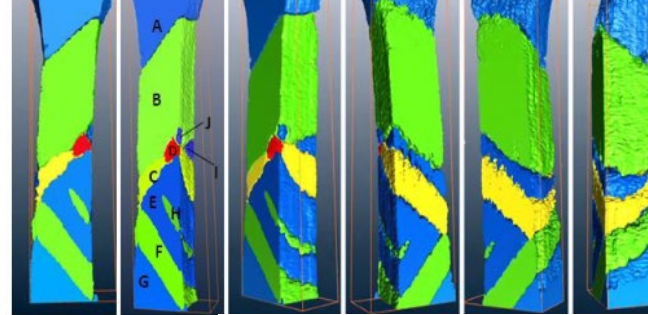
FIB applications examples

Micromechanical specimen fabrication



Casari et al., *Acta. Biomater.*, **120** (2021) Schwiedrzik et al., *Nat. Mat.*, **13** (2014)

FIB tomography + HR-EBSD



Della Ventura et al., *Mater. Des.*, **197** (2014)

3D nano structuring

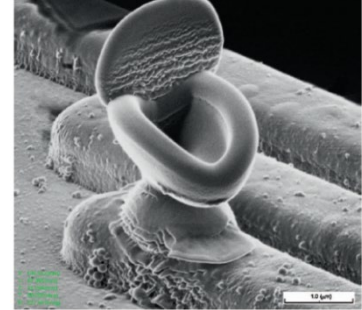
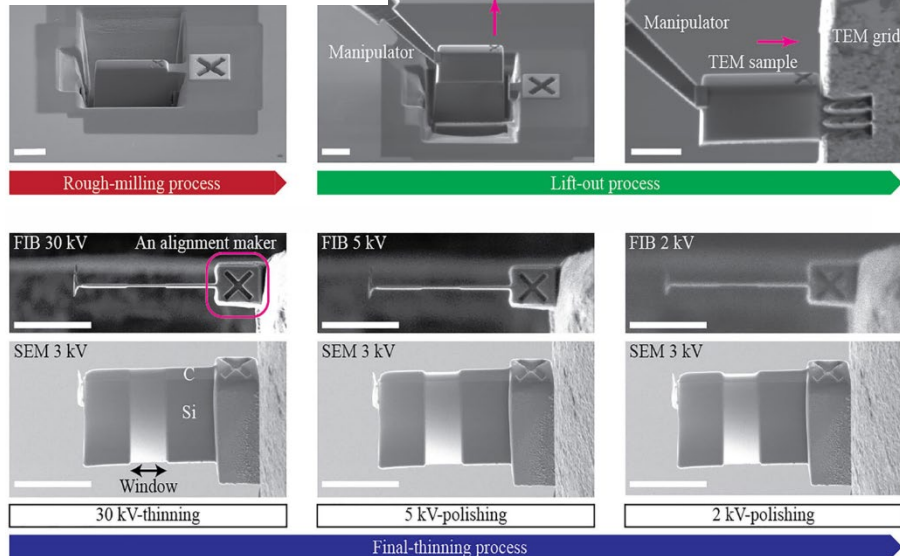


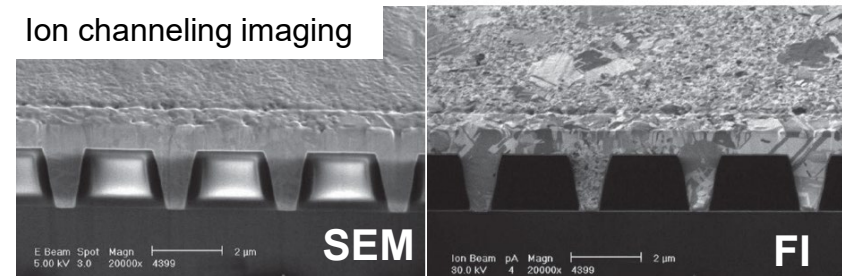
Image from Seiko instruments

TEM lamella preparation

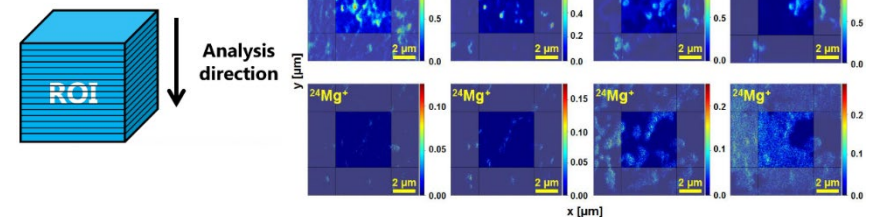


Tsurusawa et al., *Sci. Rep.*, **11** (2014)

Ion channeling imaging



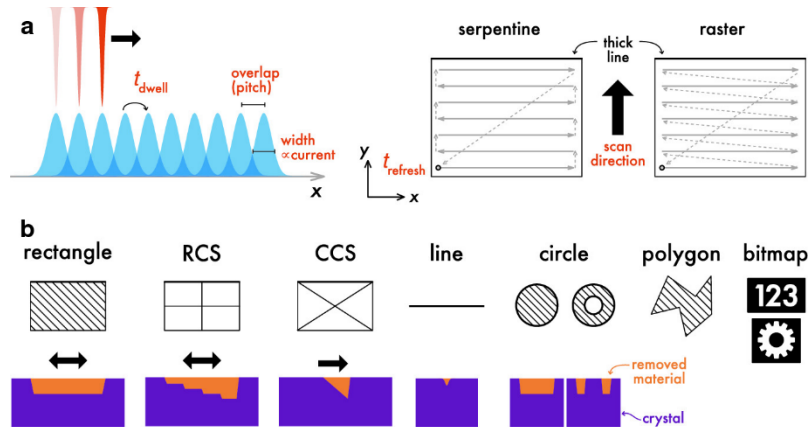
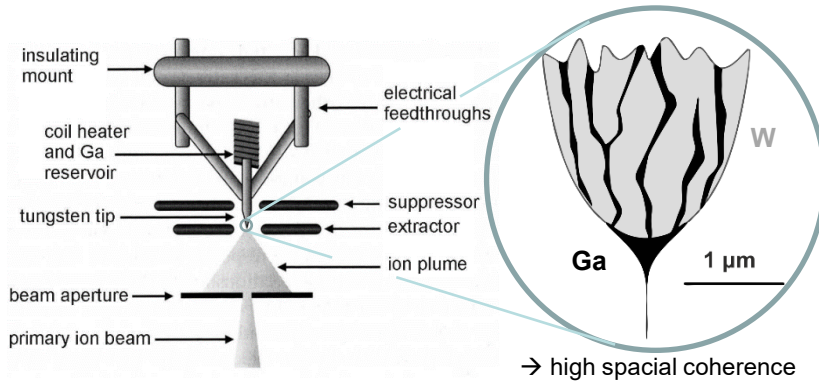
TOF-SIMS



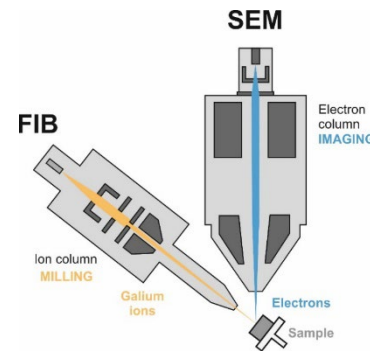
Priebe et al. EUFN Workshop poster (2018)

A quick overview on FIB

Schematic of a liquid-metal ion source (LMIS)

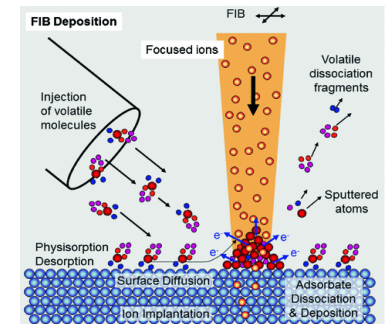
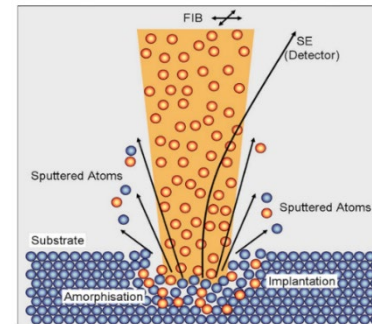
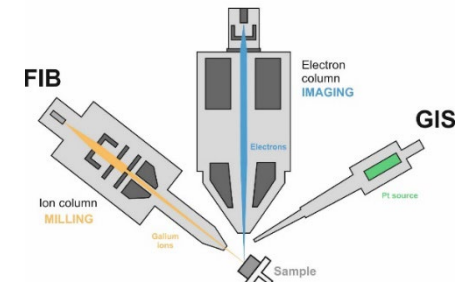


Standard milling



Gas induced deposition & etching

e.g. Pt, C, W e.g. Cl₂, XeF₂



Utke et al., *J. Vac. Sci. Technol. B* 26 (2008)

Parameters we can control

- Dwell time
- Dose
- Spacing
- Beam irradiation path

Different FIB types and ion sources

Liquid metal ion source (LMIS)

Bassim et al., *MRS Bulletin* (2014)

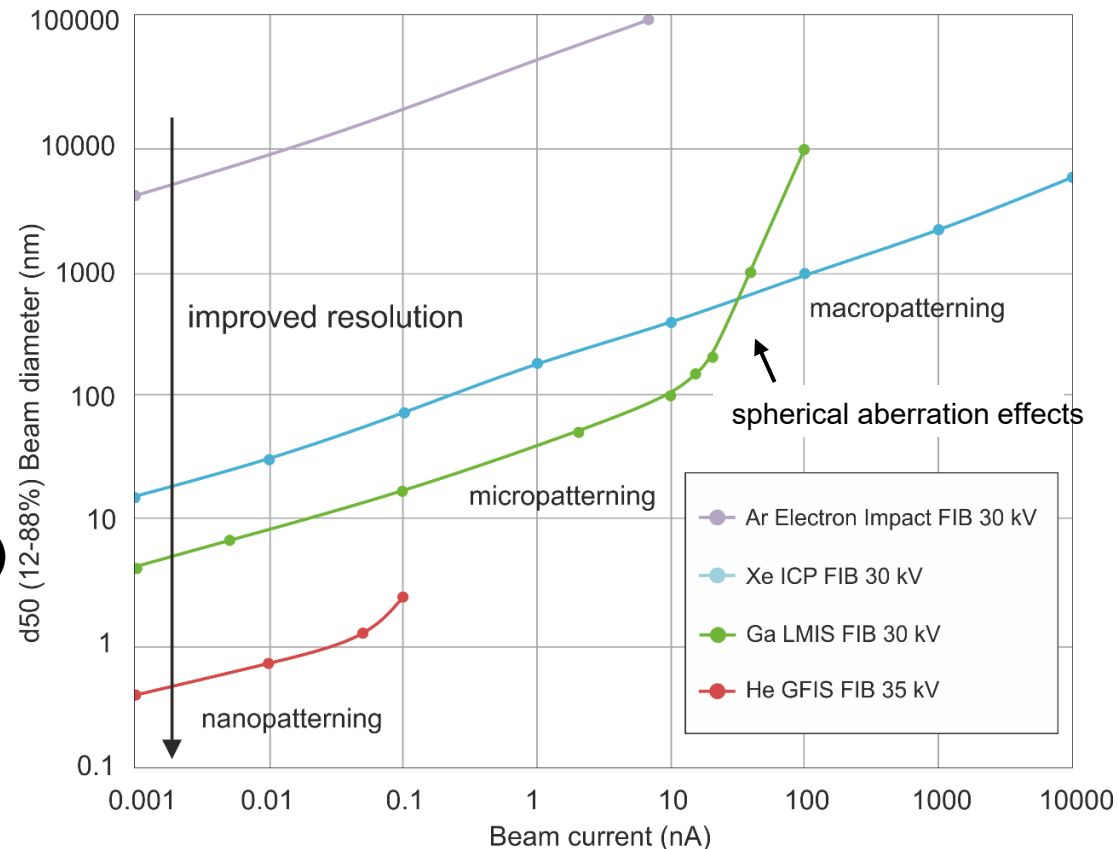
- Typically Ga
 - High stability
 - Alloy sources: Au, Bi, Si,
- Current range: 1 pA - 100 nA

Inductively coupled plasma (ICP)

- Typically Xe
- Current range: 100 pA – 1 μ A

Gas field ionization source (GFIS)

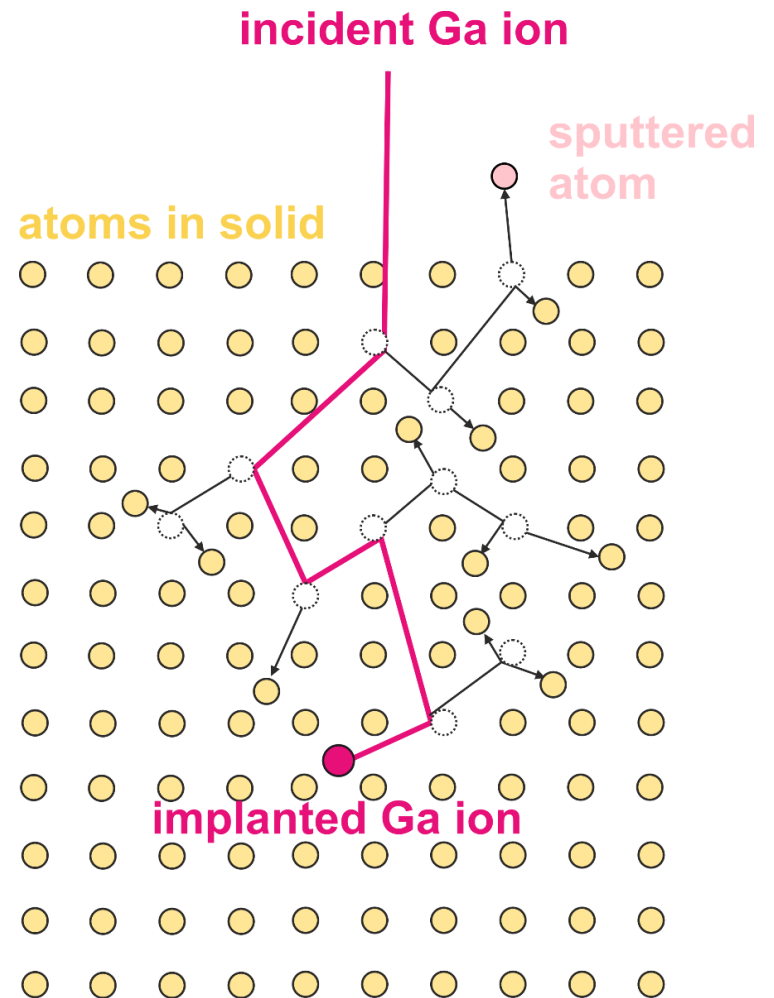
- Typically He or Ne
- High spatial resolution (<0.35 nm)
- Imaging insulating samples
- Current range: 0.1 pA – 100 pA



Collision cascade model

Ion-solid interaction

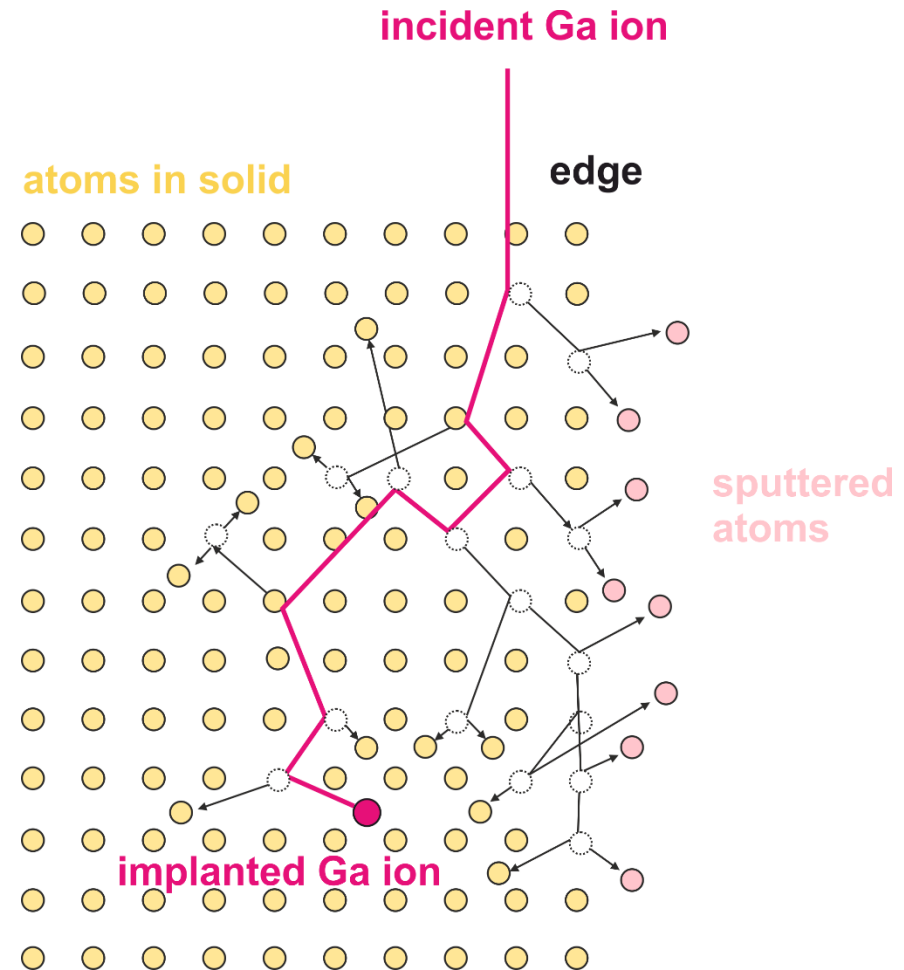
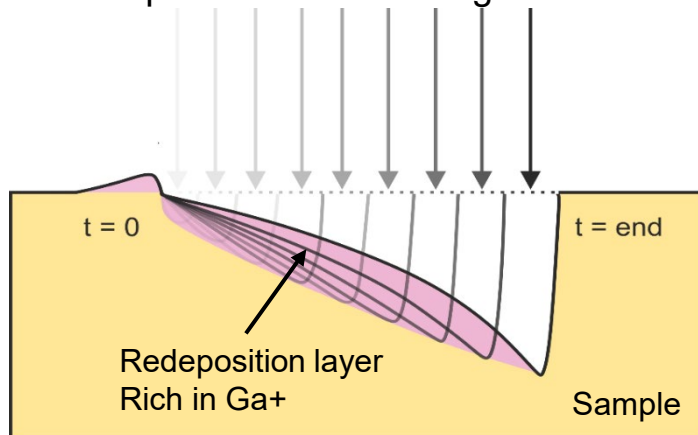
- Surface atoms can be ejected (sputtering /milling)
- Ca. 30 ps process
- Relaxation after first ca. 10 ps
- Implanted ions
- Amorphization
- Electrical damage zone
- 30 kV Ga⁺ on Si: ca. 30 nm
- Different possible sources: Ga, Xe, H, etc...



Milling at an edge

Edge effect

- Increased sputter yield (ca. 8x)
- Topographic effect (corner will mill even faster)
- Scan strategy matters
- Line-by-line milling is efficient
- Redeposition becomes significant



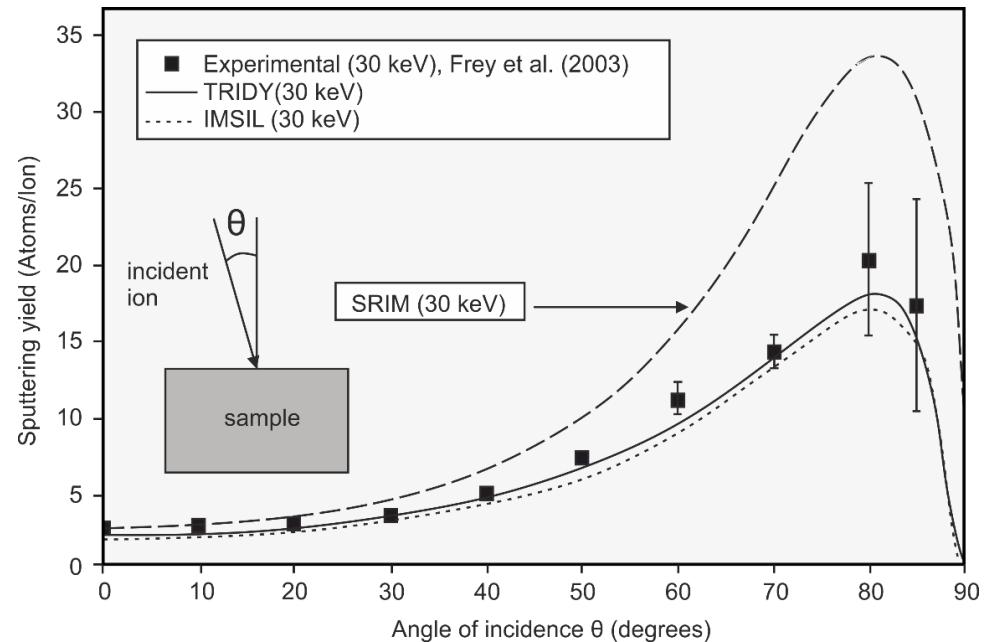
Tangential irradiation: increased sputtering rate

Sputter yield and sputter rate

Sputtering by 30 kV Ga⁺ normal incidence

Element	Sputter yield* Y (-)	Sputter rate** S ($\mu\text{m}^3/\text{nC}$)
C	1.69	0.18
Al	3.47	0.29
Si	2.78	0.24
Ti	2.28	0.46
Cr	5.15	0.10
Au	15.75	1.50
W	7.59	0.12

Max efficiency for an incident angle of 75-80°

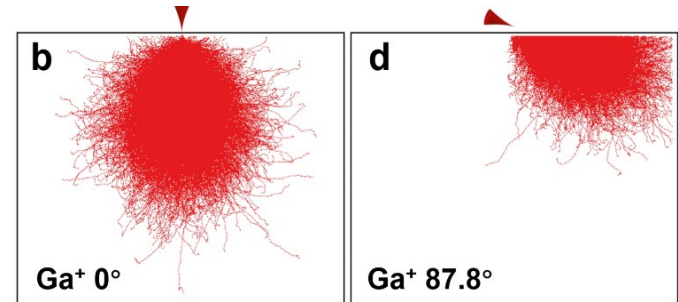


Sputtering yield Y : atoms sputtered per incoming ion

Sputtering rate S : volume removed per dose

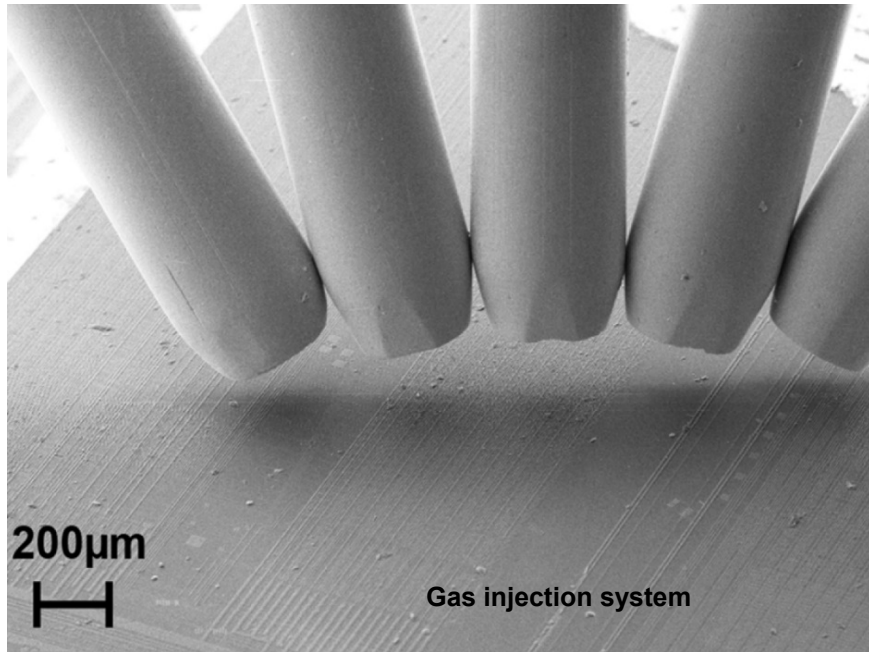
* SRIM data from: , Lucille A. Giannuzzi and Fred A. Stevie, Introduction to focused ion beams, Springer, 2005.

** Ivo Utke, Stanislav Moshkalev, Phillipp Russell, Nanofabrication using focused ion and electron beams, Oxford University Press, 2012.



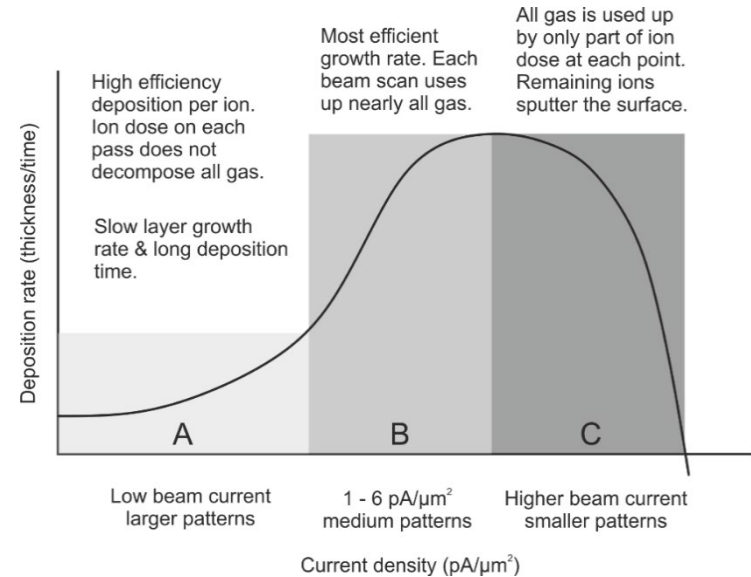
Liu et al., *Sci. Rep.*, **10** (2020)

Deposition induced by FIB



Deposited species is not pure: lots of Ga and C

Deposition without surface damage only by eBeam dep

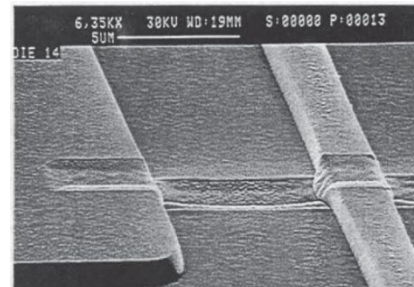


Good parameters for Ga⁺ ion Pt deposition

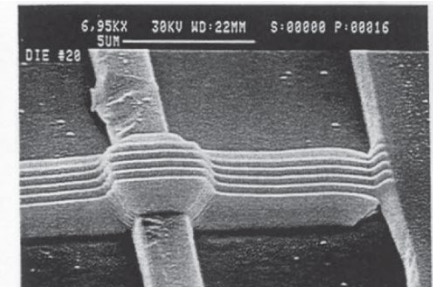
- Ion probe current = Area * 5 pA/µm² (i-dep)
- Pixel fill factor = 40 - 70 %
- Short dwell time (0.2 µs for Pt)

Good parameters for e- Pt deposition

- Electron probe current 2000 pA @ 5 kV (e-dep)



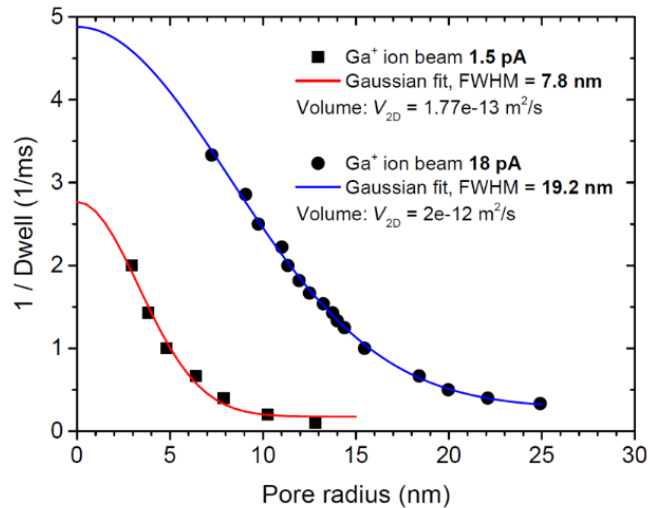
Bad parameters



Good parameters

Beam profile

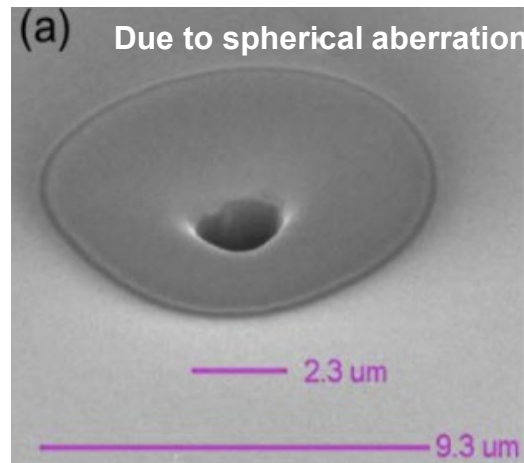
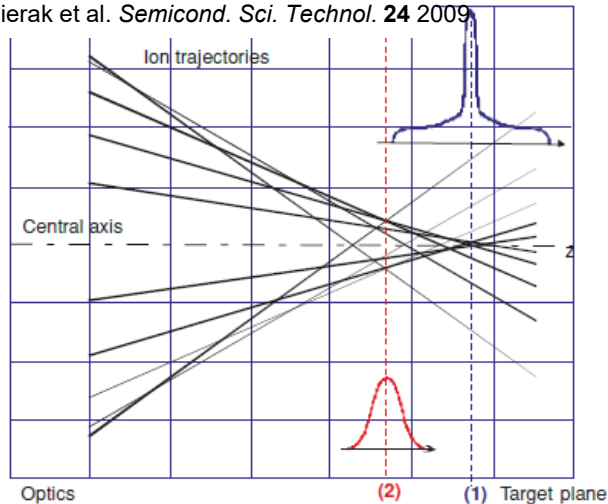
Shorubalko et al., *J. Nanotechnol.* **8** (2017)



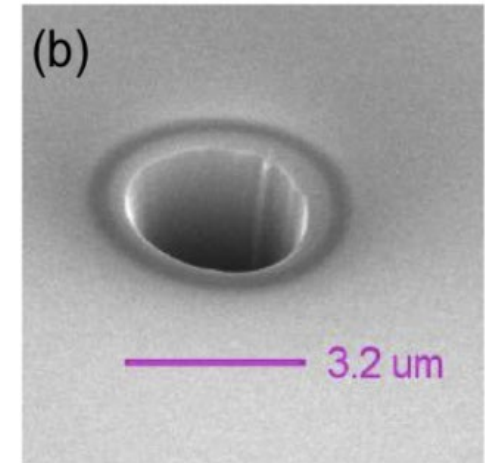
Beam traces

- Electromagnetic lenses $\rightarrow$ high current $\rightarrow$ **spherical aberration**
- Rounding of the milled edges
- Circle of less confusion at high currents lead to a better result than Gauss optimum

Gierak et al. *Semicond. Sci. Technol.* **24** 2009



27 nA no defocus

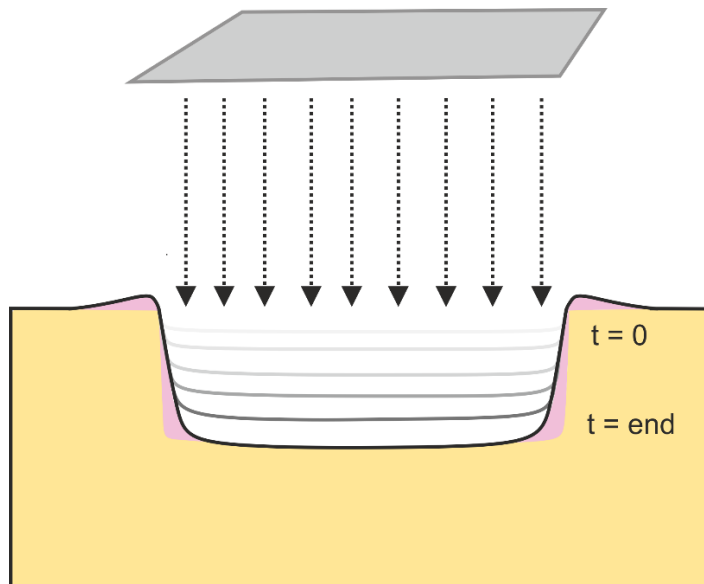


27 nA defocus

Two main milling strategies

Layer-by-layer

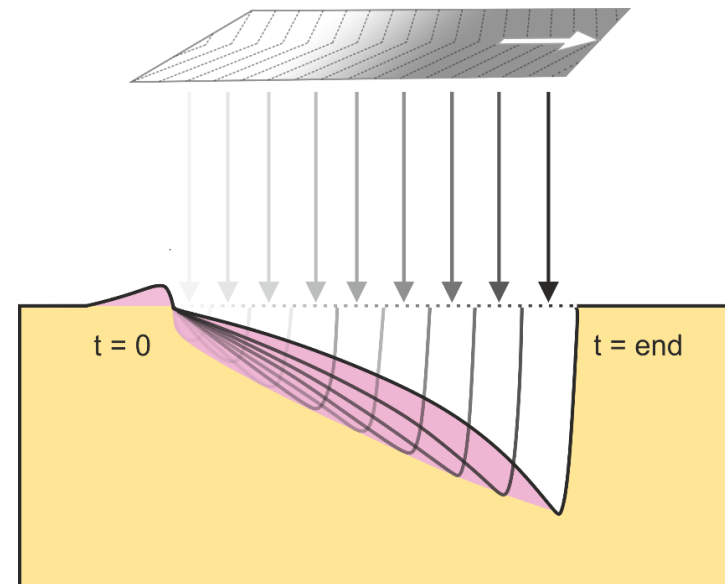
- Form a parallel floor
- Imaging
- Delayering
- Low redeposition but much slower



Layer-by-layer cut

Line-by-line (polishing)

- Tilted floor: shallow to deep
- Used for cutting (cross sections /polishing)
- Fast removal of large volumes
- Redeposition on floor and sides



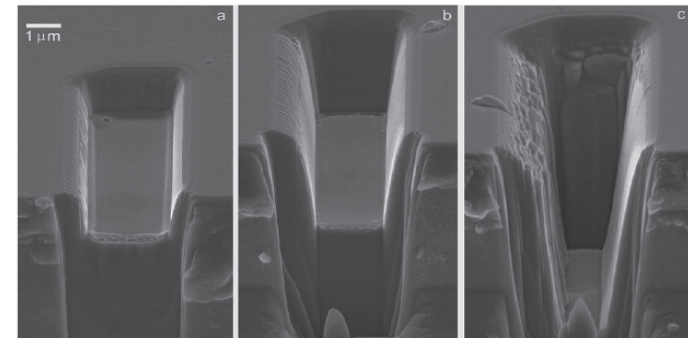
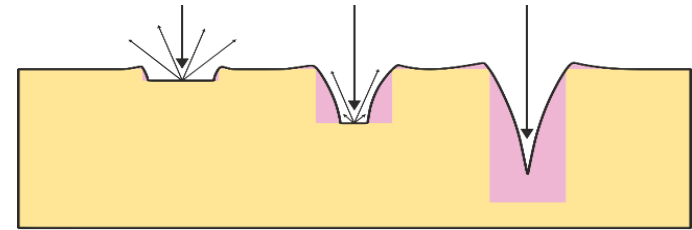
Line-by-cut (polishing)

Aspect ratio and tapering

Redeposition limits the milling aspect ratio

When the rate of redeposition equals the rate of sputtering: dynamic equilibrium (V-shaped trench).

- Generally maximum aspect ratio holes $\sim 5:1$
- Can be improved to $\sim 10:1$ with gas enhanced etching
- For small holes with size below 100 nm maximum aspect ratio of 1:1.

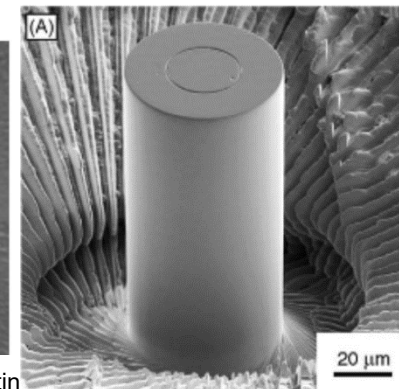
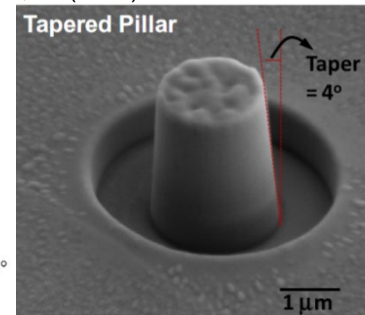
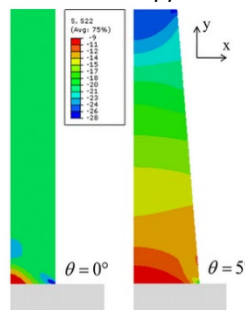


(100) Si milled at normal incidence by applying: (a) single fluence, (b) double fluence, and (c) triple fluence of Ga⁺ ions at 25 keV.

Sidewall tapering

- Material dependent
- Typical values $\sim 1-4^\circ$
- Some extreme cases e.g. Zn $\sim 14^\circ$

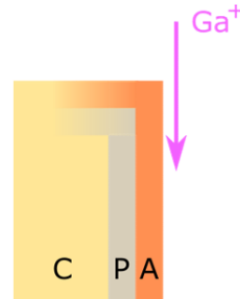
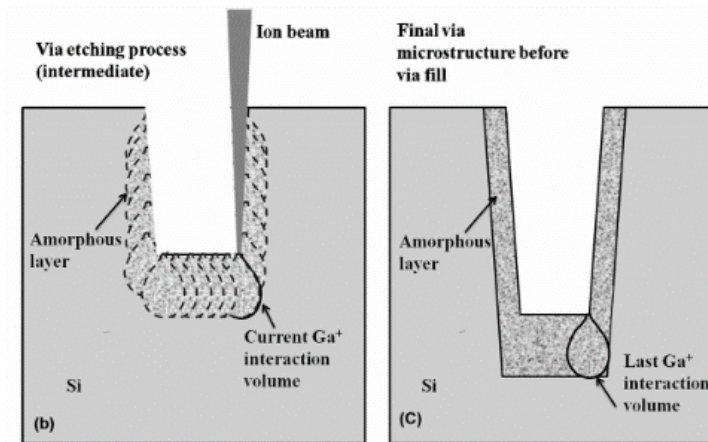
Fei et al. *J. Appl. Mech.*, **79** (2012)



Inhomogeneous axial stress during micromechanical testing

Solution: overtilt to compensate (if possible)

Amorphization layer



Reducing the amorphous layer

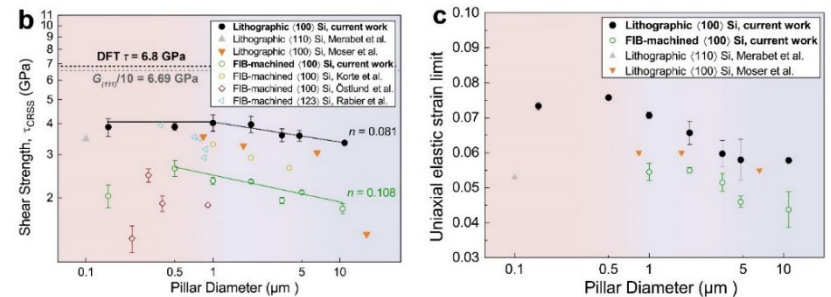
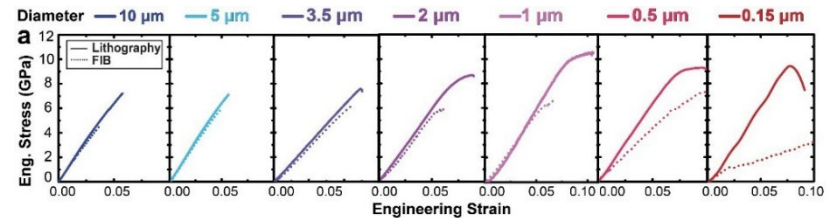
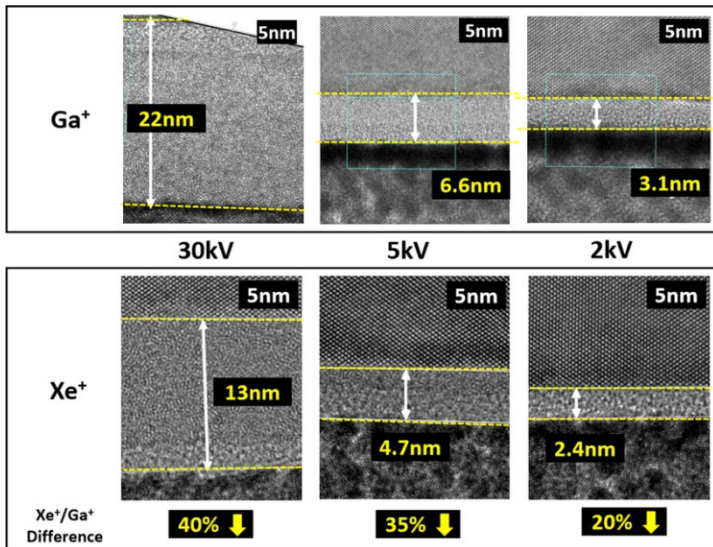
Low kV milling

- Larger ion
- Lower angles

Damage zones in a crystalline solid C

A = amorphized

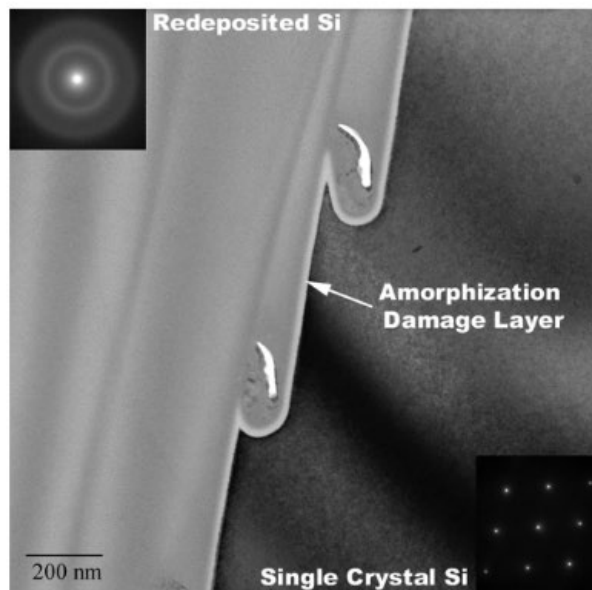
P = point defects



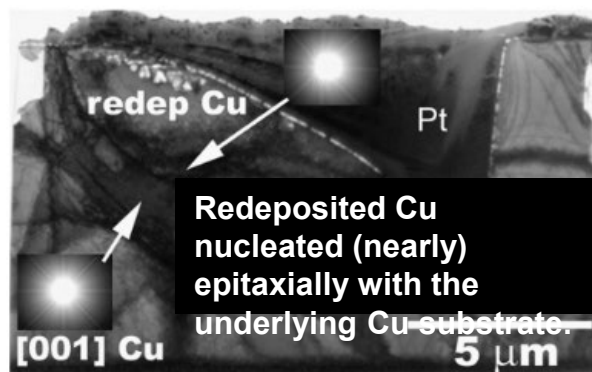
Does FIB induced damage influence the material behavior?
Something to keep in mind...

Amorphization on EBSD imaging

Matteson et al. *J. Electron. Mater.* 31 (2002)



a



b

Amorphous layer thickness

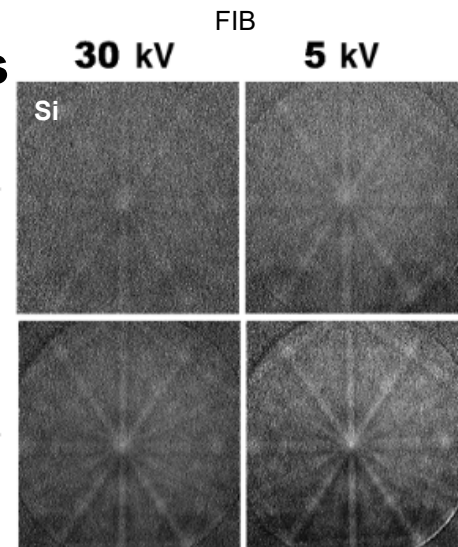
- ion energy (low kV: **thin layer**)
- angle of incidence (tan: **small**)
- ion species
- target material

Metastable phase

Formation depends on complexity and size of the unit cell

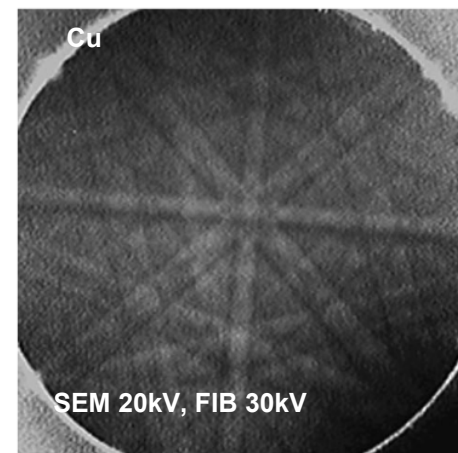
Not always amorphous?

Small unit-celled, as well as broad phase field materials are more difficult to amorphize.

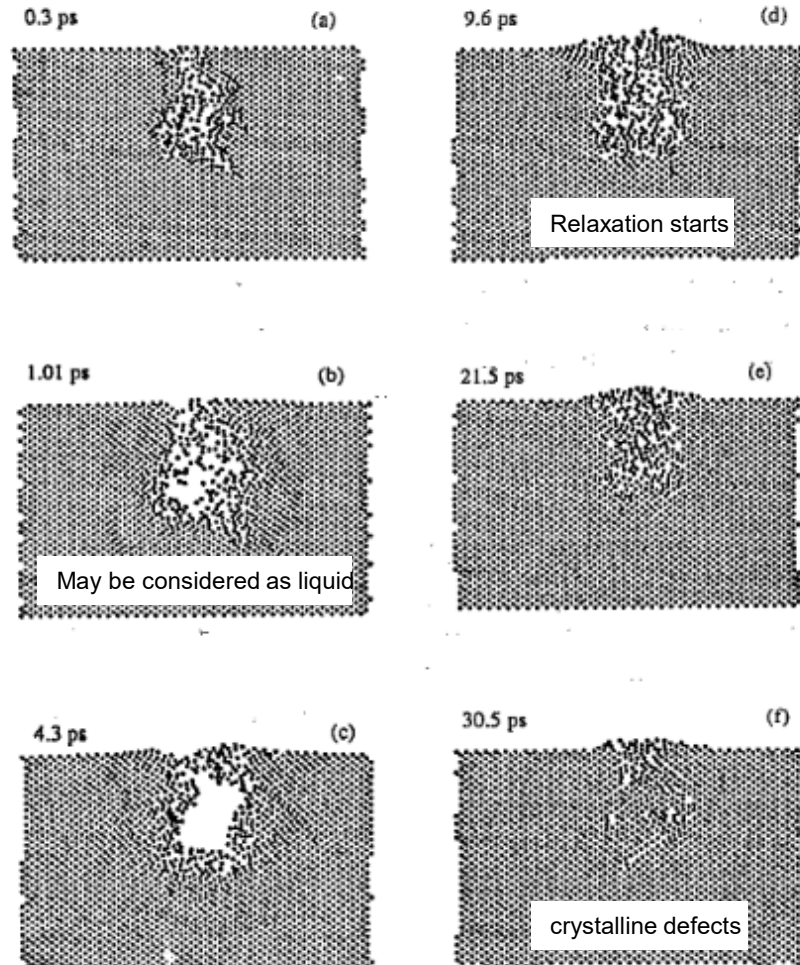


SEM

30 kV

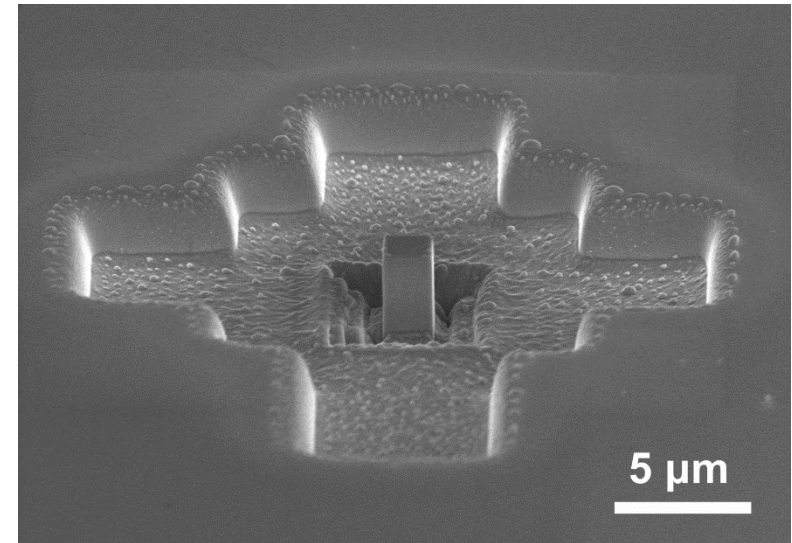


Local heating



MD simulation Au particle 10 keV on a (100) Au surface
Averback and Ghaly, *J. Appl. Phys.* 76 (1994)

- Heating **restricted** to the region defined by the **collision cascade** (a few tens of nm)
- If low dwell time < 5 μ s $\rightarrow$ most likely negligible for most materials.
- Be aware of polymers and low melting point materials (e.g. GaAs bleeding $\rightarrow$ reduce current)



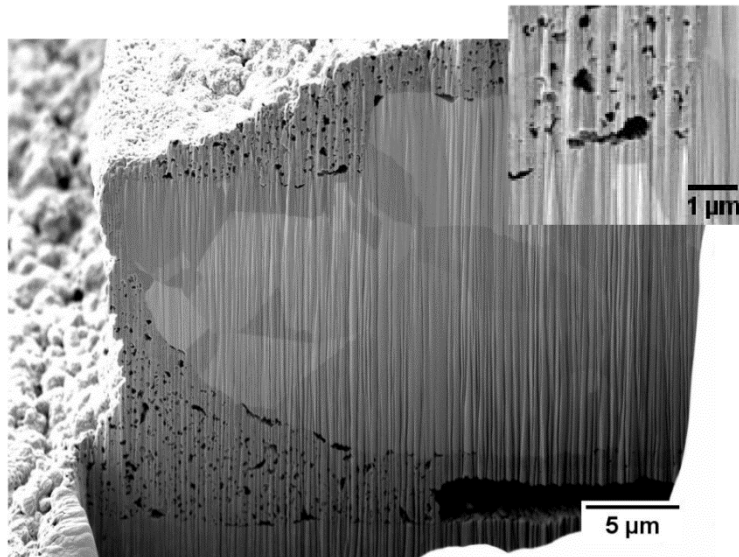
GaAs micropillar showing signs of bleeding due to local heating during milling

Curtaining

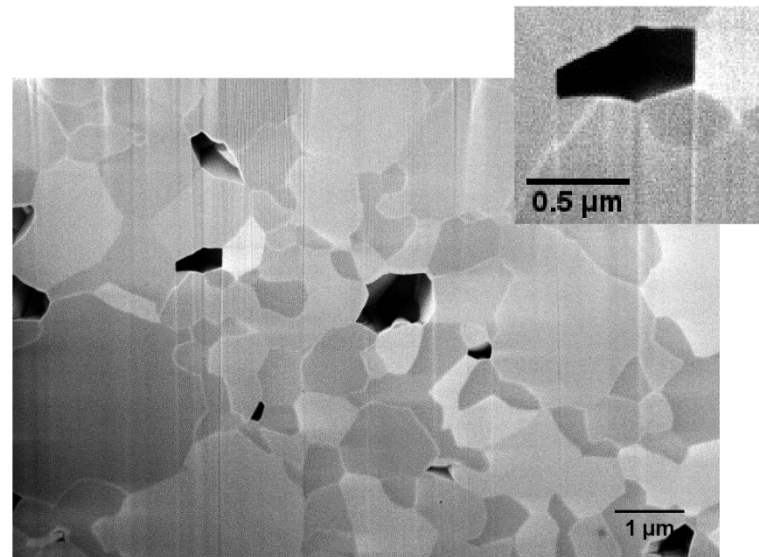
Curtaining (aka waterfall effect)

Milled face shows lines (more / less material was removed)
Irregular non flat surface: bad for quantitative image analysis

- Porous materials
- Rough surfaces
- Height steps (e.g. gates in semiconductors)
- Composites of hard and soft material
- Low kV milling



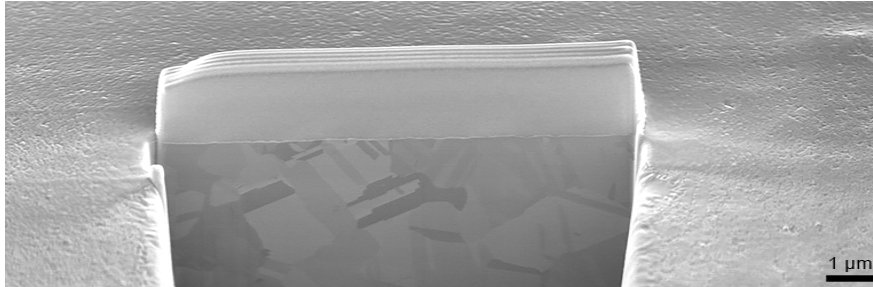
Porous ceramic on Ni: strong curtaining due to rough surface



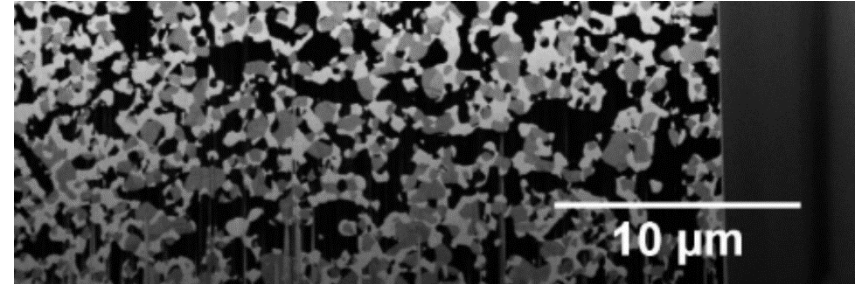
Porous cermet: mild curtaining due to pores

Curtaining is created by **spatial variation of the sputter rate** of the sample and **the modulation of the current density by forward scattering of the ions**.

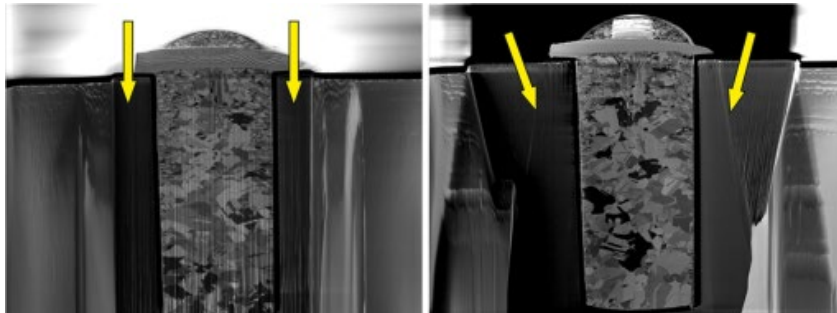
How to reduce curtaining?



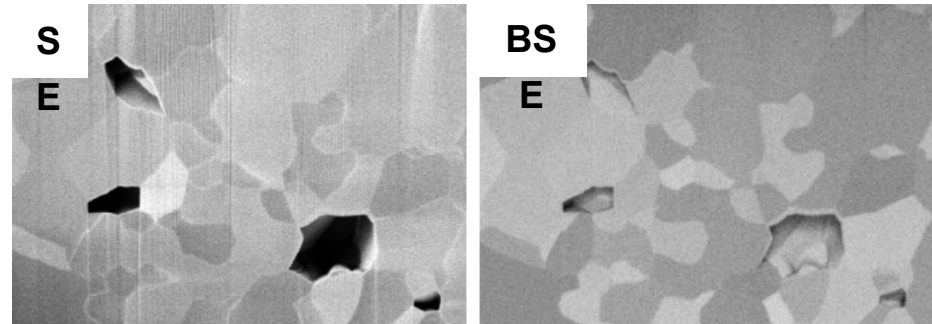
Thick protection layer



Resin infiltration



Rocking stage, sample rocking



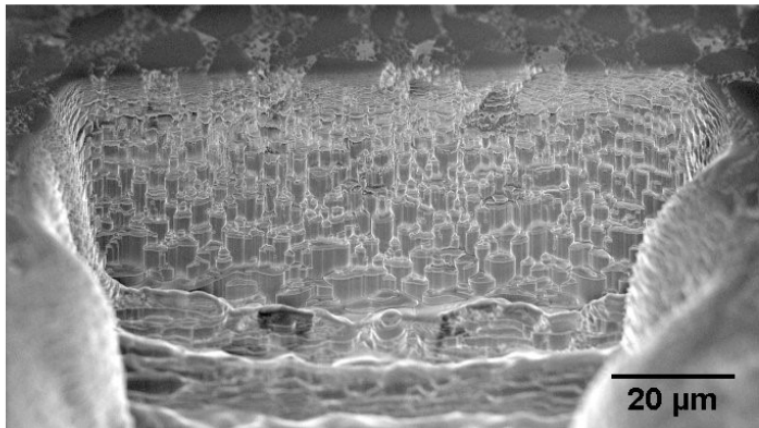
Hide it: BSE signal or post processing

1. Thick and dense FIB-deposited protective layer (W or Pt)
2. Stage rocking/tilting the specimen in the image axis
3. Increase the minimum dose
4. Resin infiltration
5. Backscattered electrons
6. Postprocessing

Rippling and how to avoid it?

Ion rippling

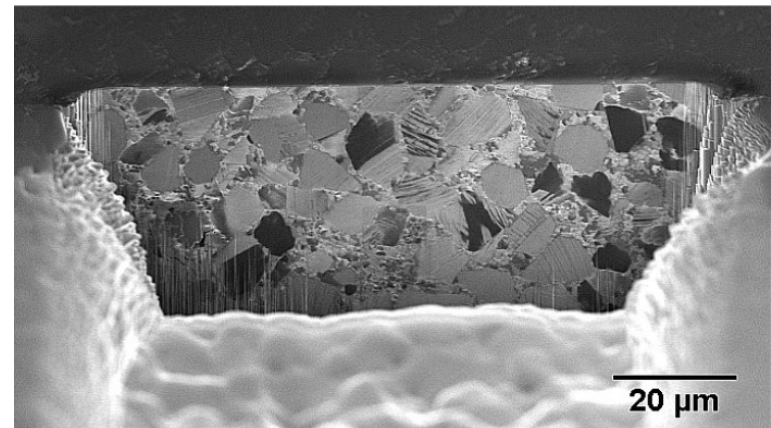
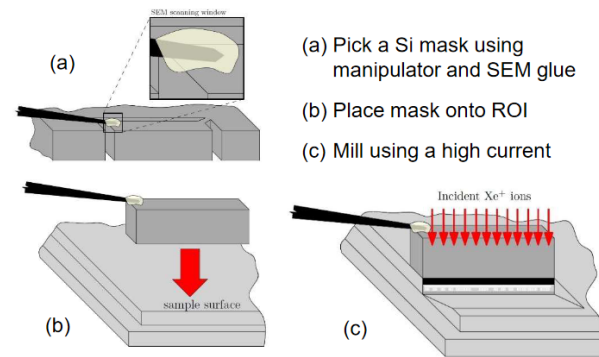
- Self organization
- Instability of process
- Material dependent
(silicate rocks are bad Al and Si are fine)
- Ion dependent (size of ion vs atoms in solid)
- Current density dependent
- Mostly see it in Xe PFIB



Massive ion rippling in SiC sample, microstructure completely obscured.

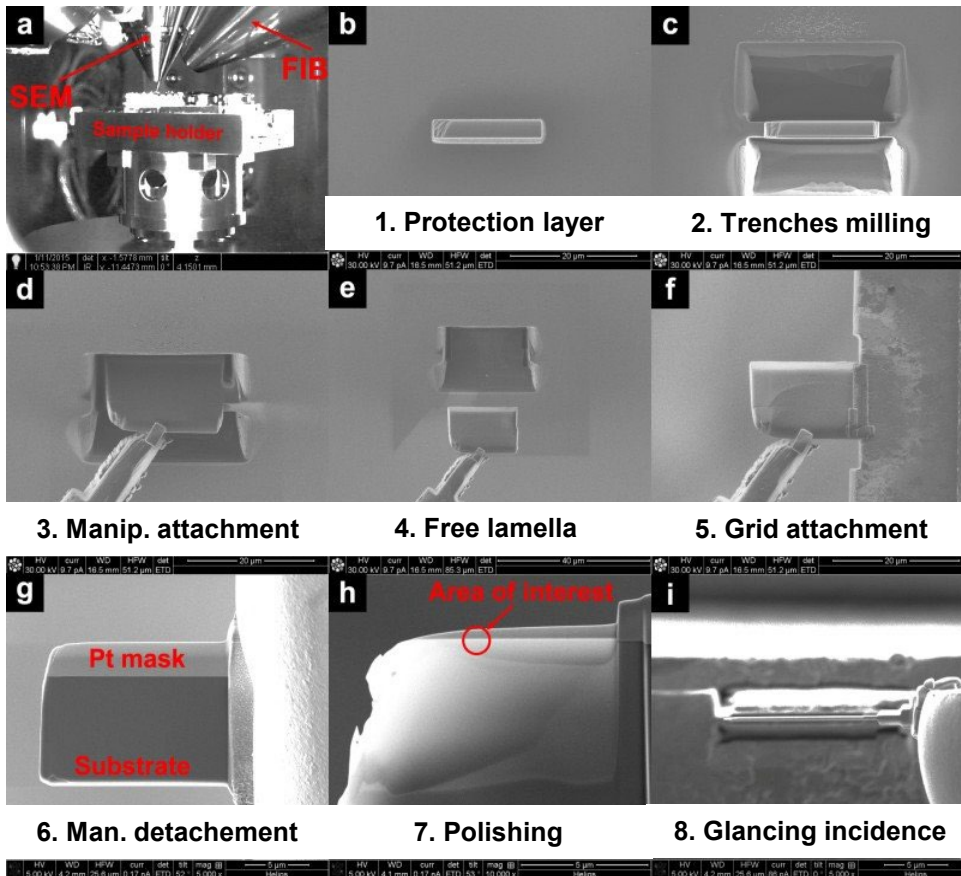
Solution

- Thick protection cap will help
- However masking works best



Cross section polished using Si mask, microstructure clearly visible.

Examples of TEM lamella prep.



Trenches milling

Mostly work at 30 kV (most materials behave ok)

Leave a few μm from ROI at high currents (beam traces)

Sequentially reduce current e.g. 10 nA, 2nA, 200 pA

2 μm lamella ok to lift-out

Keep in mind the aspect ratio (tall lamella $\rightarrow$ larger trench)

Polishing and thinning

30 kV 1nA (1 μm thickness) $\rightarrow$ 100 pA (100 nm thickness)

Overtilt to keep the surface parallel (most cases $\pm 2^\circ - 3^\circ$)

Grazing incidence 2-5 kV $\pm 5-7^\circ$ (1 - 3 min)

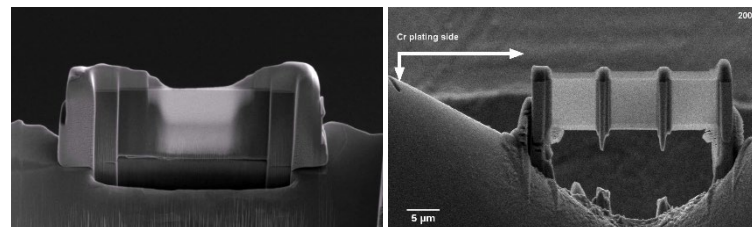
Pt. Deposition

First perform e-beam deposition (5 kV, 2000 pA)

Ion deposition 10-15 kV will be denser

Thick is good!

- Ion current = Area $\times$ 5 pA/ μm^2
- Pixel fill factor = 40 - 70 %
- Short dwell time (0.2 μs for Pt)

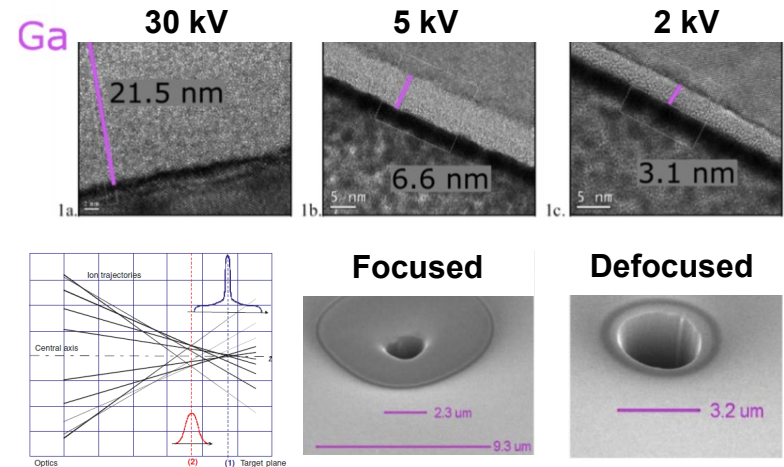


You can also attach the lamella on top of the grid instead of the side

Final tricks and tips

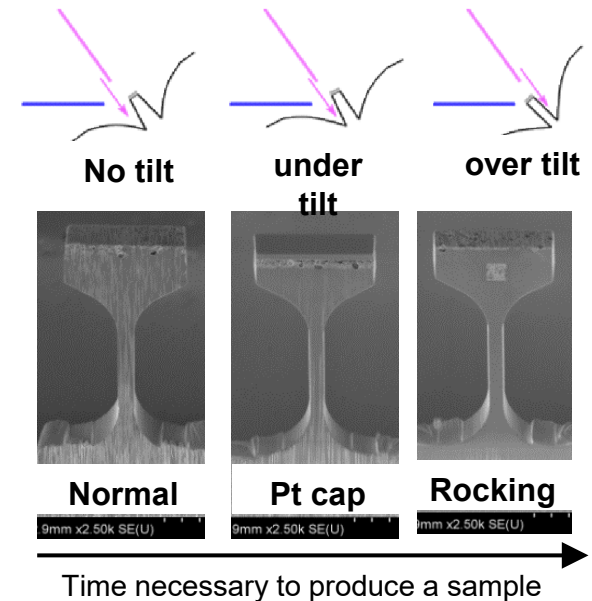
General tips:

- Small currents for high precision
- Large currents for speed
- Polishing for faster milling rate
- Defocus on high currents
- Tilt sample into beam for perpendicular cut
- Short dwell time for high control of shape/depth
- Long dwell time for hard materials /speed
- Several passes for nanopatterning to avoid redeposition
- Well tuned beam (e.g. dose and dwell time for GIS)



Pitfalls and possible solutions:

- **Damaged layer and ions implantation** → low kV
- **Tapering** → overtilting, reduce current
- **Curtaining** → protection cap, stage rocking
- **Redeposition** → Several milling steps, reactive gas



Laser ablation

Brief History of LASER

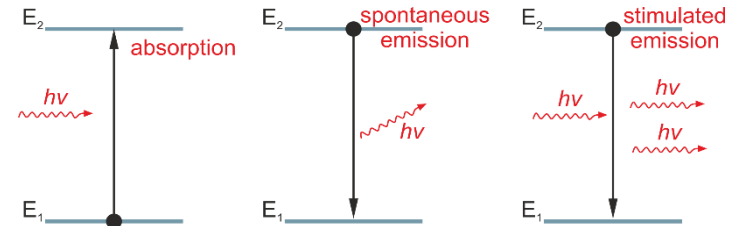
1917 *Zur Quantentheorie der Strahlung (Albert Einstein)*
→ stimulated emission of electromagnetic radiation

1928 Rudolf Ladenburg first observed stimulated emission

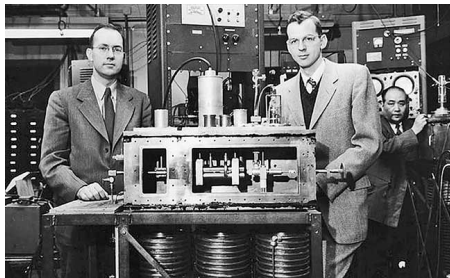
1950s Charles Townes (Columbia University) → MASER:
“microwave amplification by the stimulated emission of radiation.”

Gordon Gould (PhD @ CU) → LASER

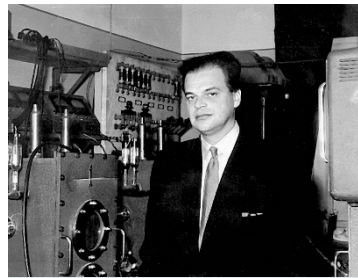
1964 Nobel Prize in Physics



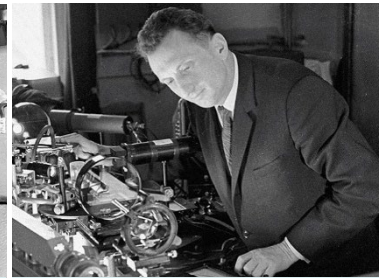
Albert Einstein and Rudolf Ladenburg
(Princeton Symposium, 1950)



Charles Townes and Gordon Gould



Nikolay Basov



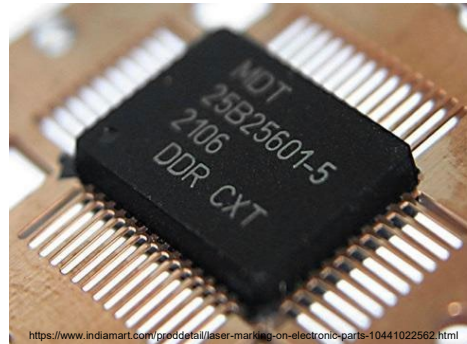
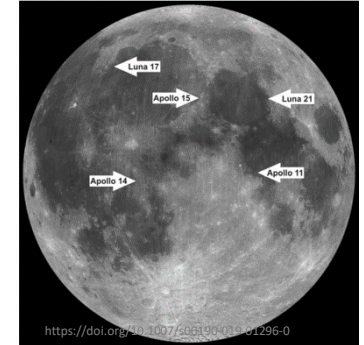
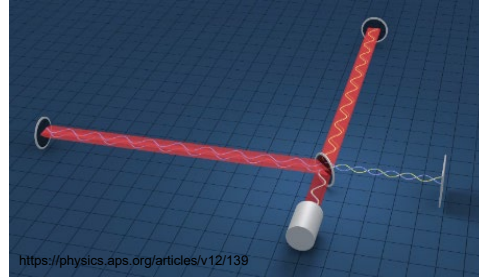
Aleksandr Prokhorov

1960s Hughes Research Laboratories in Malibu/ Bell Labs (New Jersey)/ General Electric Research and Development Center in Schenectady (New York) / ...

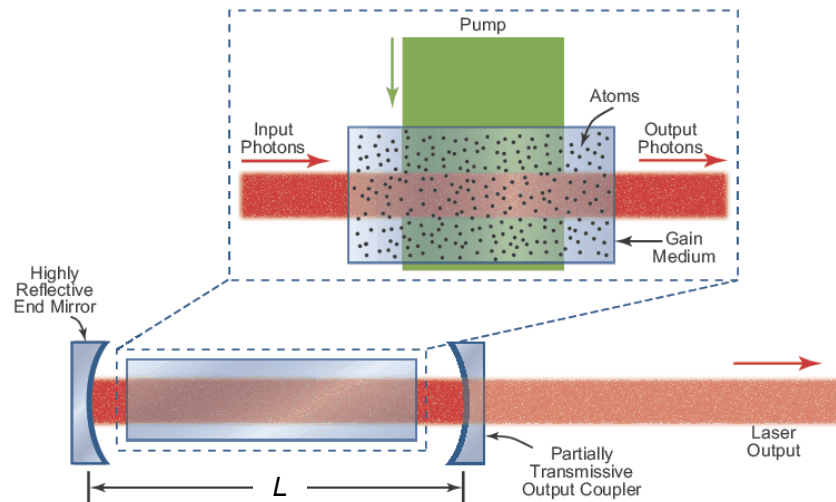
“... a solution looking for a problem”

Laser Applications

- Scientific
- Industrial
- Medical
- Military
- Commercial



Laser Physics



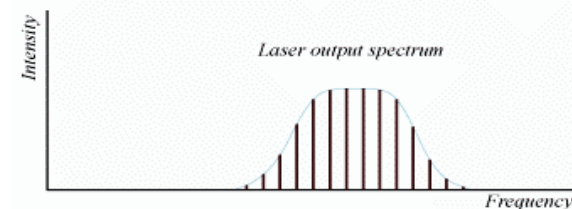
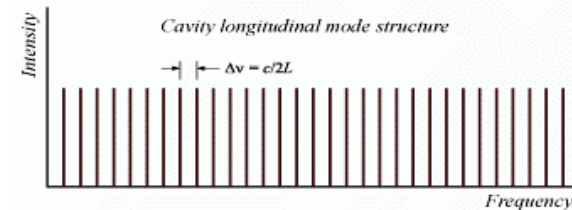
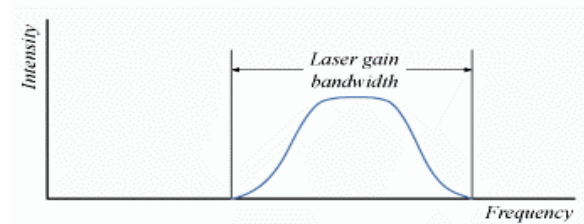
- **Optical resonator**
- **Pump** (optical/electrical/gas)
- **Gain medium**

Solid: doped crystals (Nd:YAG, $\text{Ti:Al}_2\text{O}_3$),
semi-conductors (InGaAs)

Gas: HeNe, N, Ar, CO_2

Liquid: dye solutions (rhodamine 6G)

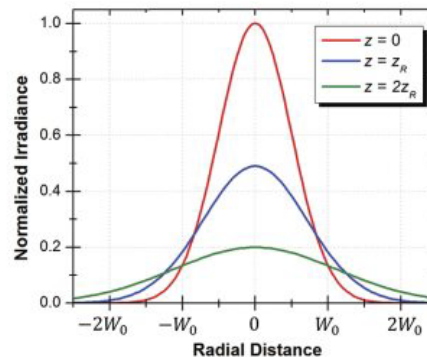
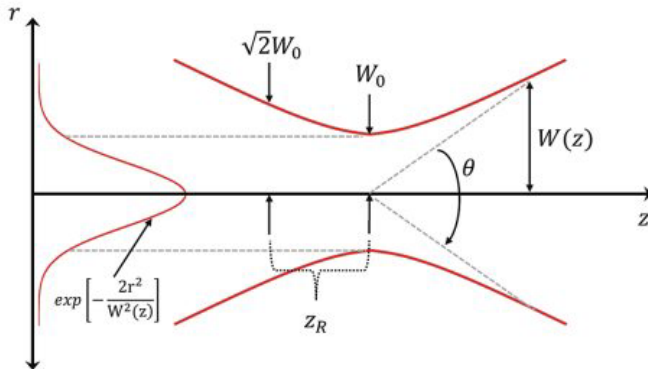
- Wavelength, λ
- Gain Bandwidth, B
- Output Power, P



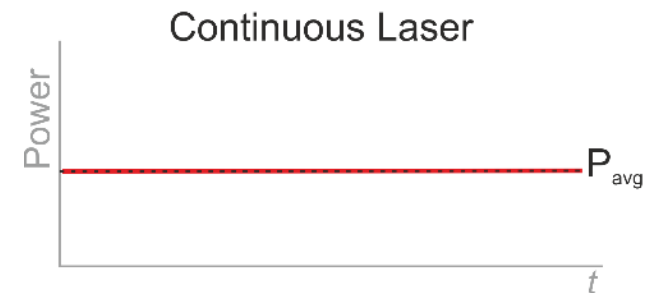
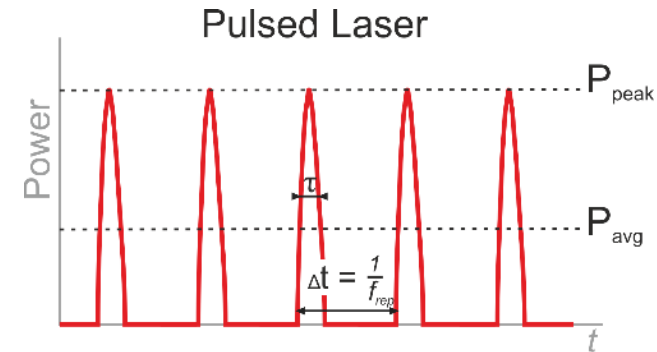
Laser Physics

- Continuous laser
- Pulsed laser (0.5 to 500 ns)
- Ultrashort pulsed laser (5 fs to 100 ps)

Gaussian laser beam profile



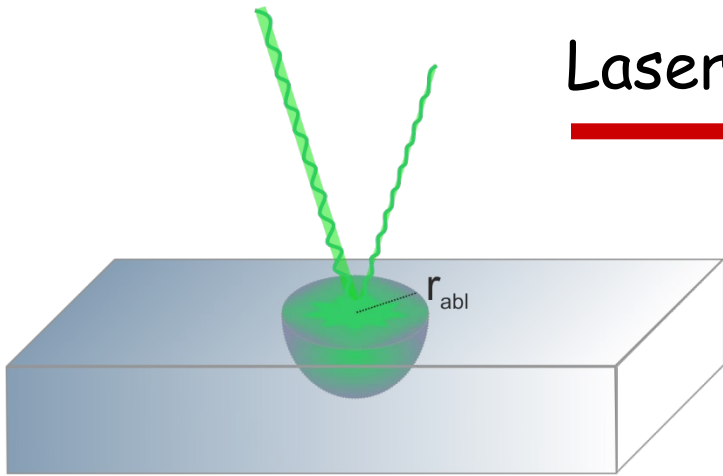
w_0 – beam waist radius



$$E_{pulse} = \frac{P_{avg}}{f_{rep}} [J]$$

$$P_{peak} = \frac{E_{pulse}}{\tau} [W]$$

Laser Ablation Principles



Laser-Induced Damage $\leftrightarrow$ Fluence [J/cm^2]

$$F_0^{peak} = 2 \frac{E_{pulse}}{\pi \omega_0^2}$$

$$F(r) = F_0^{peak} \exp\left(-\frac{2r^2}{\omega_0^2}\right)$$

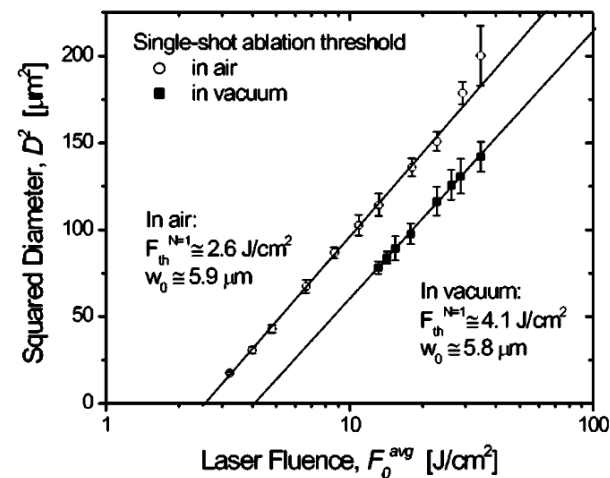
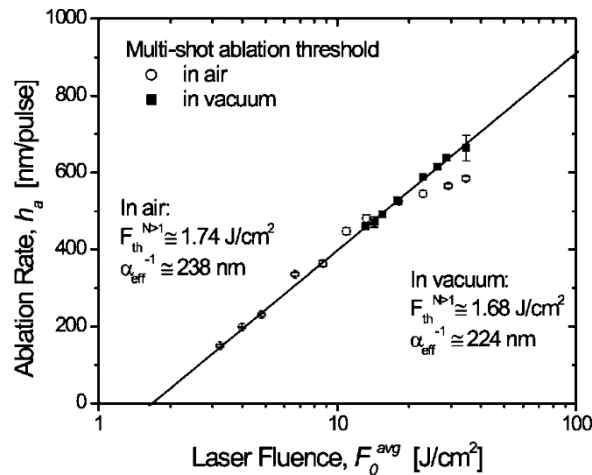
$$F_{th} = F_0^{peak} \exp\left(-\frac{2r_{th}^2}{\omega_0^2}\right)$$

Ablation rate [nm/pulse]: $h_a = \alpha_{eff}^{-1} \ln\left(\frac{F_0^{av}}{F_{th}^{N \geq 1}}\right)$ [1]

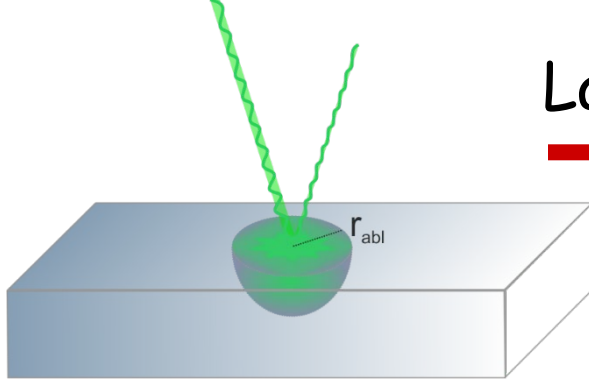
$F_0^{avg} = \frac{1}{2} F_0^{peak}$, α_{eff}^{-1} – effective optical penetration depth

Ablation "crater" diameter: $D^2 = 2w_0^2 \ln\left(\frac{F_0^{peak}}{F_{th}}\right)$

Fs-laser ablation properties of borosilicate glass [2]



Laser Ablation Principles



Interaction mechanisms x fs-laser ablation [3]

- 1) absorption of laser energy through photon-electron coupling (~ fs)
- 2) energy distribution to the lattice through electron-phonon coupling (~10 ps)
- 3) normal energy diffusion into the material through phonon-phonon collision

Laser-Induced Heat accumulation: **Two-temperature model (TTM)** [4]

$$C_e \frac{\partial T_e}{\partial t} = \nabla \cdot (k_e \nabla T_e) - g(T_e - T_p) + Q^*(x, y, z, t),$$

$$C_p \frac{\partial T_p}{\partial t} = \nabla \cdot (k_p \nabla T_p) + g(T_e - T_p)$$

[5] ablated volume $\leftrightarrow$ isolated mechanism
($T_e = T_p = T$)

$$(C_e + C_p) \frac{\partial T}{\partial t} = \nabla \cdot (k_e + k_p) \nabla T + Q(x, y, z, t)$$

[5] Bulk heat capacity: $(C_e + C_p) = \rho c$
Bulk thermal conductivity $(k_e + k_p) = k$

$$\rho c \frac{\partial T}{\partial t} = \nabla \cdot k \nabla T + Q(x, y, z, t)$$

e – electron / p – phonon

C, k – heat capacity and thermal conductivity

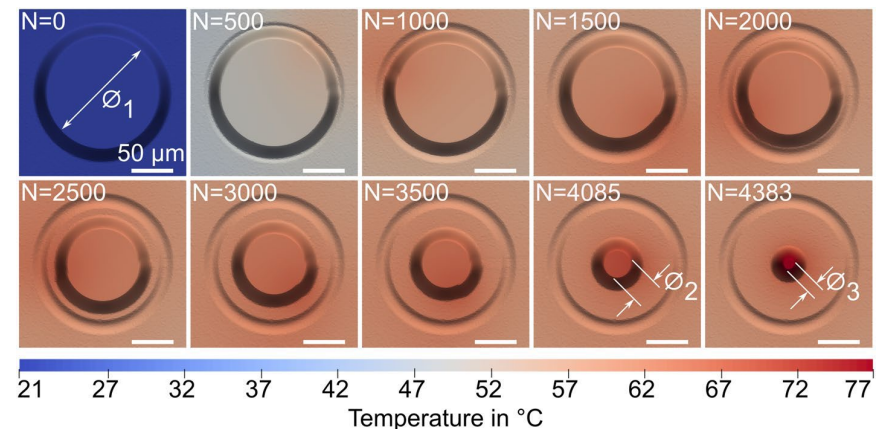
g – electron – phonon coupling

$Q(x, y, z, t)$ – energy absorption rate (spatial and temporal):

$Q(x, y, z, t) = S(x, y, z) \cdot T(t)$

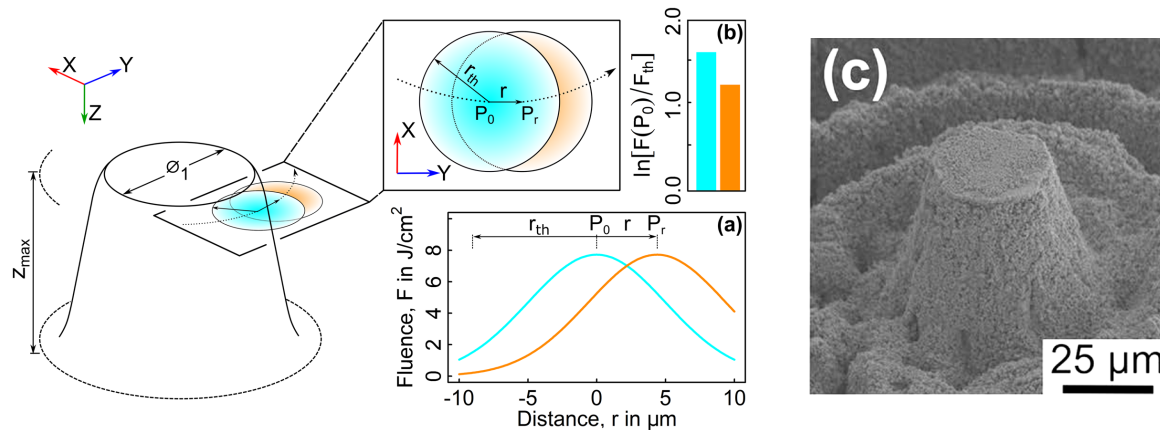
* – terms that vary between dielectrics and Me

FEM of temperature distribution during laser ablation extraction of mineralized tissue micropillar (final diameter ~12 μm) [5]

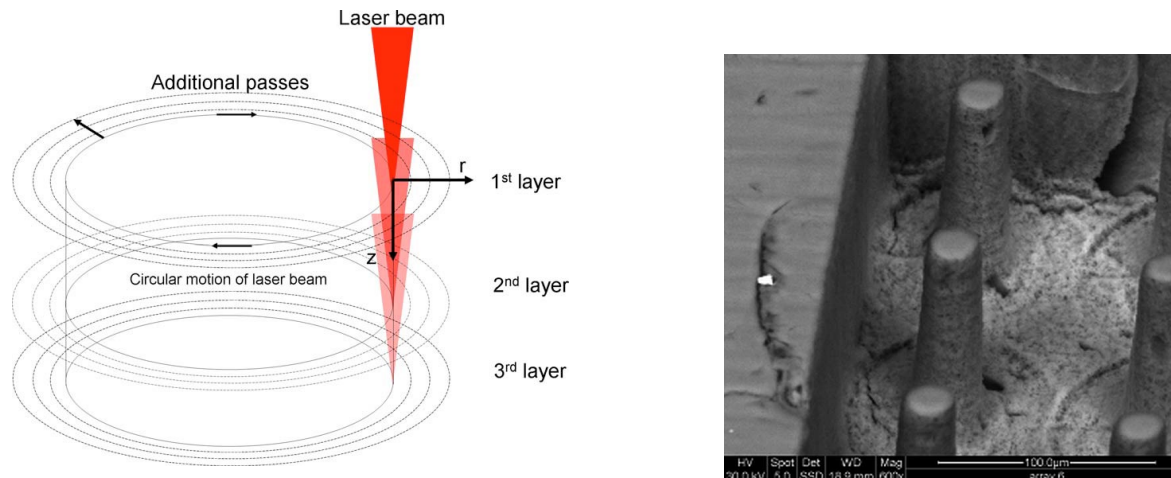


Laser Ablation for micropillar fabrication

Heat impact during laser ablation extraction of mineralised tissue micropillars [5]
(anticlockwise inward spiral pattern)

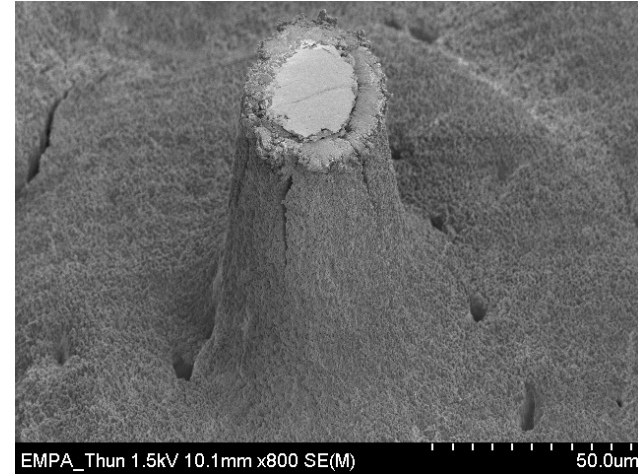
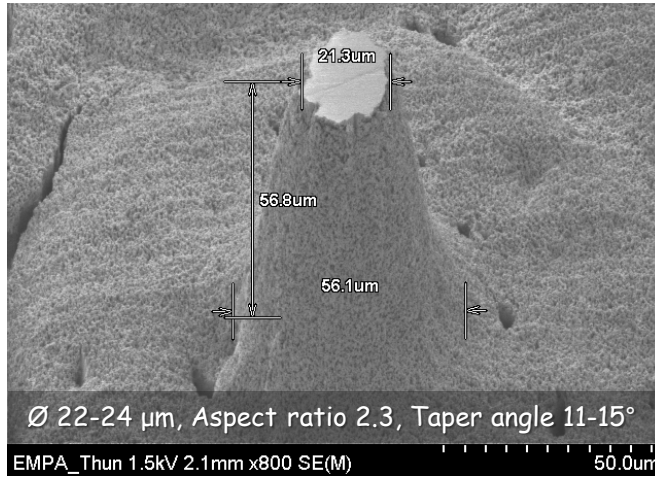


Micropillar fabrication on bovine cortical bone by direct-write femtosecond laser ablation [6]



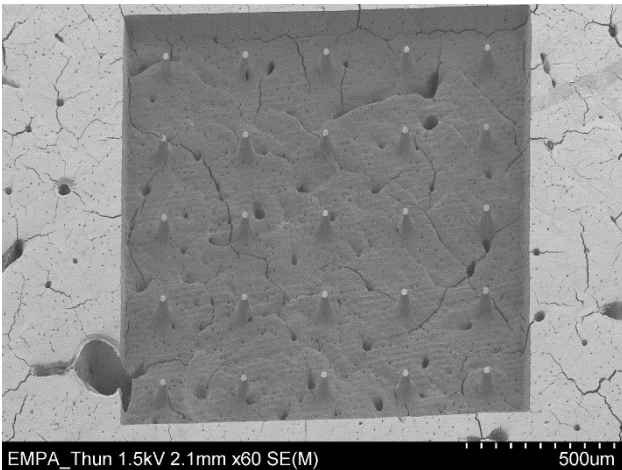
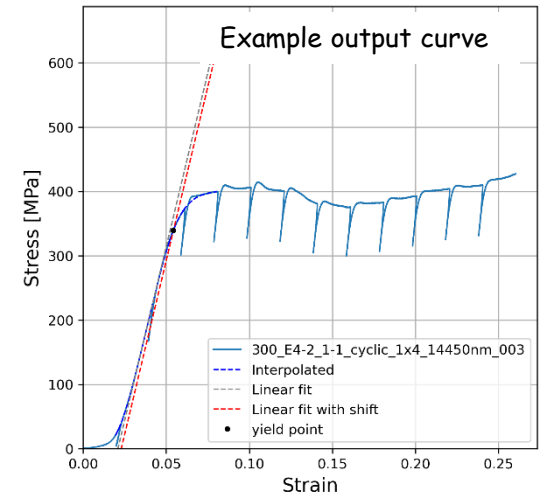
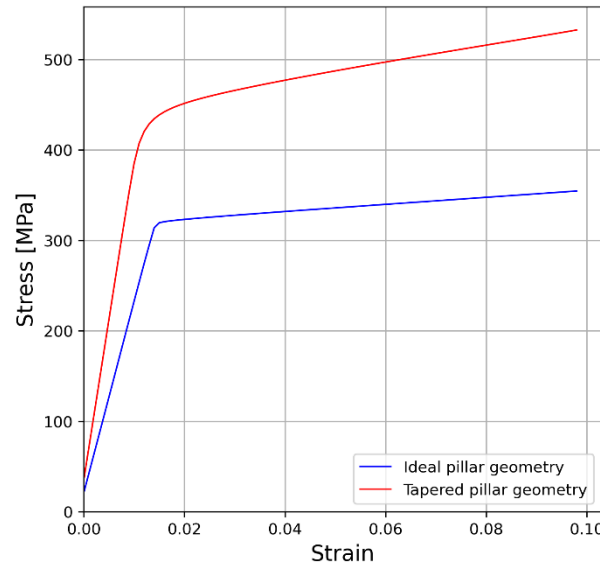
Micropillar compression

Scanning ablation @ALPS BFH :



- $\lambda = 515 \text{ nm}$
- $f_{\text{rep}} = 3 \text{ kHz}$
- $F_{\text{peak}} = 5.14 \text{ J/cm}^2$

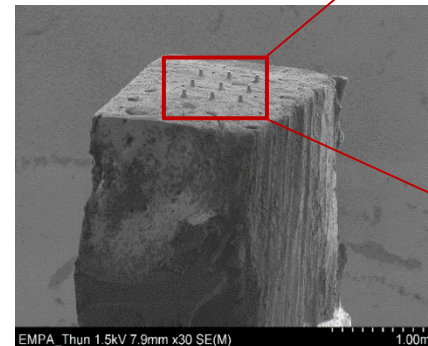
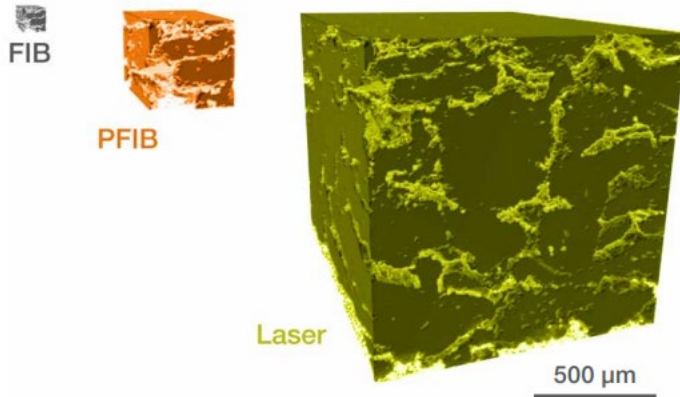
FEM of micropillar compression with different geometries



Laser Ablation for micropillar fabrication

	Ga ⁺ FIB	PFIB	Fs-Laser
Removal rate (Si)	~18 $\mu\text{m}^3/\text{s}$ 1x	~675 $\mu\text{m}^3/\text{s}$ 38x faster	~265000 $\mu\text{m}^3/\text{s}$ 15000x faster
Spot size	50nm	100nm-10 μm	10-20 μm

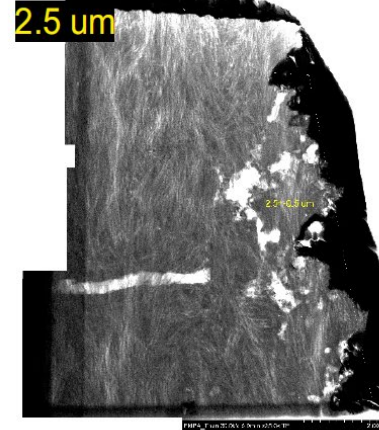
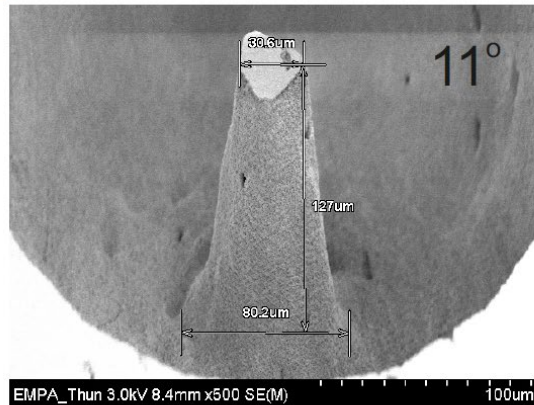
Comparison of 3D volumes milled within the same amount of time with FIB, Plasma FIB, and femtosecond laser



Surface damage

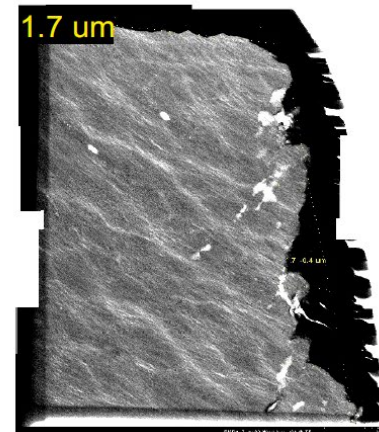
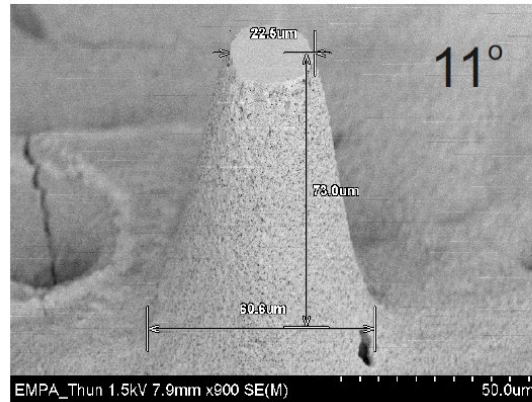
Spiral :

1W@5kHz



Bitmap:

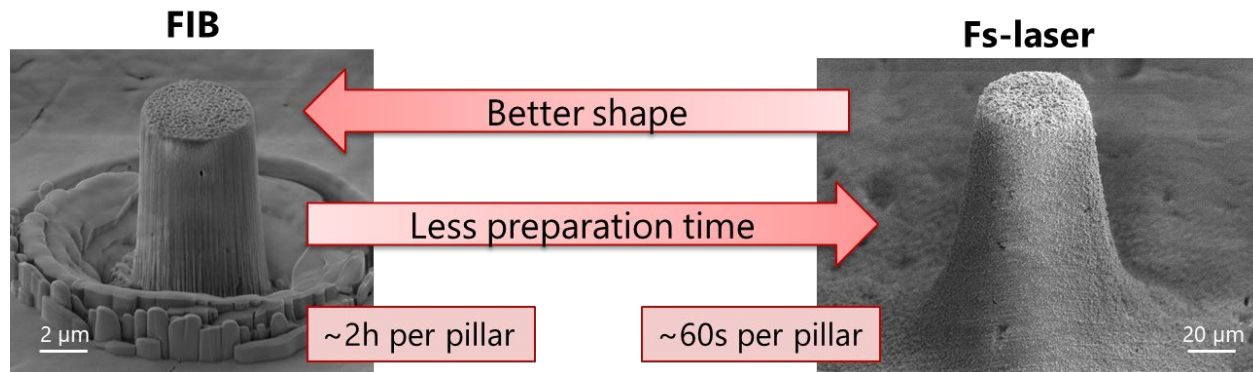
2W@3kHz



Laser Ablation for micropillar fabrication

take-home message:

- Choosing laser system for ablation (CW/pulsed)
- ★ Ultrashort pulsed laser (<100 ps)
- Choosing laser parameters for ablation (λ , τ , f_{rep} , F^{pulse} , N)
- Ways to improve micropillar geometry (focal height adjustment, beam waist radius, beam profile)

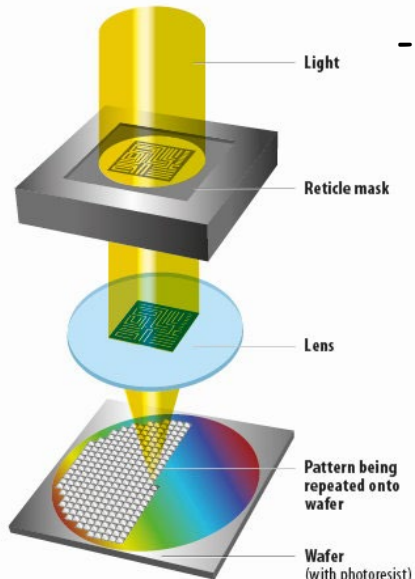


Lithography for 3D nanoscale metamaterials

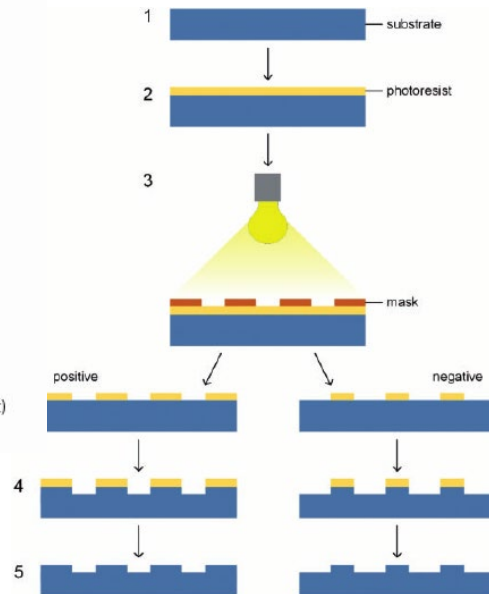
Basic idea

3 basic ingredients:

-light
-mask
-resist

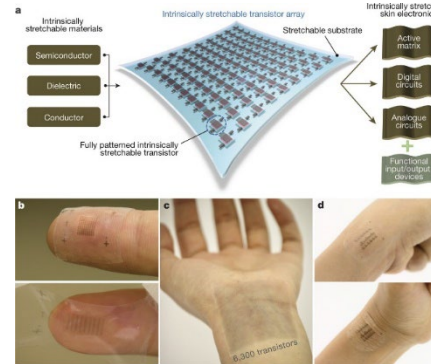


Categories

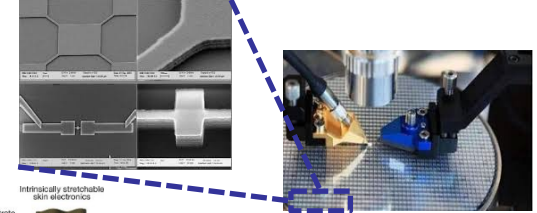


Applications

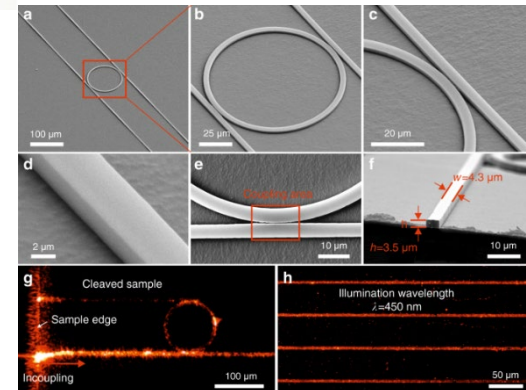
Bio-electronics



Integrated electronic circuits

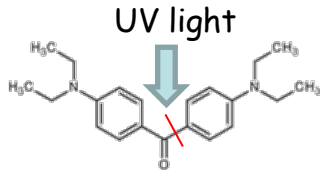


Photonic structures

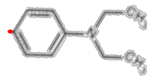


Principle of photolithography

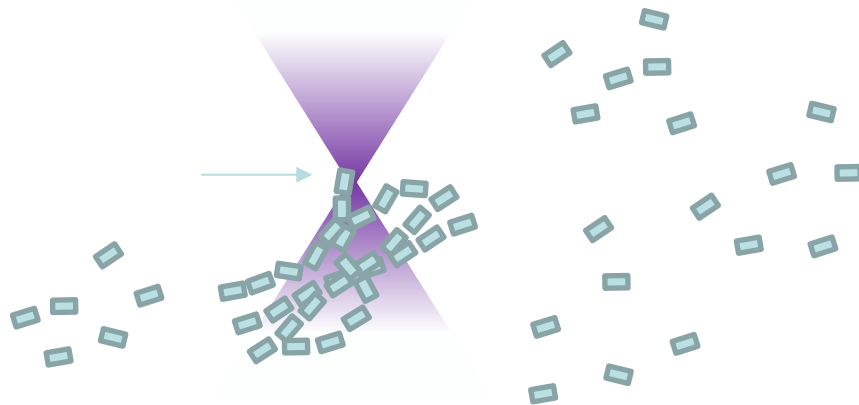
Chemistry
Photo-initiator



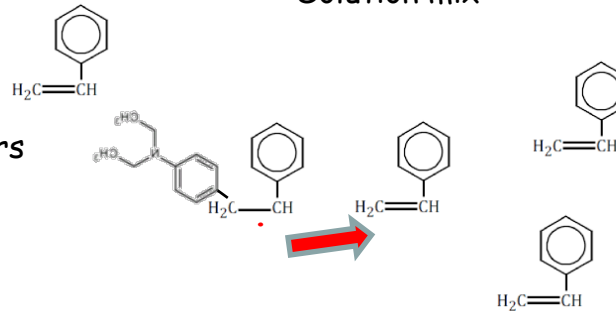
Free radicals attack monomers



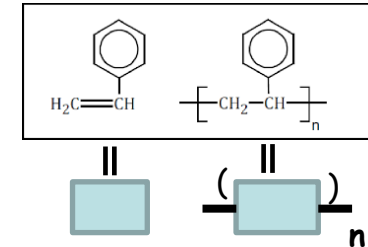
UV light



Solution mix



Monomer Polymer



Drawbacks

-2D patterns only

-Mask required for different patterns, increases the cost

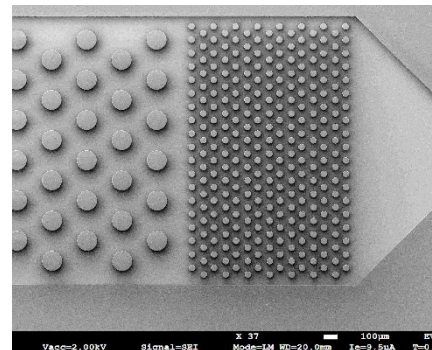
-Resolution: diffraction limited $d = \frac{\lambda}{2n \sin \theta} = \frac{\lambda}{2NA}$

$\lambda=500\text{nm}$ can be focused only down to 250nm

-Limited variety of designs

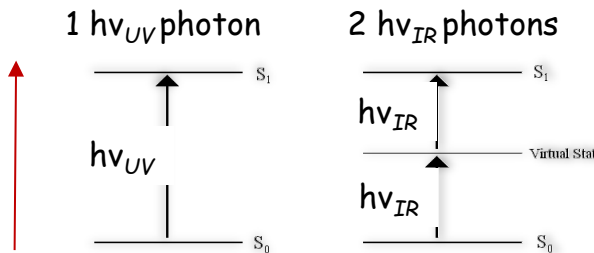
-Limited Opto-mechanics

High quality patterns

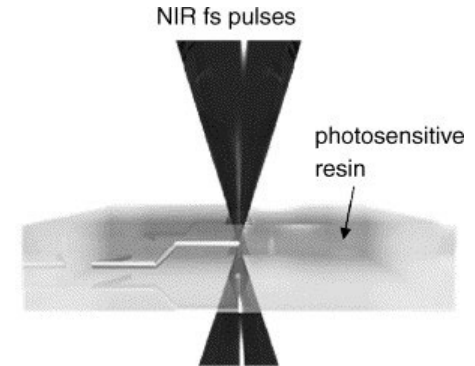
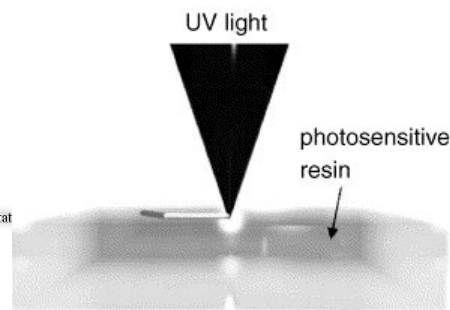


Single photon vs multiphoton

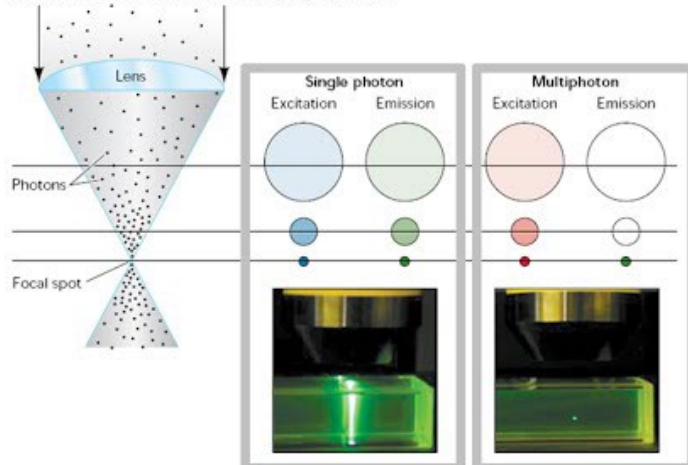
Energy needed for polymerization



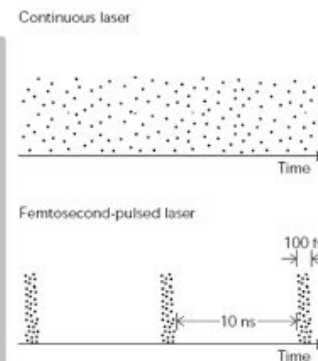
Volumetric Direct Laser Writing (DLW)



A Spatial compression of photons by objective lens



B Temporal compression of photons during femtosecond pulses



Essential and potential

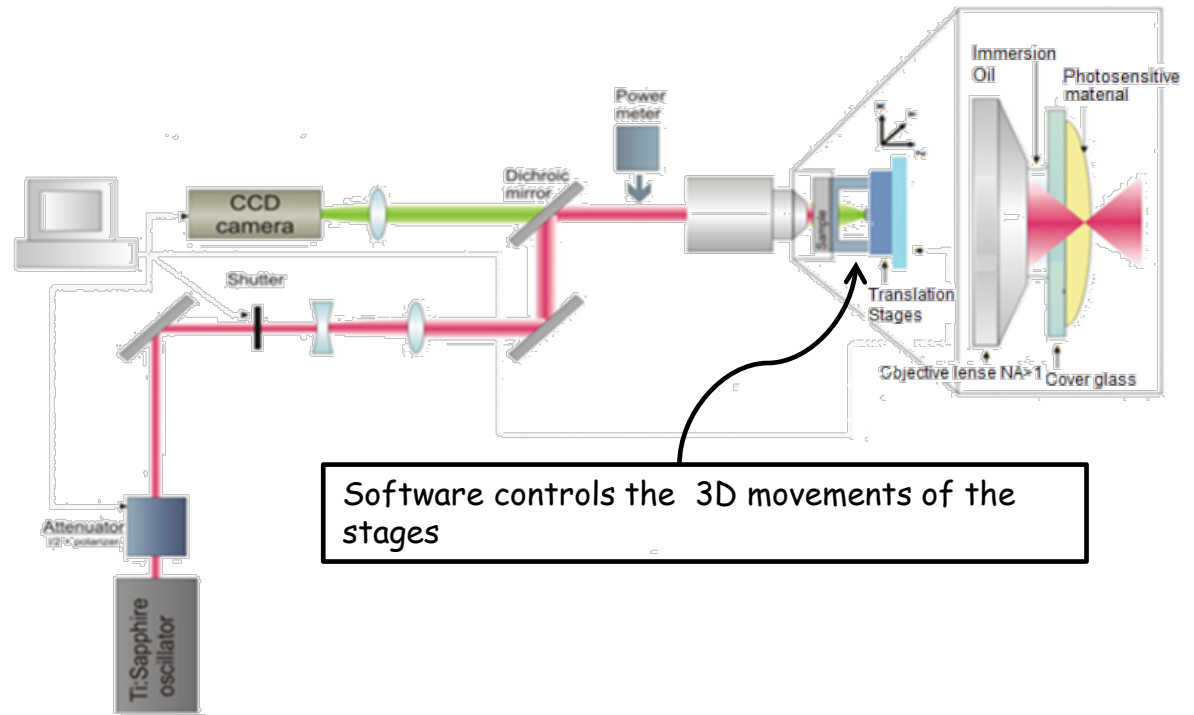
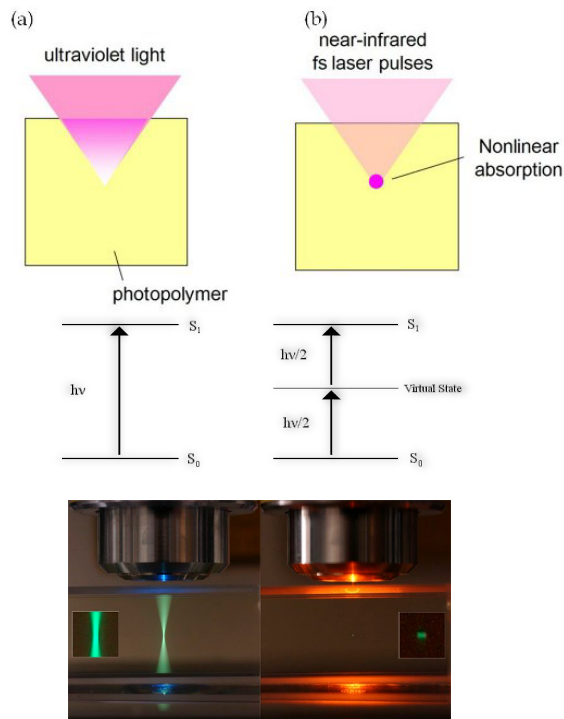
- Ultrafast laser (pulses shorter than virtual state lifetime)
- 3D patterns
- Resolution around 100nm
- Maskless
- Big variety of designs
- Relatively slow

Setup/instrumentation

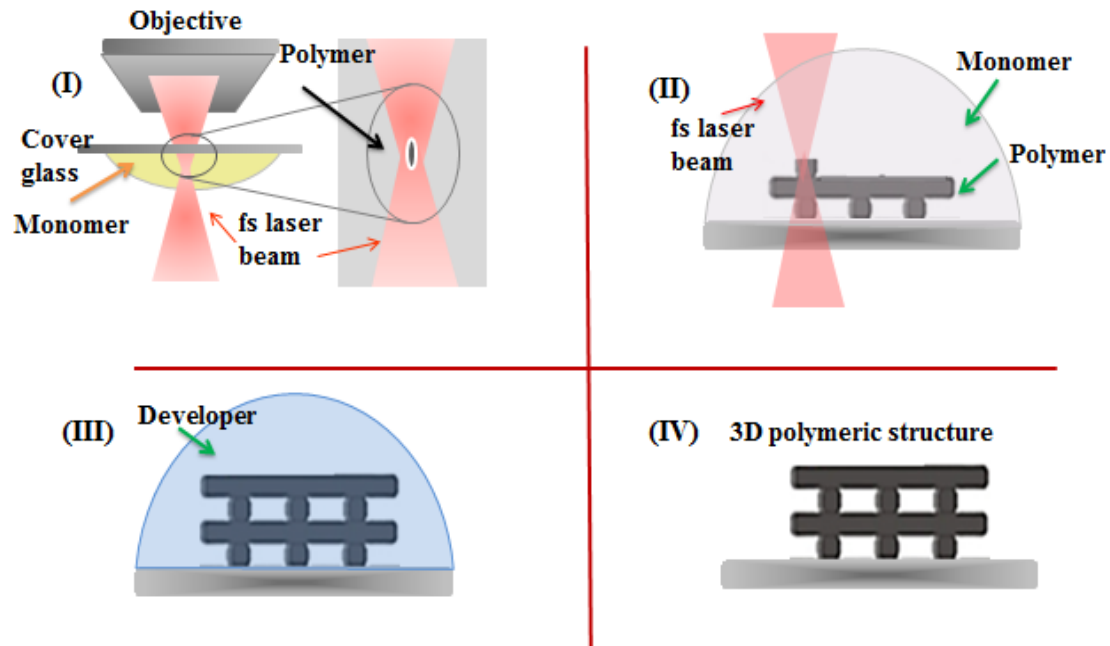
Setup

Ti: Sapphire Femtosecond Laser (800nm, 75MHz, <20fs)

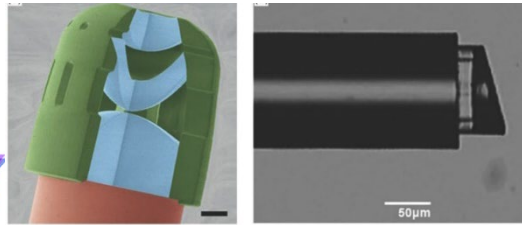
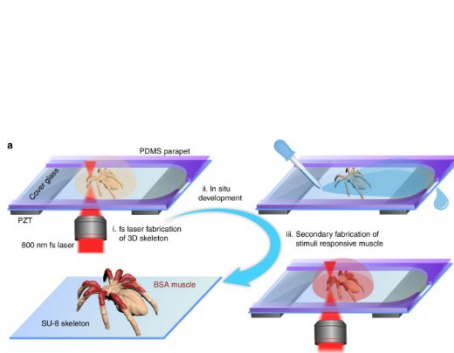
Direct Laser Writing



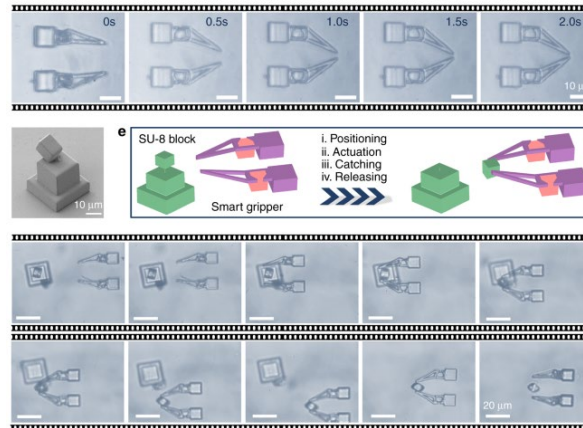
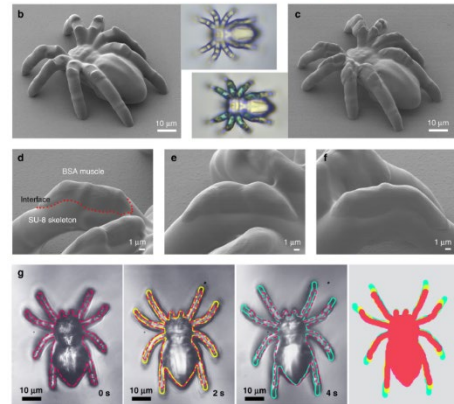
Fabrications steps



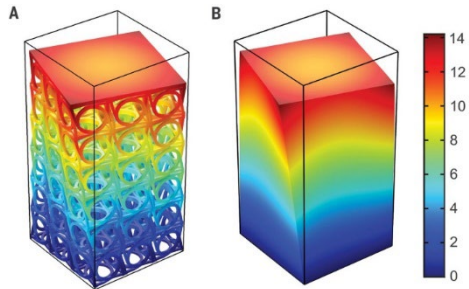
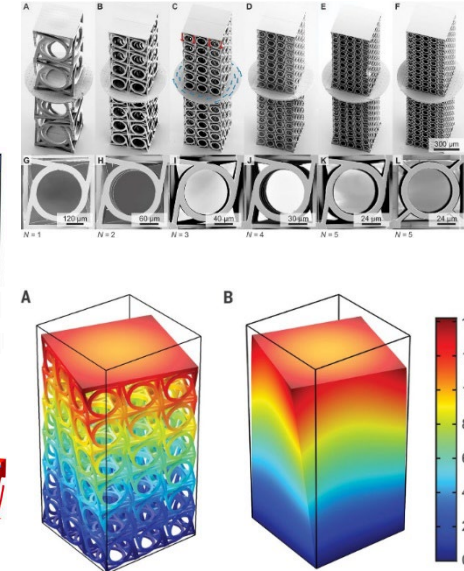
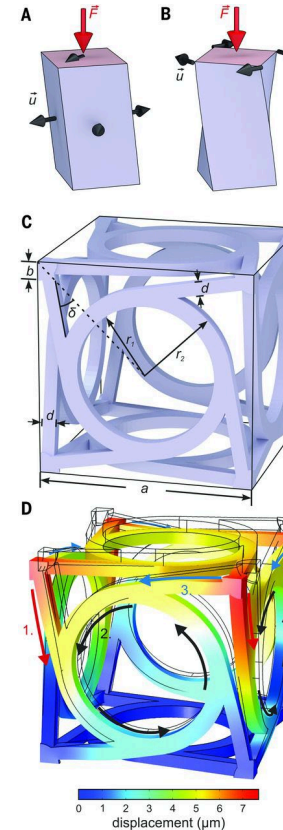
Possibilities



Adv. Opt. Mat. 7, 1800419 (2019)



Nature Commun. 11, 4536 (2020)



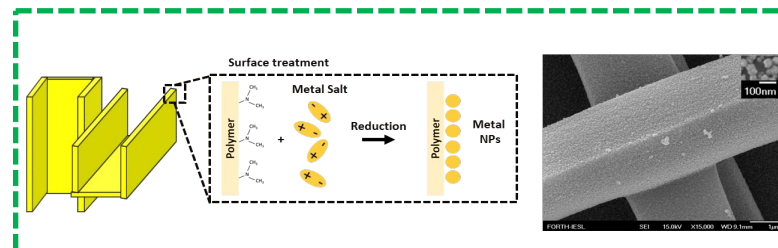
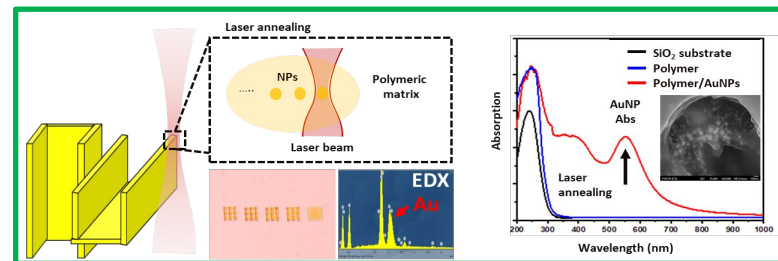
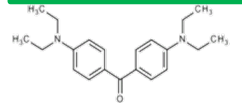
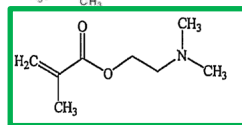
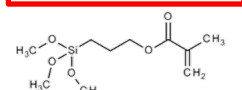
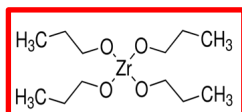
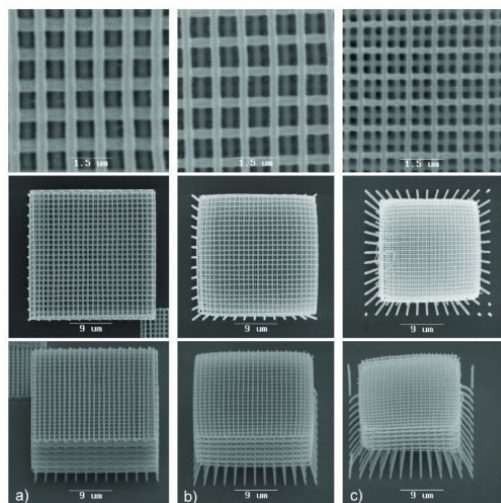
Science 358, 6366, 1072 (2017)

Actuation:

Photo, Thermal, pH, hydrophobic/ hydrophilic, abnormal mechanics

Improve/Advance materials I

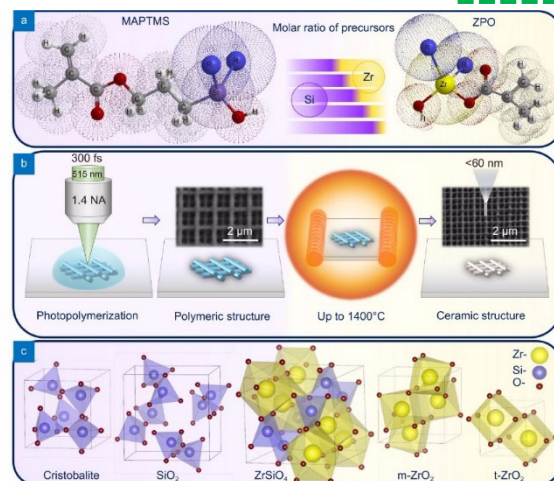
Selective plating of polymeric structures (binding of Au/Ag nanoparticles)



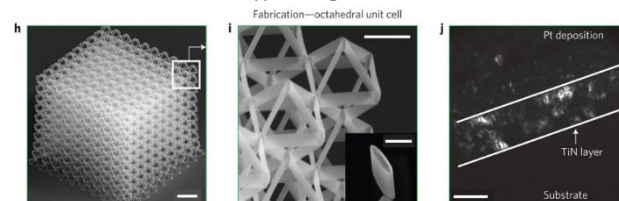
ACS Photonics 2, 287-294 (2015)
Adv. Opt. Mat. 5, 1700200 (2017)

Increasing % of inorganic material

Opt. Express 17, 2143 (2009)



Hollow ceramic 3D structures

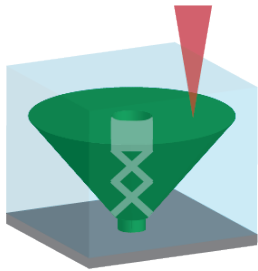
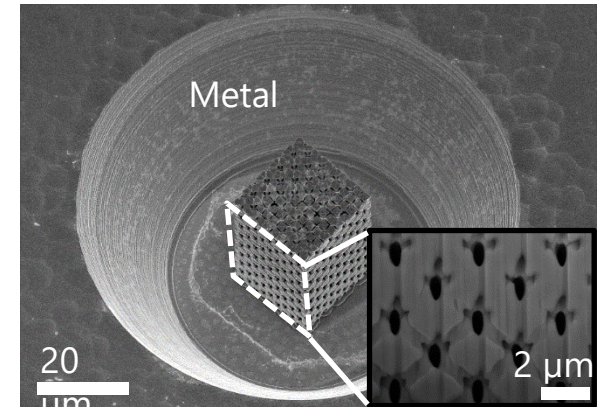
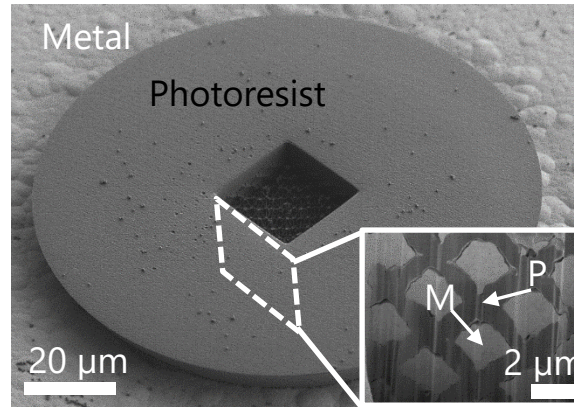
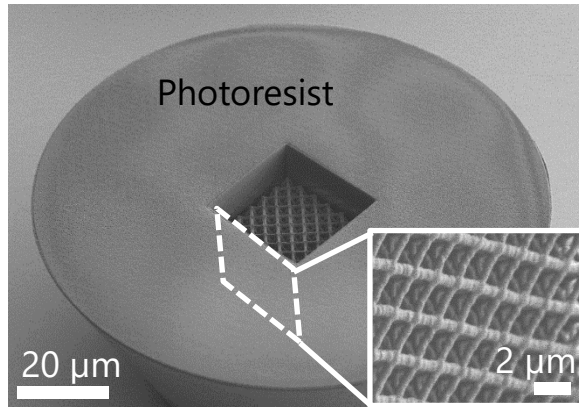


Nature Materials 12, 893 (2013)

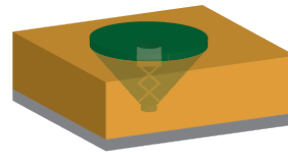
From photo resist to ceramic
Opto-Electron Adv X, 210077 (2022)

Improve/Advance materials II

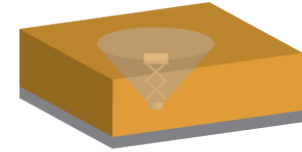
Template assisted electrodeposition:



Two-Photon
Lithography



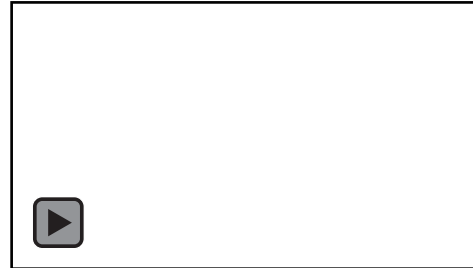
Electrodeposition



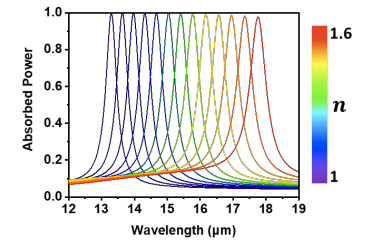
Plasma
Etching

Equipment & possibilities at Empa

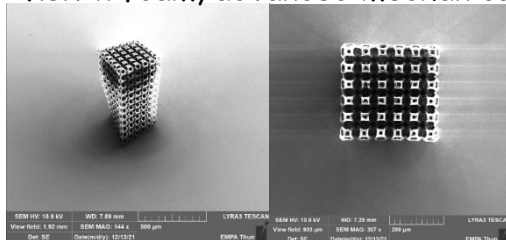
High resolution 3D printer from Nanoscribe



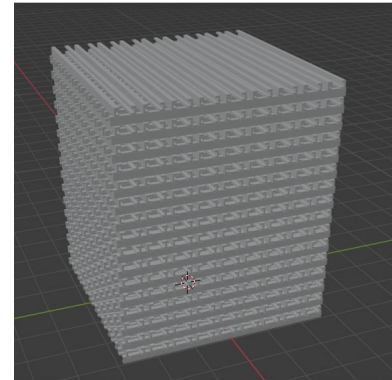
Metamaterials with enhanced light-matter interactions



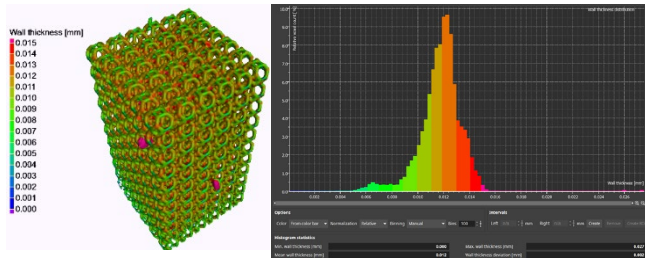
Kelvin foam, advanced mechanical properties



1mm tall
<1hr

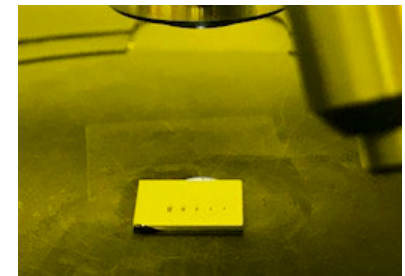
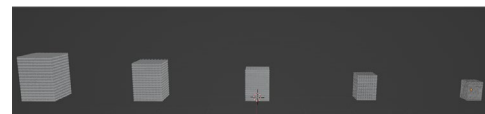


ALD deposition to increase opto-mechanics

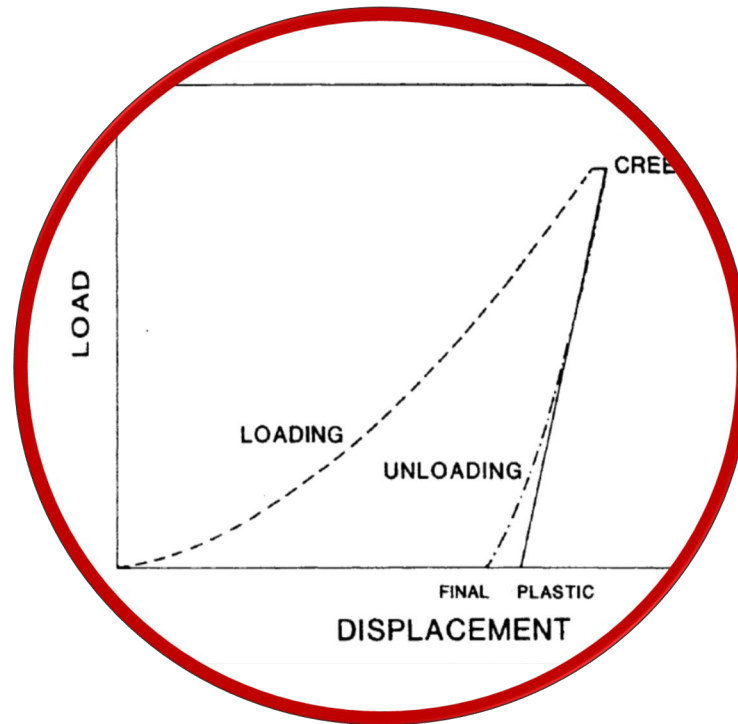


Nano-CT characterisation

Scaling



Nanoindentation



Nanoindentation

1. Definition of hardness
2. Principles of instrumented indentation
3. Load and displacement: actuation and measurement
4. What happens in the material?
5. Mechanical materials properties: beyond hardness and Youngs modulus

Hardness & Young's modulus

hardness: historical definition

Hardness	Mineral
1	Talc
2	Gypsum
3	Calcite
4	Fluorite
5	Apatite
6	Orthoclase
7	Quartz
8	Topaz
9	Corundum
10	Diamond

Mineralogist:
Metallurgist:
Machinist:

resistance to scratching
resistance to penetration
resistance to cutting



Gypsum
Calcite
Fluorite
Apatite
Orthoclase
Quartz
Topaz
Diamond

1812:

Mohs scale of mineral hardness

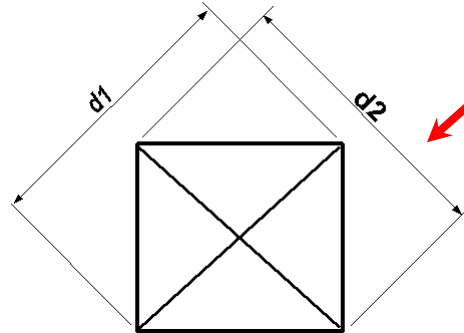
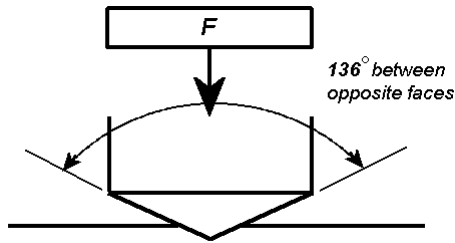
(German mineralogist Frederick Mohs (1773-1839))

Selected the ten minerals because they were common or readily available.

The scale is not a linear scale, but somewhat arbitrary.

Vickers Hardness Test

Diamond indenter body
(Vickers pyramid)



Residual impression
measured
by optical microscopy

$$HV = \frac{2F \sin \frac{136^\circ}{2}}{d^2} \approx 1.854 \cdot \frac{F}{d^2}$$

Load in kgf
d in mm

1 kgf = 9.81 N

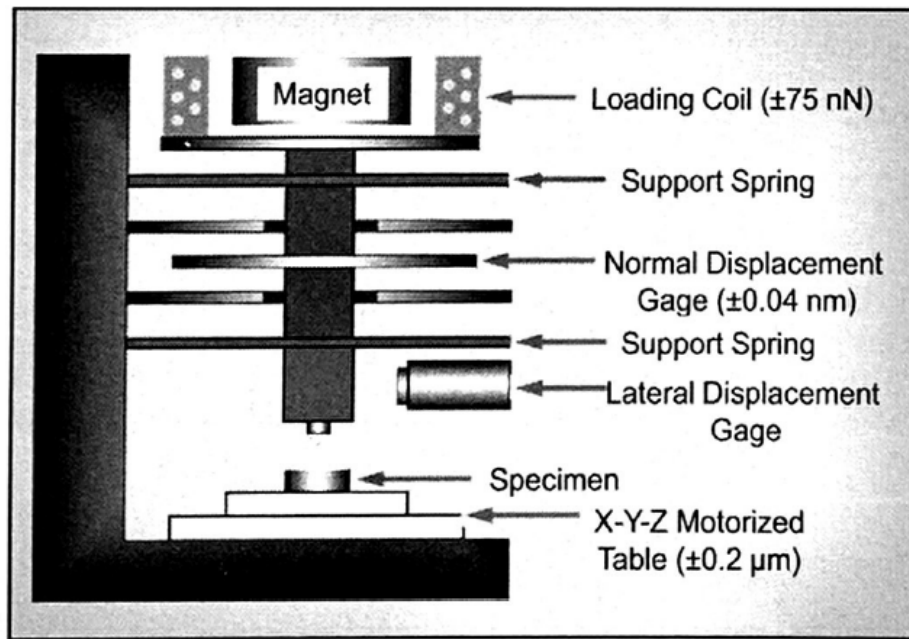
$$HV = 0.1891 \cdot \frac{F}{d^2}$$

Load in N
d in mm

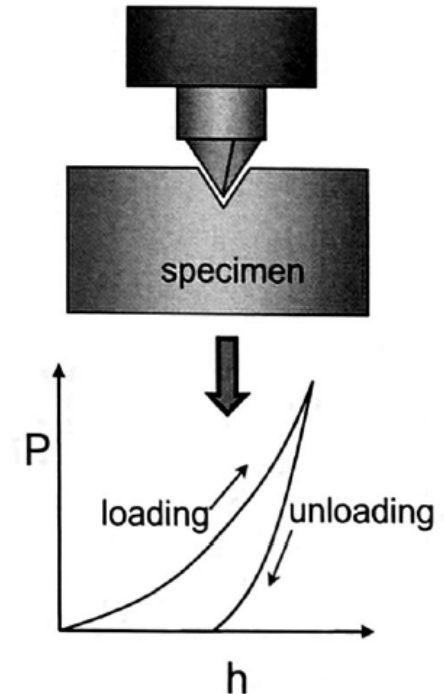
Advantage: „Easy“

Disadvantage: Operator needed for optical measurement
„NOT SUITED“ FOR SMALL OBJECTS OR THIN FILMS

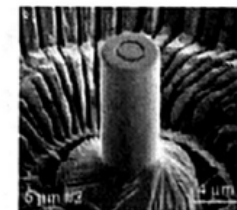
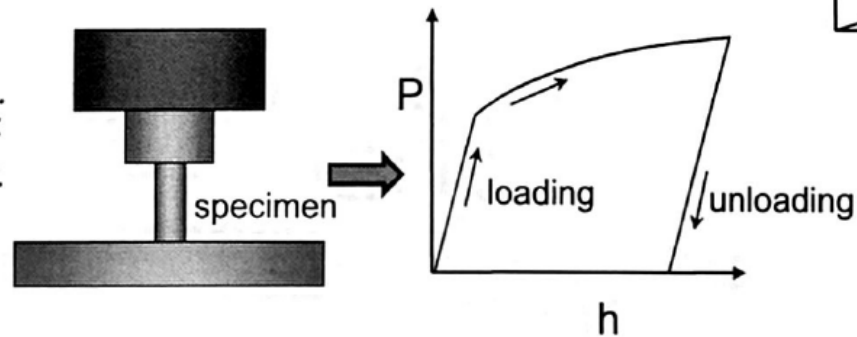
A nanoindenter



Indentation:

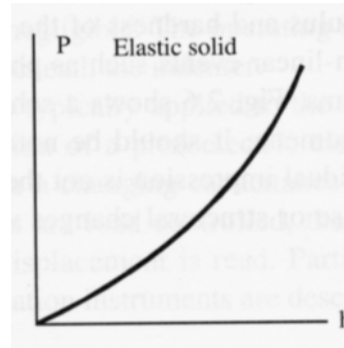


Pillar Testing

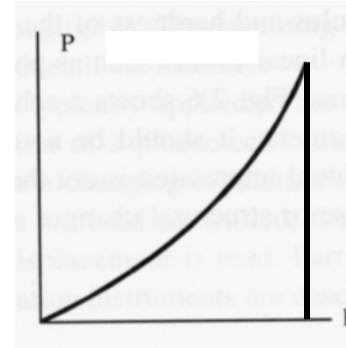


Load-Displacement curves: Typical shapes

Extreme cases:



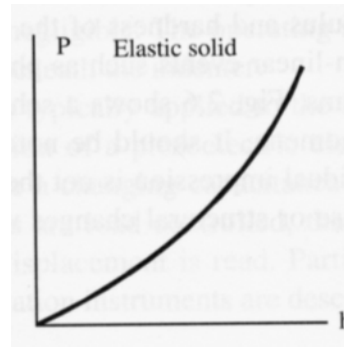
Elastic Solid



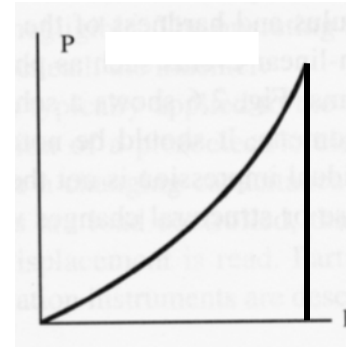
Fully plastic

Load-Displacement curves: Typical shapes

Extreme cases:

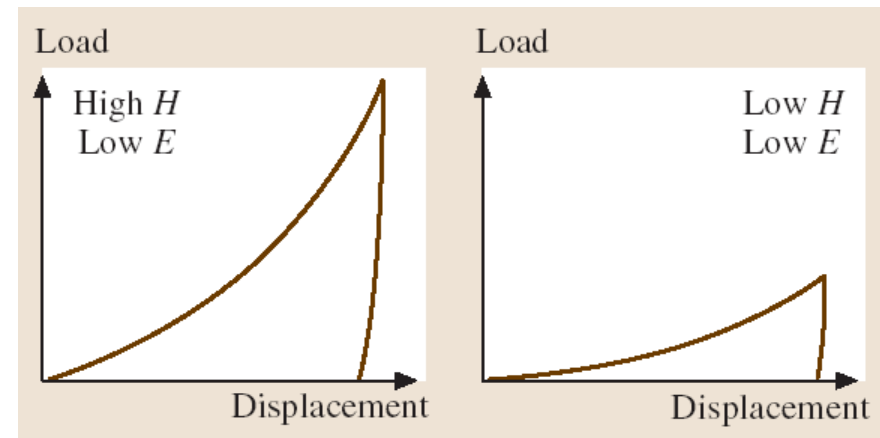
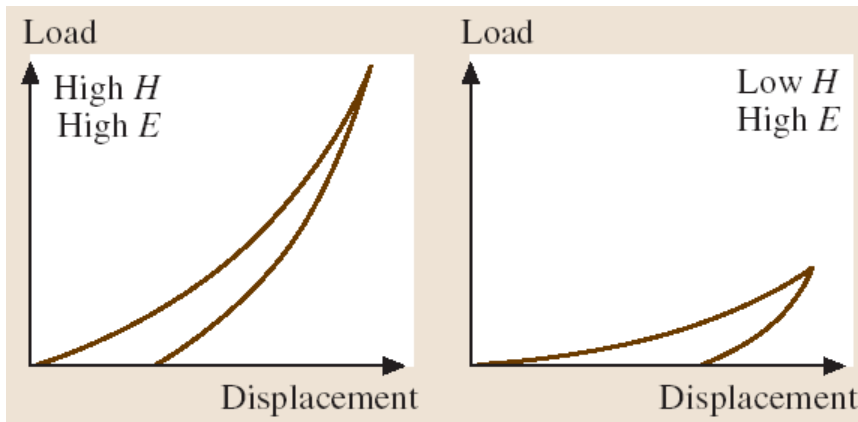


Elastic Solid



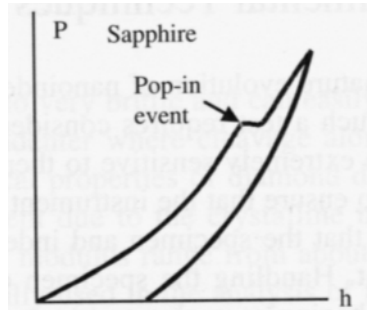
Fully plastic

Typical cases:

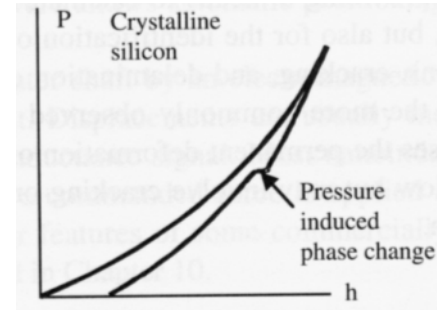


- High hardness $\Leftrightarrow$ High load needed for a given displacement
- Young's modulus depends on the shape of the unloading curve

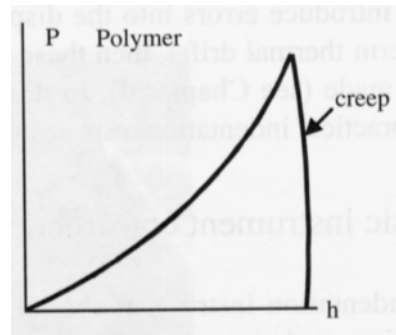
Load-Displacement curves: Typical shapes



Brittle Solid
Cracking during loading

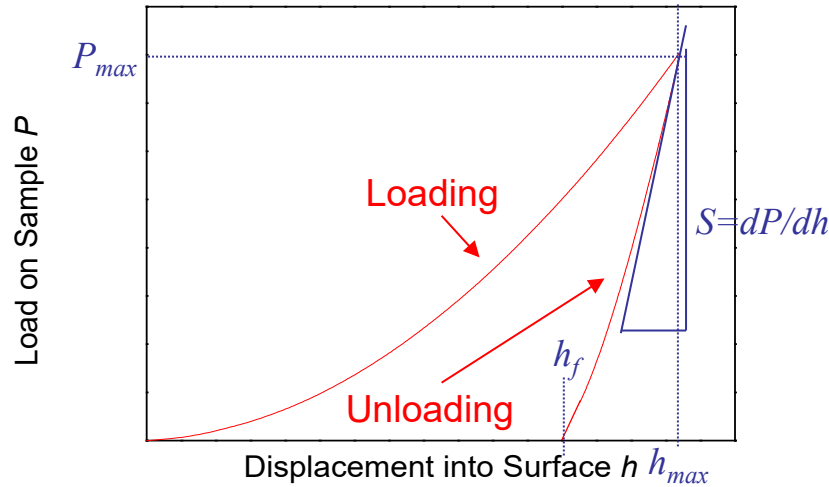


Pressure induced phase change
(Silicon)



Polymer exhibiting creep

Principle of nanoindentation



hardness:

Average contact pressure at maximum load
(projected area)

Young`s modulus:

proportional to slope of unloading curve

Basic equations

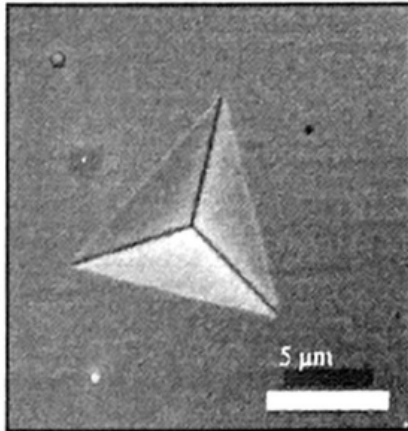
$$E_r = \frac{\sqrt{\pi} \cdot S}{2 \cdot \sqrt{A}}$$

$$\frac{1}{E_r} = \frac{(1-\nu^2)}{E_{Indentation}} + \frac{(1-\nu_i^2)}{E_i}$$

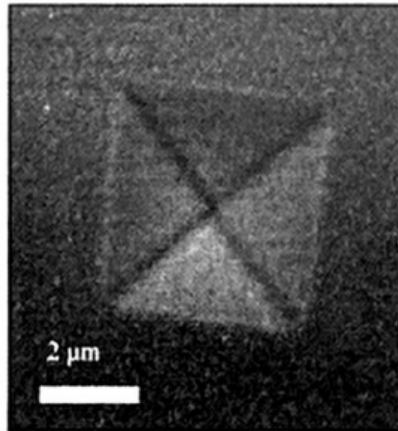
$$H = P/A$$

What determines the contact area?

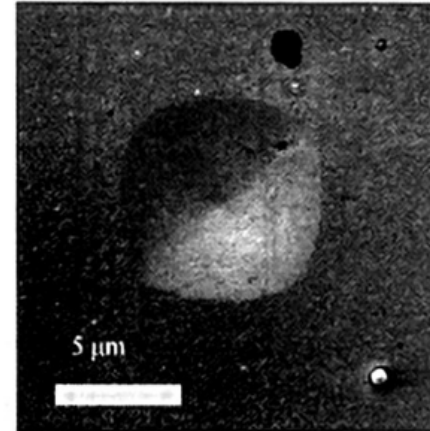
indenter geometries



Berkovich



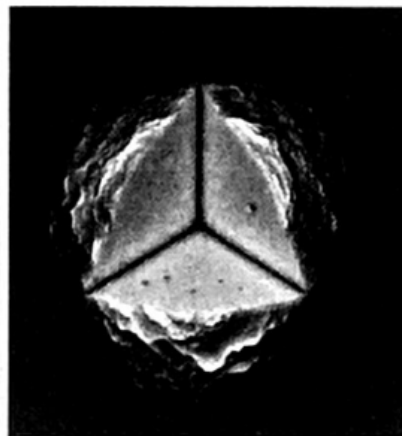
Vickers



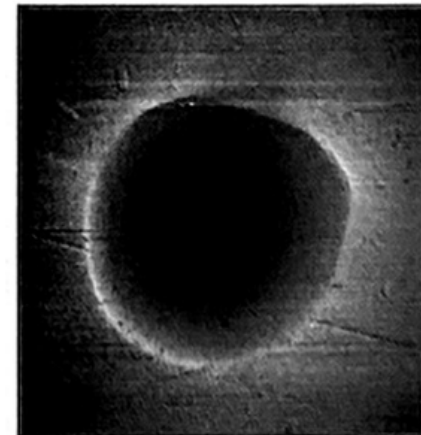
Conical



Berkovich
indenter

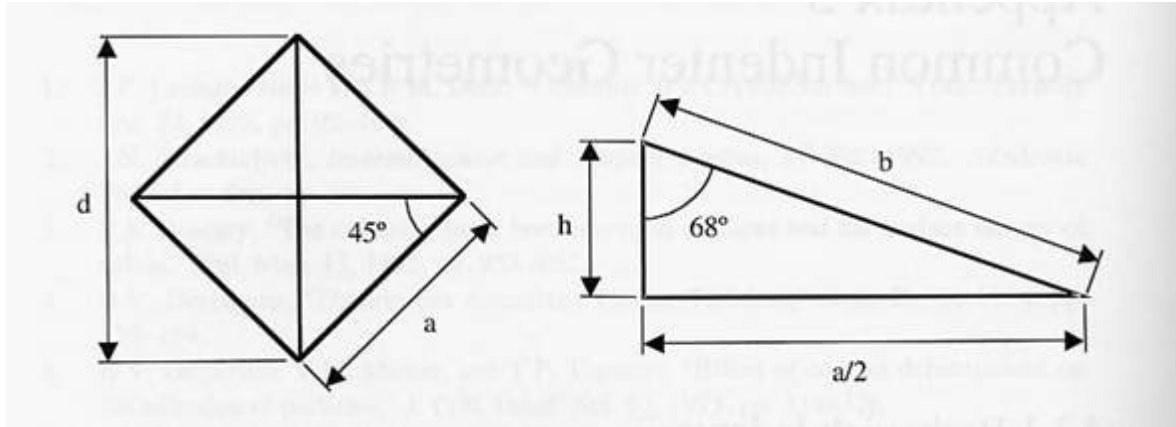


Cube-corner



Spherical

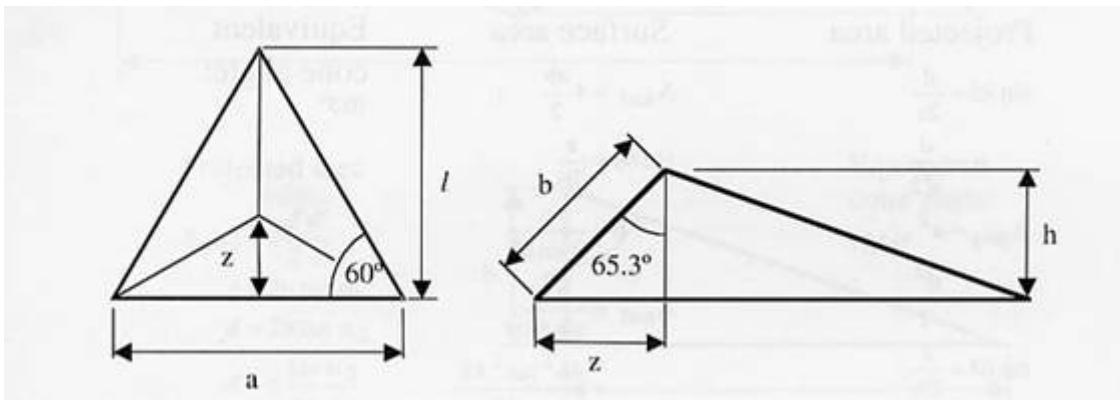
Tip area function assuming ideal geometries



Vickers:

$$A_{\text{proj}} = 24.504 h^2$$

$$A_{\text{surf}} = 26.429 h^2$$



Berkovich:

$$A_{\text{proj}} = 24.56 h^2$$

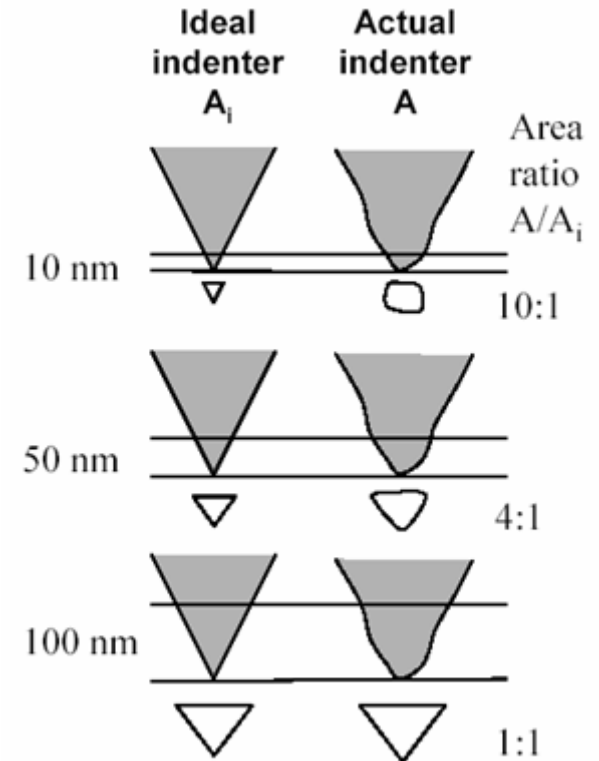
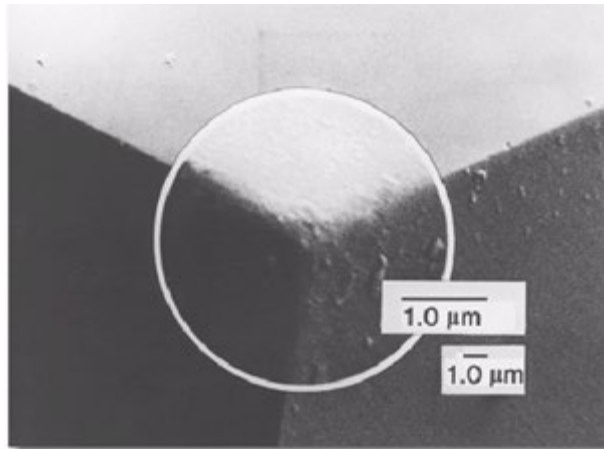
$$A_{\text{surf}} = 27.05 h^2$$

Determination of the tip area function $A(h_c)$

Projected contact area needed for the determination of H and $E_{\text{Indentation}}$ is calculated using the displacement into the surface
Crucial point: Exact tip geometry has to be known for this procedure!!

For small indentation depths ($<1\mu\text{m}$) the assumption of having an ideal tip is not valid.

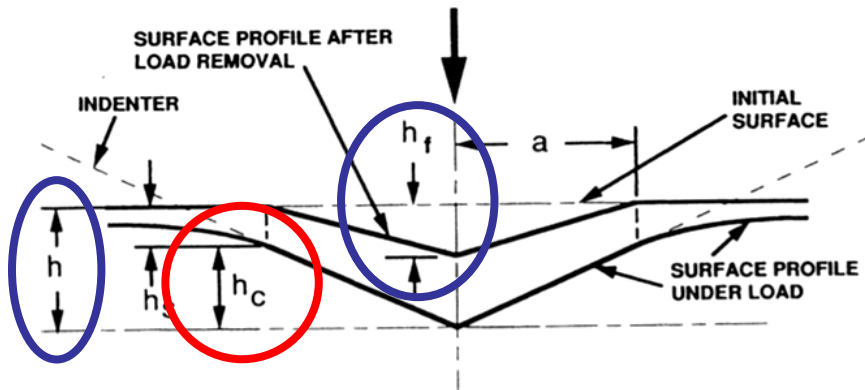
Berkovich: tip roundness between 20 and 100 nm



=> Tip area function

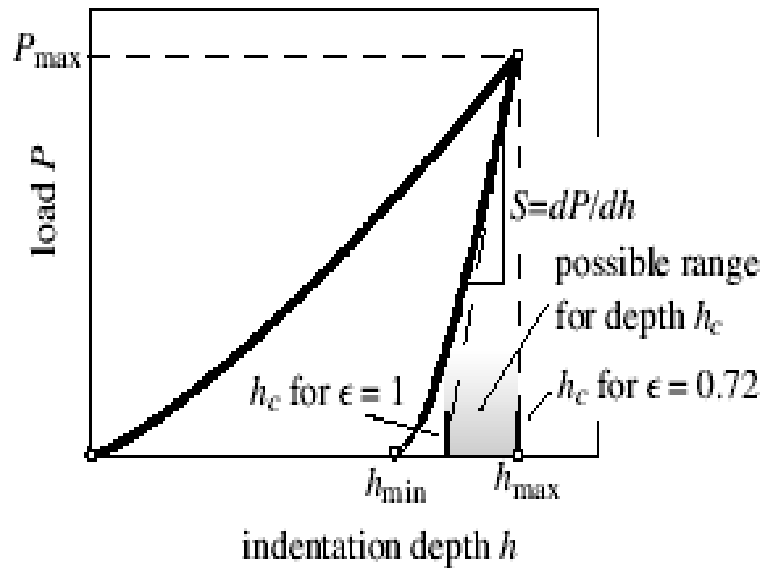
$$A(h_c) = a_0 \cdot h_c^2 + \sum_{i=0}^n a_{i+1} \cdot h_c^{1/2^i}$$

Contact depth



Take into account elastic sink-in!

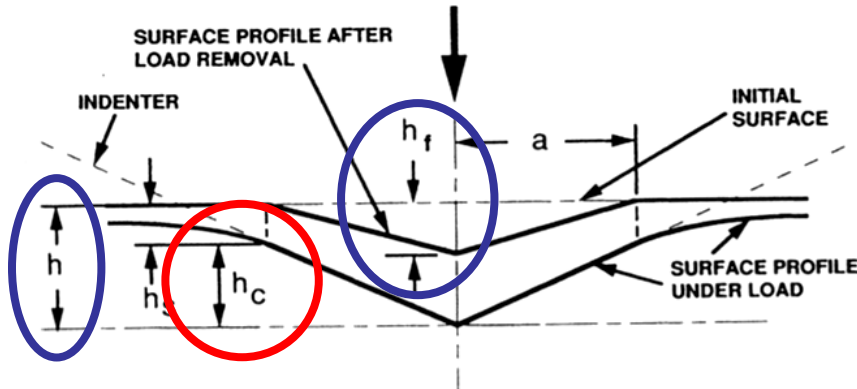
h maximum depth
 h_f residual depth
 h_c contact depth



$$h_c = h - \epsilon \frac{P_{\max}}{dP/dh}$$

ϵ is geometry factor to adapt
 "Flat Punch" model to Berkovic indenters

Summary: evaluation of load displacement curves



Fit unload curve

$$P = B(h - h_f)^m$$

$$S = dP/dh \longrightarrow S = Bm(h_{\max} - h_f)^{m-1}$$

Projected contact area

$$h_c = h - \varepsilon P/S$$

$$A(h_c) = a_0 \cdot h_c^2 + \sum_{i=0}^n a_i \cdot h_c^{1/2^i}$$

a_i is determined from measurements on reference materials (Fused Silica)

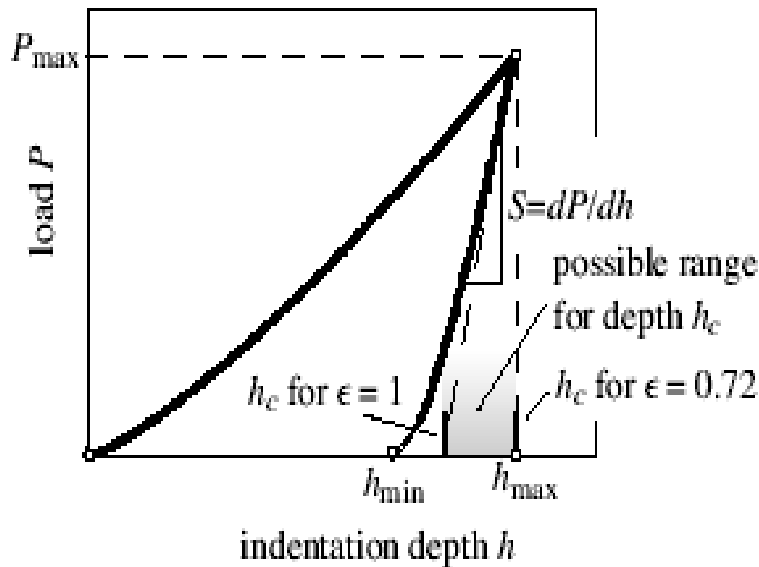
Ideal Berkovich tip: $a_0=24.5$ / $a_i=0$ für $i>0$

Summary H and E

$$E_r = \frac{\sqrt{\pi} \cdot S}{2\beta \cdot \sqrt{A(h_c)}}$$

$$\frac{1}{E_r} = \frac{(1-\nu^2)}{E_{\text{Indentation}}} + \frac{(1-\nu_i^2)}{E_i}$$

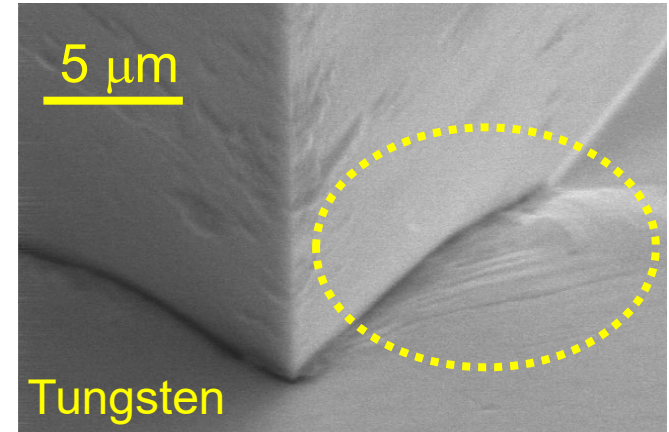
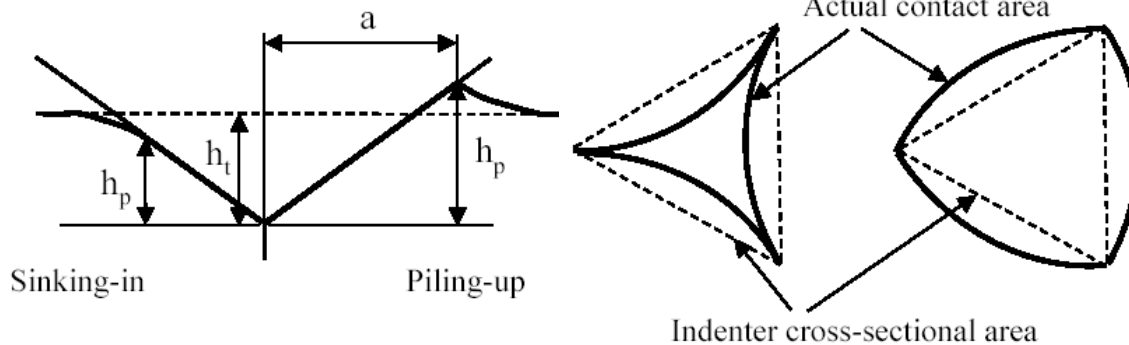
$$H = P/A(h_c)$$



ε, β are geometry factors to adapt flat punch model to Berkovic geometry

Limit of the model: Pile-up and Sink-in

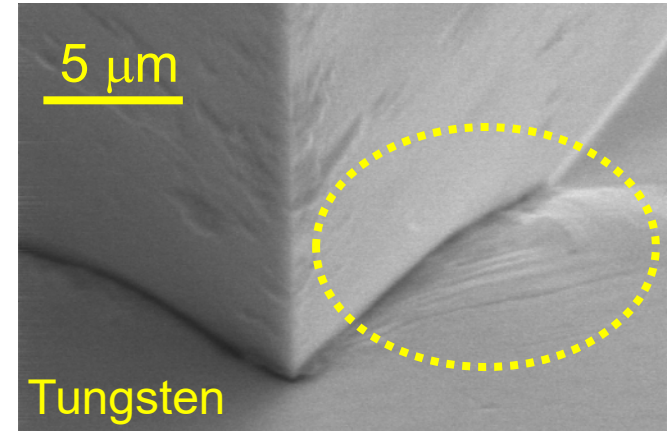
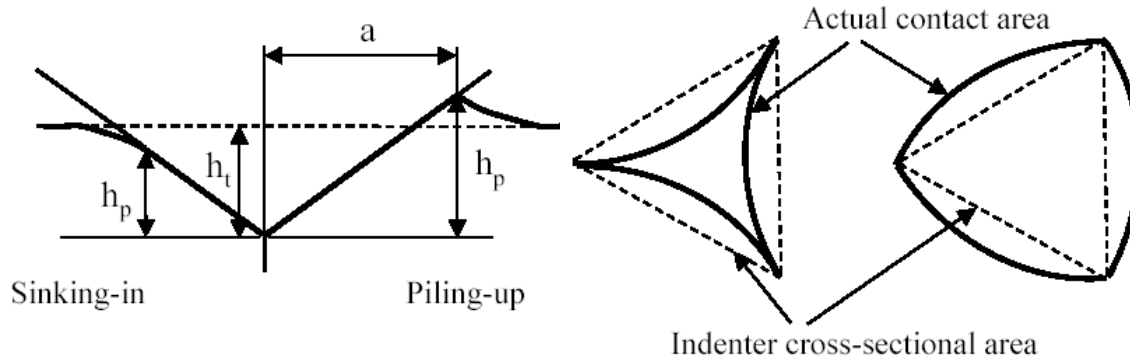
Depending on strain hardening behavior,
pile-up or sink-in phenomena influence the contact depth value
=> errors in contact area => errors in hardness and modulus



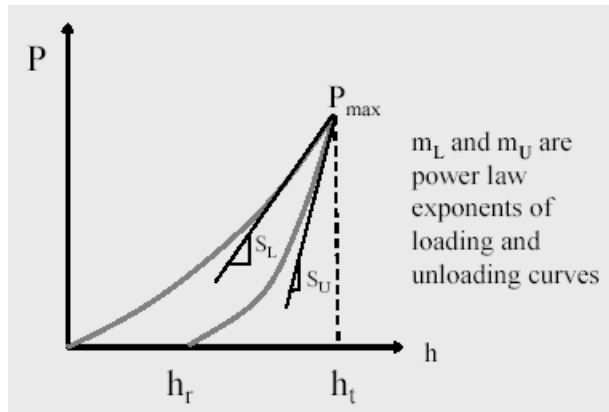
SEM image of cube corner
indent into tungsten

Limit of the model: Pile-up and Sink-in

Pile-up or sink-in phenomena influence the contact depth value
 $\Rightarrow$ errors in contact area $\Rightarrow$ errors in hardness and modulus



Correction factor: (from finite element simulations)



$$\frac{S_L}{S_U} = \frac{m_L}{m_U} \left(1 - \frac{h_r}{h_t} \right)^{1.35}$$

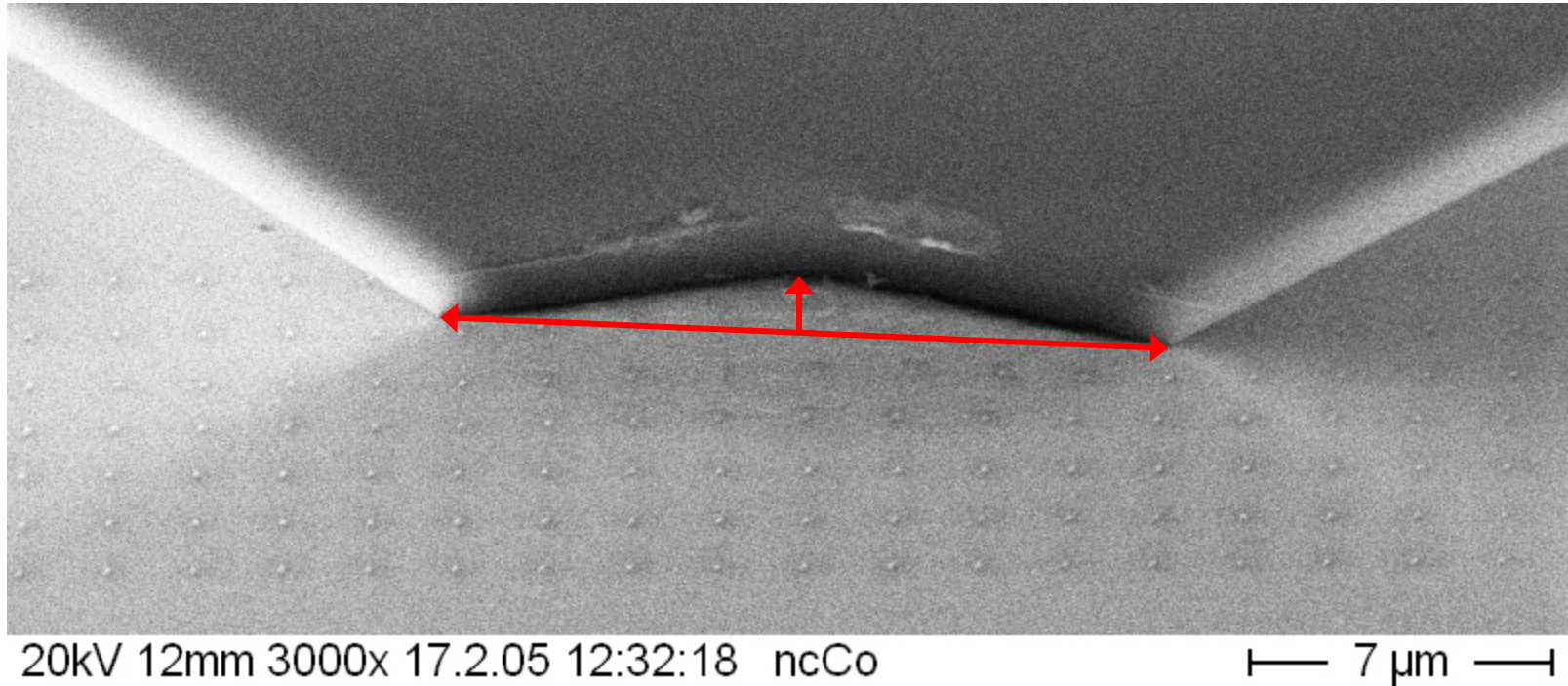
Residual depth

Total depth

1.35

Pile-up, Sink-in factor.
 Can be applied to
 calculated contact area to
 obtain true contact area.

in-situ measurement of contact area



contact area of PDMS

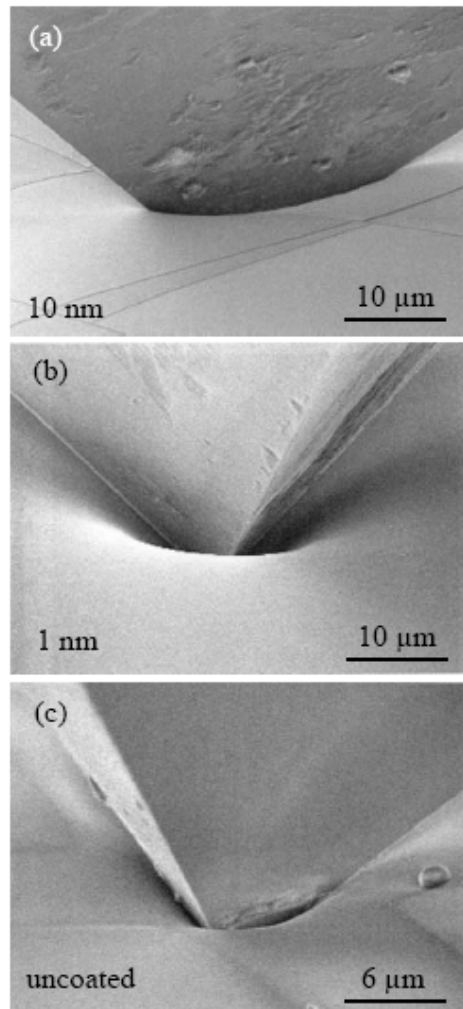
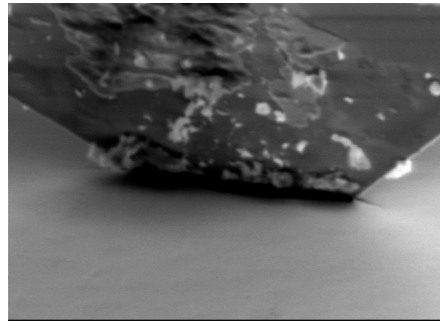


Figure 8

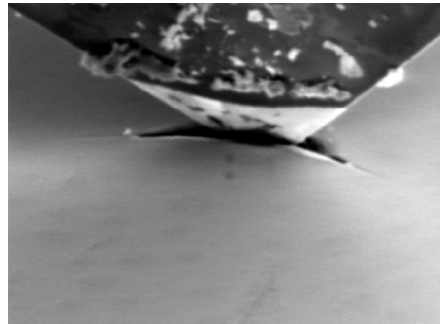
influence of gold layer

J. Deuschle (2008) J. Mater. Res.

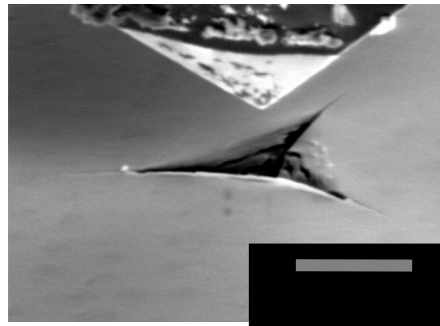
contact area of glassy carbon



max.load

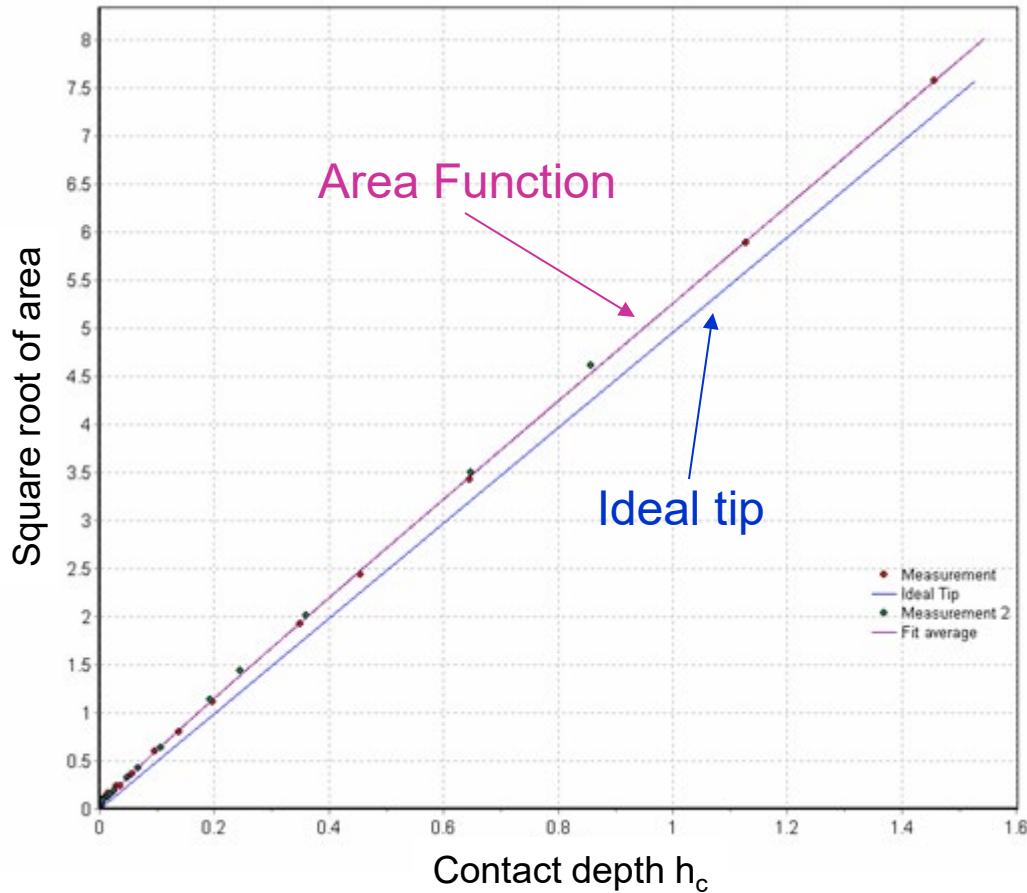


partial
unloading



complete
unloading

Determination of tip area function $A(h_c)$



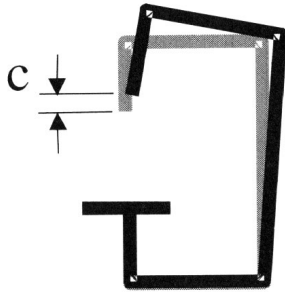
Reference Material (typical):
Fused silica $E = 72 \text{ GPa}$
 $H = 9 \text{ GPa}$

Load-Displacement curves are measured at different loads (500mN - 300 μ N)

Coefficients are adjusted to obtain the best modulus and hardness values

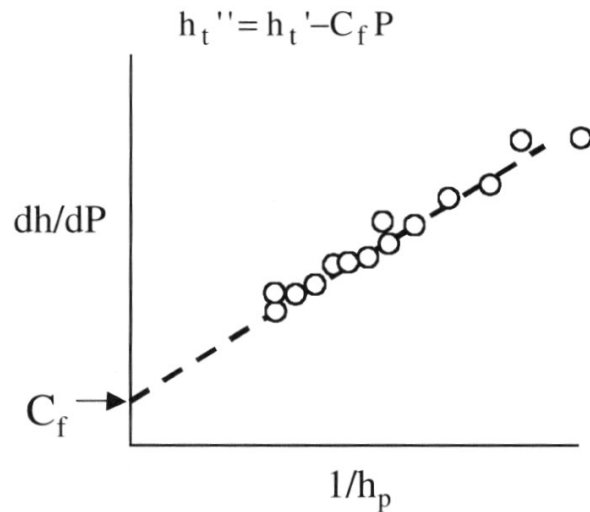
$$A(h_c) = a_0 \cdot h_c^2 + \sum_{i=0}^n a_{i+1} \cdot h_c^{1/2^i}$$

Instrument stiffness



Problem: stiffness of the machine

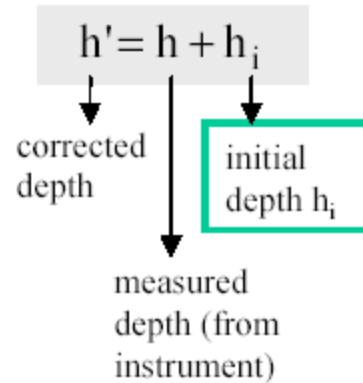
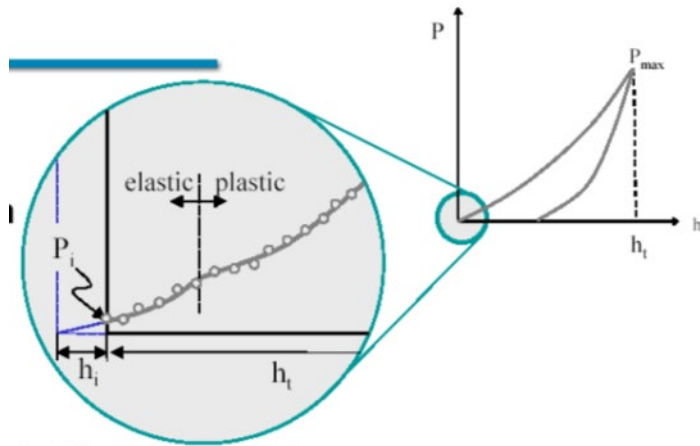
$$\frac{dh}{dP} = \frac{1}{S} + C_f$$



Use reference materials with known E

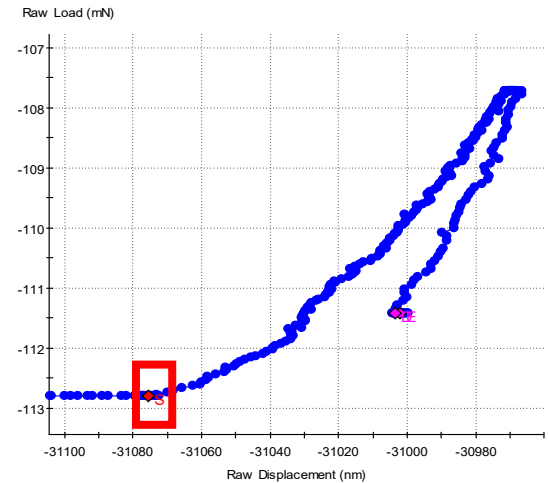
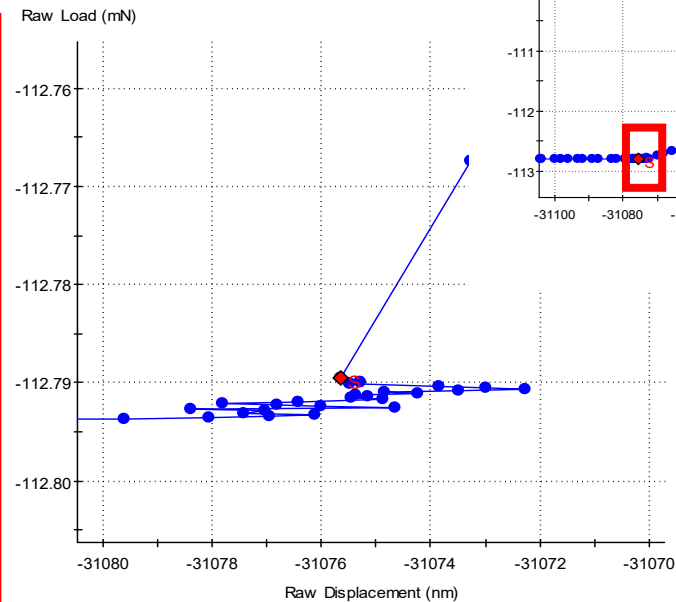
$$\frac{dh}{dP} = \sqrt{\frac{\pi}{24.5}} \frac{1}{2E^*} \frac{1}{h_p} + C_f$$

Surface Detection



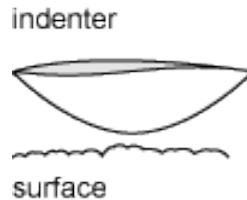
How to estimate h_i ?

1. Estimate by eye the intercept with the depth axis
2. Fit a polynomial by least squares method to the initial loading data and extrapolate back to zero load.
3. Fit the Hertz equations for elastic contact to the initial loading data and extrapolate back to zero load.



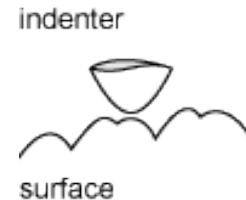
Surface Roughness

Low surface roughness



well-defined

High surface roughness



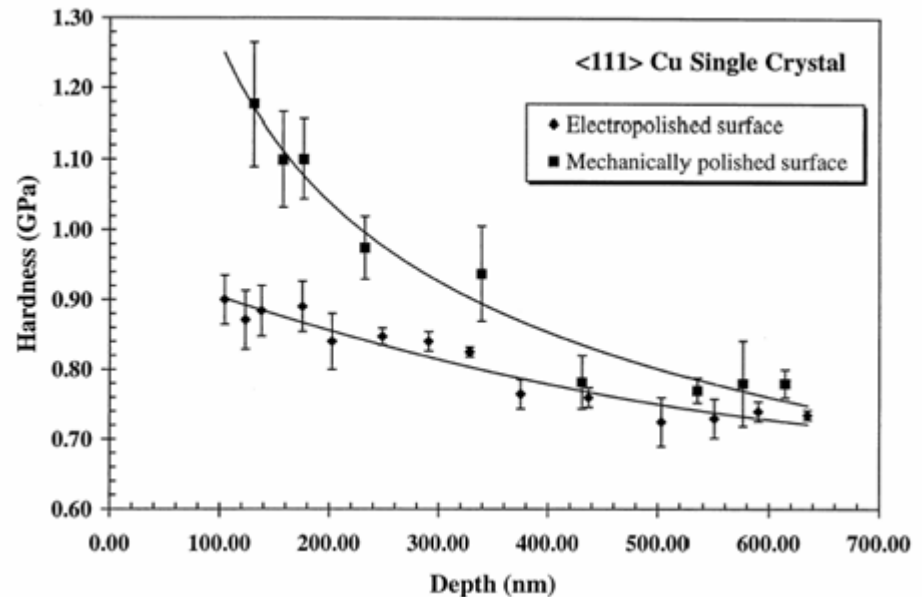
ill-defined

Contact Area

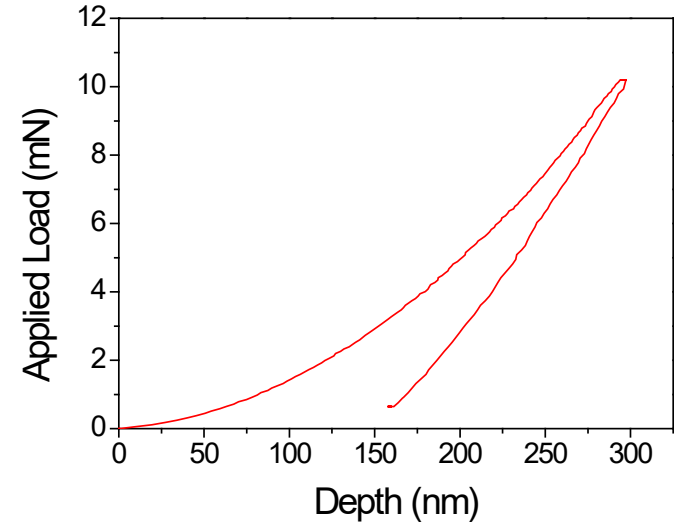
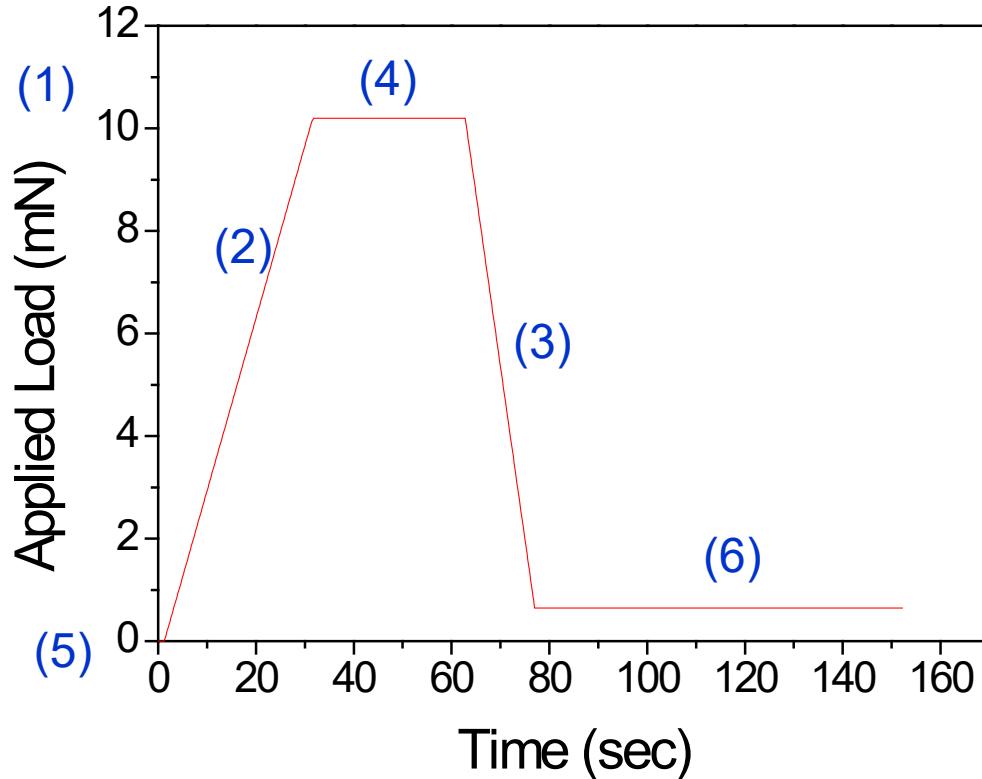
"Rule": $h_{\max} > 20 R_a$

Caution:

Polishing the sample surface results in a reduced roughness but considerable work hardening may occur



"A typical measurement"

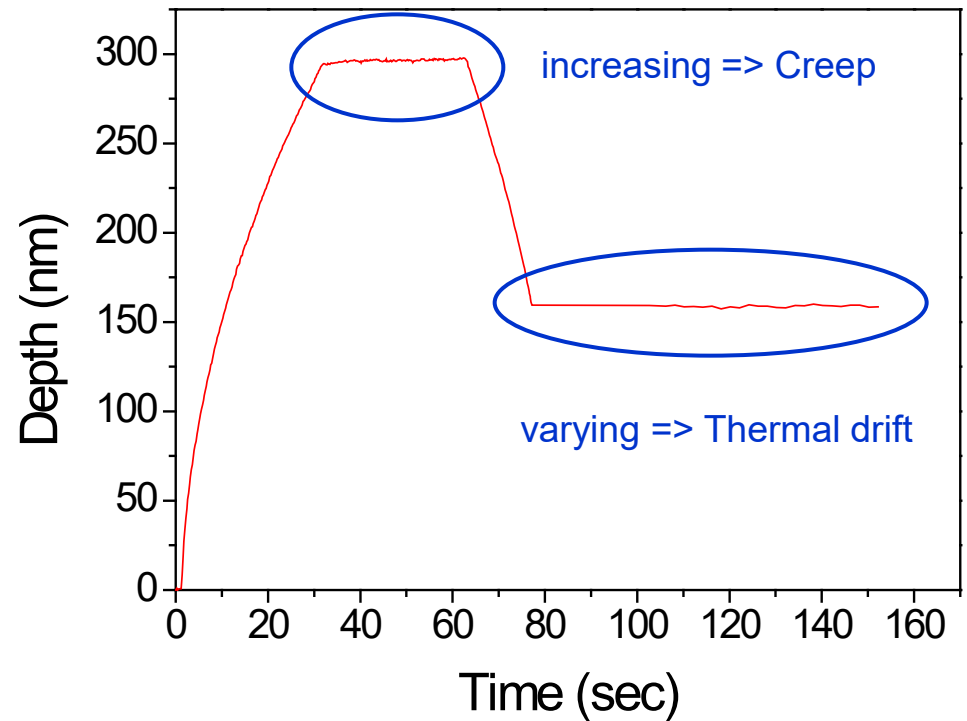
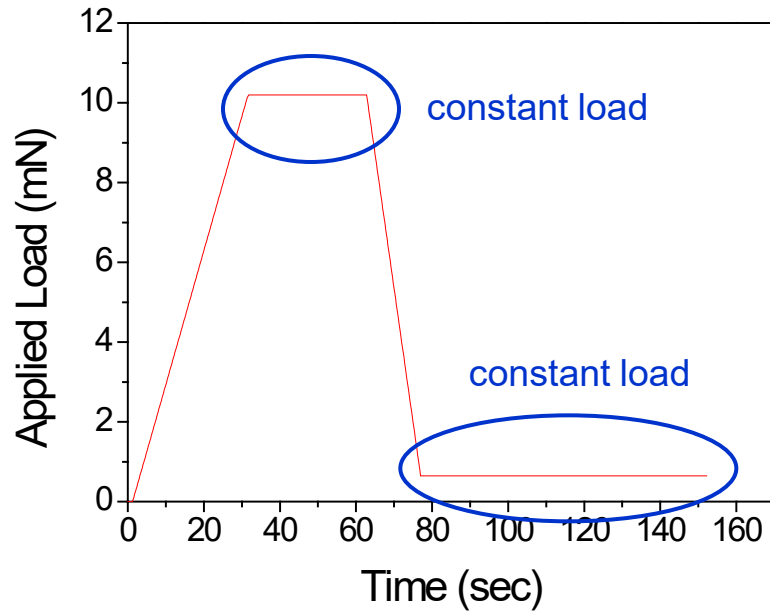


The loading - unloading cycle is **load-controlled**
Normally a **constant load rate** is used

Input Parameters:

- (1) $F_{\max}$
- (2) Time to load / loading rate
- (3) Time to unload / unloading rate
- (4) Hold period @ $F_{\max}$
- (5) Load for 2nd hold period after partial unloading
- (6) Period of 2nd hold period after partial unloading

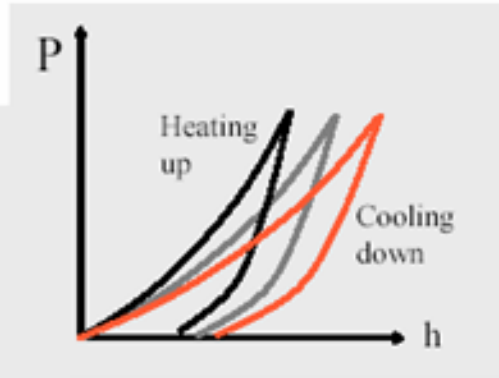
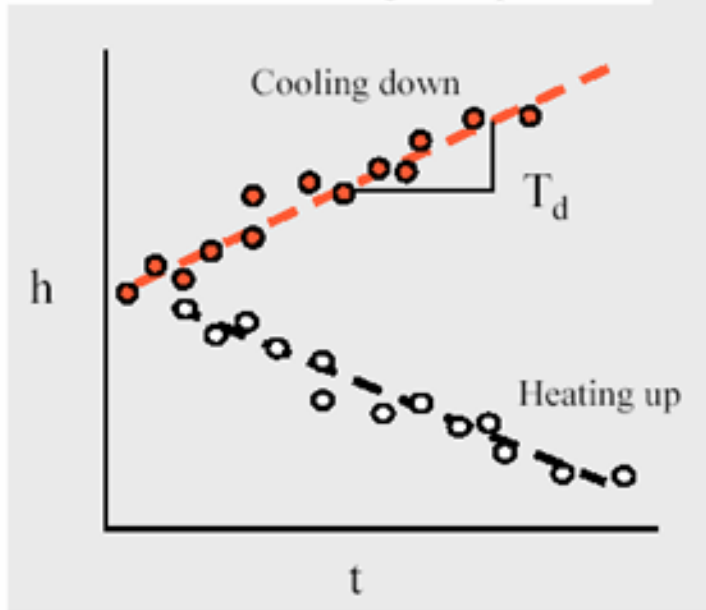
Hold periods



Thermal drift

How to estimate T_d ?

Thermal drift data taken during “hold” period



$$h' = h - T_d \cdot t$$

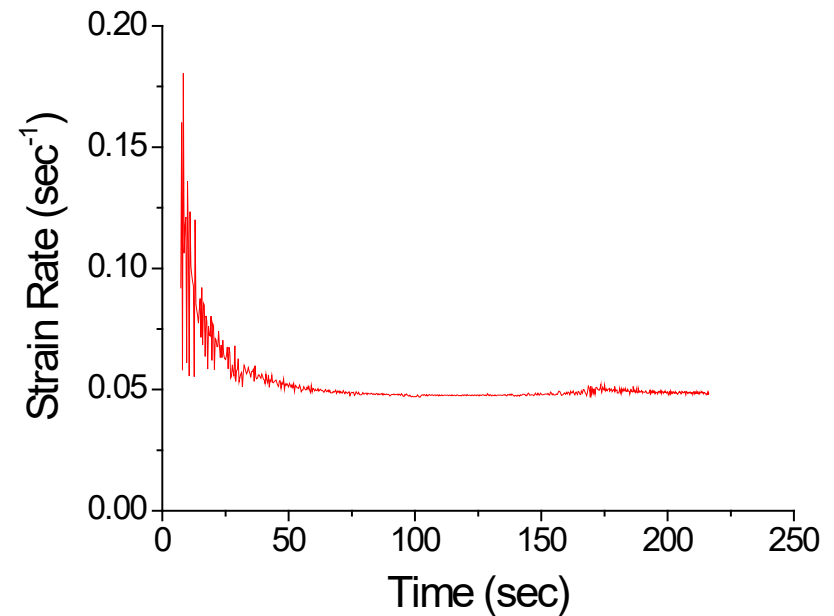
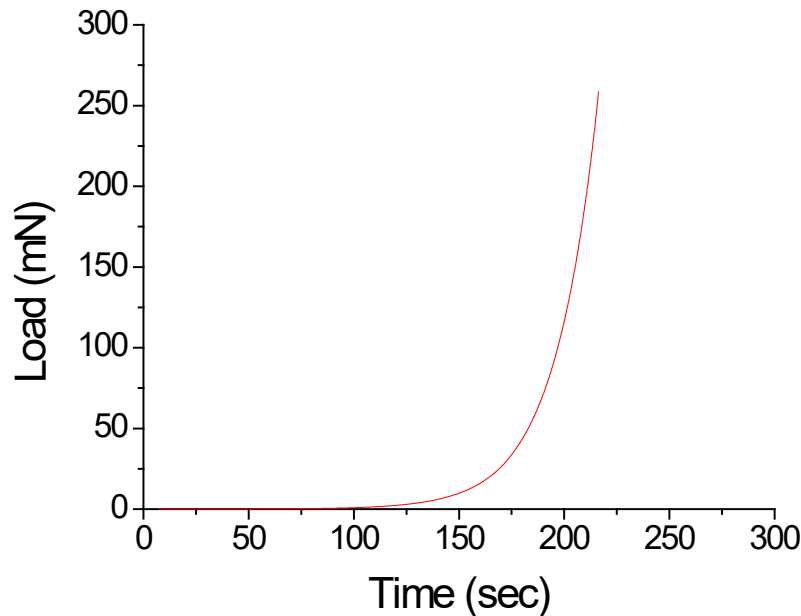
Typical drift values below 0.05 nm/sec
(for MTS Nanoindenter @ Empa Thun)

Strain rate in indentation tests

Tensile are normally performed with displacement-controlled machines at constant strain rate

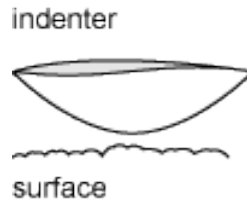
With pyramidal indenters it is possible to perform constant strain rate experiments with load-controlled nanoindenters

$$\dot{\varepsilon} = \frac{\dot{P}}{P} = \frac{\dot{h}}{h} = \text{const}$$



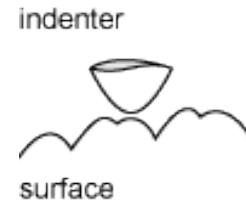
Surface Roughness

Low surface roughness



well-defined

High surface roughness



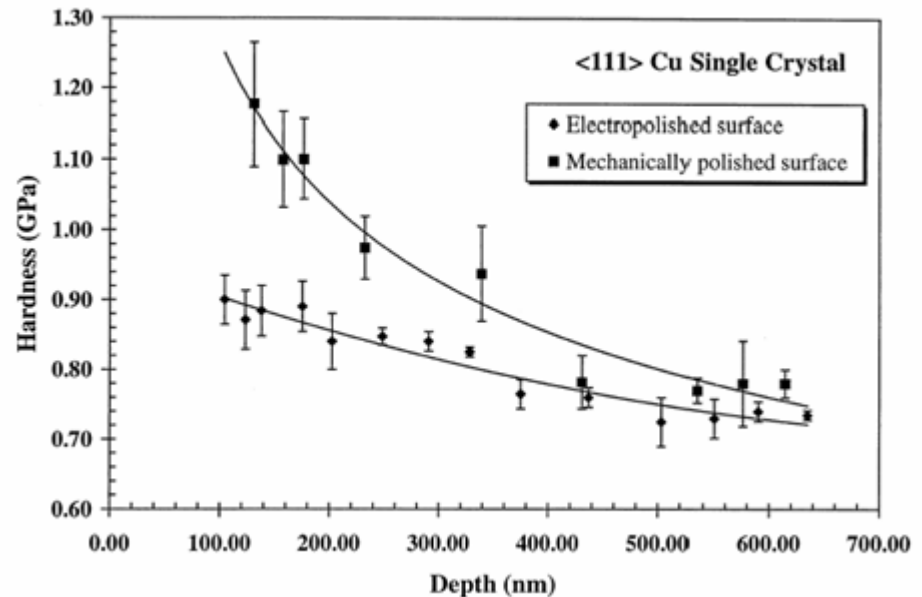
ill-defined

Contact Area

"Rule": $h_{\max} > 20 R_a$

Caution:

Polishing the sample surface results in a reduced roughness but considerable work hardening may occur

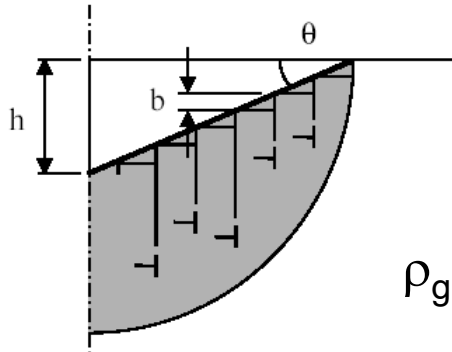


Indentation Size Effects (ISE)

Soft materials (Fe, Al, Cu) with surface contaminant layers (oxides, carbides)

=> dislocations pinned

=> higher hardness values @ small indentation depths



$$\rho_g = \frac{3}{2bh} \tan^2 \Theta$$

ρ_g : dislocation density

circular dislocation loops serve to increase the effective yield strength

Nix & Gao, J. Mech. Phys. Solids 46(3) 1998, pp. 411-425

Other ISE effects:

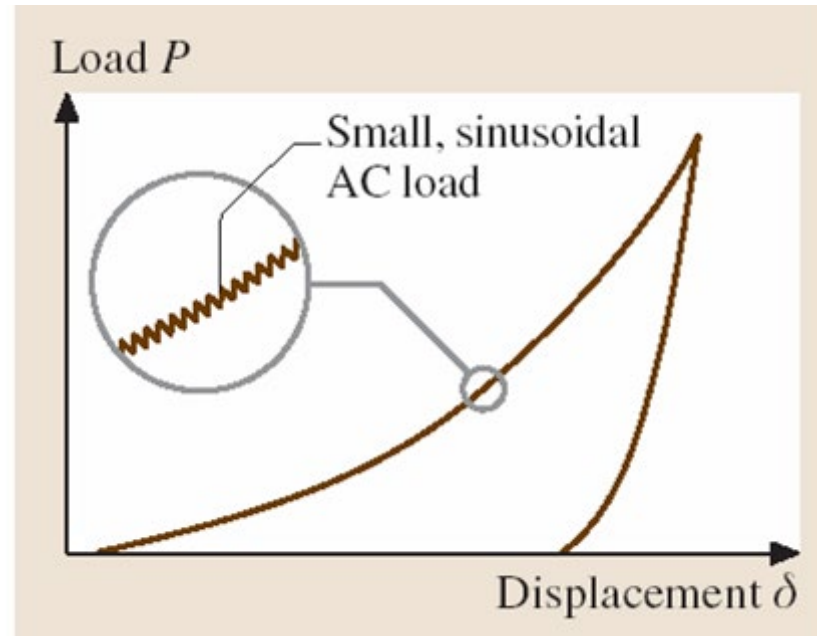
- Real shape of indentation pyramid
- Sample surface roughness, surface polishing

Dynamic Contact Models

Principle:

A small AC load is superimposed to the DC load.

The contact stiffness S can be calculated throughout the complete indentation cycle.



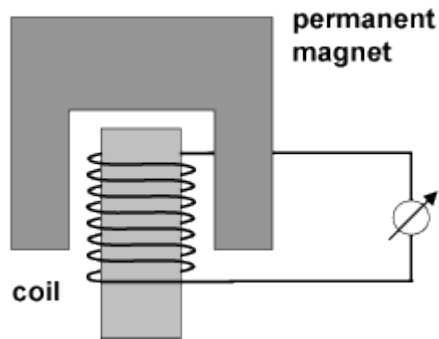
Typical frequency ≈ 60 Hz

The AC load creates a dynamic system with the sample acting as a spring with stiffness S (contact or unloading stiffness) and the nanoindentation system as a series of springs and dampers

Nanoindentation equipment: load actuation & displacement measurement

Load actuation

Electromagnetic actuation:



Advantages:

- long displacement range (up to mm)
- linear current-load behavior
- wide load range (mN to N)
- can be used to pick up the indenter as well as to push it down

Disadvantages:

- large ($\approx 10\text{cm}$) and heavy (order of kg)
- current in the coil $\Rightarrow$ heat $\Rightarrow$ thermal drift

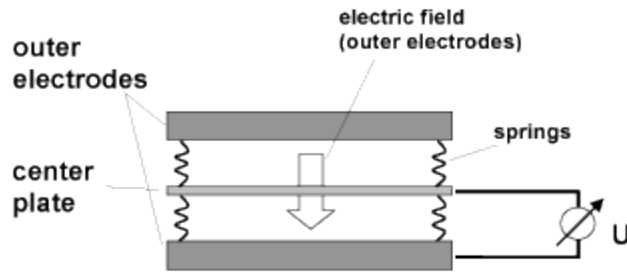
By varying the current, I , through the coil, a magnetic field is generated that interacts with the magnetic field of the permanent magnet.

Calibration:

Hanging a series of weights to the indenter shaft and recording the load current to lift the shaft to the same position for each weight

Load actuation

Electrostatic actuation:



Advantages:

- small size of the system (in the order of tenths of mm)
- good temperature stability

Disadvantages:

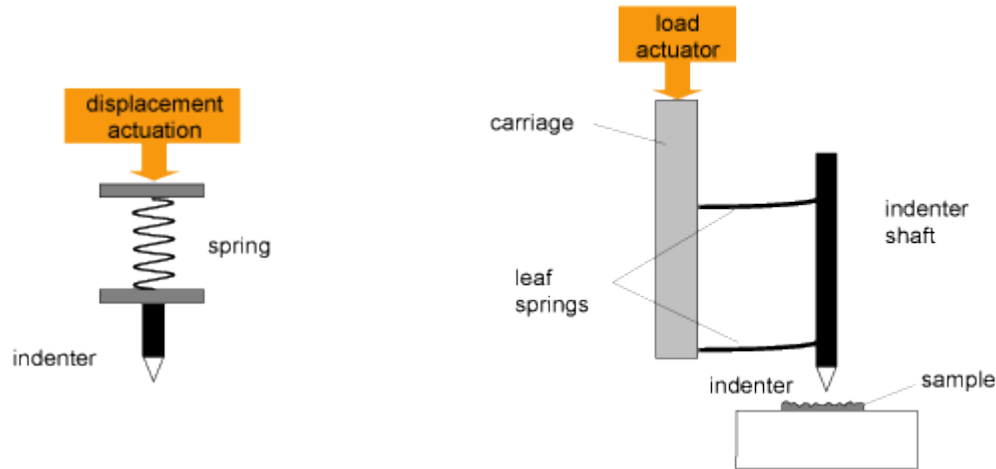
- limited load range (in the order of tenths of mN)
- limited displacement range (in the order of tens of μm)
- usually only one possible direction of tip movement

The load is applied by an electrostatic force generated between the center plate and the upper or lower plate of a three-plate transducer system.

The applied load is proportional to the square of the voltage.

Load actuation

Actuation through springs:



Range vs. resolution: In principle, very high force resolutions are possible. Cantilever based devices commonly report force resolutions in the pN range, or better. However, as resolution goes up, range goes down.

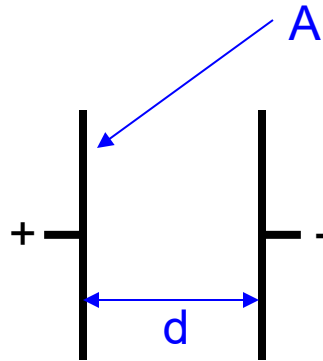
Geometric stability: One of the main disadvantages of using a cantilever is that the tip rotates as the load on it is increased.

Force response: The higher the force resolution, the greater are the displacements needed to change that force. The rate at which the force can be changed is limited in such machines.

Calibrations: Force calibration depends on accurate knowledge of the spring constant and the relative displacements.

Displacement measurement

Capacitive:



Parallel plate capacitor:

$$C = \frac{\epsilon\epsilon_0 A}{d}$$

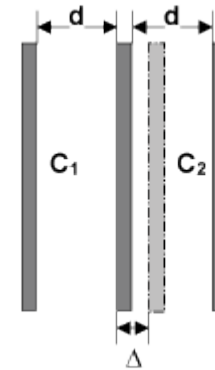
Advantages:

- can be of very high precision (resolution below $1\text{\AA}$)
- small size

Disadvantages:

- total displacement range is relatively small

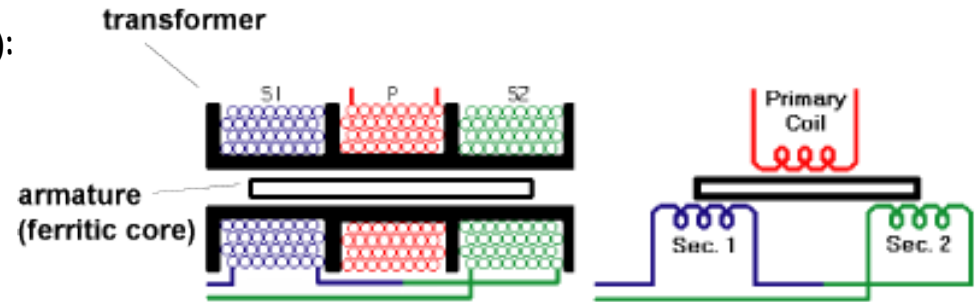
Differential capacitor:



Difference between C_1 and C_2 is measured

Displacement measurement

Linear Variable Differential Transducer (LVDT):



Advantages:

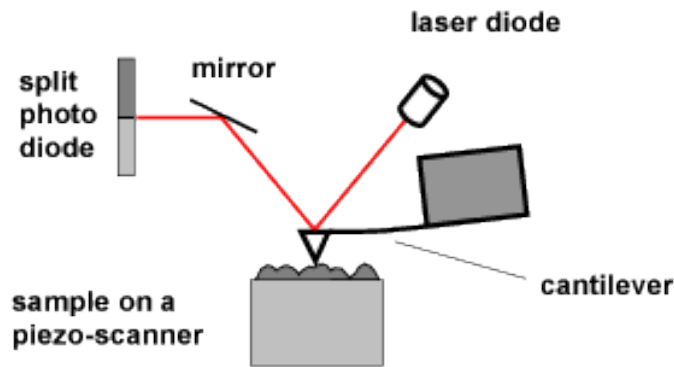
- relatively low cost due to its popularity
- solid and robust, capable of working in a wide variety of environments
- high signal to noise ratio
- negligible hysteresis
- no permanent damage to the LVDT if measurements exceed the designed range

Disadvantages:

- lower resolution compared to a capacitive displacement measurement
- core must be in contact (directly or indirectly) with the measured surface

Displacement measurement

Optical lever methods:



Used for example to measure the deflection of beams
Most of the Atomic Force Microscopes (AFM) use this principle

Very high sensitivity (below $1\text{\AA}$) is possible

Nanoindentation other experimental aspects

Nanoindentation experiments on thin films

Advantage:

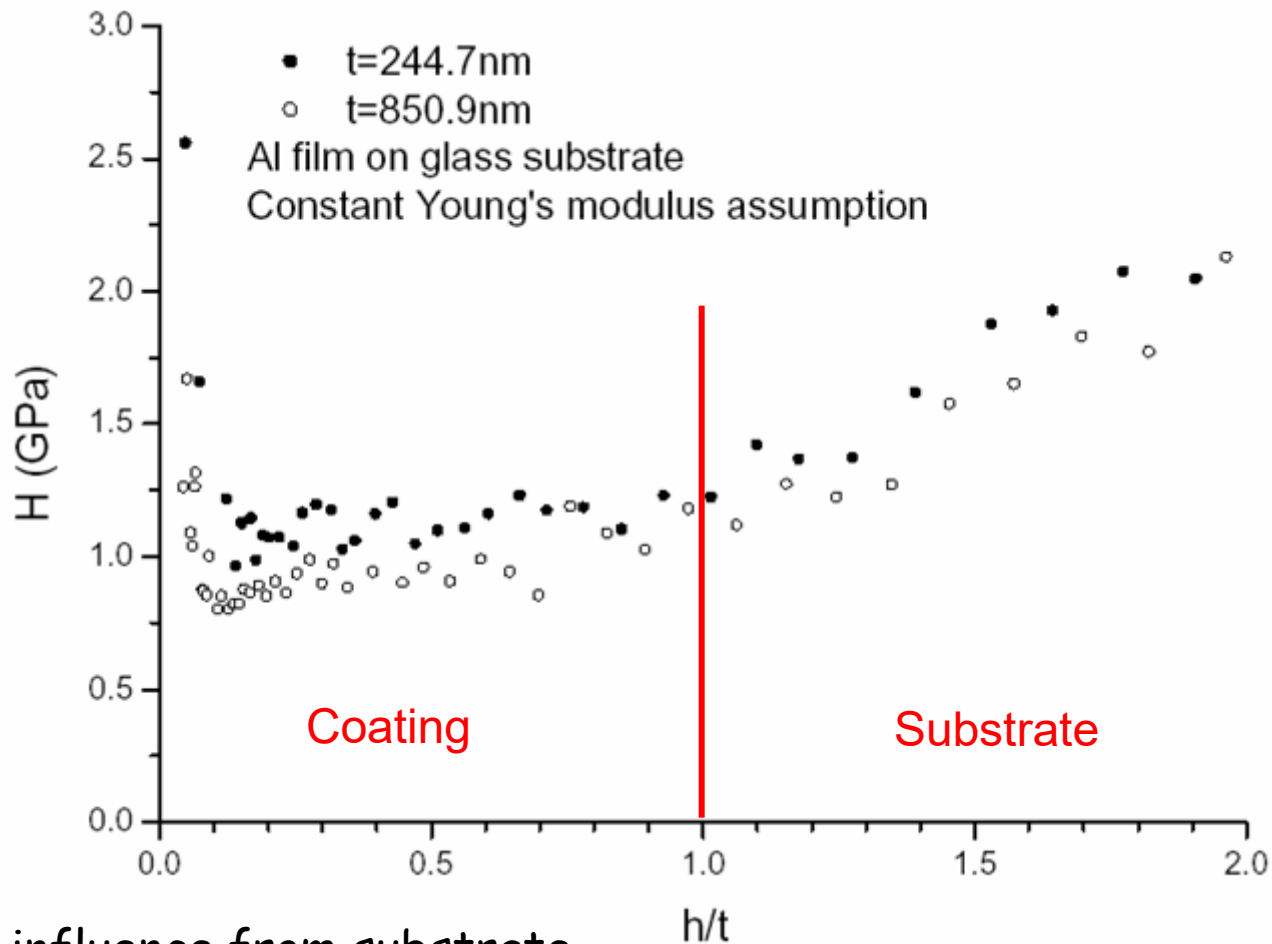
Film can be measured without removing it from the substrate
Spatial distribution in lateral & vertical direction



Chief difficulty:

Avoiding unintentional probing of substrate properties

$1\mu\text{m}$ Al on glass (soft coating on hard substrate)



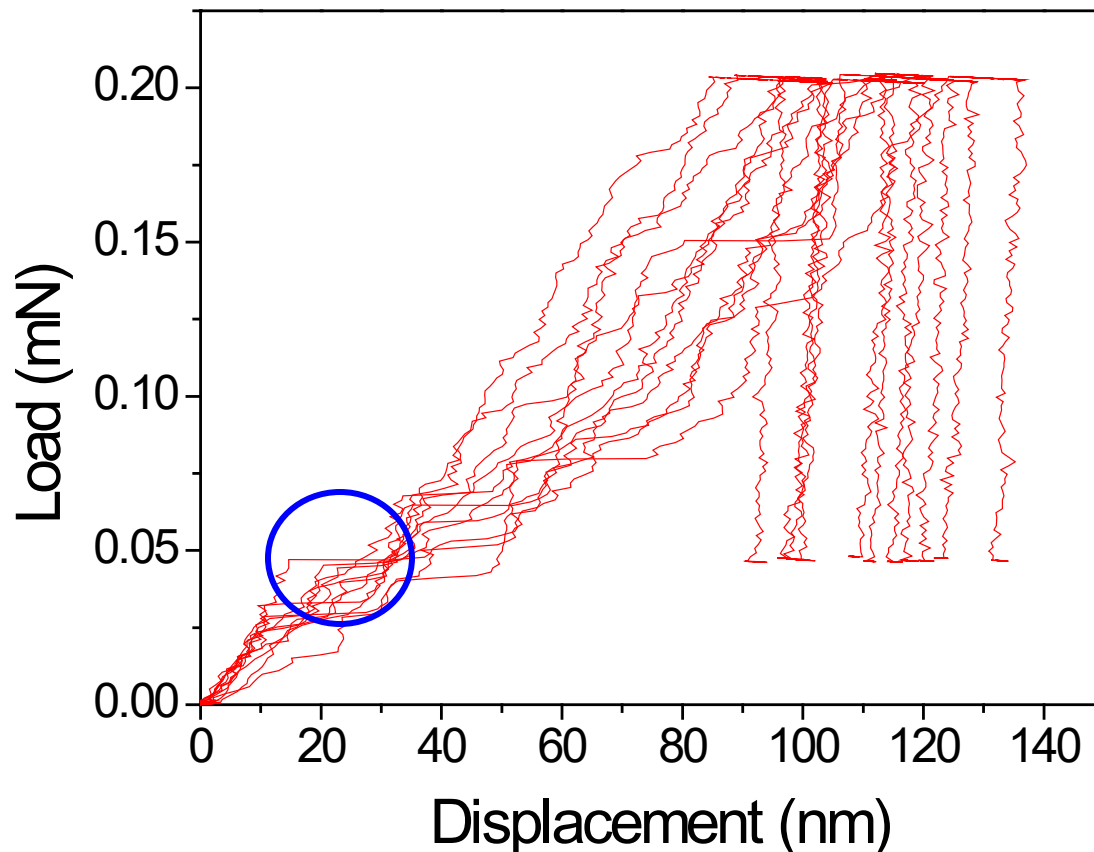
- almost no influence from substrate
- ISE observable

S. Chen, L. Liu, T. Wang, *Acta Mater.* 52 (2004), p. 1089

1 μm Al on glass: effects of surface oxide layer

Surface oxide layer breaks under load resulting in "pop-in" features in the load-displacement curve

=> Evaluation of H and E is not possible at small penetration depths



Considering the substrate properties

Soft film on hard substrate:

$$H_{eff} = H_s + (H_f - H_s) \cdot \exp \left\{ - \frac{Y_f}{Y_s} \frac{E_s}{E_f} \left(\frac{h}{t} \right)^2 \right\}$$

Hard film on soft substrate:

$$H_{eff} = H_s + (H_f - H_s) \cdot \exp \left\{ - \frac{H_f}{H_s} \frac{Y_s}{Y_f} \sqrt{\frac{E_s}{E_f}} \frac{h}{t} \right\}$$

H_{eff} : hardness of film-substrate combination

Y : Yield stress

h : penetration

t : film thickness

„Finite element simulation of indentation experiments“

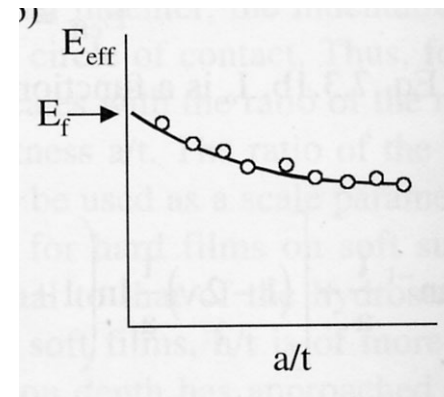
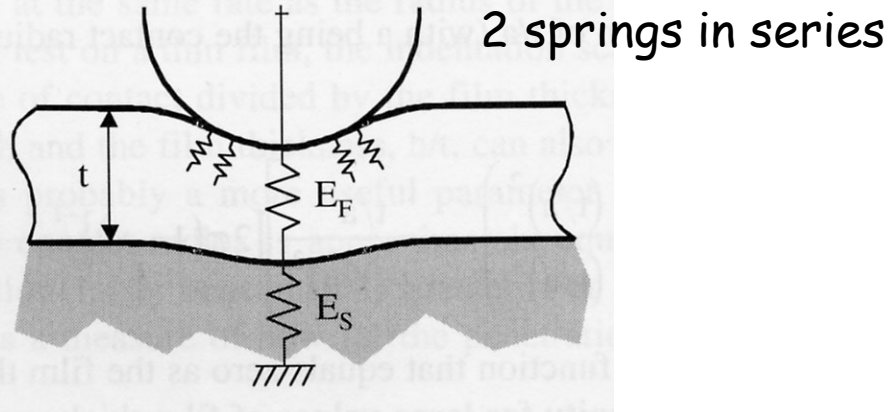
Bhattacharya and Nix, Int. J. Solids Structures 24, 1988, 1287-1298

BUT:

There is no one relationship which covers a wide range of materials behaviour

Young's modulus

Evaluating E for different a/t values
 $\Rightarrow$ extrapolating to E_f



$$\frac{dP}{dh} = \beta \frac{2}{\sqrt{\pi}} E_{eff} \sqrt{A} \left[\frac{\frac{(1-\nu_s^2)}{E_s} + \frac{(1-\nu_i^2)}{E_i}}{\frac{(1-\nu_f^2)}{E_f} (1 - e^{-\alpha t \sqrt{A}}) + \frac{(1-\nu_s^2)}{E_s} (1 - e^{-\alpha t \sqrt{A}}) + \frac{(1-\nu_i^2)}{E_i}} \right]$$

a : penetration depth
 β : depends on the indentation tip geometry

t : coating thickness
 α : empirical constant, determined from a series of experiments

R.B. King, Int. J. Solids Structures 23(12), 1987, pp. 1657-1664

"1/10-rule of thumb"

Rule of thumb by H. Bückle:

($h_{\max}$ = maximal depth; d_{Film} = film thickness)

$$h_{\max} \leq 0.1 \cdot d_{\text{Film}}$$

For the "extreme" cases of a hard film on a soft substrate (Nanocomposite coatings)
or for a soft coating on a hard substrate (Al on glass)

this rule may be too lax or too strict

H. Bückle, VDI Berichte, Vol. 41, p. 14, 1961

Choice of applied maximal load / maximal displacement

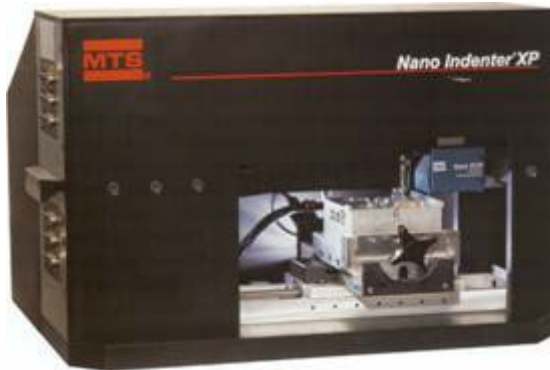
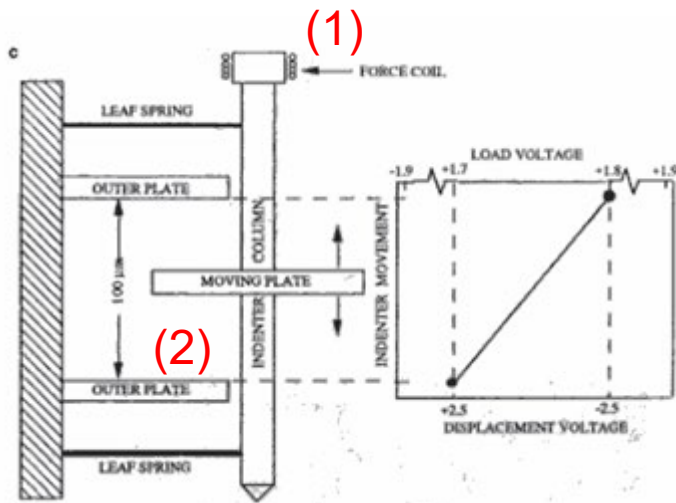
Rules:

- Displacement $> 20 R_a$
- Displacement $\leq 10\%$ of coating thickness

Nanoindentation equipment: Commercial Nanoindenters

MTS Nanoindenter XP / KLA Tencor NanoIndenter G200

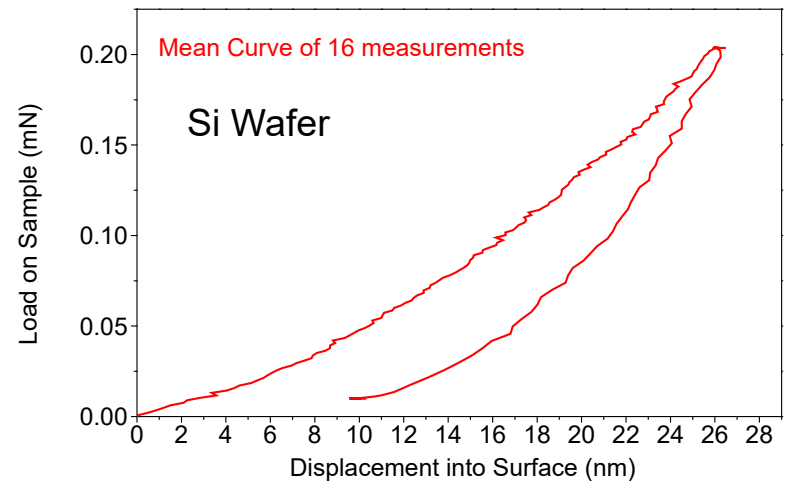
- (1) Inductive Force Generation
- (2) Capacitive Displacement Measurement



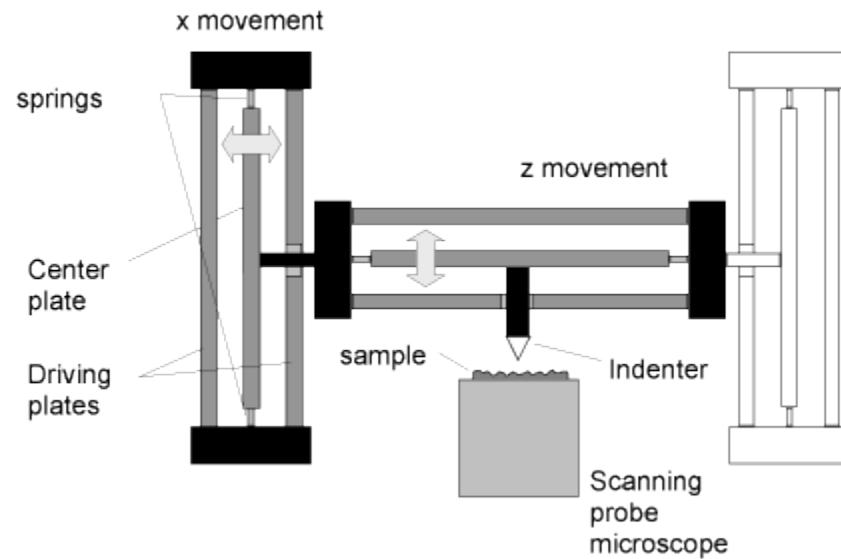
MTS Nanoindenter XP installed @ Empa in Thun:

Displacement resolution	< 1 nm
Maximum indentation depth	500 μm
Minimal peak load	100 μN
Maximum load	500 mN
Load resolution	50 nN
Lateral position accuracy	$\pm 1 \mu\text{m}$

Scratch option with lateral force measurement
(Measurement of Friction Coefficient)



Hysitron Tribo-Scope / Ubi



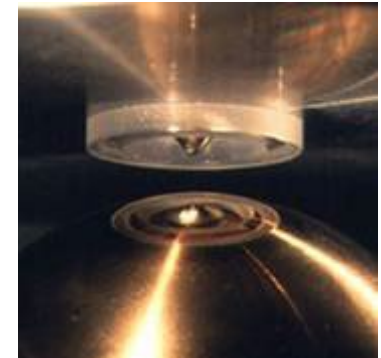
Transducer Specifications

- Load
 - Resolution: $<1\text{nN}$
 - Noise Floor: 100nN
- Displacement
 - Resolution: 0.0002nm
 - Noise Floor: 0.2nm
 - Drift: $<0.05\text{nm/sec}$

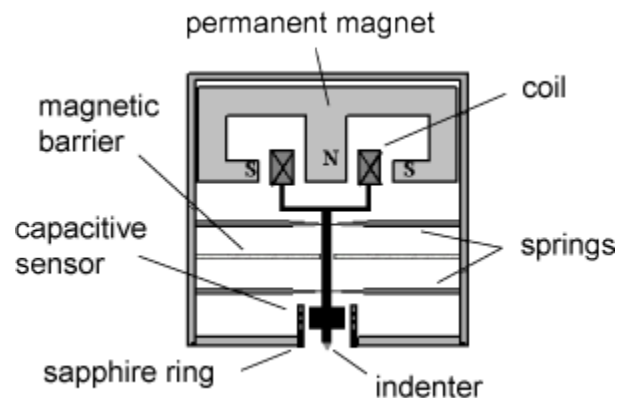
CSM Instruments Nano-Hardness Tester

Indentation Testers Specifications

	Micro	Nano
Maximum Load	10 N (30 N optional)	500 mN
Load Resolution	100 μ N	0.04 μ N
Maximum Depth	0-20 & 0-200 μ m (1 mm optional)	0-20 & 0-200 μ m
Depth Resolution	0.04 & 0.3 nm	
XY stages (*)	120 mm x 20 mm	
XY resolution	0.25 μ m	

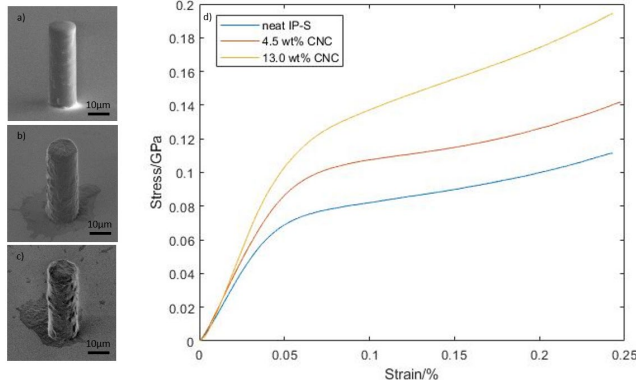
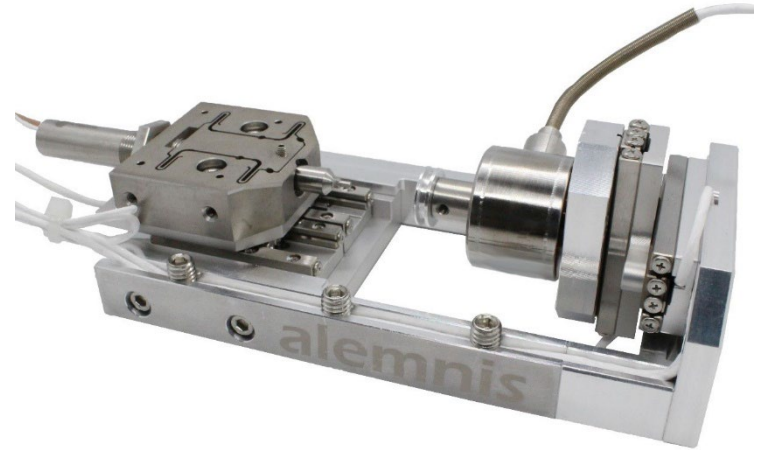


Surface reference by contact with a ring



Alemnis in situ indenter

- In situ experiments inside the SEM
- Actuation: Piezo
- Load, displacement measurement: Strain gauges
- Load range: Up to 2.5N
- Load noise: 4 μ N
- Displacement range: 40 μ m
- Displacement noise: 0.2 nm

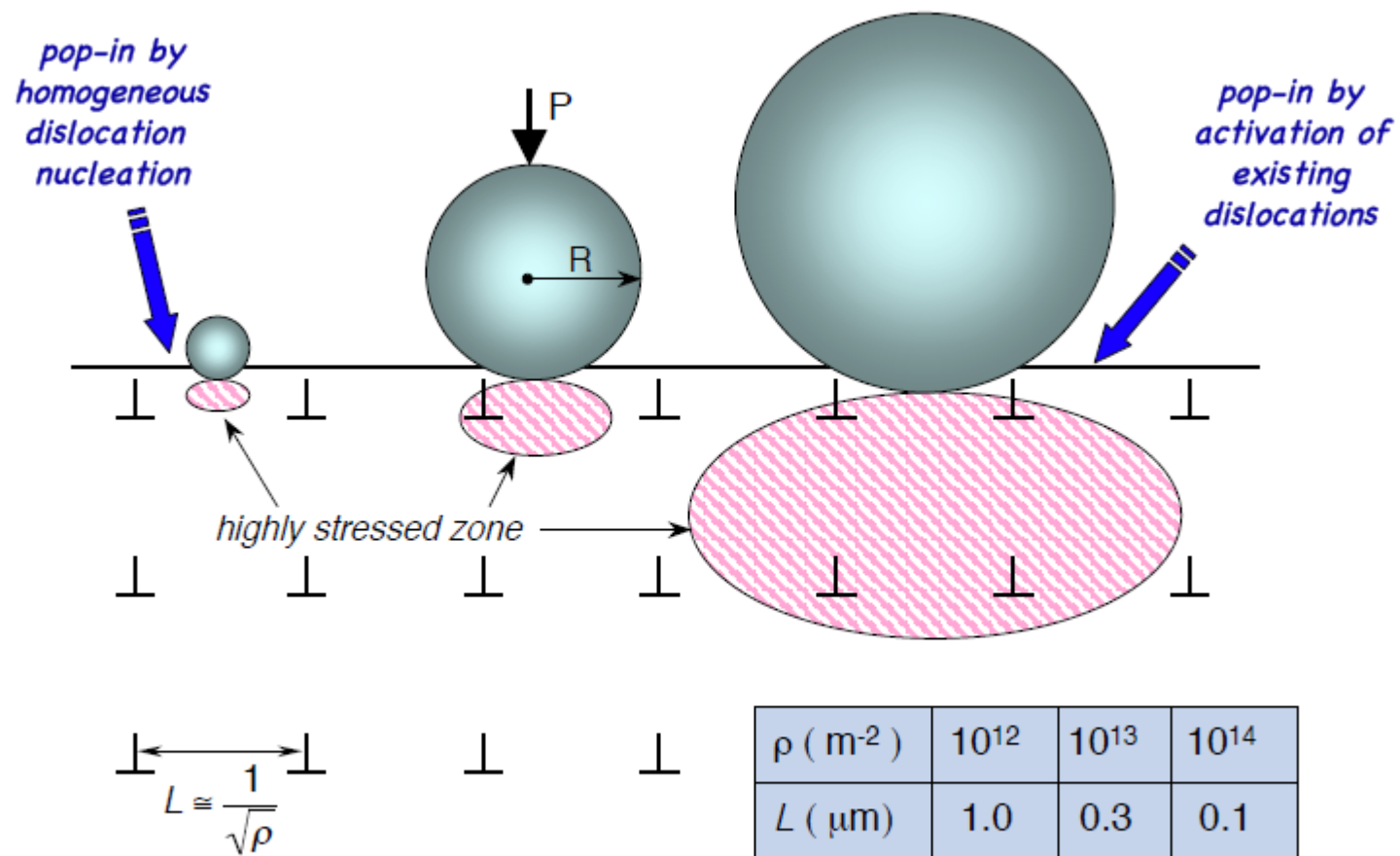


- Variable temperature modules (-150°C to 1000°C)
- High strain rate module (up to 1000/s)

What happens in the material?

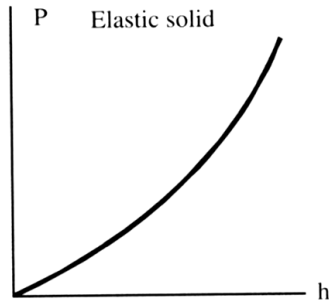
- Sink-in and pile-up around the tip (α -Me)
- Densification and phase transformation (Remy)
- Dislocation nucleation and grain boundary sliding (Sid etc.)
- Creep
- Crack propagation

Pop-in and defect microstructure

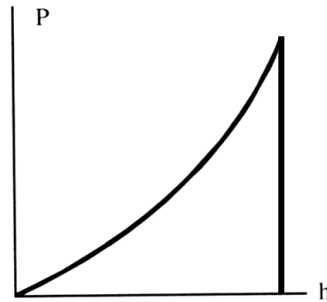


40

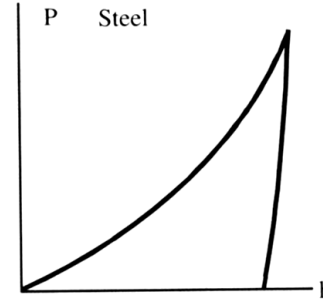
Typical load displacement curves



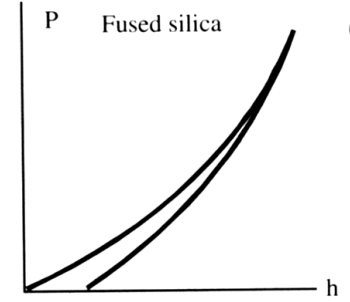
Elastic Solid



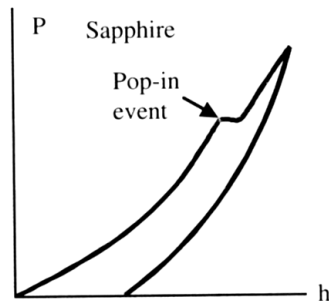
Fully plastic



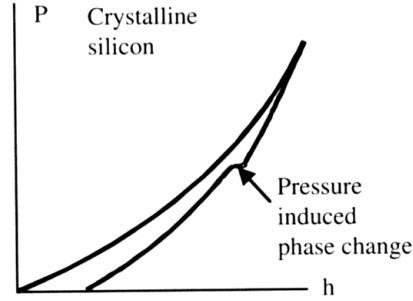
Ductile Solid
(Steel)



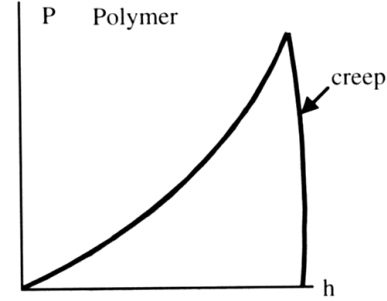
Brittle Solid
(Fused Silica)



Brittle Solid
Cracking during
loading



Pressure induced
phase change
(Silicon)



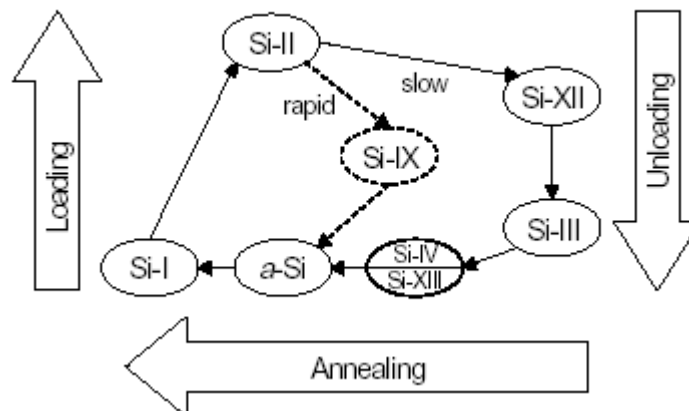
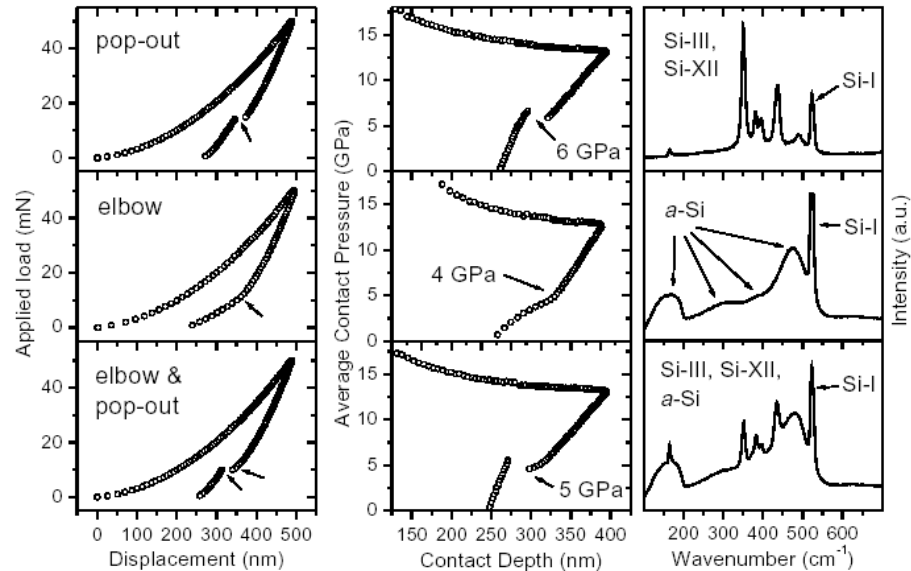
Polymer exhibiting
creep

phase transformations in silicon

slow ($<1\mu\text{m/s}$)

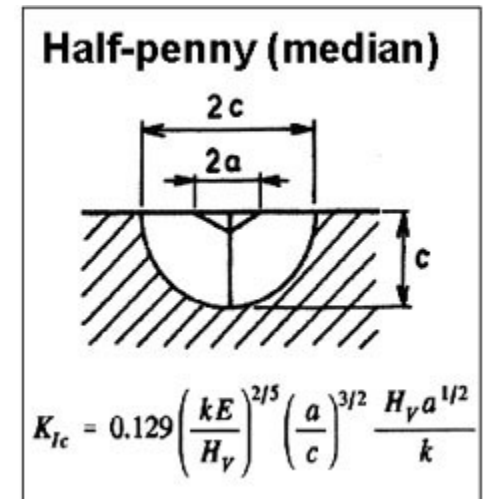
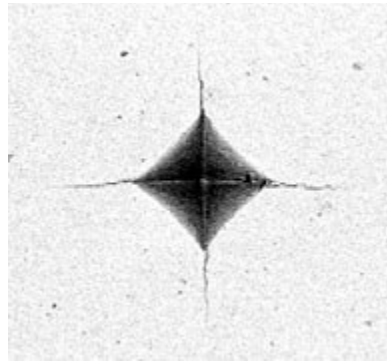
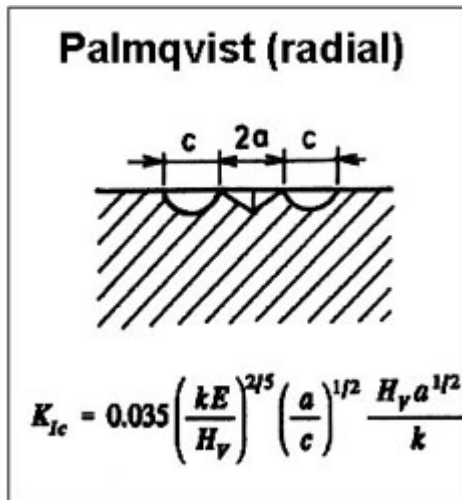
fast ($>100\mu\text{m/s}$)

intermediate



Fracture toughness

Determine K_{Ic} : measure size of residual impression a and crack length c

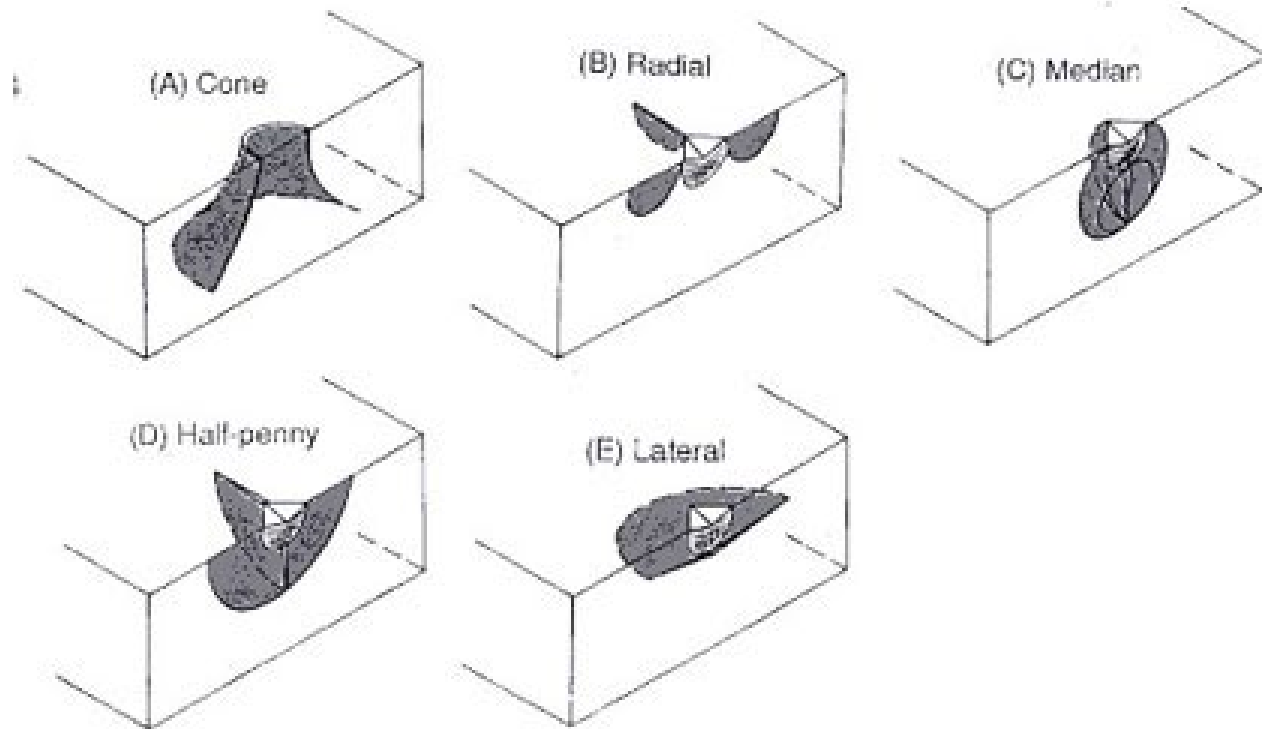


Probleme:

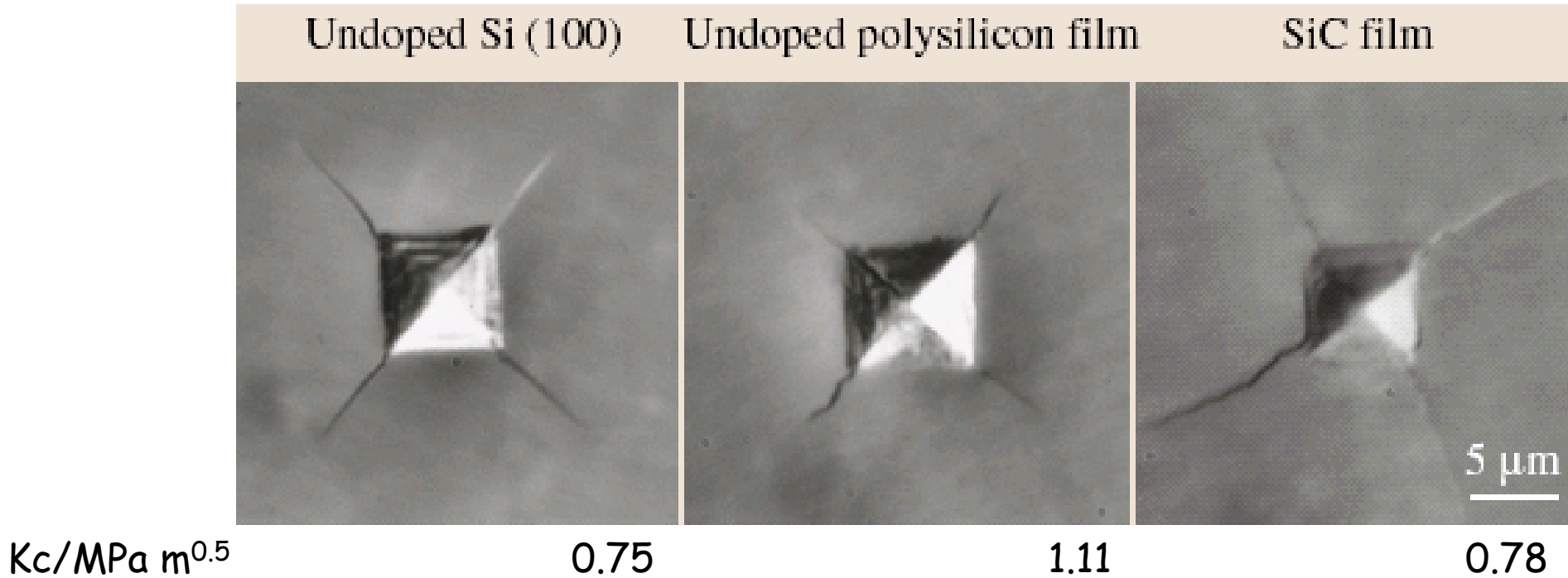
- precision of length measurement
- Crack morphology
- When is crack generated?
- Model is material specific

Fracture toughness

Possible crack modes



Example: fracture toughness



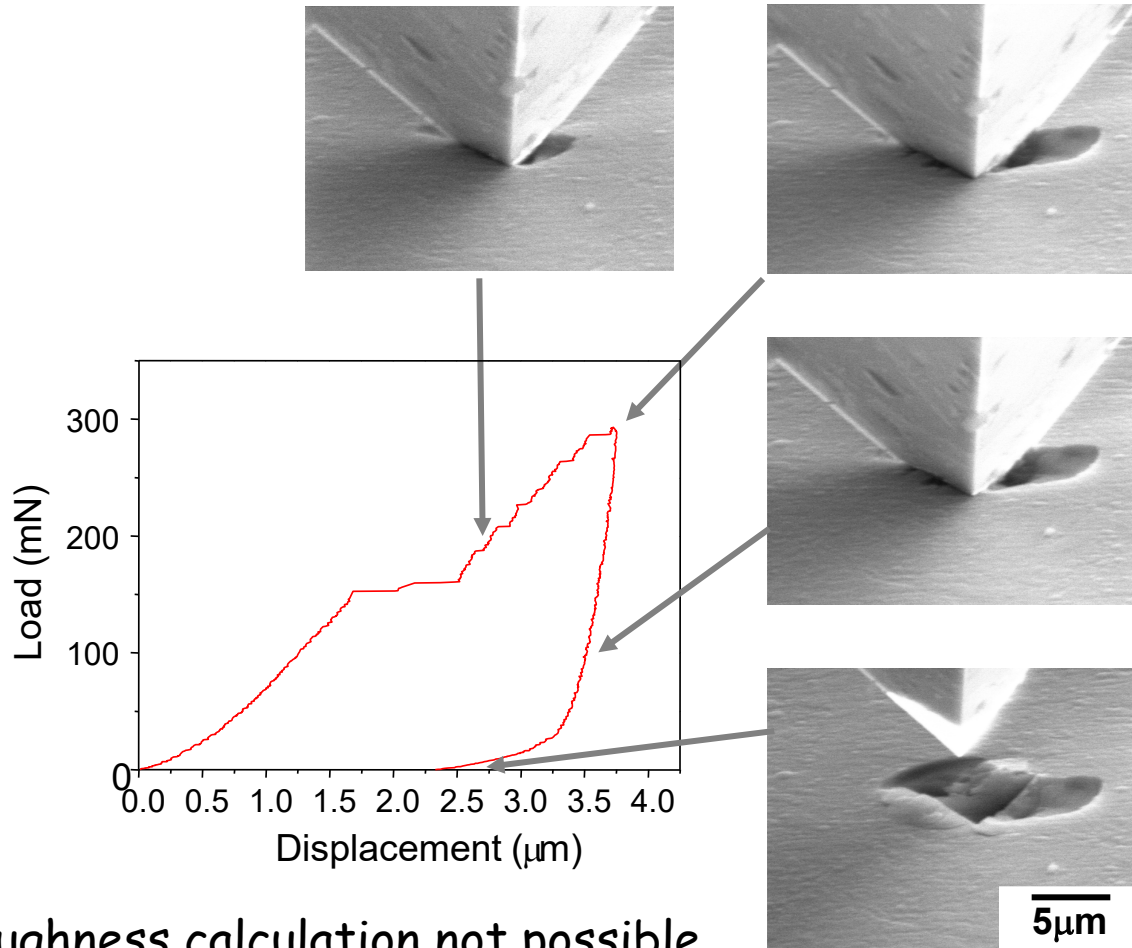
(Half- penny crack model)

Example: fracture toughness

Indenter:
cube corner

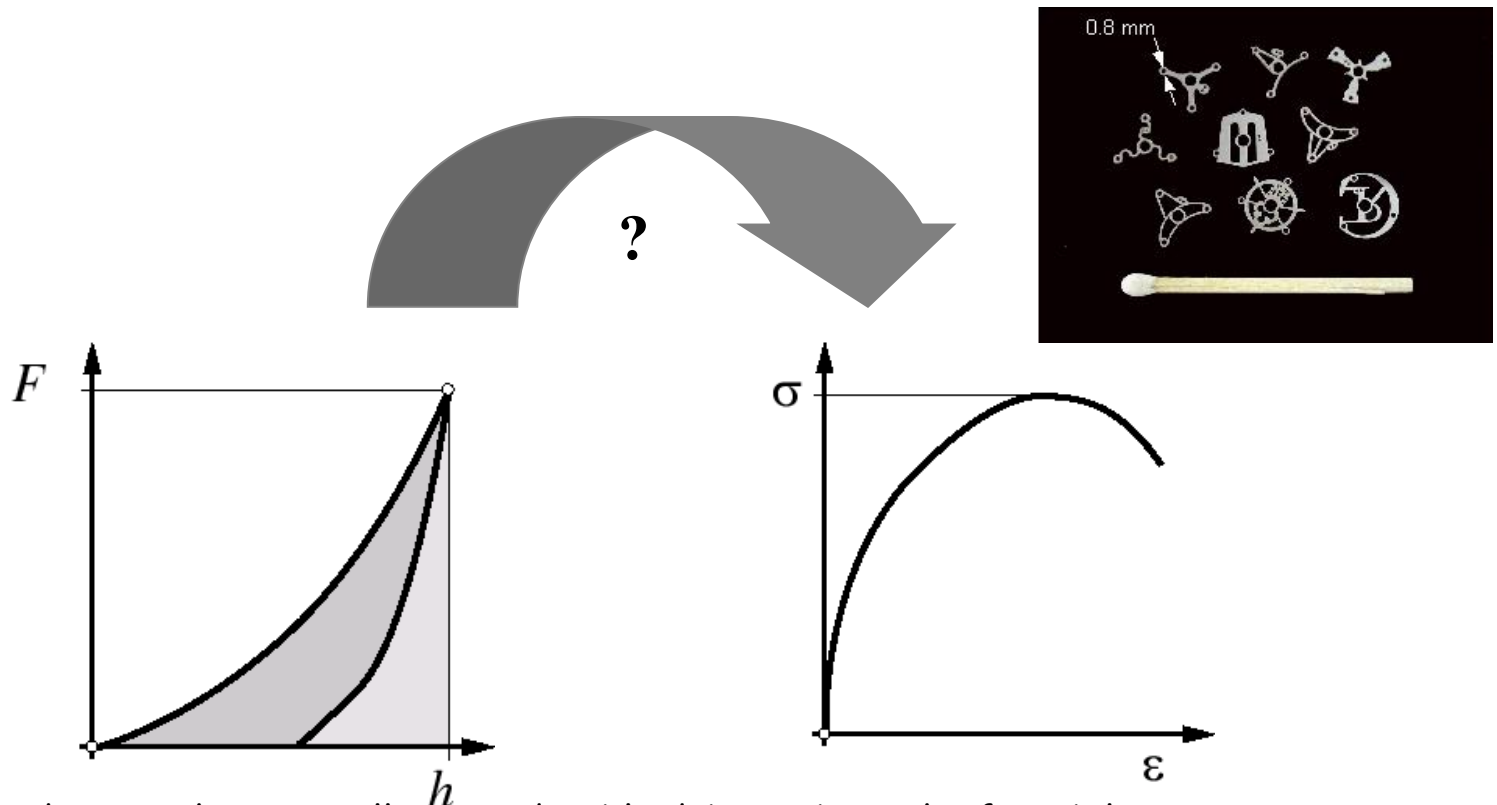
coating:
a-C:H mit 20at% Si
3.5 μm thickness
H=8GPa

Substrate:
(X155CrMoV121)



Lateral cracks form: toughness calculation not possible

Stress-strain curves from indentation

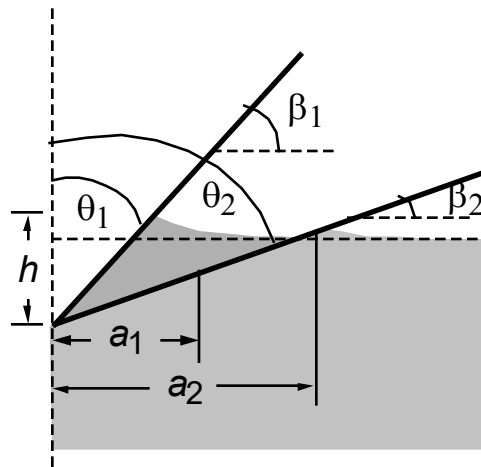


For conical type indenters, there is still a considerable debate about the feasibility:
Cheng YT, Cheng CM (1999) J Mater Res 19, 3493.
Venkatesh TA, Van Vliet KJ, Giannakopoulos AE, Suresh S. (2000) Scripta Mater 42, 833

Can we at least develop a robust practical method by pure numerical methods?

idea: combination of experiments

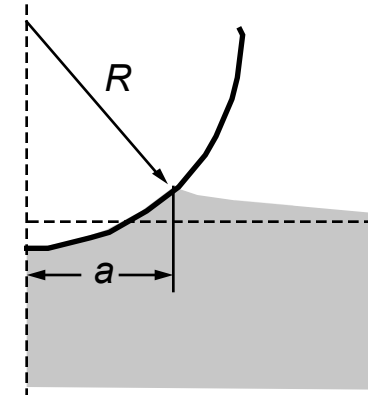
Conical tips



$$\varepsilon \sim \frac{h}{a} \sim \cot \Theta$$

Johnson, K.L.: Contact Mechanics, 1985.

Spherical tip

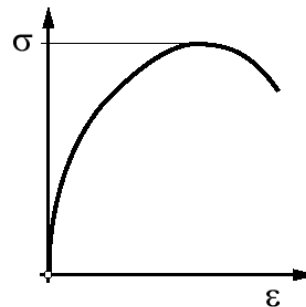


$$\varepsilon \sim \frac{a}{R}$$

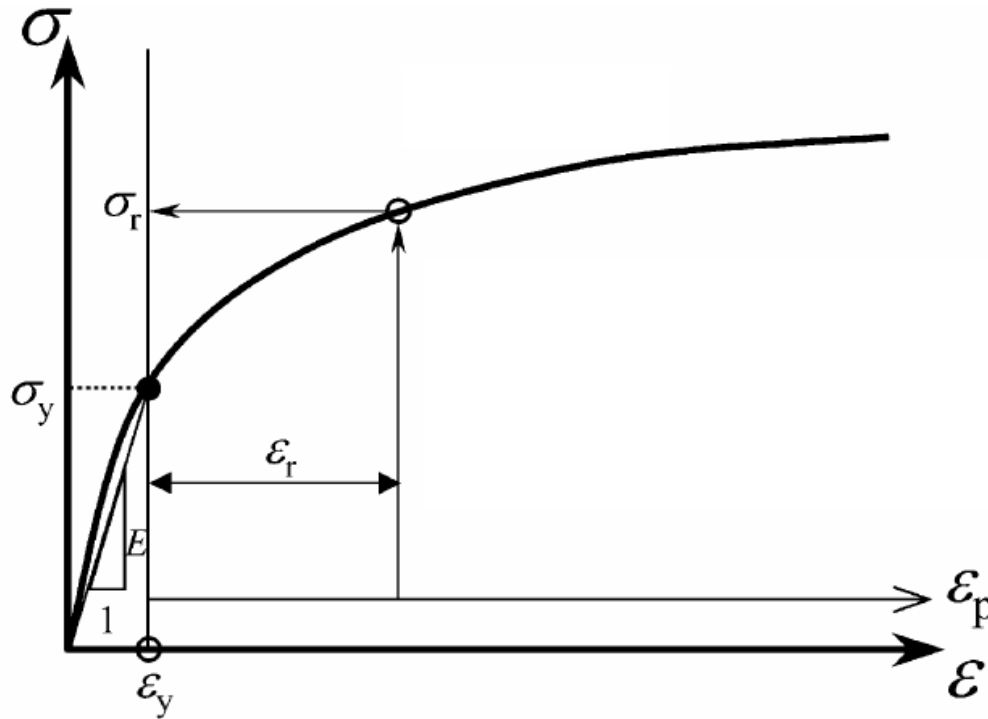
Tabor, D.: The Hardness of Metals, 1951

combine several
experiments
with varying cone angle

combine
load displacement data
with residual profile



some definitions



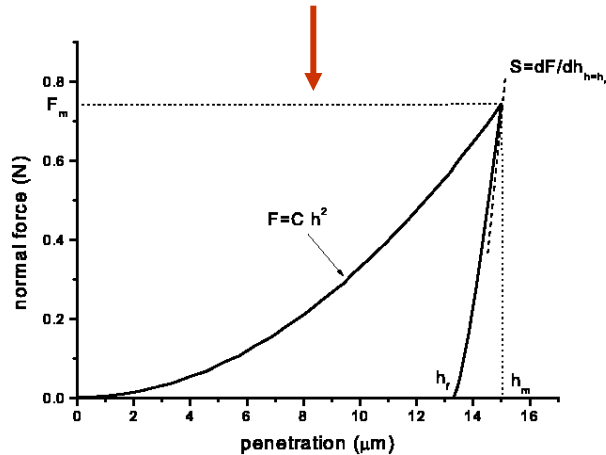
$$\sigma = \begin{cases} E\varepsilon & \varepsilon < \varepsilon_y \\ \sigma_y \left(1 + \frac{E}{\sigma_y} \varepsilon_{pl} \right)^n & \varepsilon > \varepsilon_y \end{cases}$$

ε = true strain, σ = true stress

We can describe the elastic behavior by E (ν)
and the plastic behavior by σ_y and n

conical tips: algorithm I

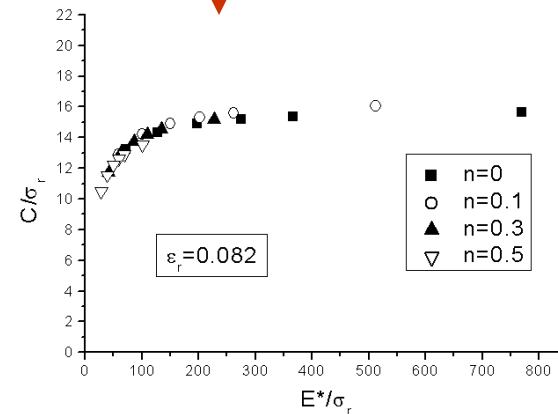
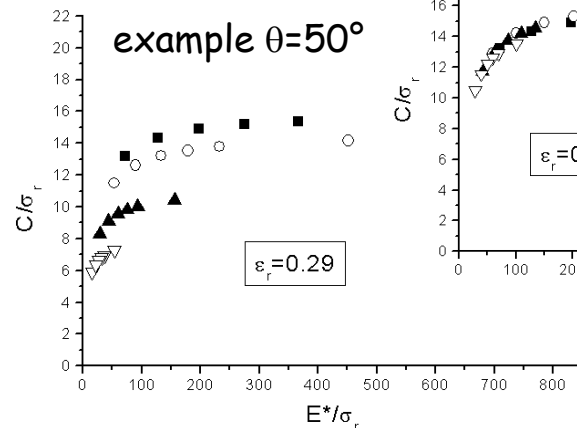
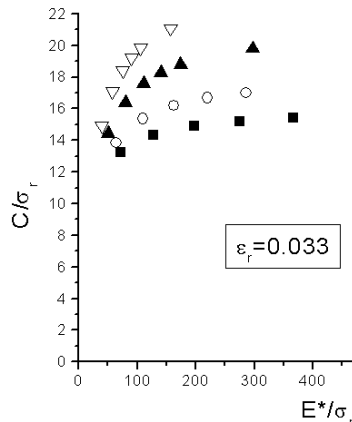
parametric finite element calculations of a large set of typical E , σ_y , n and θ



modeling of loading: $F = Ch^2$

for a given θ find representative ε_r for that all C/σ_r and E^*/σ_r are superimposed on one curve (Dao, Acta Mater. (2001),
 -> average total strain independent on strain hardening

F-h for a large number of combinations of σ_y , n , θ

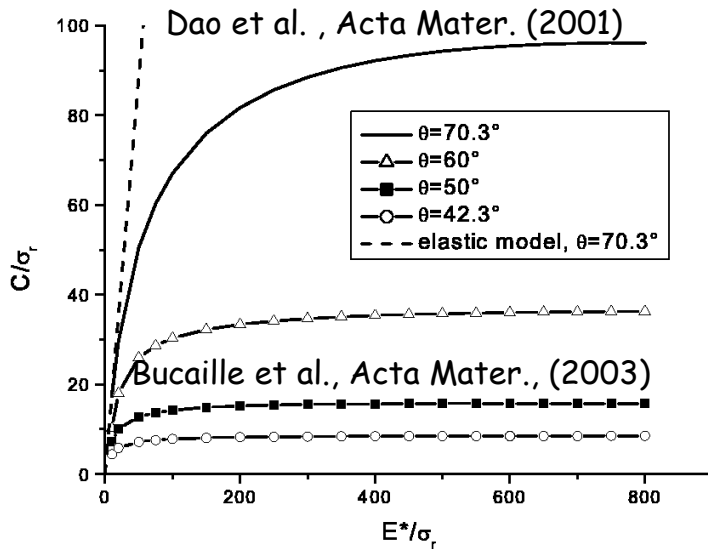


calculate back σ_y , n

Dao, Acta Mater. (2001)

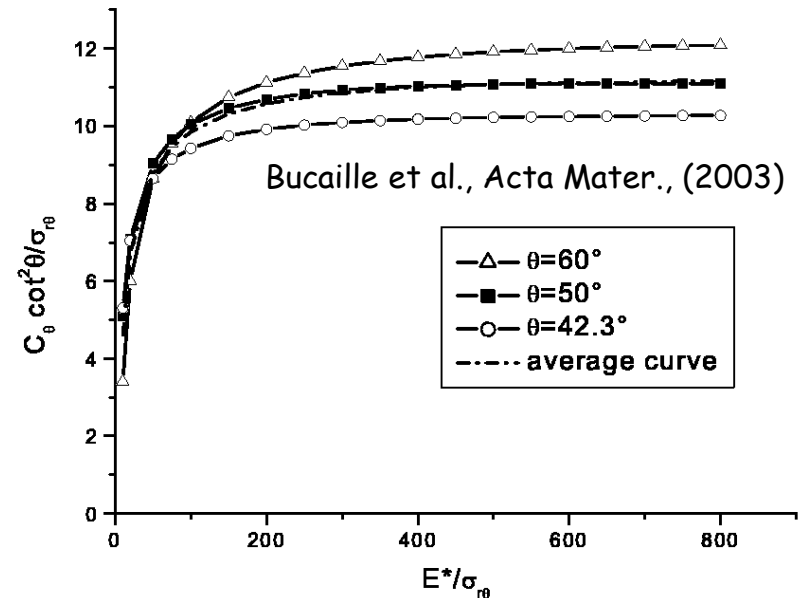
conical indenters: algorithm II

normalized dimensionless functions



master curve:

relation between C and E , σ_r for any value of θ



representative **strain**

$$\varepsilon_r = 0.105 \cot \theta$$

and the corresponding **stress** computed with the Π_1 function below:

$$\Pi_{1\theta} = \frac{C_\theta}{\sigma_{r\theta}} = \tan^2 \theta \left\{ 0.02552 \left[\ln \left(\frac{E^*}{\sigma_{r\theta}} \right) \right]^3 - 0.72526 \left[\ln \left(\frac{E^*}{\sigma_{r\theta}} \right) \right]^2 + 6.34493 \left[\ln \left(\frac{E^*}{\sigma_{r\theta}} \right) \right] - 6.47458 \right\}$$

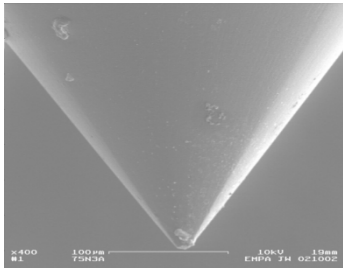
-> two simple formula based on E^* , C_θ from experimental load-displacement data

conical indenters: experiment

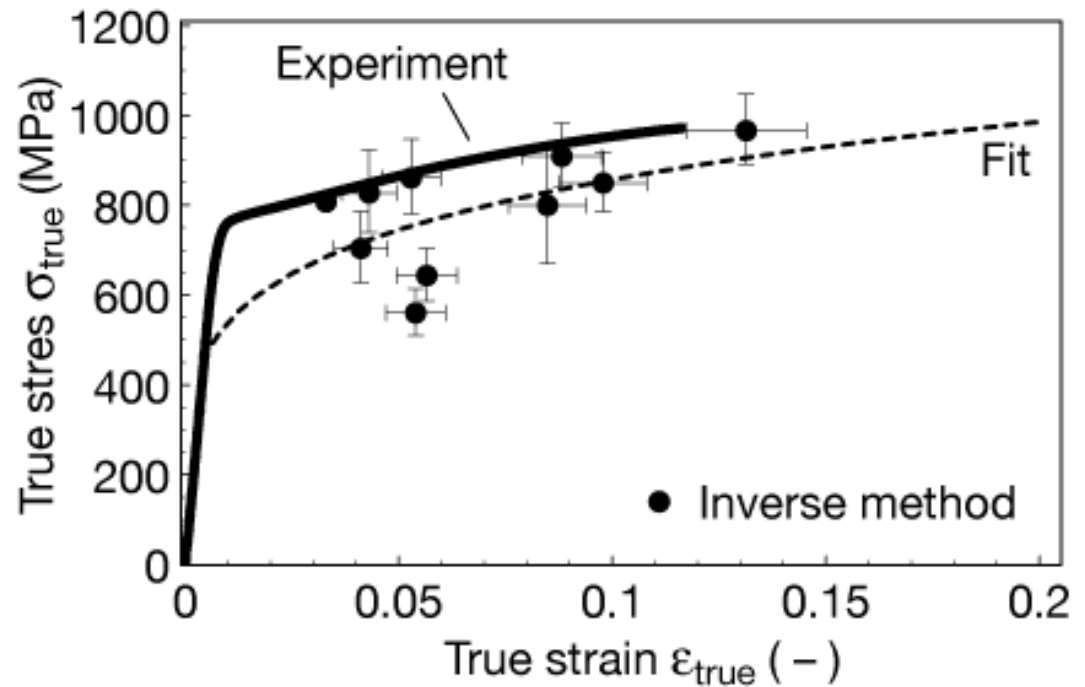
Material:
CuCo₂Be

Indentation tests:
Zwick Universal
hardness tester

Diamond tip geometries
38.6°, 47.0°, 50.0°, 51.0°, 61.7°
62.8°, 63.3°, 67.7°, 68.6°

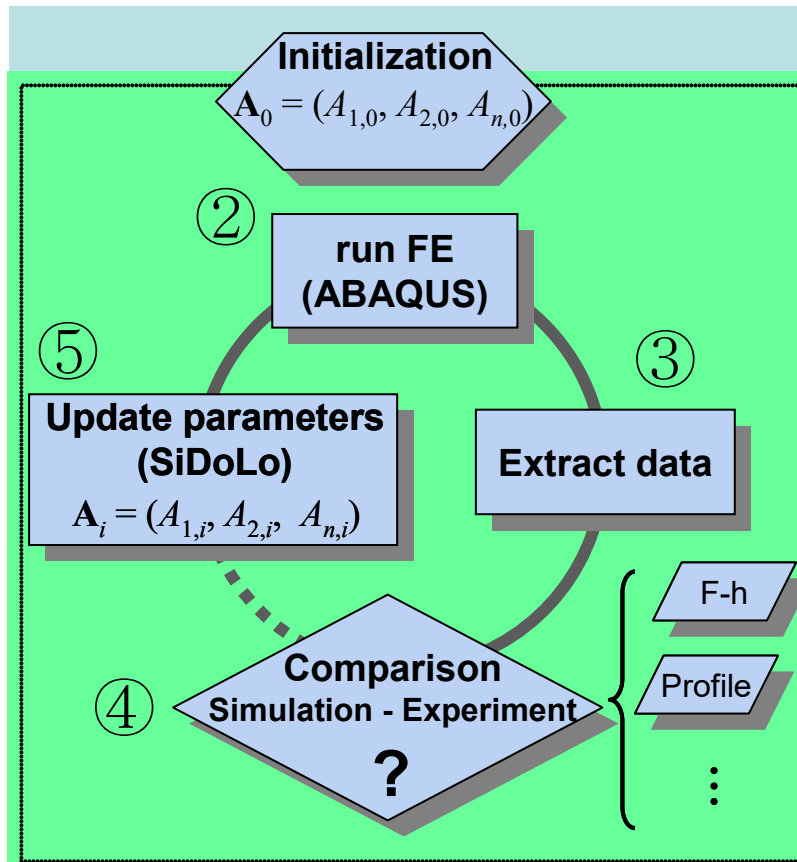


& Vickers (70.3°)

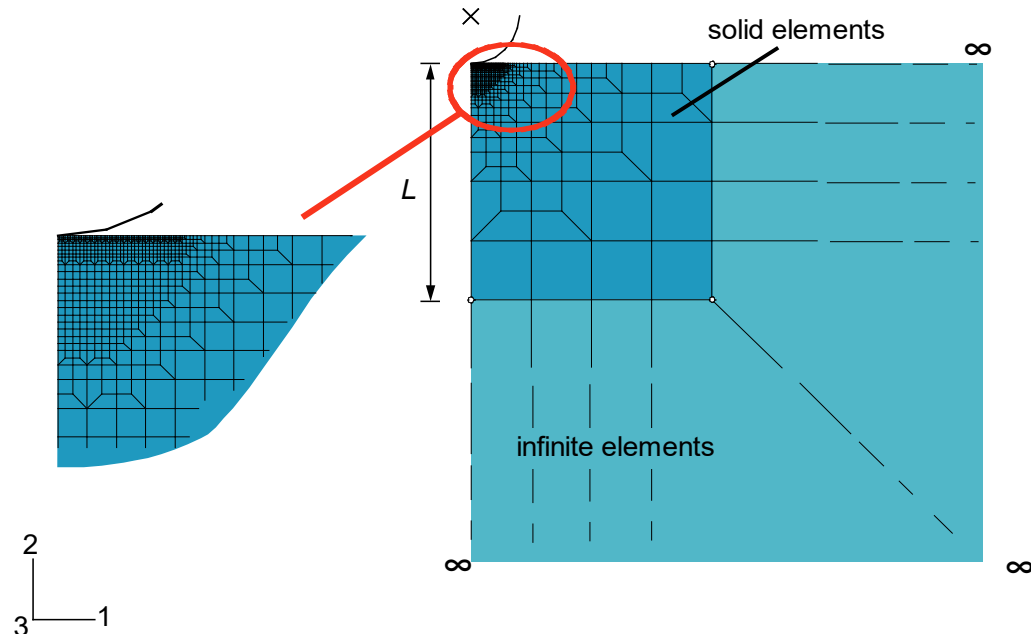


spherical indentation: algorithm I

idea: optimize FE calculation until calculated data fit experimental topography and load-displacement data

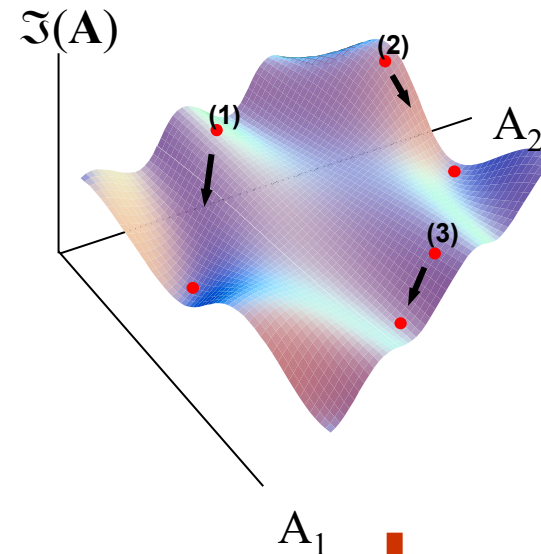
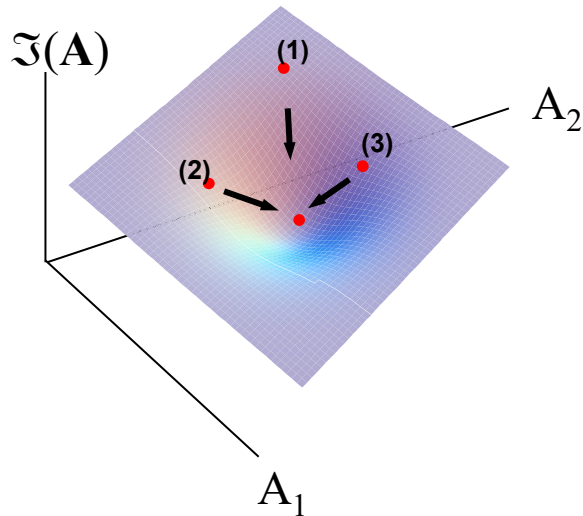


FE meshing scheme optimized for minimizing computing time; combination of finite and infinite axisymmetric elements (to eliminate boundary effects (i.e. $u_i = 0$) on numerical solution)



spherical indentation: algorithm II

But: Ill conditioned problem?



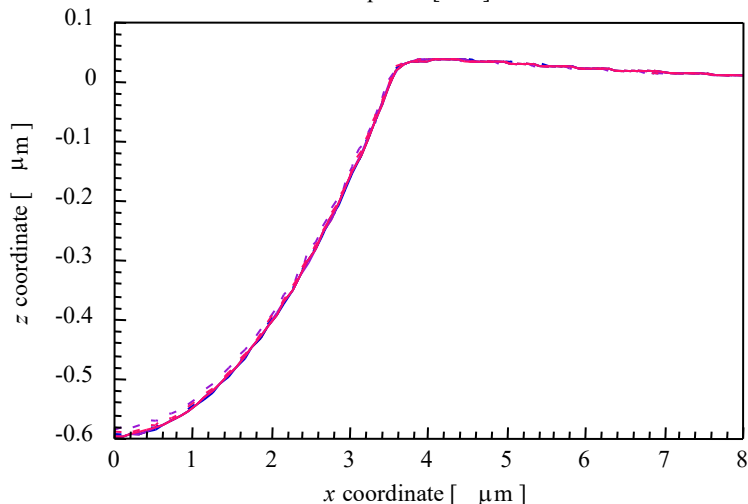
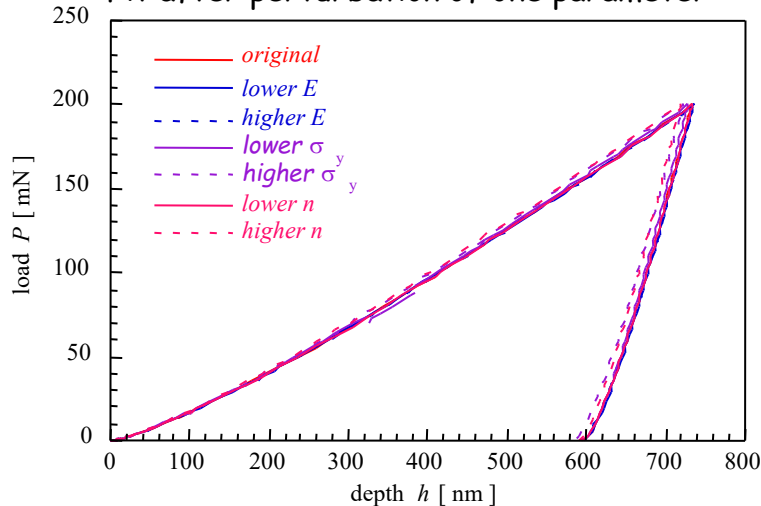
Fitting: Vary systematically parameters around starting point and “go a step in direction of the deepest gradient” to minimize the difference $J(\mathbf{A})$ between simulated and experimental data

Practical guideline: Good estimation of initial values and stabilization of the solution by increasing the number of fitted parameters

spherical indentation: model calculation

Validation: Fit back a finite element calculation after starting from 10% perturbation

Fit after perturbation of one parameter



Material model:

$$\sigma = \begin{cases} E\varepsilon \\ \sigma_y \left(1 + \frac{E}{\sigma_y} \varepsilon_{pl} \right)^n \end{cases}$$

Example calculation (perturbation of all param.):

	E [GPa]	σ_y [MPa]	n []
Original	210	500	0.3
Start	189	450	0.27
Fit	208	494	0.30

spherical indenters: experiment

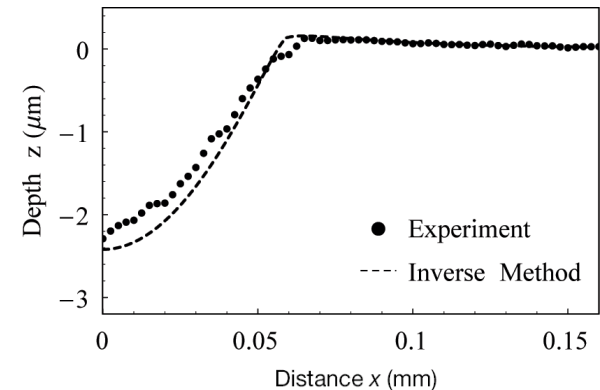
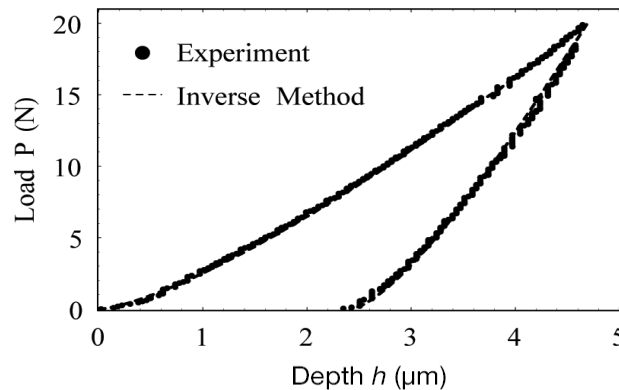
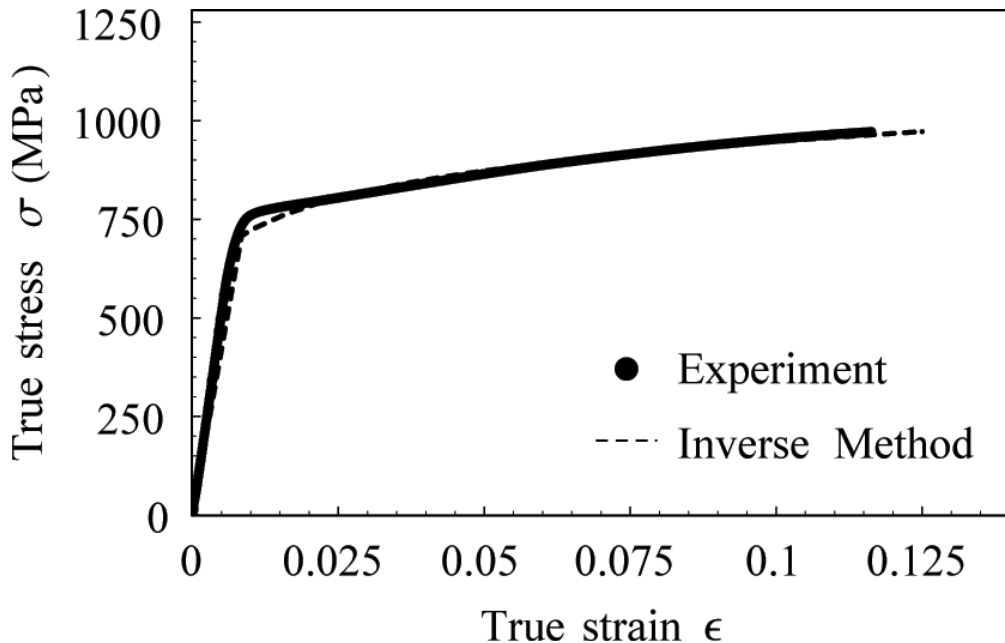
material:
CuCo2Be

indentation tests:
universal
hardness tester

topography measurements:
laser profilometer

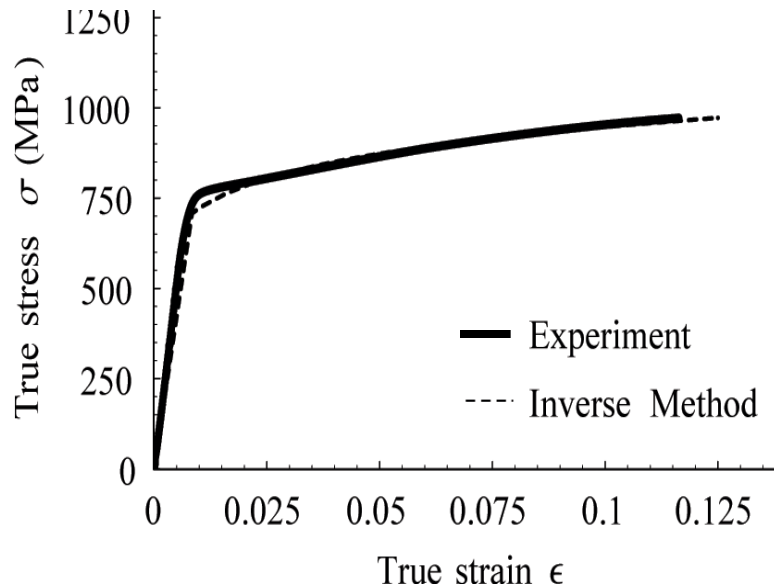
tip geometry:
WC sphere $R=0.5\text{mm}$

fit:
perturbation of
all parameters
by 10%

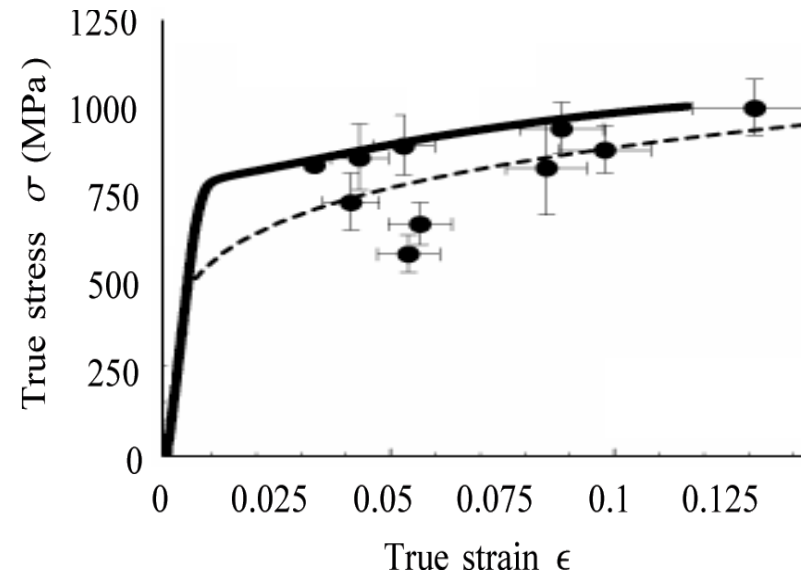


comparison of "conical" and "spherical" indentation methods

sphere & residual imprint



multiple cones



outlook: application to coatings

material:

NiCo electrodeposited coating

tensile tests:

UV-Liga tensile test bars

indentation tests:

MTS nanoindenter XP

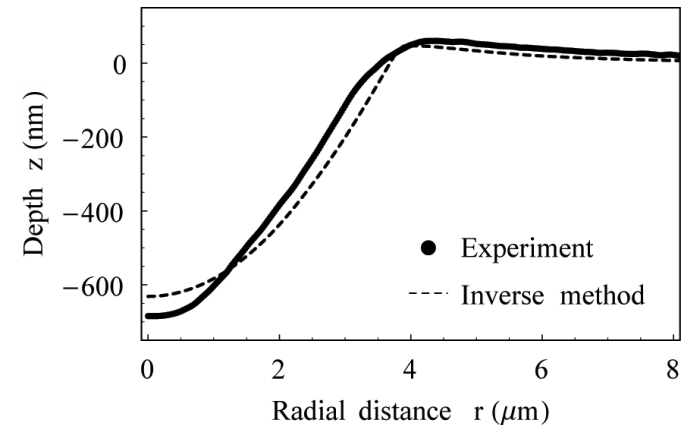
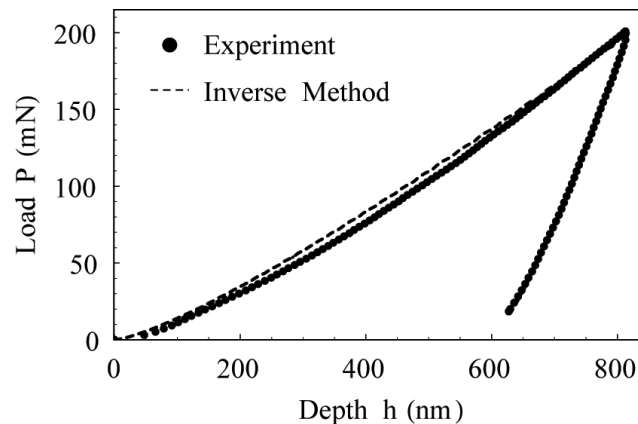
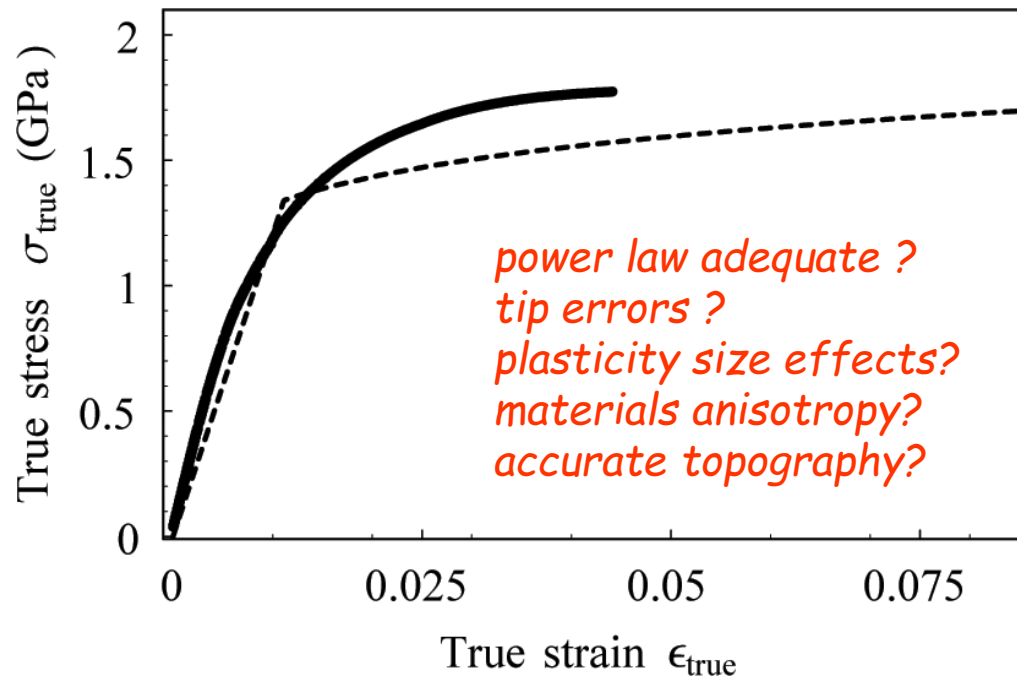
topography measurements:

AFM

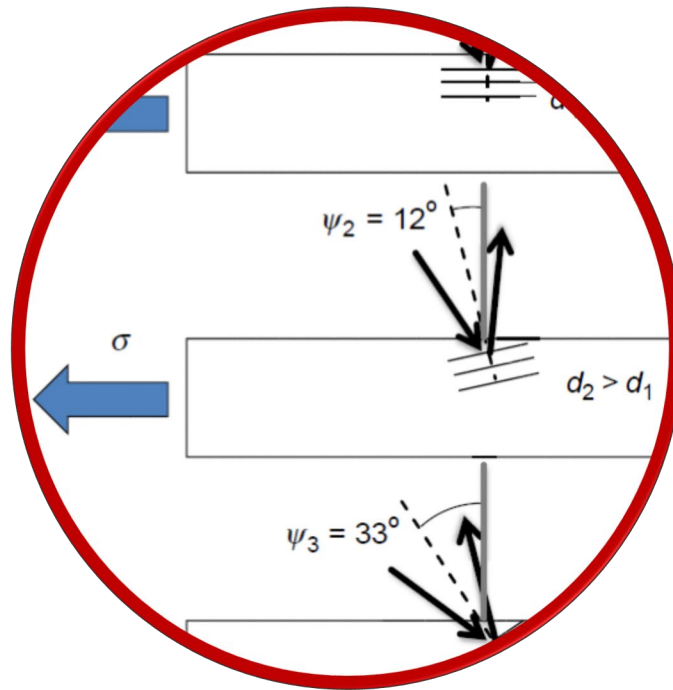
tip geometry:

diamond

spherical $R=10\mu\text{m}$



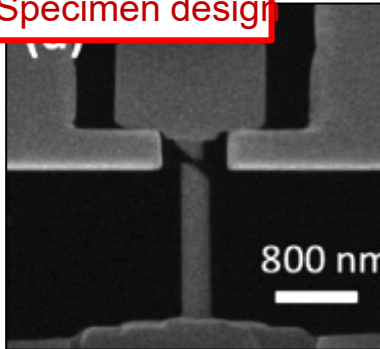
Microscale tensile testing



Challenges of small scale tension

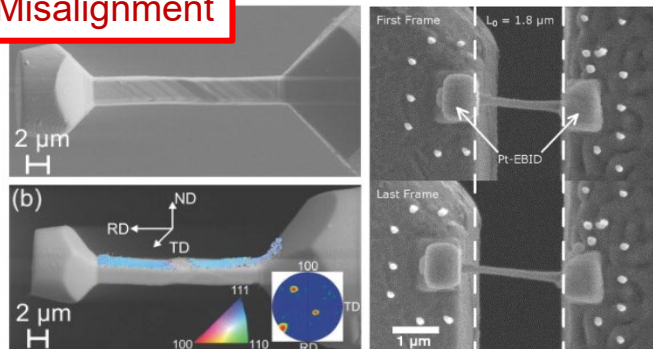
Possible issues in small scale tensile experiments:

Specimen design



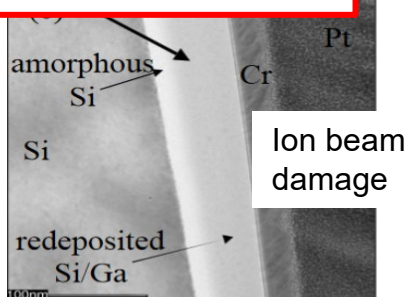
Liontas et al. *Acta Mater.* (2016)

Misalignment



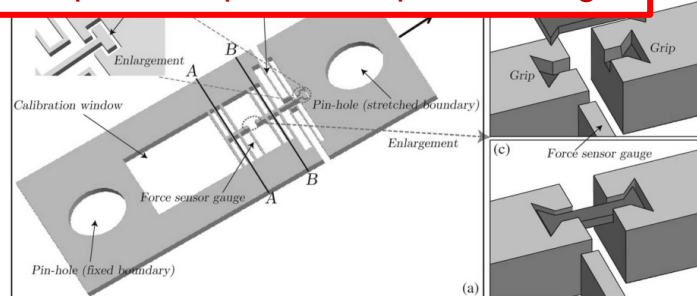
Kiener et al. *Acta Mater.* (2008) Magagnosc et al. *Sci. Rep.* (2015)

Fabrication artifacts



Rajsiri et al. *Microsc Microanal* (2002)

Complex setup and sample handling



Kang et al. *J. Microelectromech. Syst.* (2010)

Requirements for the new methodology:

1. Minimise stress concentrations.
2. Optimise specimen fabrication via FIB.
3. Ensure alignment.
4. Avoid complex and tedious manipulations. No single use setups.

In situ tension: single-crystal copper samples

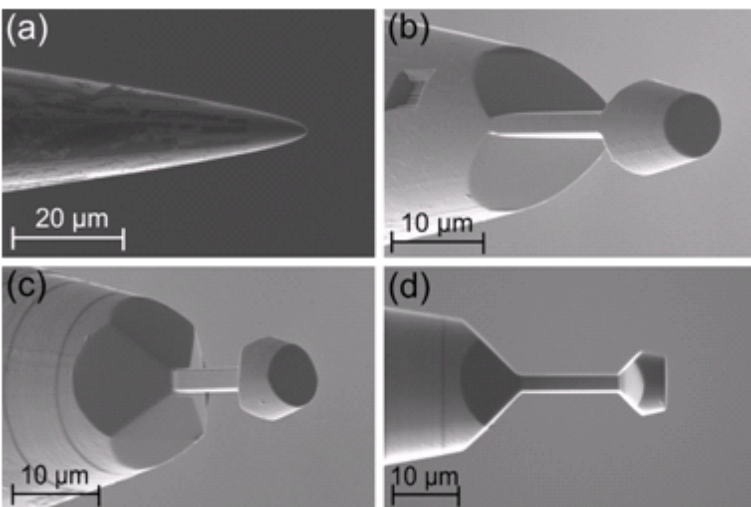


Fig. 1

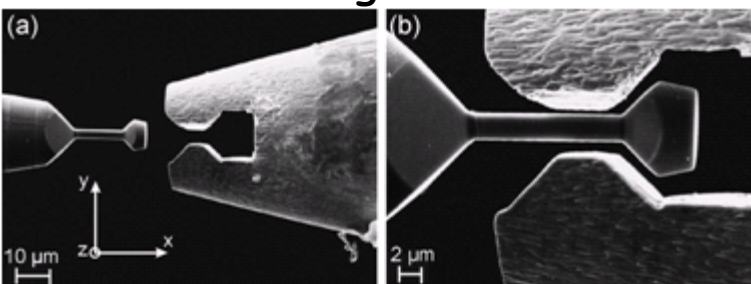


Fig. 2

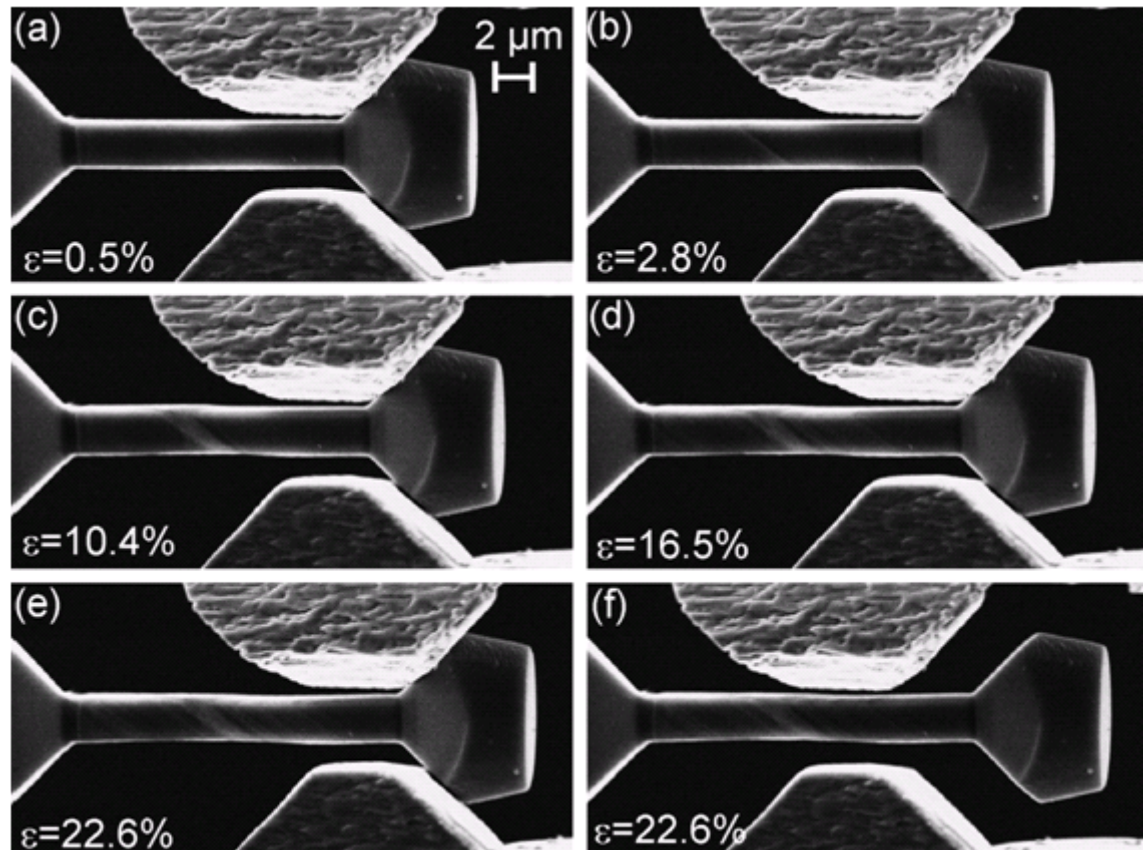
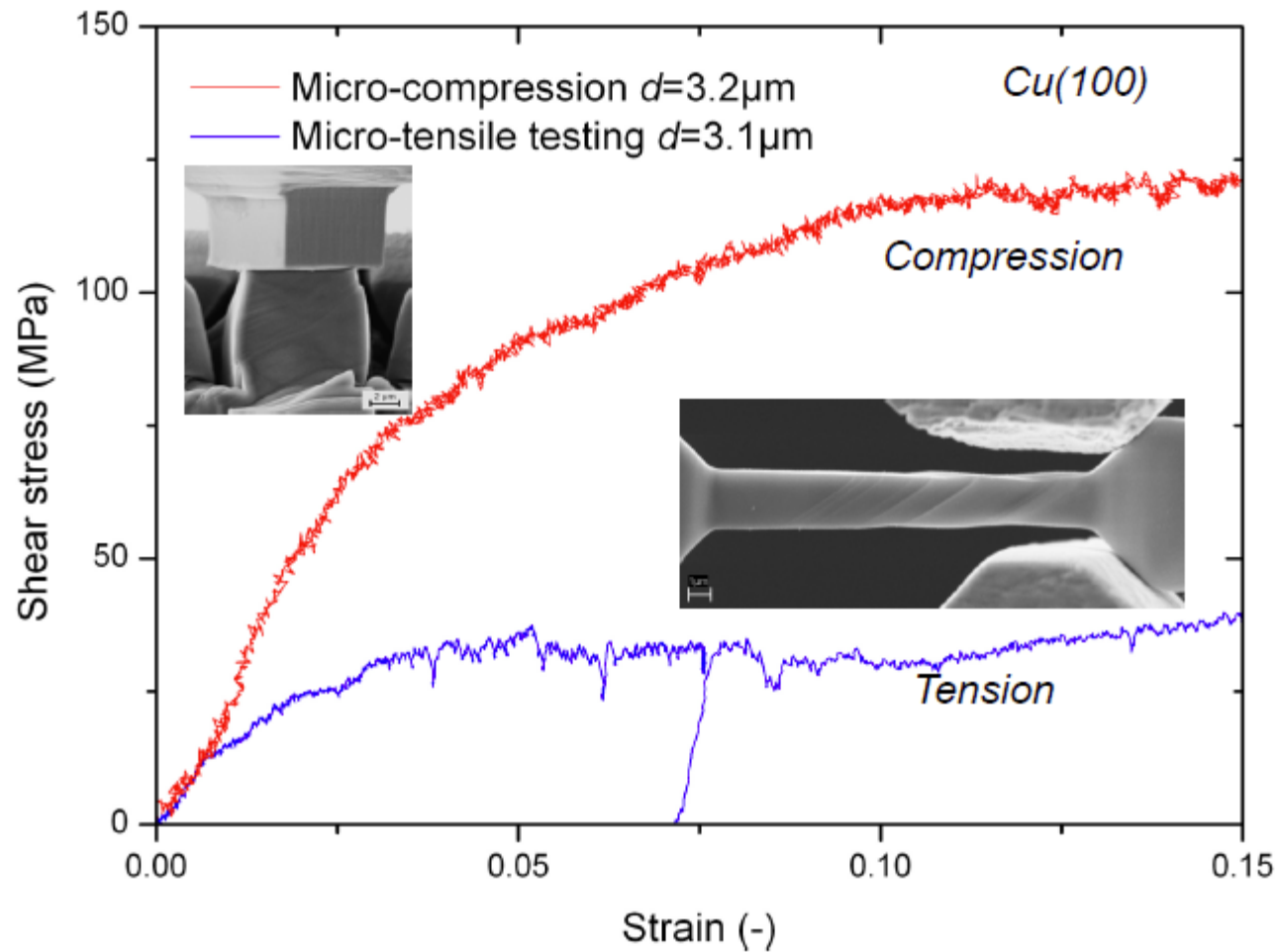


Fig. 3

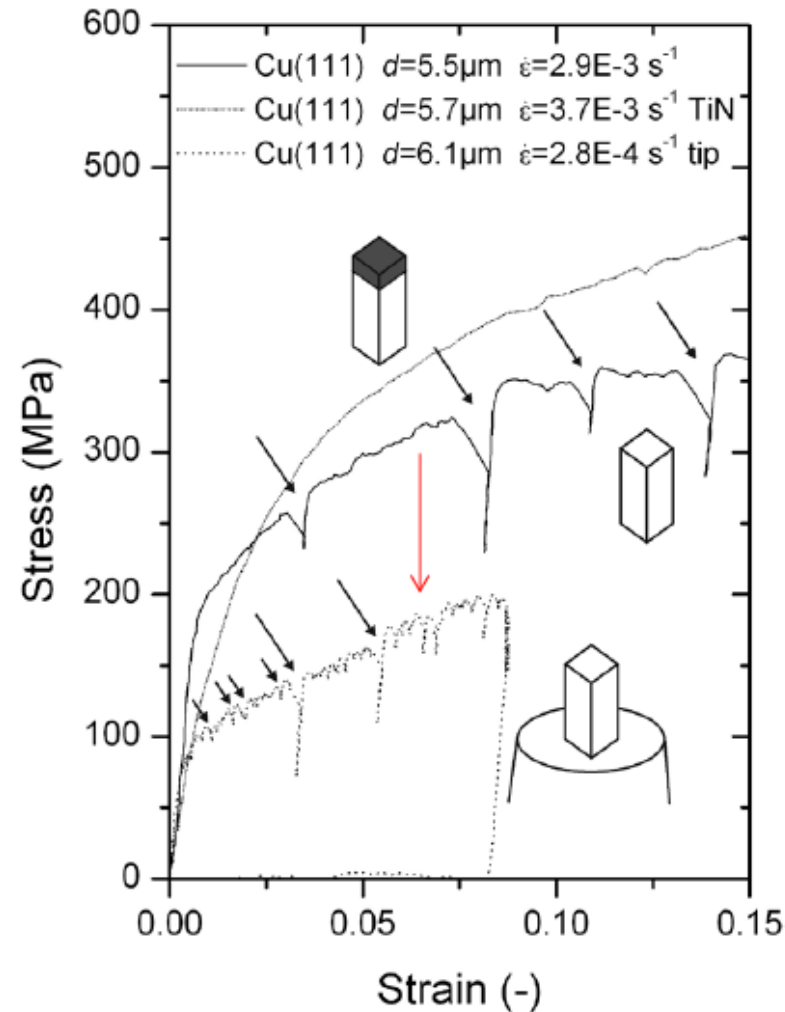
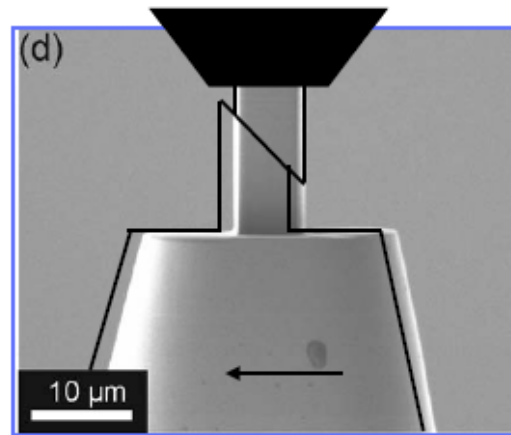
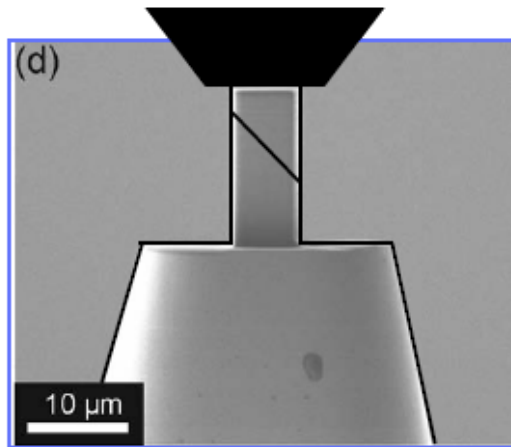
Compression versus tension

Cu(100) oriented tensile and compression sample



Compression versus tension

Micro-compression of a *Cu(100)* sample with reduced lateral stiffness



Kiener D, Motz C, Dehm G. *Mater. Sci. Eng. A* 2009;505:79.

Rotation of the Crystal Lattice

The slip direction rotates towards the tensile axis

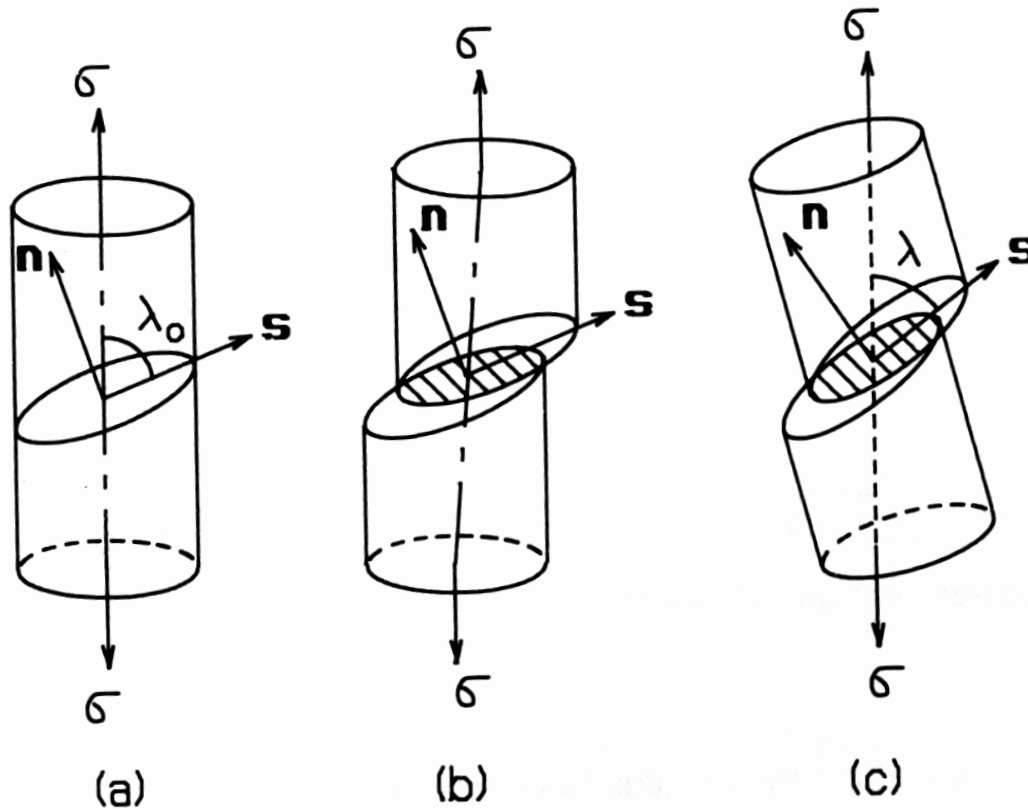
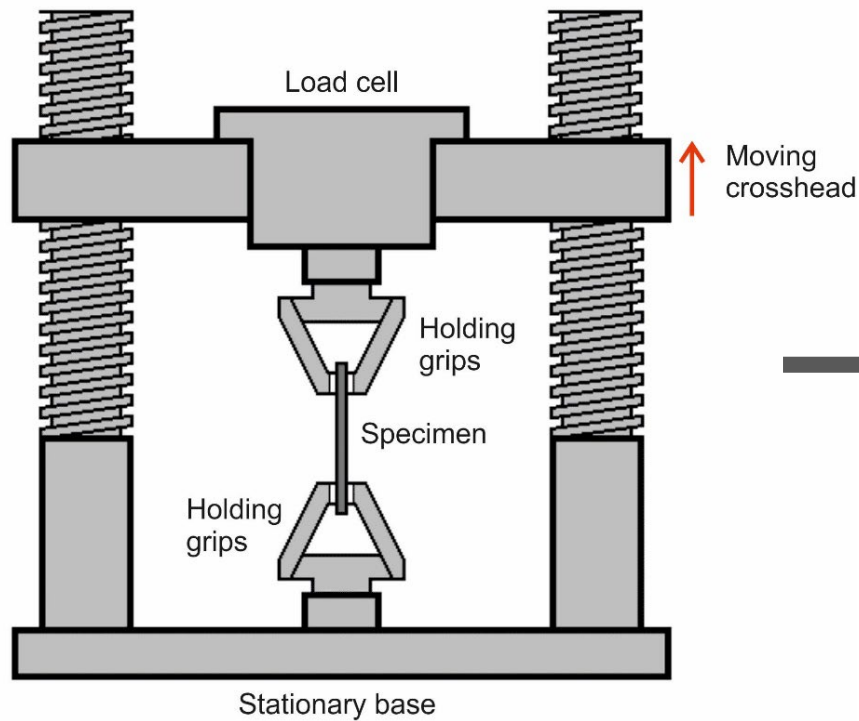


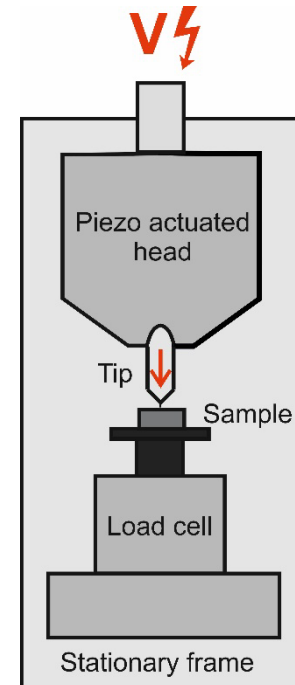
Fig. 10.5 Rotation of crystal lattice in tension

Standard testing at the microscale?

Macroscale



Microscale

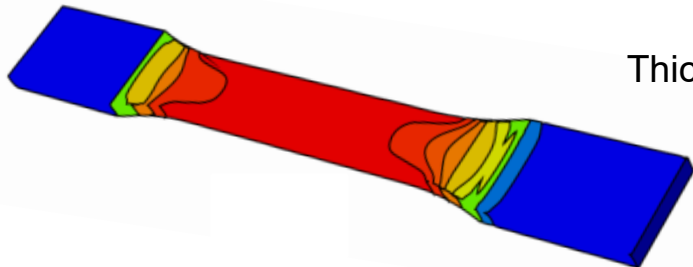


Standard testing at the microscale?

Macroscale

ASTM 638 Type V

Free end



Free end

Variables

Thickness, width, radius, head shape

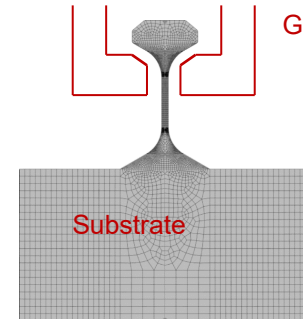


Microscale

New specimen geometry

Free end

Gripper



Substrate

Goals

Homogeneous stress in the gauge section

Low stress concentration at the radius (<5%)

Constraints

Maximum gauge dimensions = 10 μm (**lamella**)

On bulk substrate (**no micromanipulation**)

FIB milling fabrication

Specimen design

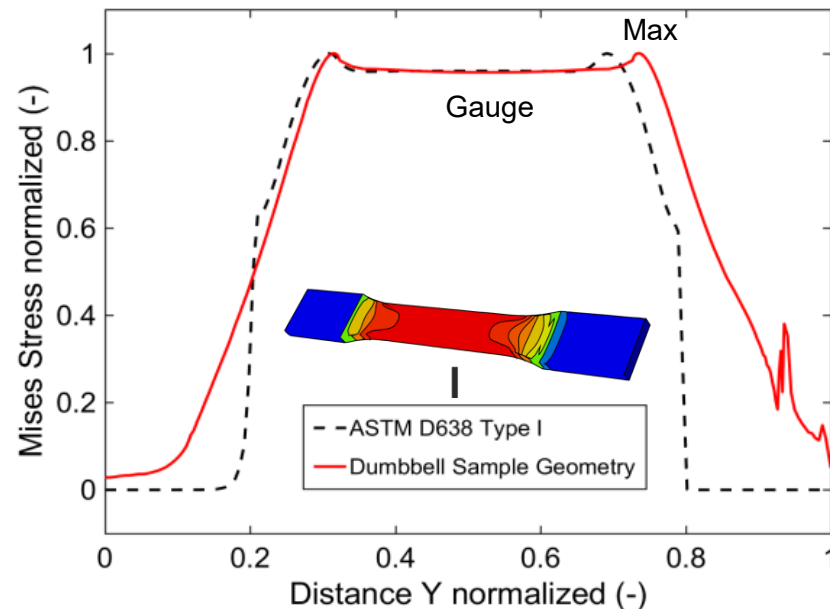
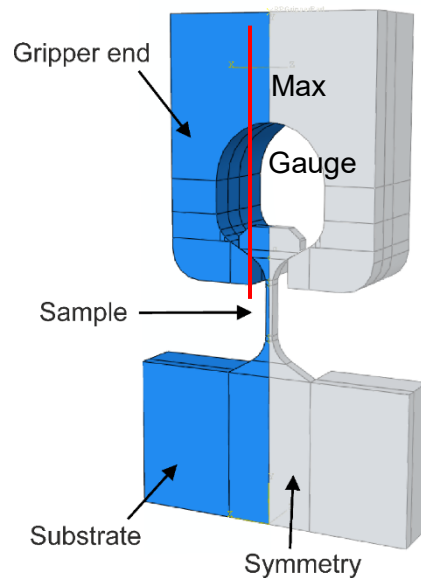
- **Finite elements analysis using Abaqus/Standard:**

- $\frac{1}{4}$ Symmetry, C3D8R (no bending).
- Linear elastic material properties.
- Hourglassing controlled and checked with ALLAE/ALLIE < 1%.

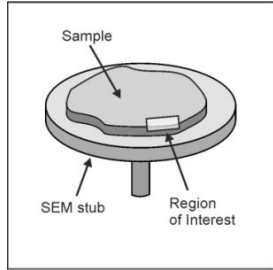
Variables: thickness, width, radius, head;

Fixed: gauge length = 10 μm

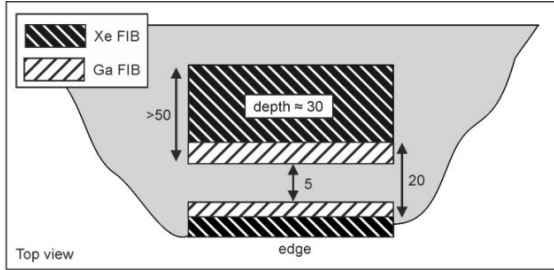
Output: stress profile and stress concentrations



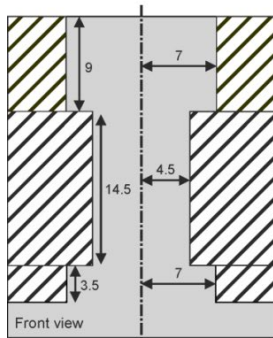
Specimen design



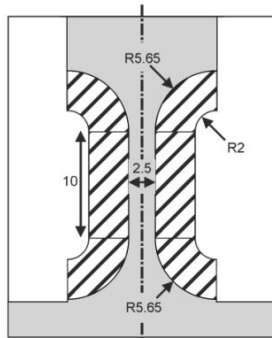
0. Sample on a SEM stub



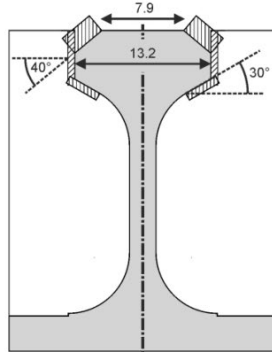
1. Xe plasma-FIB milling (30kV, 30 nA) and Ga FIB (30kV, 4.5nA and 0.7nA)



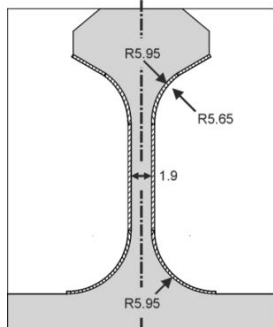
2. Coarse polishing A (30kV, 4.5nA)



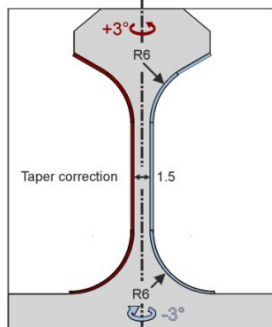
3. Coarse polishing B (30kV, 4.5nA)



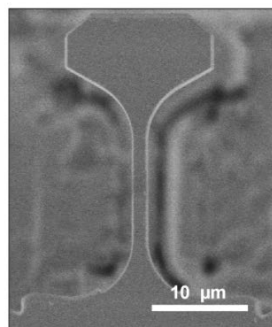
4. Fine polishing A (30kV, 0.7nA)



5. Fine polishing B (30kV, 0.7nA)



6. Taper polishing (15kV, 0.4nA)



Final geometry

Homogeneous stress profile in the gauge

Gauge section length $\geq 4w$.

Low stress concentration at the radius

$$K_c = \frac{\sigma_{max}}{\sigma_{gauge}}$$

Maximise geometry ratio r/w ($r/w \geq 4 \rightarrow K_c < 1.05$).

K_c independent of thickness.

Fabrication via FIB possible

Optimized FIB Milling protocol

Microtensile gripper

Requirements: Solutions:

Mounting
Handling



Large base

No creep
Reproducible



Silicon

Alignment
Gripping

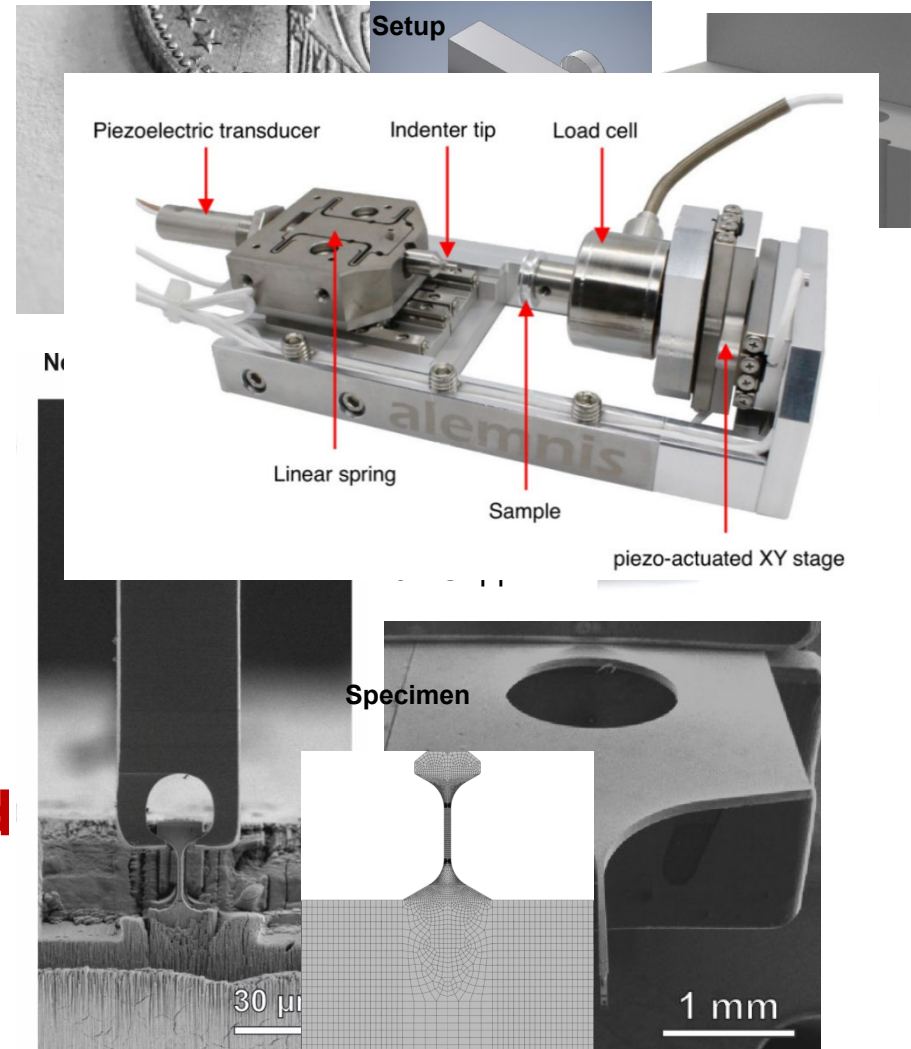


Needle shaped

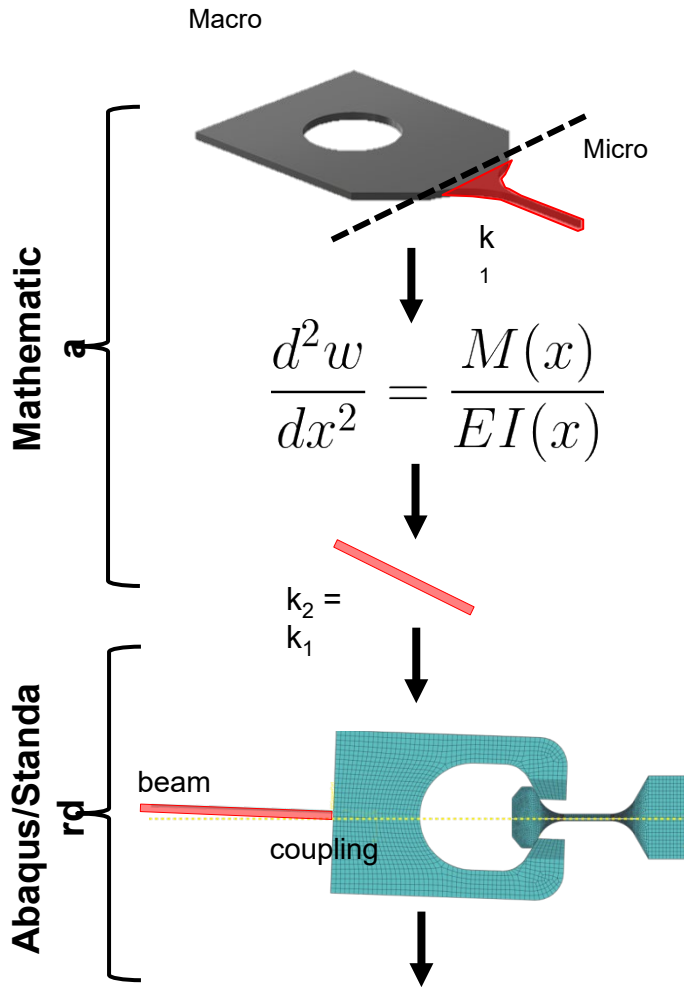
Lateral compliance



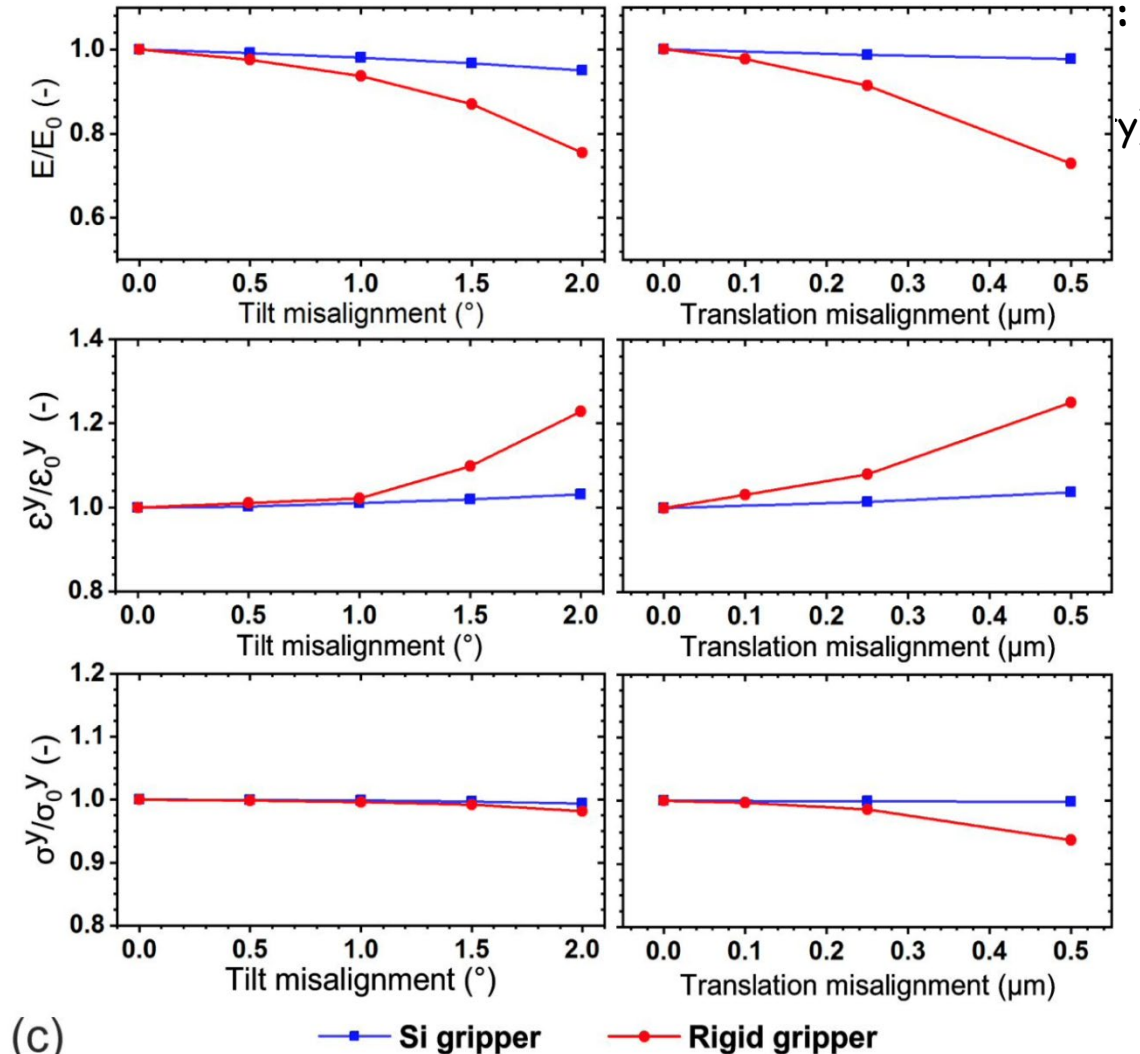
Self-aligning



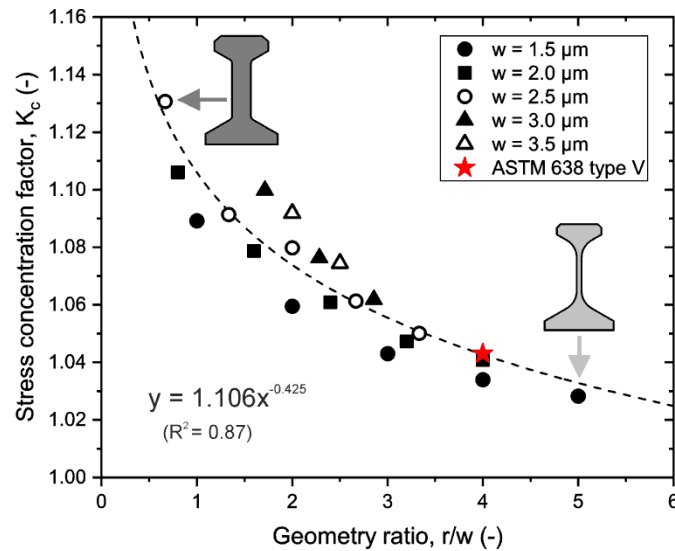
Misalignment tolerance



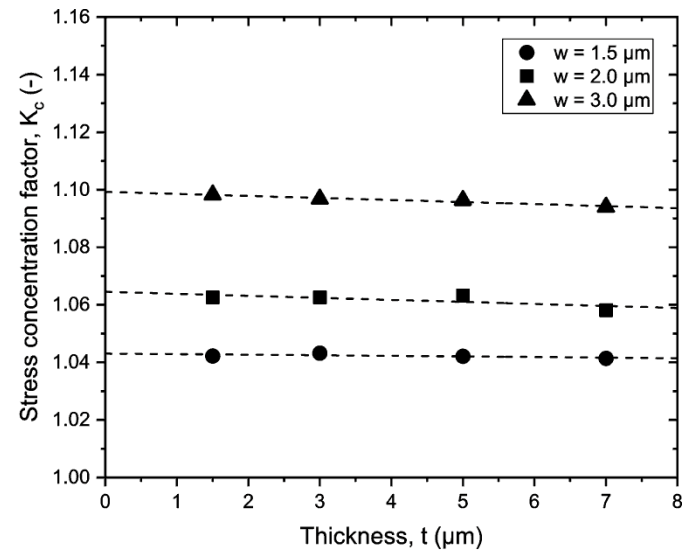
Which needle shape is better?



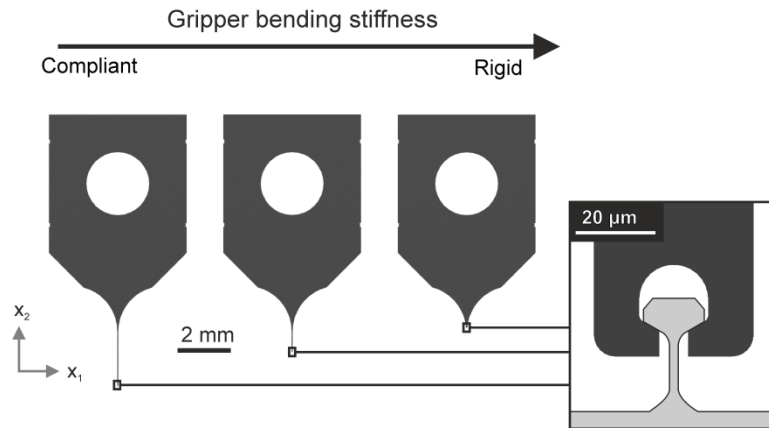
Misalignment tolerance



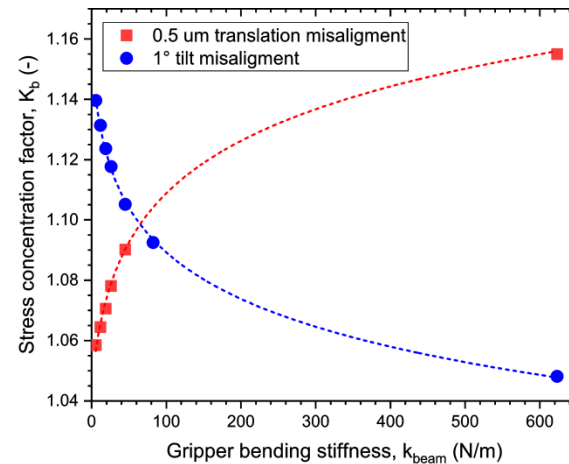
(a)



(b)



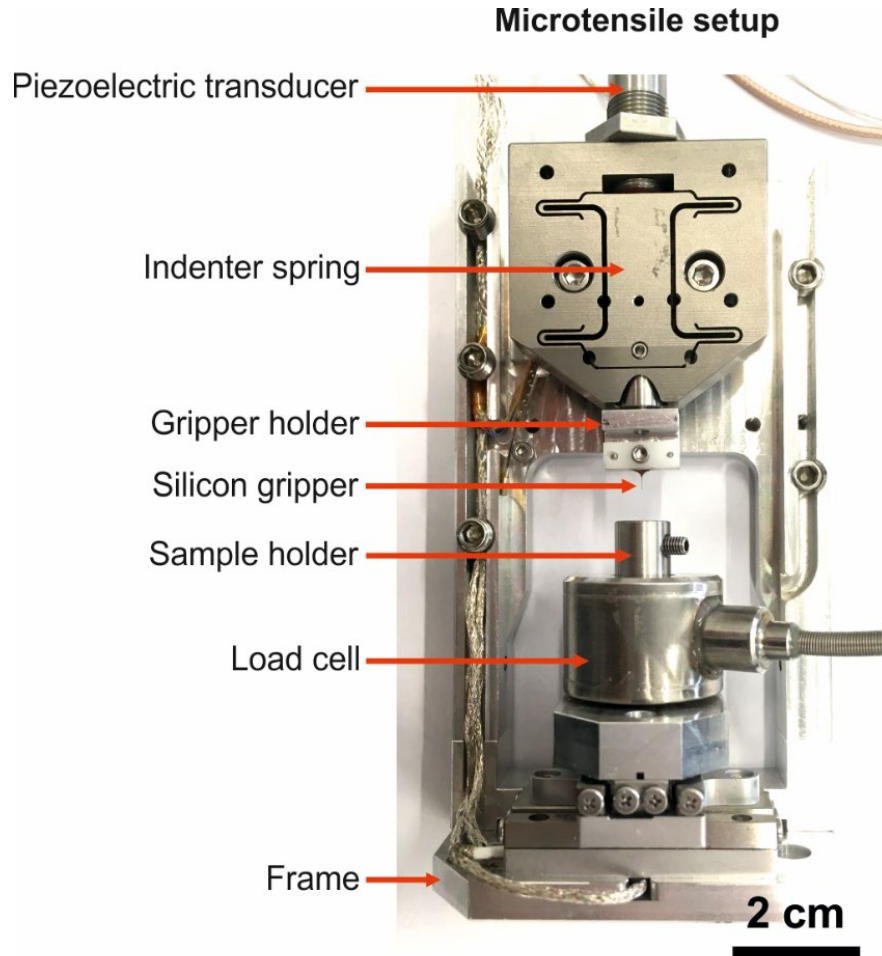
(a)



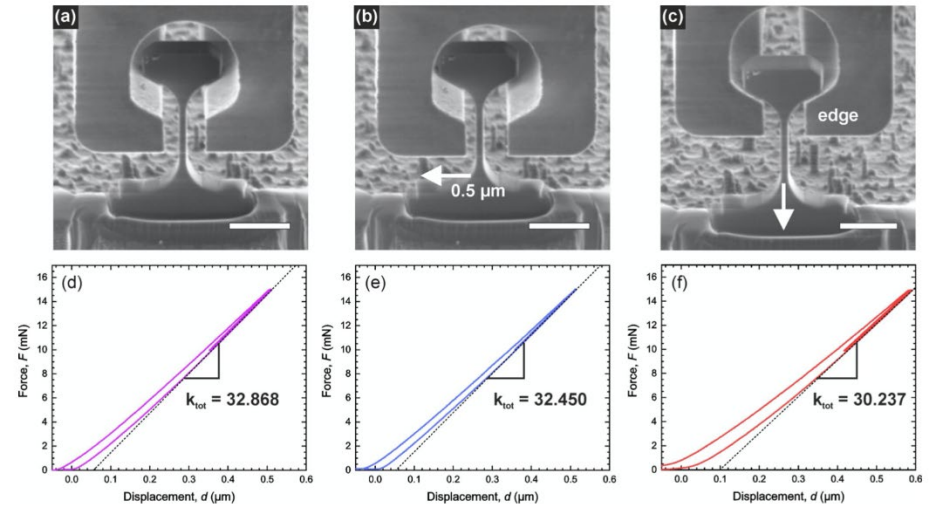
(b)

Microtensile setup

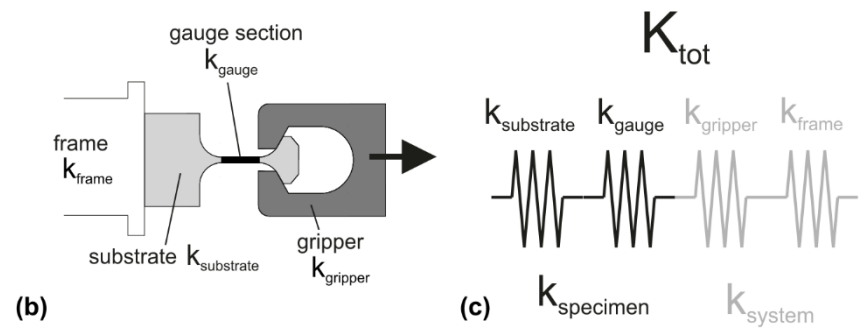
Setup components



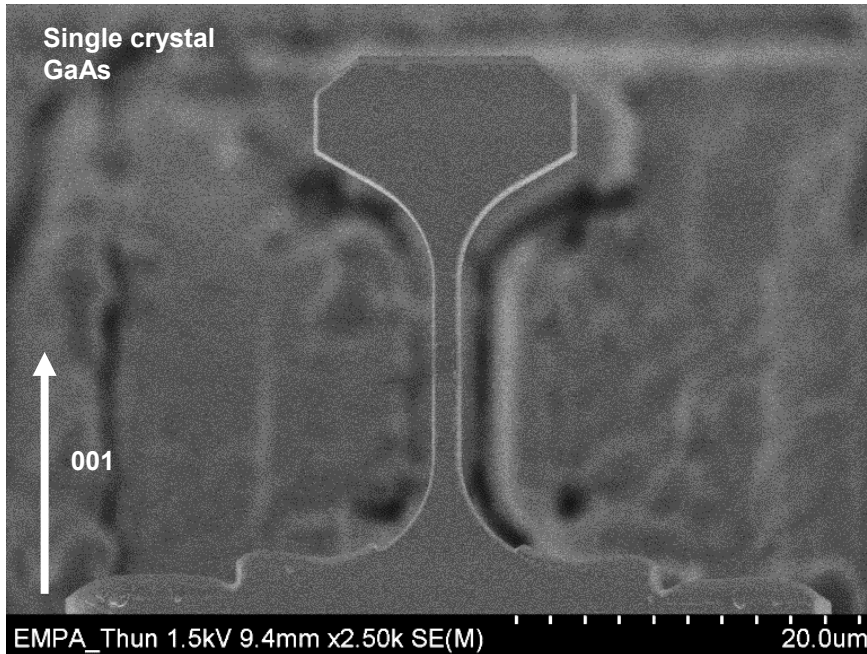
Misalignment tolerance



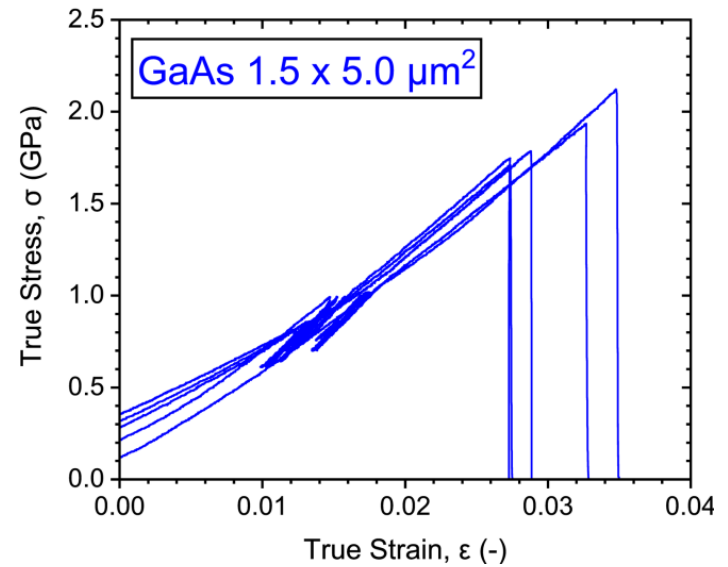
Compliance correction



Validation and application



Experiment on GaAs

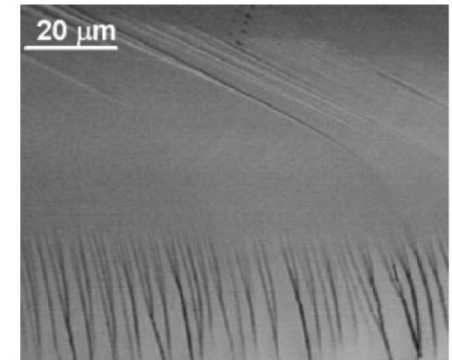
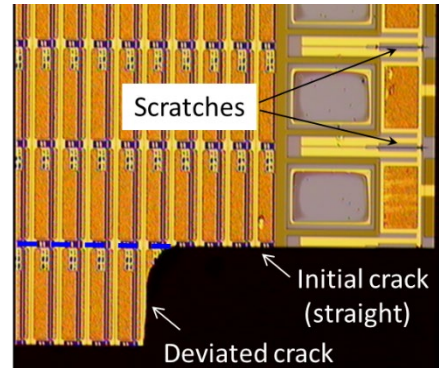
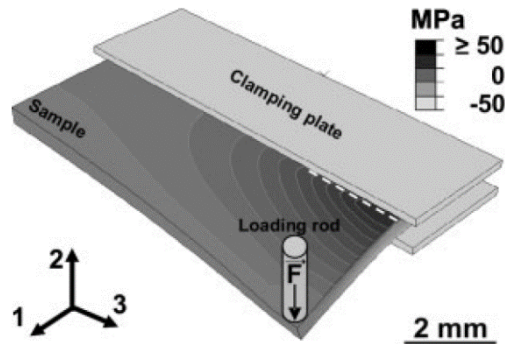


Property	Literature value (GPa)	Measured (N = 5) (GPa)	Relative difference (%)
Elastic modulus E_{001}	85.5		

More information in: A self-aligning microtensile setup: application to single-crystal GaAs microscale tension-compression asymmetry, *Journal of Material Research*, **34**, 2019

Case study: Tension-compression asymmetry in GaAs

Miniaturized components from GaAs

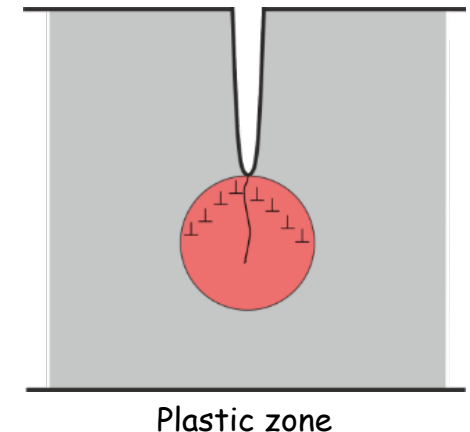


Wasmer et al., Adv. Eng. Mat.,
2005

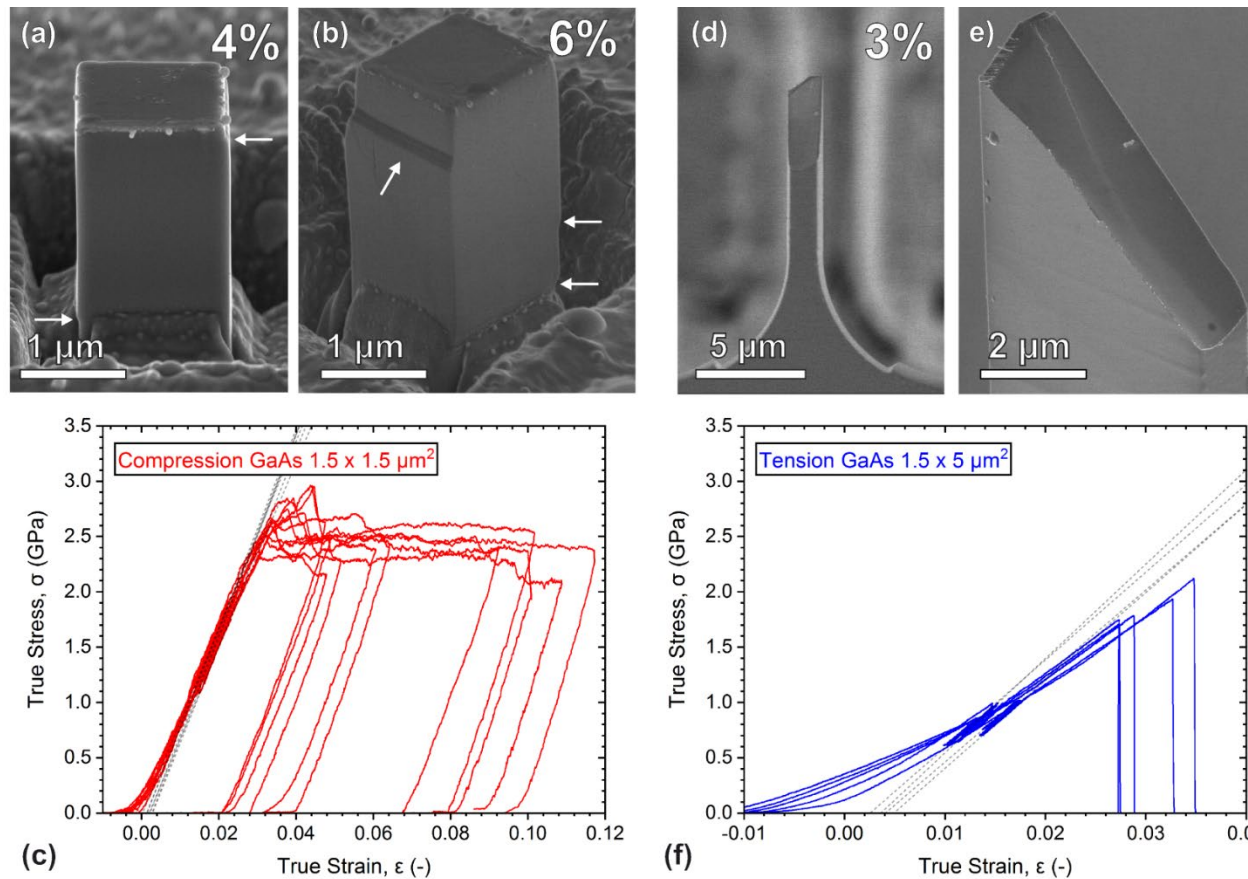
Production of microelectronic components:

- Cleavage of patterned GaAs wafers
- As component size decreases, cleavage becomes unstable
- Cleavage surface shows irregularities and defects
- Crack deviates from ideal path

→ Under which conditions does plasticity occur at small scales?



Tension-compression strength asymmetry in GaAs



- GaAs tested along the [001] crystallographic orientation.
- Specimen sharing a common thinnest dimension of $1.70 \pm 0.19 \mu\text{m}$.
- Plasticity in compression, brittle failure and lower strength in tension

Deformation and failure mechanisms of GaAs

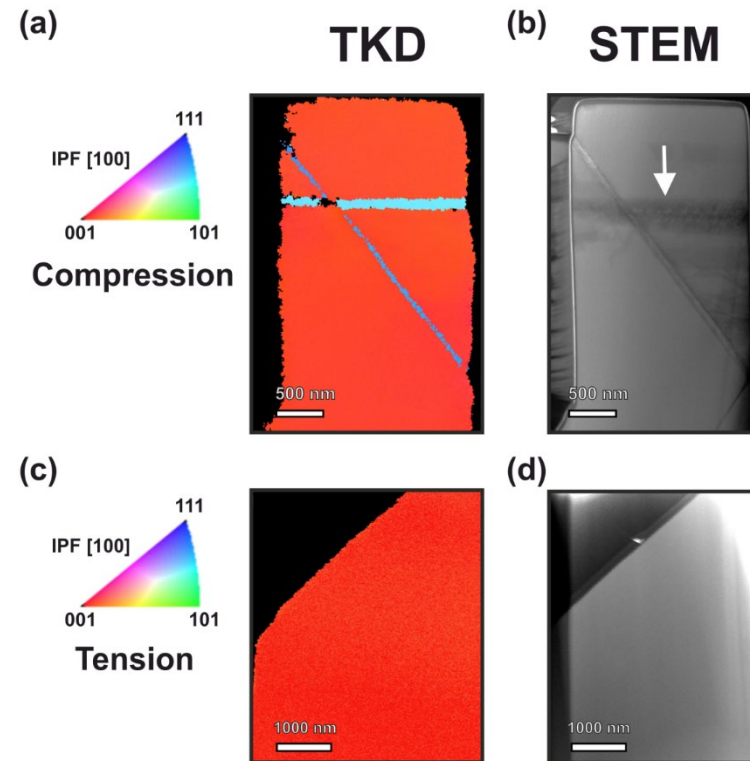


- In tension, failure is brittle
- No measurable dislocation activity in tension

- Estimated process zone size:

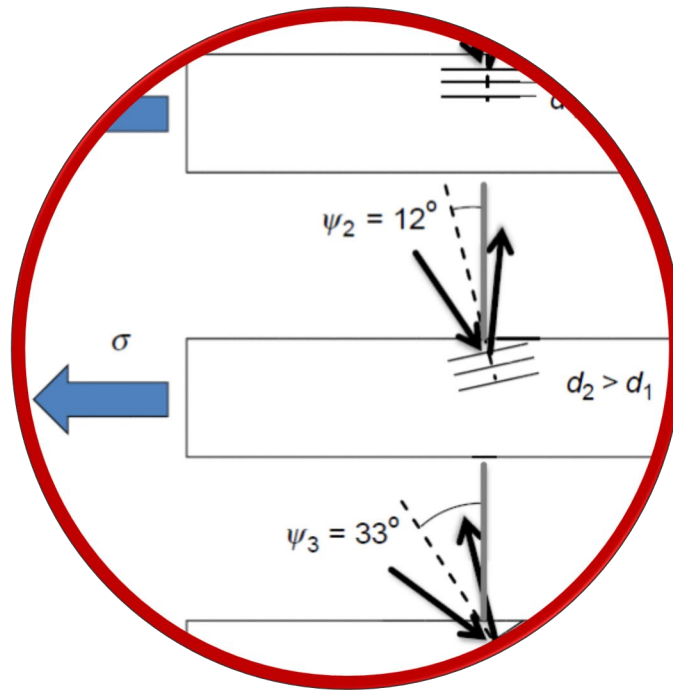
$$r_{pl} = \frac{1}{2\pi} \left(\frac{K_{Ic}}{\sigma_y} \right)^2 = 1.5 \text{ nm}$$

- Plastic deformation by twin formation in compression
- The critical size for brittle-to-ductile transition in compression lies between $1.7 \text{ }\mu\text{m}$ and $2.3 \text{ }\mu\text{m}$.
- Irregular fracture pattern likely related to mixed mode loading

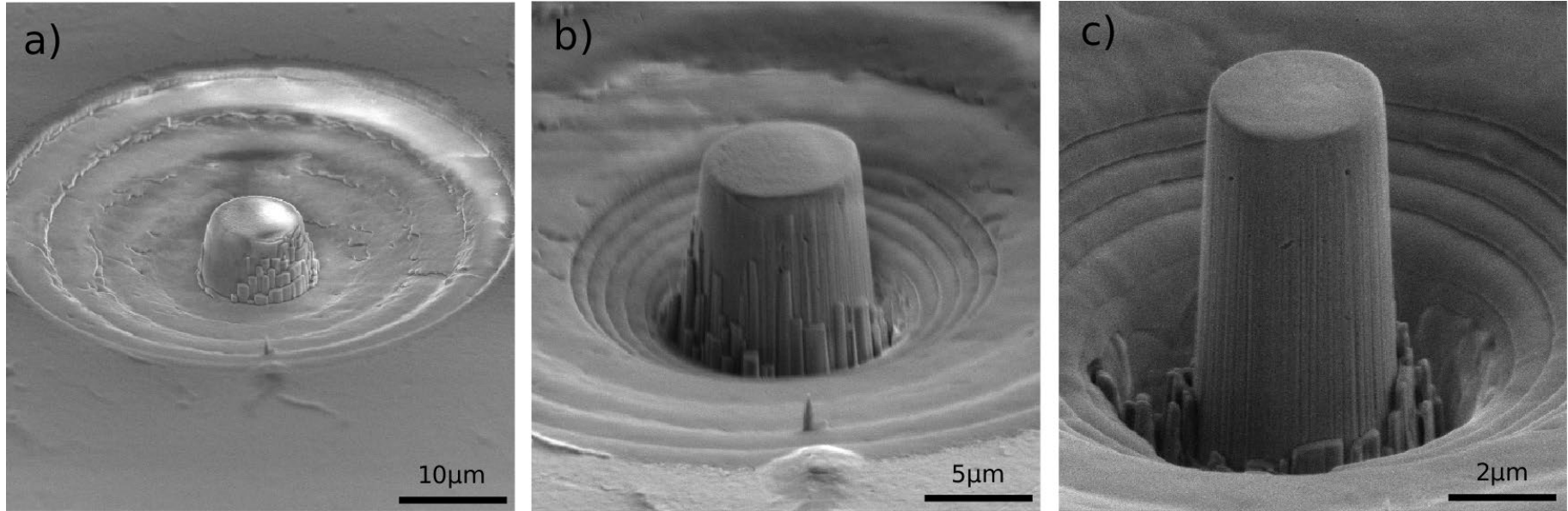


Casari et al., J Mat Res, 2019

Microcompression testing

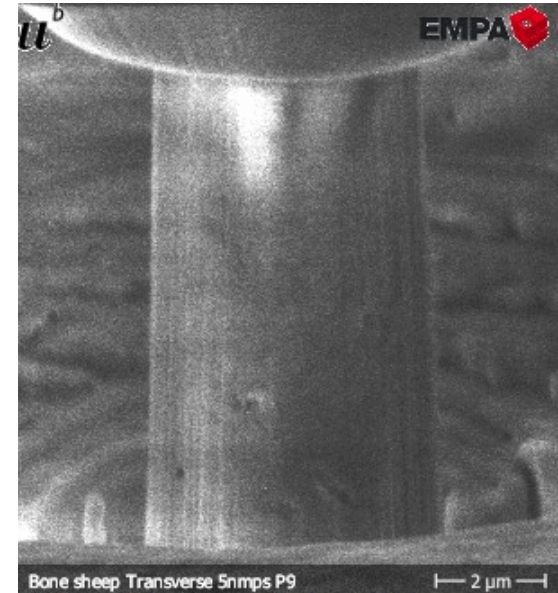
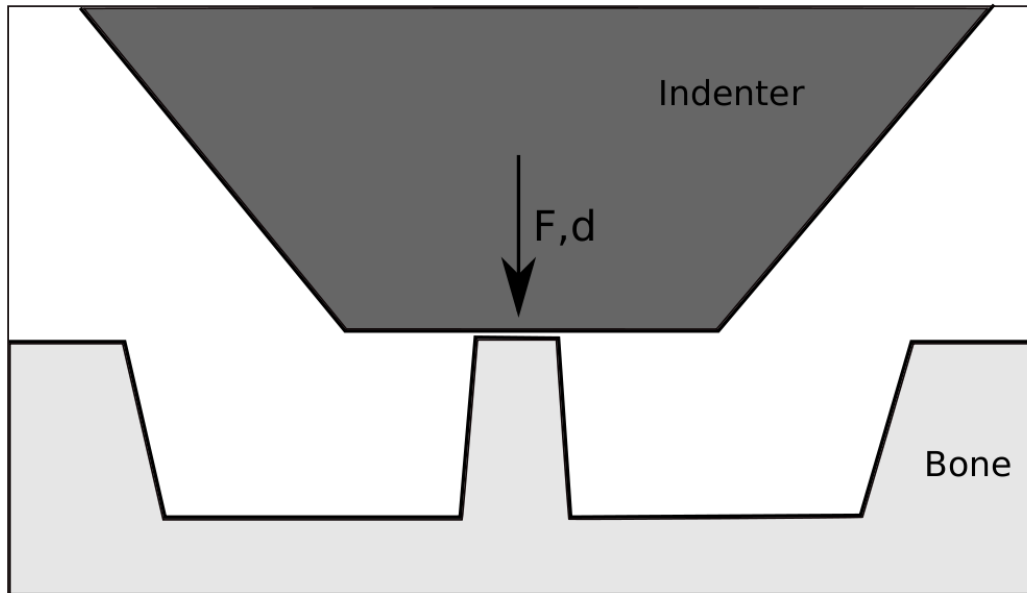


Micropillar Compression



- Micropillars machined using focused ion beam
- Compression with flat punch diamond indenter
- Uniaxial stress state → straightforward interpretation

Micropillar Compression

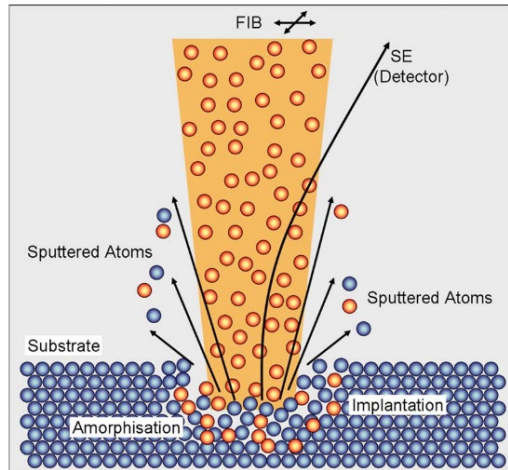


Schwiedrzik et al., Nat. Mat. (2014)

- Micropillars machined using focused ion beam
- Compression with flat punch diamond indenter
- Uniaxial stress state → straightforward interpretation

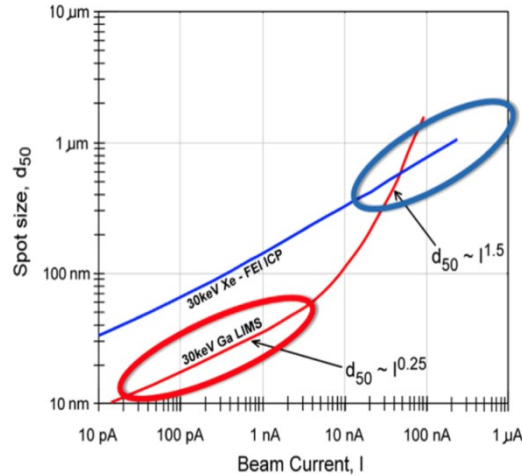
Micropillar compression (and analysis)

Focused ion beam (FIB) milling



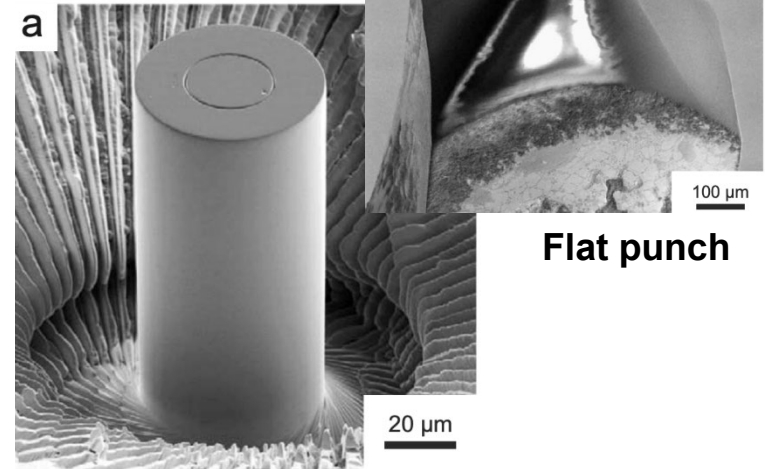
Utke et al. *J. Vac. Sci. Technol. B* (2008)

Different types of FIB



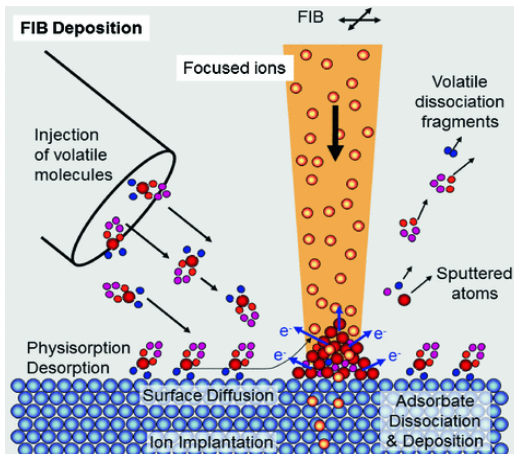
Smith et al. *J. Vac. Sci. Technol. B* (2006)

Compression sample



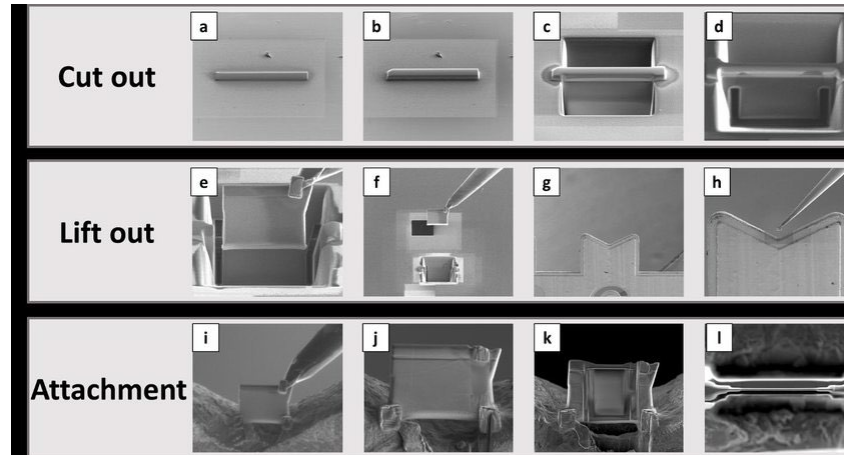
Uchic et al. *Science* (2004)

FIB induced deposition



Shorubalko et al. *Helium Ion Microscopy*, Springer (2016)

Transmission electron microscopy (TEM) lamella preparation



Abed, MSc thesis, University of Toronto (2007)

Micropillar

Compression

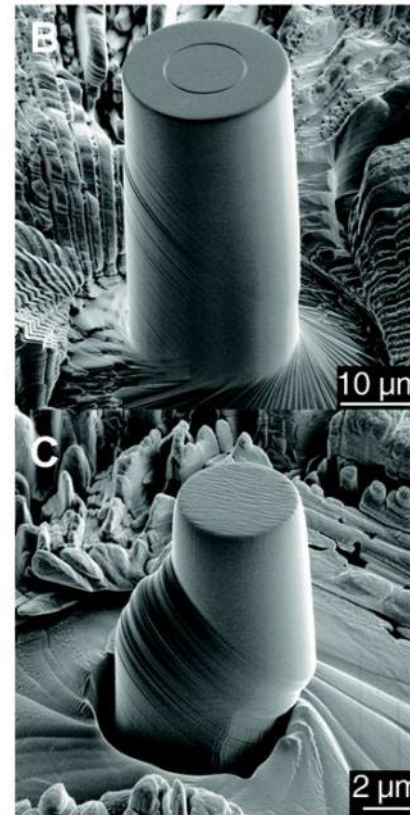
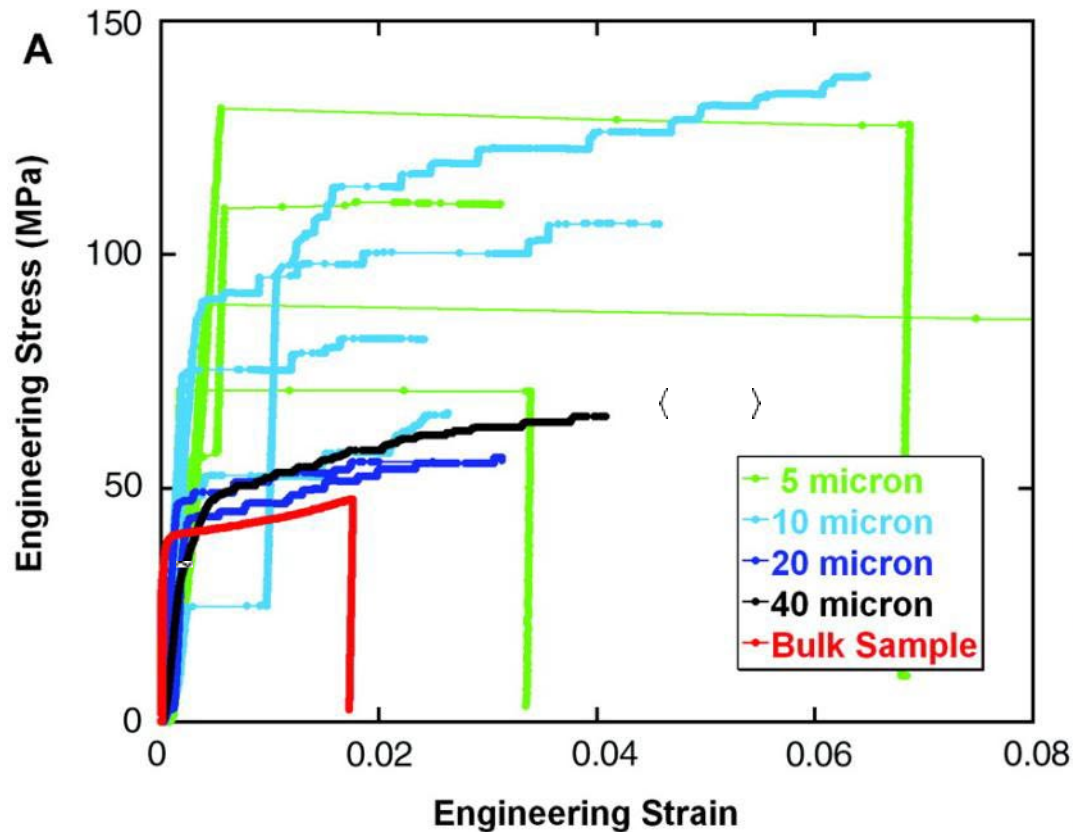
Straightforward
Post-yield behaviour

TEM

Microstructure
Deformation mechanisms

**Powerful
combination for
micro-nanoscale**

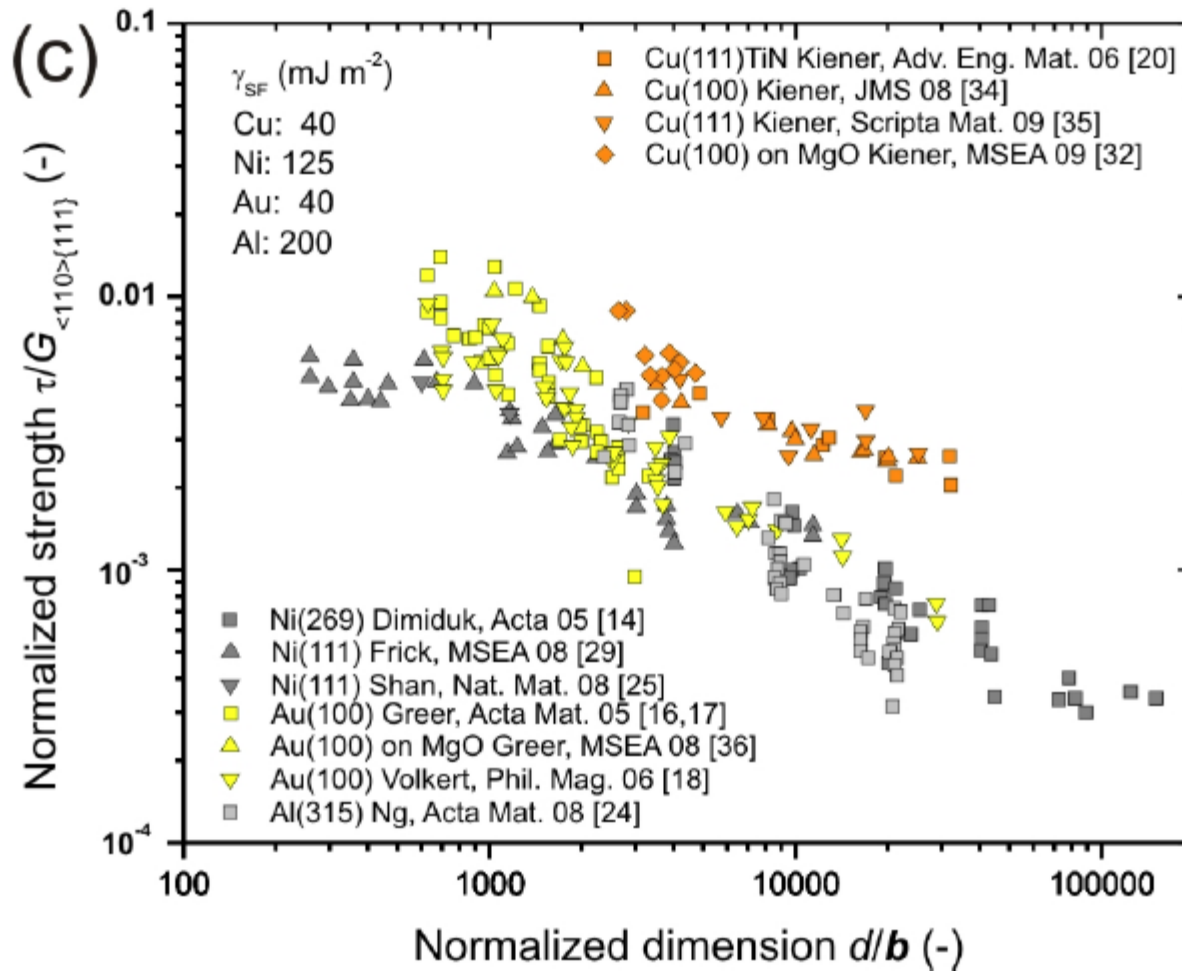
Micro compression tests



MD Uchic, DM Dimiduk, JN Florando, WD Nix (2004) Sample dimensions influence strength and crystal plasticity, *SCIENCE* 305, 986-989

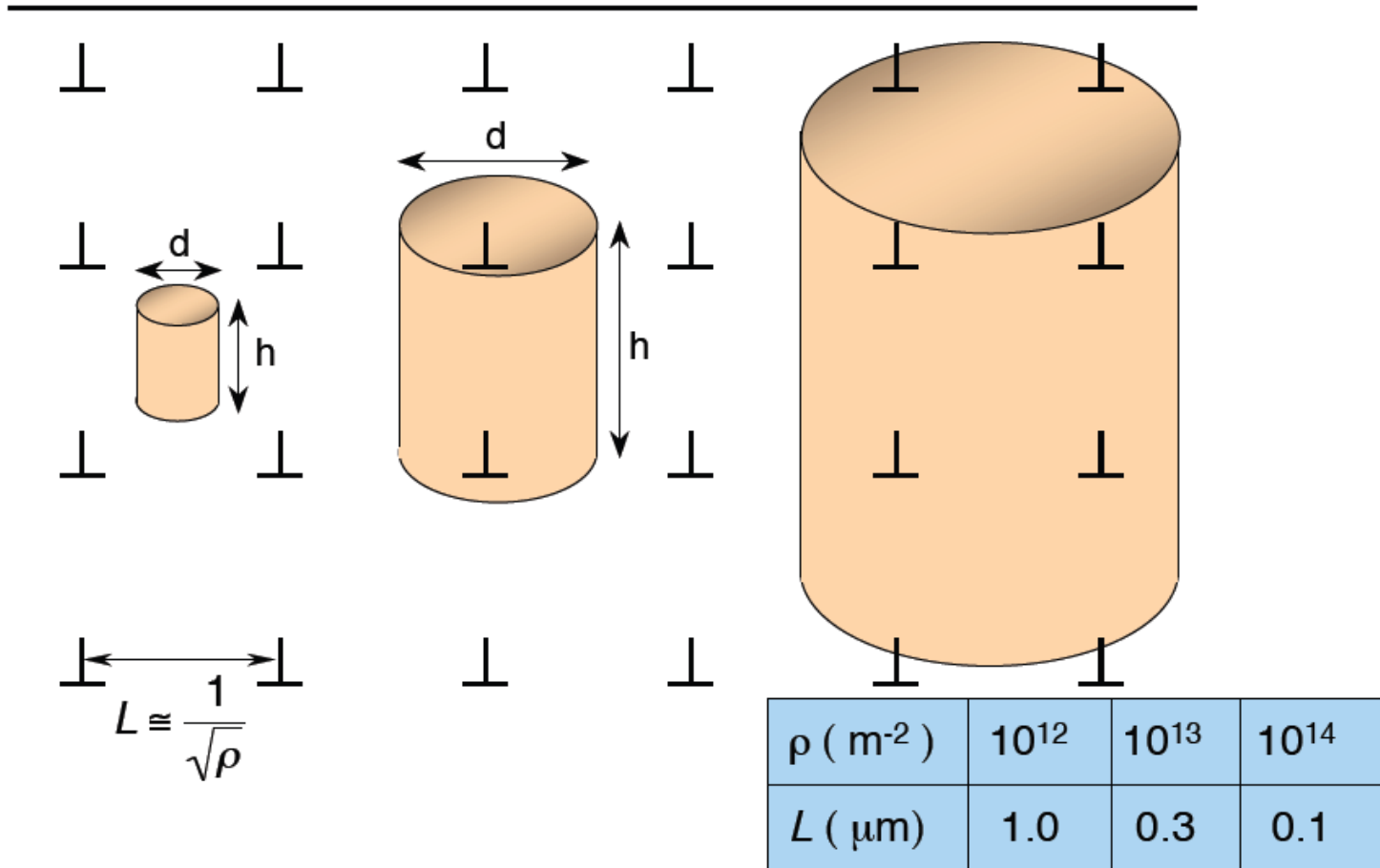
Smaller is stronger

Smaller is stronger



Kiener D, Motz C, Dehm G, Pippan R. *Int. J. Mat. Res.* 2009;100.

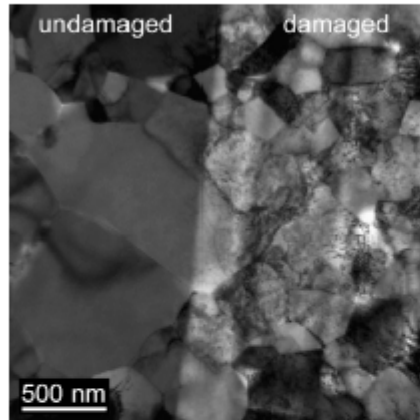
Micro compression tests



Extrinsic influences of FIB milling on pillar strength

Kiener et al, *Mater. Sci. Eng. A* 459, 262 (2007)

TEM of polycrystalline Cu



Important parameters

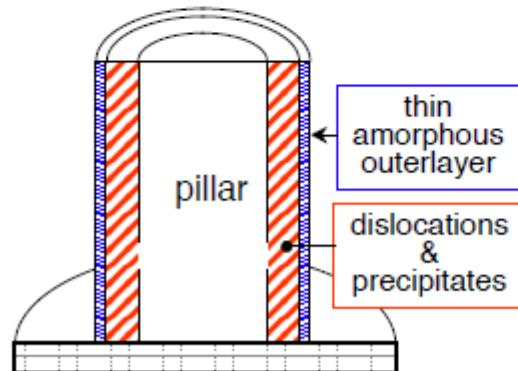
- beam incidence (normal/grazing)
- accelerating voltage (5–30 kV)
- current (10–2000 pA)

Strengthening mechanisms

- hard amorphous film formation
- dislocations
- precipitation

Conclusions

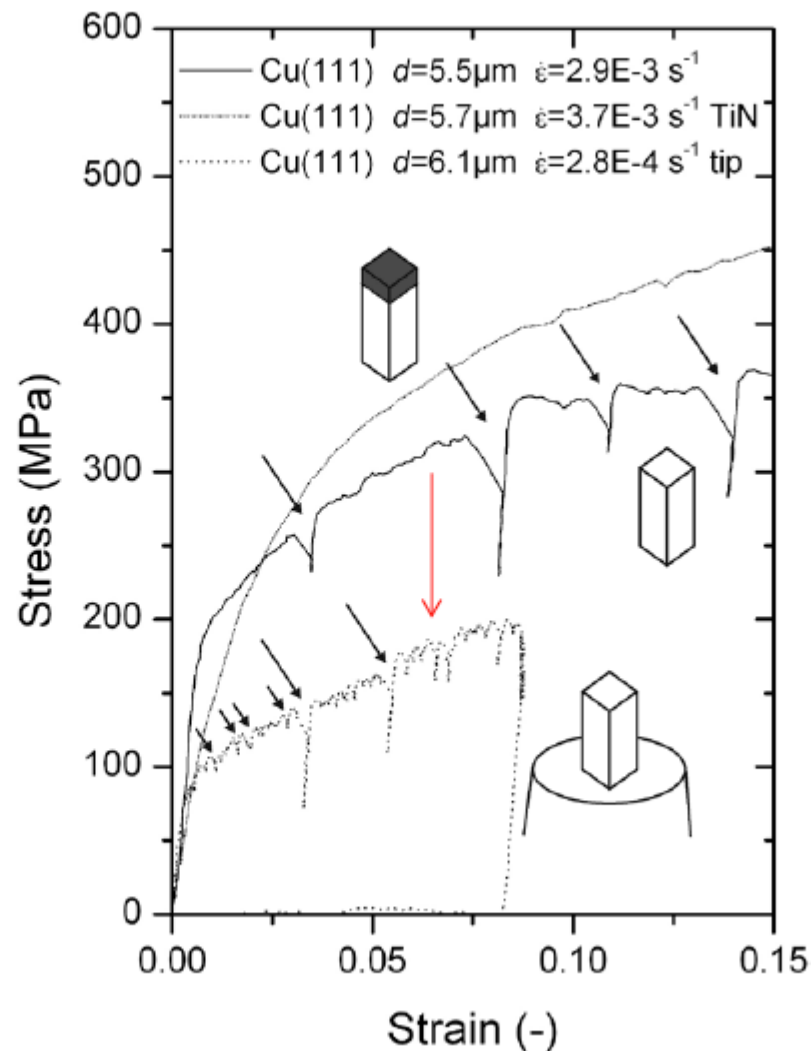
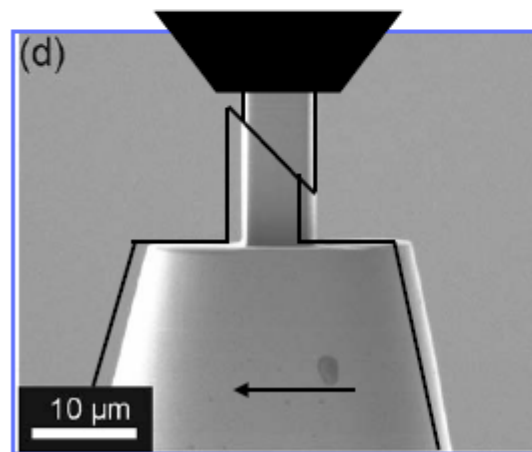
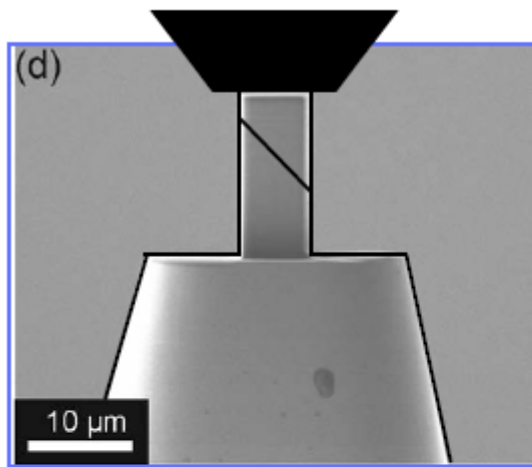
- FIB damage reduced at smaller accelerating voltages & grazing incidence
- even for grazing incidence, effects on strength may be significant for submicron pillars



20

Influence of lateral stiffness

Micro-compression of a **Cu(100)** sample with reduced lateral stiffness



Kiener D, Motz C, Dehm G. Mater. Sci. Eng. A 2009;505:79.

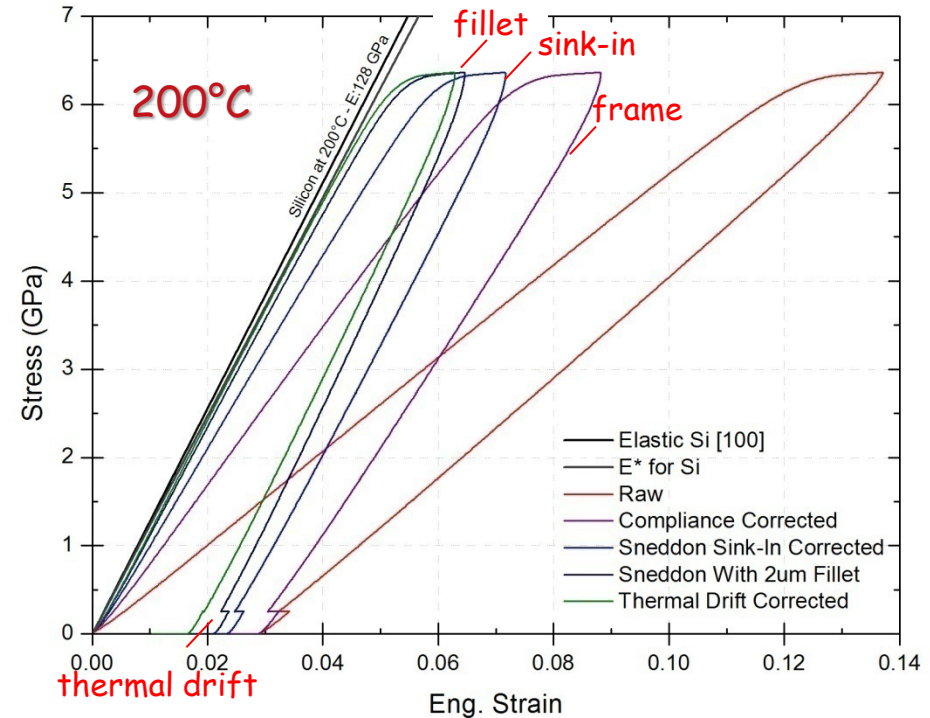
the pain with the strain (-measurement)

Comparison of Modulus values from 200°C,
6.8μm diameter pillar with various corrections:

- Indenter frame compliance
- Pillar sink-in into substrate
- Correction for fillet on pillar base
- Correction for contact thermal drift
- (60 second, constant-load hold period at 1/20th of maximum load during unloading)
- Effective modulus correction using high temperature indenter modulus

$$E^* = \left(\frac{1-\nu_i^2}{E_i} + \frac{1-\nu_s^2}{E_s} \right)^{-1}$$

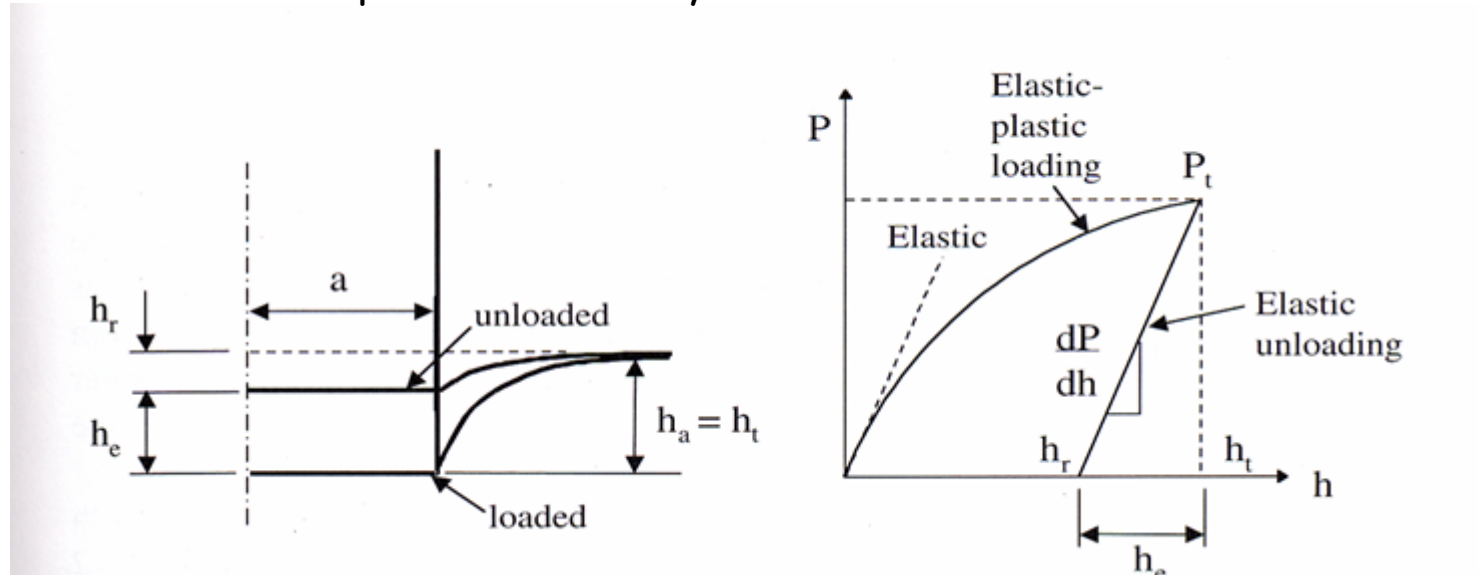
Values converge towards ideal single crystal [100] value. Micro-pillars are not an ideal geometry for Young's modulus measurements, but it is possible.



Alternative: Digital image correlation

"Flat Punch"-Model

Elastic-plastic contact cylinder on flat



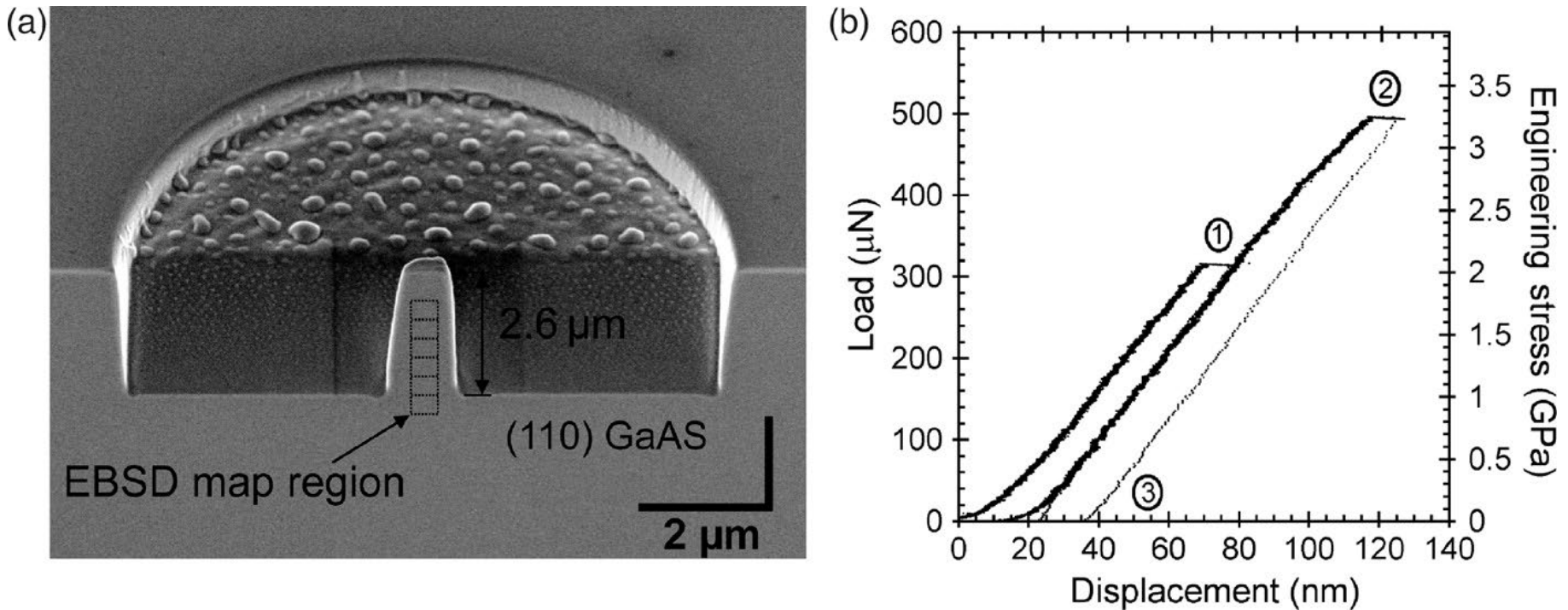
In elastic case:

$$\frac{dP}{dh} = 2aE_r = 2E_r \frac{\sqrt{A}}{\pi}$$

i.e. Young's modulus can be derived from slope of unloading curve:

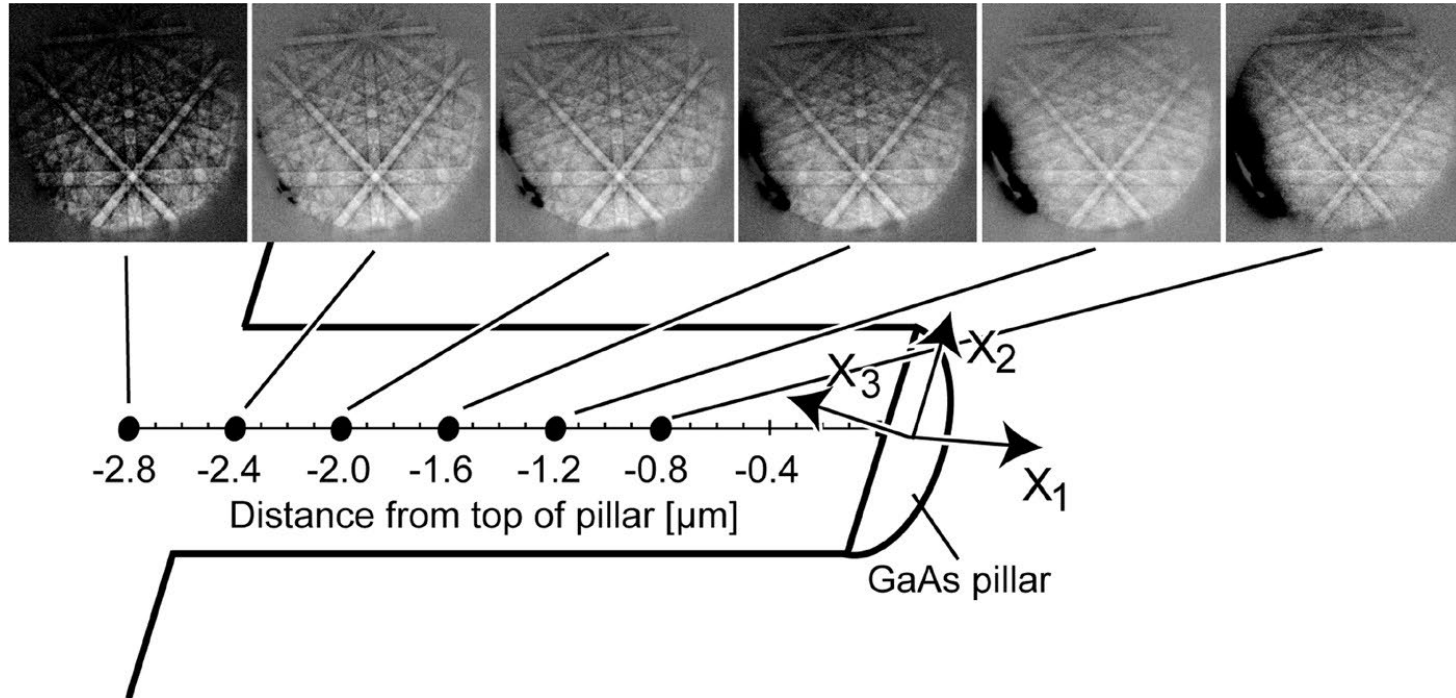
$$E_r = \frac{dP}{dh} \frac{\pi}{2\sqrt{A}}$$

Uniaxial strains in micropillar compression



(a) SEM image of the GaAs micropillar taken at a 45° tilt. The front side of the pillar where the EBSD measurements were acquired is a (110) cleaved surface. (b) GaAs pillar compression data where numbers 1-3 indicate the points at which EBSD mapping occurred. Load-displacement data were converted to engineering stress data. Engineering stress was calculated by dividing the load by pillar cross-sectional area.

Uniaxial strains in micropillar compression



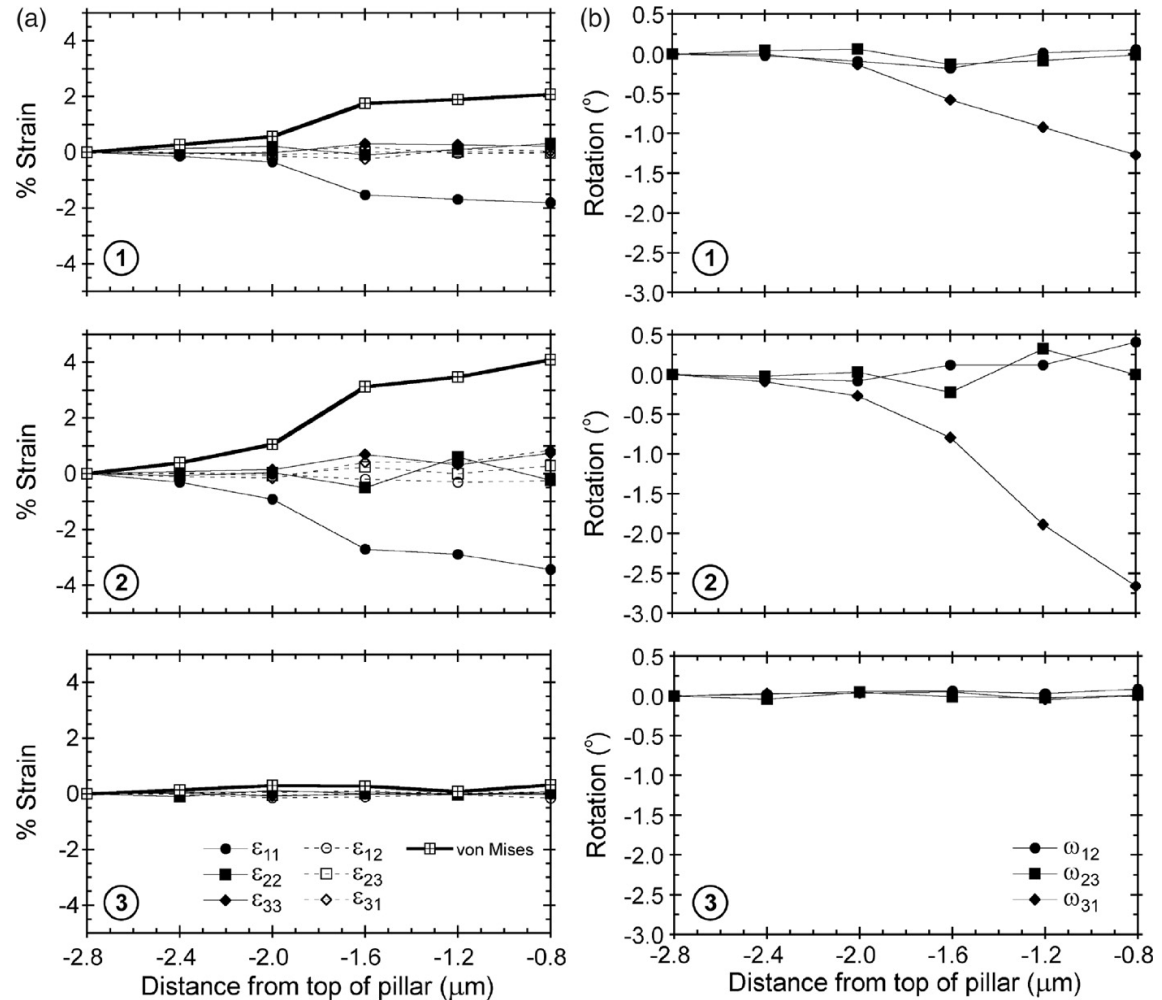
Schematic of the GaAs micropillar with the placement of the EBSD measurements used in and . The EBSD cross-correlation technique measures the bands shift between the patterns of the respective EBSD patterns (distances from -0.8 to $-2.4 \mu\text{m}$ from the top of the pillar) compared to the reference pattern below the base of the pillar at $-2.8 \mu\text{m}$ from the top of the pillar.

Uniaxial strains in micropillar compression

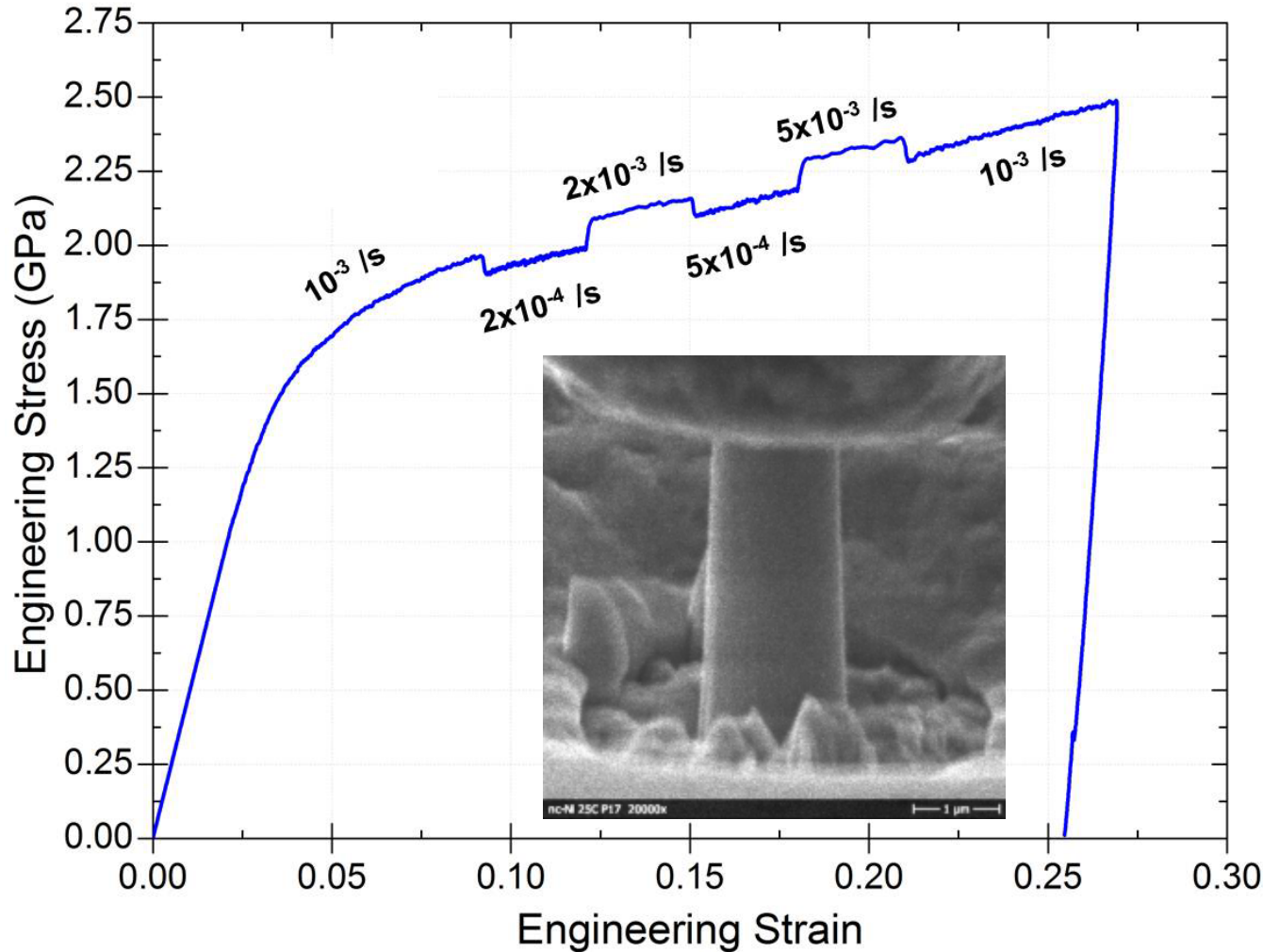
Stresses calculated with the EBSD cross-correlation compare well with the engineering stress values from the nanoindenter load-displacement data.

Elastic strain is found to be mostly homogeneous, but strain inhomogeneities are found at the pillar base due to its non-symmetrical geometry resulting from FIB fabrication.

Lack of shear stress indicates that despite imperfect geometry, misalignments and/or bending, the assumption of uniaxial stress can be considered appropriate to within 10%.



Strain rate jump tests



the strain rate
sensitivity exponent

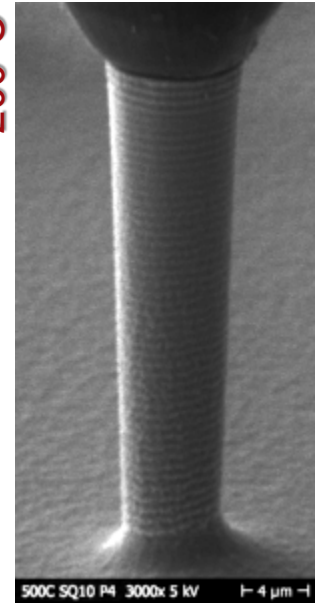
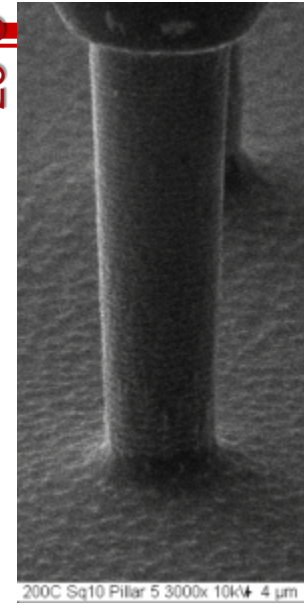
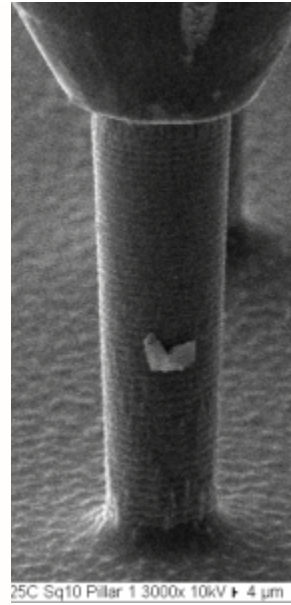
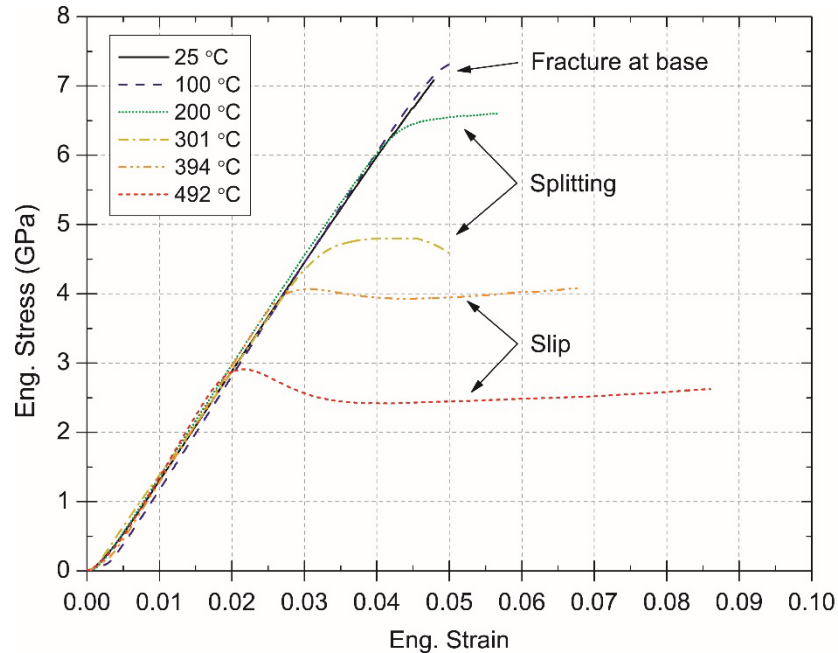
$$m = \frac{d(\ln \sigma_f)}{d(\ln \dot{\epsilon})}$$

the activation volume

$$V = \frac{\sqrt{3}k_B T}{m \cdot \sigma_f}$$

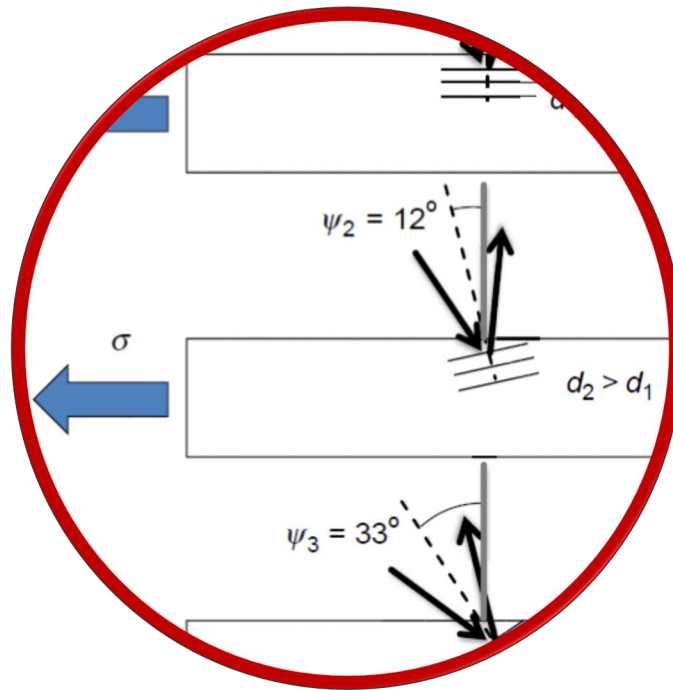
- Strain rate jump tests are straightforward to perform using a displacement control.
- Strain rate sensitivity values are obtained by linear extrapolation to the elastic limit.

Example: microcompression of silicon <001>



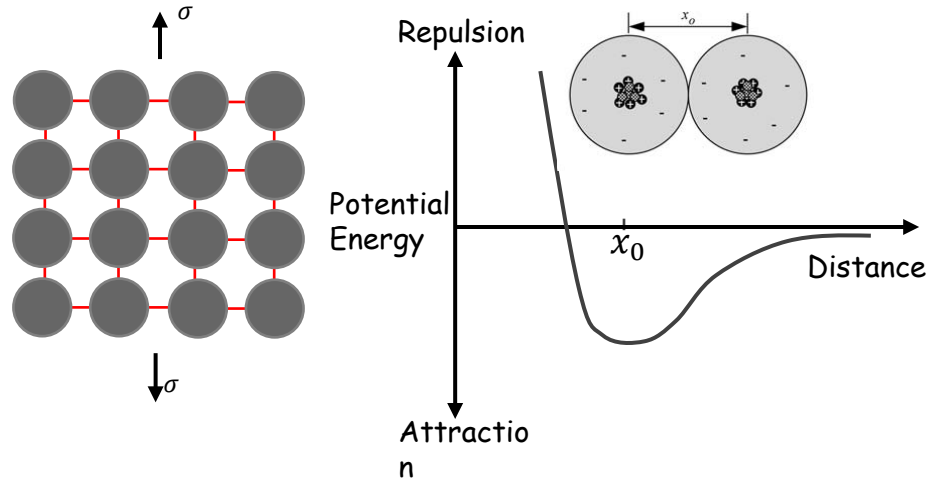
- Transitions between in deformation mechanisms between fracture, splitting, and plasticity directly observed.
- At a diameter of 6.7 μm , the brittle-ductile transition is seen at $\sim 400^\circ\text{C}$ - significantly lower than bulk silicon.
- Classic upper yield point consistent with dislocation nucleation observed during plastic deformation due to system's intrinsic displacement control.

Microscale fracture testing



Why fracture mechanics?

Ideal

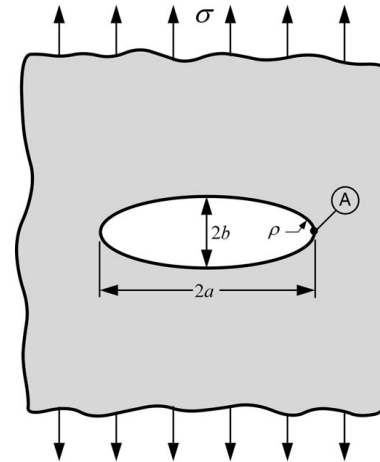


- Perfect material
- σ equal to cohesive stress σ_c to break bonds

Theoretical fracture stress:

$$\sigma_f = \sigma_c \approx \frac{E}{\pi}$$

Real



Sources of flaws:

Casting;
Particle decohesion;
Plastic deformation
...

- Material with flaw
- Stress concentration at flaws
- σ much smaller than σ_c

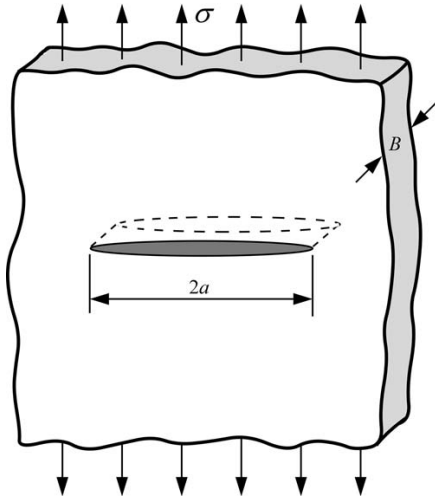
Fracture stress with a flaw:

$$\sigma_f \approx \sigma_c \cdot \sqrt{\frac{x_0}{4a}} \quad \begin{matrix} \sim 10^{-10}m \\ \sim 10^{-6}m \end{matrix} \quad \sim 0.01\sigma_c$$

Study the material resistance to preexisting crack propagation

Two ways to understand fracture toughness

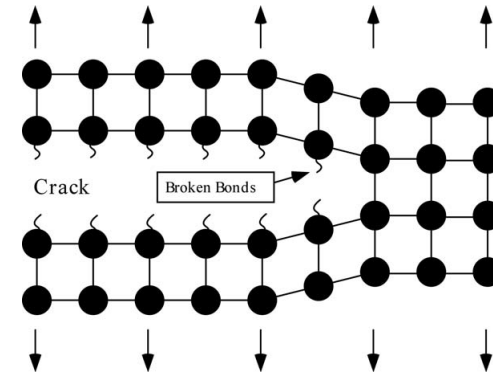
➤ Energy balance: G



Crack can only grow when this process causes the total energy to decrease or remain constant.

Unit: J/m^2 $-\frac{d\Pi}{dA} \geq 2\gamma_s$

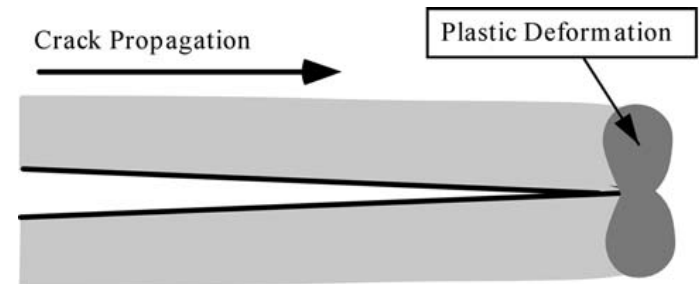
$-\frac{d\Pi}{dA} = G$ energy release rate;



Griffith's theory

*Ideal elastic brittle material

$$G_c = 2\gamma_s$$



Irwin and Orowan theory

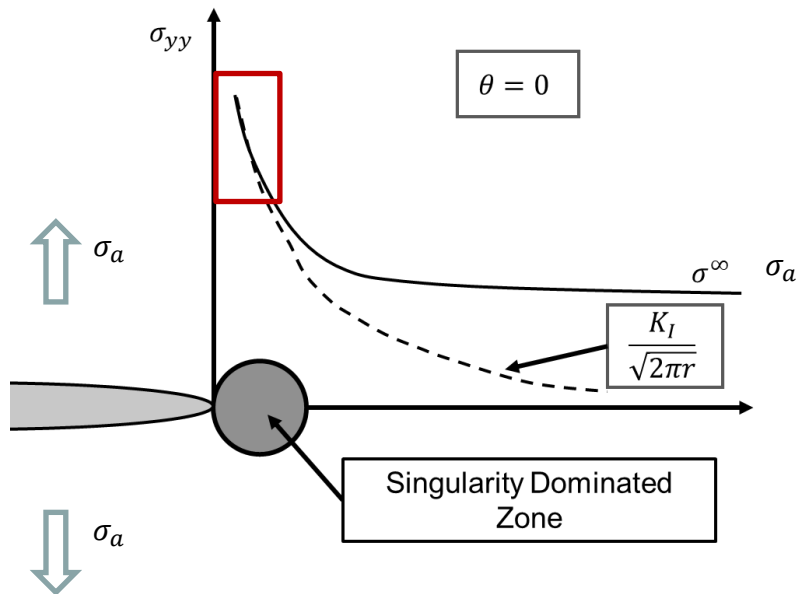
*Metals with plastic zone

$$G_c = 2(\gamma_s + \gamma_p)$$

Two ways to understand fracture toughness

➤ Stress distribution: K & J

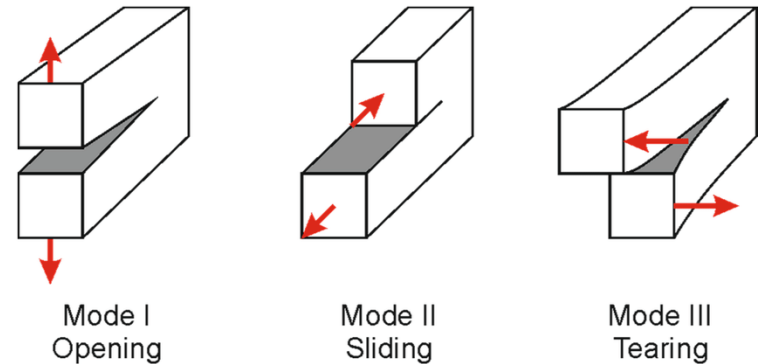
Linear elastic material:



$$\sigma_{ij}(r, \theta) = \frac{K_{I,II,III}}{\sqrt{2\pi r}} f_{ij}(\theta)$$

$K_{I,II,III}$: stress intensity factor

Unit: $\text{MPa} \cdot \text{m}^{0.5}$

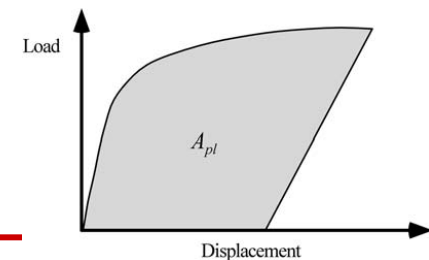


Elastic Plastic Material:

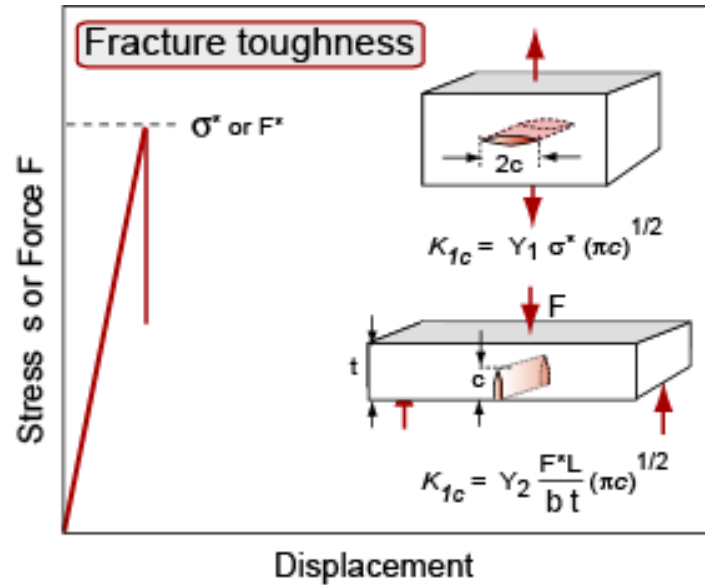
$$\sigma_{ij}(r, \theta) = c \left(\frac{J}{r} \right)^{\frac{1}{1+n}} s_{ij}(\theta)$$

$$J_{pl} = \frac{\eta A_{pl}}{B b_0}$$

Unit: J/m^2



Two ways to understand fracture toughness



Definition:

Fracture toughness

$$K_{Ic} = Y_1 \sigma^* \sqrt{\pi c}$$

Plane stress: $E' = E;$

Fracture energy or toughness

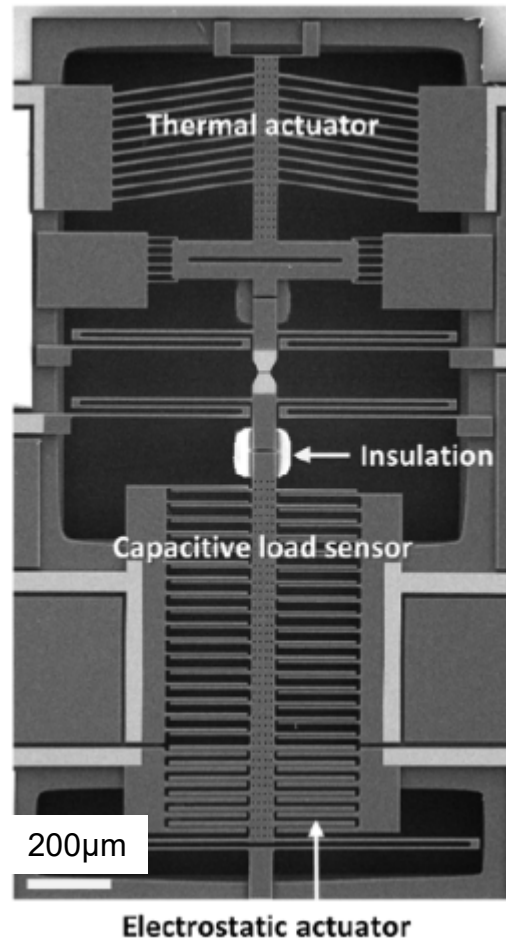
$$G = \frac{K_c^2}{E}$$

Plane strain: $E' = \frac{E}{1 - \nu^2}$

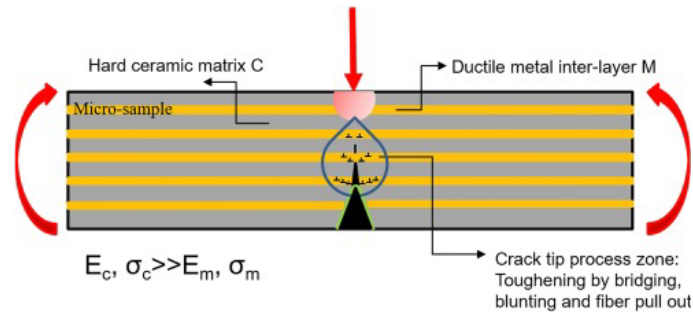
Range of fracture toughness:

0.3 MPa m^{1/2} (glass) to 200 MPa m^{1/2} GPa (steel)

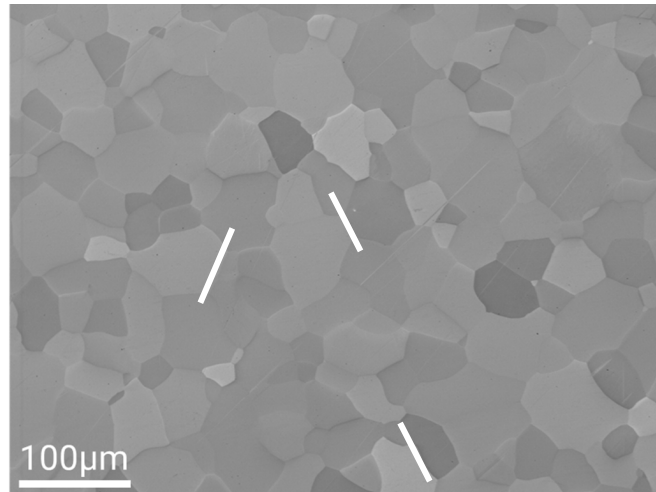
Why microscale fracture testing?



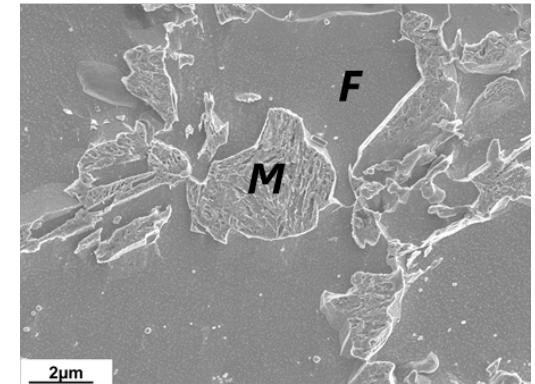
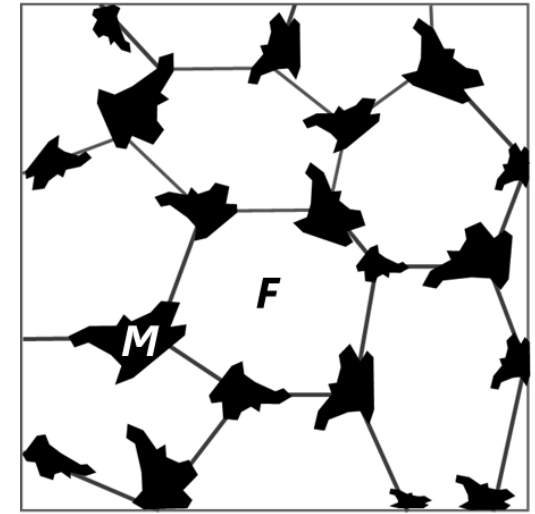
MEMS actuator/sensor



Thin film coating



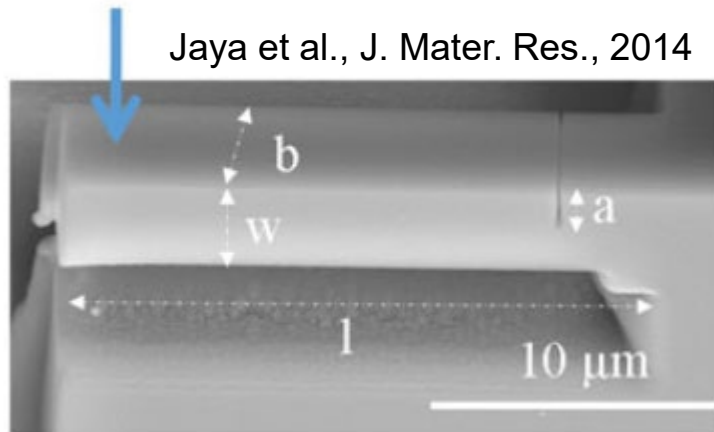
Interface/boundary brittleness



Damage sensitive phase

Testing methods

➤ Single cantilever bending

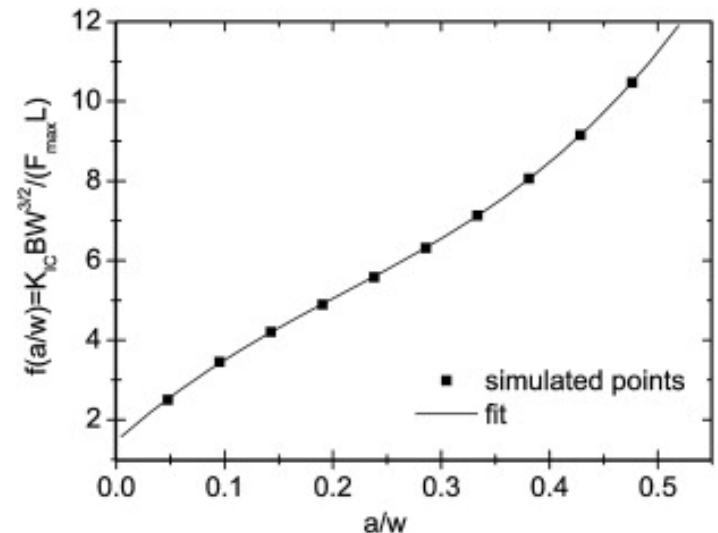
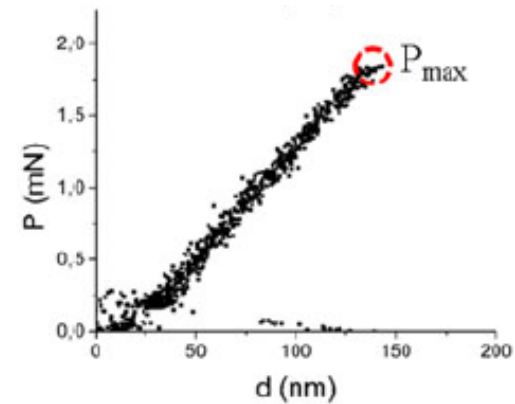


Linear Elastic Fracture Mechanics (LEFM)

$$K_I = \frac{P_{max} l}{b w^{3/2}} f\left(\frac{a}{W}\right)$$

$$f\left(\frac{a}{W}\right) = 1.46 + 24.36 \left(\frac{a}{W}\right) - 47.21 \left(\frac{a}{W}\right)^2 + 75.18 \left(\frac{a}{W}\right)^3$$

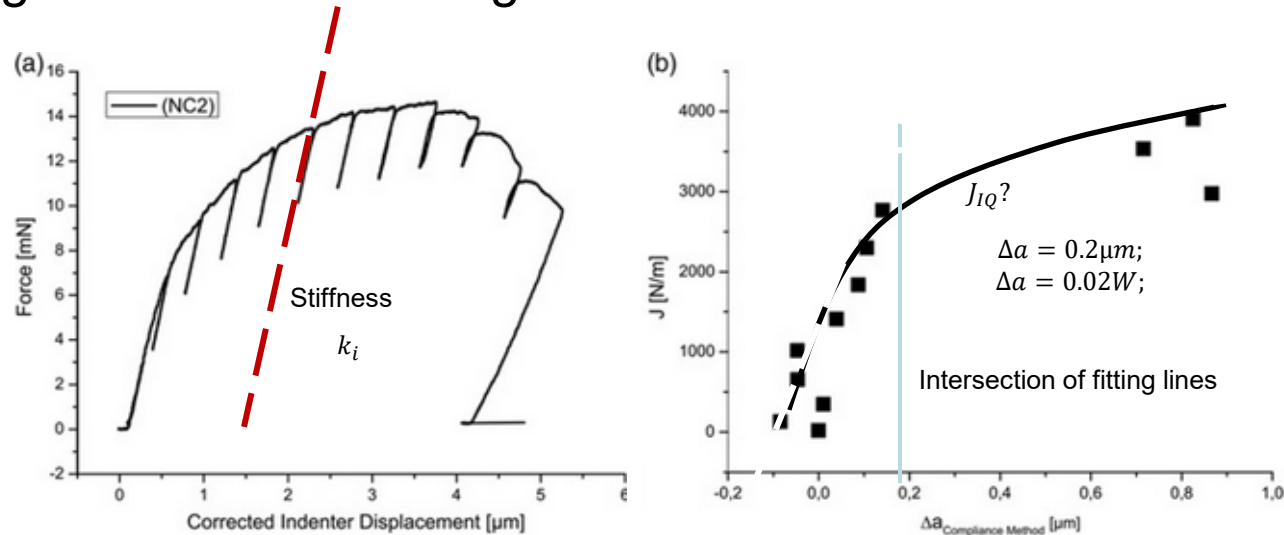
↙ Geometry factor function



Matoy et al., Thin Solid Film, 2009

Testing methods

➤ Single cantilever bending

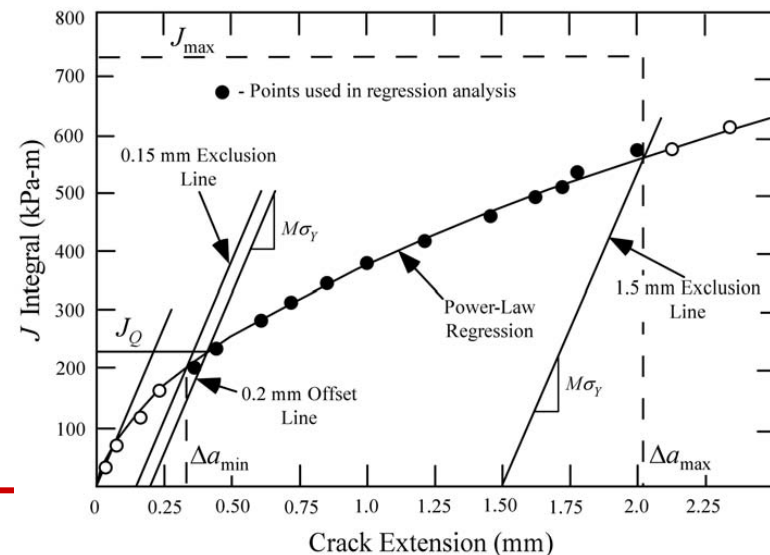


Wurster et al., Philosophical Magazine, 2012

$$J = J_{el(i)} + J_{pl(i)}$$

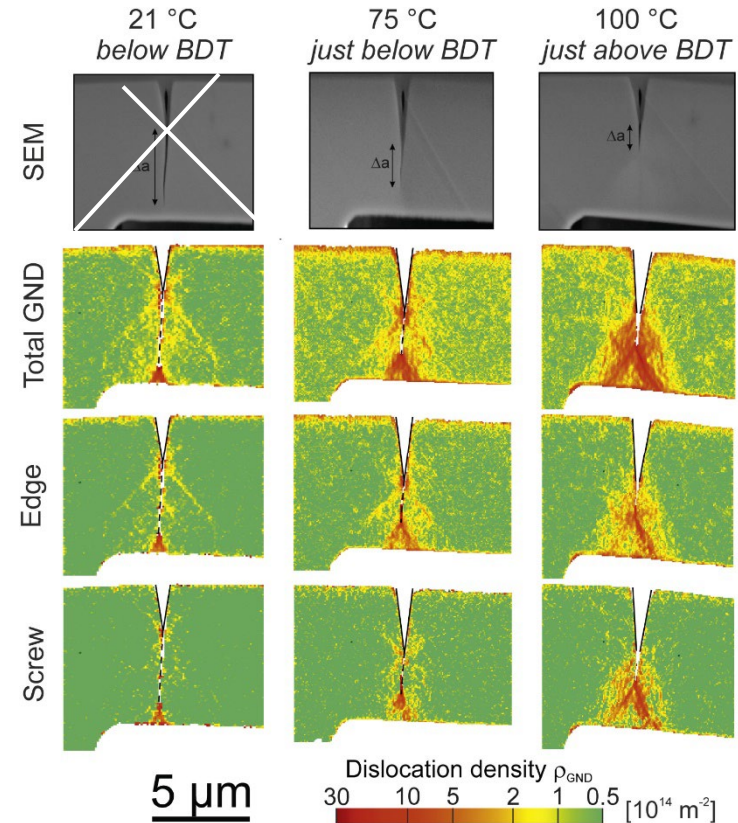
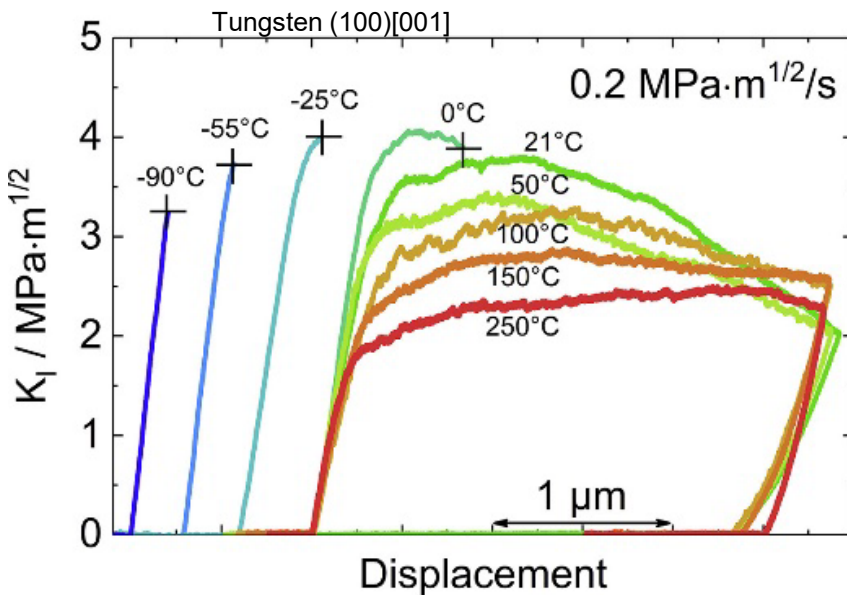
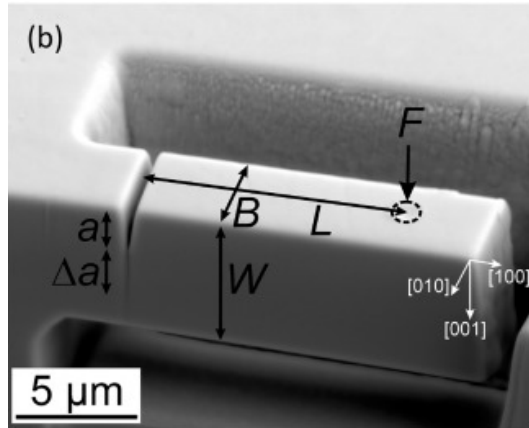
$$J_i = \frac{(K_{(i)})^2 (1 - \nu^2)}{E} + \frac{\eta A_{Pl(i)}}{B(W - a_0)}$$

$$a_i = W - \sqrt[3]{\frac{4k_i L^3}{BE}}$$



Testing methods

➤ Single cantilever bending - Temperature



Edge dislocation caused slip trace at low T;

Testing methods

➤ Single cantilever bending-Size effect

Plastic zone size (Irwin):

Plane stress:
$$r_y = \frac{1}{2\pi} \left(\frac{K_I}{\sigma_{YS}} \right)^2$$

Plane strain:
$$r_y = \frac{1}{6\pi} \left(\frac{K_I}{\sigma_{YS}} \right)^2$$

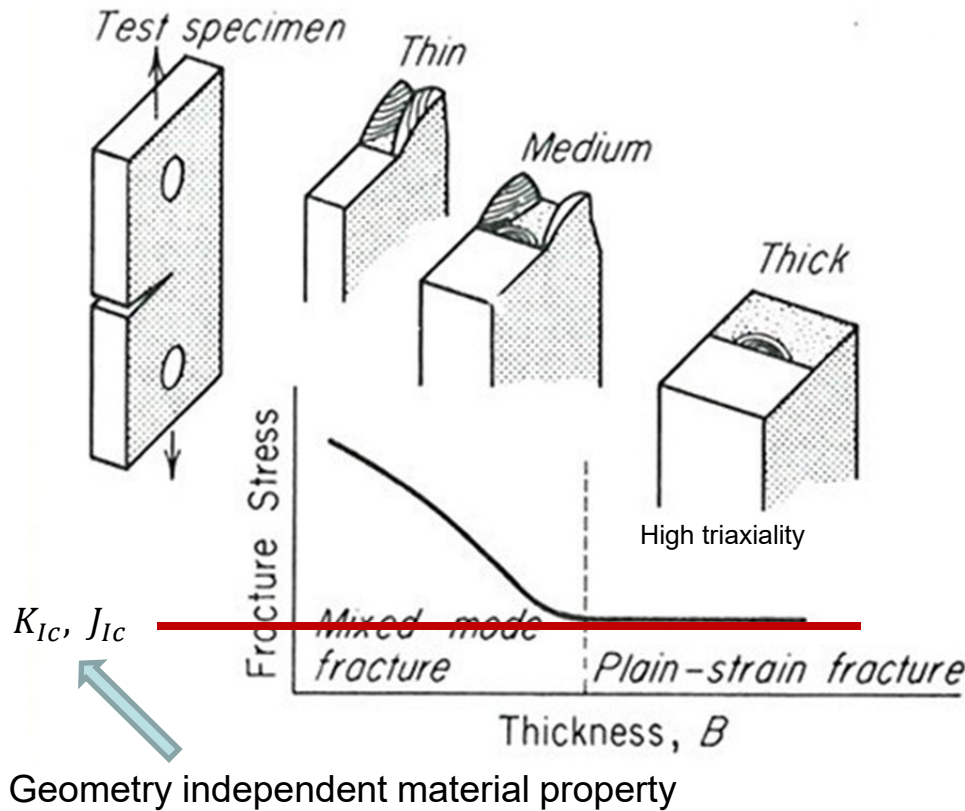
1/3 in size!

ASTM E399 LEFM:

$$B, a \geq 2.5 \left(\frac{K_Q}{\sigma_{YS}} \right)^2 \gg r_y$$

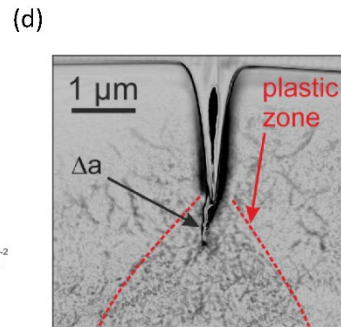
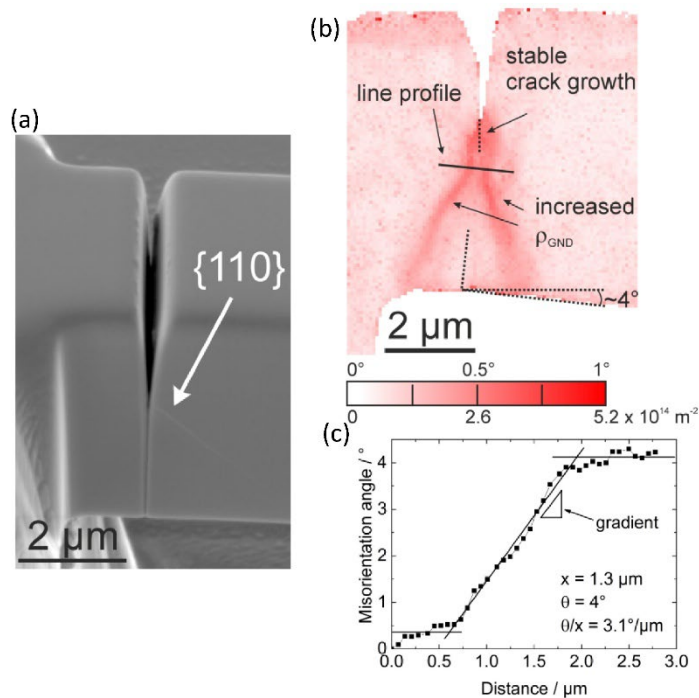
ASTM E1820 EPFM:

$$B, b_0 \geq \frac{25J_Q}{\sigma_Y}$$

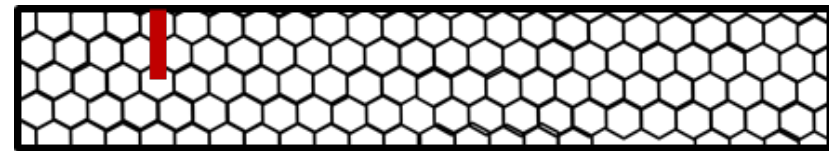
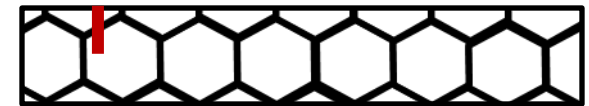


Testing methods

➤ Single cantilever bending-Size effect



2) Characteristic Microstructure

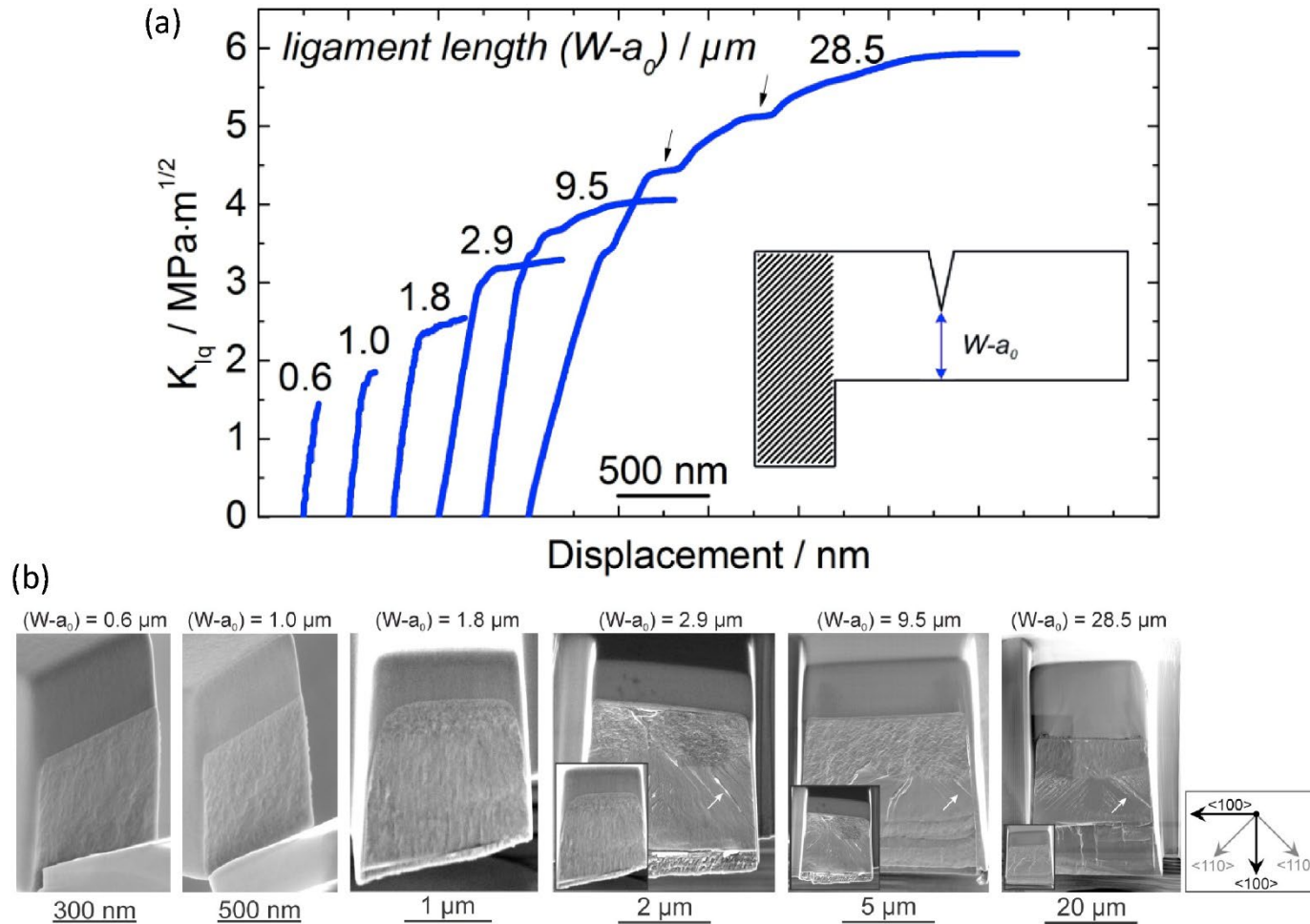


Plastic zone size already comparable to sample dimension!!!

1) Stress state caused dimension dependence

Testing methods

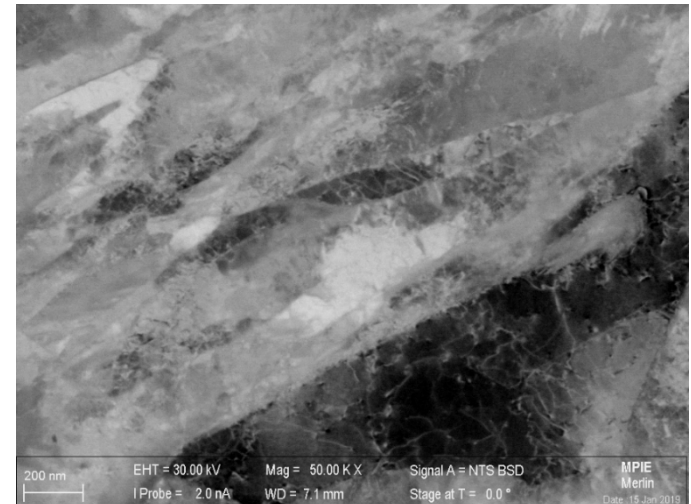
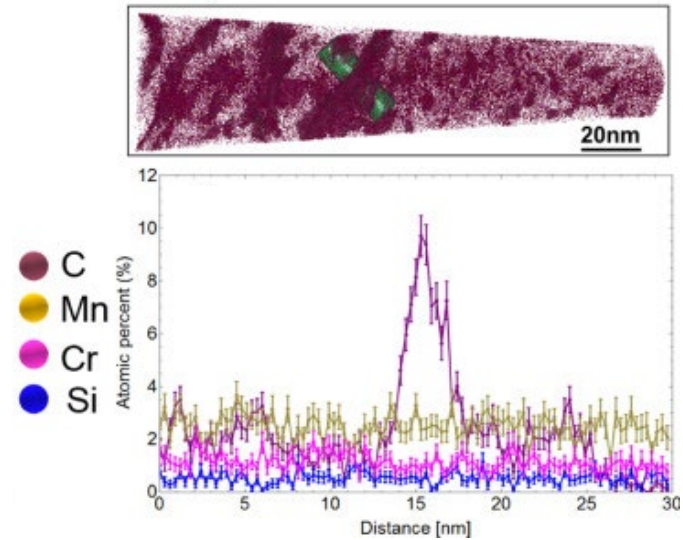
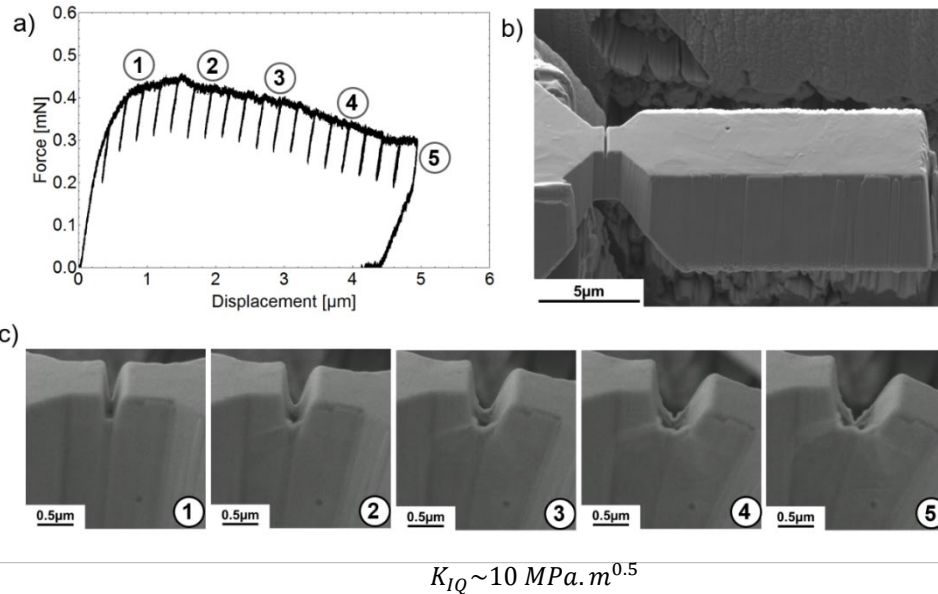
➤ Single cantilever bending-Size effect



3) Dislocation availability caused dimension dependence

Testing methods

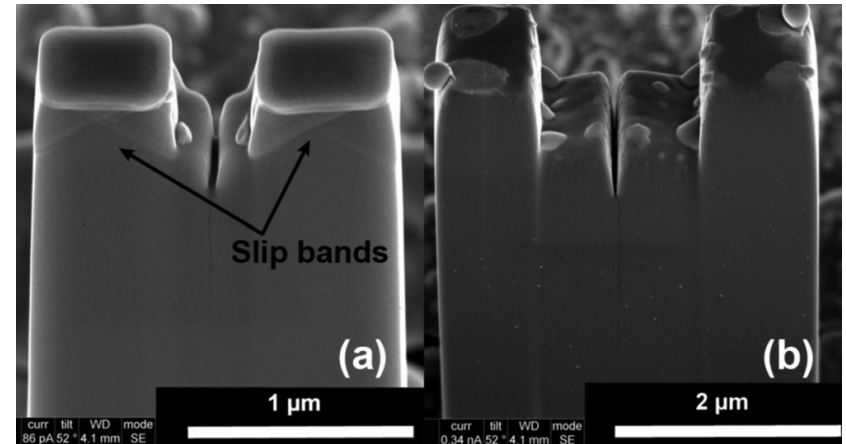
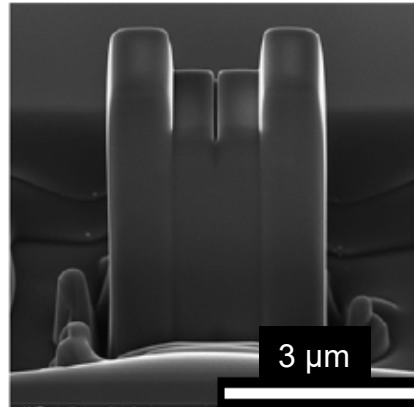
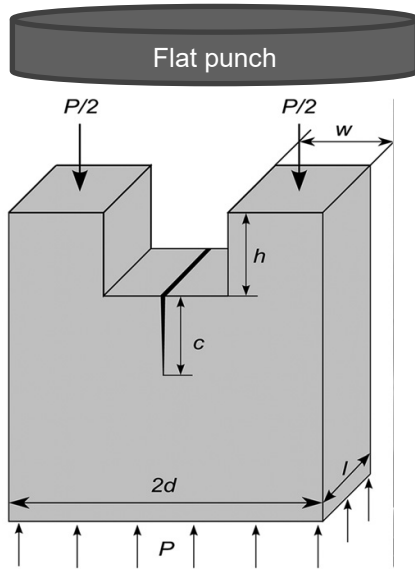
➤ Single cantilever bending-damage sensitive phase



Martensite sensitive to damage initiation;
But not cause catastrophic fracture.

Testing methods

➤ Double cantilever beam



Liu, S., et al. Applied Physics Letters, 2013

$$K_{IC} = \sqrt{3} \frac{(e - \mu h)}{bd^{3/2}} P_c$$

Pros:

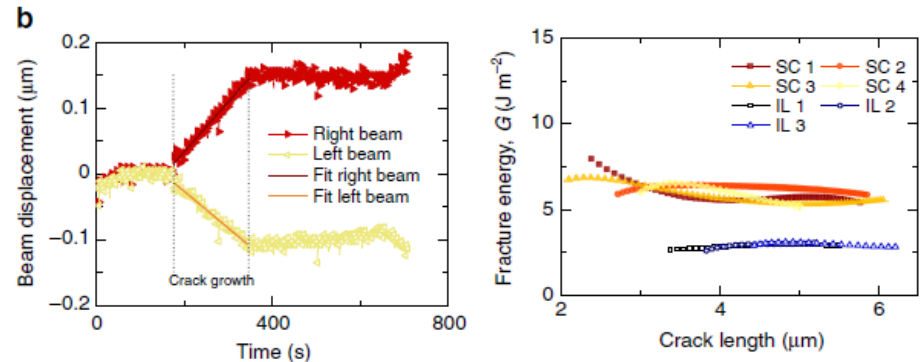
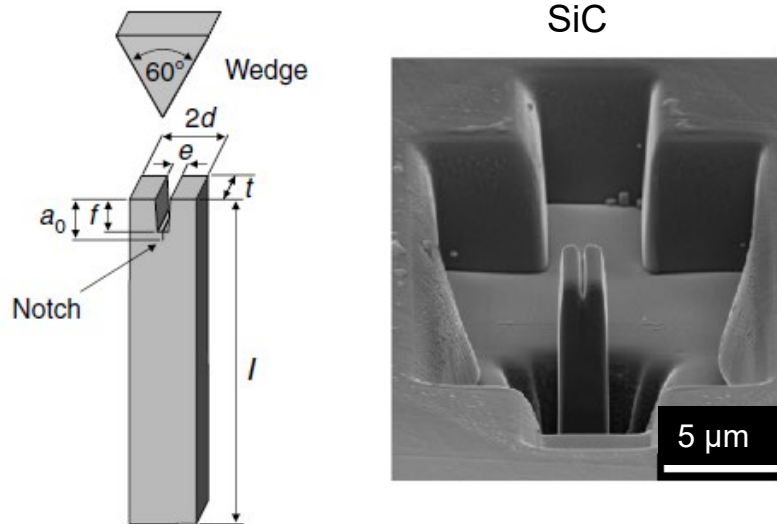
- 1) Less FIB time
- 2) More stable crack propagation
- 3) Free of residual stress

Cons:

- 1) Friction coefficient between tip and test sample required
- 2) Currently only applicable to brittle materials
- 3) Success rate low

Testing methods

➤ Double cantilever beam



$$G = \frac{3Ed^3\delta^2}{8a^4} [1 + (1 + \nu)(d/a)^2]$$

Pros:

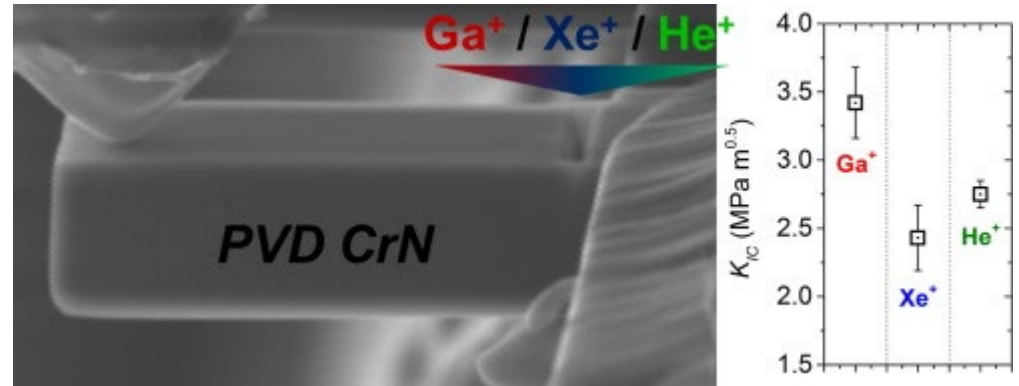
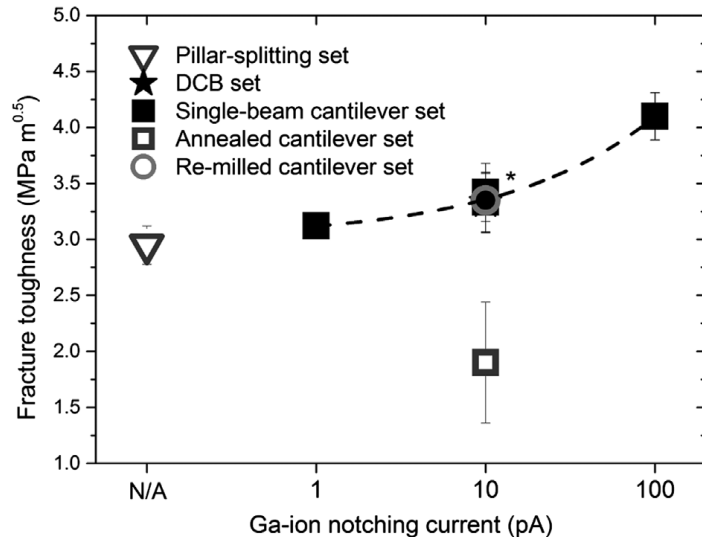
- 1) No need to consider friction
- 2) Observation of stable crack propagation
- 3) Interfacial toughness measurement

Cons:

- 1) Tracking the beam displacement δ
- 2) Alignment issue with wedge

Testing methods

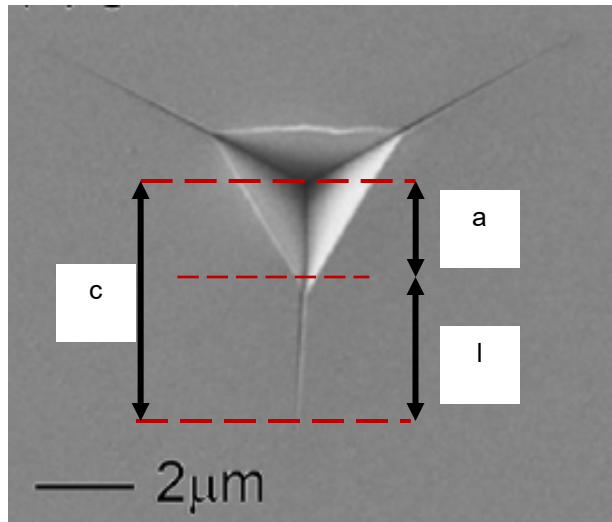
➤ FIB influence



- Apparent fracture toughness depends on
 - Ion species (implantation depth, ion damage cascade)
 - Ion current during notching (damage, notch root radius)

Testing methods

➤ Nanoindentation



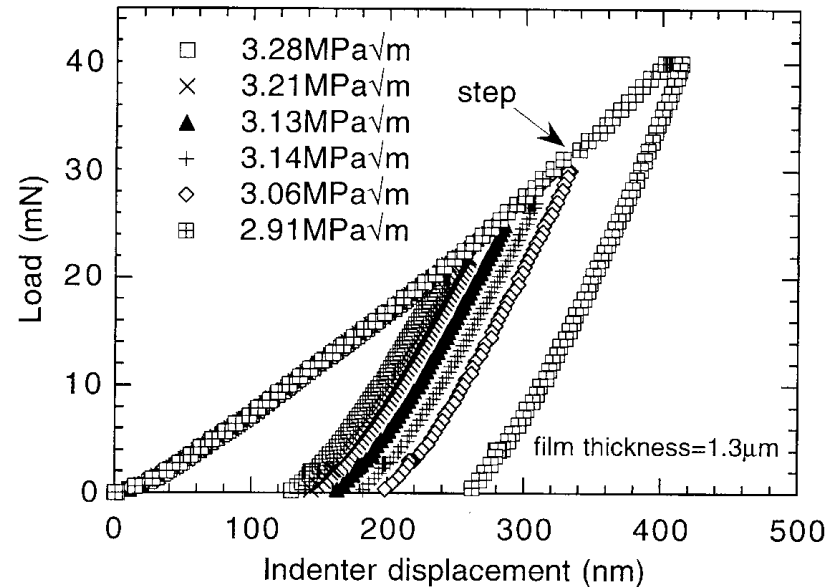
Crack on soda-lime glass

Cuadrado, N., et al. Journal of the European Ceramic Society 2015.

$$K_c = \alpha \cdot \sqrt{\frac{E}{H}} \cdot \frac{P_{max}}{c^{3/2}}$$

Pros:

- 1) No FIB induced damage
- 2) Simplicity



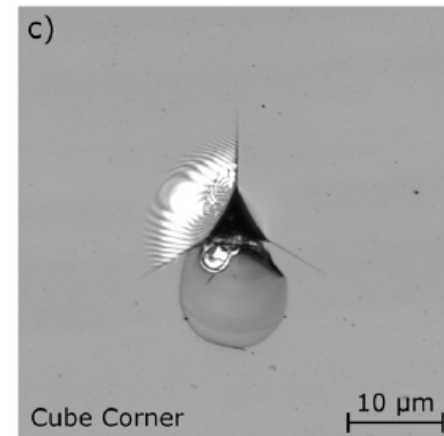
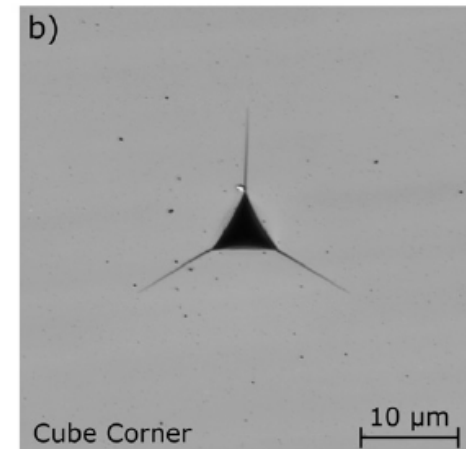
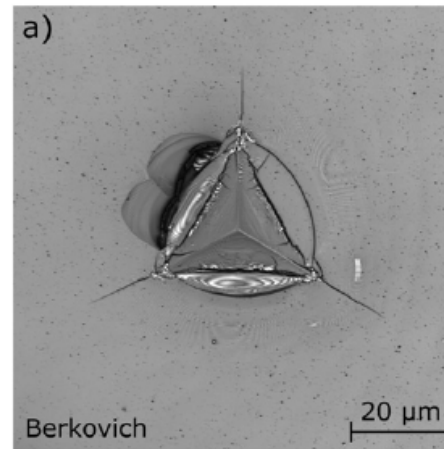
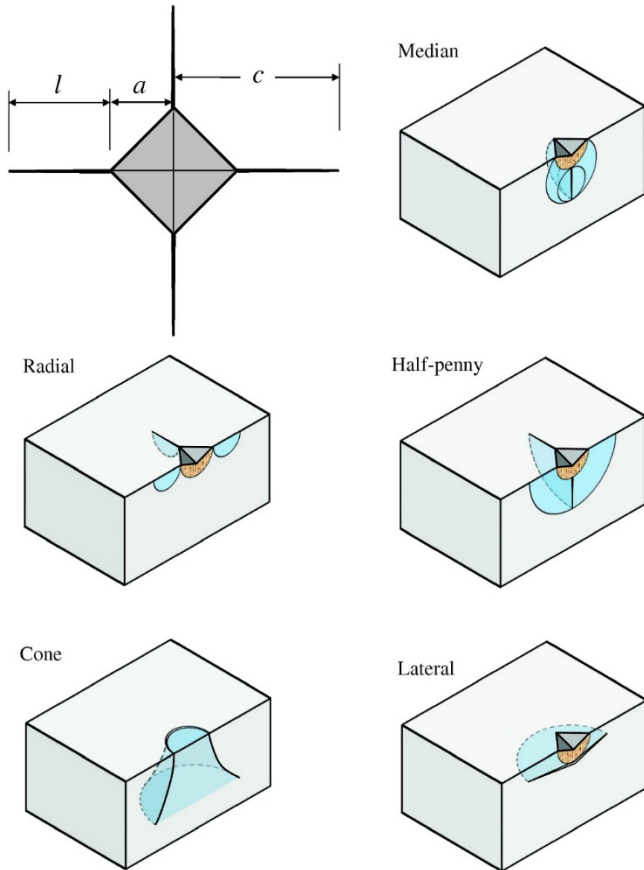
Scharf et al., Journal of Vacuum Science & Technology A: Vacuum, Surfaces, and Films (1997).

Cons:

- 1) Residual stress/substrate effect
- 2) Only applicable to brittle materials
- 3) Need special crack system

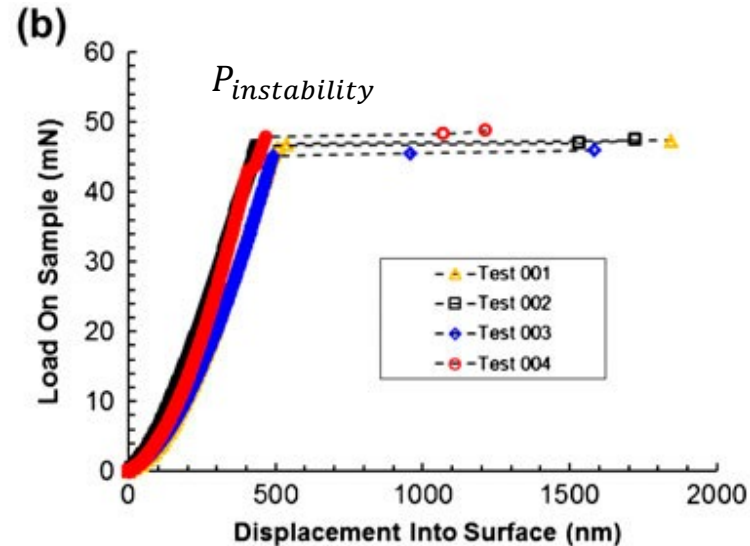
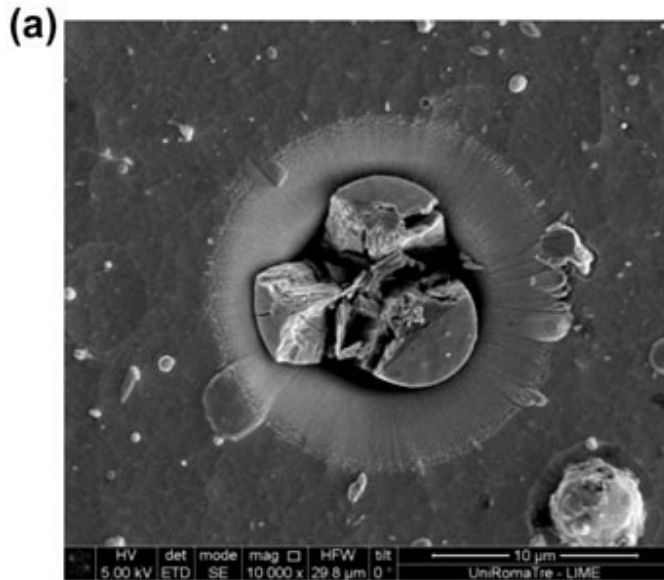
Testing methods

➤ Nanoindentation



Testing methods

➤ Pillar splitting



$$K_c = \gamma \cdot \frac{P_c}{R^{3/2}}$$

$$\text{Aspect ratio: } \frac{z}{D} \geq 1$$

Pros:

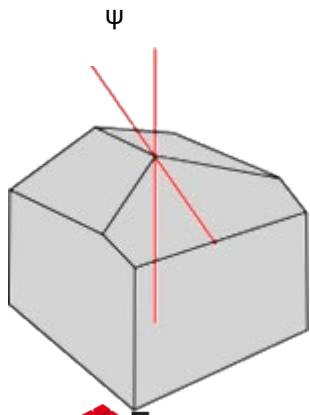
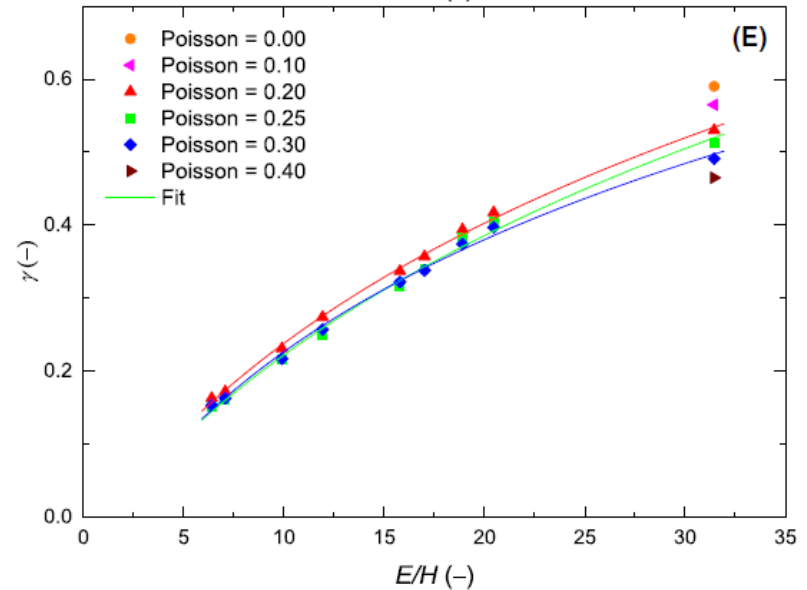
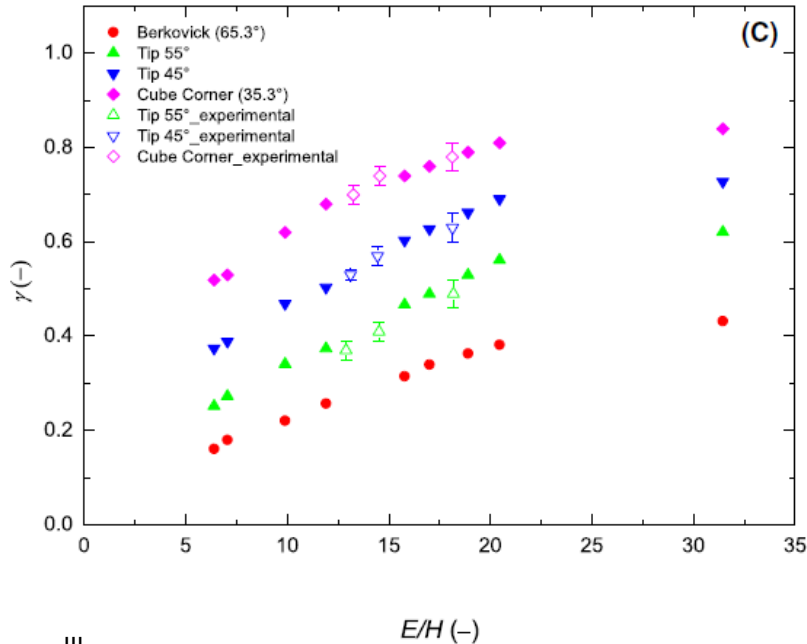
- 1) Less FIB induced damage
- 2) No measurement of crack length
- 3) No substrate/residual stress influence

Cons:

- 1) Alignment when doing indentation
- 2) Cohesive zone FEM for evaluation γ
- 3) Only applicable to brittle materials

Testing methods

➤ Pillar splitting



$\Psi=65.3^\circ$: Berkovich tip

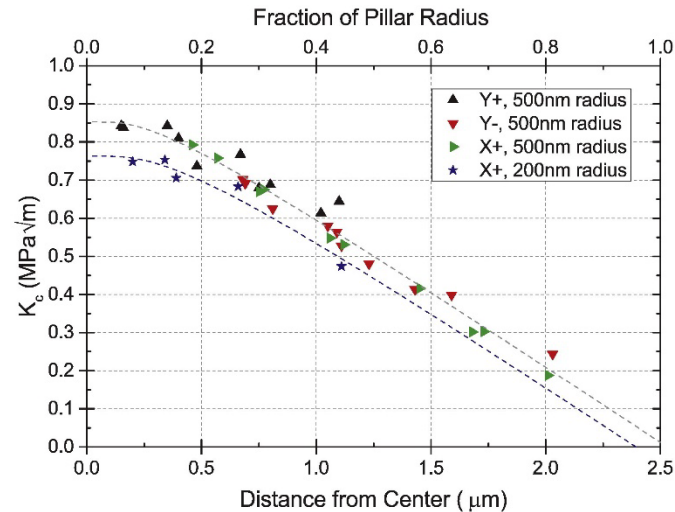
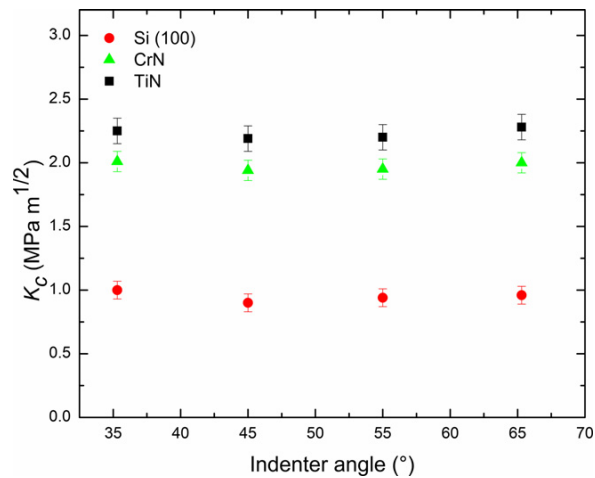
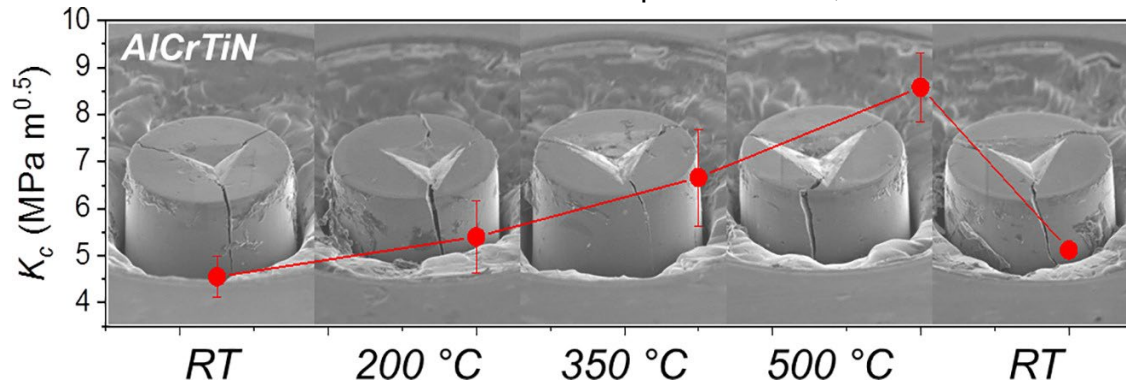
$\Psi=35.3^\circ$: Cube corner tip

- 1) γ increases with the E/H ratio;
- 2) γ decreases with indenter angle;
- 3) Poisson's ratio has little influence on γ .

Testing methods

➤ Pillar splitting

J.P. Best et al. Scripta Materialia, 2019

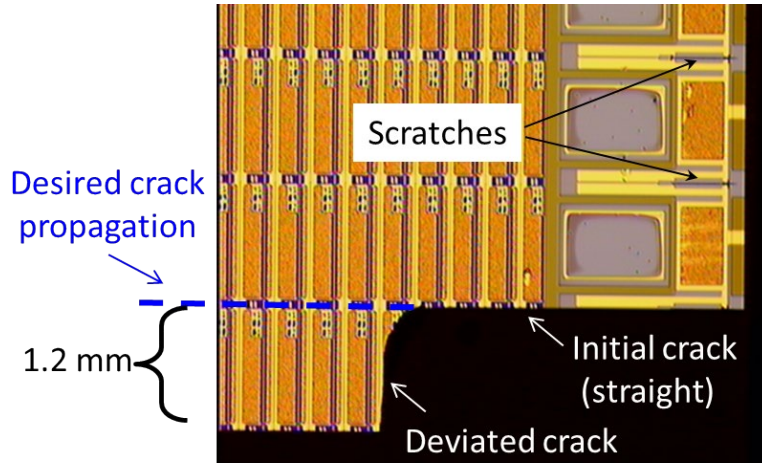


Summary and comparison

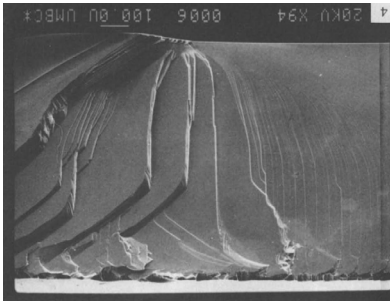
Testing methods	Input parameter	Indenter type	Stable crack growth	Free of residual stress	Interface	Tolerance to misalignment	FIB influence
Single cantilever	$P_{\max}, a$	Wedge/Cone	×	✓	✓	✓	×
Double cantilever	$P_{\max}, \mu$	Flat punch	✓	✓	~	×	×
Naoindentation	$P_{\max}, a$	Berkovich/cube	×	×	×	~	✓
Pillar splitting	$P_{\max}, \gamma$	Berkovich/cube	×	✓	×	×	×

Mode-dependent fracture toughness testing at the micro-scale

Motivation: Cleavage of GaAs

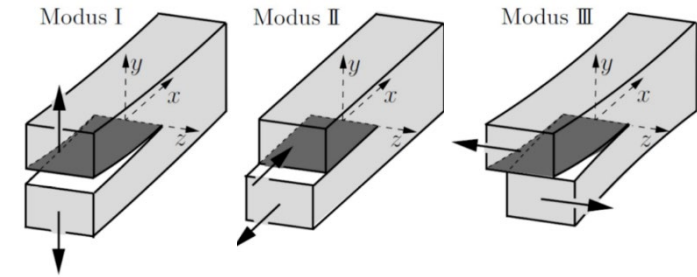


II-VI LASER ENTERPRISE

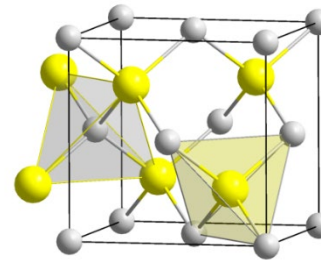


Belsinger et al., Eng. Fract. Mech., 1994
River-lines on GaAs fracture surface

Change from
mode I (start) to
mode III (end)



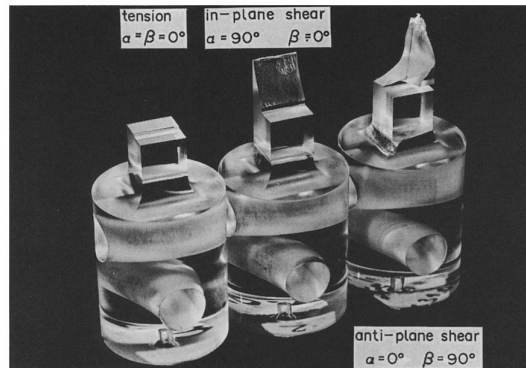
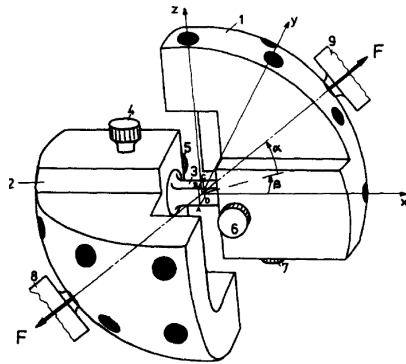
Different fracture modes



GaAs:
"ZnS" sphalerite structure
{110}-cleavage planes
 $K_{Ic} = 0.31 \dots 0.46 \text{ MPam}^{0.5}$

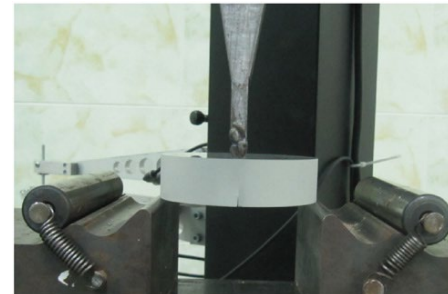
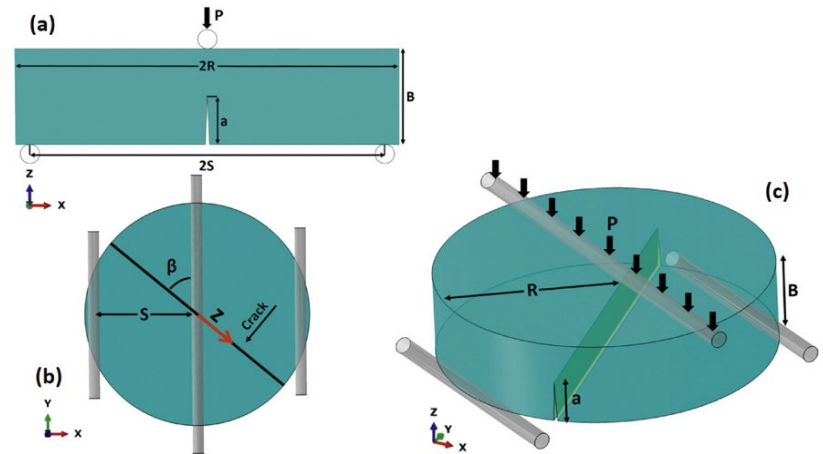
Macroscopic mode-dependent testing

Mixed mode study



Richard and Kuna, *Eng. Fract. Mech.*, 1990
All fracture-mode specimen

Mode I and III study



PMMA specimen subjected to pure mode I



asphalt specimen subjected to pure mode III

Aliha et al., *Materials and Design*, 2015
Edge Notched Disc Bend (ENDB) specimen for mode III

Maximum tangential stress criterion

Mode I

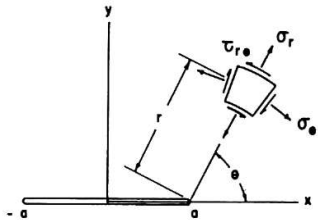


Fig. 1 Stress components near the crack tip in cylindrical coordinates

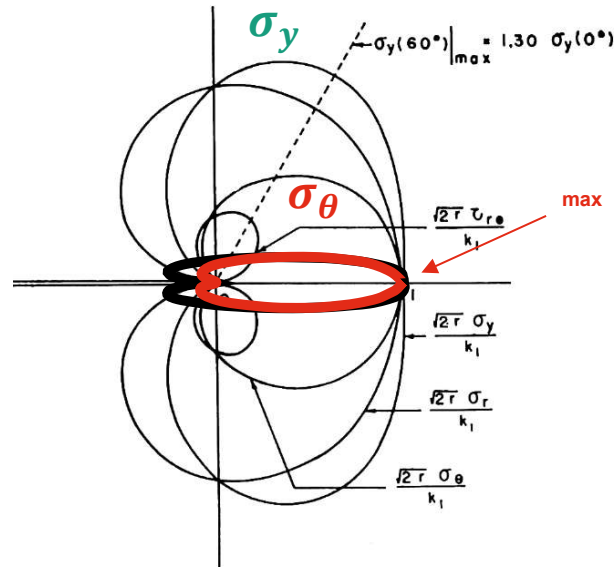


Fig. 2 Polar plot of stresses near the crack tip for symmetric case ($k_2 = 0$)

Mode II

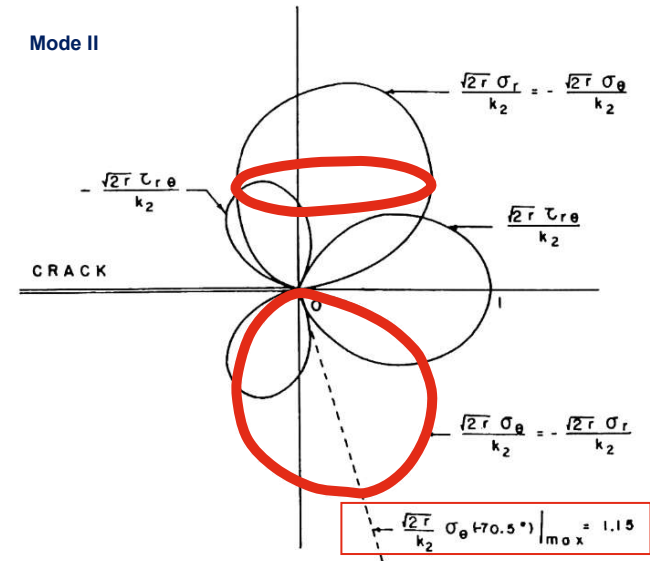


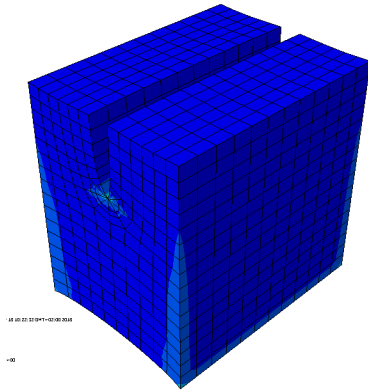
Fig. 3 Polar plot of stresses near the crack tip for skew-symmetric case

Erdogan and Sih, Journal of Basic Engineering, 1963

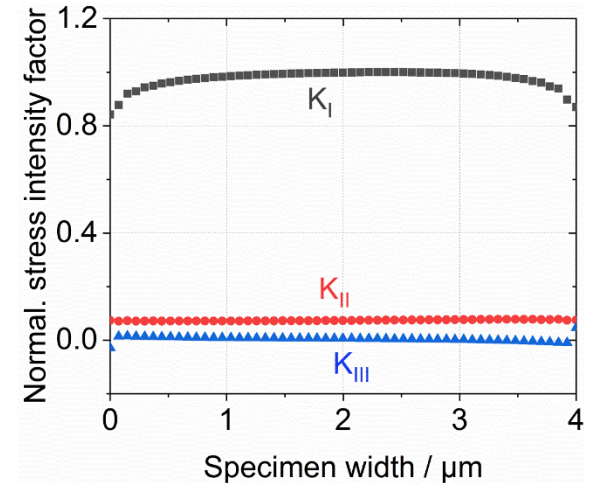
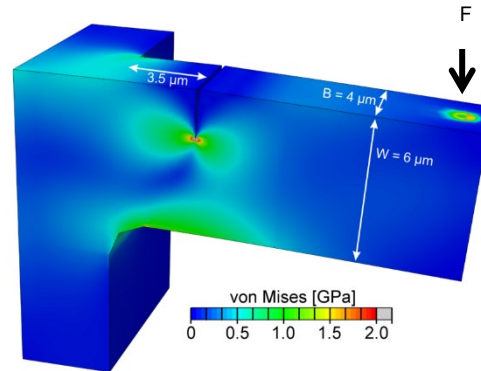
- Crack extension in a large plate subjected to general plane loading
- **Skew-symm. plane loading** leads to crack deflection under $\sim 70^\circ$, crack growth not in initial plane

Mode I / K_I - FE model

Ideal case



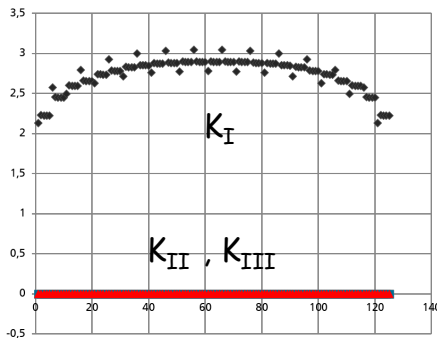
microcantilever



- For: $0.2 < a/W < 0.5$
- $L > 3(W-a)$

$$K_I = \frac{F L}{B W^{3/2}} f_{KI}(a/W)$$

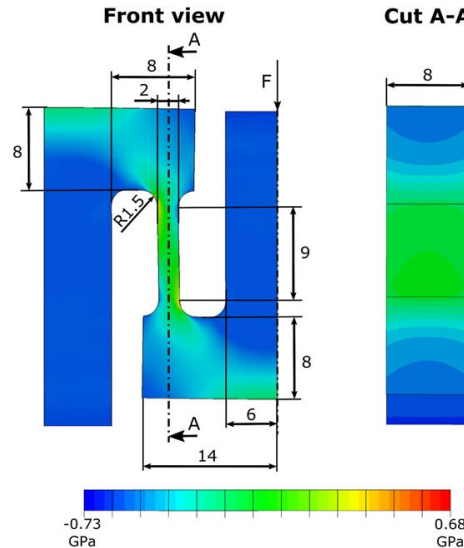
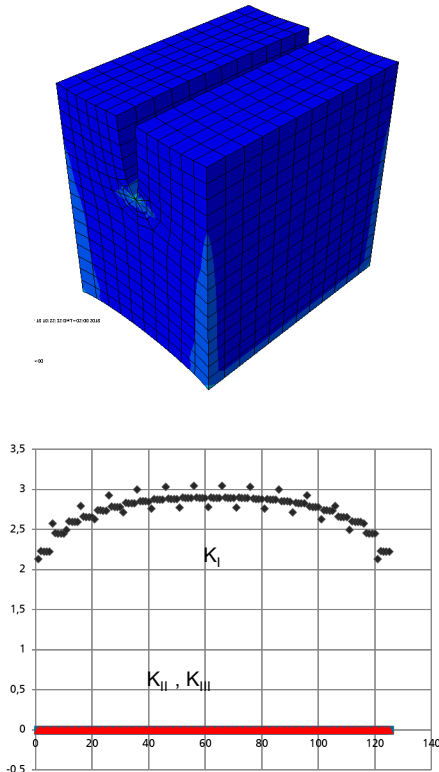
- K_I 10 times higher than K_{II} and K_{III}
- K_I , K_{II} and K_{III} nearly constant along the sample width B
- Slightly influenced by free surfaces



Mode I / K_I - FE model

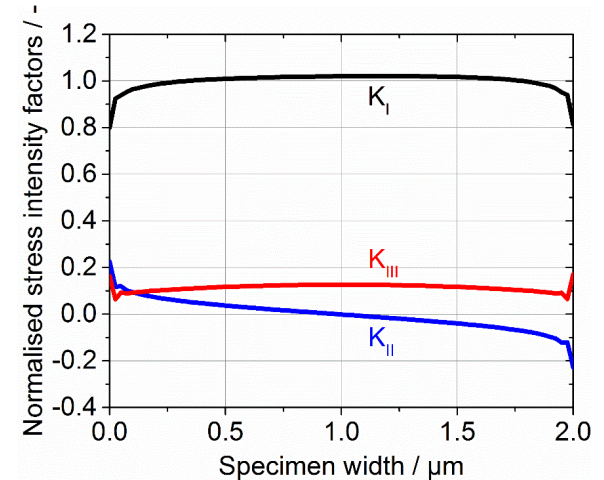
Ideal case

μ -SENT geometry



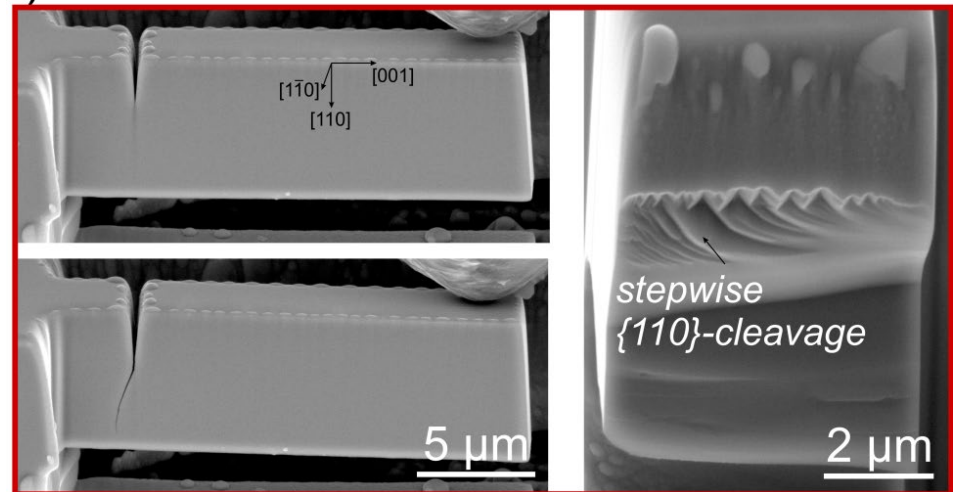
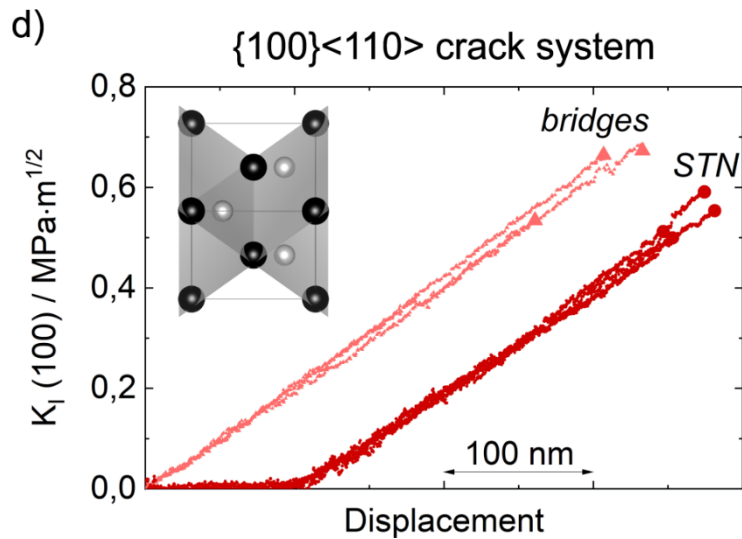
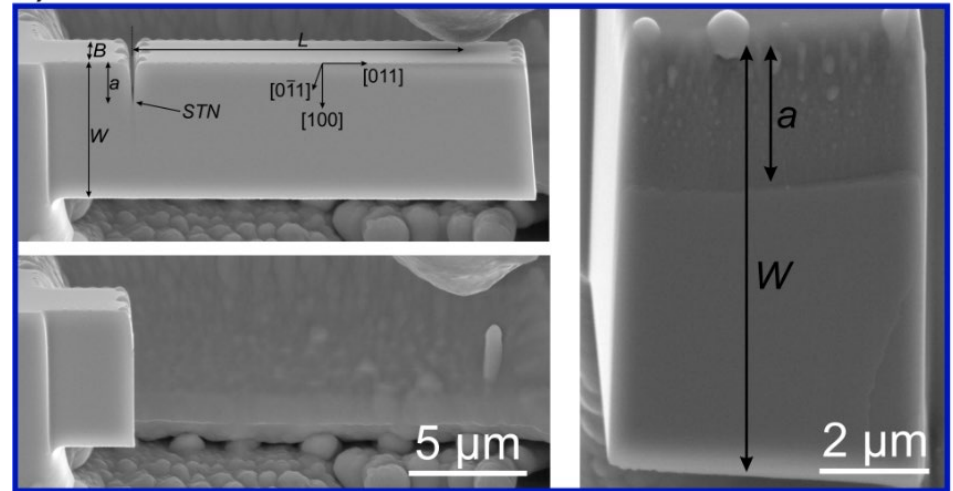
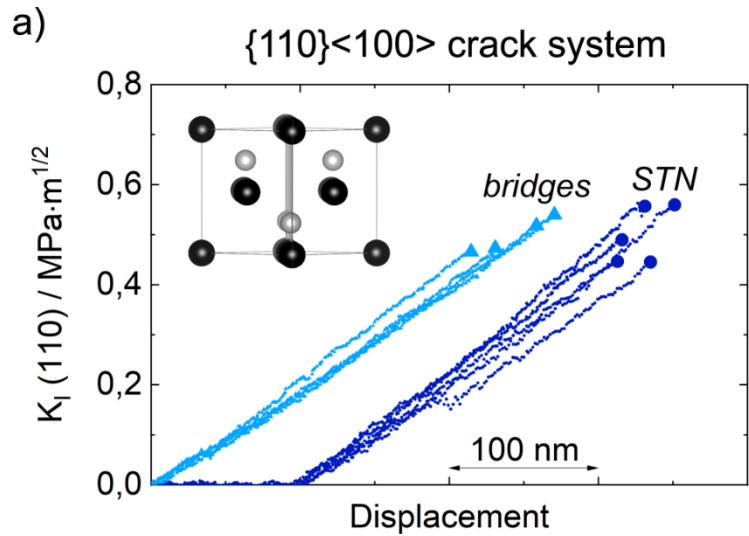
$$f(a/W) = 2.485 \left(\frac{a}{W}\right)^2 + 2.721 \left(\frac{a}{W}\right) + 0.401$$

$$K_I = \frac{F L}{B W^{3/2}} f_{KI}(a/W)$$



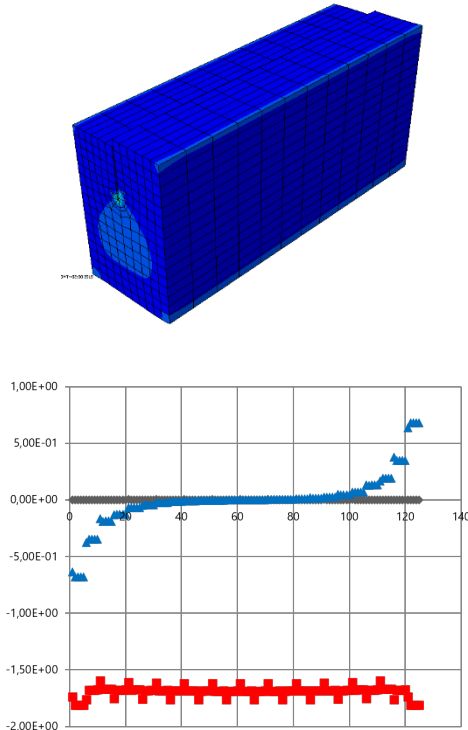
- Notched with $a/W = 0.25$
- Pure-tensile stress field at notch tip
- Low geometry factor and similar to SENT for small a/W ratios

Mode I fracture testing

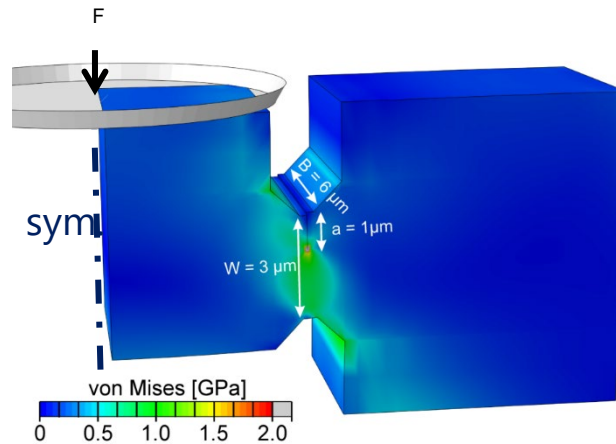


Mode II / K_{II} - FE model

Ideal case



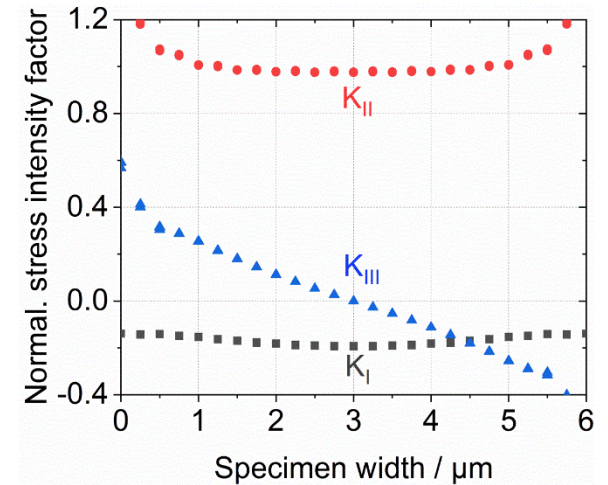
Symm. in-plane shear sample



- For: $0.2 < a/W < 0.5$

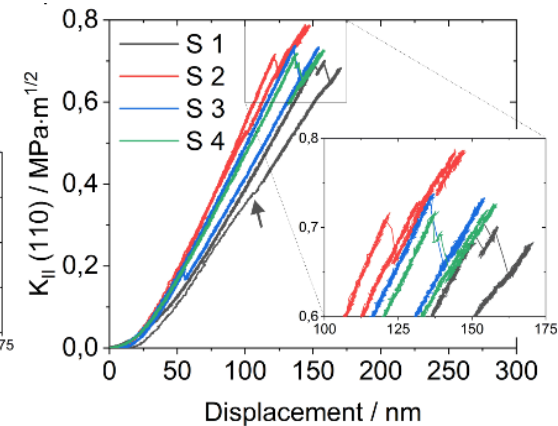
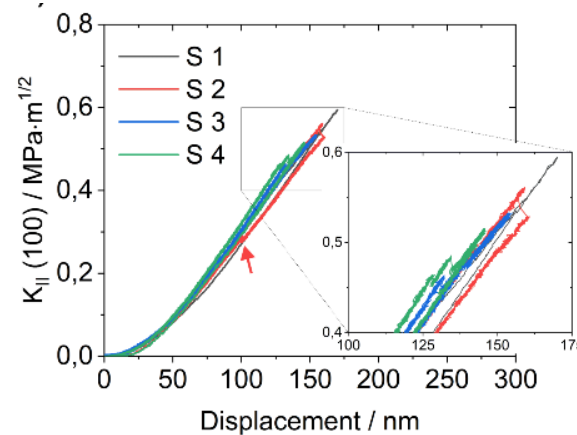
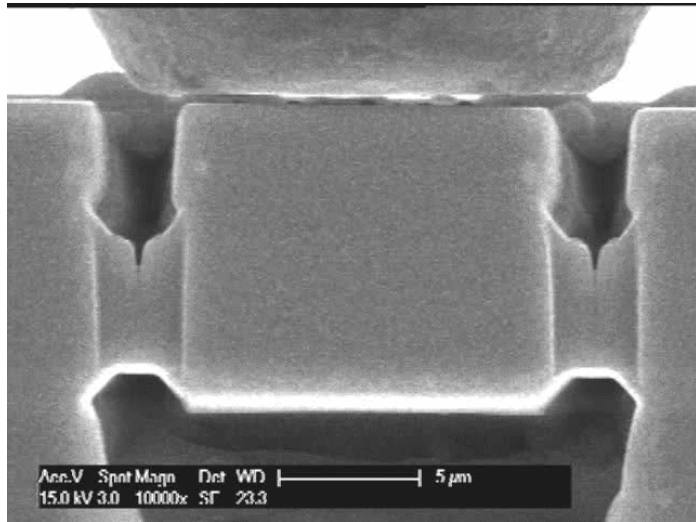
$$f(a/W) = 4.14(a/W) + 0.87$$

$$K_{II} = \frac{F}{2B\sqrt{W}} f_{KII}(a/W)$$

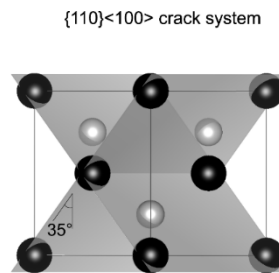
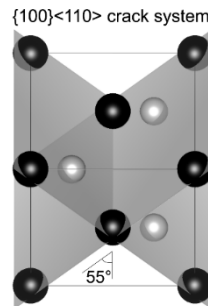
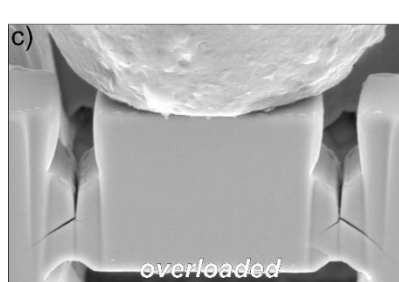


- K_{II} predominant
- K_I negligible, slightly negative because of compressive stresses
- $K_{III} = 0$ (average), due to Poisson ratio still present

Mode II fracture testing

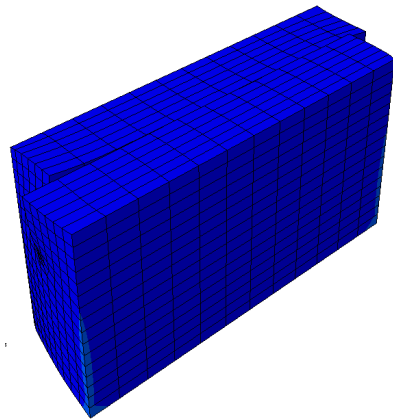


- Inclined $\{110\}$ planes in both cases
- Sliding does not occur in a homogeneous crystal
- The angle between notch direction and crack propagation direction for:
 $\{110\}\langle 100 \rangle$: $45^\circ \rightarrow$ "tougher"
 $\{100\}\langle 110 \rangle$: $60^\circ \rightarrow$ closer to MTSC

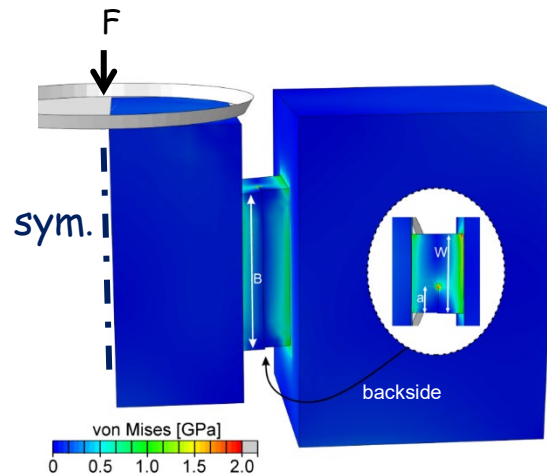


Mode III / K_{III} - FE model

Ideal case



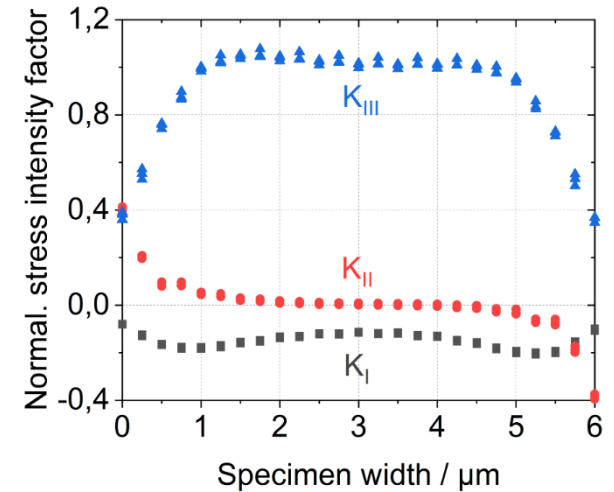
Symm. out-of-plane shear sample



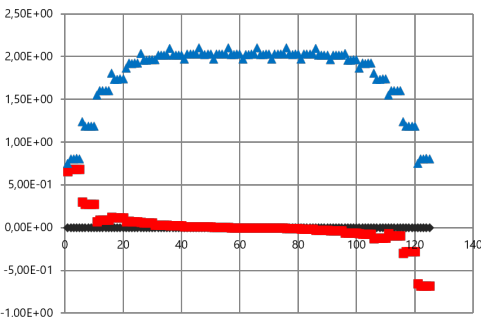
- For $0.2 < a/W < 0.5$

$$f(a/W) = 4.13(a/W) + 1.15$$

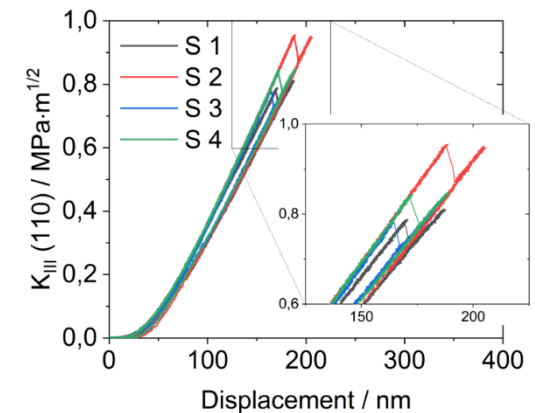
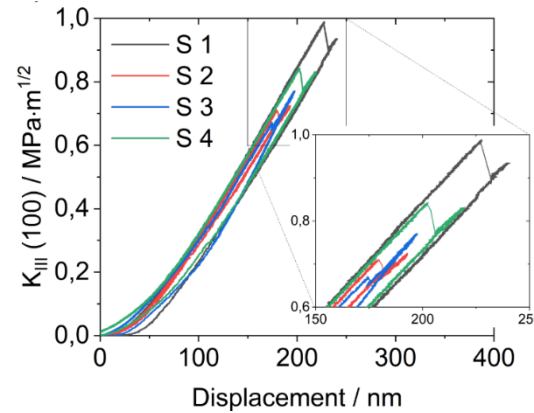
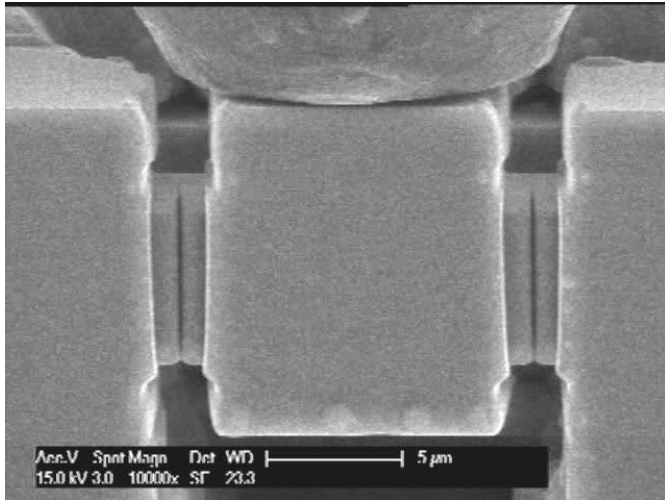
$$K_{III} = \frac{F}{2B\sqrt{W}} f_{K_{III}}(a/W)$$



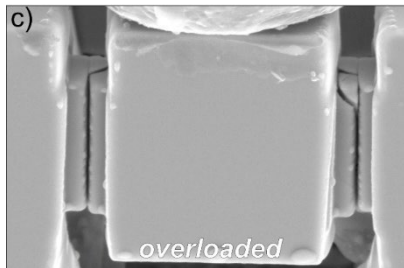
- K_{III} predominant
- K_I negligible, slightly negative because of compressive stresses
- $K_{II} = 0$ (average)



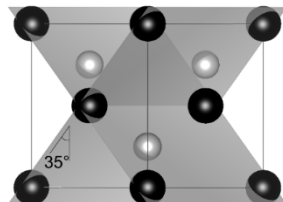
Mode III fracture testing



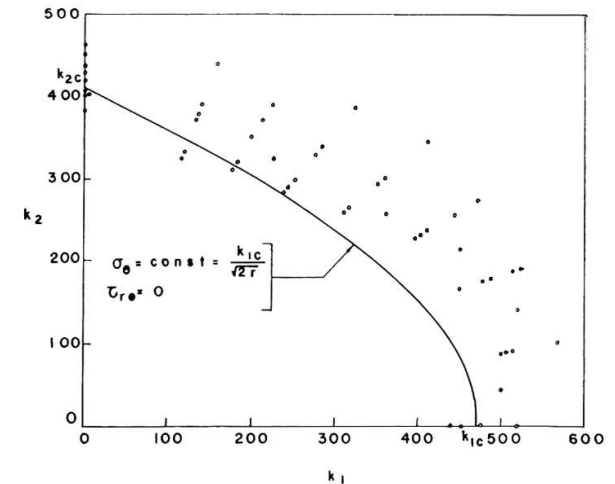
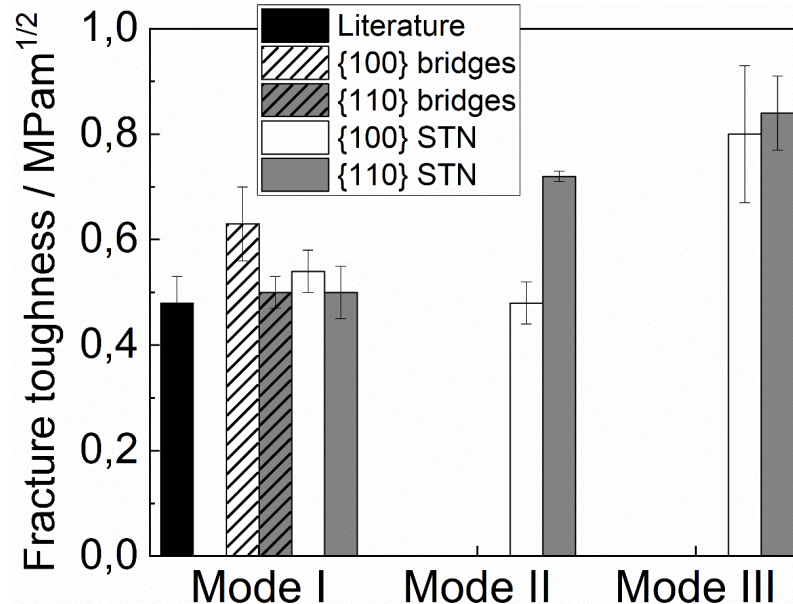
- Inclined $\{110\}$ planes in both cases
- Sliding does not occur
- Complex fracture pattern
- Again: $\{110\}\langle 100 \rangle$ slightly tougher than $\{100\}\langle 110 \rangle$ crack system



$\{110\}\langle 100 \rangle$ crack system



Overview - fracture toughness



Erdogan and Sih, Journal of Basic Engineering, 1963
 KI versus KII at the beginning of crack extension in a cracked plate under plane loading

- Literature data: Double-cantilever beams (DCB) and indentation fracture match results of this study
- Mode I lower bound, mode II and mode III higher

Summary and Conclusions : GaAs single crystals

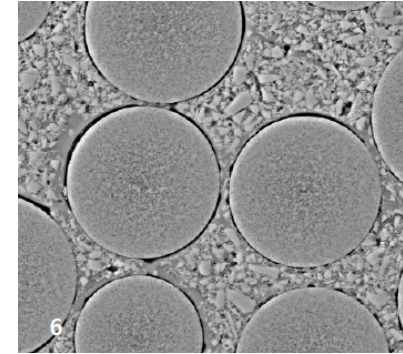
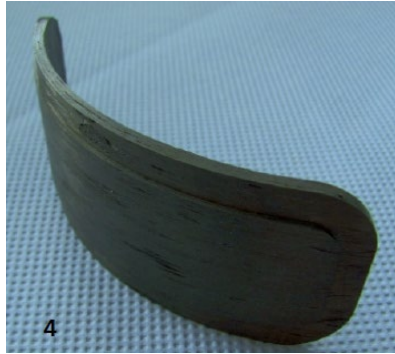
- New micro-scale specimens for pure mode II and pure mode III fracture experiments were designed and characterized by FE modeling.
- Mode I, II and III fracture testing of anisotropic single crystal GaAs was performed focusing on crack systems $\{100\}\langle 110\rangle$ and $\{110\}\langle 100\rangle$.
- Critical stress intensity factors of GaAs were determined for mode I, II and III loading and fracture patterns were analyzed.
- Mode I: Careful when studying anisotropic material and when applying the bridge notch (can influence fracture toughness)
- Mode II and III results confirm macroscopic studies and GaAs shows fracture anisotropy because of the preferred $\{110\}$ cleavage planes

Willkommen
Welcome
Bienvenue



Studying interface fracture in ceramic matrix composites

Ceramic matrix composites



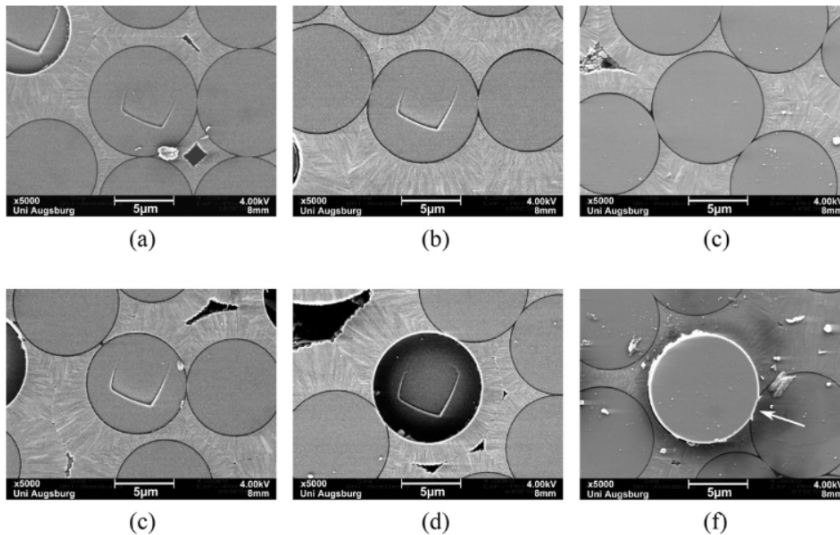
Curved tile made of SiC/SiC

SiC pipe reinforced with SiC/SiC

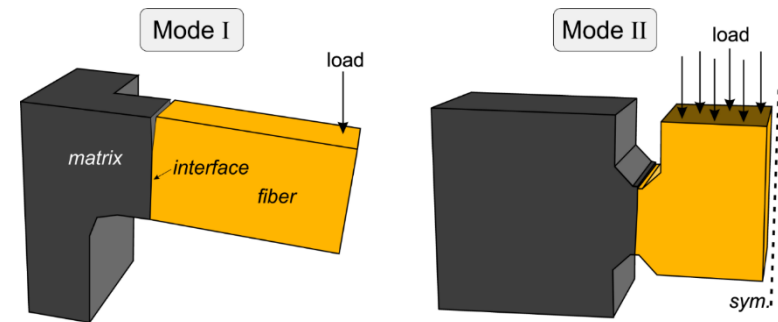
SEM image of microstructure

- Good high temperature mechanical properties, improved fracture and damage tolerance, good corrosion resistance in air and fuel gas atmosphere
- Complex shaping and geometries possible (large and thin walled components)
- Applications: Aero-engines, nuclear technology (radiation tolerance)
- SiC fibers surrounded by a SiC matrix with a Pyrolytic carbon (PyC) interphase
- Knowledge of interface toughness necessary to allow proper design of components

Failure along PyC interphase: Push-out test



Mueller et al., J. Eur. Ceram. Soc., 2013

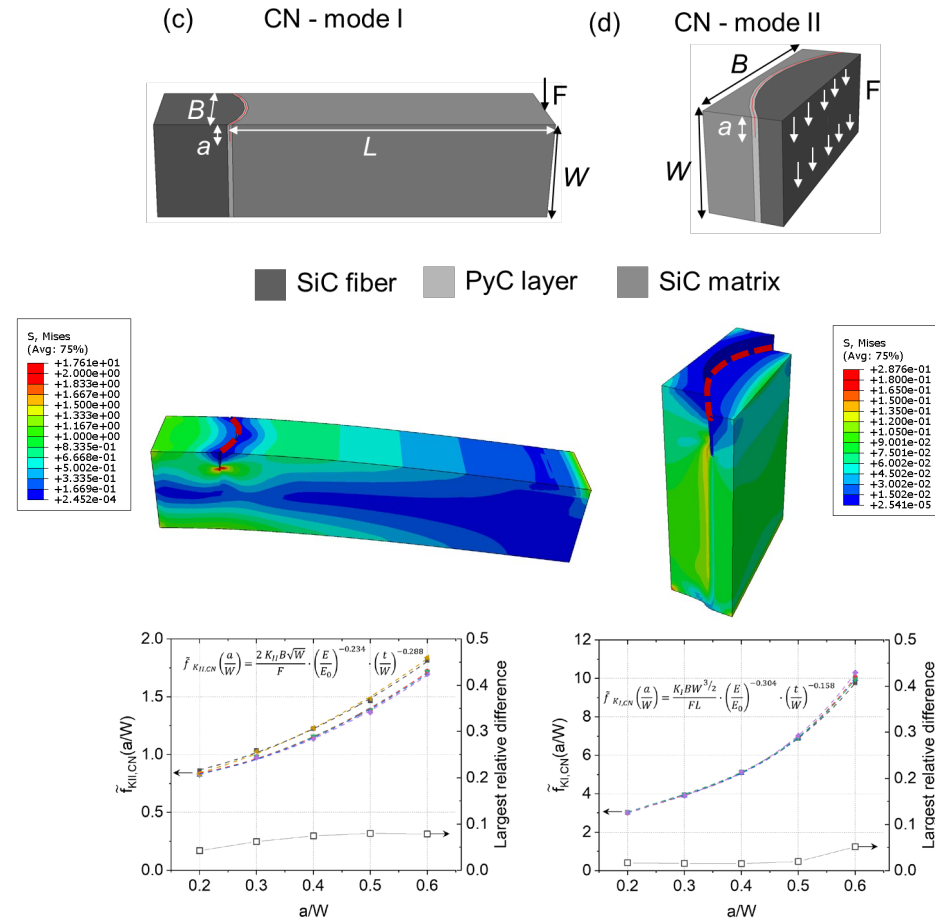


Ast, Schwiedrzik et al, Mat. & Des., 2020

- State of the art: Fiber push-out tests using a flat punch indenter
- Assumptions concerning stress distribution and crack growth along interface
- Can novel micromechanical specimens measure local fracture toughness more accurately?

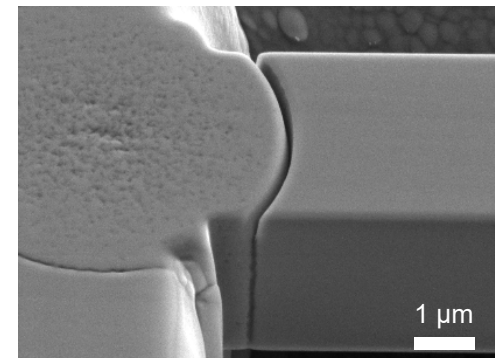
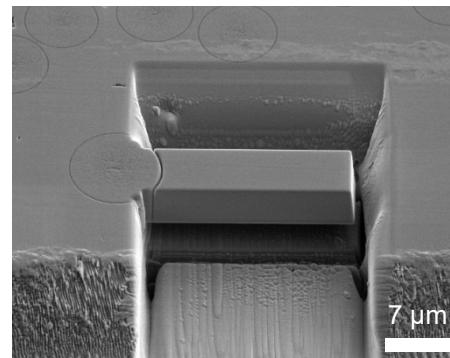
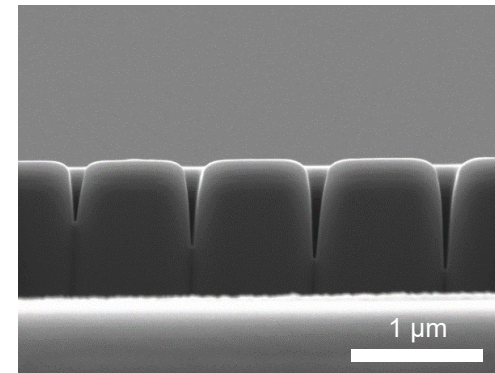
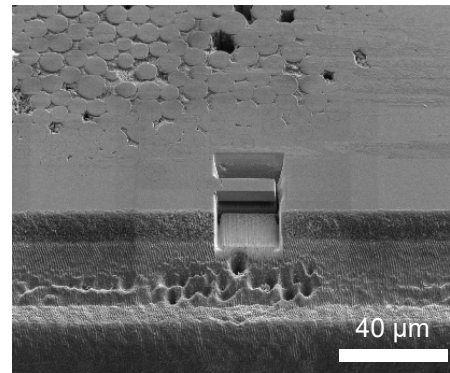
Micromechanical sample development

- Parametric finite element simulations were performed varying notch depth, interphase thickness, and interphase stiffness
- Analysis of KI,II,III distributions at the notch root show that one mode is dominating in each specimen geometry
- The geometry factor was determined and normalized by the varied parameters to allow quantitative analysis of the experimental force measurements



Micromechanical sample preparation

- Ps laser ablation of large areas to create edge near visible SiC fibers in correct orientation
- FIB milling from top and side to create freestanding cantilevers at a fiber-matrix interface
- Optimization of FIB settings at low currents to generate sharp and reproducible notches with known depth
- Cutting of curved notches along the PyC interphase

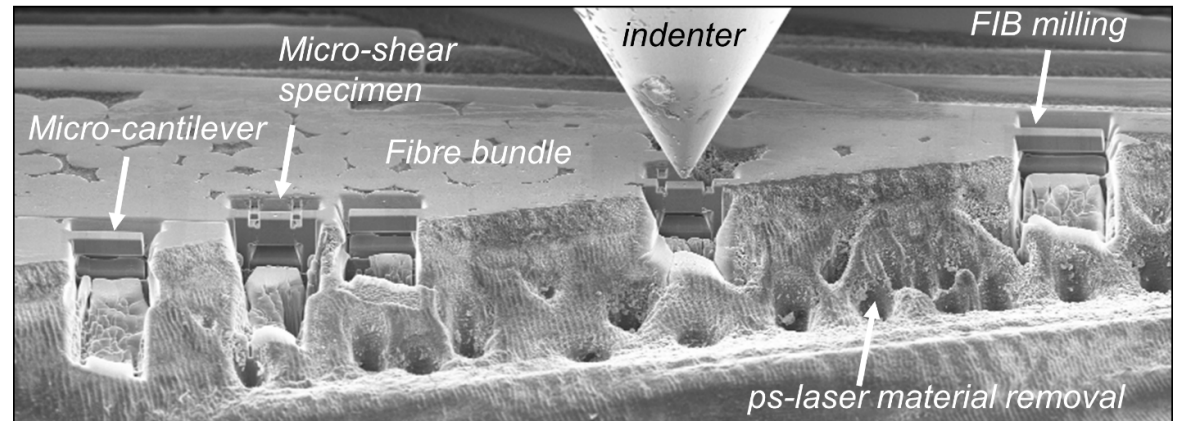
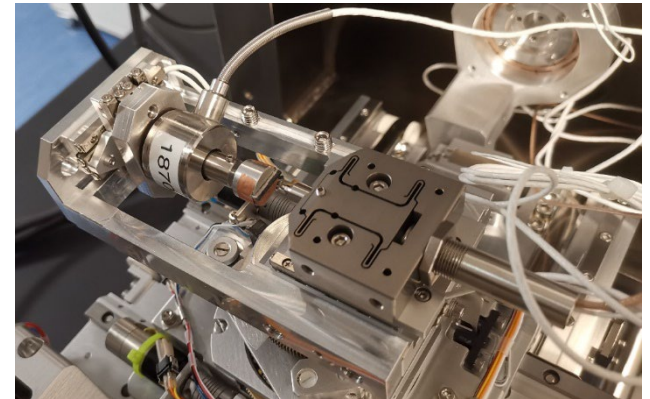


Experimental setup

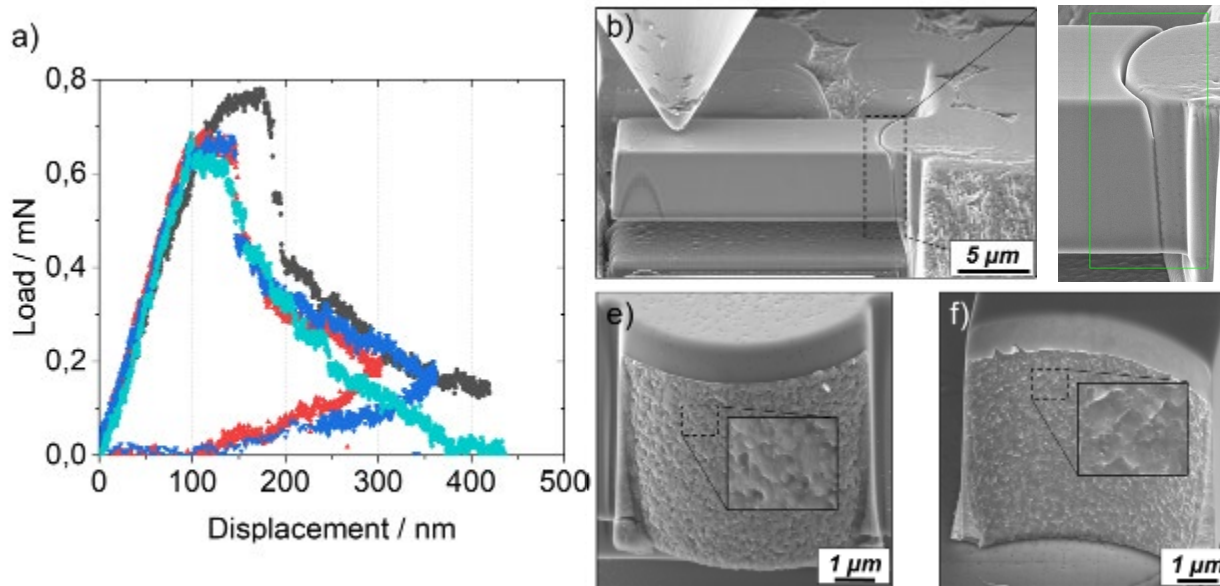
- Preparation of notched and unnotched specimens loaded in bending and shear
- Testing inside a SEM using an in situ micromechanical tester
- Simultaneous measurement of force and displacement allows fracture mechanical analysis



FIB/SEM Crossbeam Auriga

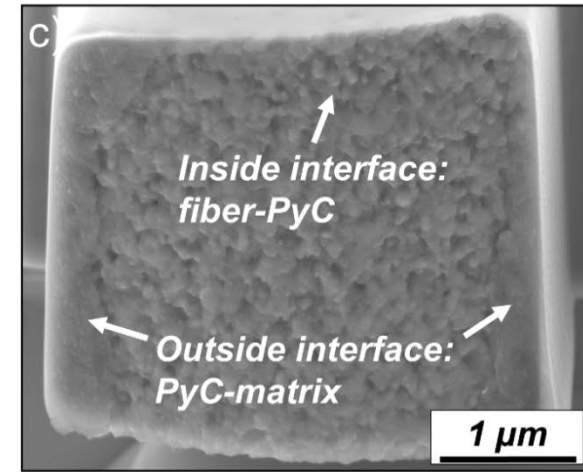
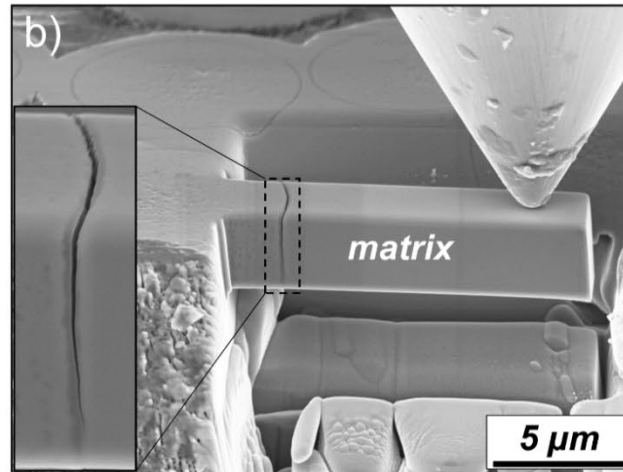
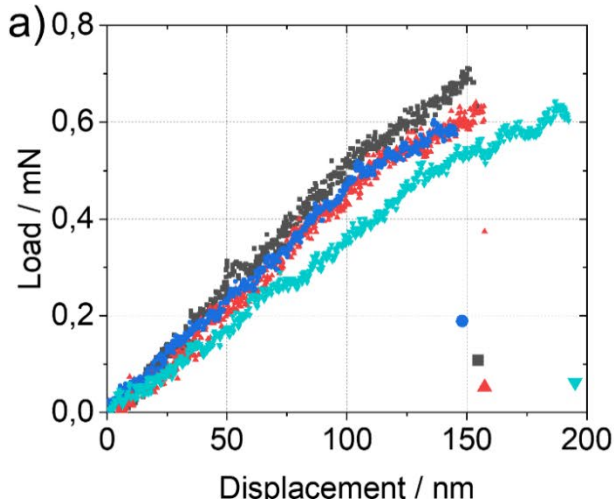


Interfacial fracture toughness - mode I



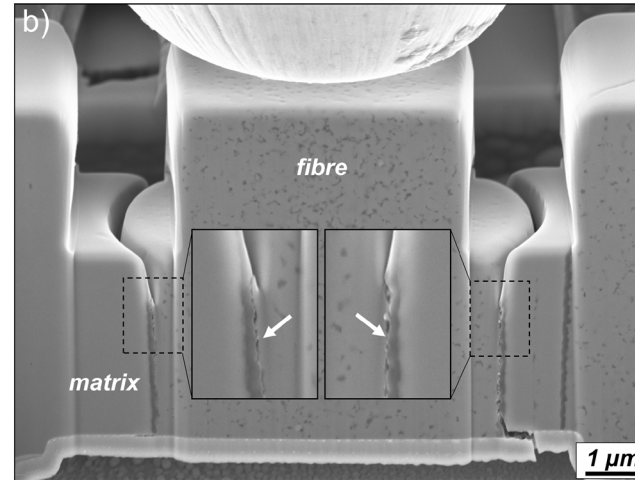
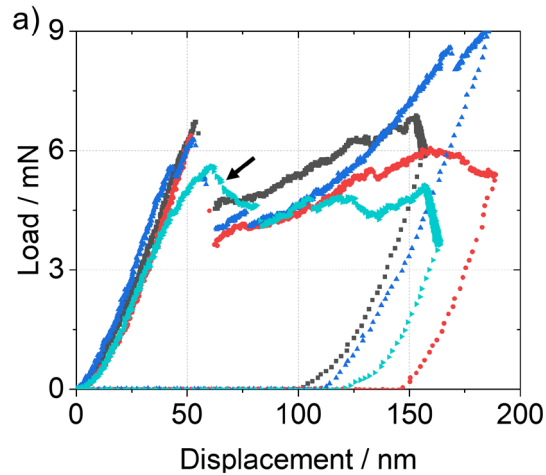
- Consistent fracture loads: K_{IC} of PyC interphase is $0.24 \pm 0.02 \text{ MPa}\sqrt{\text{m}}$
- Fracture surfaces are consistent with fiber-PyC interface failure
- Crack deviates from fibre-PyC interface at free edges due to complex stress state

Interfacial strength - bending



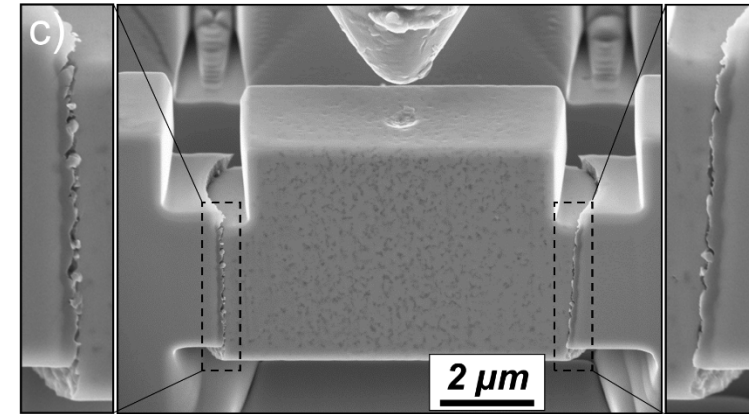
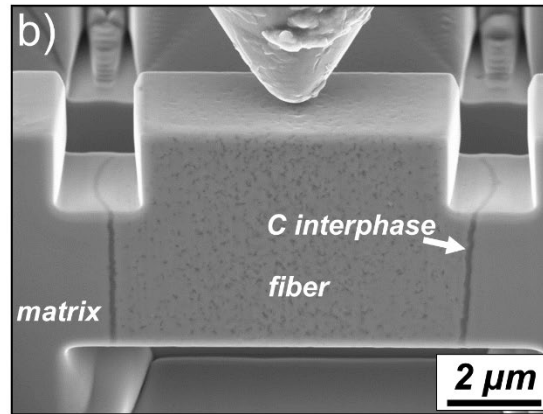
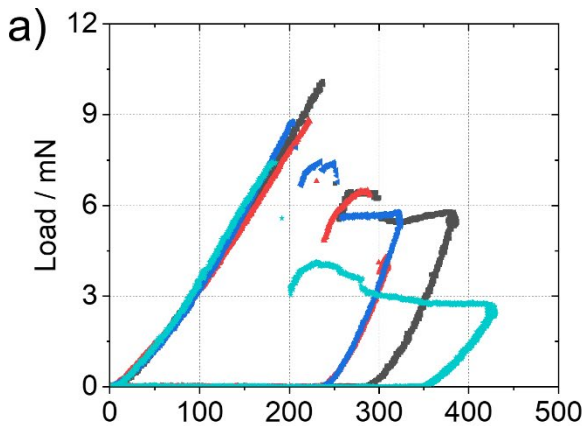
- Loading of unnotched cantilevers allows measuring interfacial strength
- PyC interphase strength in bending: 934 ± 137 MPa
- Failure along the fibre-PyC interface on the inside, crack deviates at free edges

Interfacial fracture toughness - mode II



- K_{IIC} of PyC interphase: $0.17 \pm 0.06 \text{ MPa}\sqrt{\text{m}}$
- Higher variation likely due to small deviations from perfect symmetry
- Crack travels consistently along the fibre-PyC interface

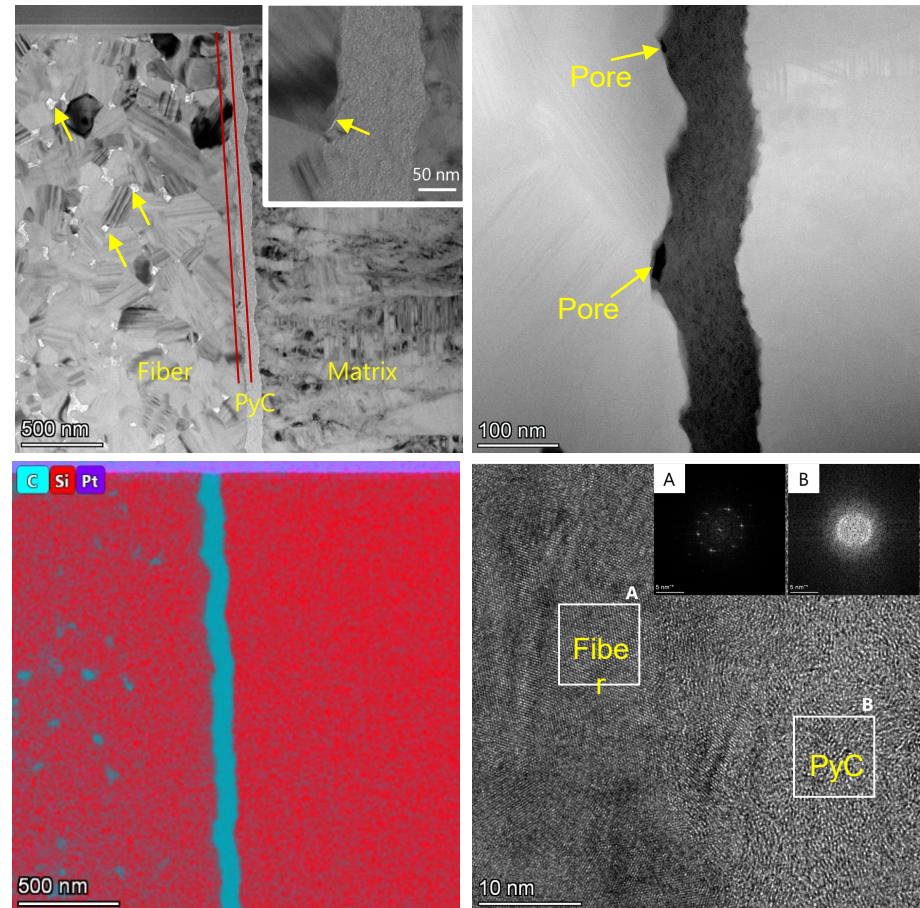
Interfacial strength - shear



- Loading of unnotched shear specimens allows measuring shear strength
- PyC interphase strength in shear: 418 ± 36 MPa
- Cracking in shear mode along fibre-PyC interface

Why does the fibre-PyC interface fail?

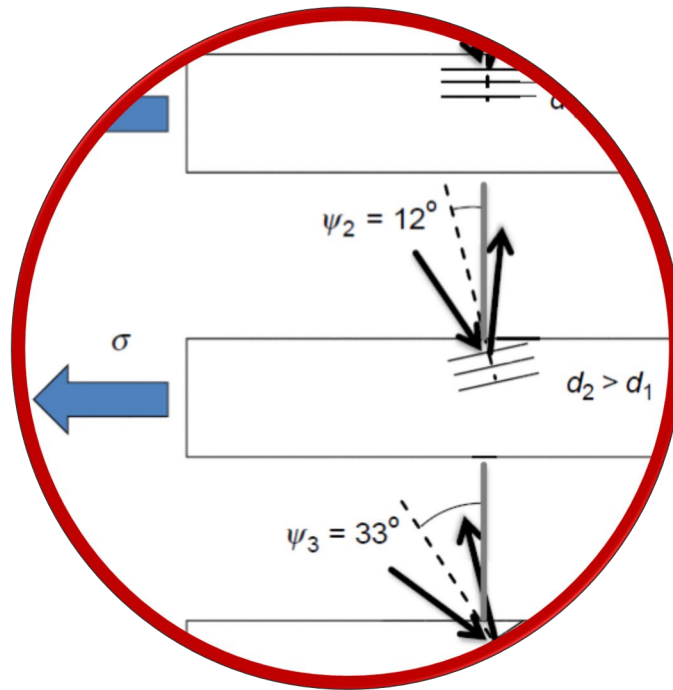
- TEM lamellae were prepared by a FIB lift-out technique
- TEM highlights different grain structure of fiber and matrix
- Interface between SiC fiber and PyC interphase is rougher compared to the PyC-matrix interface
- Crack in a confined system steers towards the more compliant phase
- Theoretical defect size was computed to be around 20nm-
- Local defects of 20-50nm size can be found at the fiber-PyC interface



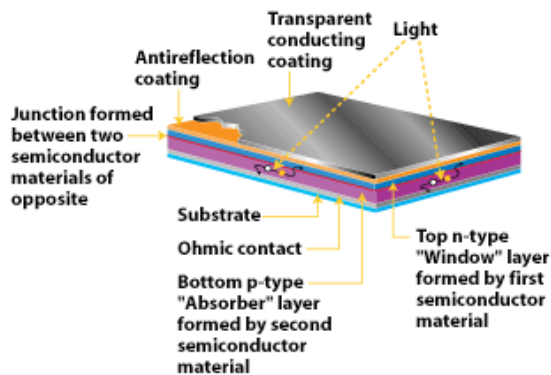
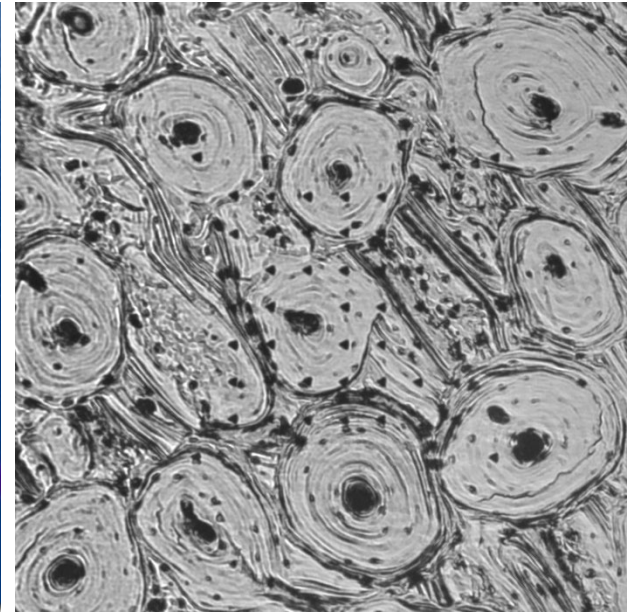
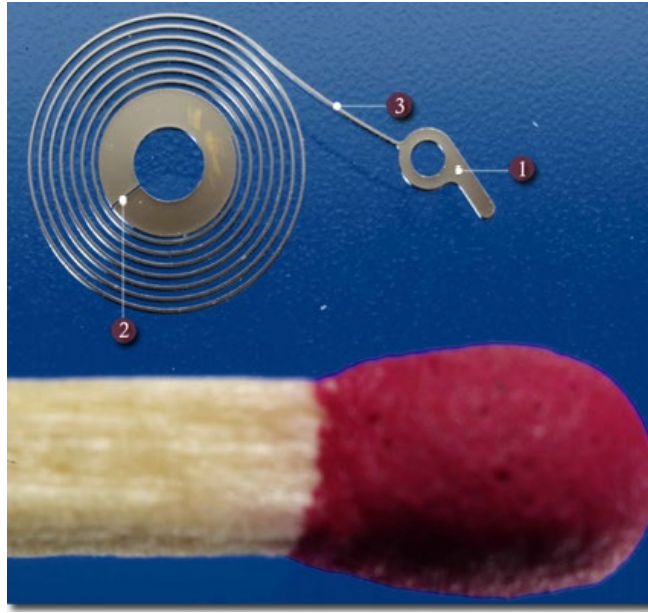
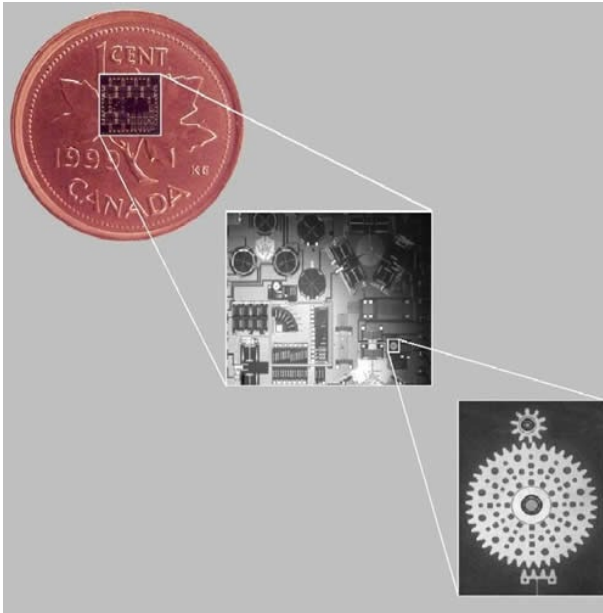
Summary

- Novel FIB-based specimens were developed that allow probing of local mechanical properties of thin interphases
- Mechanical characterization of the specimen geometries by parametric finite element analysis allows interpreting the experiments in a quantitative fashion
- Consistent measurements of fracture toughness and strength under bending and shear loading can be obtained
- PyC interphase is stronger and tougher in bending than shear
- Failure always occurs at the fibre-PyC interface
- Bending strength is consistent with flaw size found in TEM analysis

Micromechanics at extremes



Need for nanomechanical testing



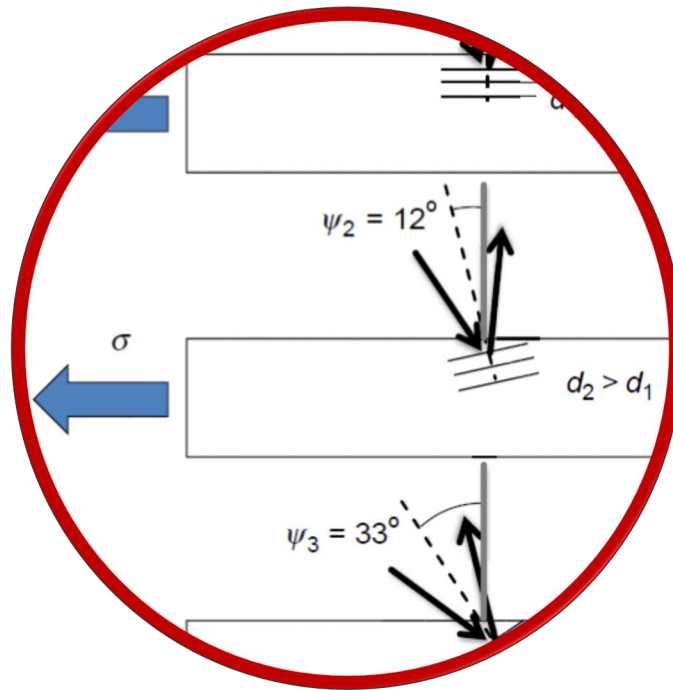
Cases where bulk testing is not possible:

- Thin films
- Microcomponents/MEMS devices

Small length-scale testing

- Individual phases of complex materials
- Size effects

High temperature testing



Motivation for high temperature testing

- Measurement of properties at application relevant temperatures
- Identification of ductile-brittle transition temperatures
- Measurement of mechanical properties at varying temperature allows identifying deformation mechanisms based on thermal activation analysis

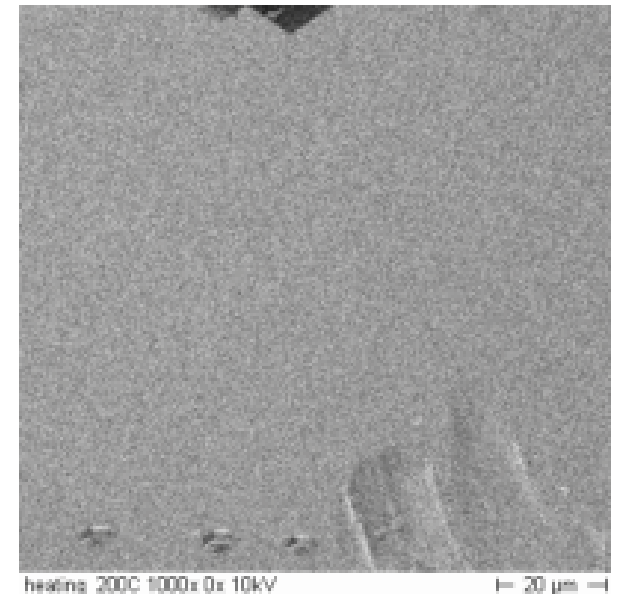
Challenges in elevated temperature nanoindentation

$$\text{experimental data} = \text{mechanical deformation} + \underbrace{\text{thermal drift behavior}}_{\text{artifact of measurement due to thermal effects}} + \underbrace{\text{noise}}_{\text{instability of the measurement system}}$$

thermal drift - drift occurs due to thermal expansion of the indenter tip and load frame

system stability - control electronics and sensors must be protected from heat to avoid noise in experimental data

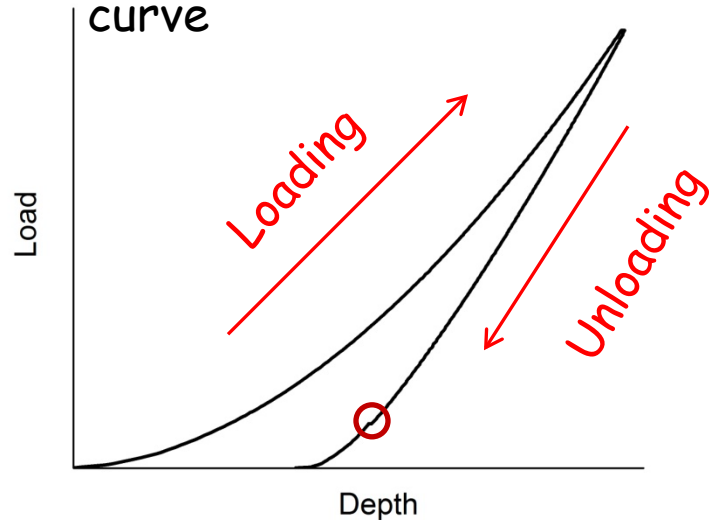
sample and tip oxidation - undesirable for surface property testing. **oxidative loss of diamond** will damage the indenter tip at high temperatures (>400 degrees C) without high **vacuum** or **high purity inert gas**.



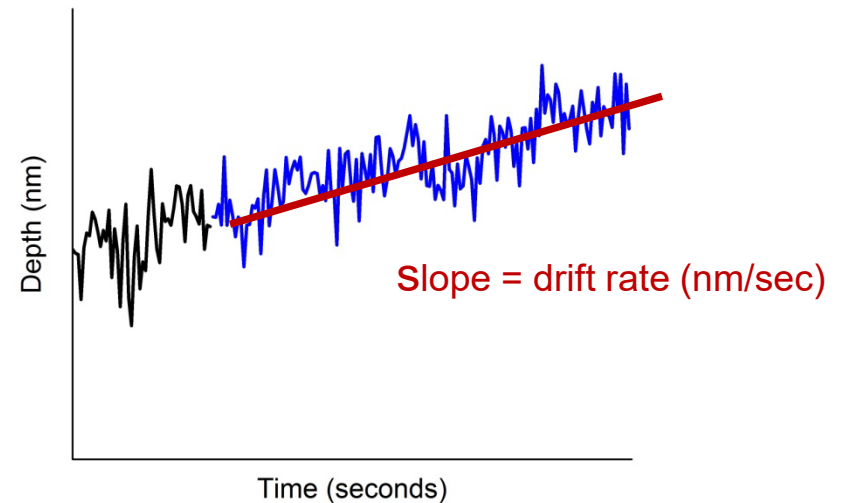
frame drift on heating to 200°C

thermal drift

schematic load-displacement curve



thermal drift data



- thermal drift measured at 10% peak load value during unloading, typically less than 0.05nm/sec at RT

effect of drift rate on hardness

$$H = \frac{F_{max}}{A_c}$$

Let's assume an ideal tip area function for simplicity. $A_c = 24.5 d^2$

Let maximum depth of penetration be 'd' nm.

Let drift rate be 'r' nm/sec

Let loading time be 't' sec

Let the data unaffected by drift be represented by H_1

Let the data affected by drift be H_2

Therefore, change in hardness due to thermal drift (ΔH) = $(H_2 - H_1) / H_1$

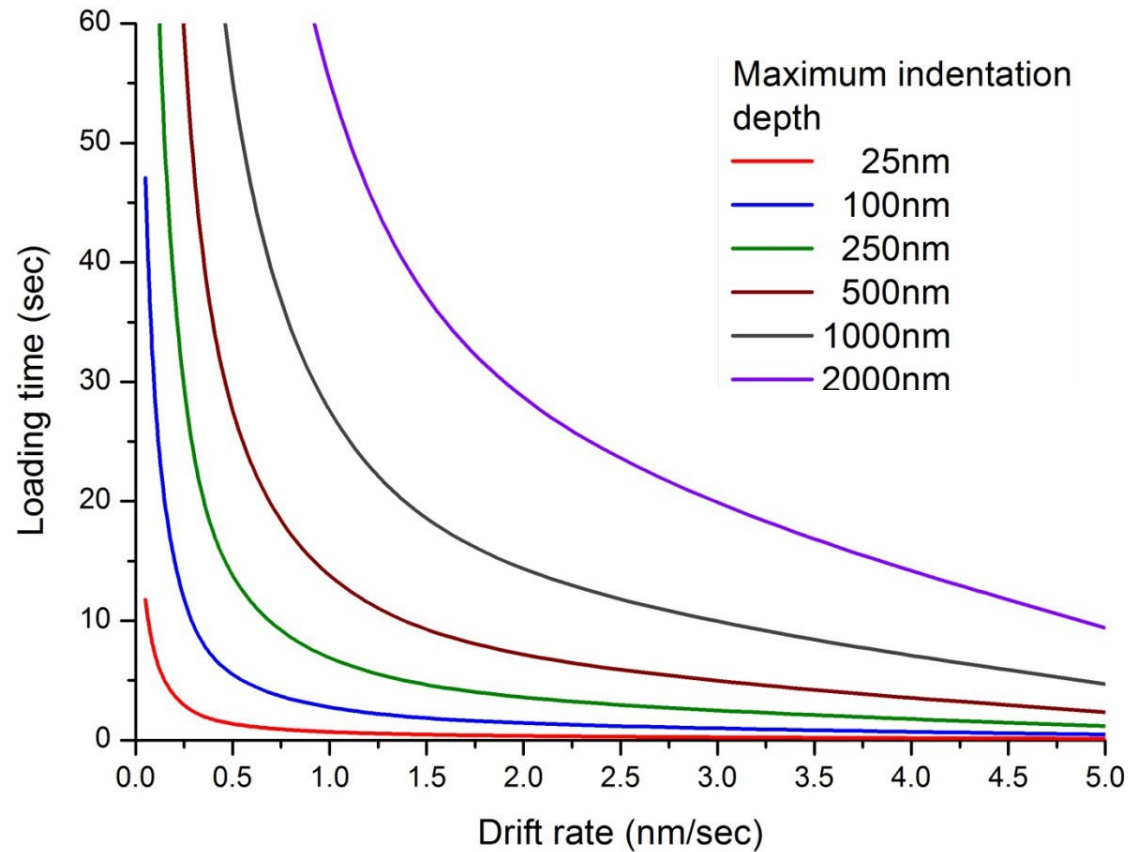
If we take 5% change in hardness as the tolerance in our experiments:

$$\Delta H = \frac{H_2 - H_1}{H_1} = \frac{\frac{F_{max}}{24.5 (d+rt)^2} - \frac{F_{max}}{24.5 d^2}}{\frac{F_{max}}{24.5 d^2}} = 0.05$$

Rearranging, we obtain:

$$1.05r^2t^2 + 2.1 drt + 0.05d^2 = 0$$

hardness with 5% error



low thermal drift rates essential for accurate property measurement at elevated temperatures and low indentation depths

solutions to overcome thermal drift

frame drift

- (a) long stabilization times to heat the tip passively - sample heating
- (b) heating both indenter and sample separately - indenter tip heating
- (c) active surface referencing - differential displacement measurement

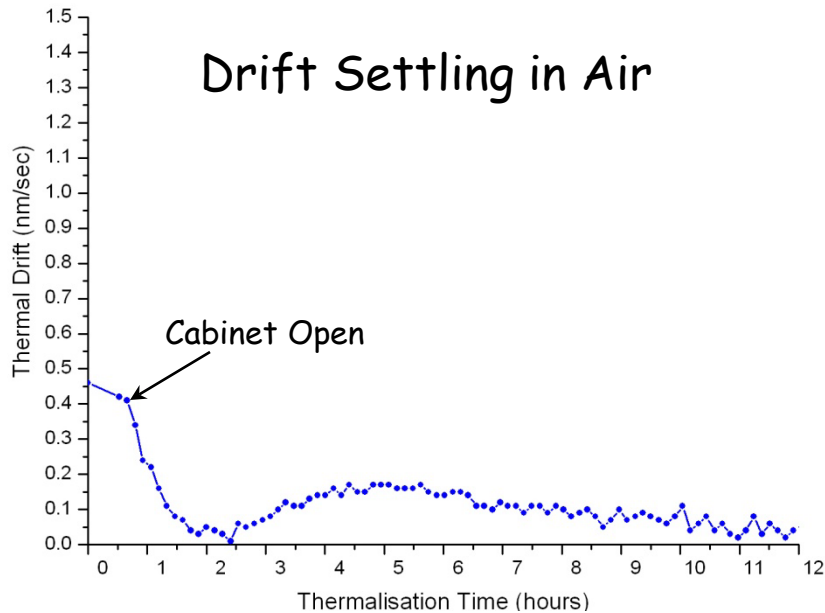
contact drift

- (a) tip-sample surface temperature matching - Calibrating and matching the tip temperature with the sample surface temperature in case of sharp tips
- (b) use flat punch - constant contact area during indentation/micro-pillar compression

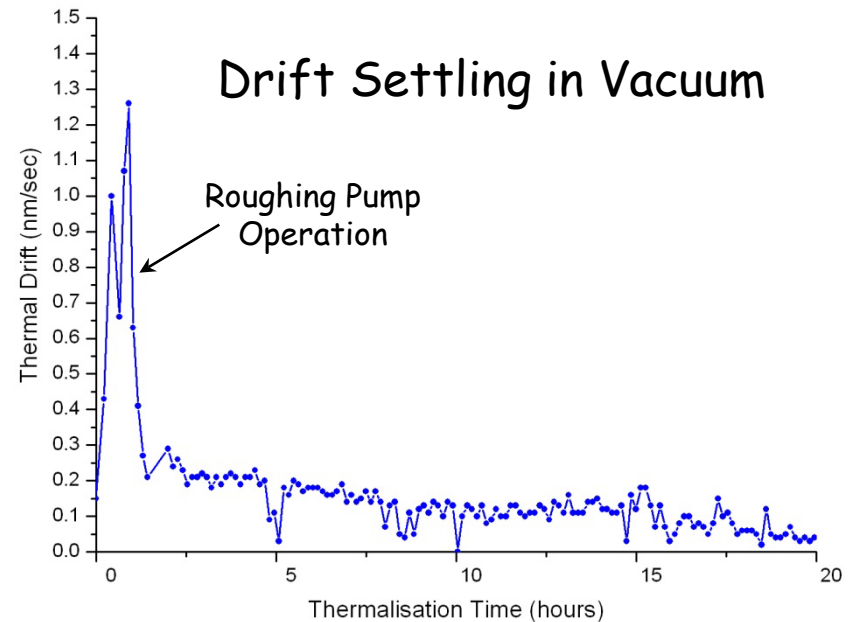
electronic drift

- (a) contain heating locally to avoid heat flow back to the sensors/electronics by water cooling

System Thermal Drift

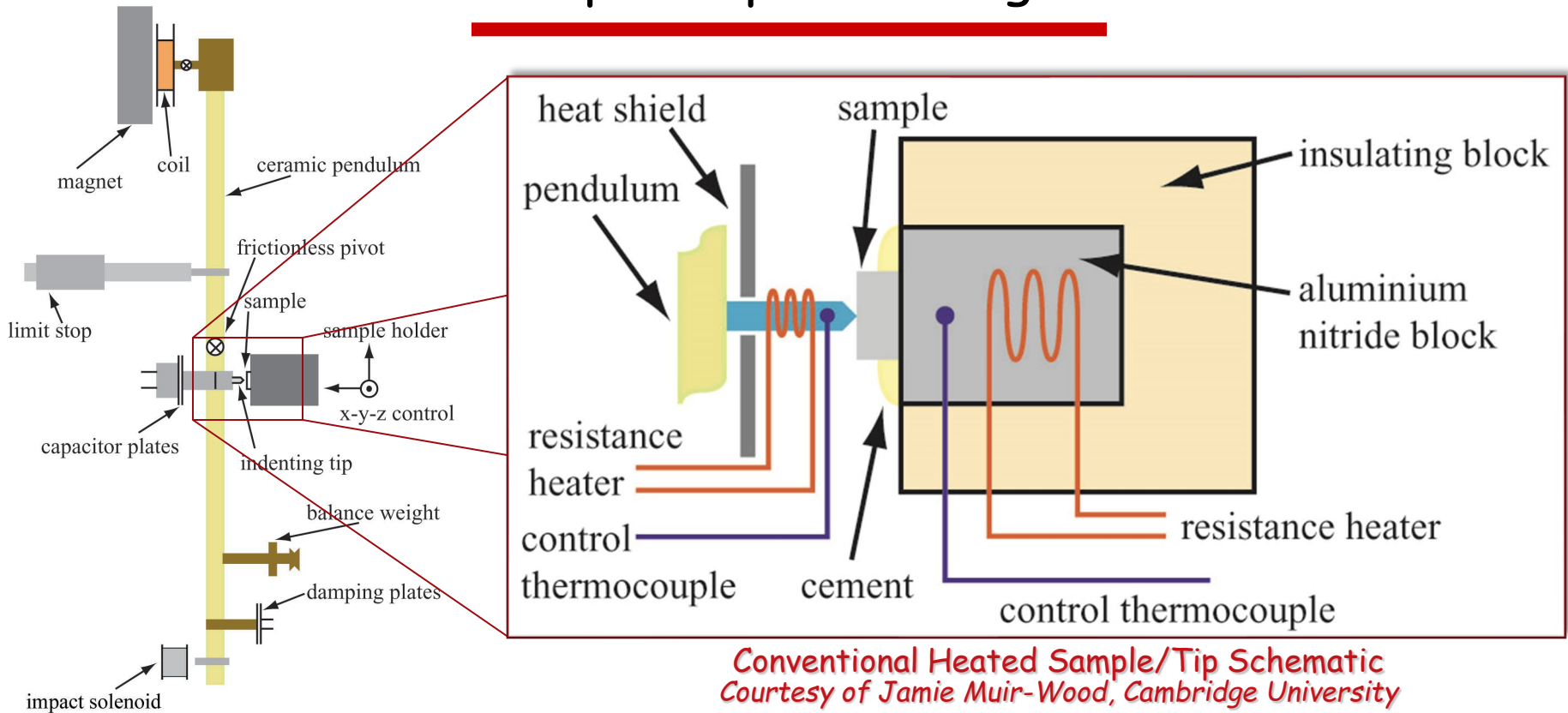


- Air currents from opening cabinet disturb system.
- System temperature achieves uniformity by convection.



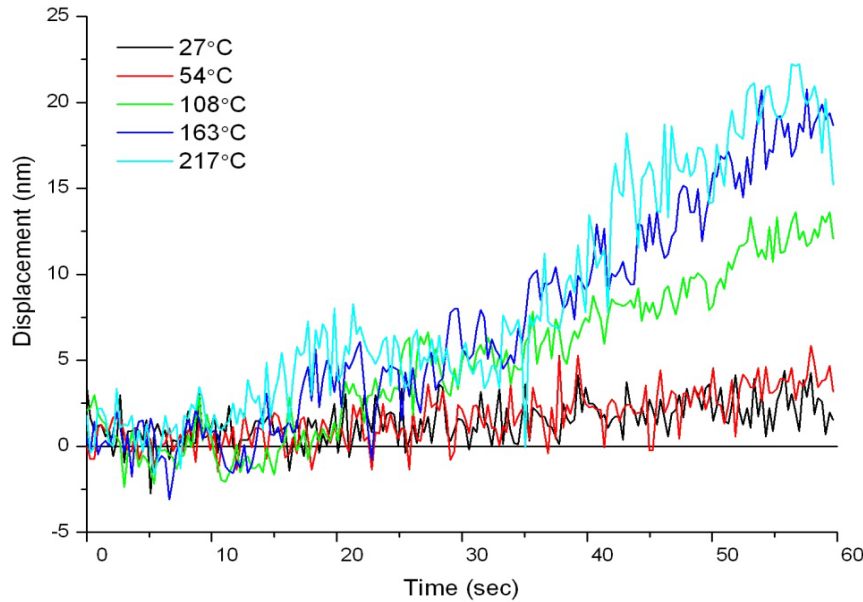
- Vacuum pumping disturbs system.
- System temperature reaches uniformity by conduction.

Tip-Sample Heating

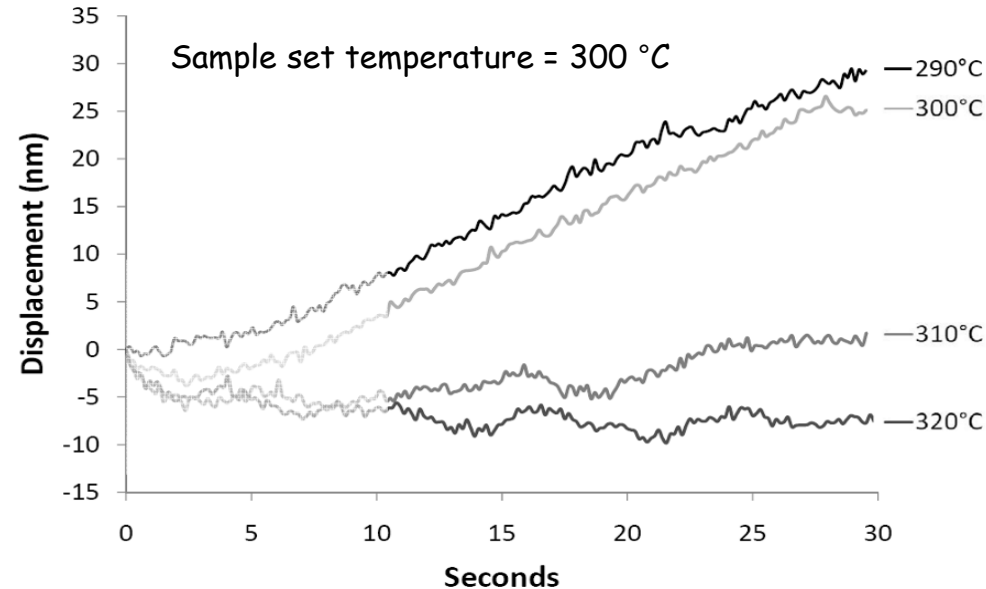


- Heater schematic for a MicroMaterials Ltd. Nanoindentation system.
- Horizontal indentation plane avoids convective heating problems in atmosphere; vacuum removes convection.
- Tip and Sample heaters/thermocouples on independent feedback loops allow testing to be performed at thermal equilibrium.

Thermal Drift at Elevated Temperature



Tip and Sample at Nominal Temperature

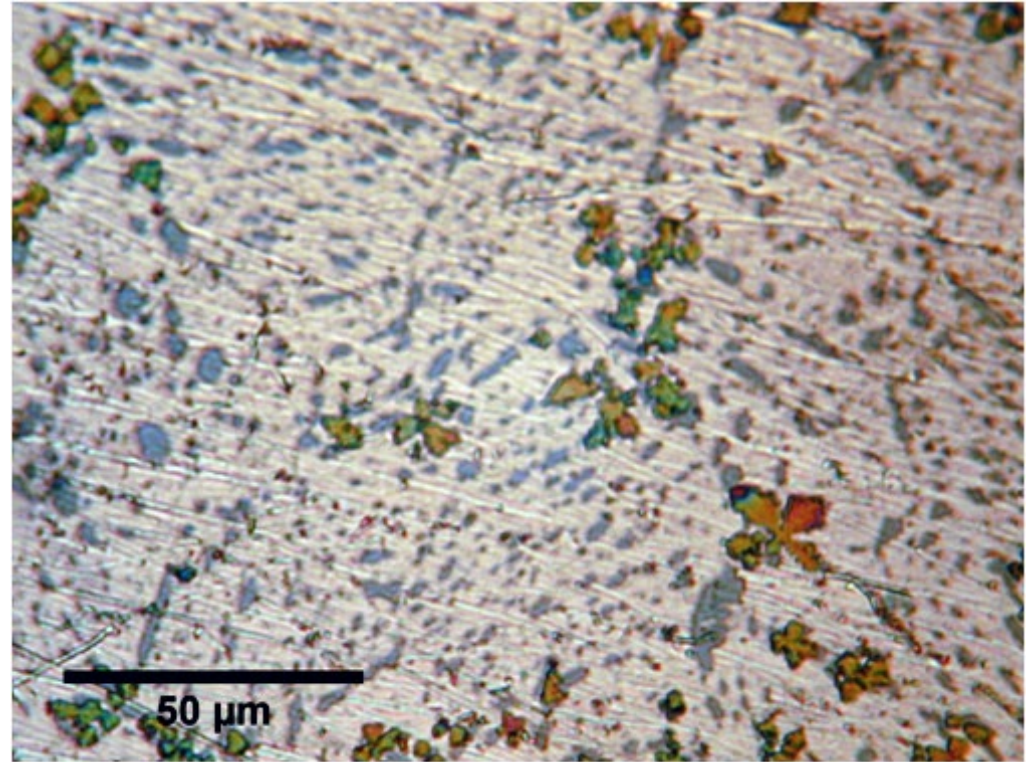


Tip Temperature Tuned
Courtesy of Sandra Korte, Cambridge University

- Heating both the sample and tip is required to achieve thermal equilibrium, but this does not immediately remove issue of drift.
- Thermocouples cannot be placed exactly at the contacting surfaces, so there is a gradient between measured and true temperature.
- True tip temperature can be calibrated, but variation in sample geometry and thermal conductivity prevent simple sample temperature calibration.
- Assuming drift is zero at thermal equilibrium, varying sample temperature until drift drops to zero achieves both zero drift and accurate sample temperature.

Sample Oxidation

- Nearly all metals oxidise.
- Reactive metals such as Samarium can oxidise quickly at room temperature.
- Careful preparation is required to maintain the oxide thickness on a sample does not interfere with indentation measurements.



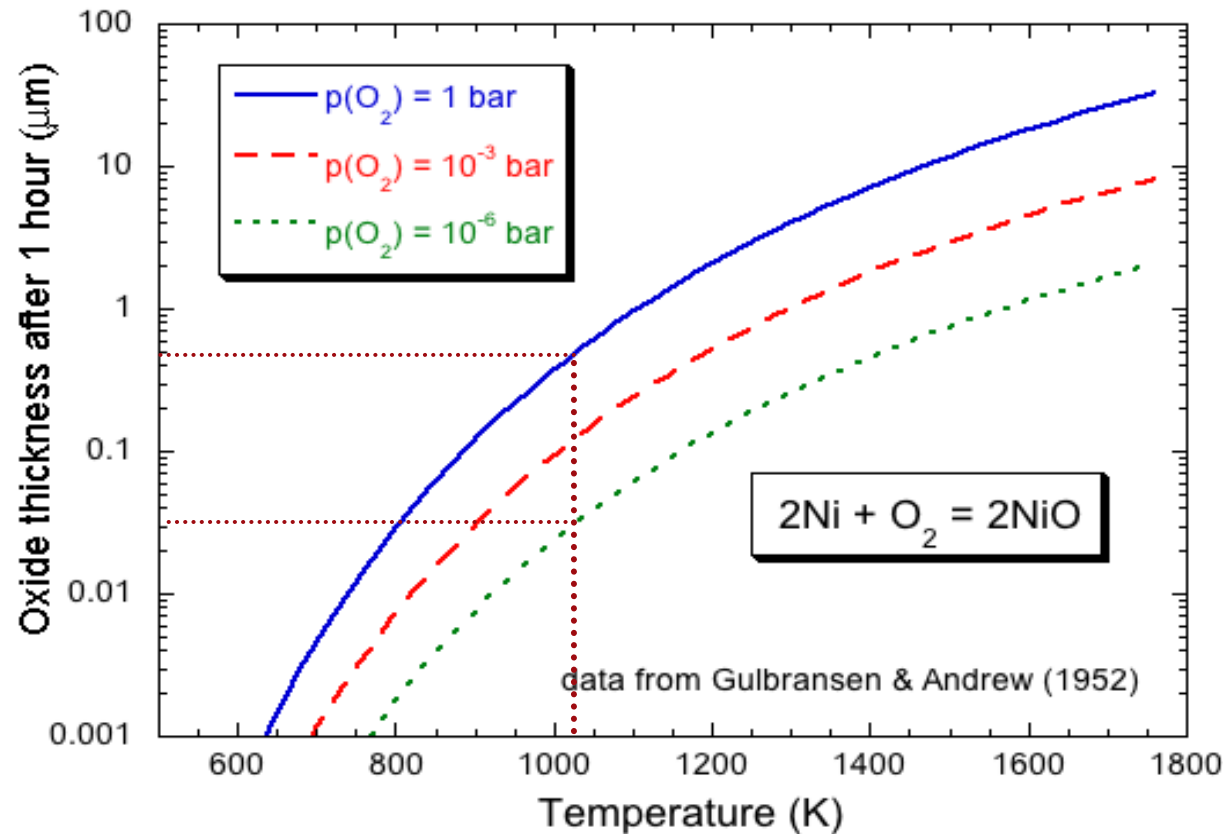
Samarium: 1 hour after polishing in air.

Sample Oxidation: Theory

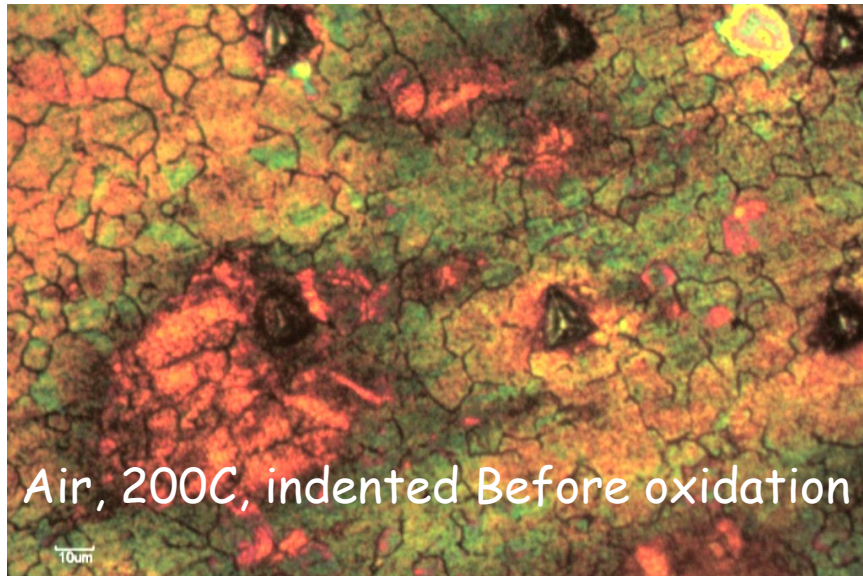
- At elevated temperatures, oxidation rates increase.
- Nickel Oxide growth rates exhibit parabolic kinetics,

$$x \propto \sqrt{t}$$

- After 1 hour at 750°C, oxide thickness:
 - ~450nm at 1 bar of O₂
 - ~30nm at 10⁻⁶ bar of O₂
- High vacuum allows nano-micro scale mechanical testing at elevated temperatures with minimal interference from surface oxide layer and prevents oxidative erosion of small features of interest.

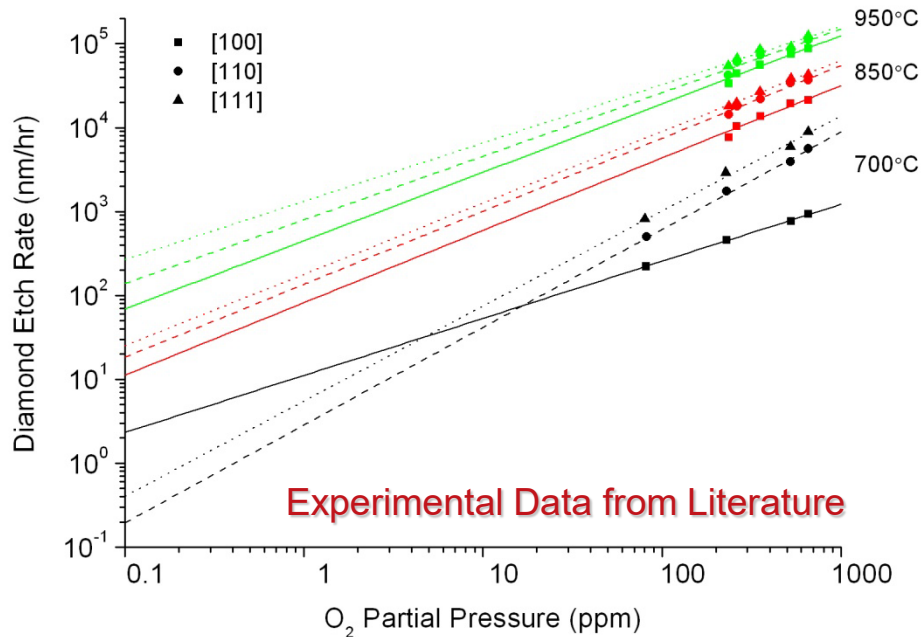


Sample Oxidation: Prevention

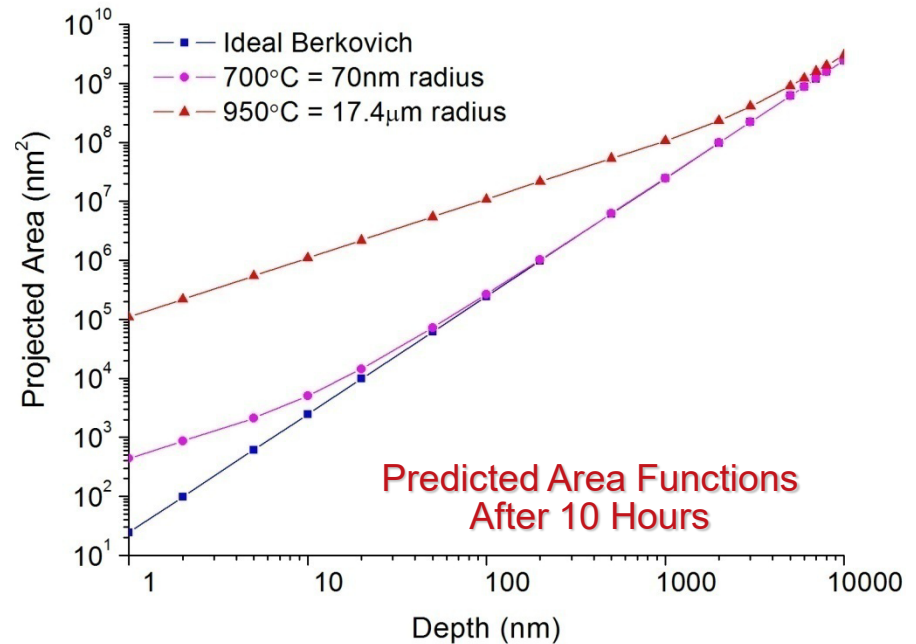


- Copper indented in Air at 200C oxidised rapidly within 12 hours.
- Indents made prior to oxidation show some preferential oxidation.
- Indents performed in High Vacuum show no visible evidence of oxidation. Any oxide is too thin for optical interference colors to form.

Tip Oxidation: Theory

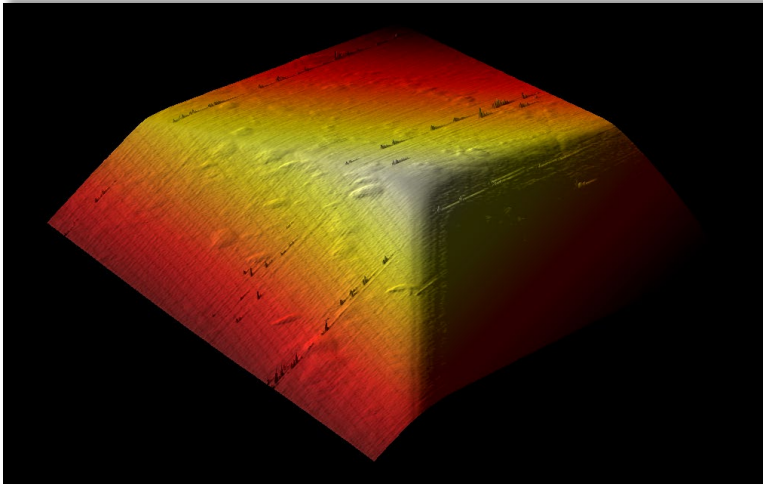


- Literature data (Evans and Phaal, 1962) taken under flowing O_2 at varying pressure and temperature.
- Extrapolated mean diamond etch rates between low angle crystal faces :
 - 0.5nm/hr at 700°C.
 - 150nm/hr at 950 °C.

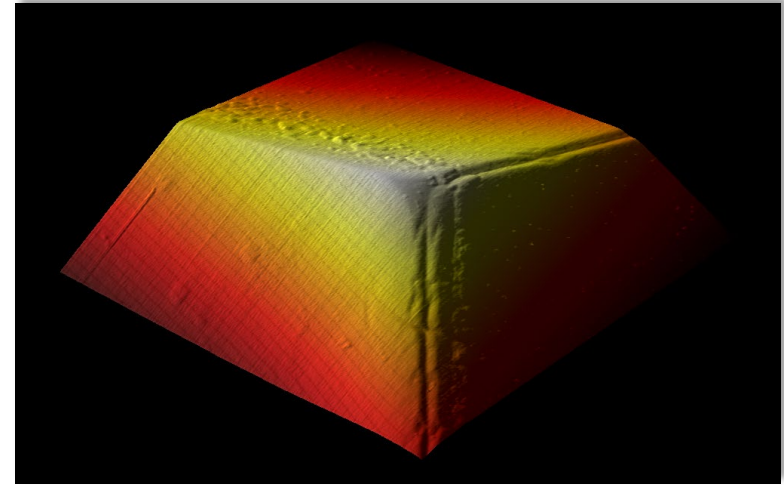


- Area functions calculated by approximating tip as conospheroid with tip blunted by etch distance.
 - Blunting at 700°C is tolerable or within manufacturing limits.
 - Blunting at 950°C is excessive.

Tip Oxidation - Morphology

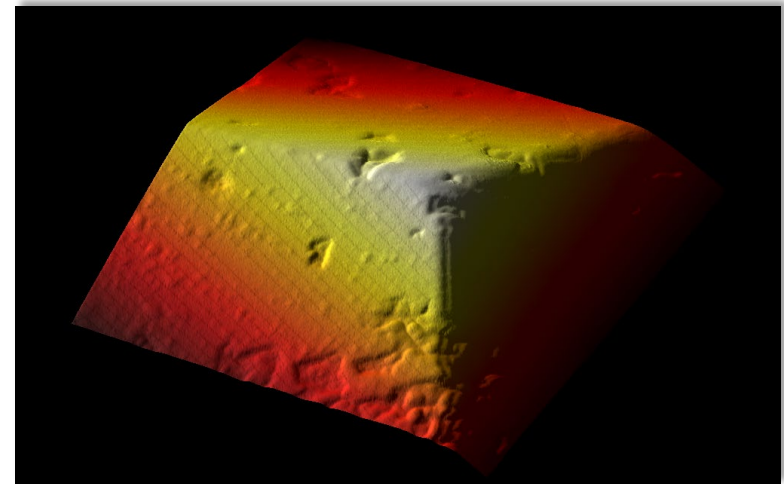


450C in Air for 30 minutes



750C in Argon (15ppm O₂) for 90 minutes

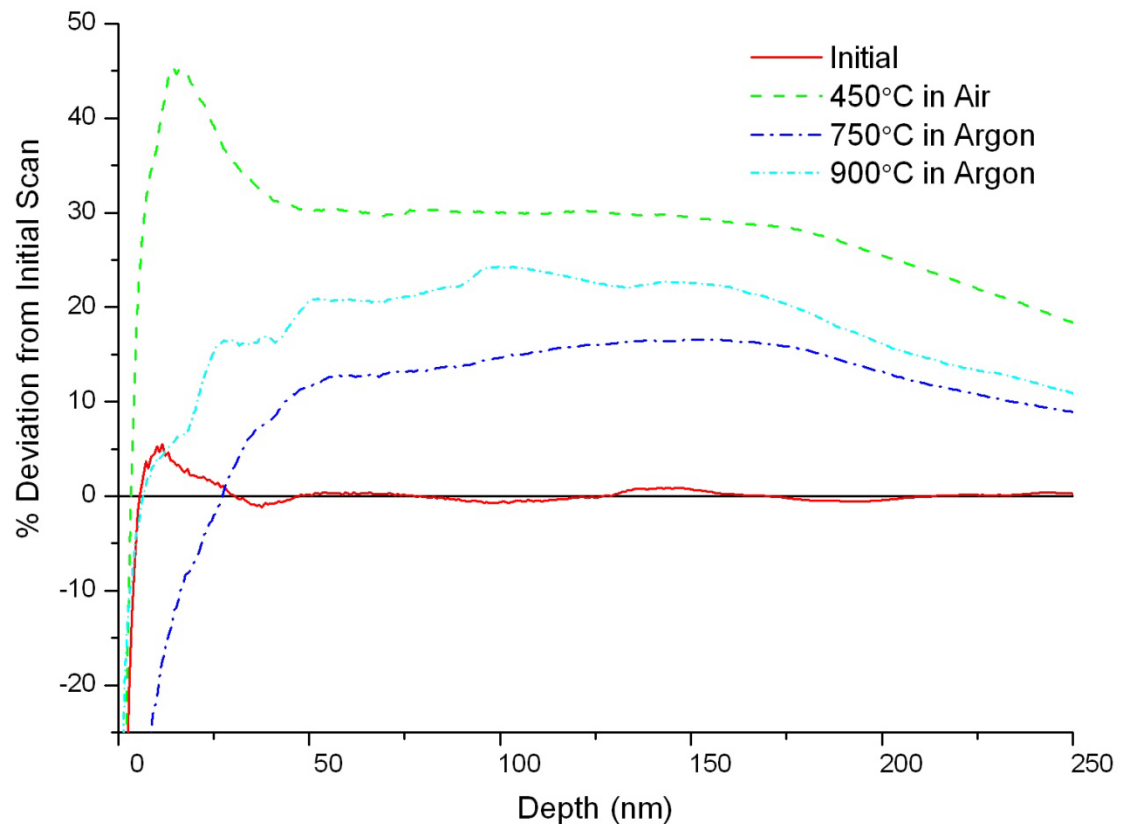
- Berkovich tip exposed to three successive atmospheres.
 - 30min in Air at 450°C.
 - 90min in Argon at 750°C.
 - 90min in Argon at 900°C.
- Faces get smoother after exposure below 900°C, though edges blunt.
- Pitting is observed after 900°C exposure.



900C in Argon (15ppm O₂) for 90 minutes

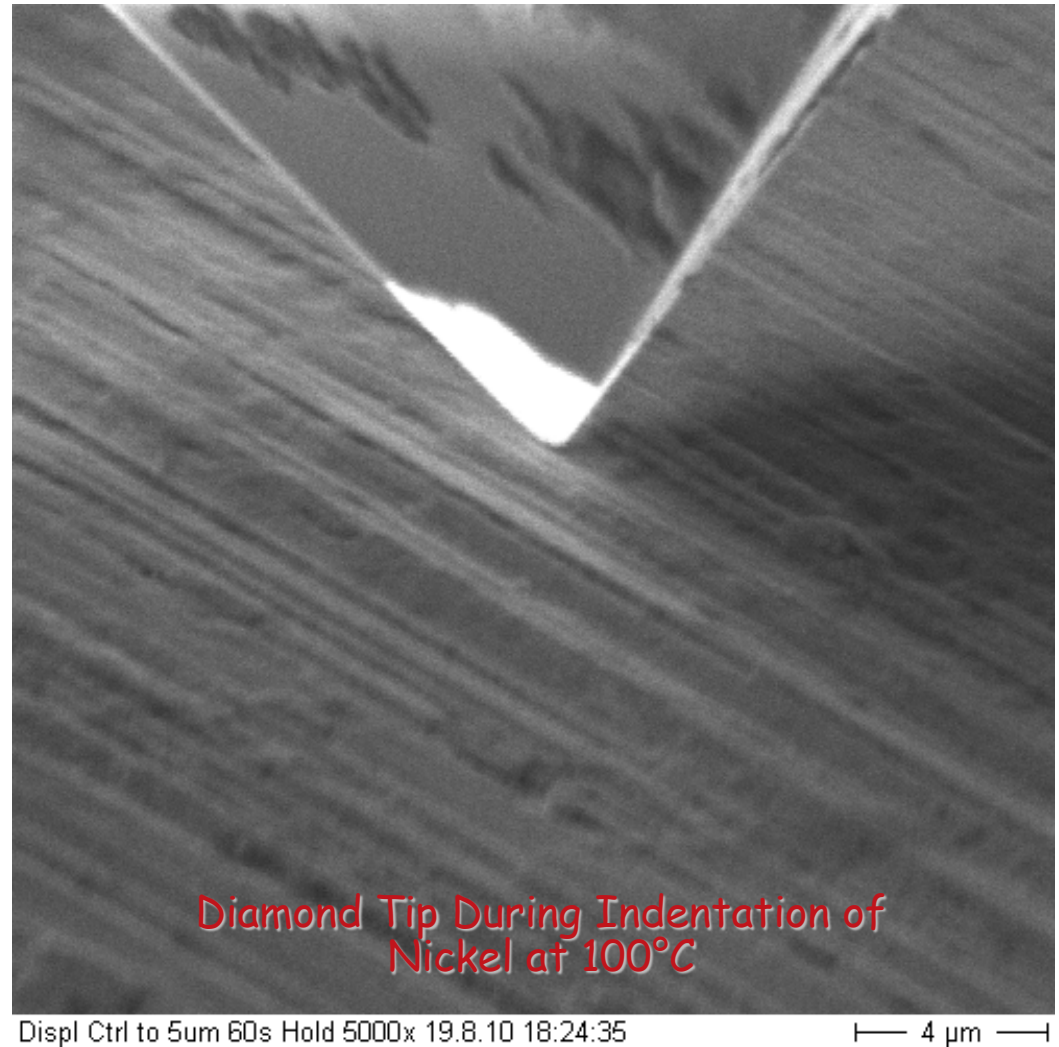
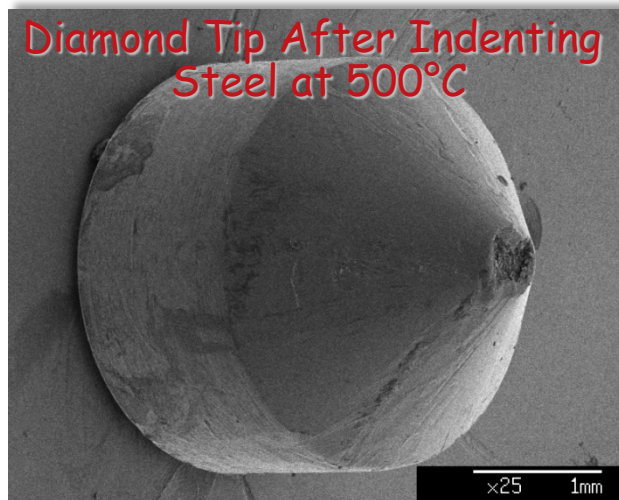
Tip Oxidation: Area Functions

- Initial blunting at intermediate temperature appears the worst.
- At elevated temperatures, the tip can appear to become sharper due to edge grooving, but this is not real sharpening.

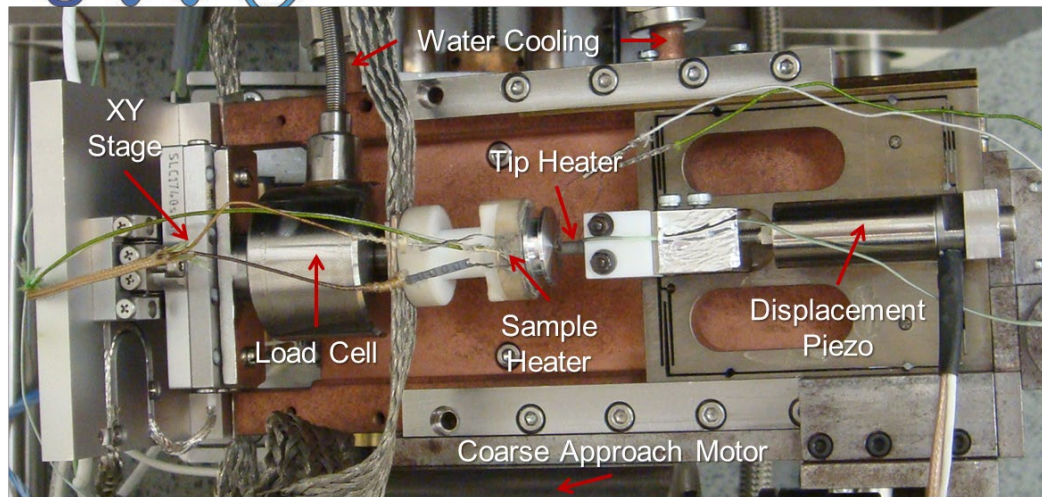
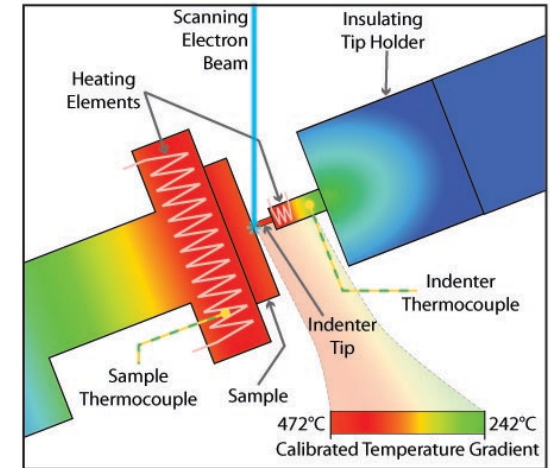
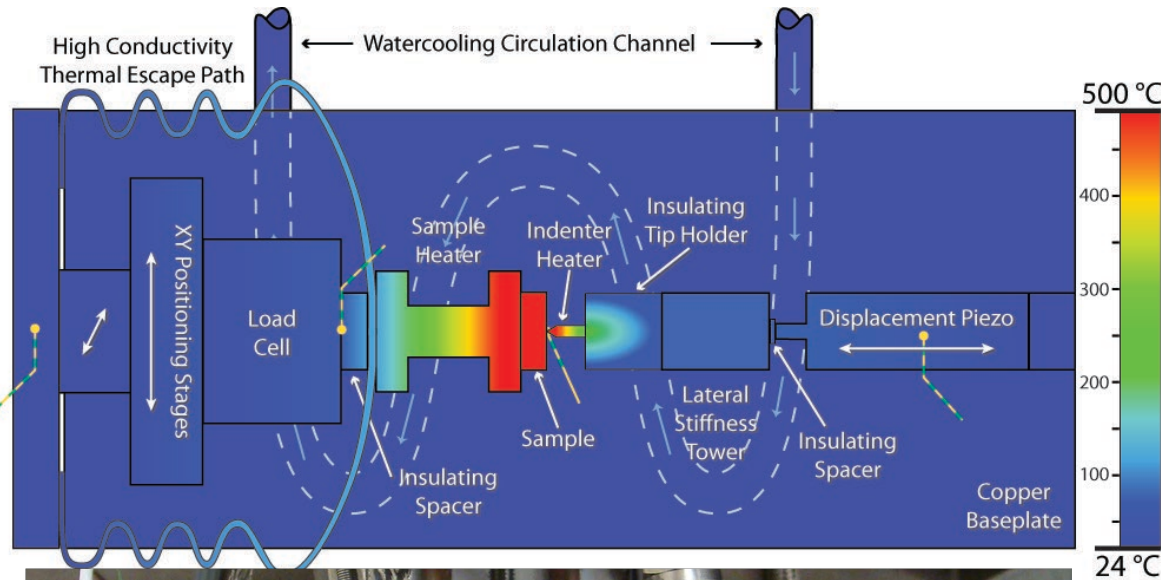


Change in Diamond Area Functions after Oxidation

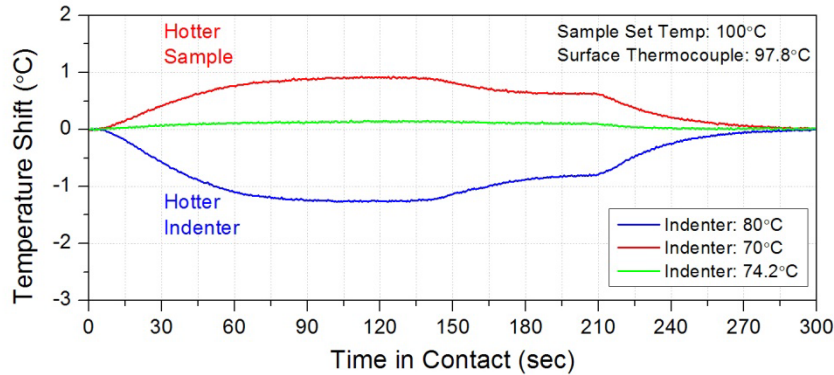
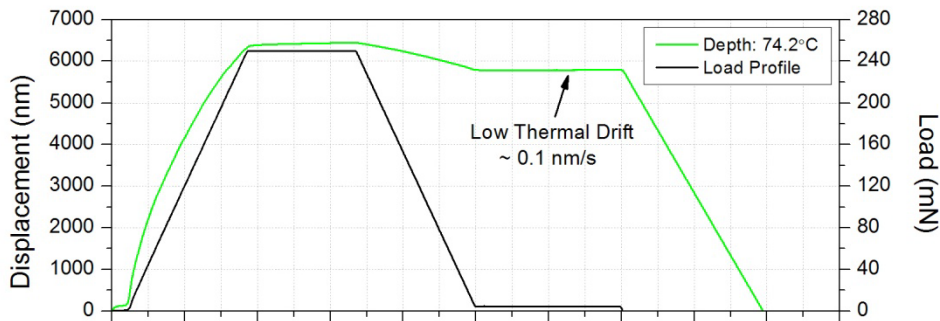
Tip-Sample Reactions



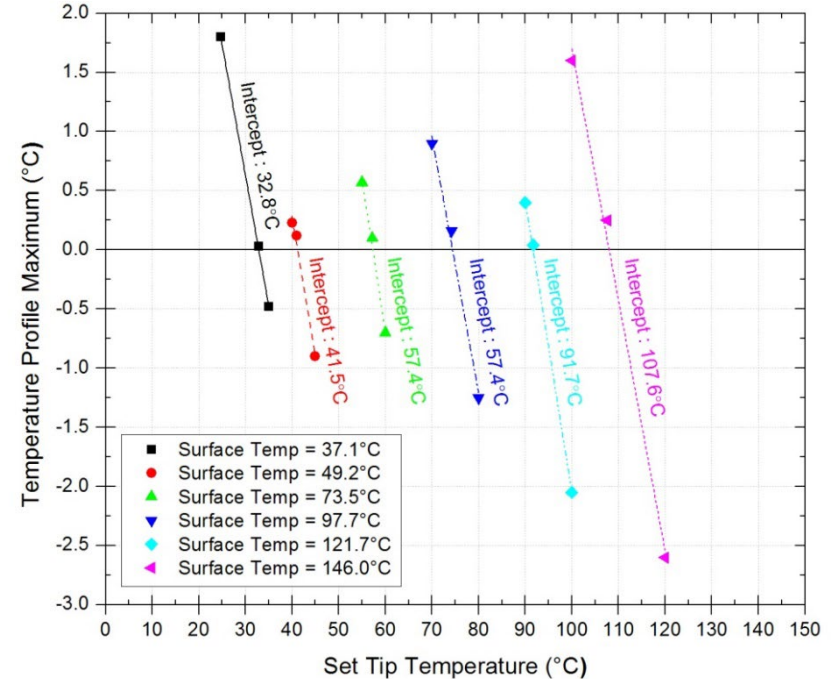
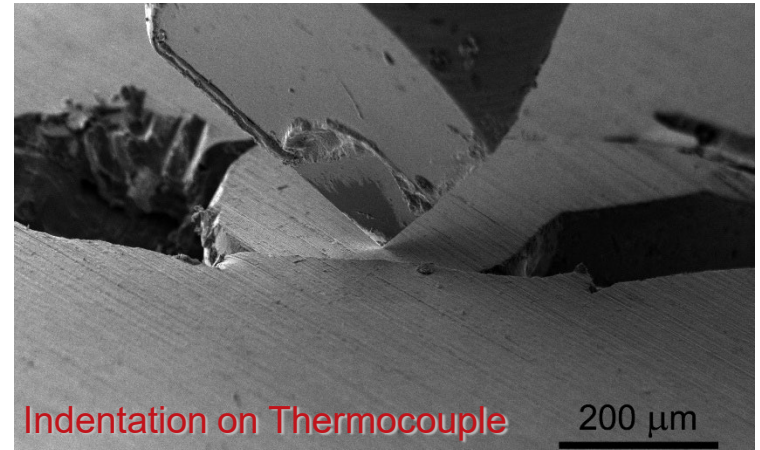
in-situ SEM high temperature system



indenter temperature tuning



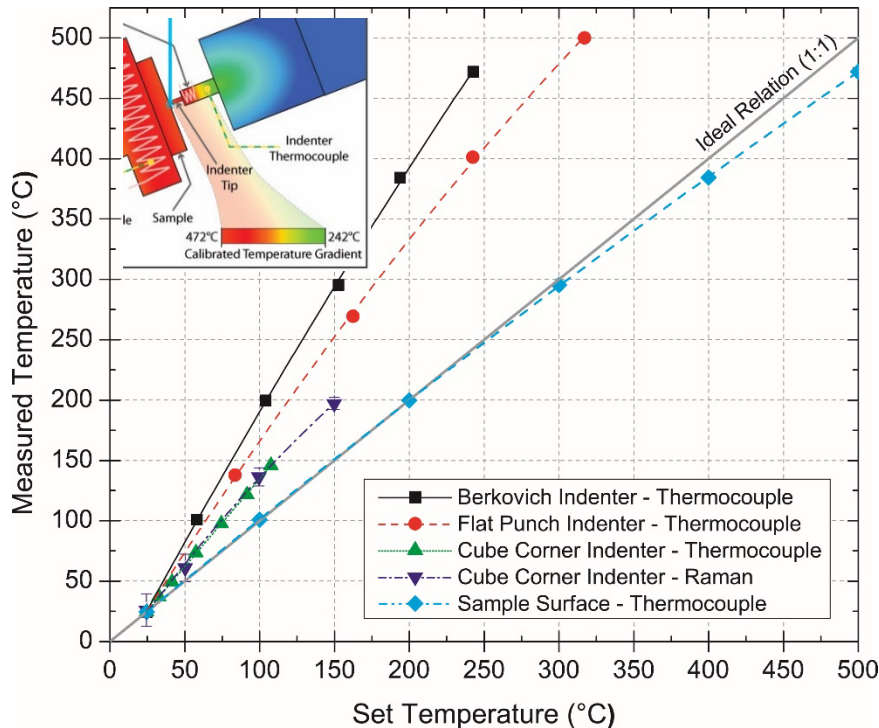
*Displacement vs. Temperature Change



JM Wheeler, P Brodard & J Michler,
Phil Mag, 92 (2012) 3128-3141.

temperature distribution

Indenter Temperature Calibration Curves

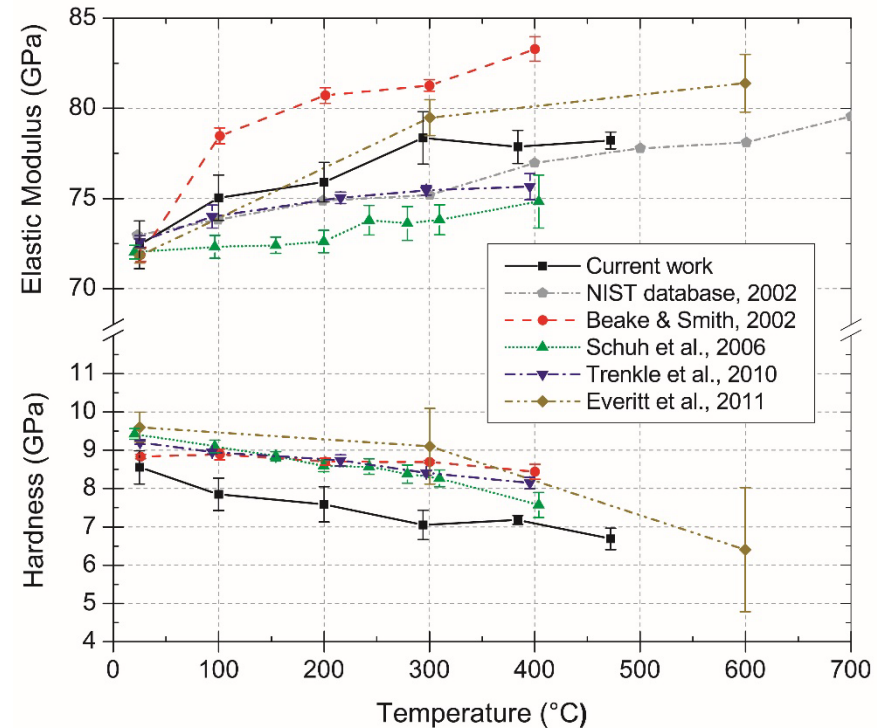
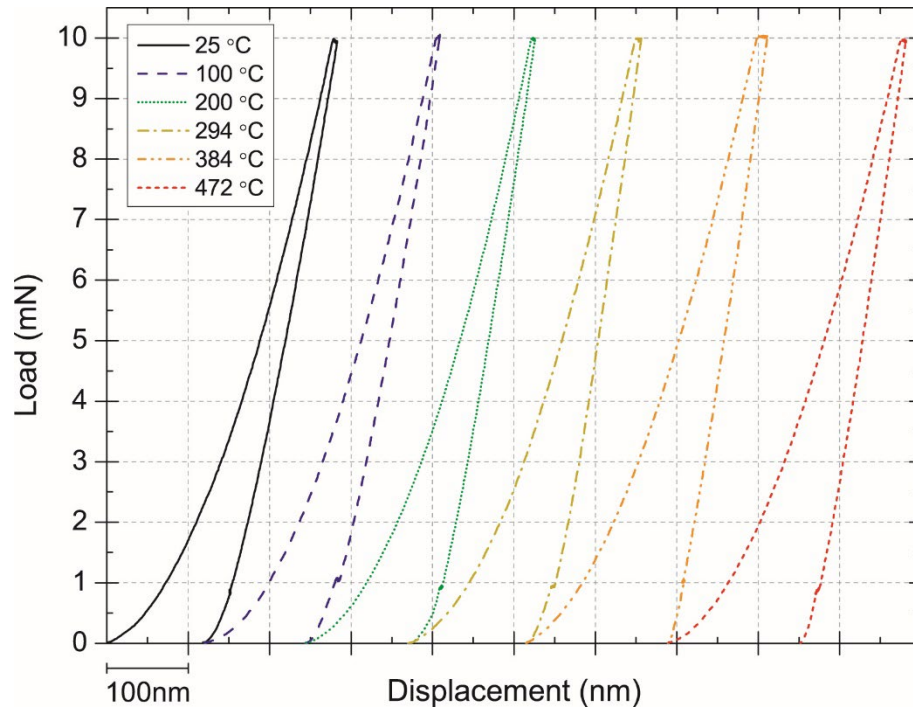


Good agreement between both Raman and thermocouple indentation measurements.

Larger value expected, since heating element is closer to the indenter than the indenter's control thermocouple in this tip design

Allows much greater temperature accuracy in elevated temperature indentation.

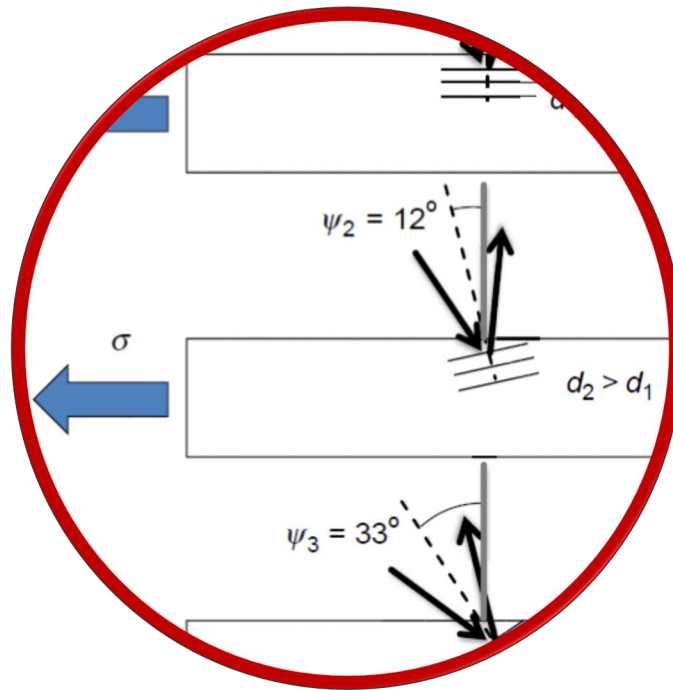
nanoindentation of fused silica



Load-displacement curves are shown uncorrected for drift, with a 60s hold period. All thermal drift is < 0.05 nm/s.

Good agreement is observed between elastic modulus results on fused silica of the current work

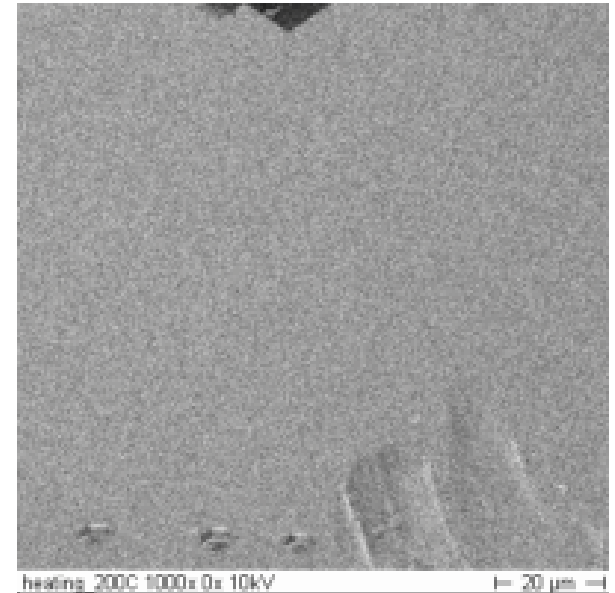
Cryogenic temperature testing



Challenges in low temperature indentation

➤ Thermal drift

- **Frame drift** – displacements due to thermal contraction within the indenter frame components
- **Contact drift** – displacements due to thermal expansion/contraction in sample and tip due to heat flow from temperature mismatch
- **Electronics drift** – Change in electronic signals with time or temperature
- Control electronics and sensors must be protected from cryogenic temperatures

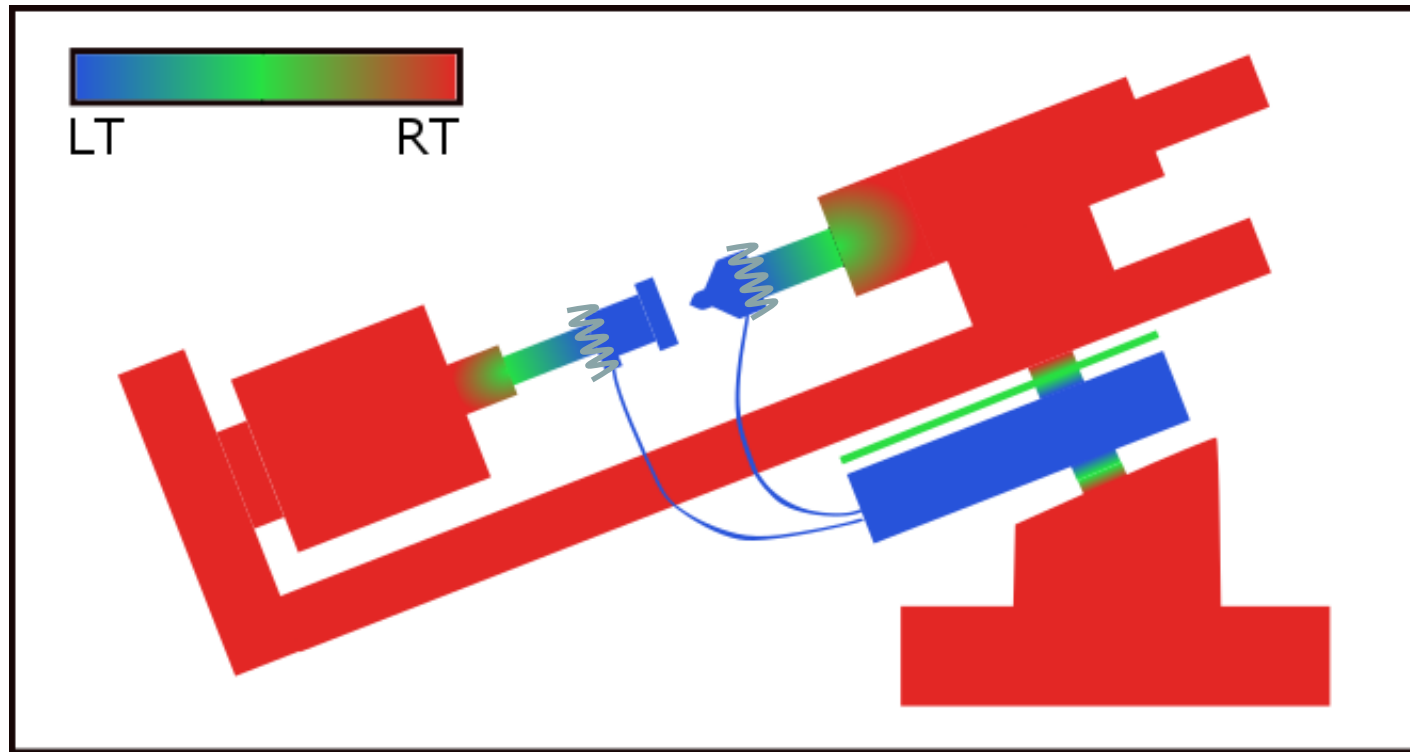


Thermal drift for ΔT of 200K
Courtesy of J.M. Wheeler

➤ Condensation

- **Condensation** on the sample surface is undesirable for surface property testing

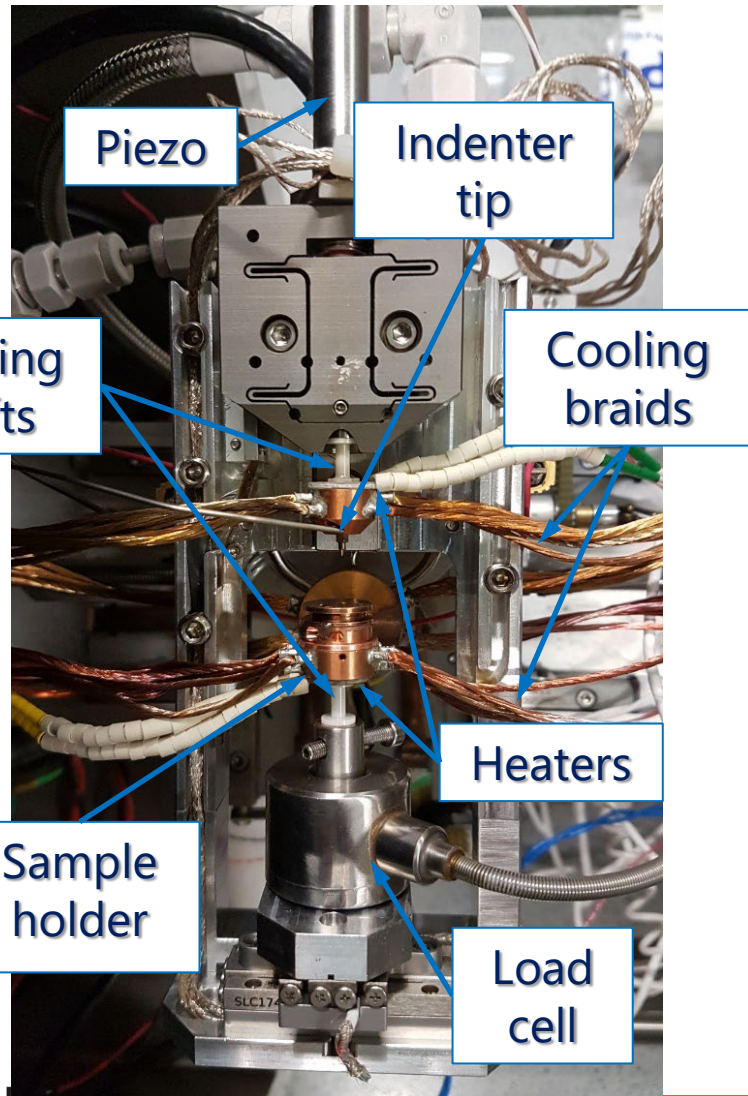
Thermal management concept



Local cooling of nano-contact:

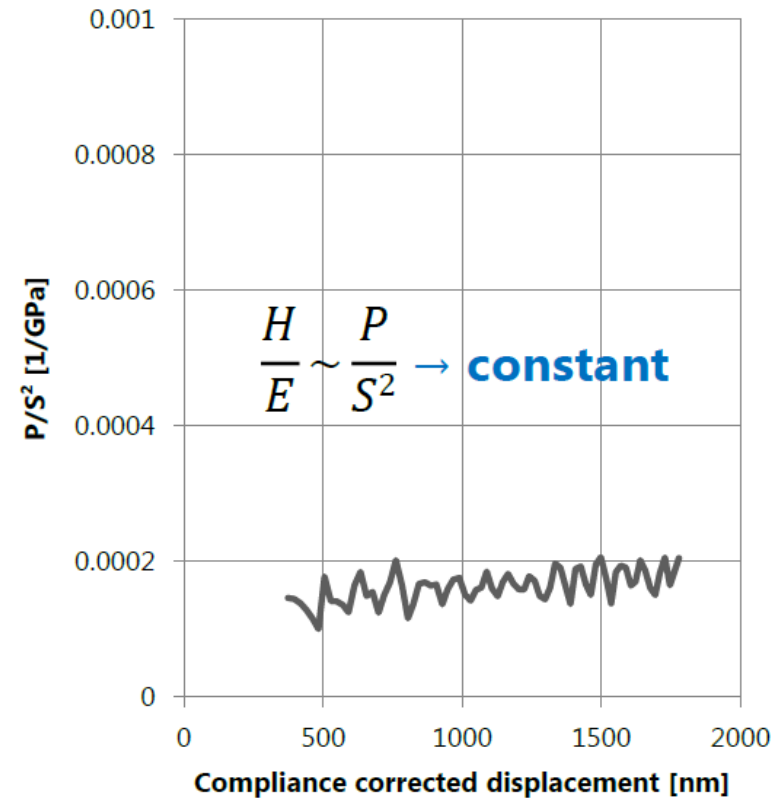
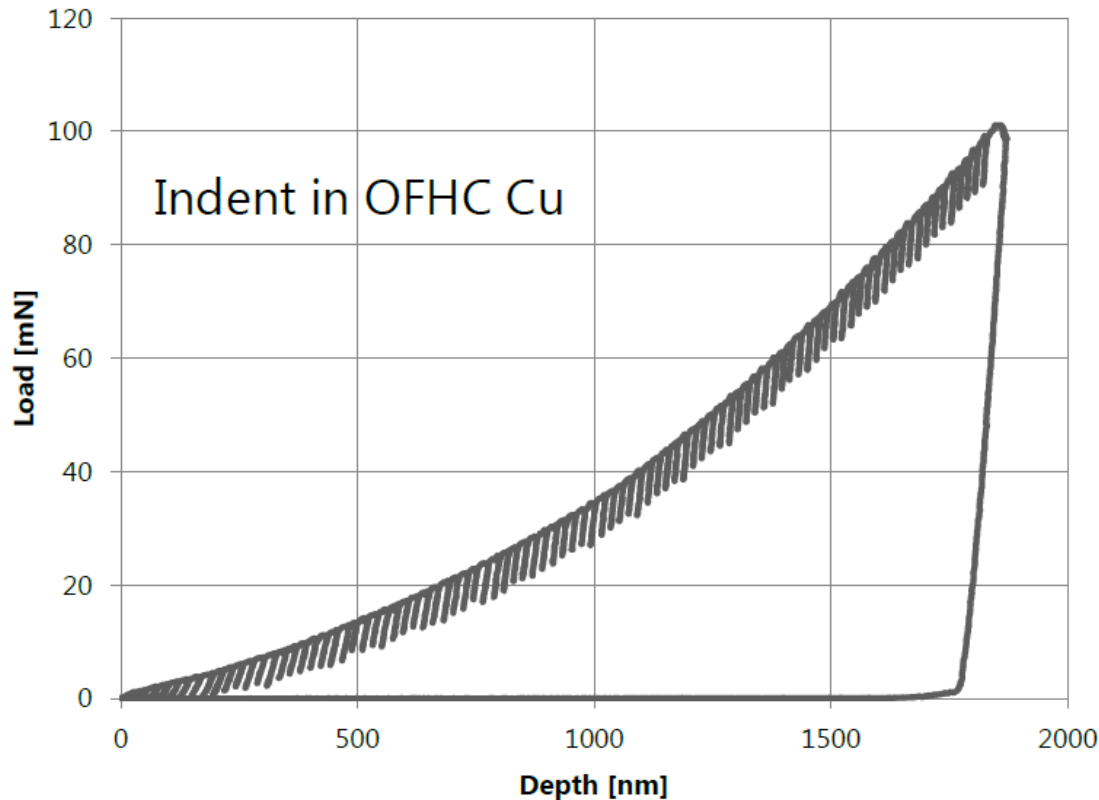
- Cold finger linked by conductive braids to sample and tip: stable base temperature.
- Miniaturized heaters for sample and indenter for independent temperature tuning.
- Thermal isolation and radiation shielding of the cooled parts to reduce frame drift and protects sensitive electronics.

In situ quasistatic cryoindenter setup



- Temperature range: 120K to 300K
- Strain rate range: 10^{-4} to 10^{-1} s
- Load range: 500mN
- Nitrogen pumped through cold finger cooling indenter tip and sample holder to a base temperature of 120K
- Thermocouples and heaters on tip and sample holders allow independent temperature control
- Isolating shafts and heaters in closed loop control keep the frame at constant temperature

Frame compliance measurement



- Room temperature drift < 5 nm/min
- Frame compliance: 4.85nm/mN (14% increase compared to standard setup)

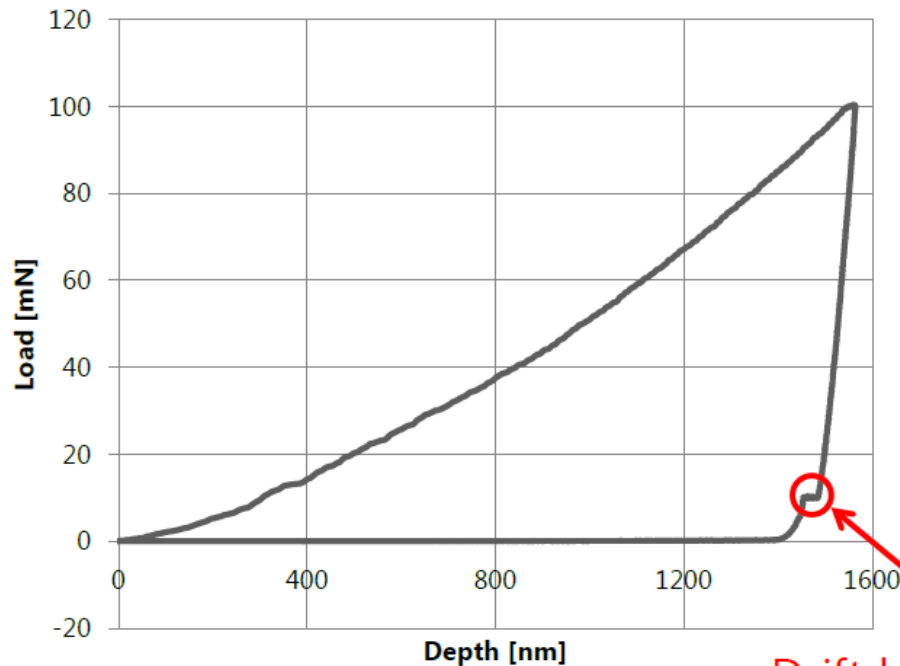
Hardness: 1.2 GPa

Young's modulus: 115 GPa

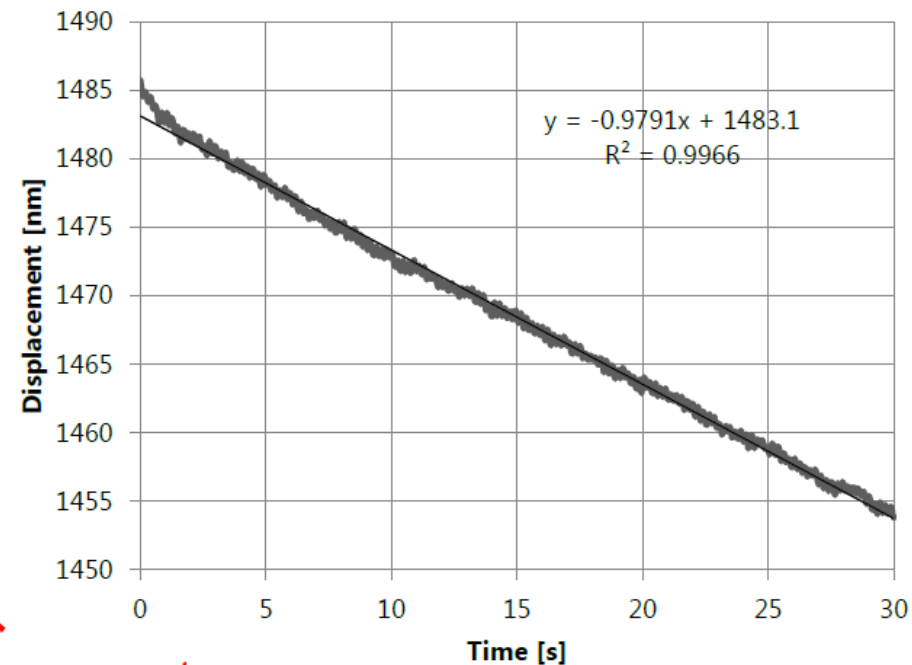


Compares well with literature [Schuh2006]

Thermal drift measurement



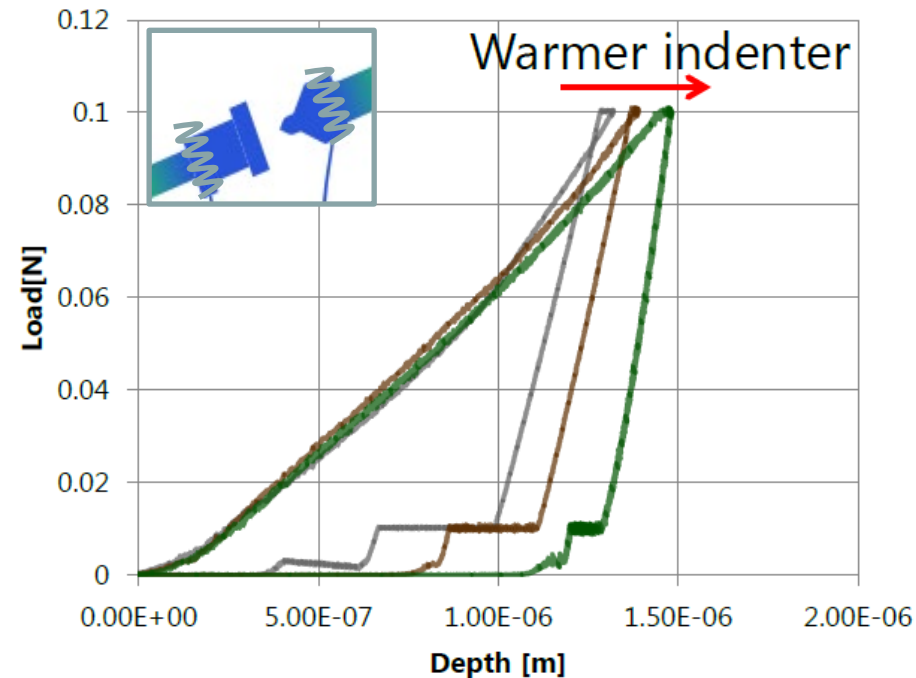
Drift hold segment



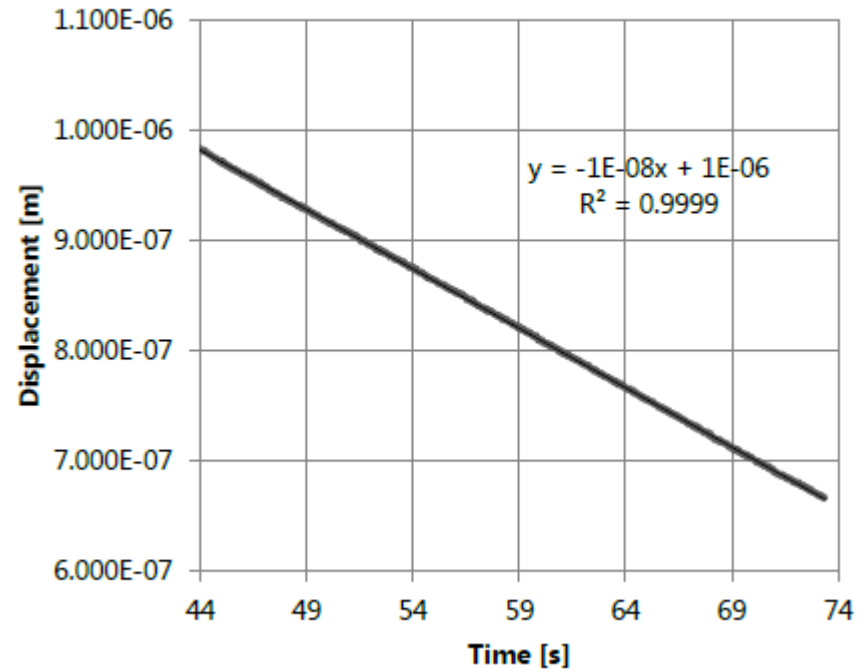
- Thermal drift measured experimentally by recording the change in depth with time at 10% of peak load during unloading.
- As the applied load is very low and the material is not expected to creep during unloading, the displacement is assumed to occur due to thermal drift.
- After 3-4 hours of stabilization, a drift of <60nm/min is reached

Drift tuning and correction at -150°C

Raw data



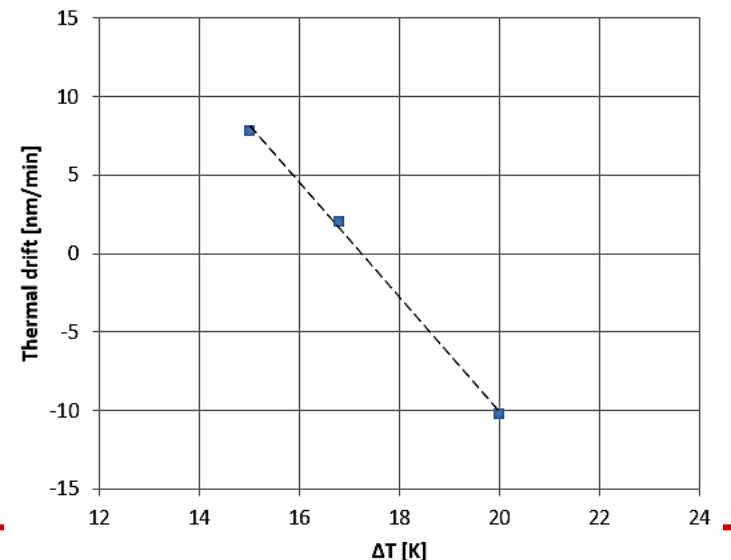
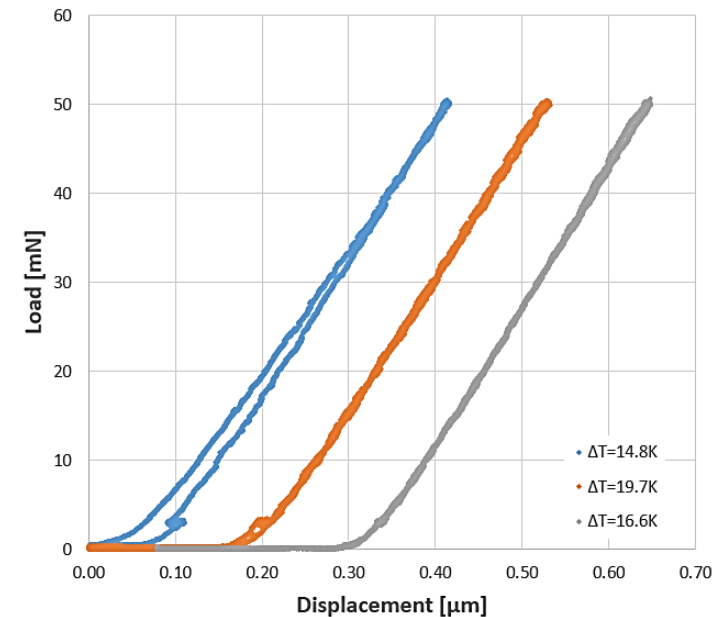
Drift measurement



- By changing the indenter or sample temperature, the drift can be tuned
- Linear drift → drift correction possible

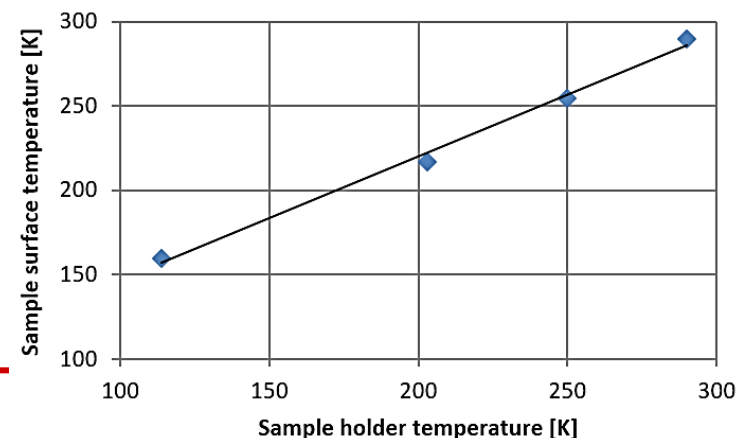
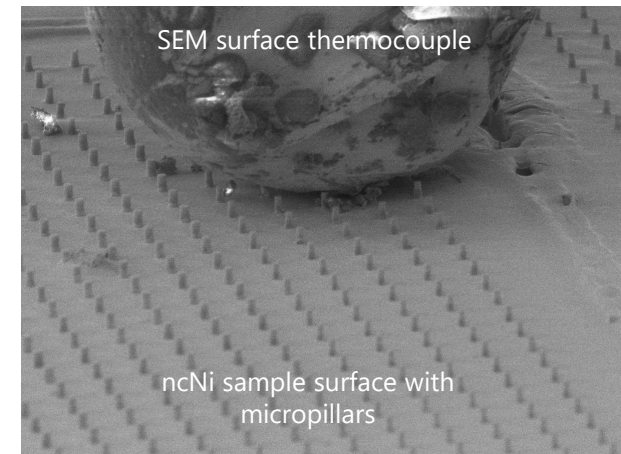
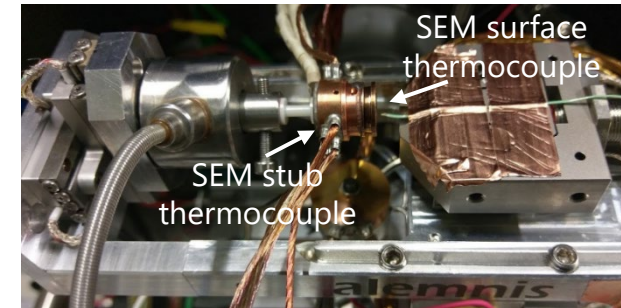
Temperature matching for drift minimization

- Indentations are performed with a 2 minute drift measurement at 5% of the maximum load
- Measurements are performed at different ΔT
- Thermal drift as low as 2 nm/min can be reached

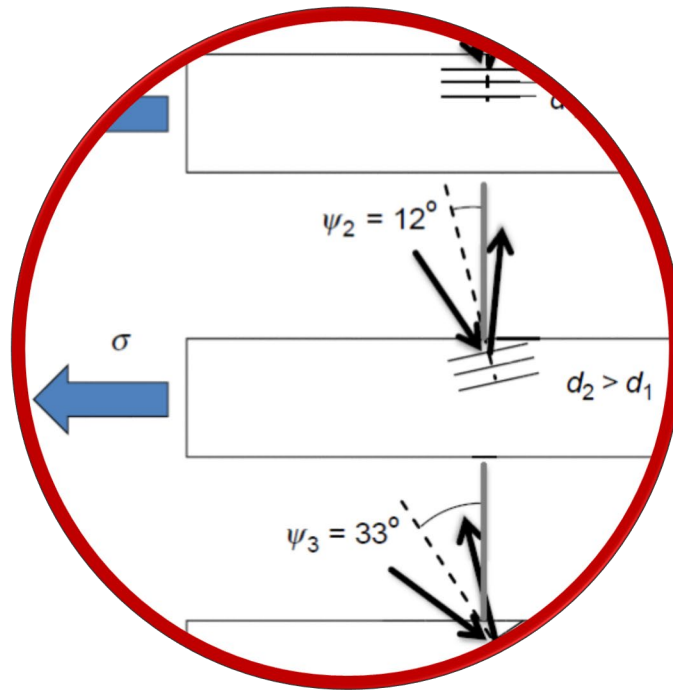


Temperature at the nanocontact

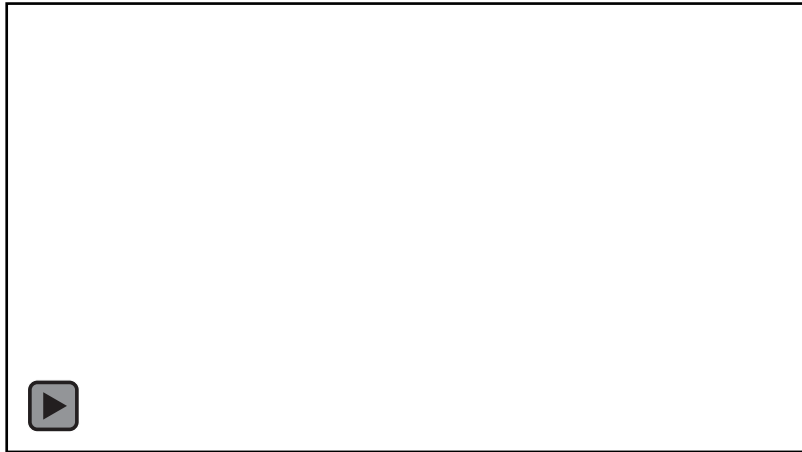
- Replaced tip by a thermocouple
- Direct measurement of sample surface temperature
- Determine calibration curve to sample holder temperature
- Plotting the tip temperature for which minimum drift was observed vs. sample surface temperature allows calibrating the tip temperature



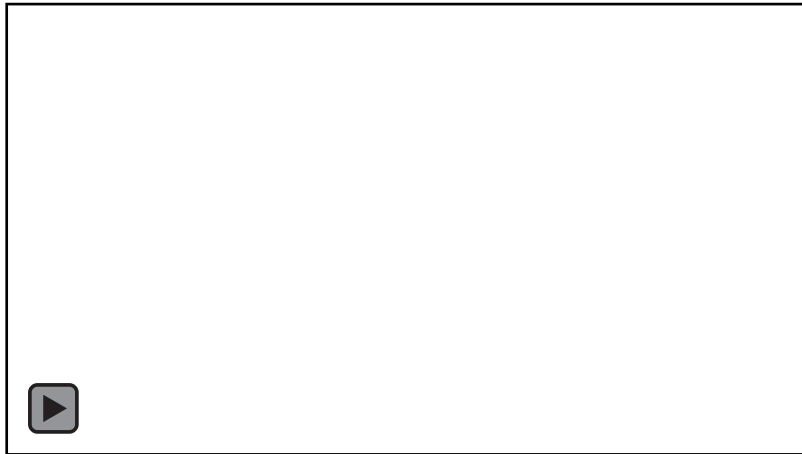
High strain rate testing



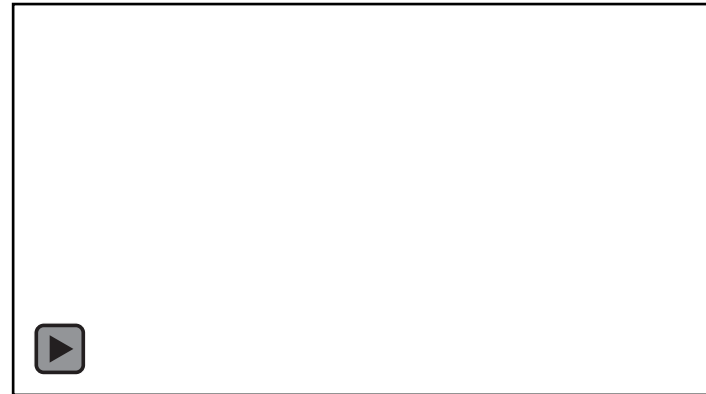
high strain rate (HSR) testing - MOTIVATION



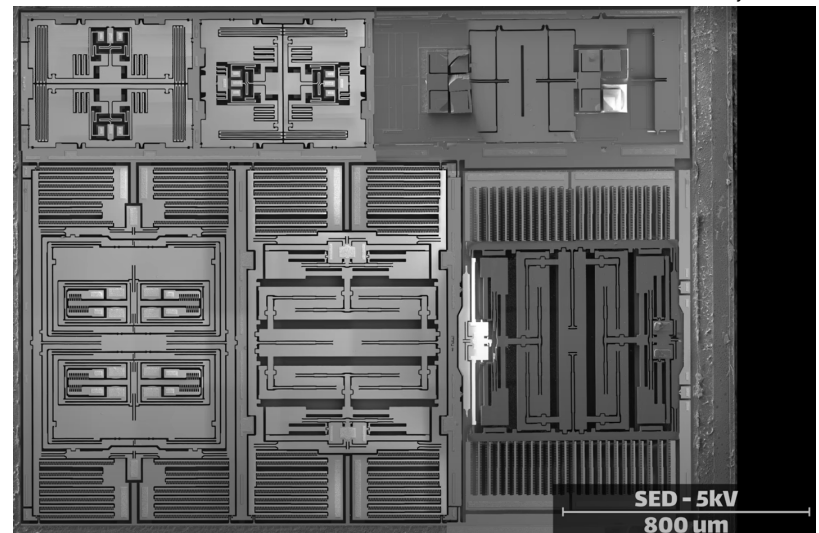
Courtesy: Animal planet



Courtesy: Spinlaunch

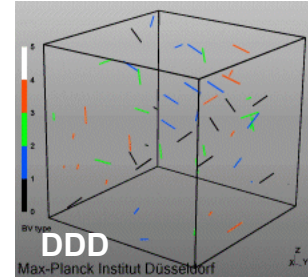
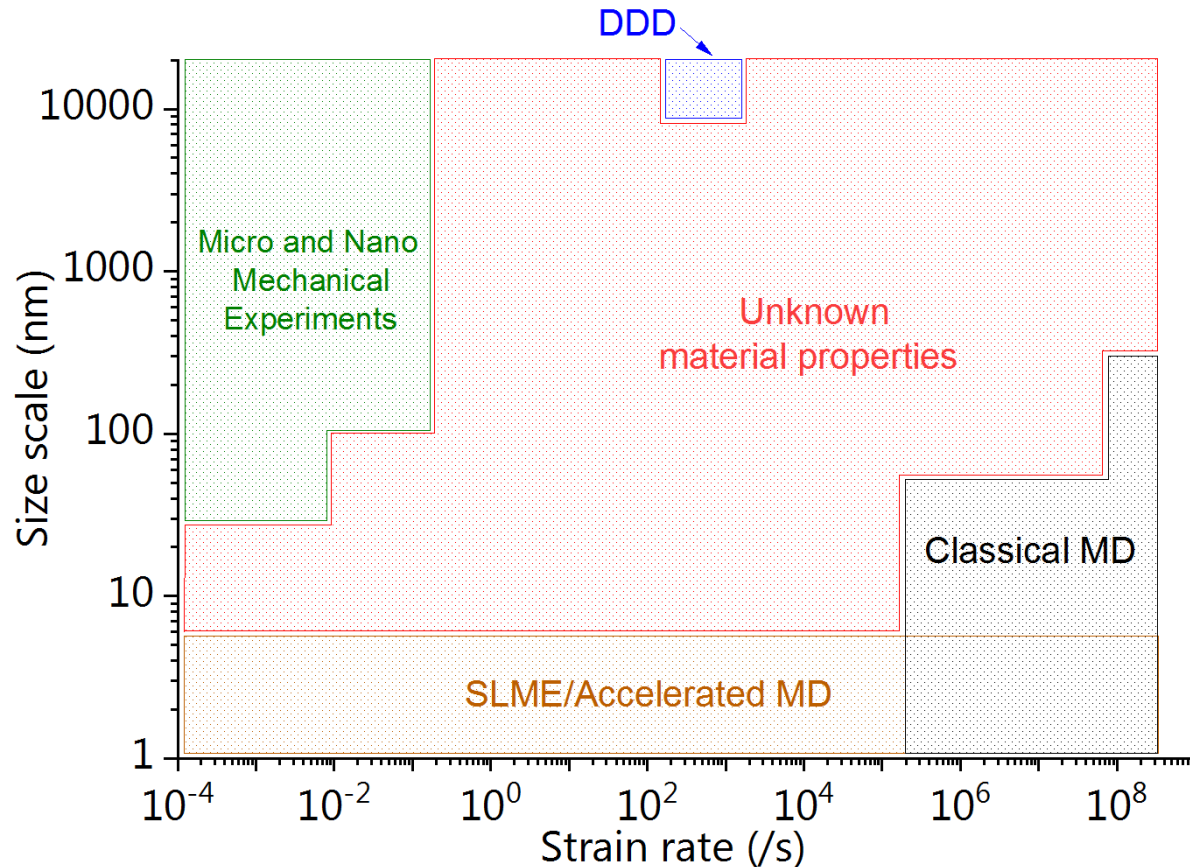
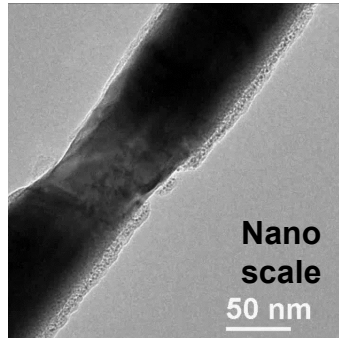
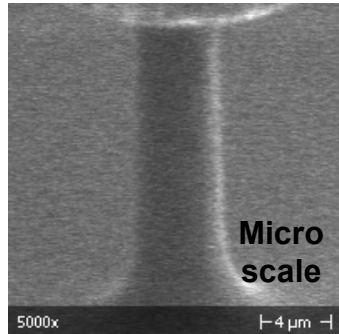


Courtesy: Bosch



Courtesy: Zachary Tong

Why micromechanics at HSR?



High strain rate experiments help bridge the gap between experiments and simulations

Historical perspective - high strain rate testing

Macroscale HSR testing



Split-Hopkinson pressure bar



Taylor impact testing

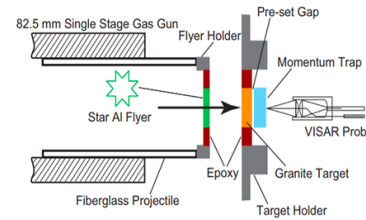
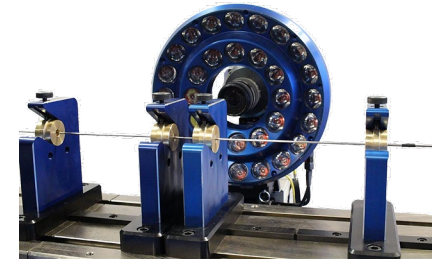


Plate impact testing



Miniaturized SHPB

Timeline

1949

1968

1980

Sample size 10-40mm

Sample size 1-5mm

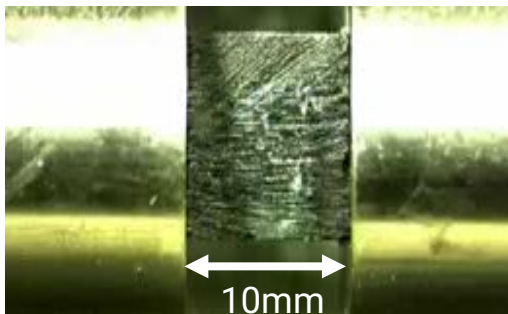
Strain rate =

1000/s

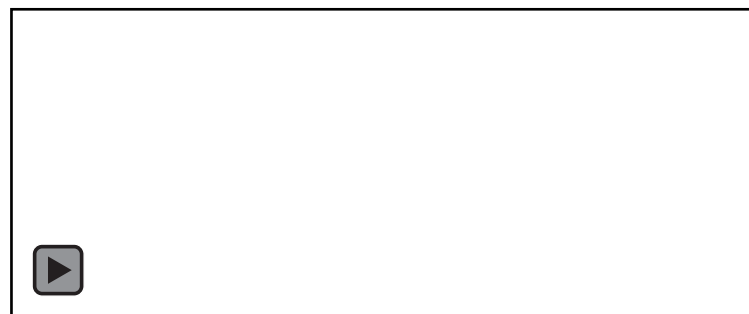
10000/s

> 100,000/s

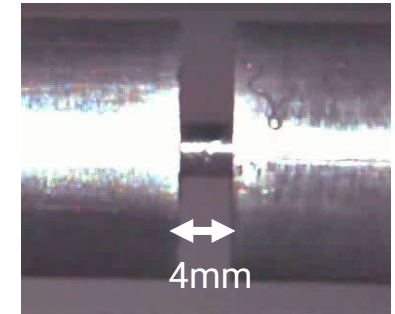
Speed/Gage length



Courtesy: REL inc



<https://youtu.be/vsLrII9OqqQ>

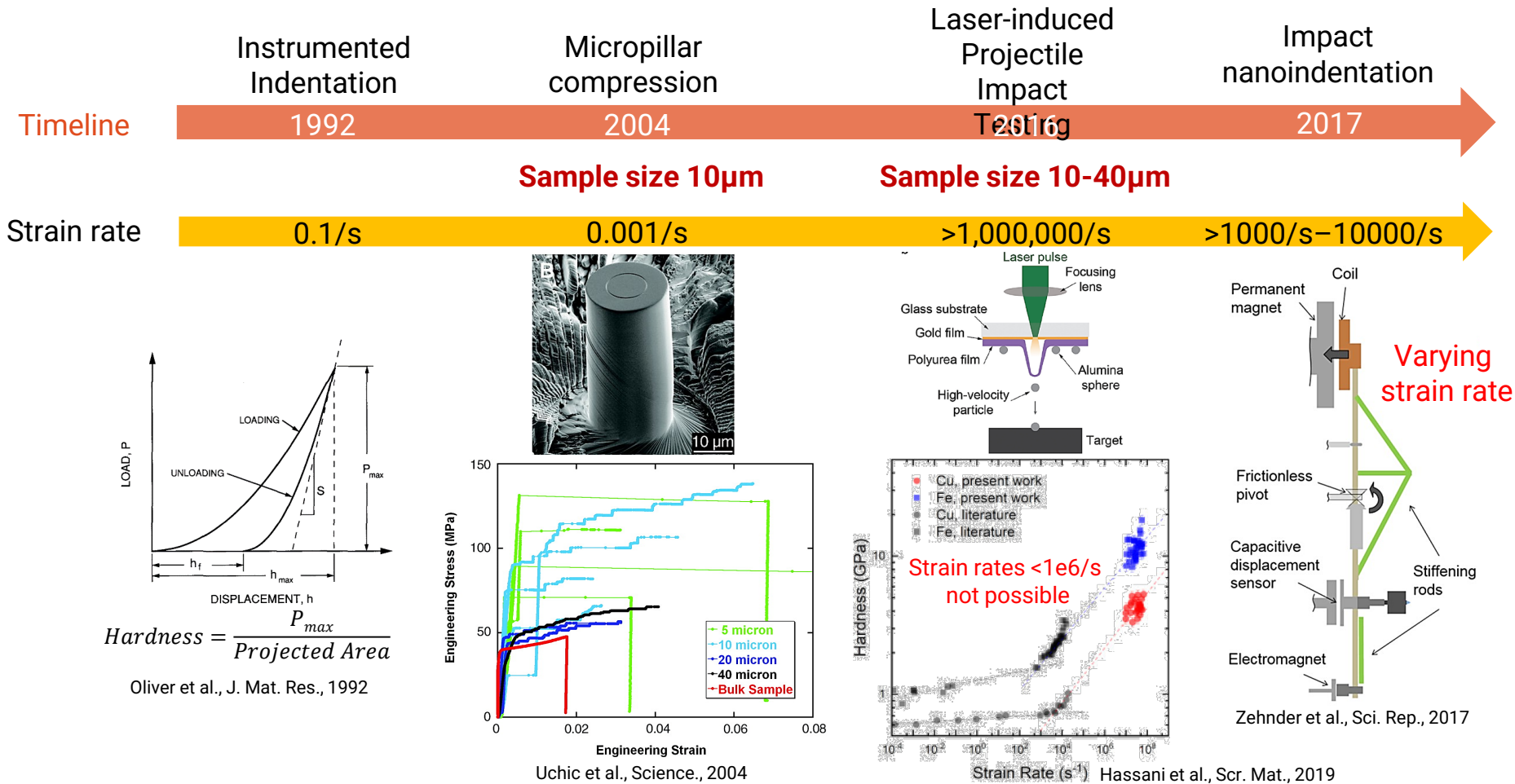


Courtesy: REL inc

- Speed of testing – **km/s** to achieve HSR
- Significant material wave propagation effects
 - **Experimental timescale $\sim$ stress equilibration time**
- Macroscale samples – Easy to fabricate
 - Polycrystalline
 - Flaws/defects
- **Prominent adiabatic heating** in samples
- Used with continuum finite element modeling (FEM)

- Speed of testing – **mm/s** to achieve HSR
- Negligible material wave propagation effects
 - **Experiment timescale $\gg$ stress equilibration time**
- Microscale samples – Difficult to fabricate
 - Isolated boundaries
 - Pristine single crystals are possible
- **Adiabatic heating less likely** in samples
- Typically linked with molecular dynamics (MD) or dislocation dynamics (DDD)
 - Strain rate gap 10 orders of magnitude

Microscale testing - State-of-the-art

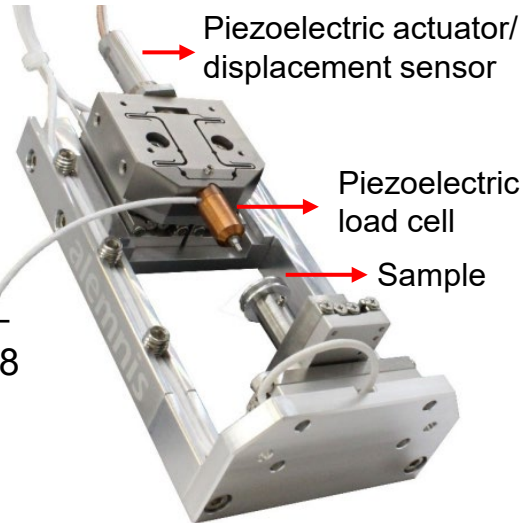


Constrain strain rate nanoindentation – Up to 100/s
Uniaxial microcompression – Up to 1000/s

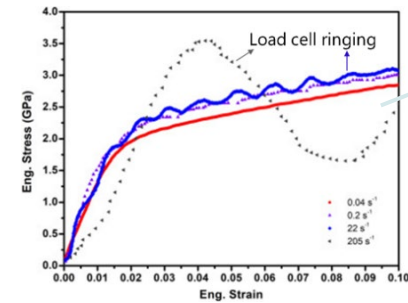
Instrumentation for high strain rates

Maximum actuation speed – 8mm/s

Strain rate range (gauge length ~4μm) – 0.0001/s to 2000/s – 8 orders of magnitude

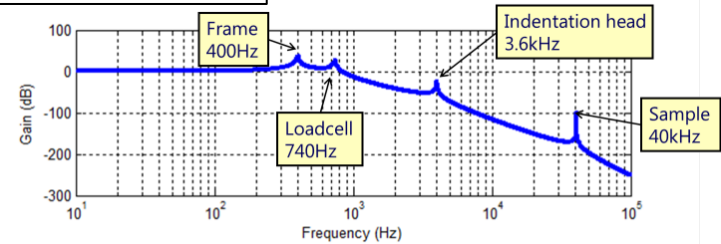


High stiffness load cell

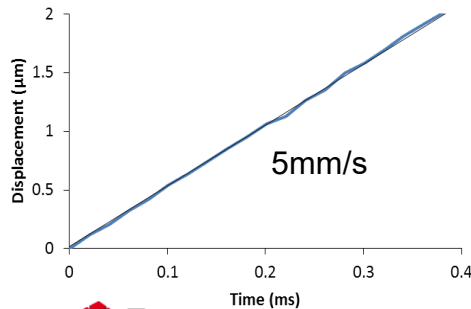


Strain gage load cell

System resonances



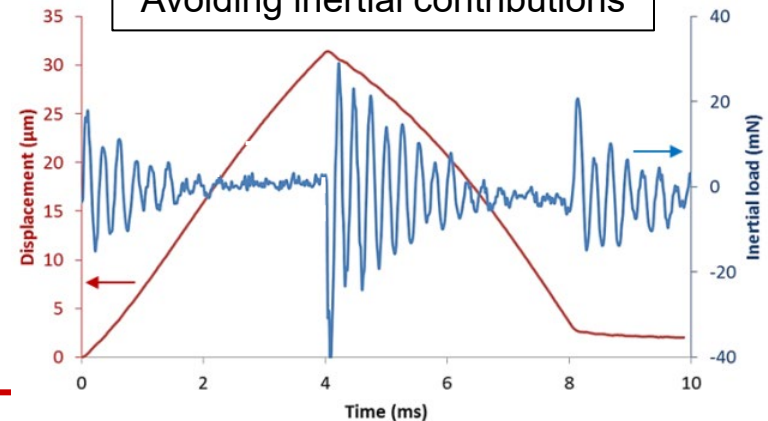
High speed actuation with accurate displacement control



High speed electronics for input and output

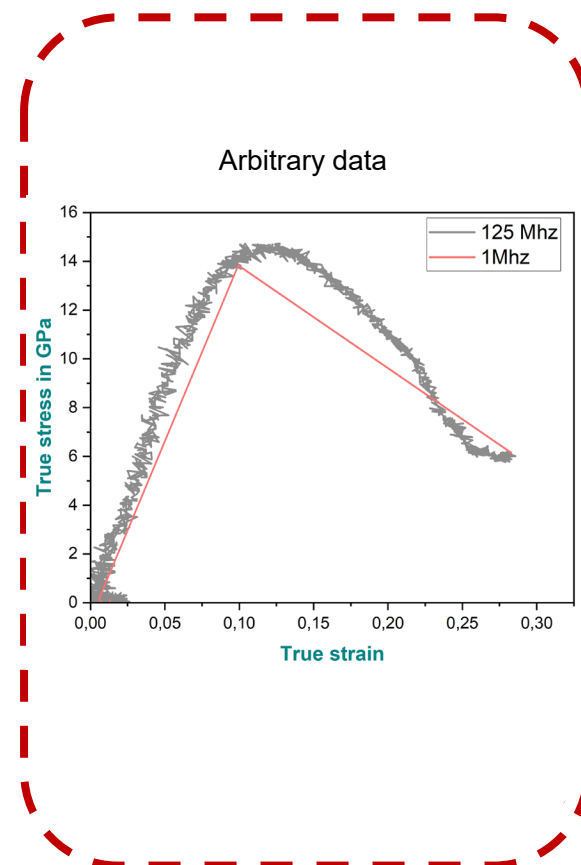
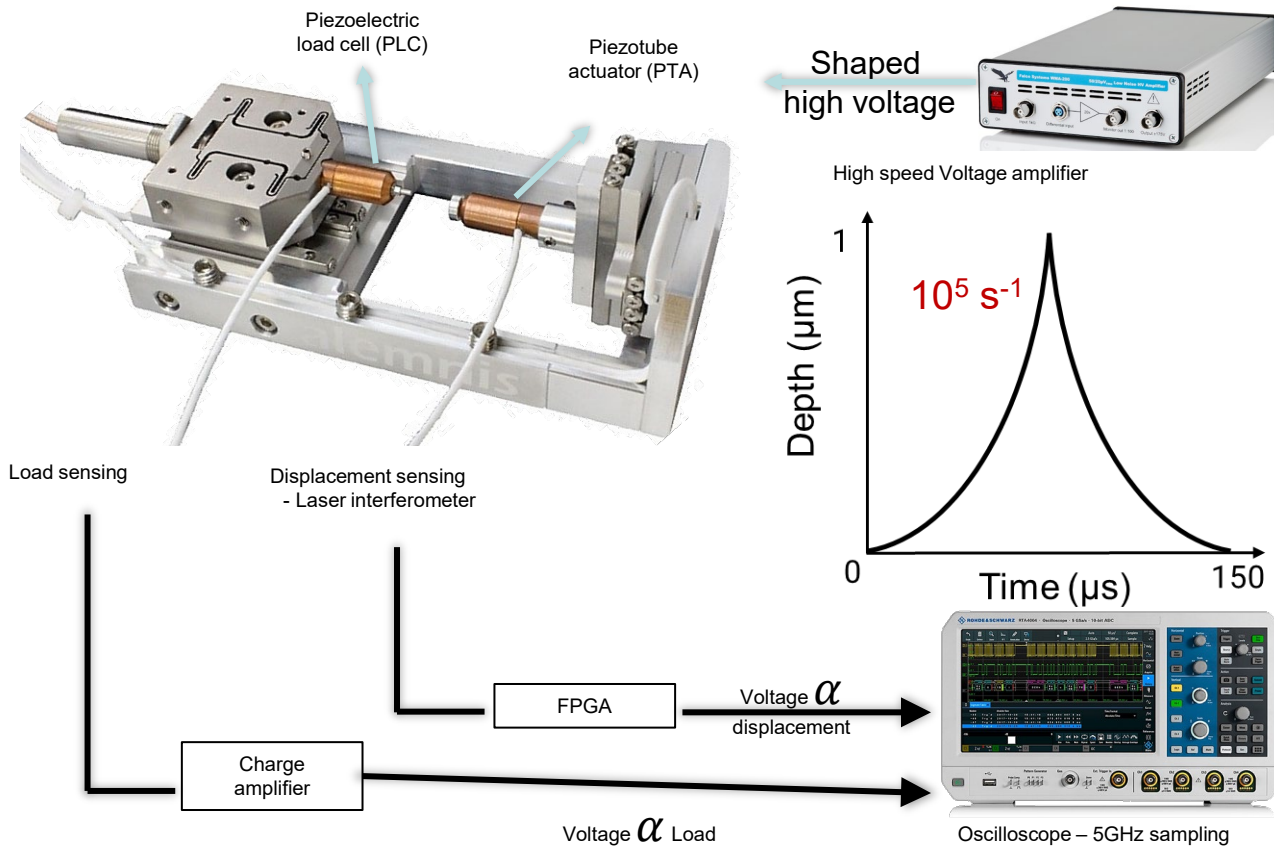


Avoiding inertial contributions



Electronics hardware

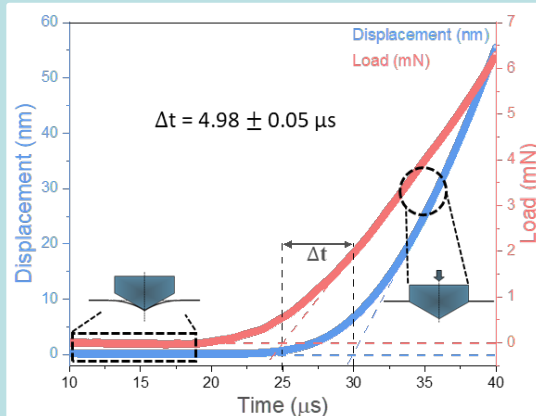
Instrumentation



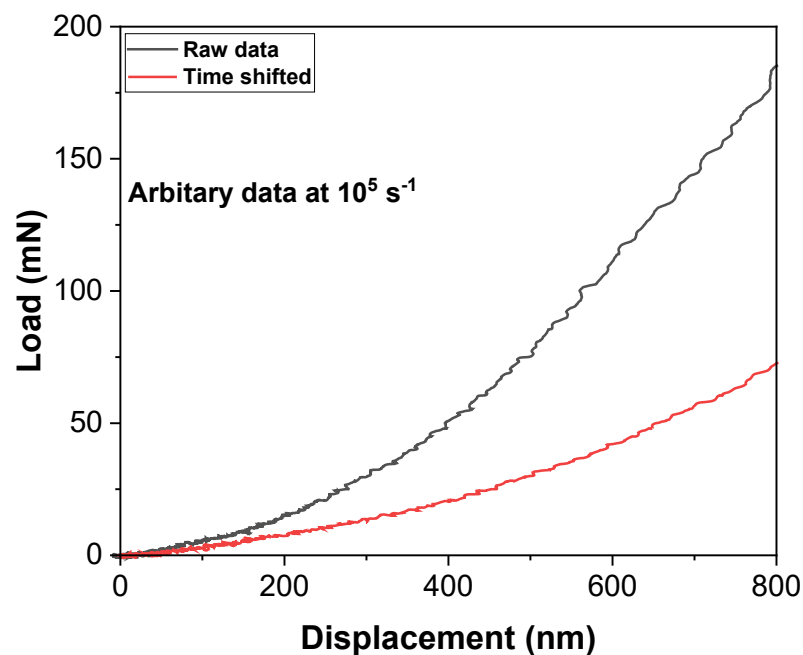
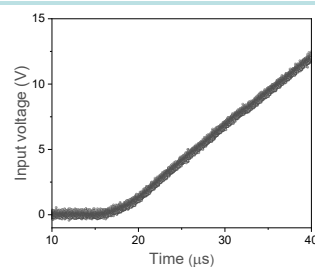
Signal processing - Time synchronization

Instrumentation

Time synchronization



Input profile



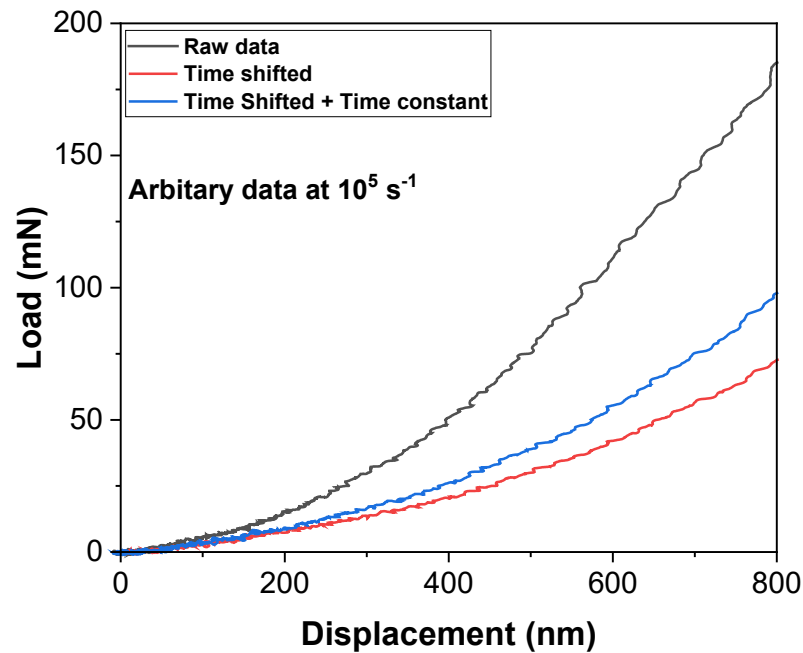
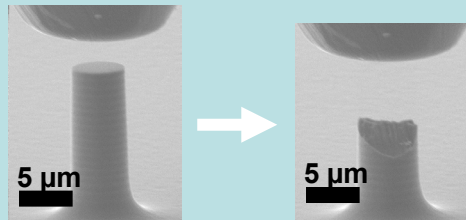
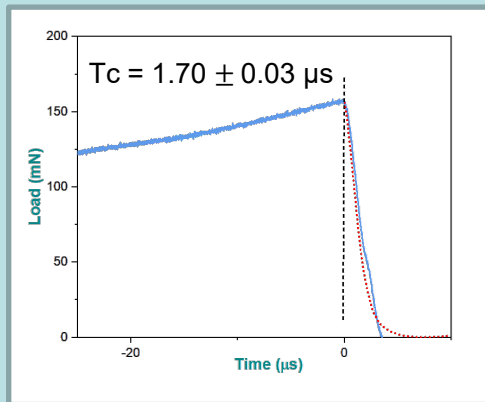
Starts affecting from strain rate:

Time shift: $\geq 10^3 \text{ s}^{-1}$

Signal processing - Time constant

Instrumentation

Time constant



Starts affecting from strain rate,

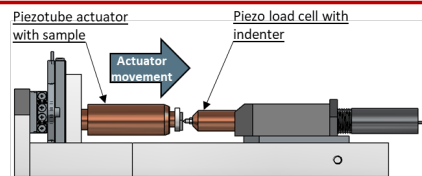
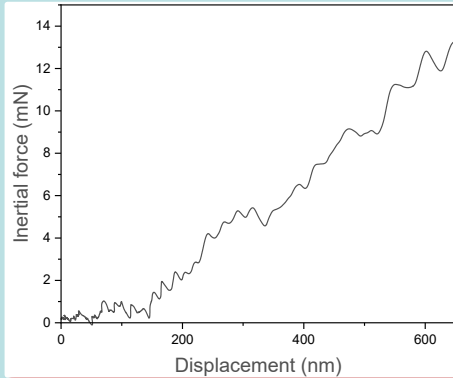
Time shift: $\geq 10^3 \text{ s}^{-1}$

Time constant: $\geq 10^4 \text{ s}^{-1}$

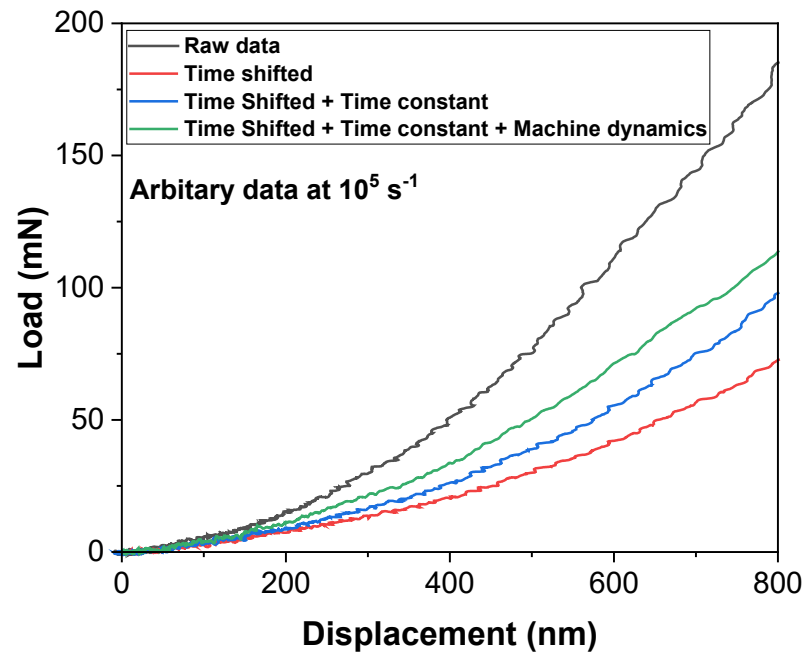
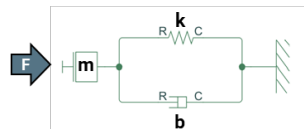
Signal processing - Machine dynamics

Instrumentation

Machine dynamics



Piezo load cell



Starts affecting from strain rate,

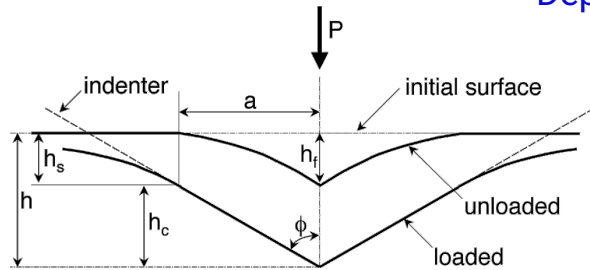
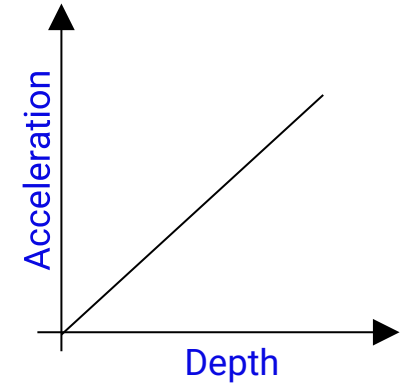
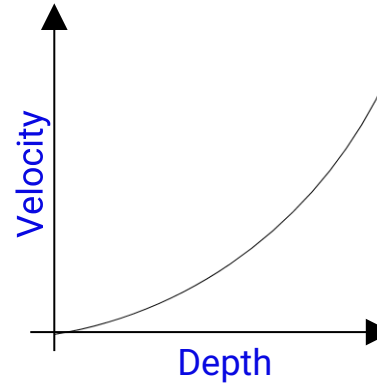
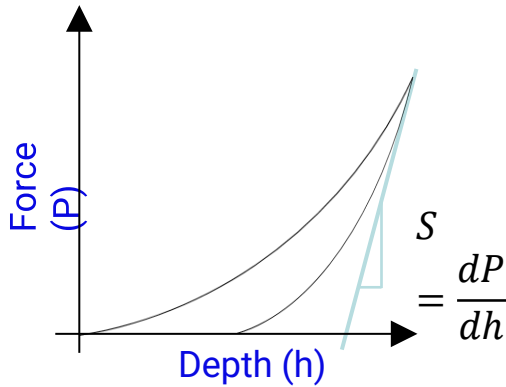
Time shift: $\geq 10^3 \text{ s}^{-1}$

Time constant: $\geq 10^4 \text{ s}^{-1}$

Machine dynamics: $> 10^4 \text{ s}^{-1}$

Constant strain rate indentation

$$\dot{\epsilon}_i = \frac{\dot{h}}{h}$$

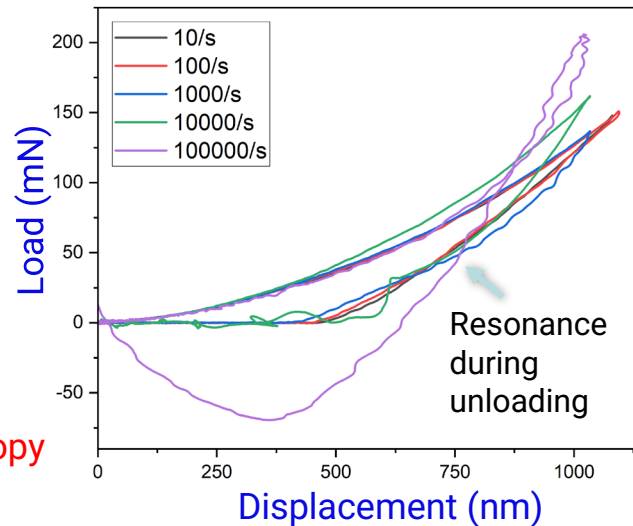


$$h_c = h - \varepsilon \frac{P}{S}$$

$$H = \frac{P}{A_c}$$

$$A_c = \sum_{i=0}^n m_i h_c^{2^{1-i}}$$

Confocal Laser microscopy



$$S = \frac{2E_r \beta \sqrt{A_c}}{\sqrt{\pi}}$$

Main assumption E_r remains constant

$$\sum_{i=0}^n m_i h_c^{2^{1-i}} - \frac{\pi \varepsilon^2 P^2}{4 \beta^2 E_r^2} = 0$$

$$A_c = \sum_{i=0}^n m_i h_c^{2^{1-i}}$$

h_c should be less than h .

Solving iteratively h_c can be determined

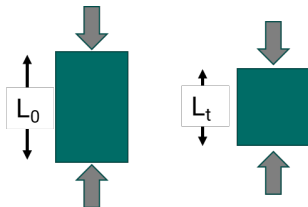
Merle et al., JMR, 2019

Constant strain rate indentation

Nano-indentation

Molybdenum high strain rate nano-indentation – How it happens?

In uniaxial experiments



For strain rate s^{-1}
Sample length 10 mm,
Input velocity ~ 1000 m/s

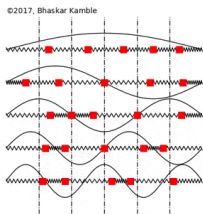
Elastic wave velocity in material ~ 3000 m/s

In indentation



For strain rate s^{-1}
Typical indent depth 1 μm ,
Input velocity ~ 0.1 m/s

Drag Strengthening

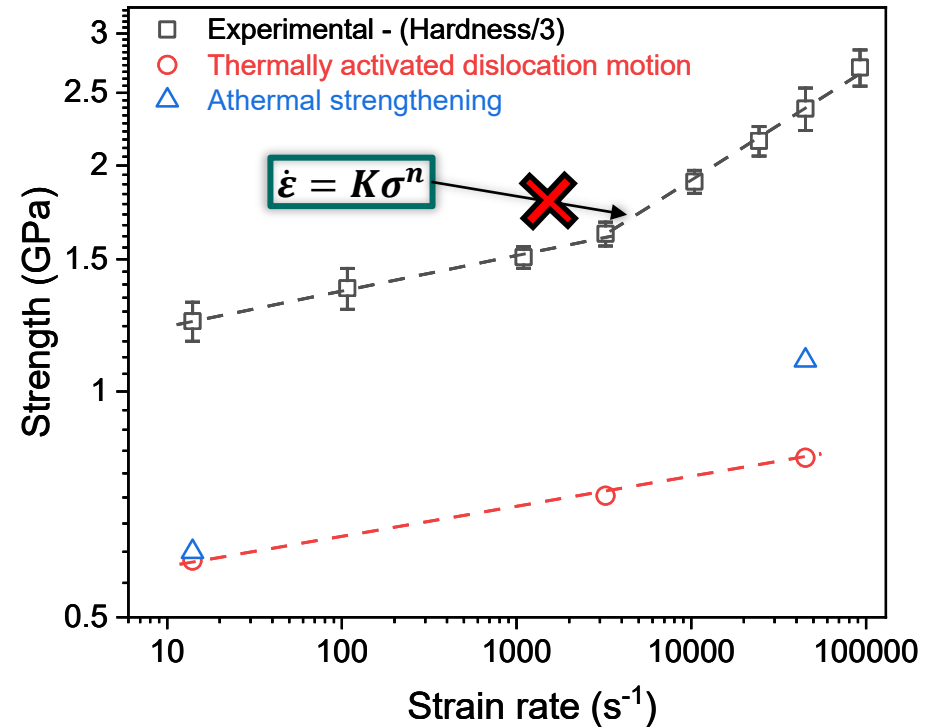


Harold and Ashby, 1982

$$\sigma_d = \frac{1.08 kT}{Ca^{\frac{1}{2}} \rho b} \dot{\epsilon}$$

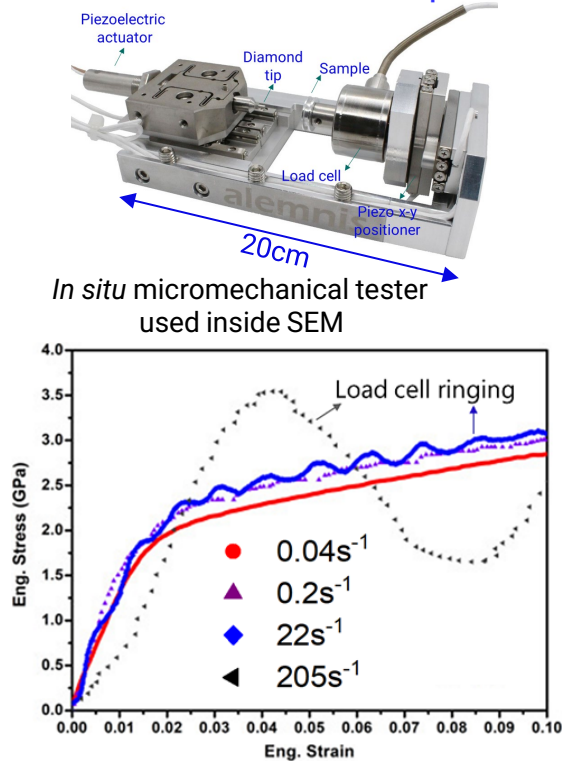
For, $\dot{\epsilon} = 10^1 s^{-1}$
 $\sigma_d = 1.03 \times 10^{-5} \text{ GPa}$

$\dot{\epsilon} = 5 \times 10^4 s^{-1}$
 $\sigma_d = 2.01 \times 10^{-2} \text{ GPa}$



High strain rate micromechanics - Methods

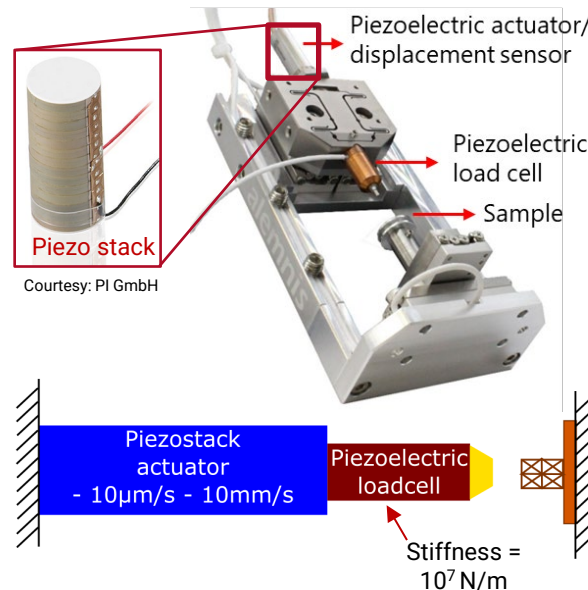
Quasi-static strain rates upto 1/s



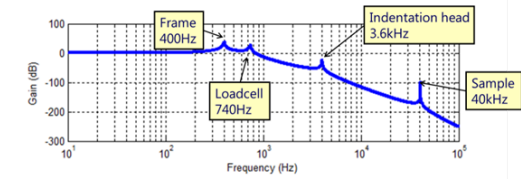
Maximum actuation speed: $10\mu\text{m/s}$
 Strain rate range: **$0.0001/\text{s}$ to $1/\text{s}$**

Guillonneau et al., Mat. and Des., 2018

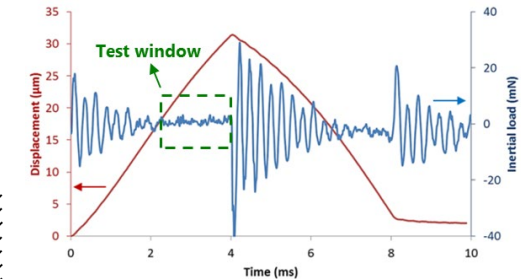
High strain rates up to 1000/s



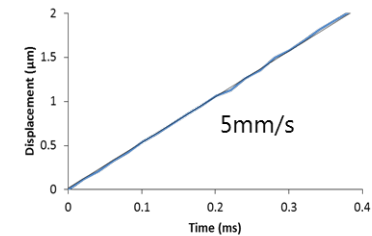
Actuation Method: *Piezoelectric*
 Displacement sensing: *Strain gage*
 Load sensing: *Piezoelectric*
 Maximum actuation speed – 10mm/s
 Strain rate range: **$1/\text{s}$ to $1000/\text{s}$**



High stiffness frame



Avoiding inertial contributions

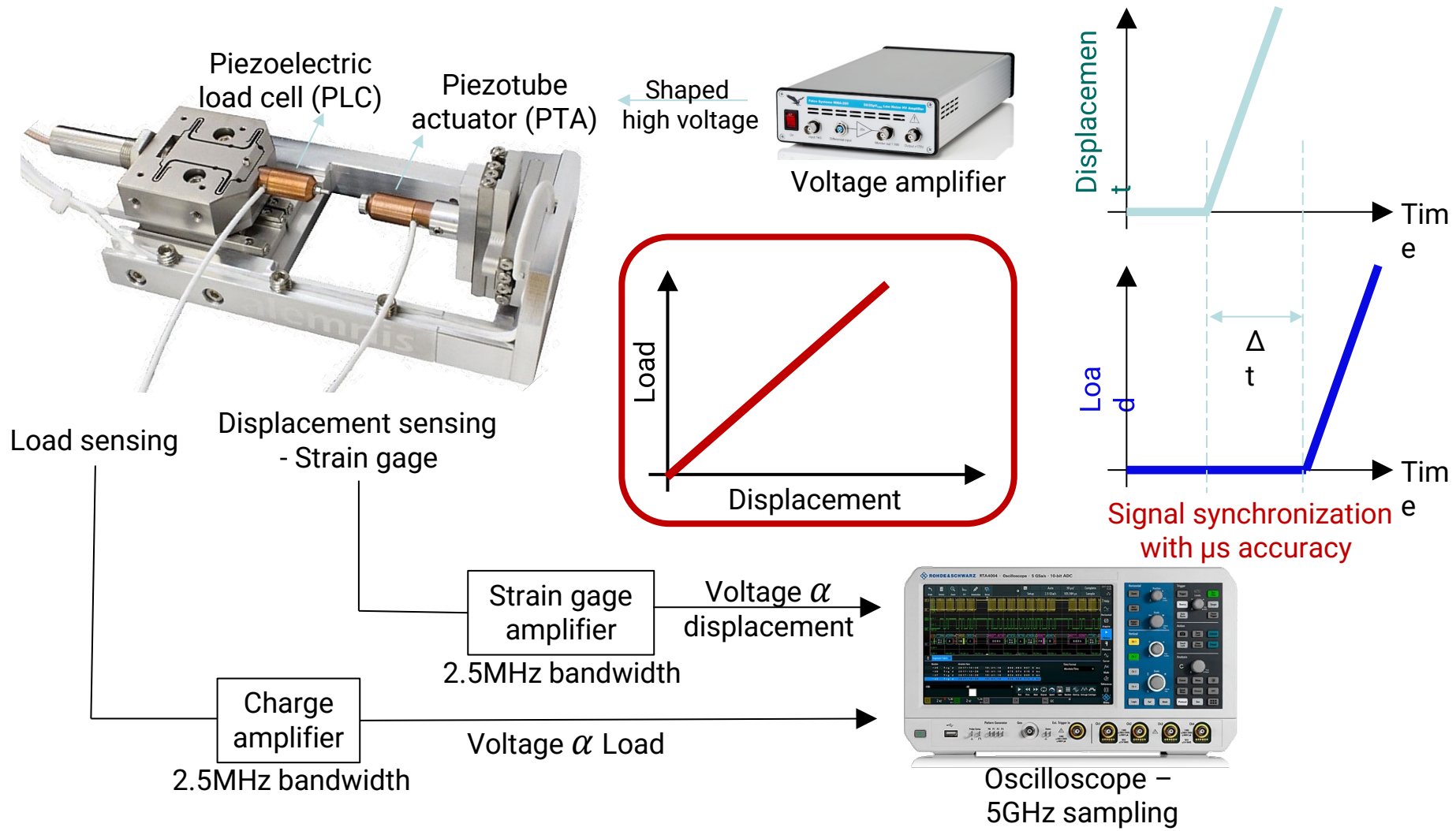


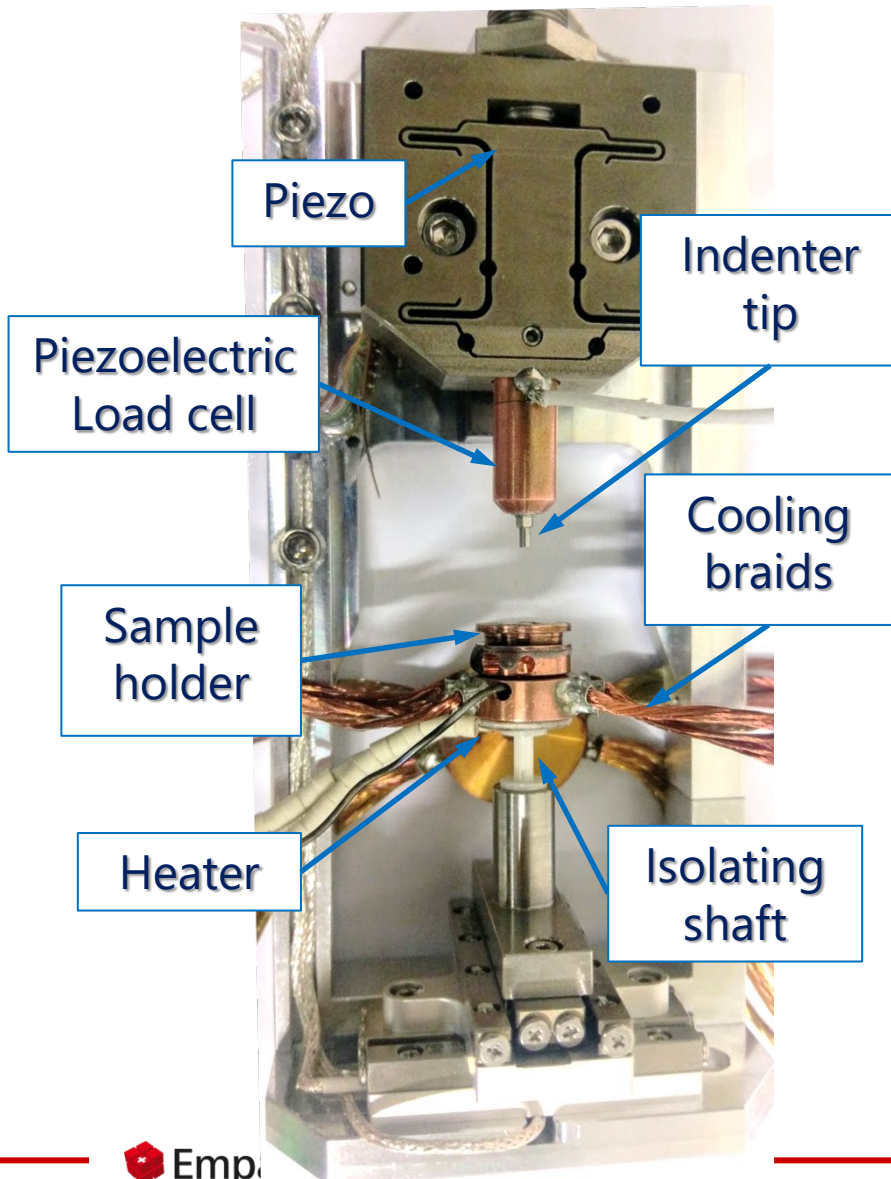
Constant strain rate
and accurate strain

Strain rates can be varied across 7 orders of magnitude

Ramachandramoorthy et al., Nanoletters., 2019
 Ramachandramoorthy et al., J. Mat. Res., 2021
 Ramachandramoorthy et al., App. Mat. Tod., 2022

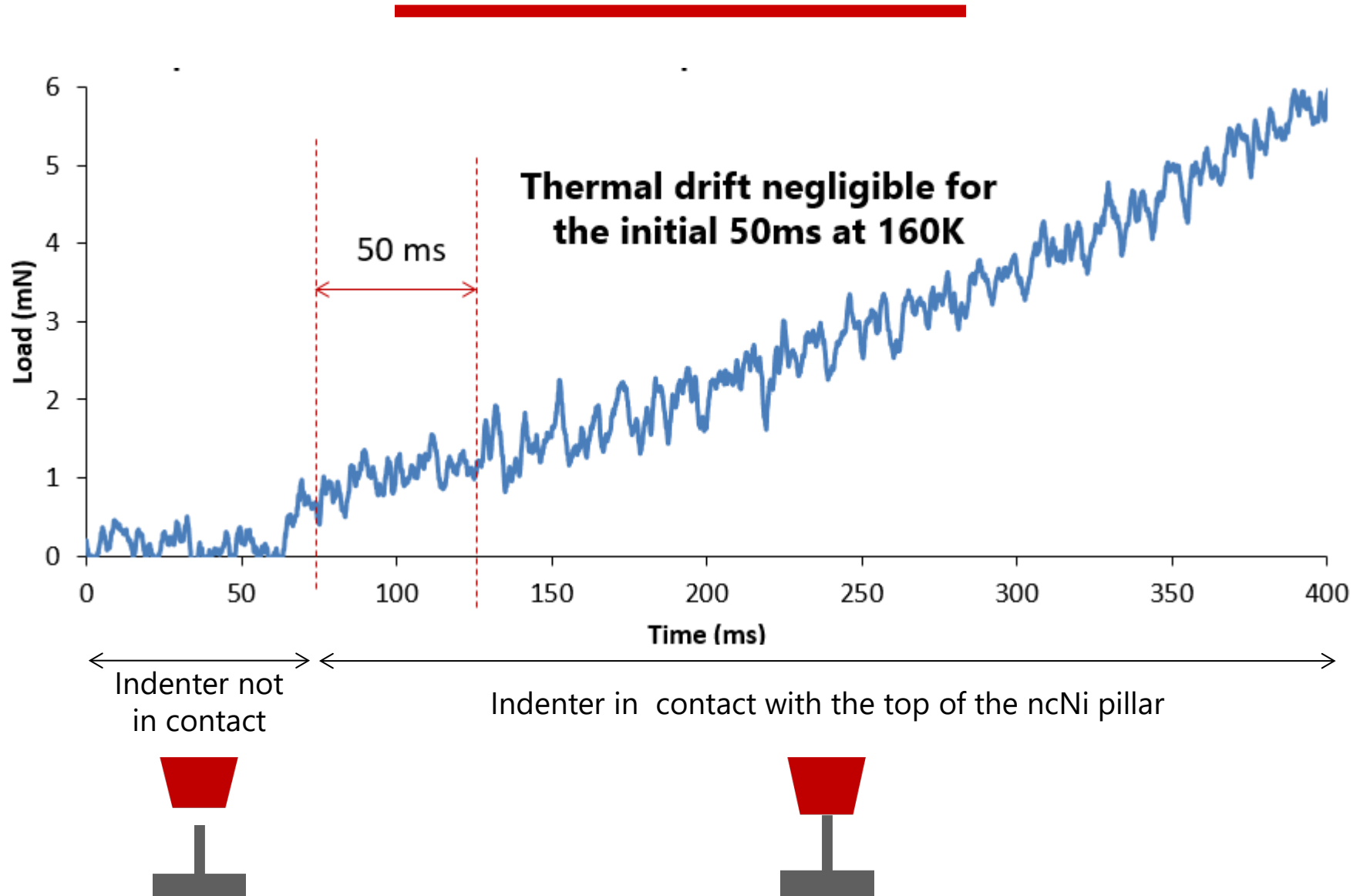
High strain rate micromechanical tester





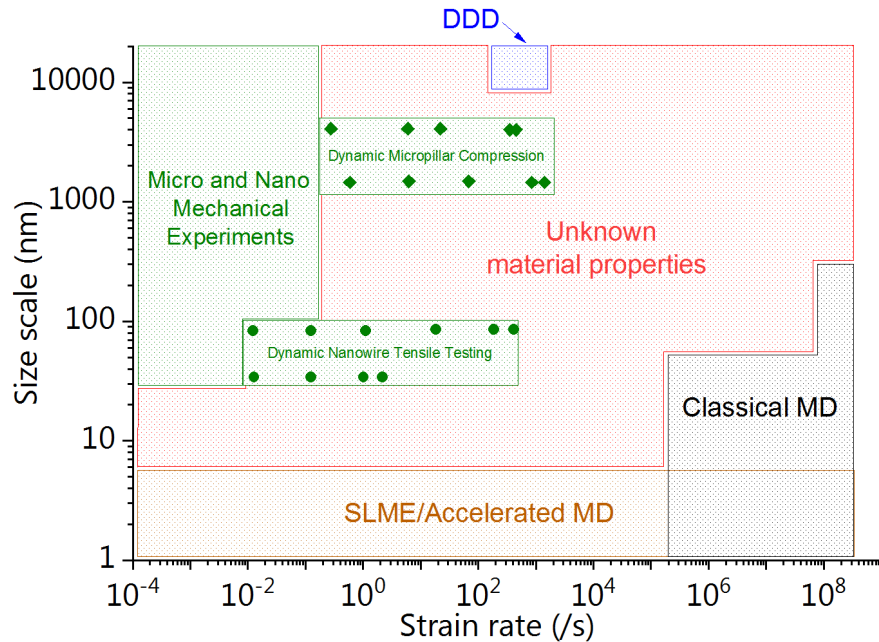
- Temperature range: 120K to 300K
- Strain rate range: 1 to 10^3s^{-1}
- Load range: 1N
- Standard load cell is replaced by a piezoelectric load sensor on the indenter tip side
- For measurement duration $< 50 \text{ms}$, thermal drift remains negligible for an uncooled tip in contact with a cold sample

Measurement duration for negligible load drift

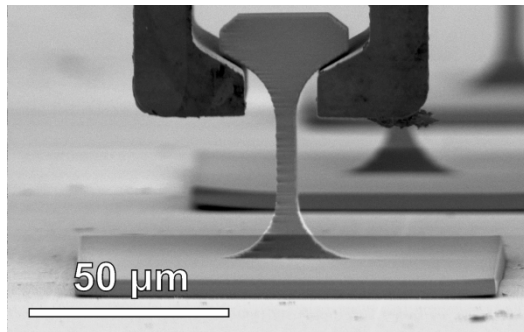


Maximum duration of cryo temperature/high strain rate tests possible – **50 ms**
Translates to a strain rate of $\sim 3/s$ for a gage length of $8\mu m$

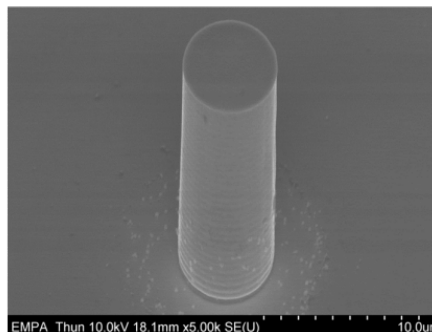
Outlook



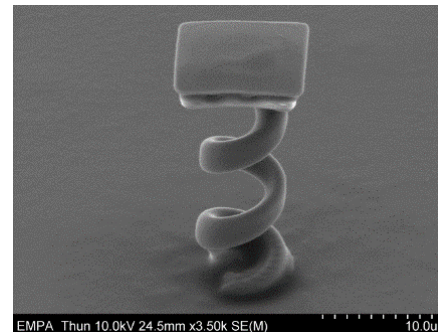
- Micromechanical investigation of deformation and failure under extreme conditions is a new phase space in small scale research
- Open questions on amorphous plasticity
 - Natural extension of dynamic testing to metallic glasses
 - What is effect of strain rate and temperature combination?



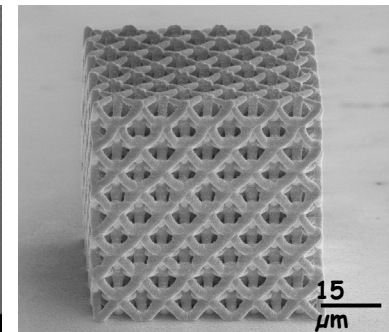
Polymer micropillars - High T + HSR



ncNi micropillars - Cryo + HSR



ncNi springs



Full metal microlattices

Case study: deformation mechanisms of nanocrystalline metals

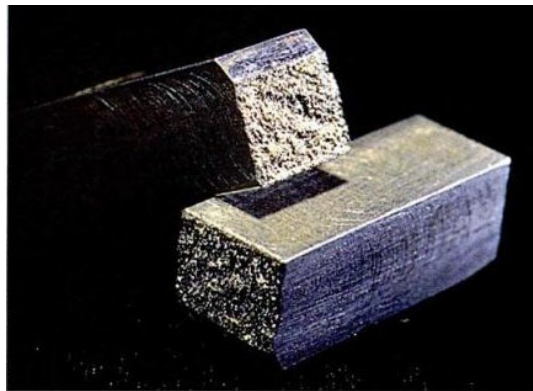
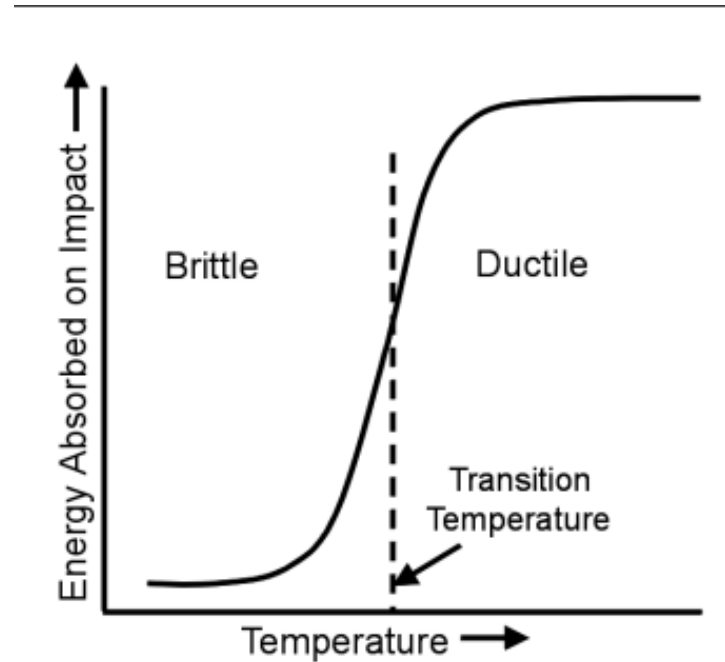
Why low temperature tests - case of the Titanic



The Titanic on the seabed, 3,784 meters deep

Charpy test results on steel samples at -4°C (the temperature of Atlantic waters when The Titanic sank)

Reference: Robert Gannon, *Popular Science*, 1995

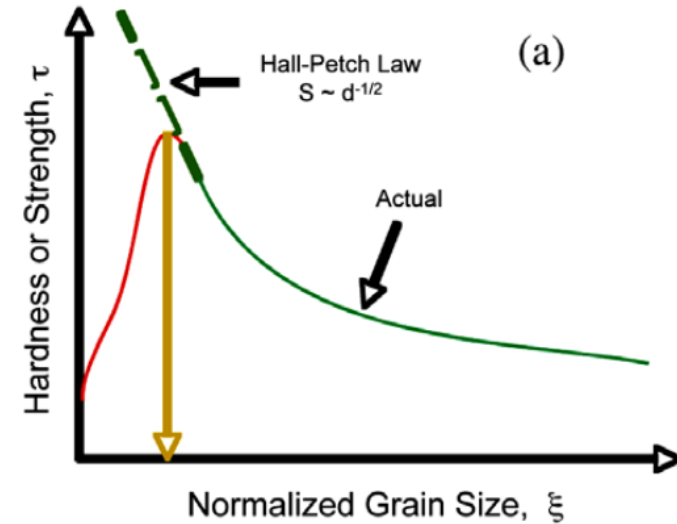
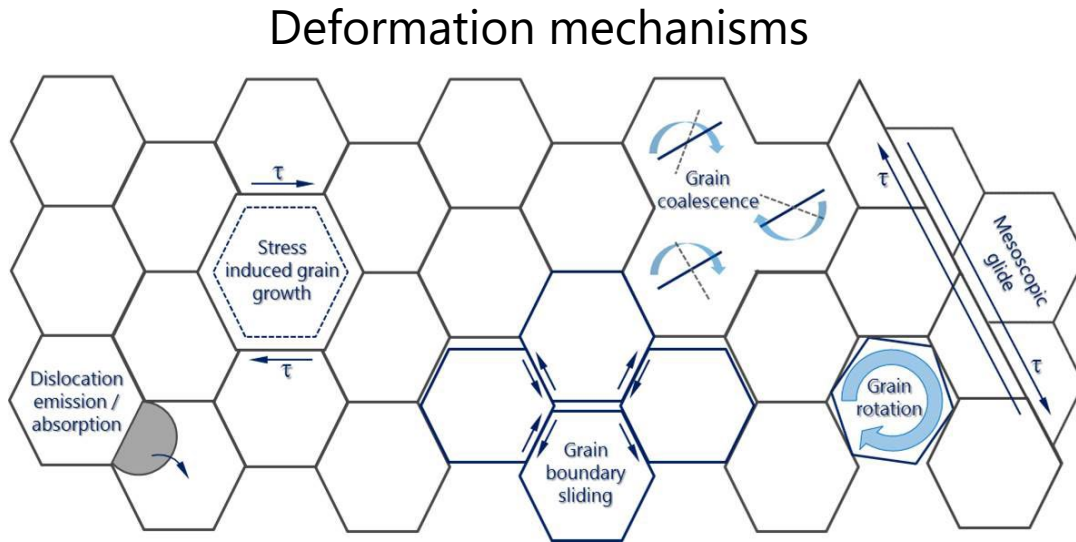


Steel from Titanic's hull – **Brittle** fracture



Modern high quality steel - **Ductile** fracture

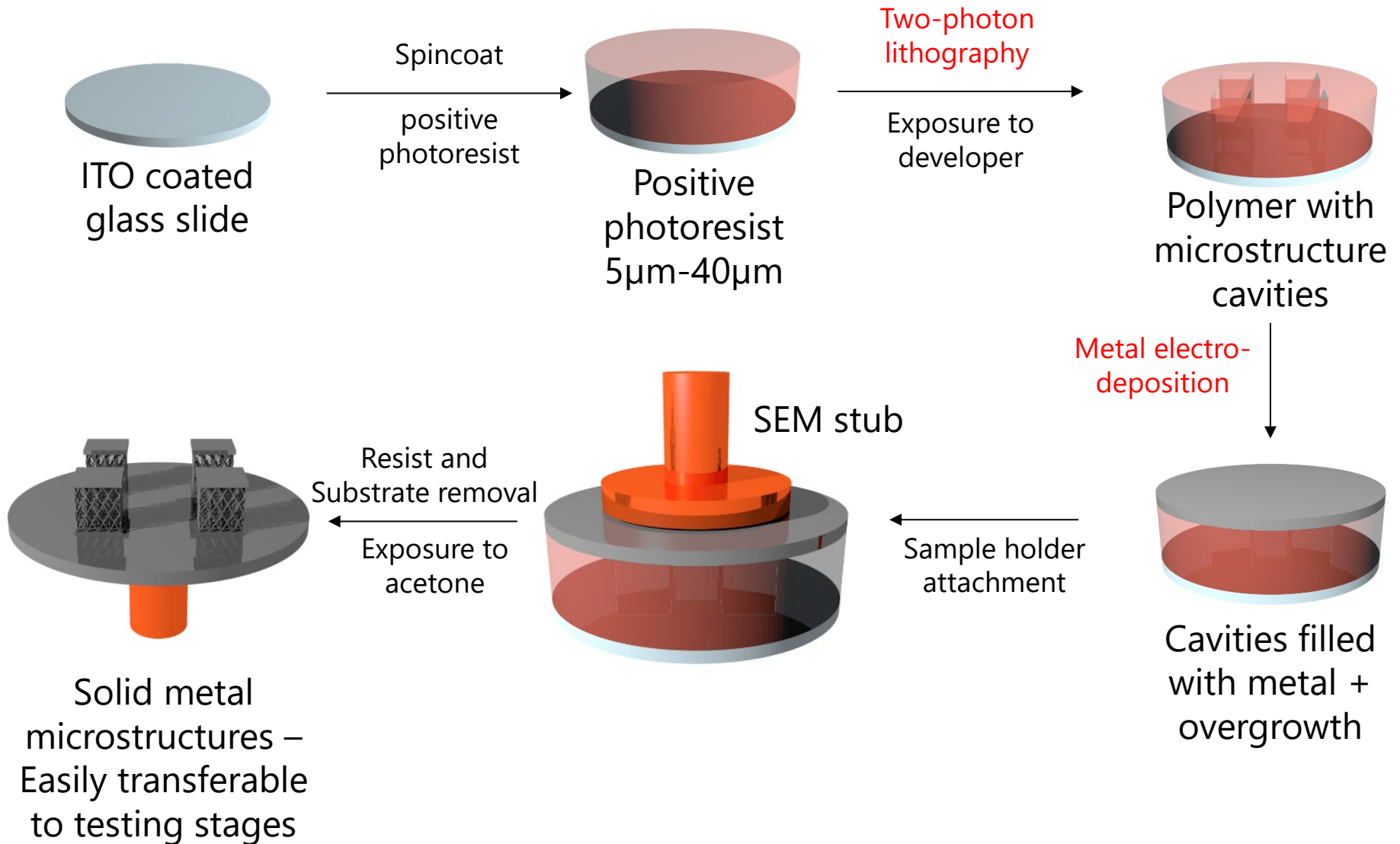
Deformation mechanisms in nc metals



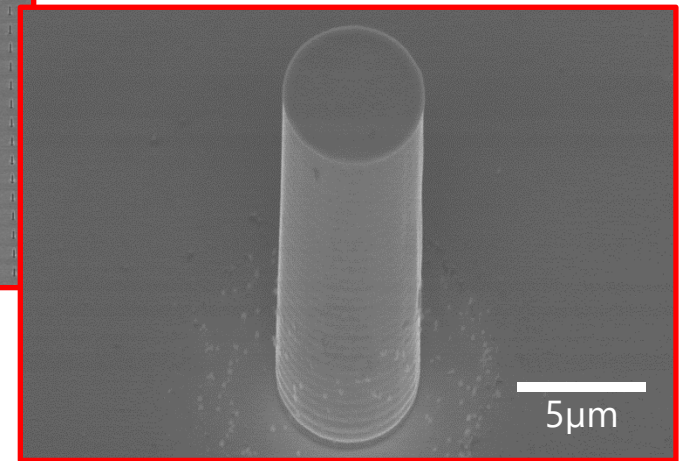
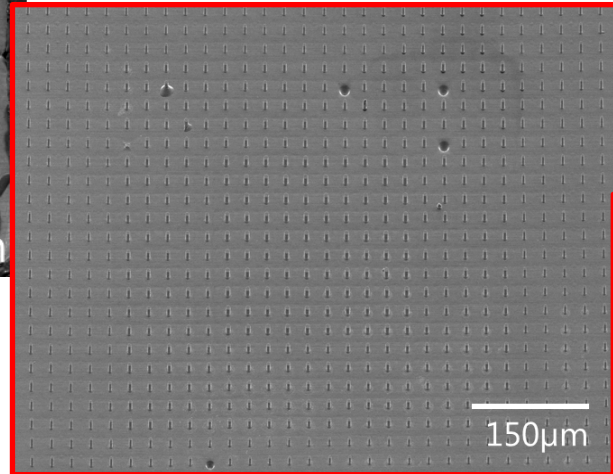
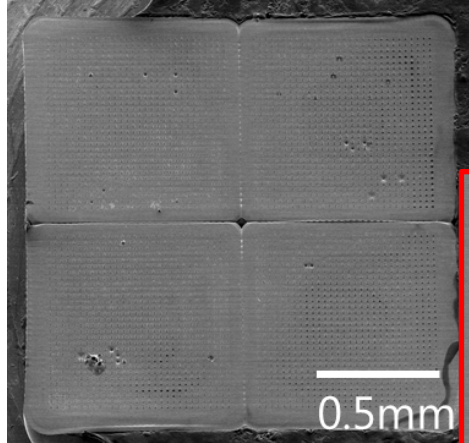
- Mechanical properties are dominated by grain size
- Pronounced time-dependent plastic behaviour
- Grain growth through FIB irradiation and at elevated temperatures
- Electrodeposition into lithography moulds
- Thermal activation analysis at low temperatures and high strain rates

Micropillar array preparation by electrodeposition

Workflow for producing nc metal microstructures



Nanocrystalline Ni micropillar array

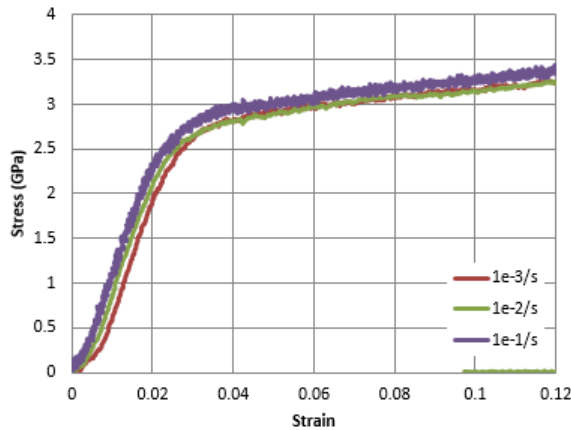


- Array of 4000 electrodeposited nanocrystalline Ni micropillars
- No FIB damage, no FIB-induced grain growth, no taper
- Grain size estimated based on XRD: ~28nm
- Random grain orientation

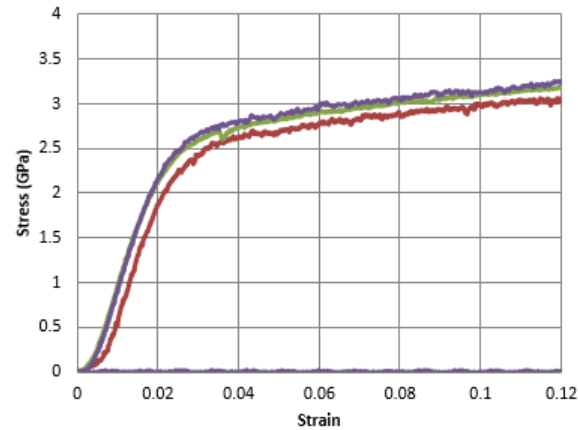
Thermal activation analysis at subambient temperatures and high strain rates

Quasistatic results

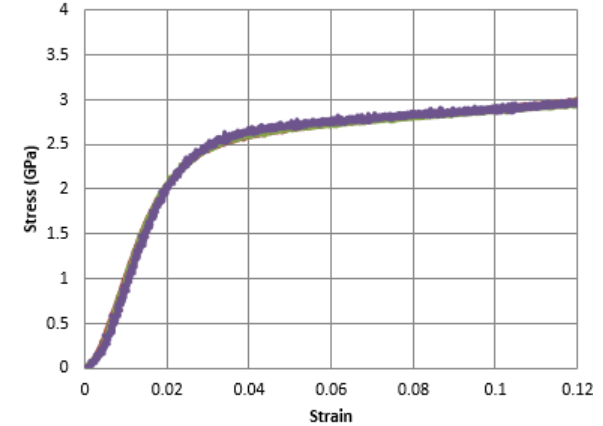
160K



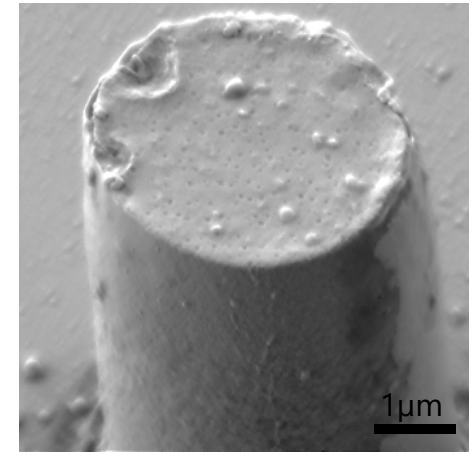
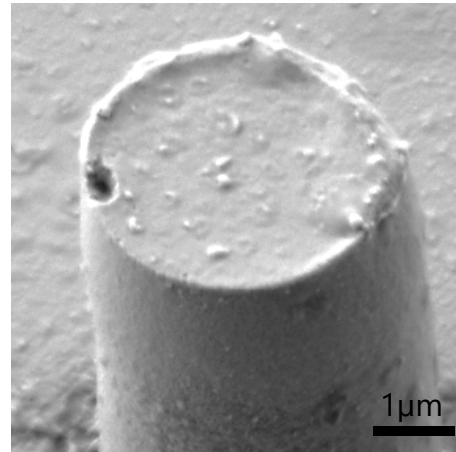
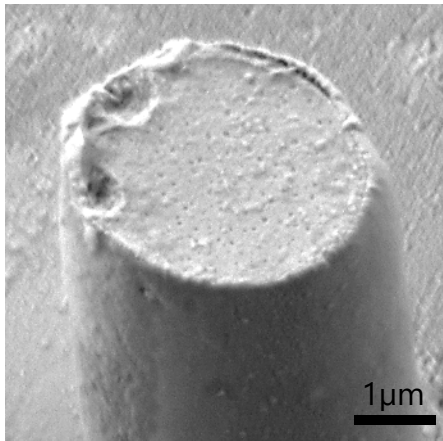
224K



300K



$10^{-3}s^{-1}$

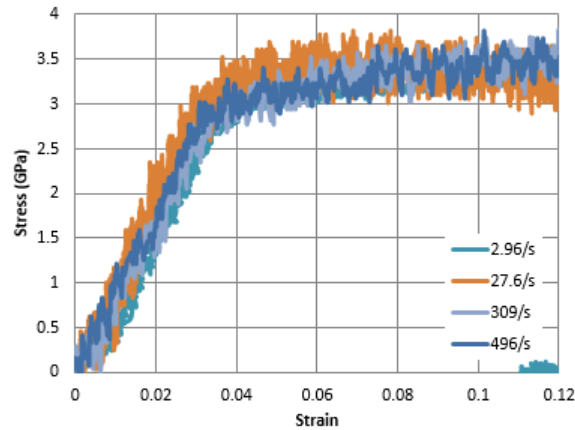


■ Homogeneous deformation, barrelling of micropillar

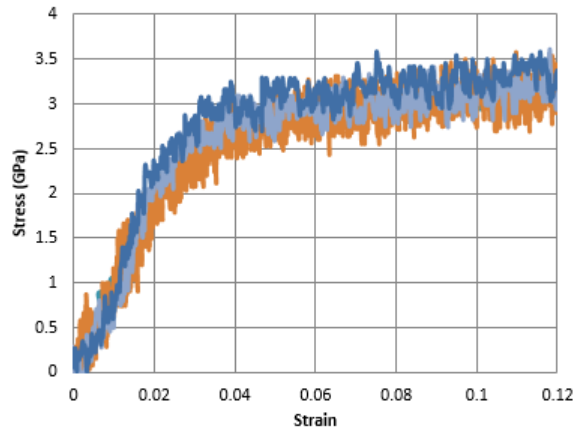
■ Weak dependence of yield stress on strain rate, strong dependence on temperature

High strain rate results

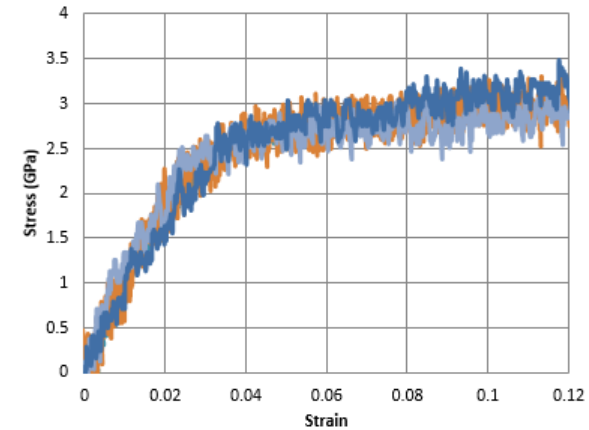
160K



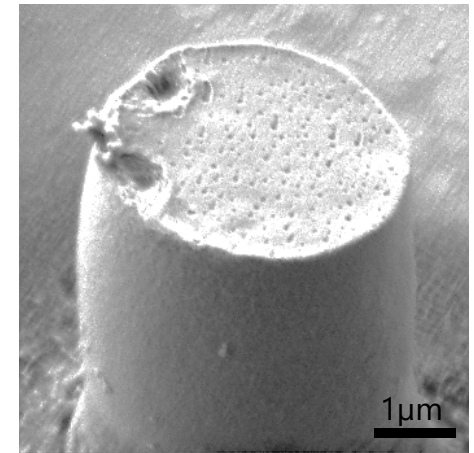
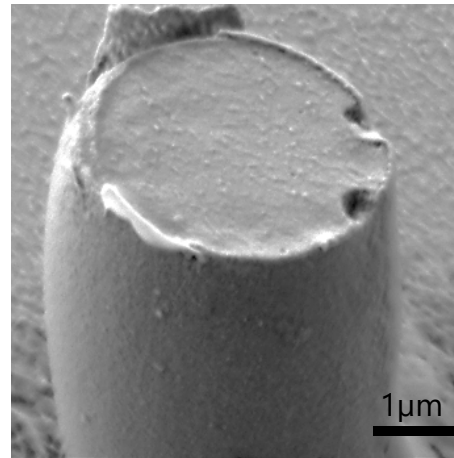
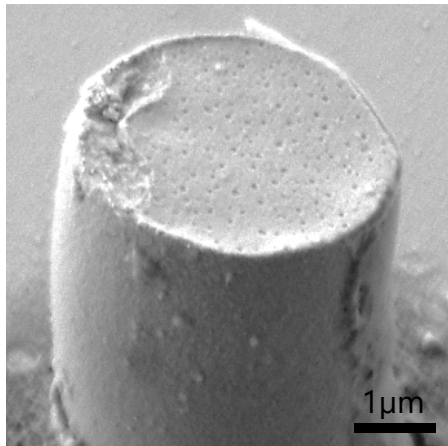
224K



300K



309s⁻¹



Results consistent with quasistatic experiments:

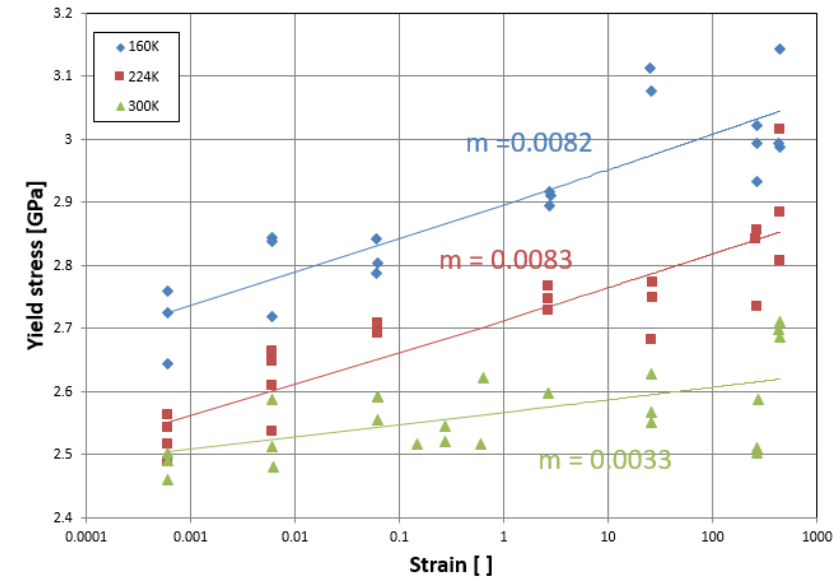
- Weak dependence of yield stress on strain rate, strong dependence on temperature

Thermal activation analysis

Strain rate sensitivity:

$$m = \frac{d \ln \sigma}{d \ln \dot{\epsilon}}$$

- m is increased at lower temperatures



Thermal activation analysis

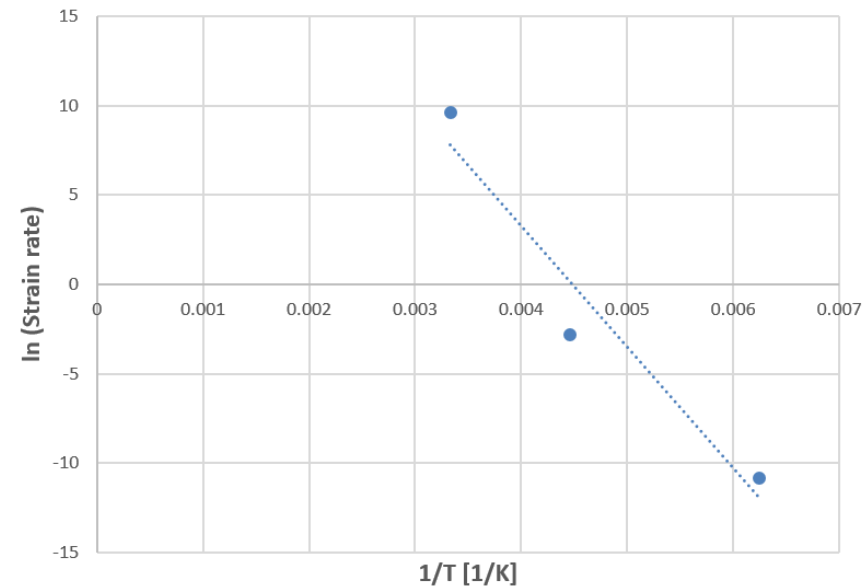
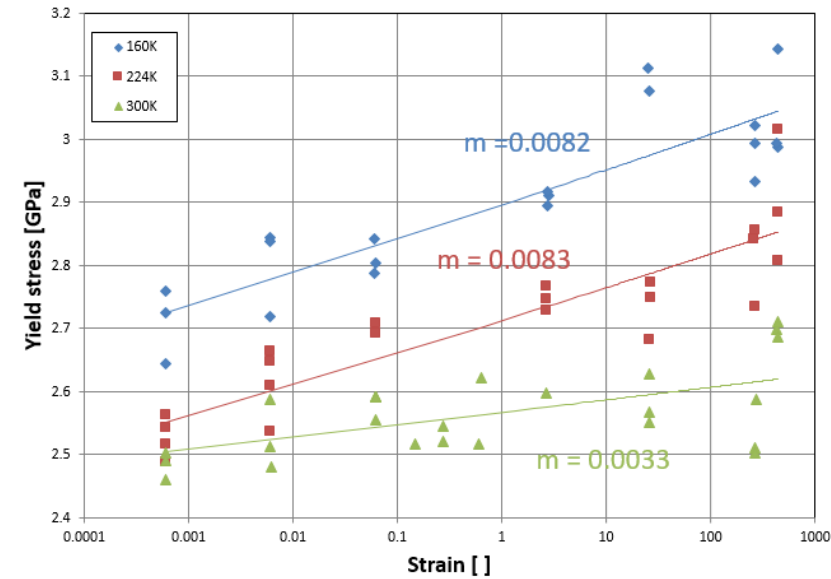
Strain rate sensitivity:

$$m = \frac{d \ln \sigma}{d \ln \dot{\epsilon}}$$

- m is increased at lower temperatures

Apparent activation energy:

- Extrapolated strain rates for $\sigma_y = 2.65 \text{ GPa}$
- Q_{app} is approx. 55 kJ/mol (0.58 eV)



Thermal activation analysis

Strain rate sensitivity:

$$m = \frac{d \ln \sigma}{d \ln \dot{\epsilon}}$$

- m is increased at lower temperatures

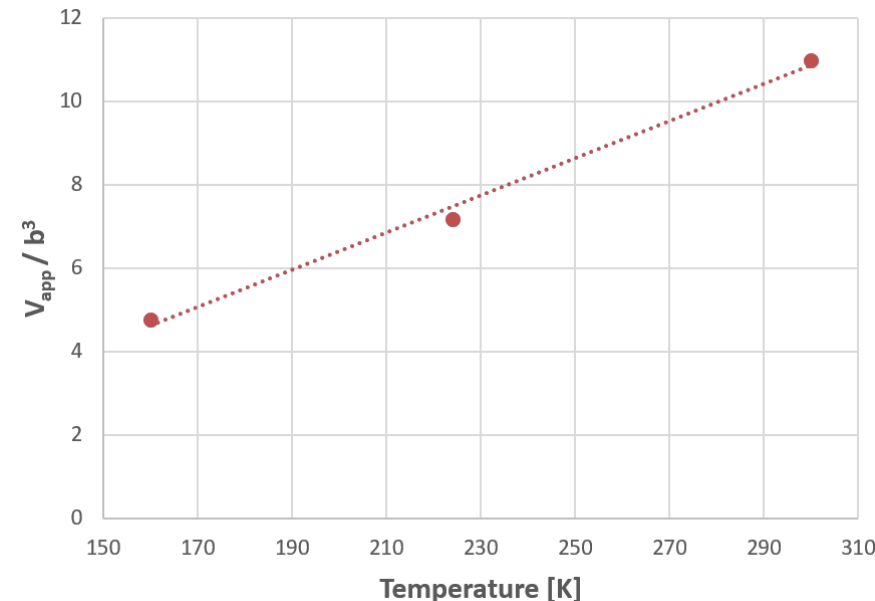
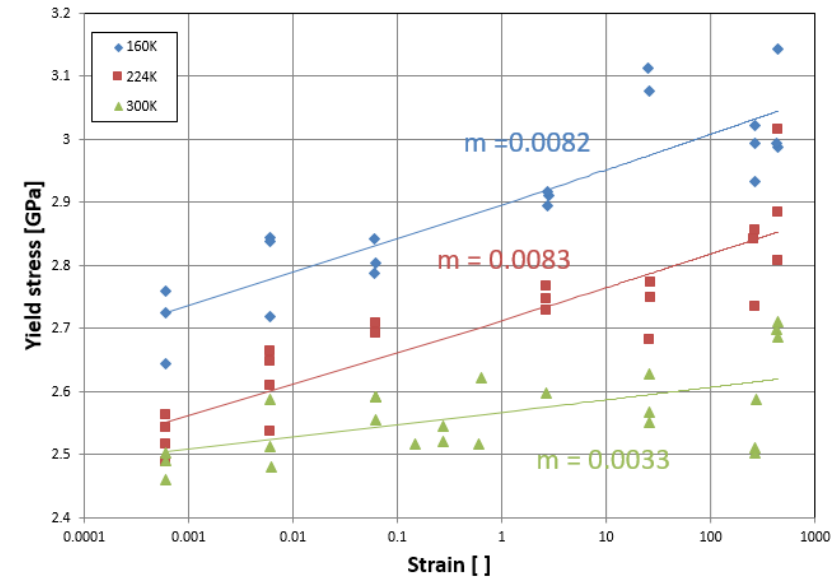
Apparent activation energy:

- Extrapolated strain rates for $\sigma_y = 2.65 \text{ GPa}$
- Q_{app} is approx. 55 kJ/mol (0.58 eV)

Apparent activation volume:

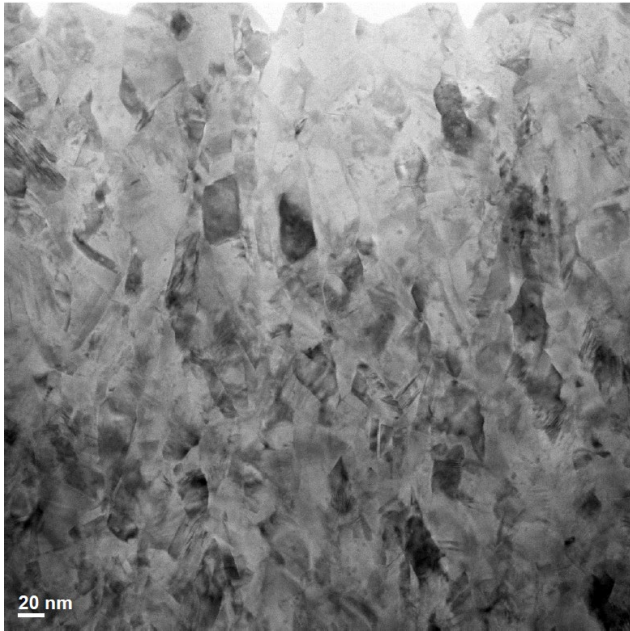
$$V_{\text{app}} = k_B T \left(\frac{d \ln \dot{\epsilon}}{d \sigma} \right)$$

- V_{app} increases with temperature from $5b^3$ at 160 K to $11b^3$ at 300 K

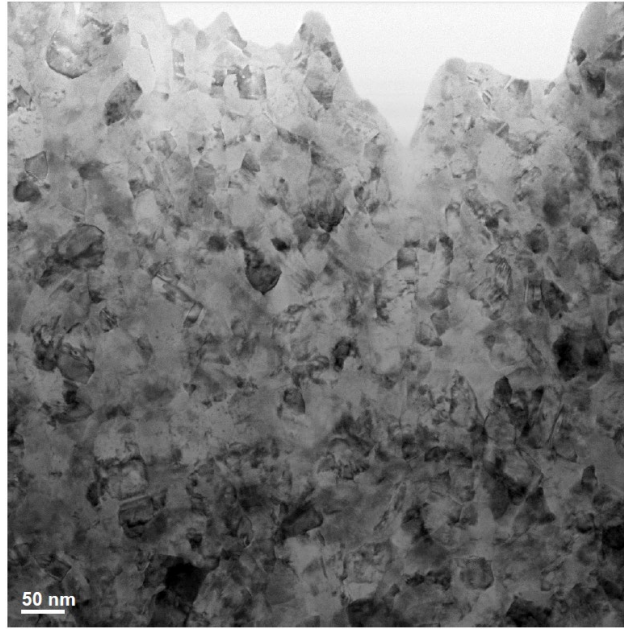


TEM analysis

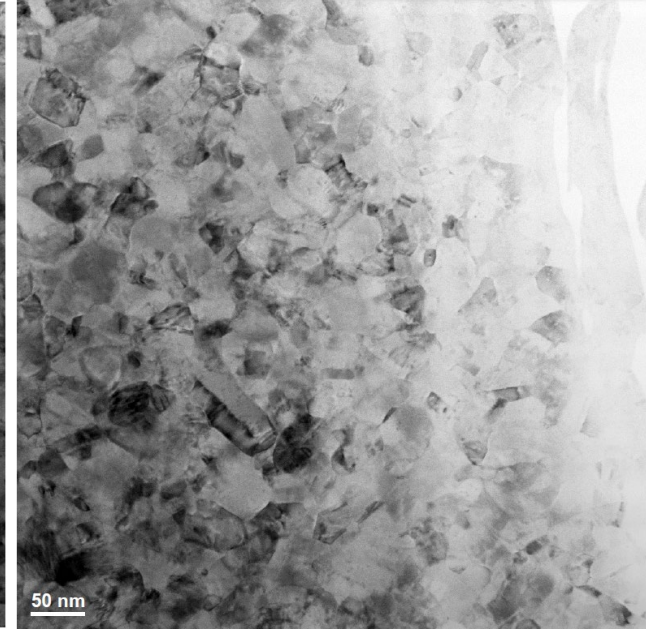
undeformed



160K, 0.001s^{-1}



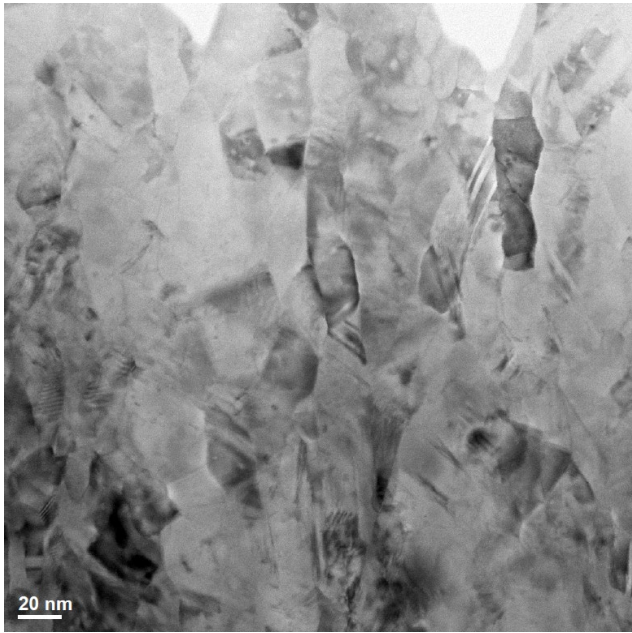
160K, 300s^{-1}



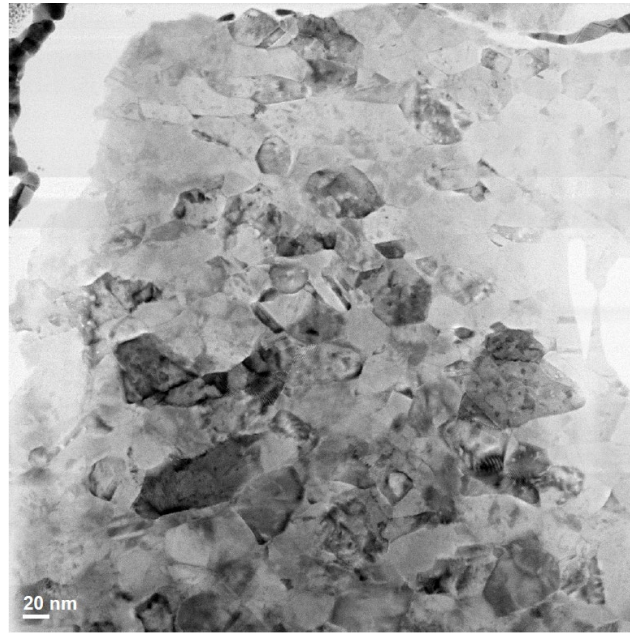
- TEM lamellae have to be extremely thin, preparation and imaging are complicated
- A change in grain shape from elongated in growth direction to oblate is observed in deformed pillars
- Grain size seems more heterogeneous in deformed pillars

TEM analysis

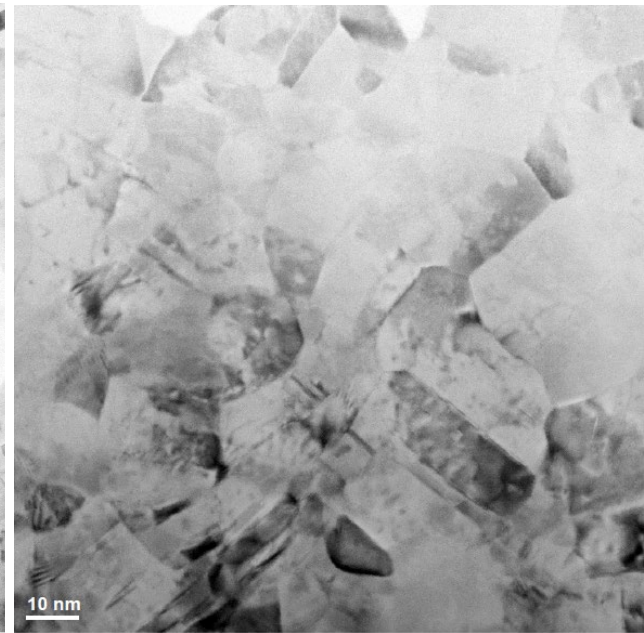
undeformed



160K, 0.001s^{-1}



160K, 300s^{-1}



- TEM lamellae have to be extremely thin, preparation and imaging are complicated
- Change in grain shape from elongated in growth direction in undeformed to isotropic/oblate in deformed pillars
- Grain size more heterogeneous in deformed pillars
- Twin density does not increase

Discussion

Electrodeposited micropillar arrays:

- Large number of specimens
- No FIB damage, no taper
- Influence of confined growth on grain structure?
 - Grains elongated in growth direction

Instrument development:

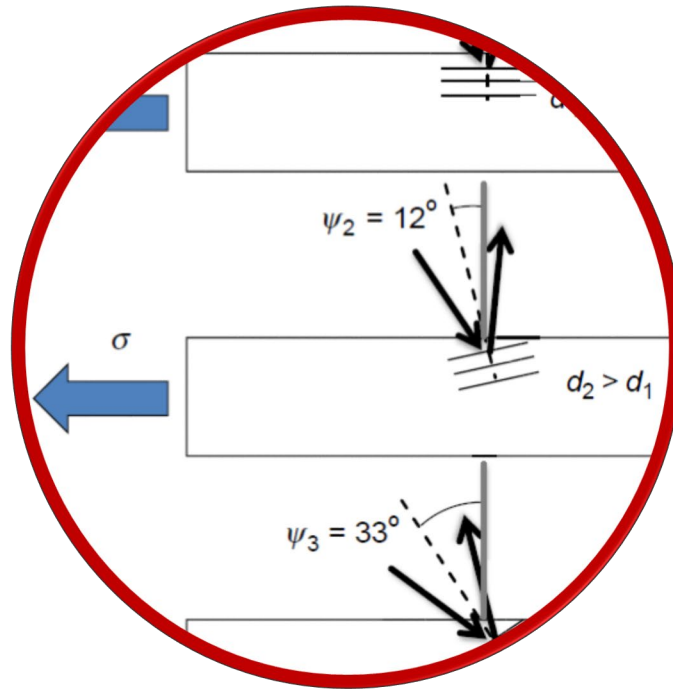
- In situ experiments at low temperatures and in large strain rate range:
 - 120K to 300K
 - Strain rate range: 10^{-4} to 10^3s^{-1}
 - Large strain rate range → even low m may be measured

Discussion

Deformation mechanisms in nc Ni:

- Activation volume and energy are consistent with dislocation nucleation, i.e. intragranular plasticity, but also other deformation mechanisms like grain boundary or defect diffusion are likely present
- Temperature dependence of activation volume reflects thermally-dependent interplay of different deformation mechanisms
- Experiments at lower temperature are needed to identify at what point thermal activation is suppressed
- Post-test TEM analysis: higher variation in grain size and change in grain shape, needs to be verified
- Next steps: TKD analysis for improved grain size and shape statistics

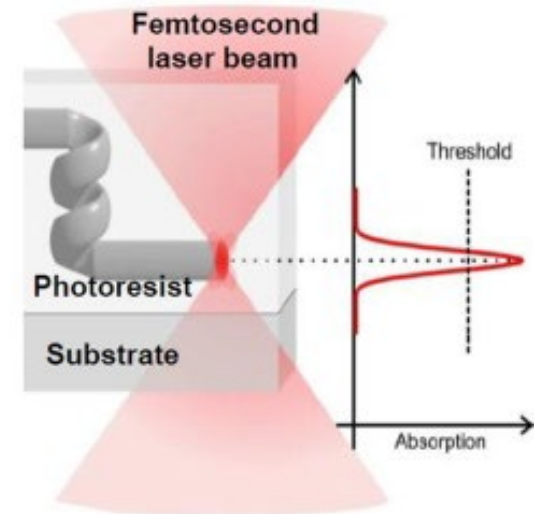
Case study: Extreme micromechanics of 3D printed polymers



Micromechanical testing at high strain rates and varying temperatures of 3D-printed polymer structures

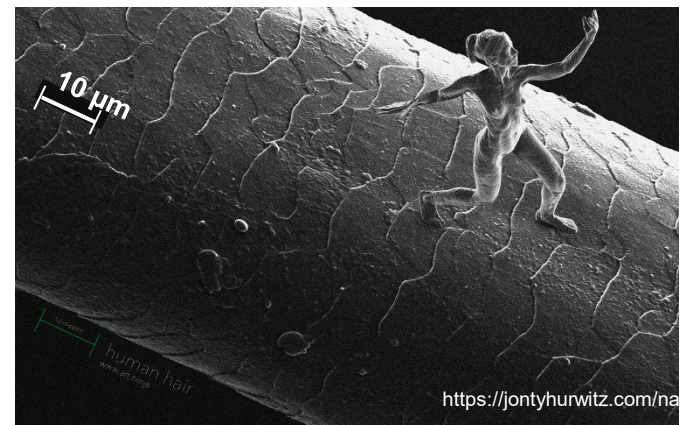
Two-Photon Lithography (2PL)

- Two-Photon Lithography Direct Laser Writing allows 3D printing of micron-sized polymer components
- A resolution in the tens of nanometres can be achieved
- Excellent for rapid prototyping, templating or surface modification



Here:

- Photonic Profession GT (Nanoscribe GmbH)
- Commercially available negative photoresist "IP-Dip" an acrylate-based polymer
- ITO covered glass substrate for improved adhesion

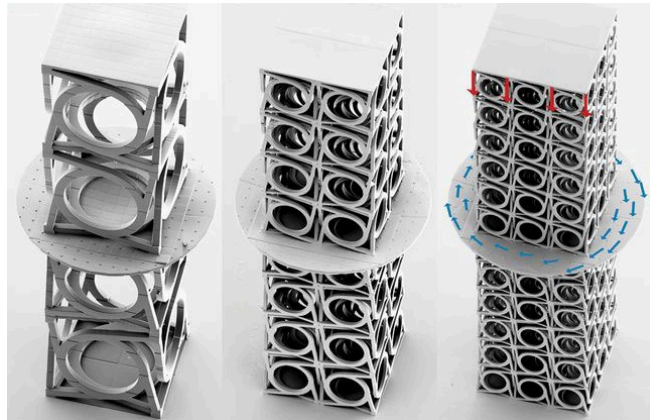


Microlattices and the Fabrication of Metamaterials

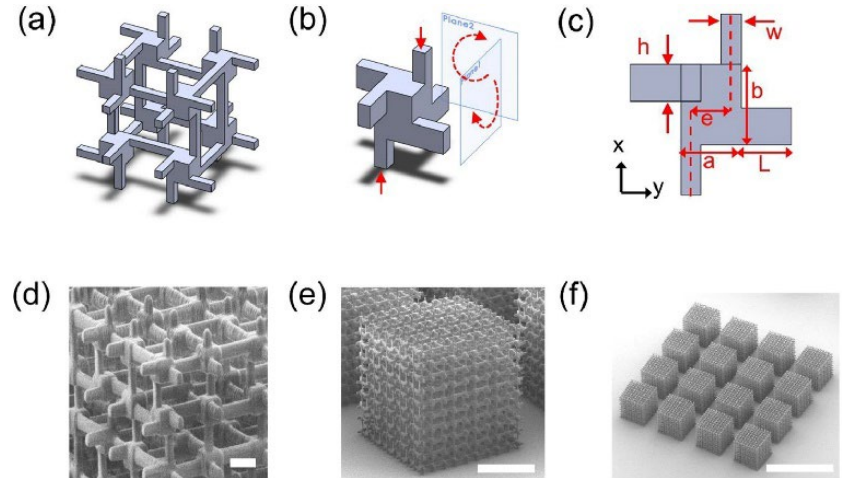
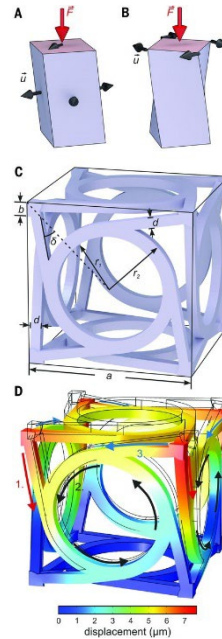
METAMATERIALS

Three-dimensional mechanical metamaterials with a twist

Tobias Frenzel,¹ Muamer Kadic,^{1,2,3} Martin Wegener^{1,2*}



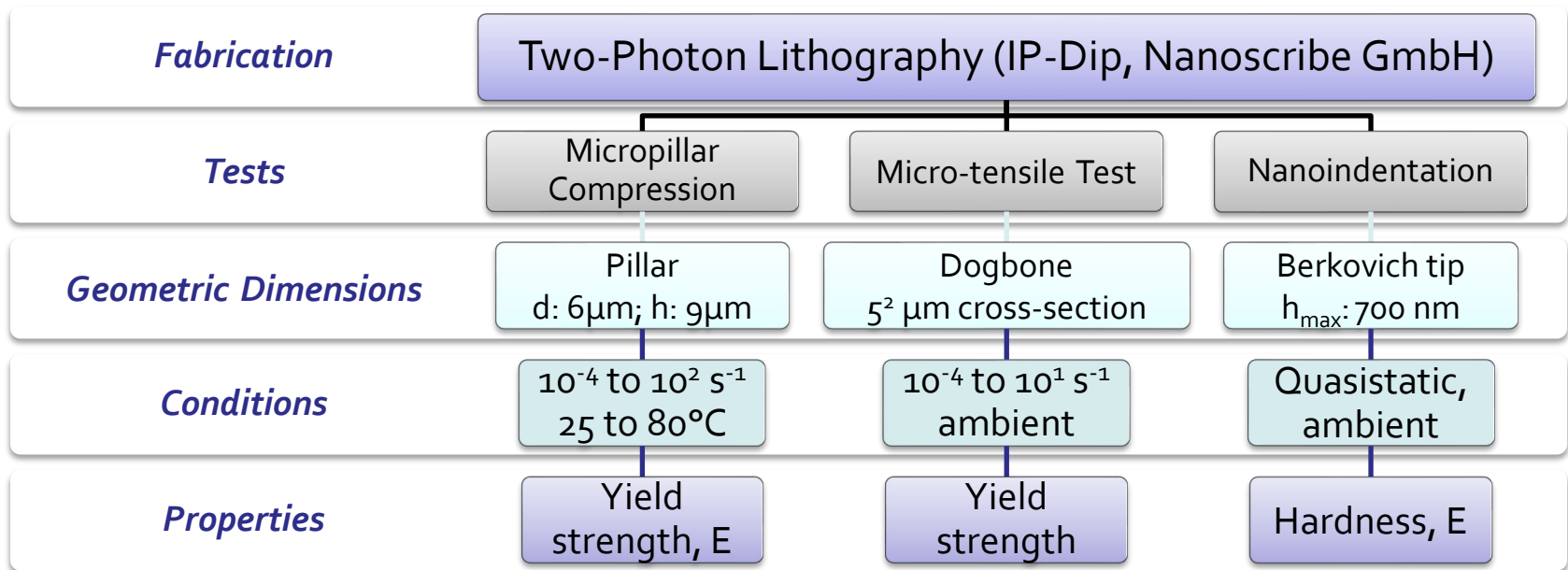
Science



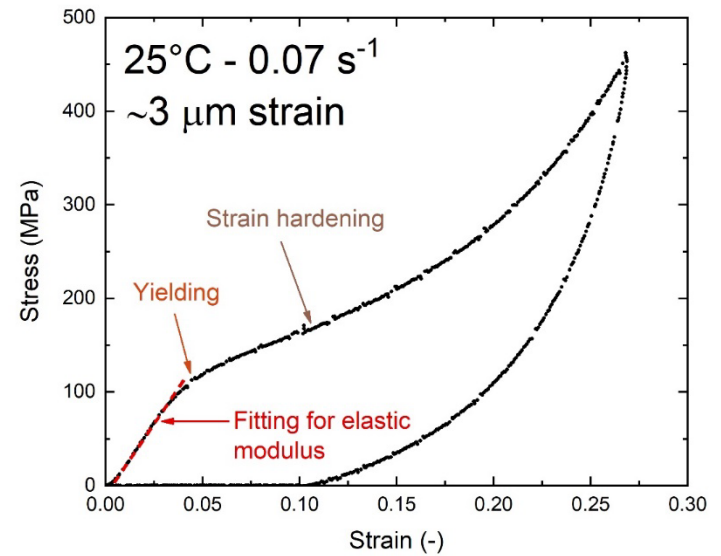
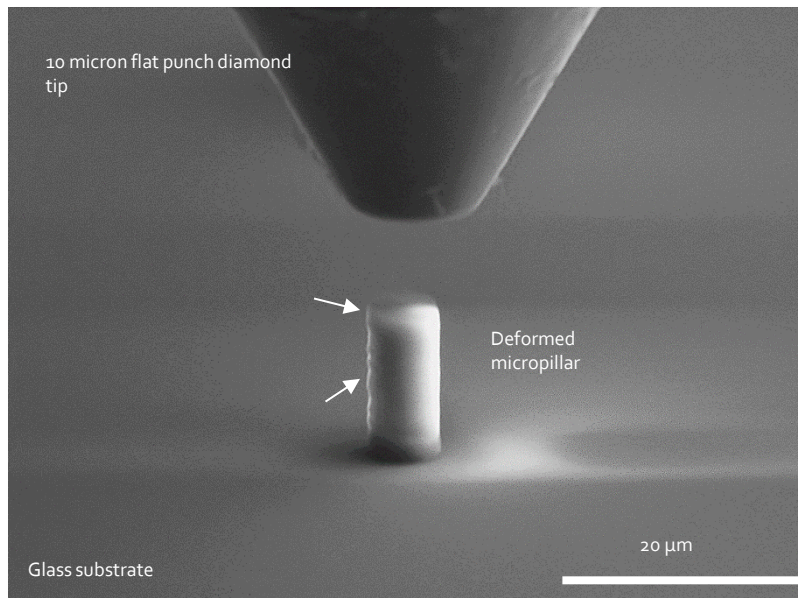
Highly porous microlattices as ultrathin and efficient impact absorbers

Chang Quan Lai^{a,b,*}, Chiara Daraio^b

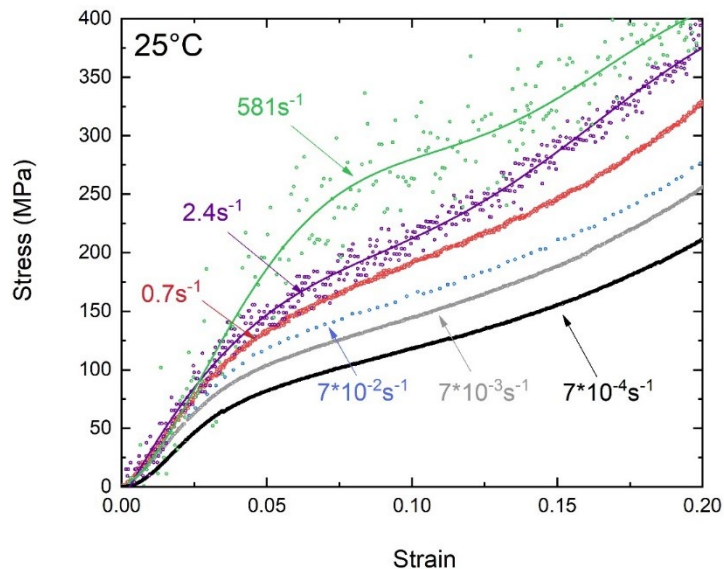
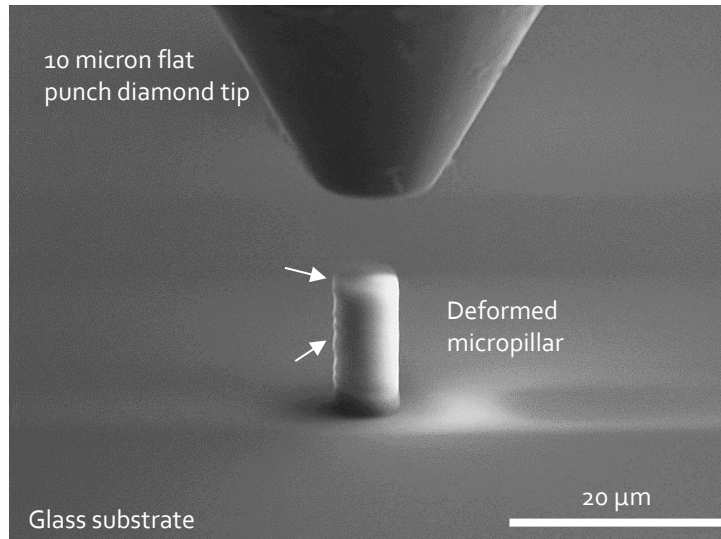
Micromechanical Testing: Experiments



Micropillar compression tests

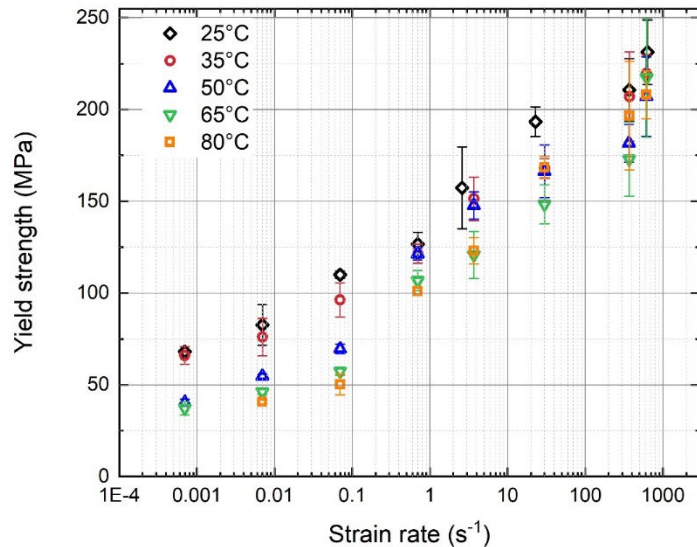


Micropillar compression tests - Strain rate dependence



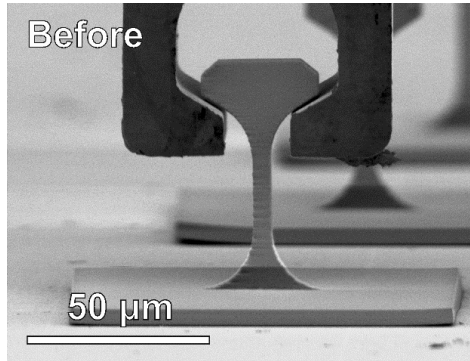
- Strong strain rate dependence in deformation behaviour of polymer pillars
- All pillars were 6 μm in diameter and 9 μm in height
- 3 Pillars were measured for each condition $\rightarrow$ measurements showed good reproducibility
- Yielding occurred above 2 mN $\rightarrow$ reliable measurement possible
- Data acquired in the quasistatic regime was corrected for thermal drift

Micropillar compression tests - Yield Strength

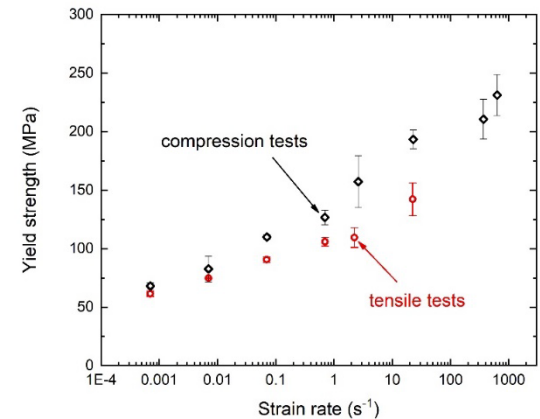
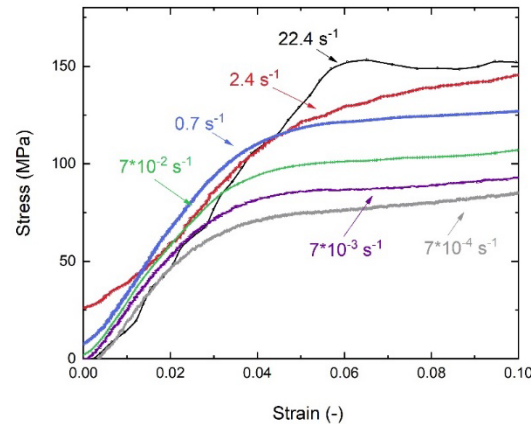
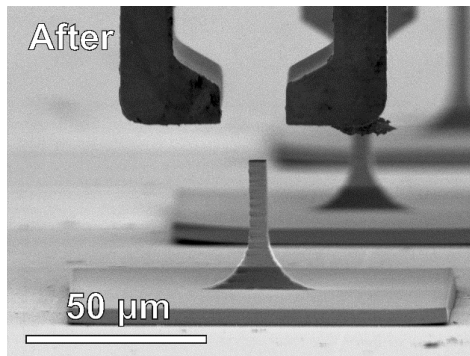


- Yield strength increases strongly with higher strain rates
- Higher temperatures lead to a significant drop in yield strength; this effect is less pronounced at higher strain rates

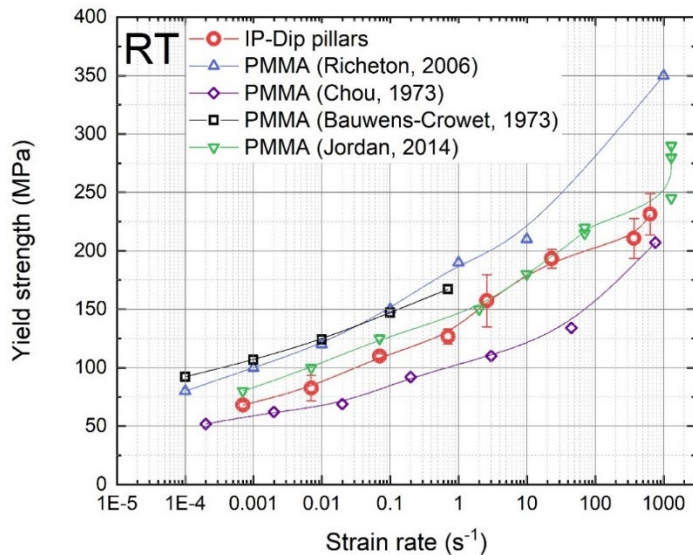
Micro-tensile test - Yield strength



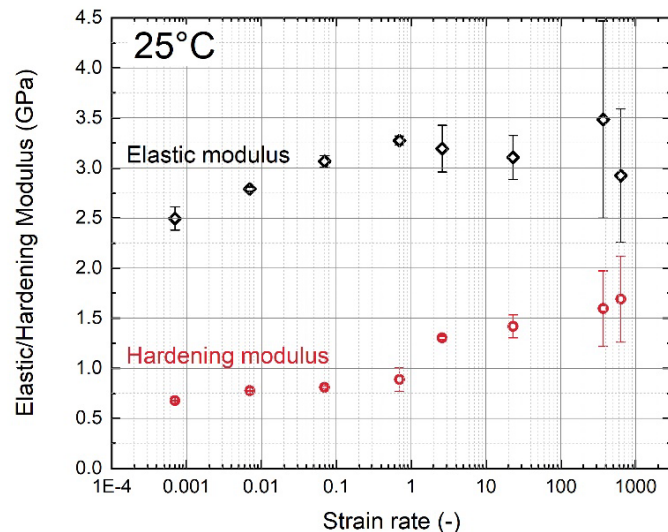
- Yield strength from tensile tests is slightly lower than from compressive tests
- Both tests give a similar strain rate dependence



Experimental observations

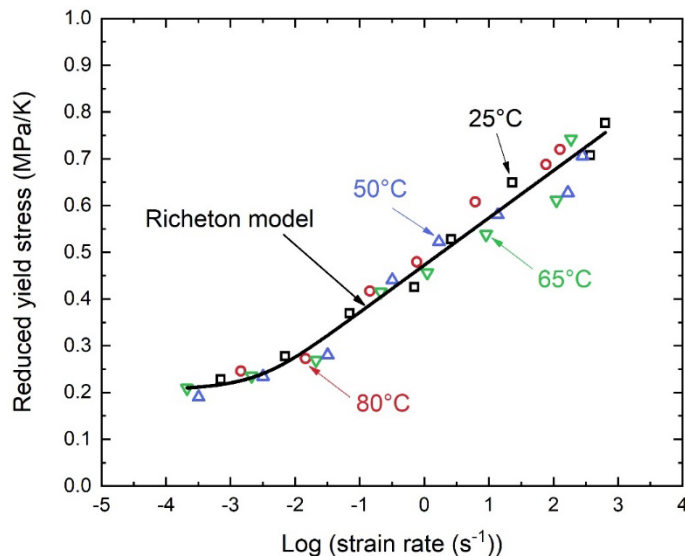
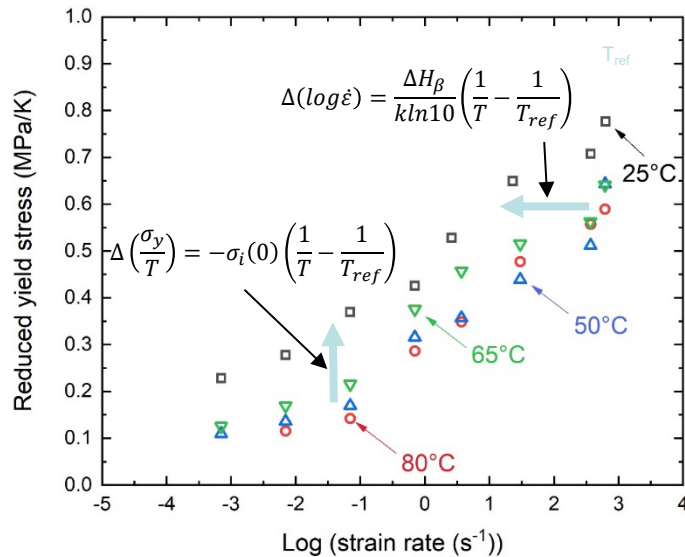


- PMMA closest chemical sibling to IP-Dip
- The strain rate dependency of yield strength in 3D printed polymer micropillars compares well to the data acquired in macroscopic compression tests of PMMA



IP Dip: Yielding occurs at higher strains and stresses, slope increases before and after yielding
→ Hardening modulus increases at higher strain rates

Strain rate - Temperature Superposition principle



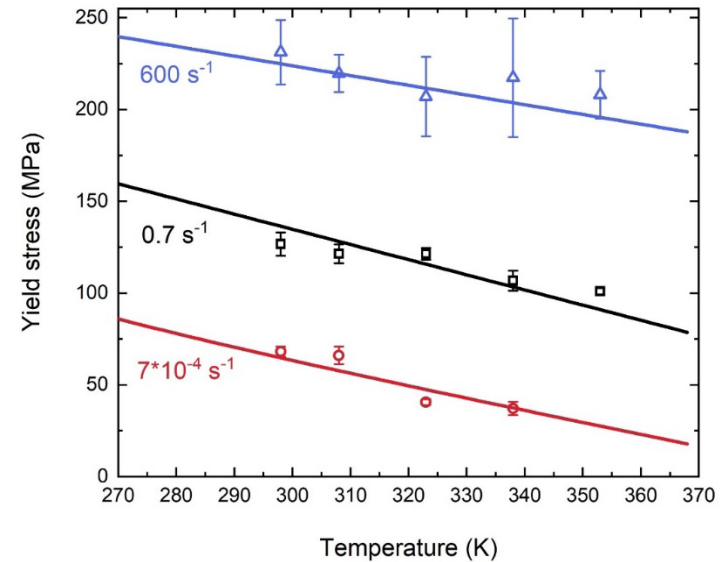
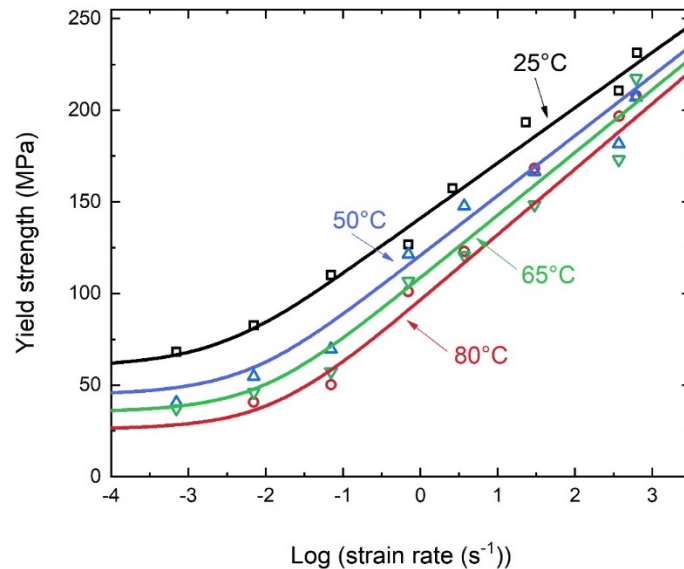
Richeton et al. (2006) propose a formulation for a cooperative model using an Eyring type equation to describe the principle of *Strain rate - Temperature Superposition* for the compressive yield strength in polymers

→ Yield stress increases with decreasing temperature and increasing strain rate

$$\frac{\sigma_y}{T} = \frac{\sigma_i(T_{ref})}{T_{ref}} + \frac{2k}{V} \sinh^{-1} \left(\frac{\dot{\epsilon}}{\dot{\epsilon}^*(T_{ref})} \right)^{\frac{1}{n}}$$

Data was fitted with the formula of Richeton model → parameters describing activation of yielding for the specific polymer are extracted

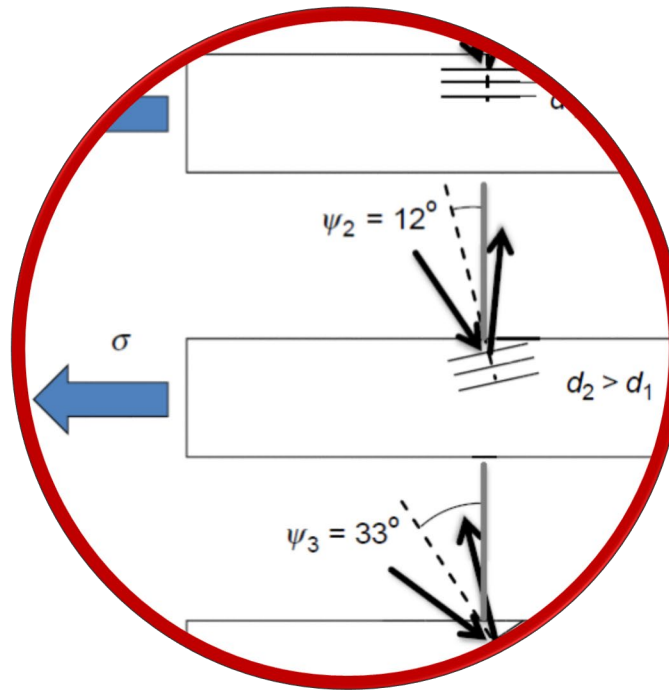
Strain rate - Temperature Superposition principle



Summary

- This is the first systematic experimental assessment on the micromechanical scale of yield strength and elastic modulus of 3D printed polymer structures investigating the effect of ***higher strain rates*** and ***temperature***.
- It could be shown that such tests can be conducted reliably ranging over 6 orders of magnitude of strain rate showing a strong strain rate dependency of yielding behaviour as well as elastic modulus. Measurements at elevated temperatures showed a strong reduction in mechanical properties of the polymer.
- A model based on the temperature-strain rate superposition principle developed for macroscopic testing was applied to describe the yielding behaviour on the microscopic scale allowing to predict properties beyond the measured scope.
- This work can be continued to more complex (lattice) geometries.

Synthesis and characterization of metal-ceramic microlattices



Controlling properties by architecture and size effects

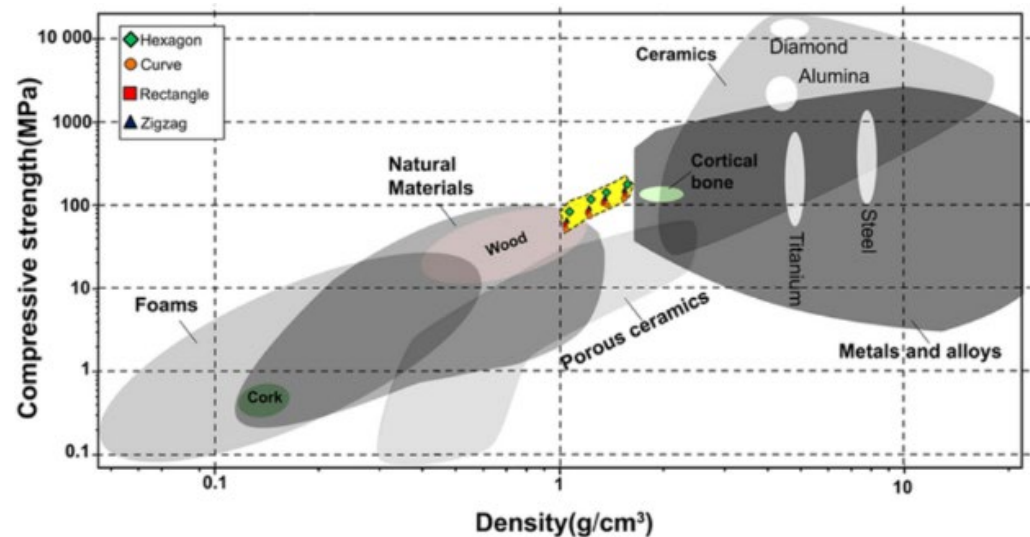


Metamaterials offer an opportunity to populate white areas in Ashby diagrams

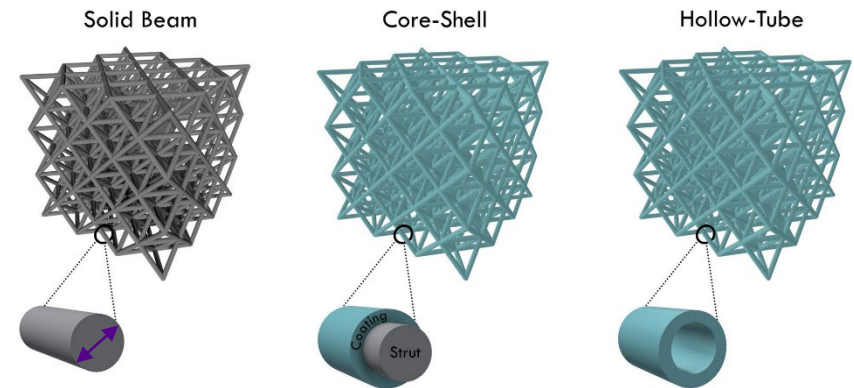
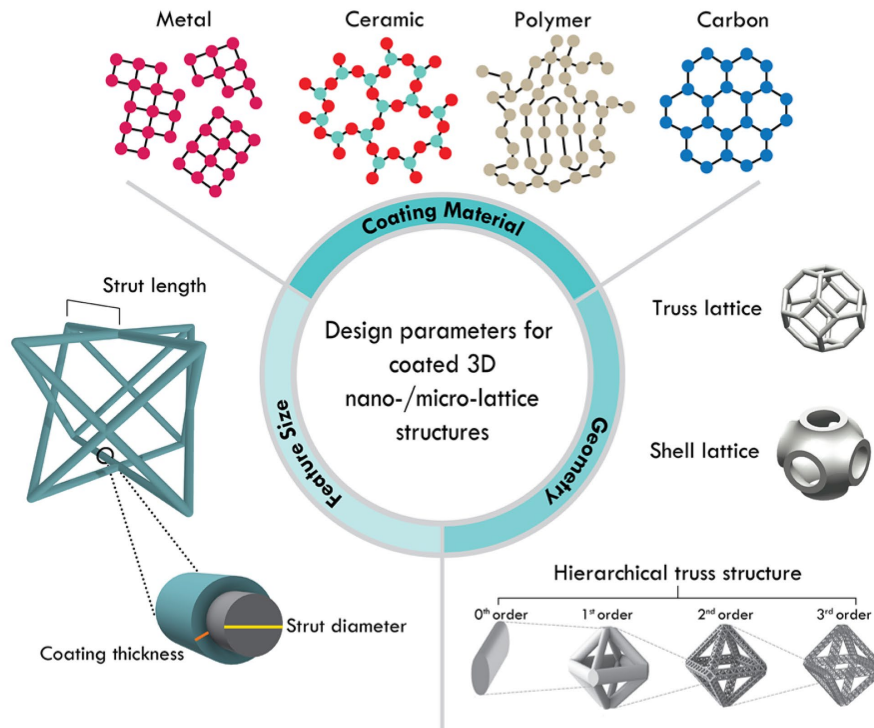
Superposition of architecture and size effects

Structures with tailored:

- Architecture
- Critical size
- Base material



Enhancing strength of micro-architectures



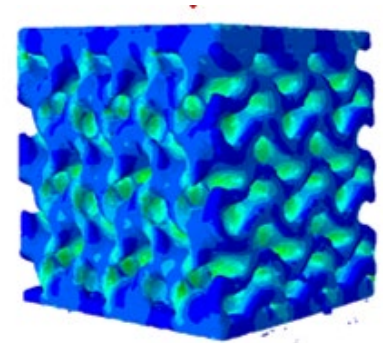
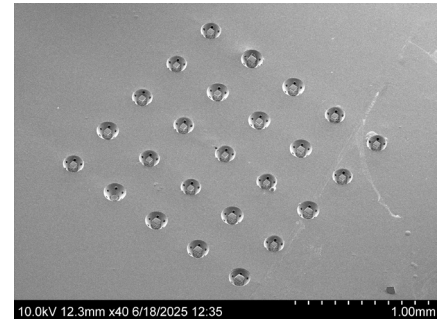
Exploit size effects and coating materials to achieve unique properties!

Adapted from: Garcia-Taormina et al. (2021) *J. Mater Res.*, 36(18), 3607-3627.

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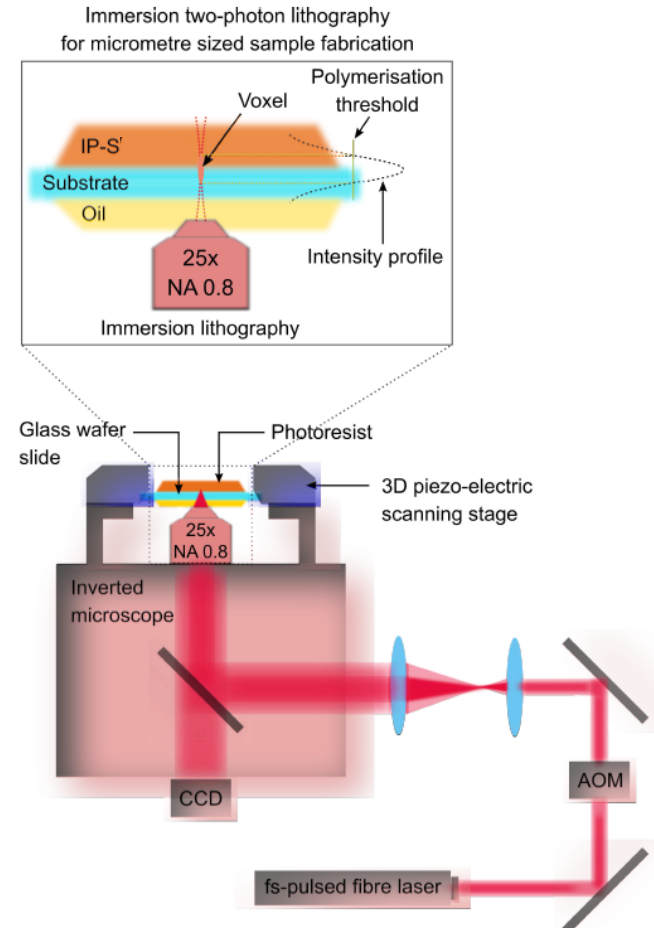
Study goals

- Develop additive manufacturing process for hybrid metal-ceramic microscale metamaterials
- Using a combination of synchrotron-based ptychography, SEM, micromechanical testing, and computational models, investigate
 - structure-property relationships and failure mechanisms
 - influence of grain structure, geometric imperfections, and defects on mechanical performance

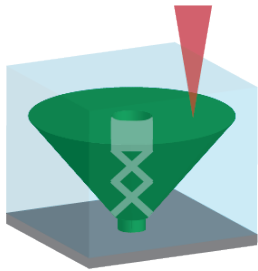
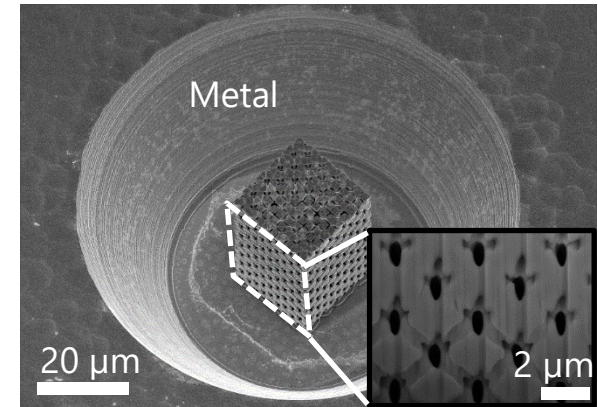
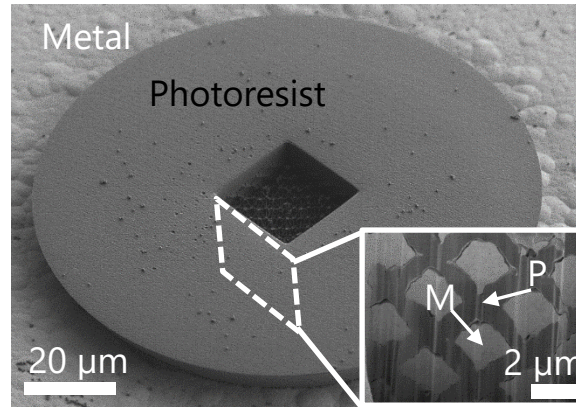
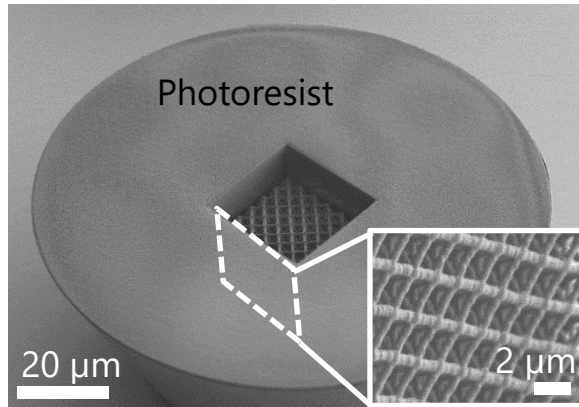


Two photon lithography

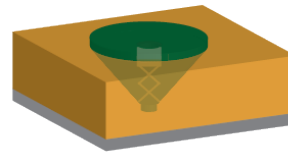
- Based on two photon absorption
- 2PP threshold determines voxel size
- Allows 3D printing below diffraction limit



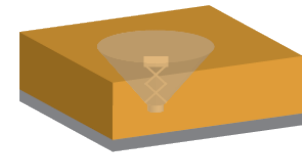
Template assisted electrodeposition



Two-Photon
Lithography



Electrodeposition



Plasma
Etching

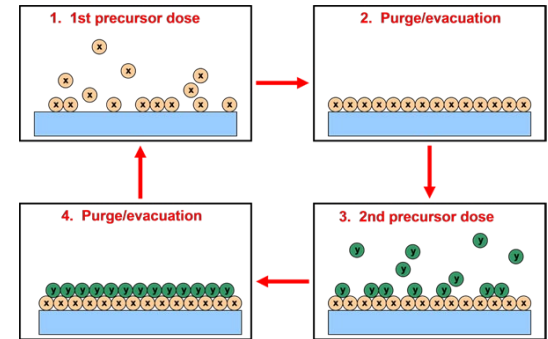
Atomic layer deposition

Atomic layer deposition:

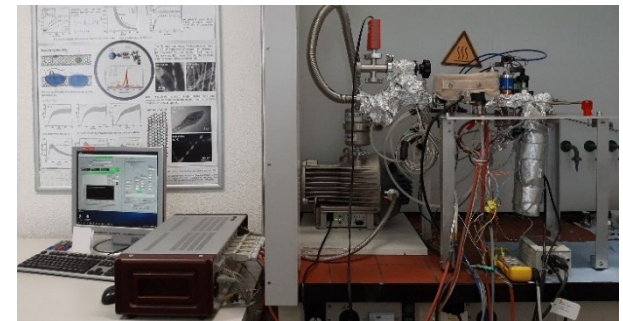
- Self-limiting process
- Sub-nm thickness control
- Conformal coatings

Process conditions:

- Deposition temperature 65°C
- 1000 cycles of
 - Trimethyl aluminum
 - H₂O
- Al₂O₃ film with 134 nm thickness

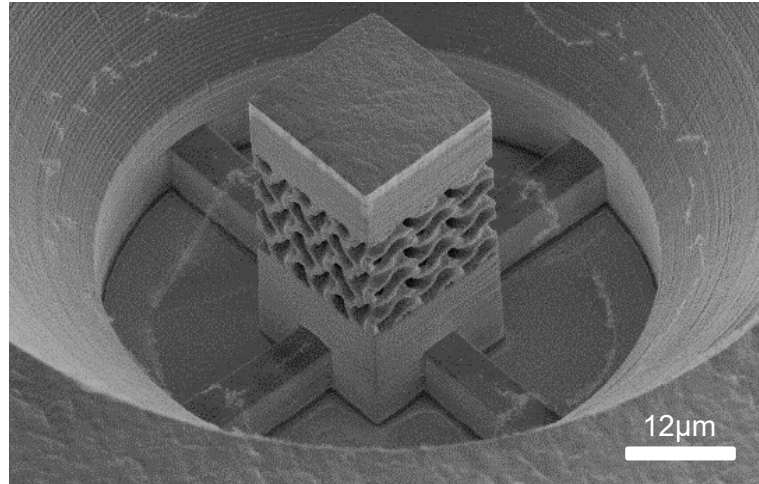


Schematics of the ALD process



ALD reactor at lab 206

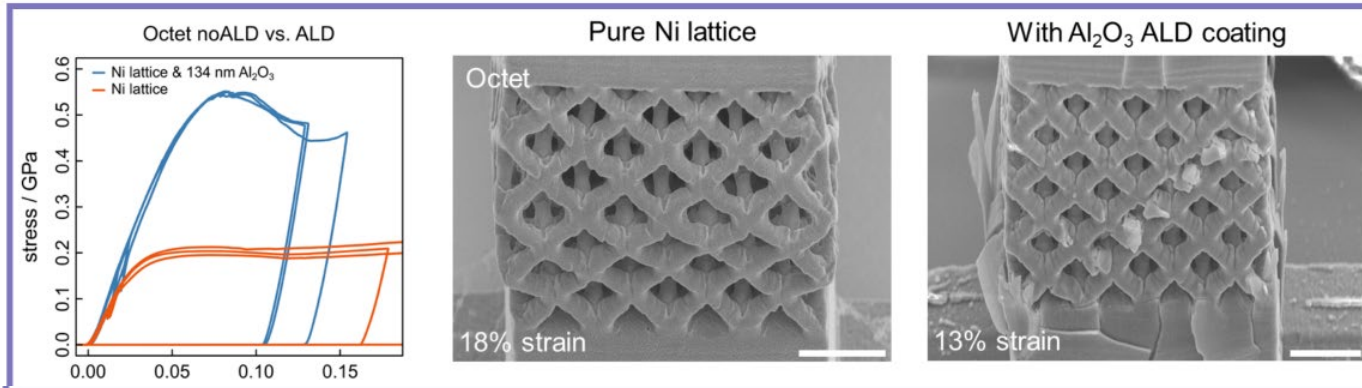
Resulting structures



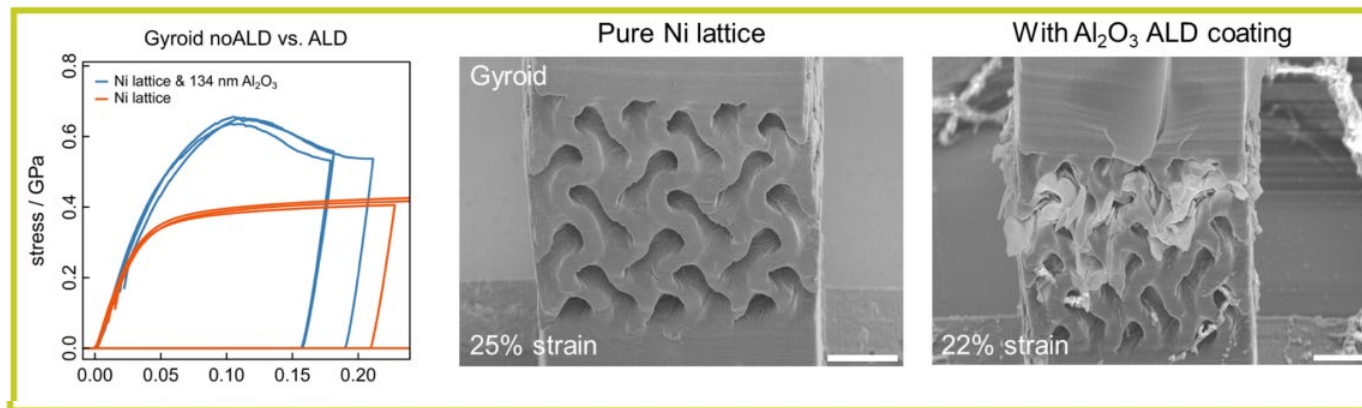
- Template assisted electrodeposition allows synthesizing metal-ceramic microarchitectures with micrometric feature sizes
- Process results in high quality, repeatable structures

Microcompression experiments

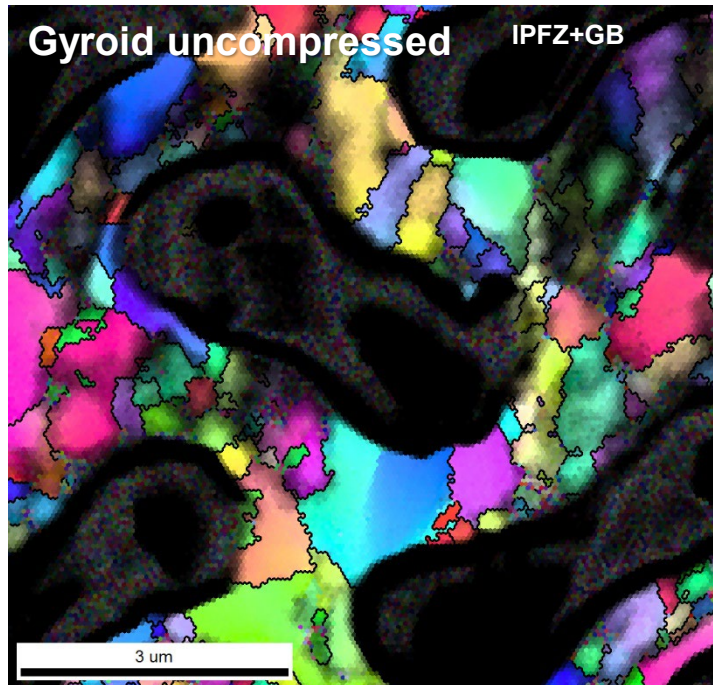
Strut based Ni vs. Ni-Al₂O₃ lattices



Surface based Ni vs. Ni-Al₂O₃ TPMS metamaterials

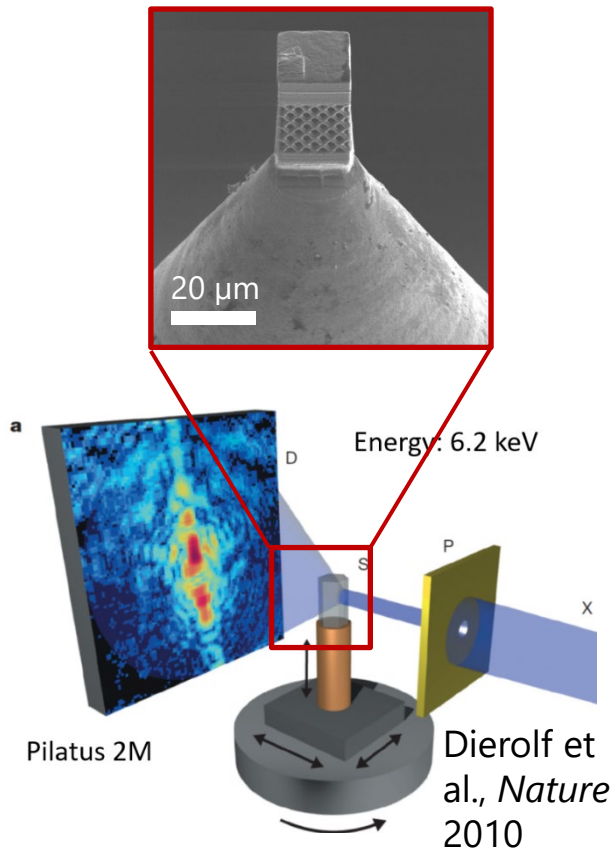


Microstructural analysis by TKD



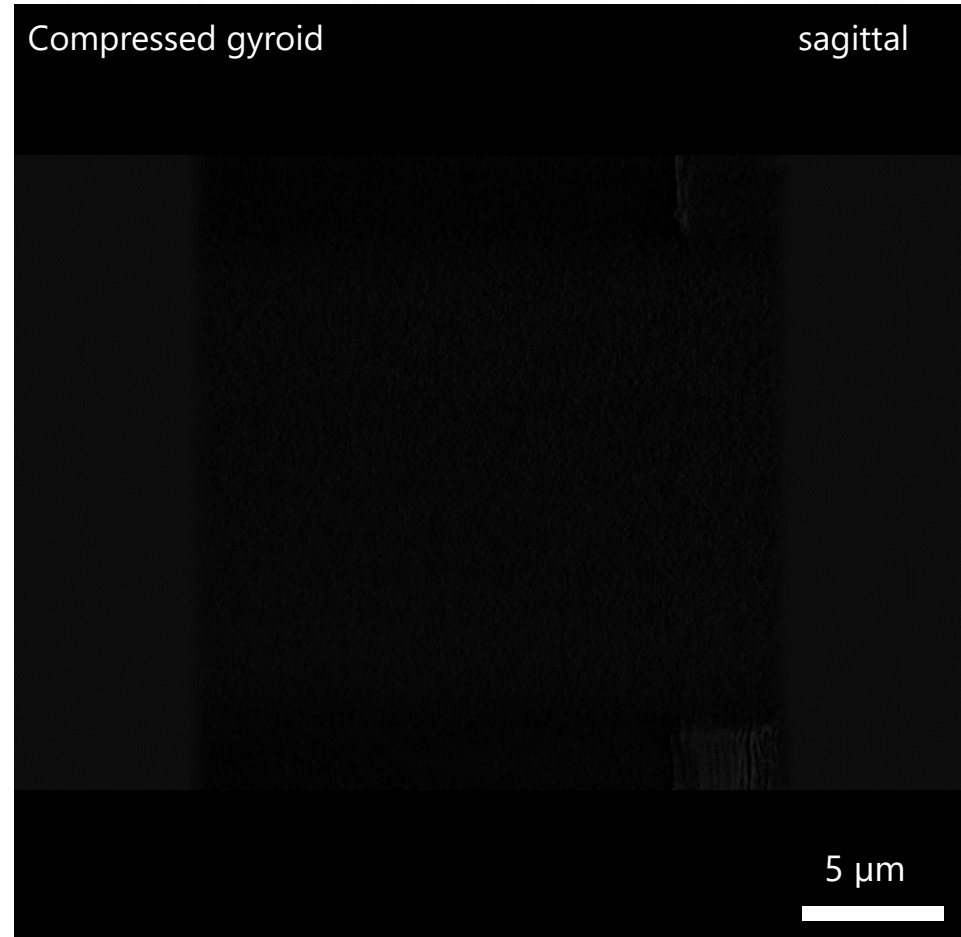
- Heterogeneous grain size between 100nm and 1μm in undeformed structure
- Deformation leads to localized grain refinement

Defect distribution and failure mechanisms



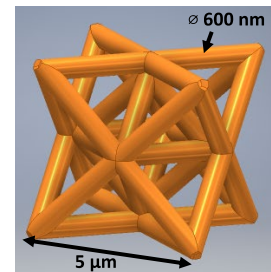
Compressed gyroid

sagittal

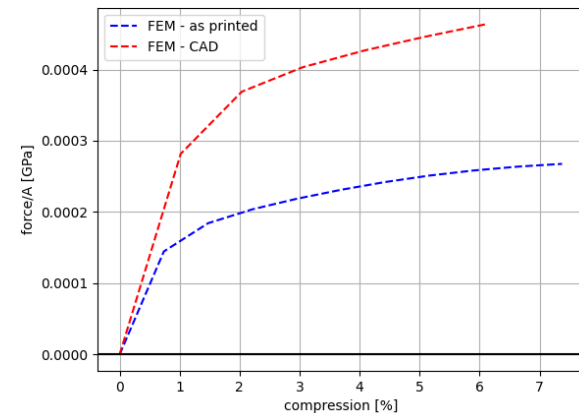
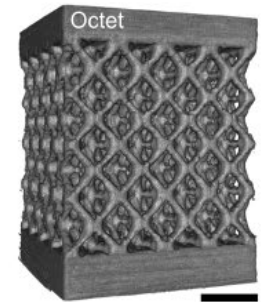


Mechanical modeling - imperfections

- Segmenting of NanoCT image dataset allows extracting the real geometry of the microstructure.
- Automatic meshing procedure from segmented images allows simulating realistic geometries.
- Comparison of ideal input structures to real 3D printed shapes shows large effect of geometric imperfections.

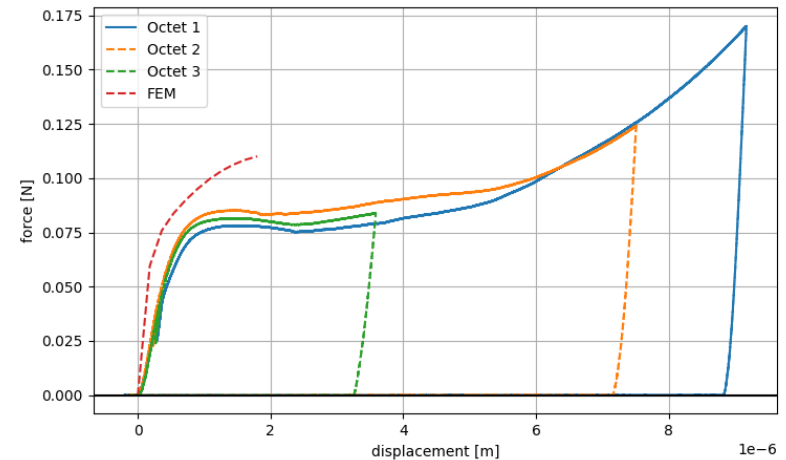
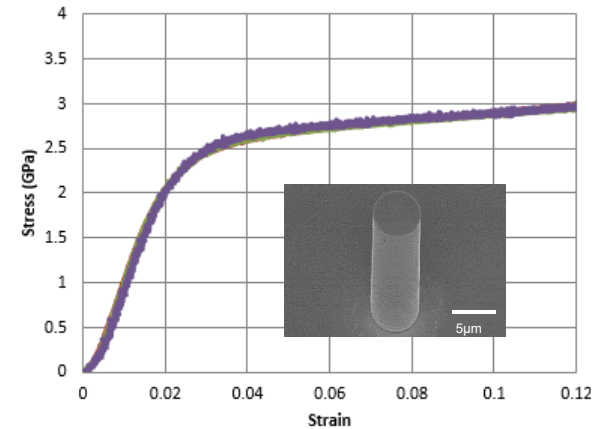


vs



Mechanical modeling - grain size

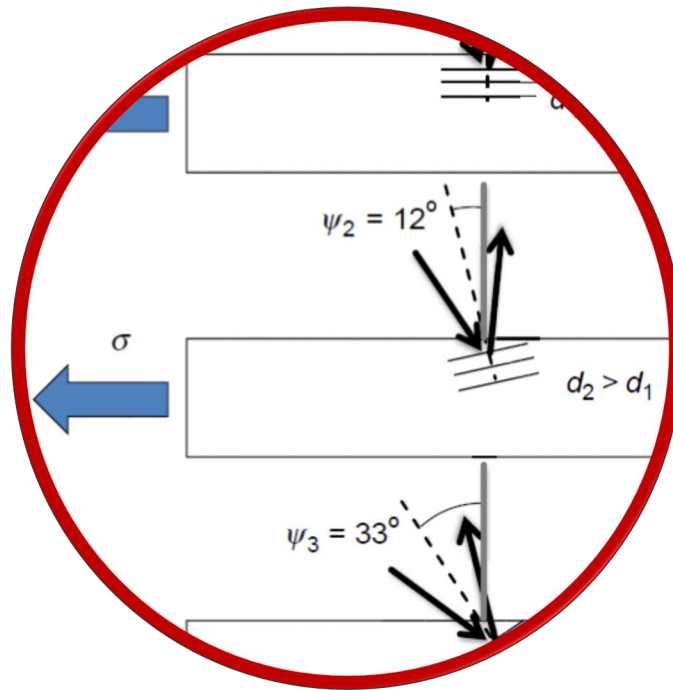
- Yield stress measured during micropillar compression was used as input for mechanical simulations.
- The large overestimation of apparent properties is caused by the differences in grain size:
 - 28 nm for the micropillars
 - >100 nm for the microlattice structures
- Adjusting the yield stress using Hall-Petch relationship yields a good fit between yield stress prediction and measurement.



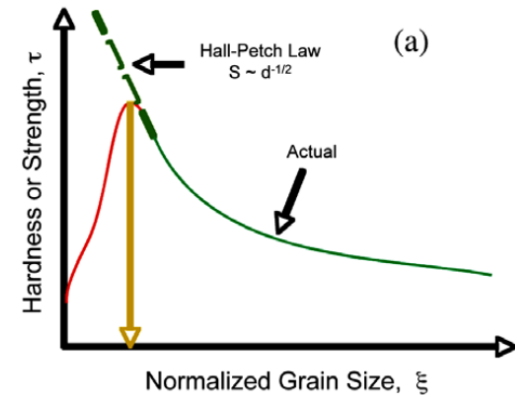
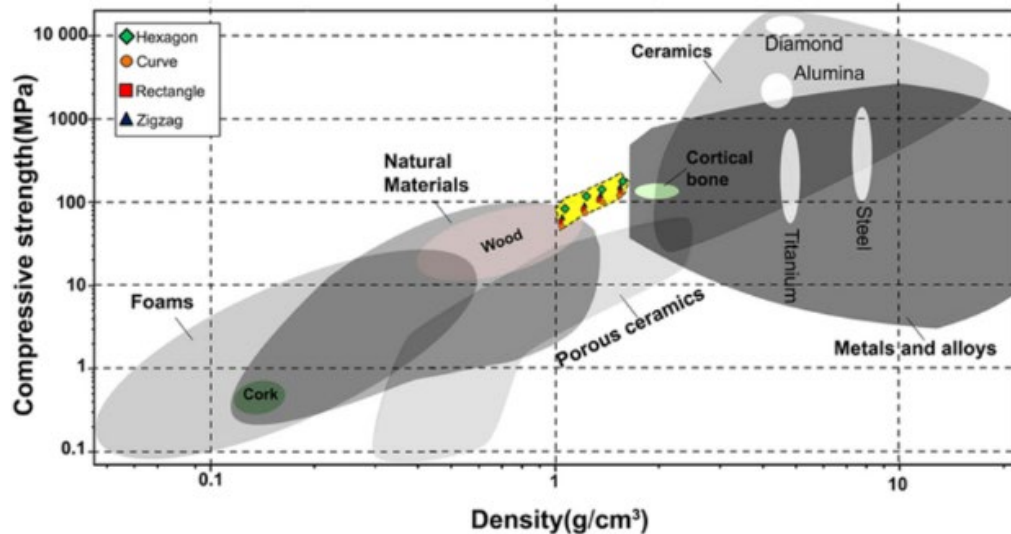
Summary

- Exploiting architecture and size effects in the design of mechanical metamaterials allows combining high strength and toughness
- Microscale additive manufacturing by TAED and ALD allows creating hybrid metal-ceramic microarchitectures and lattices
- Under mechanical loading, the Alumina coating enhances the apparent strength of the microlattice
- NanoCT shows that delamination of the Alumina coating enables ductile deformation of the metal lattice after fracture of the ceramic coating
- Mechanical modelling based on NanoCT imaging highlights the effect of geometric imperfections and grain size on the apparent behaviour
- After validation, mechanical models allow *in silico* design of architectures

Synthesis and characterization of nanocomposites

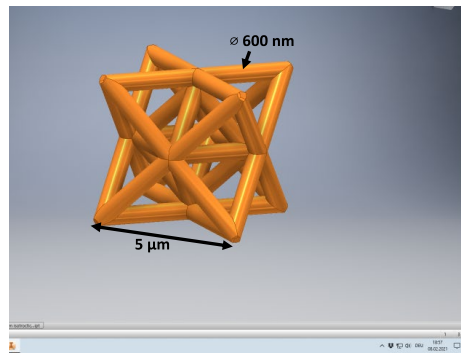


Controlling properties by architecture and size effects

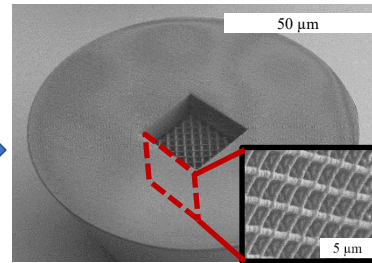


- Nanocrystalline metals feature high strength, but properties deteriorate at elevated temperature.
- Ceramics are strong in compression, stable at high temperature, and light, but brittle.
- Metal-ceramic nanocomposite to improve specific strength and thermomechanical properties?
- How to control the architecture of the nanocomposite at the nanoscale?

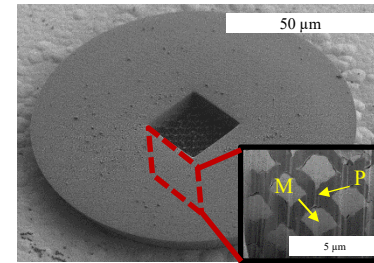
Nanocomposite synthesis by TAE and ALD



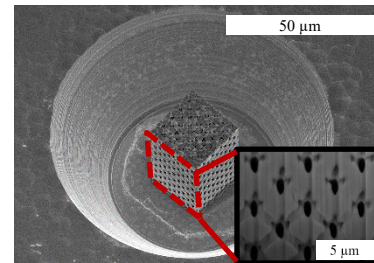
making up the reinforcement phase or the composite



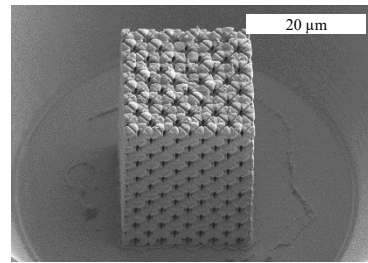
Writing the lattice geometry into large craters (both polymer) using Two-Photon Lithography



Electroplating of metallic film onto the whole surface of the chip



Removing the polymer templates by plasma etching



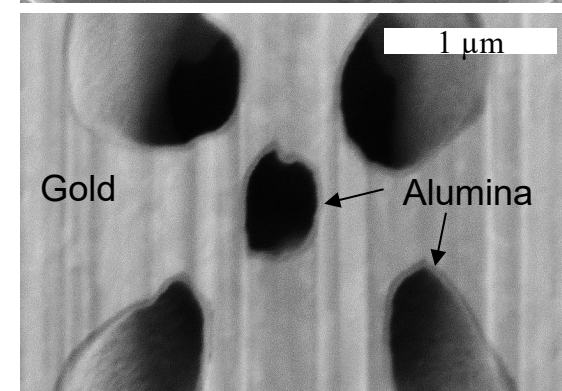
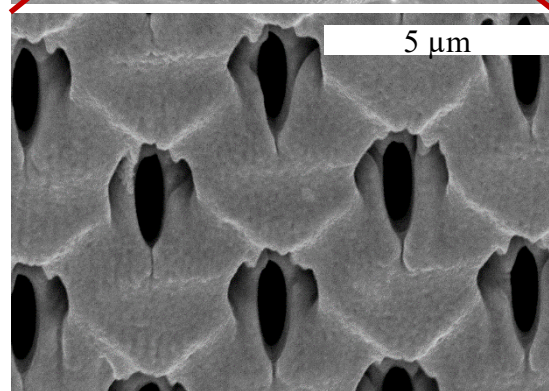
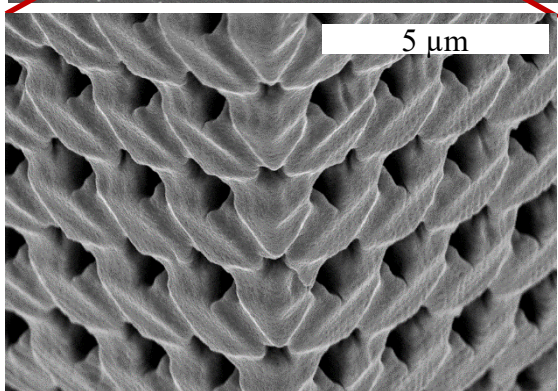
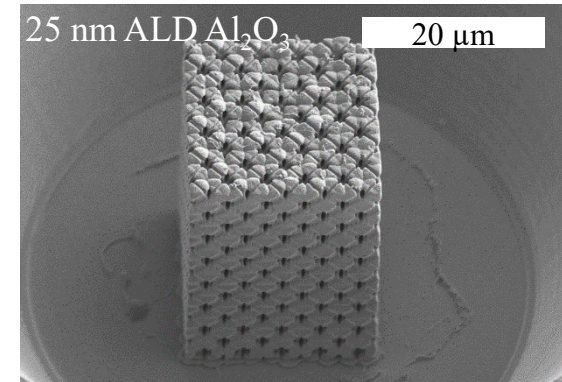
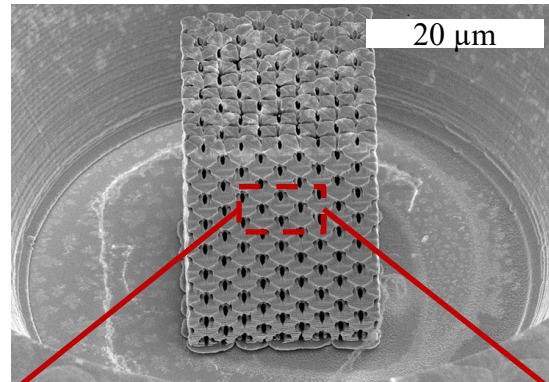
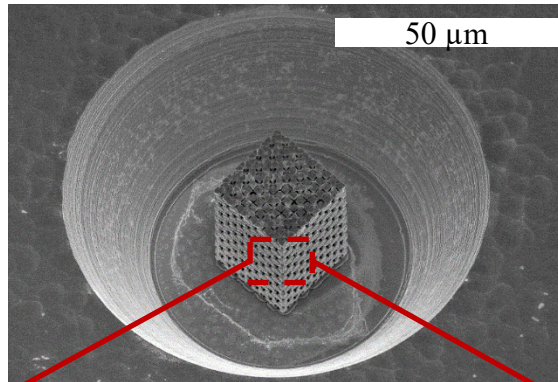
Infiltrating the metallic structures with the ceramic phase by Atomic Layer Deposition



Mechanical testing and modelling

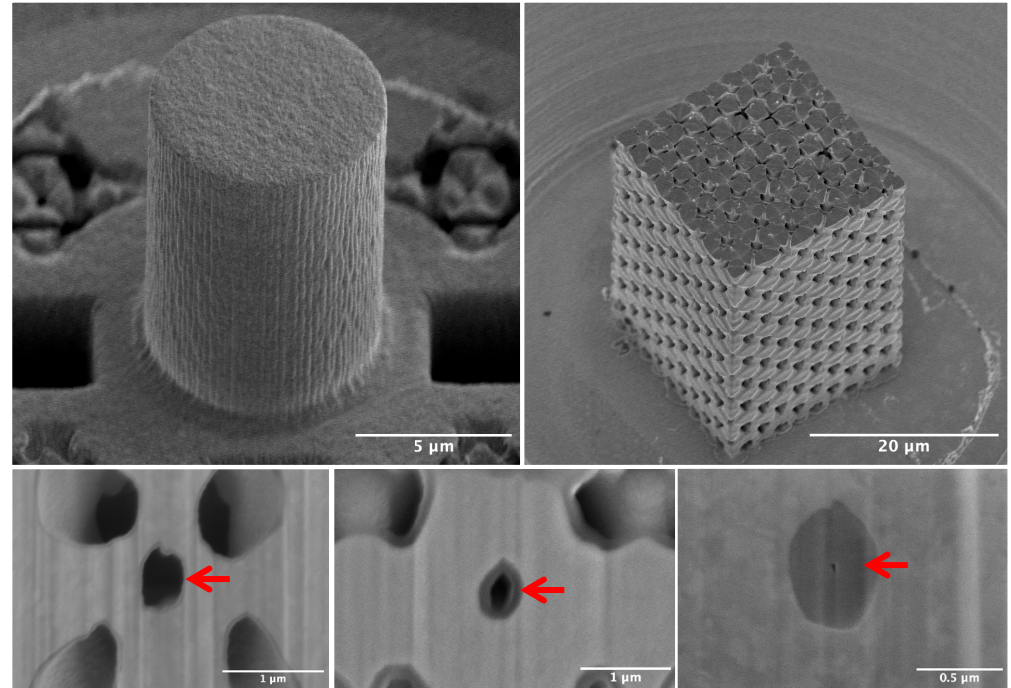
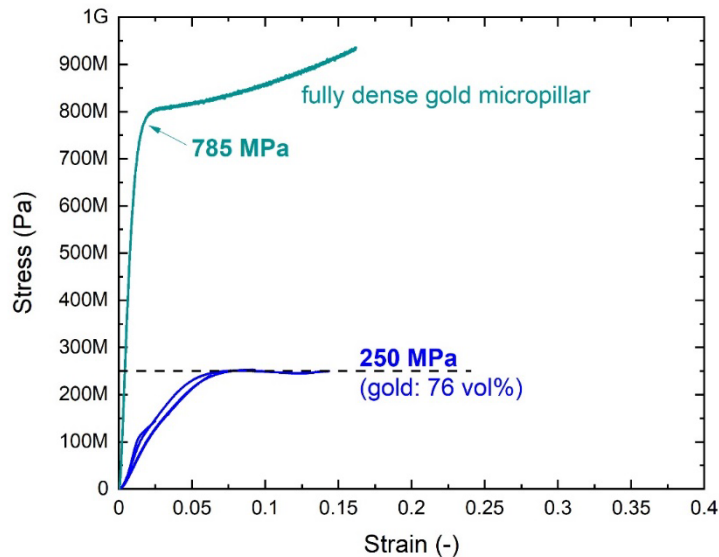
- Combination of template assisted electrodeposition of nc Au with atomic layer deposition of Al_2O_3 allows precise control of the ceramic reinforcement phase

Quality of the resulting structures



- Repeatable process, precise control of the reinforcement, but local flaws and roughness

RT mechanical performance of the nanocomposite



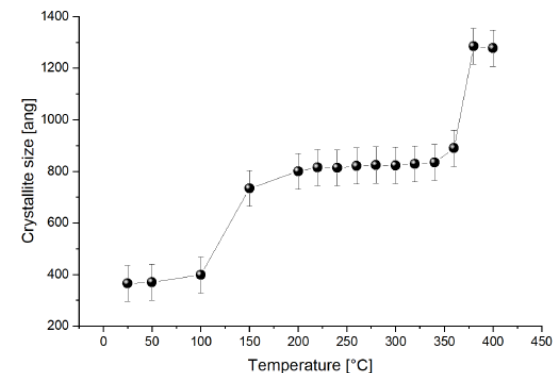
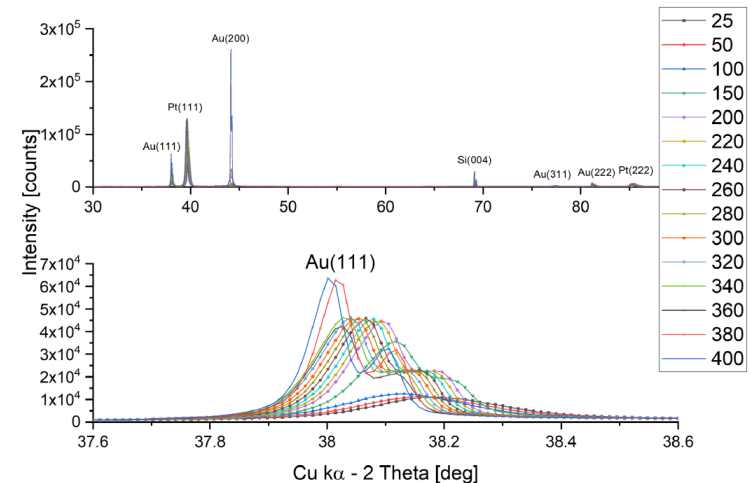
- Strength of porous Au matrix is significantly lower than the dense micropillar
- Al_2O_3 reinforcement increases specific strength by 25% from 41 to 55 MPa/(g/cm³)

Thermal stability of the nc Au matrix



X-ray diffraction to assess thermal stability of electrodeposited nc Au films:

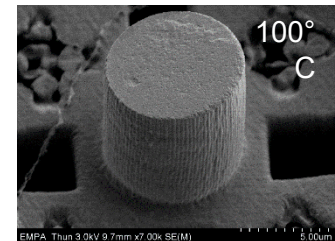
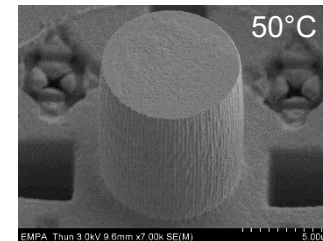
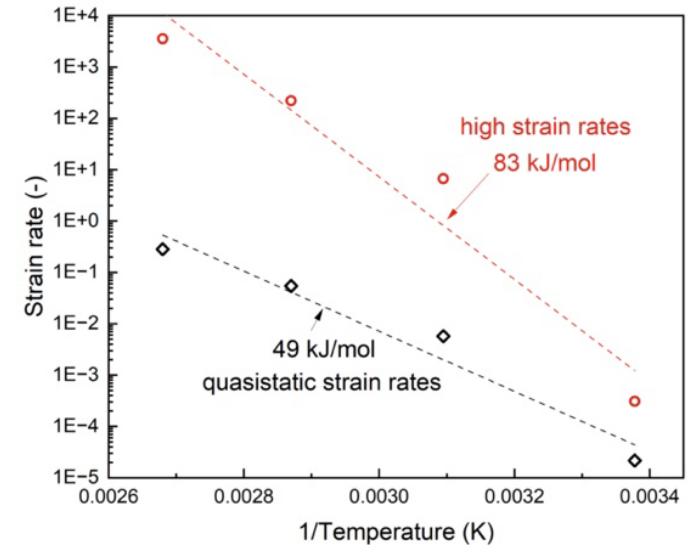
- Temperature range: 25°C to 400°C
- 10 minutes stabilization time
- Above 100°C the peak area increases and the peak width decreases corresponding to a change in grain size.
- Increase in calculated crystallite size:
 - 40nm for 25-100°C
 - 80nm for 150-350°C
 - 140nm for >400°C.
- Stable grain size in the range 25°C to 100°C



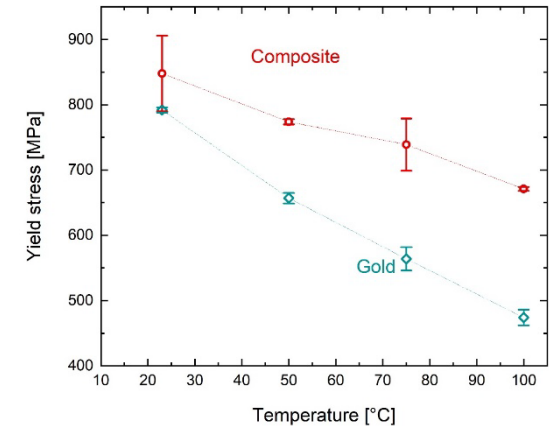
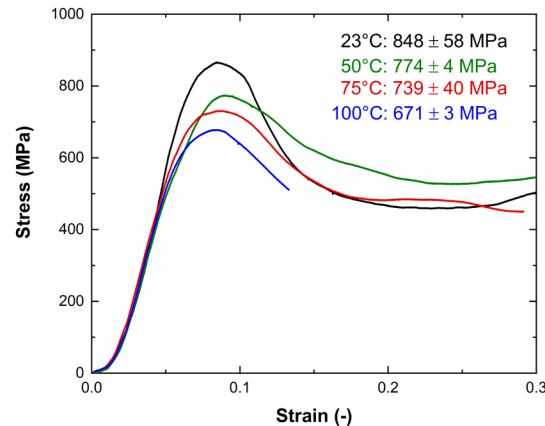
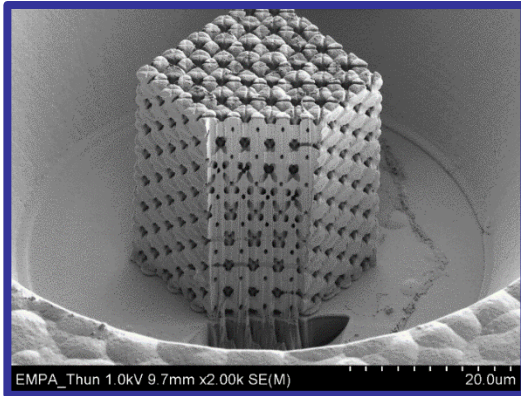
Thermal activation analysis of the nc Au matrix



- Microcompression of TAE pillars between 25°C and 100°C and $5 \cdot 10^{-5} \text{ s}^{-1}$ to 500 s^{-1}
- nc Au micropillars (grain size 40nm) exhibit a high room temperature strength up to 16Pa at high strain rates
- Strain rate sensitivity:
 - Higher m for quasistatic strain rates up to 1 s^{-1}
 - Higher m at elevated temperature (0.025 at 23°C vs 0.063 at 100°C)
- Lower activation volume at higher strain rates and at elevated temperature
- Higher activation energy at higher strain rates
- Activation parameters suggest change in deformation mechanism: grain boundary mediated plasticity at low strain rates and intra-granular plasticity mediated by heterogeneous dislocation nucleation at high strain rates



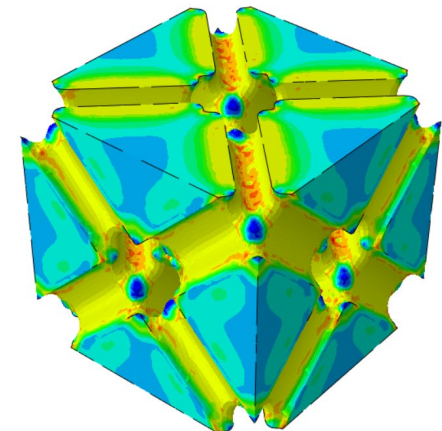
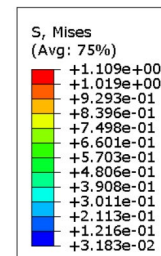
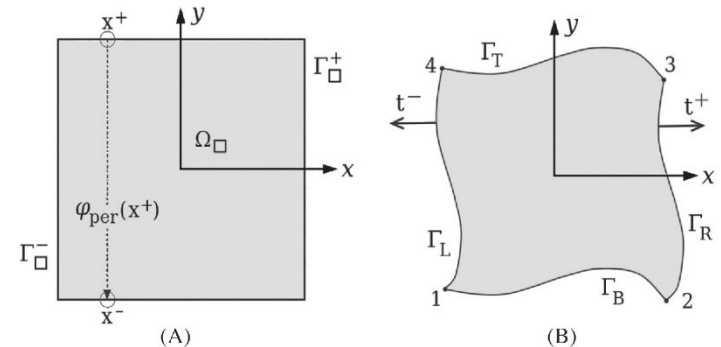
Thermomechanical performance of the nanocomposite



- Nanocomposite shows consistent strain softening behaviour at all temperatures suggesting structural damage
- Strength of nc Au pillars at 100°C is reduced by 40% compared to 25°C
- Reduction in strength of the nanocomposite with temperature is 50% lower than for pure Au.
- Specific strength of the nanocomposite at 100°C is 44.1 MPa/(g/cm³) compared to 23.3 MPa/(g/cm³) for pure Au.
- This corresponds to an increase by almost 90% in specific strength at 100°C



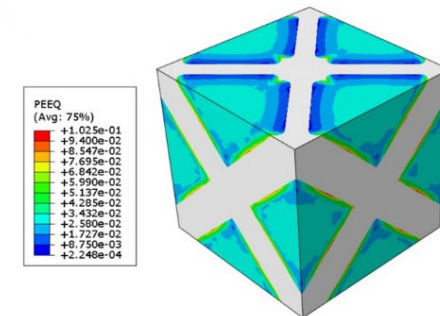
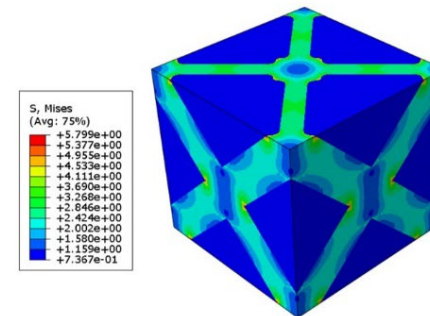
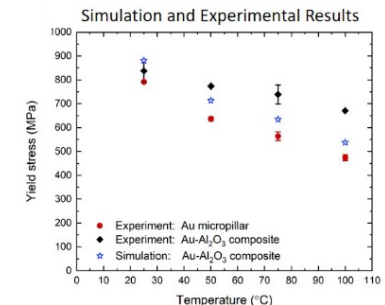
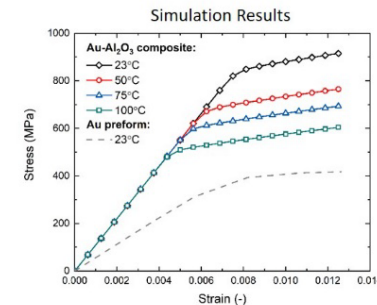
- Unit cell of the nanocomposite with idealized geometry (no roughness or flaws)
- Periodic boundary conditions representing an infinite periodic arrangement of unit cells in a material
- Elastic properties from nanoindentation:
 - Au: $E=98 \text{ GPa}$, $\nu=0.42$
 - Al_2O_3 : $E=150 \text{ GPa}$, $\nu=0.25$
- $\sigma_y(T)$ of nc Au from micropillar compression experiments
- 2.5% uniaxial strain, quasistatic strain rate
- Assumptions: Ideal interface between Au and Al_2O_3 , Al_2O_3 deforms elastically



Thermomechanics - experiment vs simulation



- Quantitative agreement of increase in σ_y due to ceramic reinforcement at 23°C with experiments
- Decrease in yield stress with temperature comparable to experiments on nc Au pillars (40%), but not nanocomposite (20%)
- Highly stressed regions at the nodes of the octet lattice. Almost 6 GPa of stress might lead to local inelastic deformation or cracking in Al_2O_3 .
- Plastic deformation in the Au matrix present in the whole volume, but significantly more pronounced near the Au- Al_2O_3 interface
- Limitations: simulated periodic material vs. Al_2O_3 coated structure in experiment, perfect interface, absence of flaws



Summary



- Novel technique to fabricate metal-ceramic nanocomposites with highly controlled architecture presented on $\text{Au-Al}_2\text{O}_3$ model system.
- The fabrication methodology is scalable, repeatable, and versatile. A main limitation is the resolution and throughput of two-photon-lithography.
- Compression studies on deposited nc Au pillars revealed remarkable yield stress reaching 1 GPa and strong dependence on temperature and strain rate.
- Activation parameters indicate a change of the deformation mechanism from grain boundary sliding at low strain rates to dislocation-mediated processes at high strain rates.
- Ceramic lattice reinforcement improved the specific strength of the composite by 25% at RT and 90% at 100°C.
- Modeling of periodic unit cells provides insights into locations of highest stress and plastic strain and allows further optimizing the nanocomposite architecture.
- This work demonstrates the potential of the combination of TAE and ALD for creating tailored nanocomposite coatings and microspecimens with improved thermomechanical performance.