Mechanical size effects: Experiments and mechanisms

By

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References

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- Bacon, D. J., and D. Hull. Introduction to Dislocations. New York, NY: Pergamon Press, 1984. ISBN: 9780080287201
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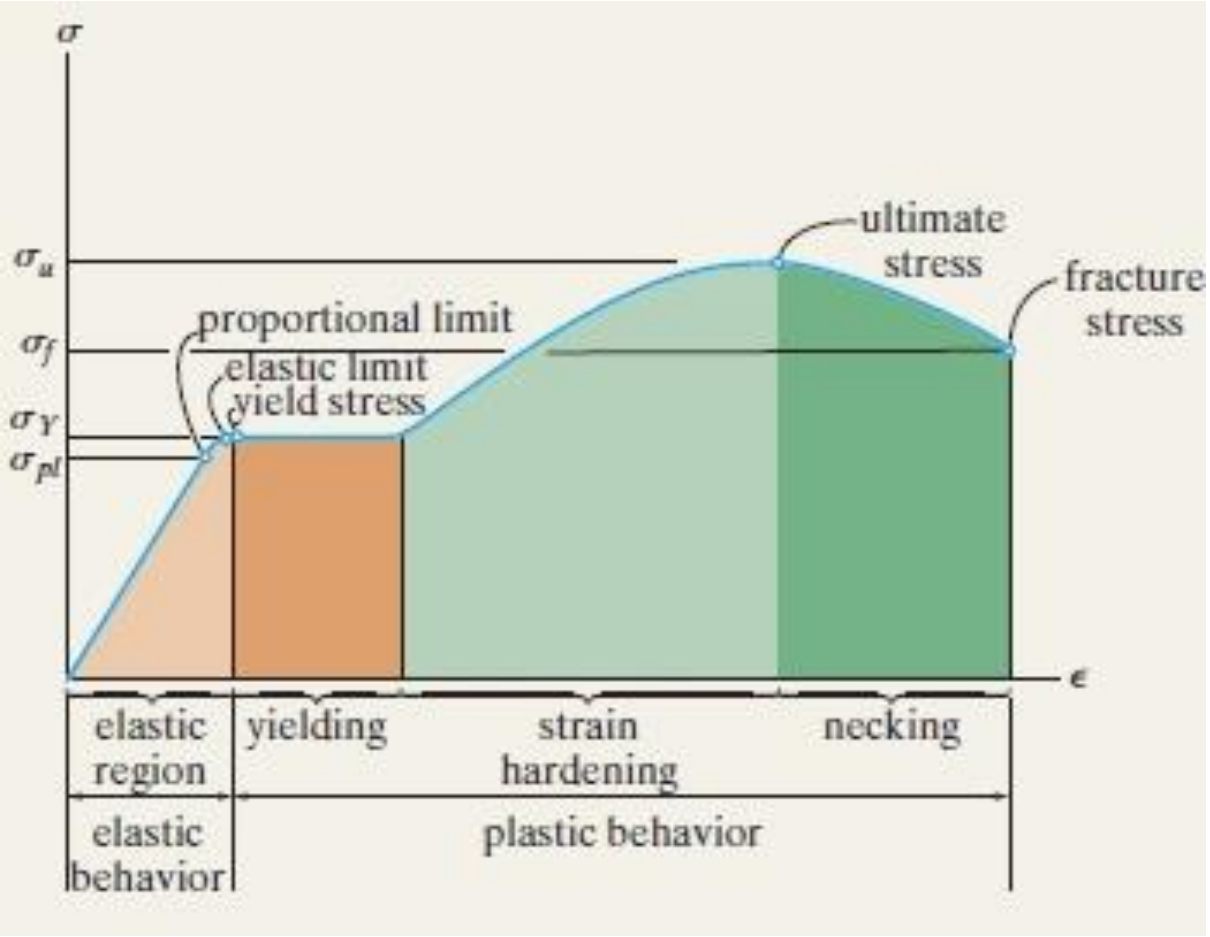
- B R Lawn and T R Wilshaw. Fracture of Brittle Solids.

- Reviews: J. Greer et al., O. Kraft et al., G. Dehm et al., A. A. Volinksy. B. N. Jaya and V. Jayaram

Revision


σ
σu
σf
σY
σpl
ultimate stress
fracture stress
proportional limit
elastic limit
yield stress
ε
elastic region
yielding
strain hardening
necking
elastic behavior
plastic behavior

Mechanical size effects – why do they occur?

Continuum mechanics (Also thought of as ‘bulk’ / macro – mechanics)

- Materials are assumed to be devoid of microstructural discontinuities
- Materials can be subdivided into infinitesimal elements with the same properties

Property ≠ f (size)

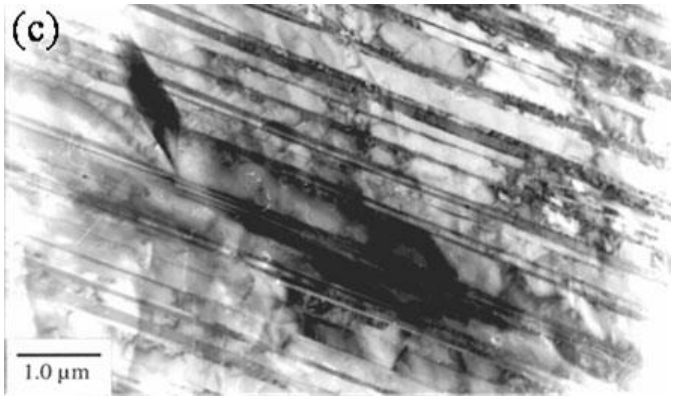
- Several observations revealed that materials behave differently at small length scales, both during intrinsic and extrinsic miniaturization

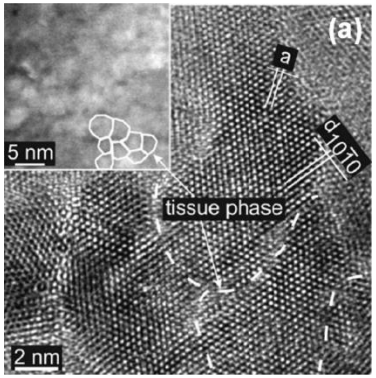
- Testing materials at the desired length scales using conventional testing methods – Miniaturization!

Motivation: Why study size effects?

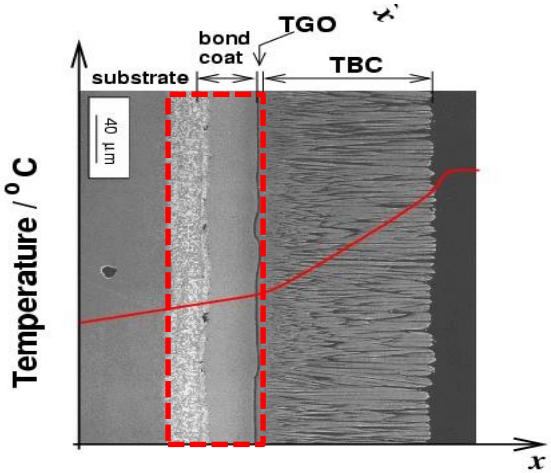
Fundamental changes in material's mechanical response observed with:

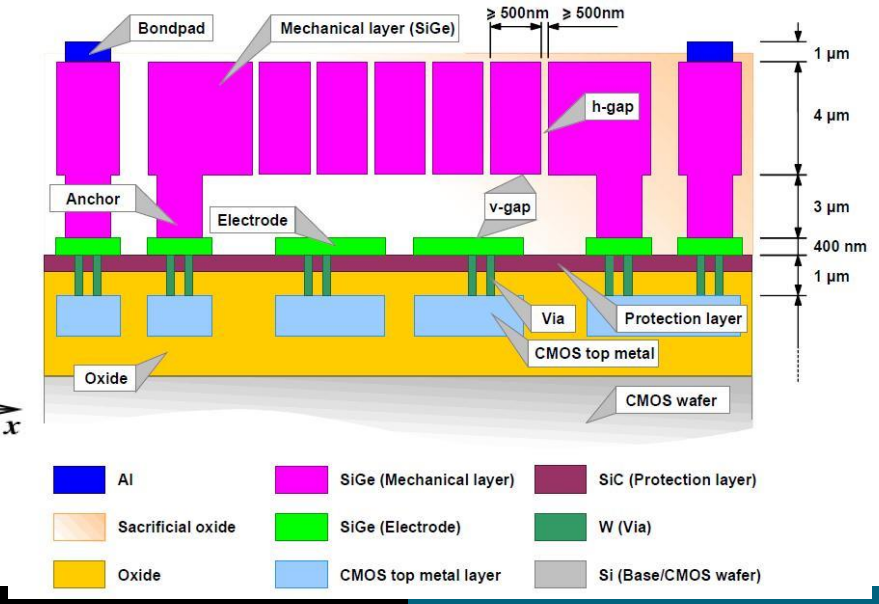
1. Decreasing internal microstructural length scale (intrinsic)

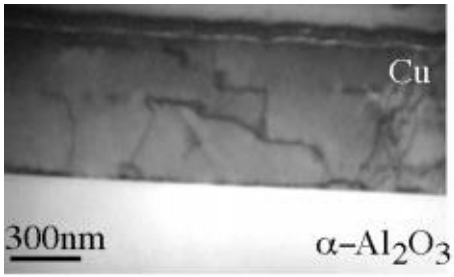




2. Decreasing external dimensions of devices and applications (extrinsic)







Motivation: Age of miniaturisation

Nanostructured materials have potential applications as :

- Builiding block for electronic devices like silicon nanowires in integrated circuits (mech. integrity, carrier mobilities under strain)

- ZnO nanowires in “nanogenerators”

(bending strength, piezoelectric properties)

- Building blocks in nanoelectromechanical systems

Magnetic nanowires for the artificial nose/ear projects

- End-effectors for scanning probe tips

(tip-enhanced Raman spectroscopy, high-aspect ratio tips)

Motivation: Why study size effects?

- Academic: To discern fundamental material behavior, which is increasingly being seen to be different at smaller length scales. Macroscopic single crystals in sizes that are suitable for conventional testing techniques are difficult to process or unaffordable.

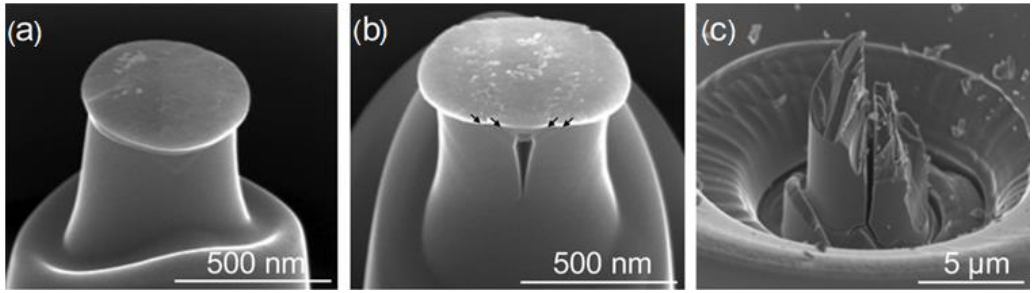
- Industrial:

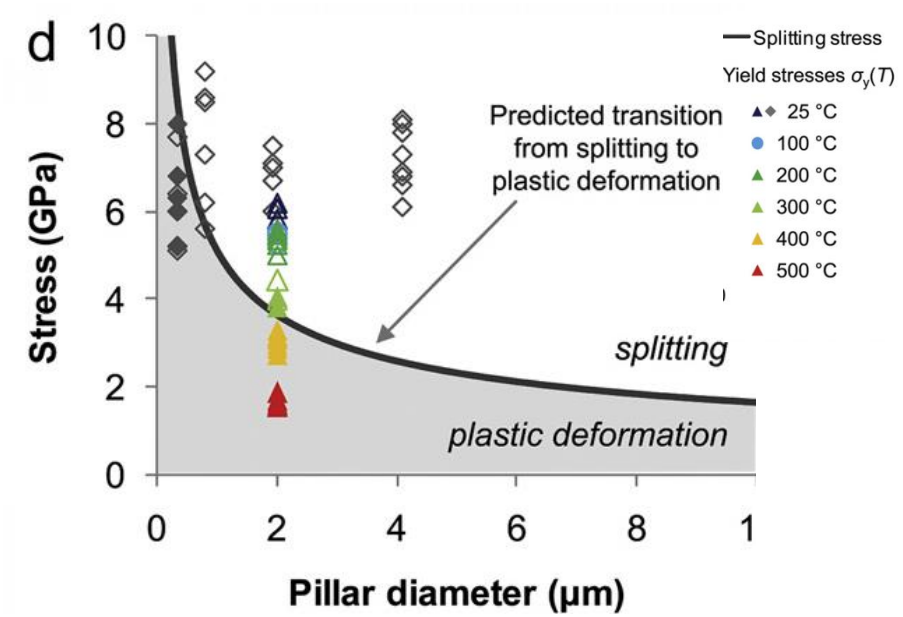
- Nanomaterials (with awesome properties) are impossibly difficult to obtain in bulk form currently. They have to be studied at the small-scale in free-standing or thin film form.
- Increasing device miniaturisation in the micro-electro-mechanical space routinely uses wafers and thin films, whose performance is directly connected to their behavior at these length scales

Size effect (extrinsic): decreasing specimen dimension

DBTT due to sample size

Si (100) Increasing pillar diameter

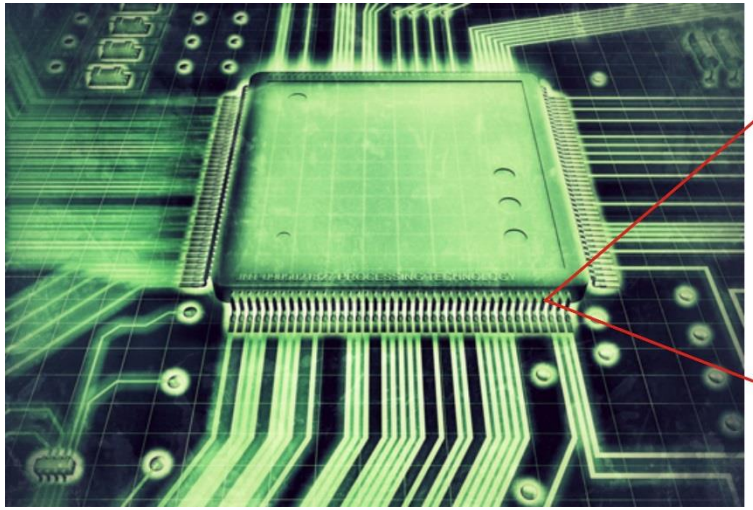


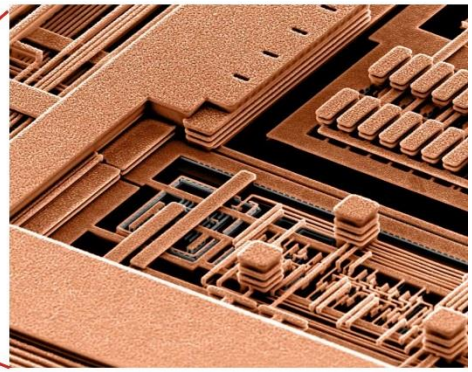


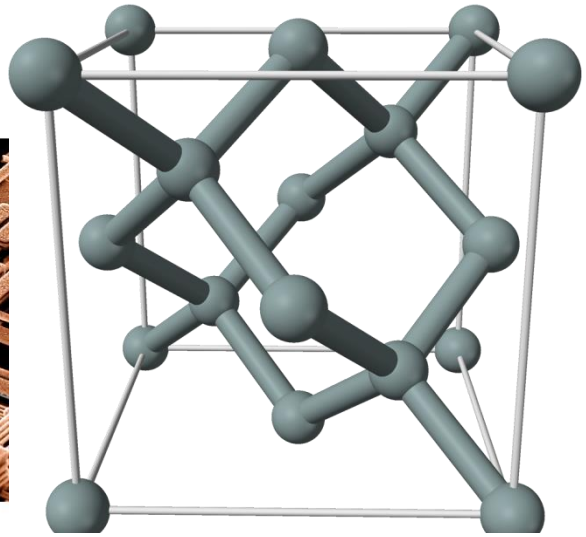
S. Korte et al., Int. J. Plasticity., 2011

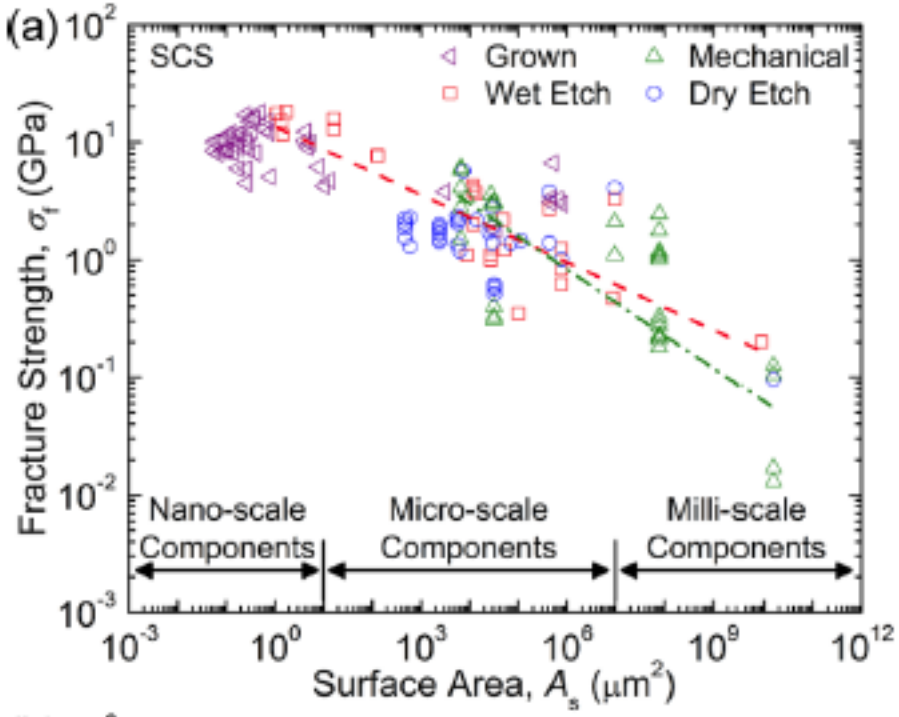
Device miniaturization and its sid(z)e effects

Si





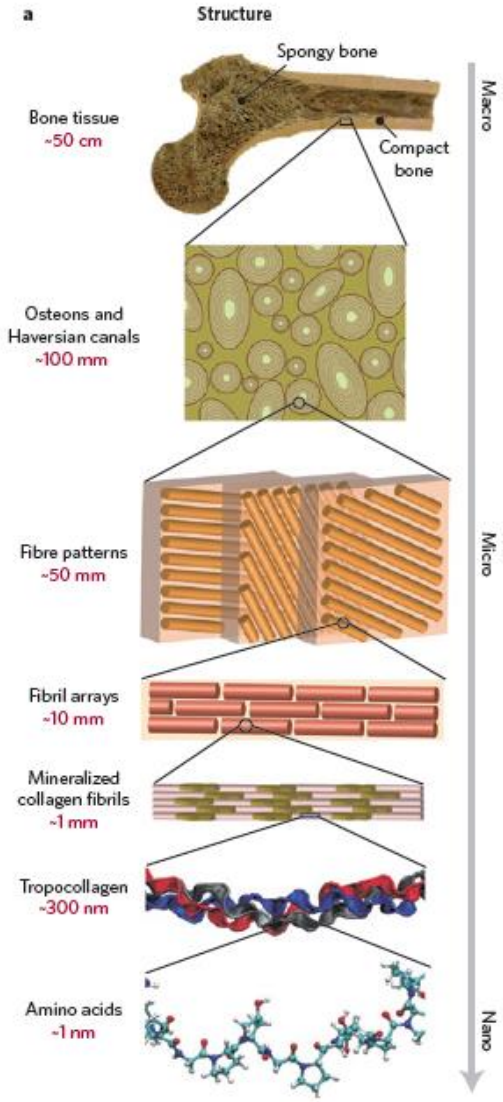


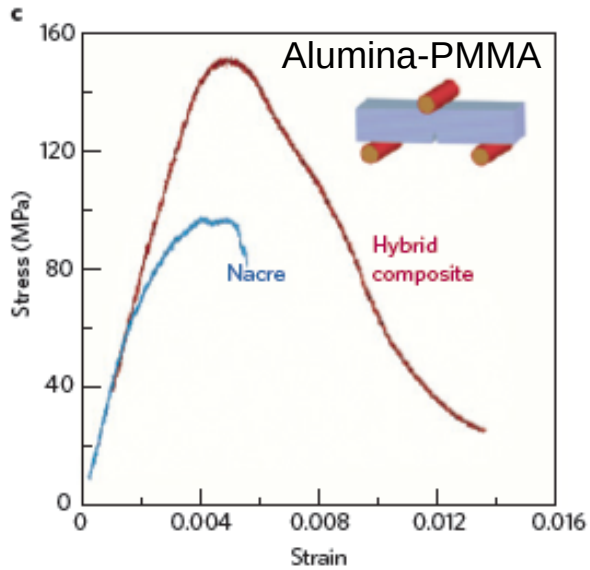


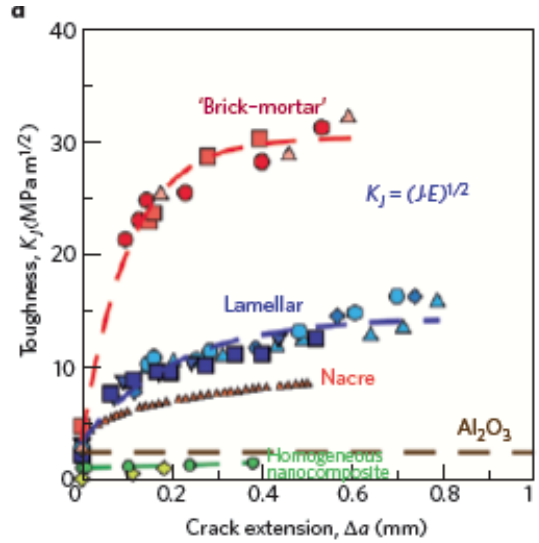
DelRio et al., Appl Phy Rev, 2015

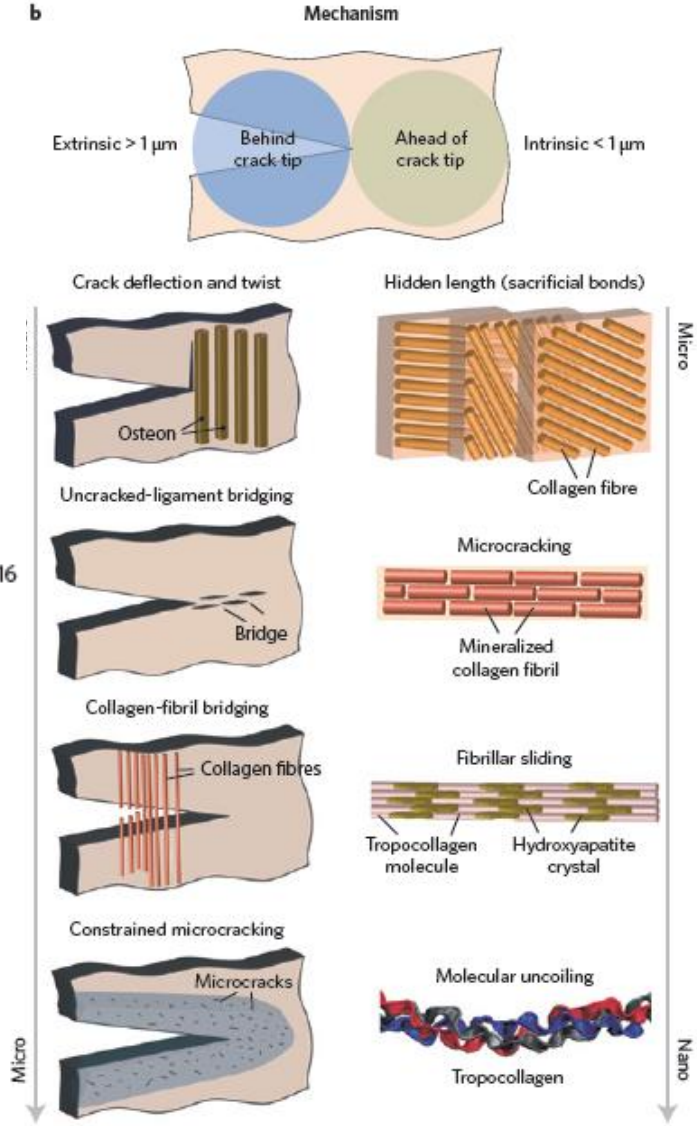
Size effect (intrinsic): Macro to meso to micro structure

Structural material: Natural: Bone









Ritchie, Nature Mater, 2011

Size effect: Change in deformation mechanism

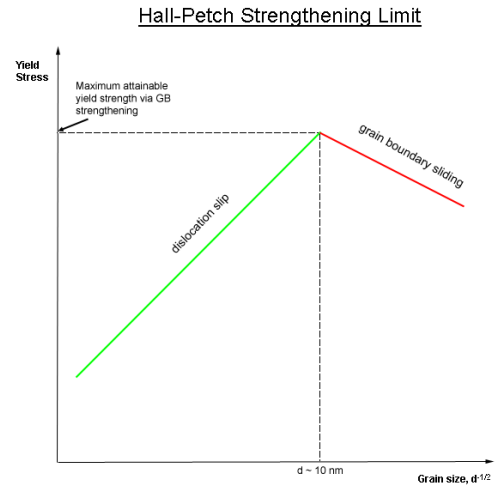
Strengthening mechanisms observed in bulk materials

- Dislocation source activation and multiplication
- Precipitation hardening
- Solid solution strengthening
- Grain boundary strengthening

In general:
Strength increases
Ductility decreases
Fracture toughness remains constant

Nanostructured materials (geometry/microstructure)

- Sputtering
- Evaporation
- Electrodeposition
- Severe plastic deformation



Wikipedia

Methods: From macro to micro-mechanics

Tension-compression

Instron UTM

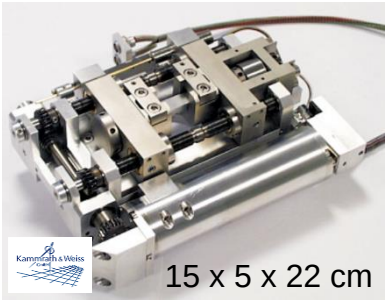
- Sample size
  ~100mmX10mmX10mm
- Load range
  0 to 5kN-500kN
- Load resolution
  (1/1000 of load cell) ~5N
- Displacement range
  0 to 100mm-150mm
- Displacement rate
  1 to 5e5 μm/min

http://www.instron.com/en-us/products/testing-systems/universal-testing-systems/electromechanical

Kamrath and Weiss

- Sample size
  60 mm X 10 mm X 3mm
- Load range
  0 to 5kN
- Load resolution
  0.2N
- Displacement range
  +/- 10mm
- Displacement rate
  6 to 1200 μm/min

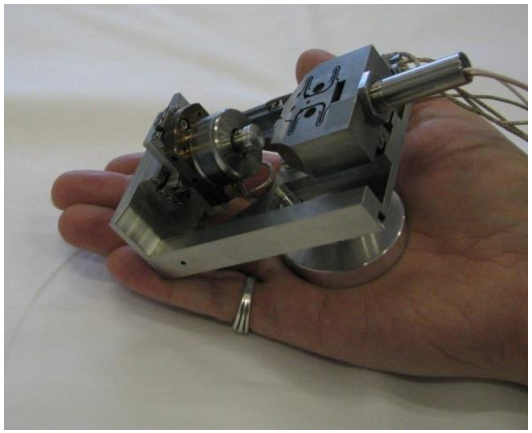
http://www.kammrath-weiss.com/en/products/materials/tensile-compression.html



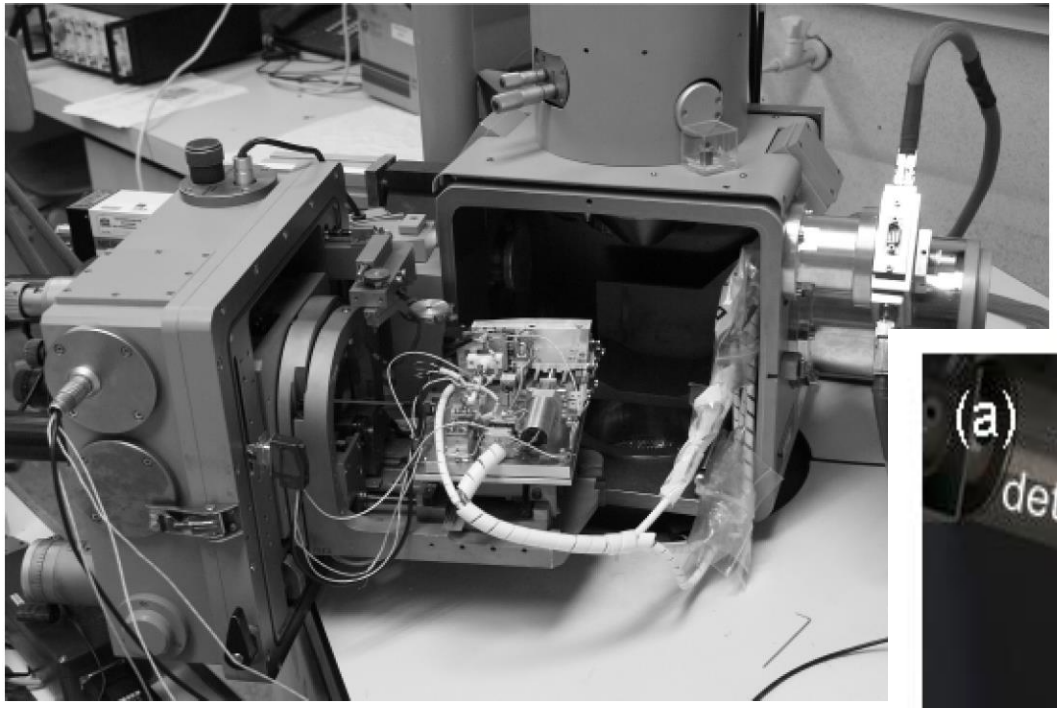
Nano Tensile

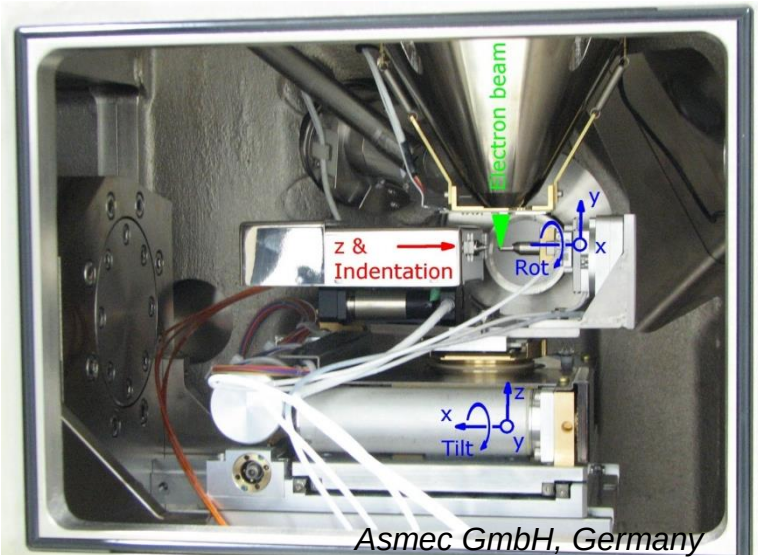
- Sample size
- Load range
  0 to 150mN - 10N
- Load resolution
  10nN - 1μN
- Displacement range
  0 to 80μm - 150mm
- Displacement resolution
  0.02 nm - 100nm
- Displacement rate
  0.1 to 20 μm/s

Hysitron nanoTensile 5000 sell sheet

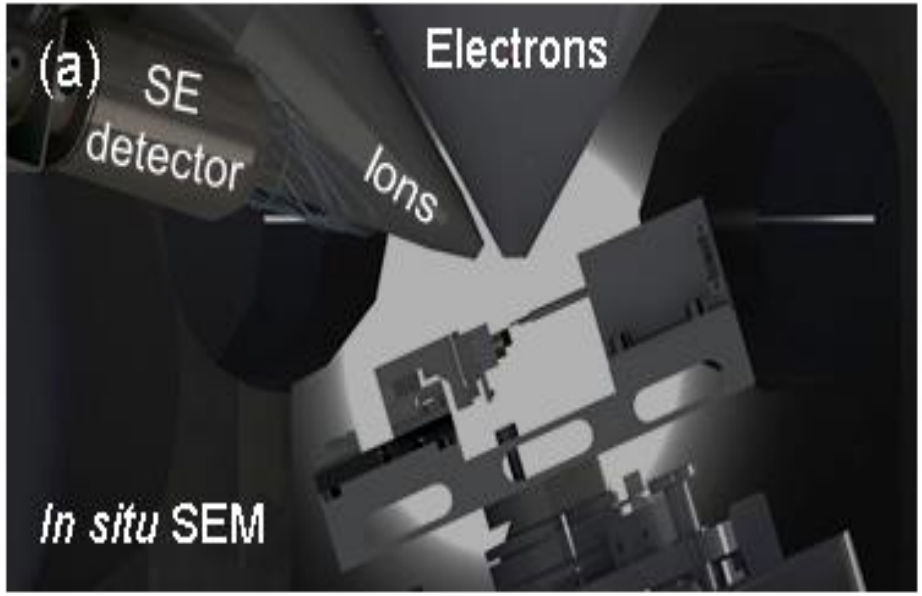


Methods: Small-scale mechanical testing





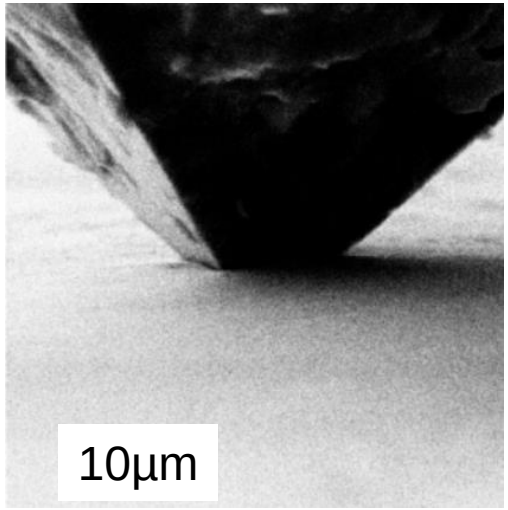
Asmec GmbH, Germany



Hysitron Inc, USA

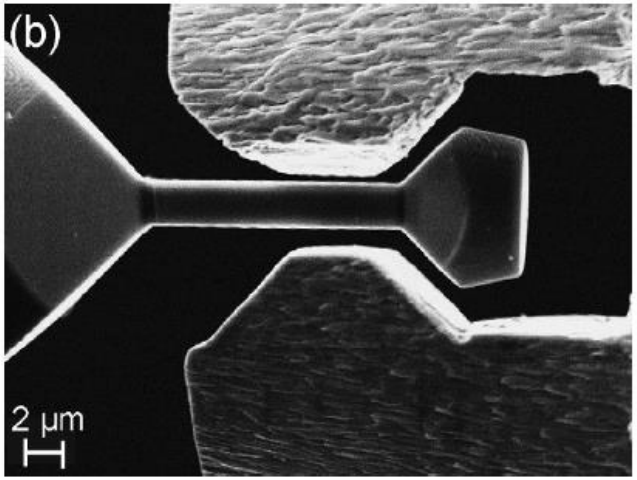
Methods: Testing techniques for deformation

Indentation

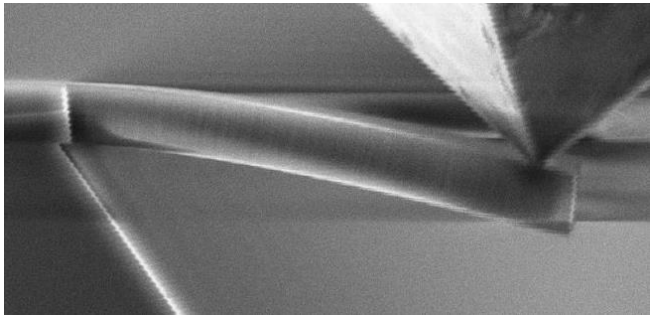


Compression Testing

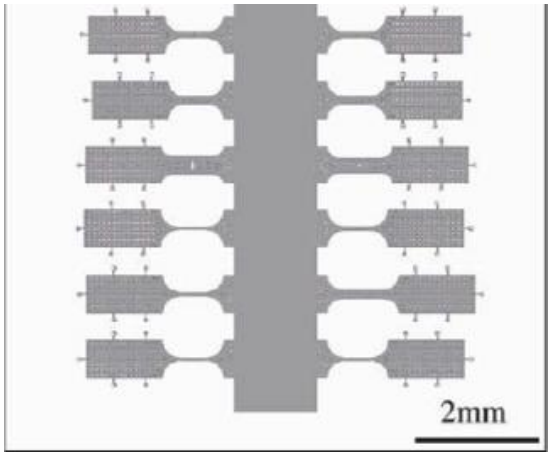
Tensile Testing



Bending Experiments

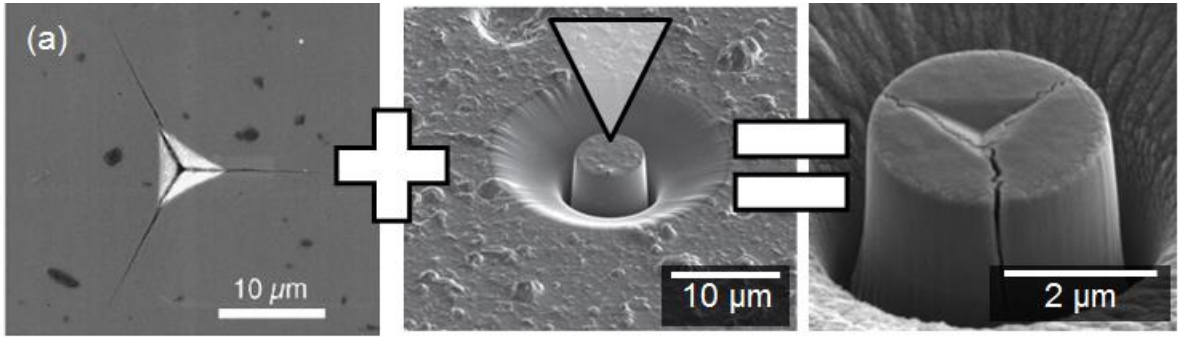


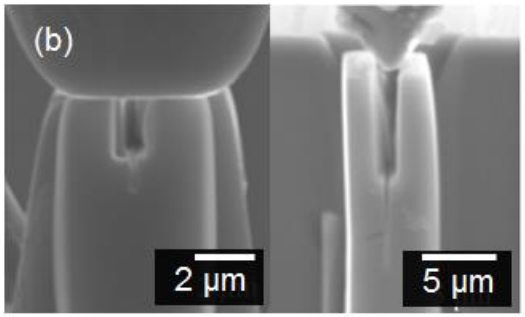
MEMS based Testing

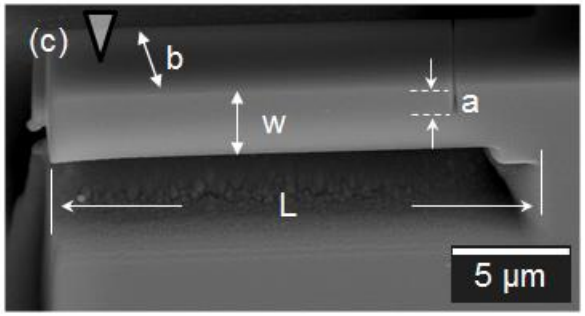


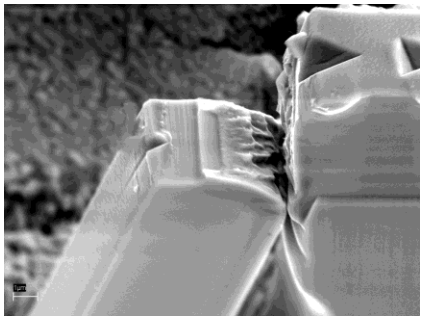
B. N. Jaya and V. Jayaram, Current Science, 2013

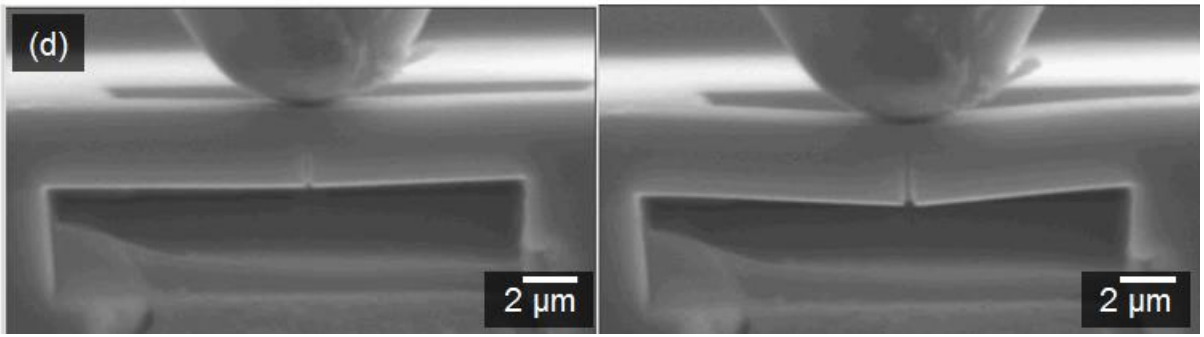
Methods: Testing techniques for fracture

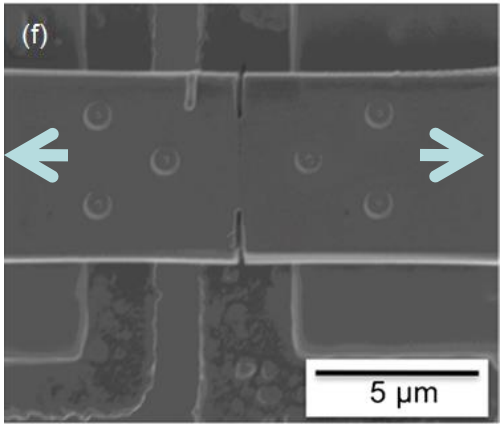






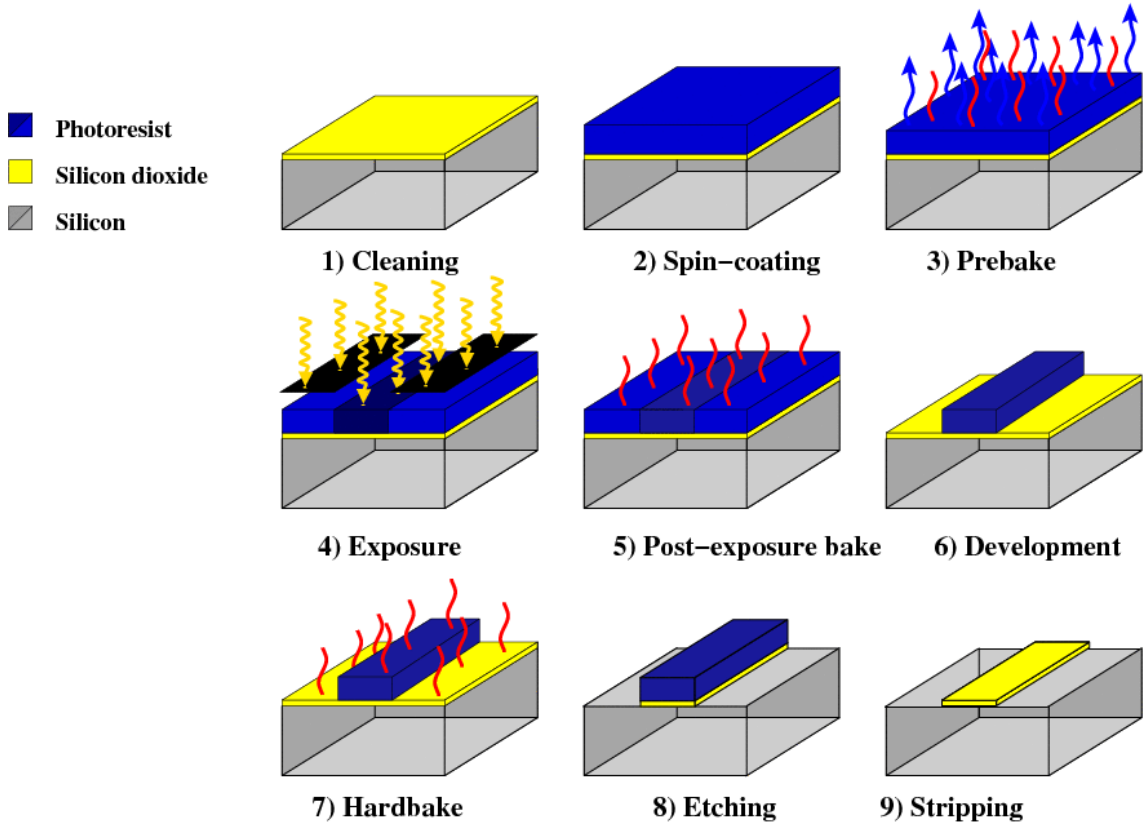




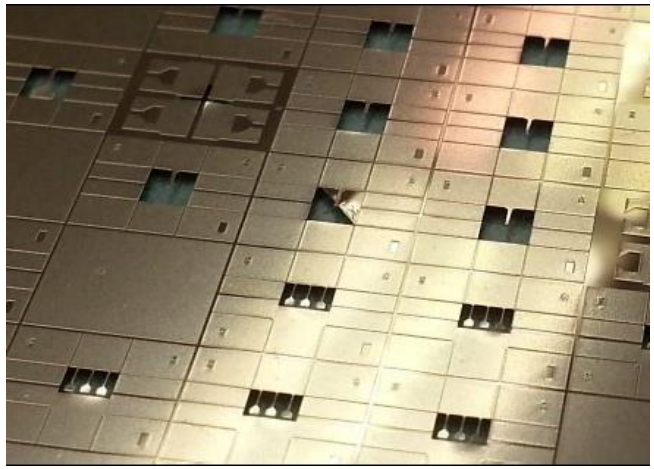


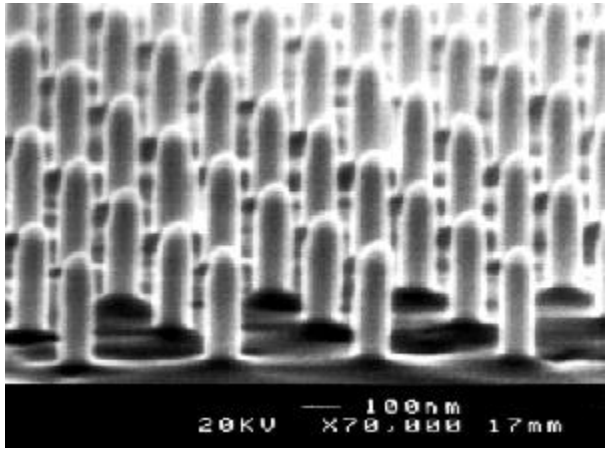
B. N. Jaya and V. Jayaram, JOM, 2015

Lithography

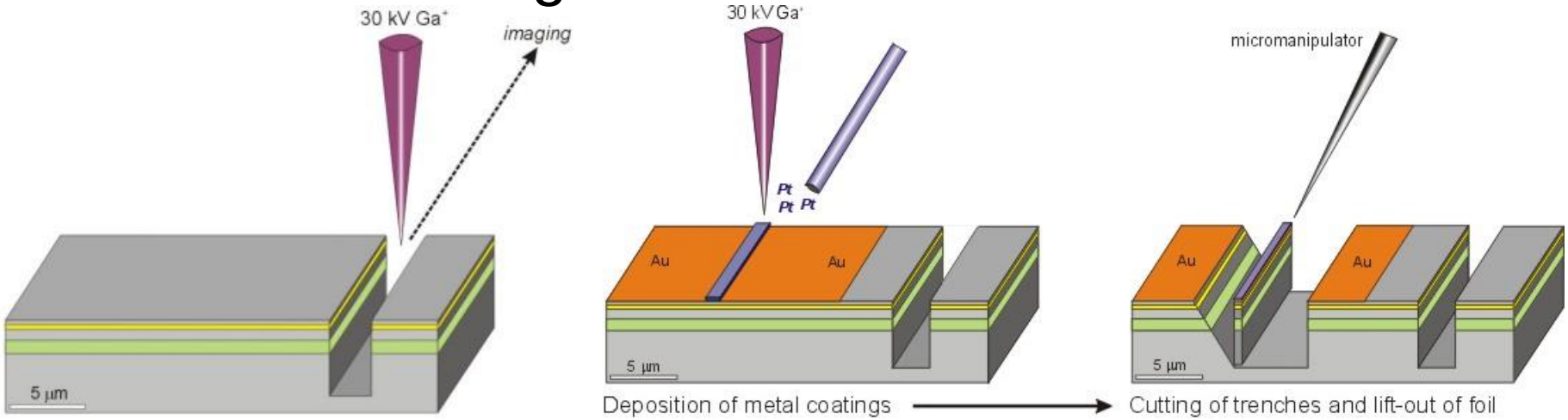


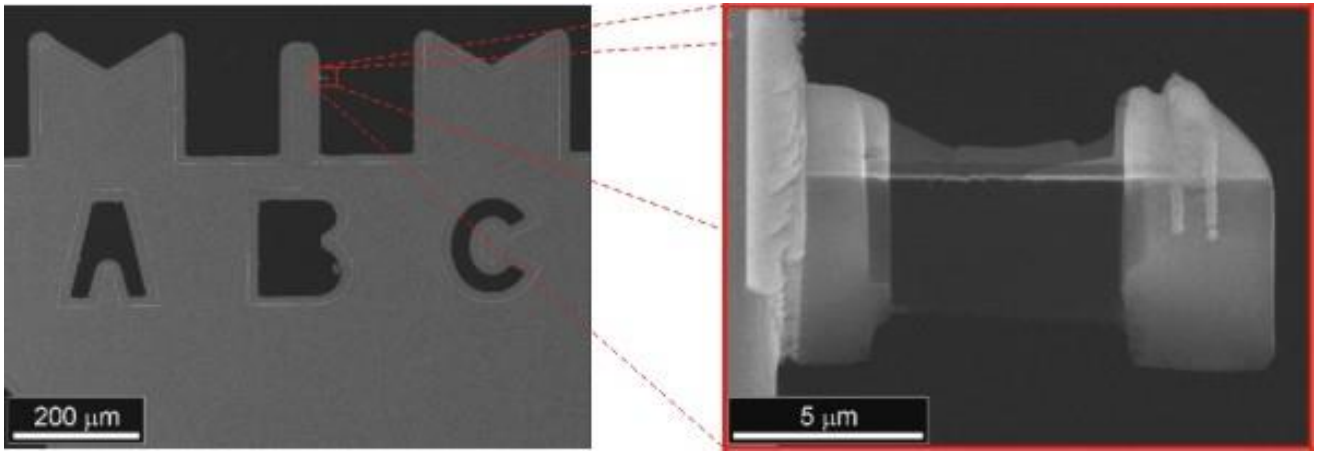
http://www.wias-berlin.de/





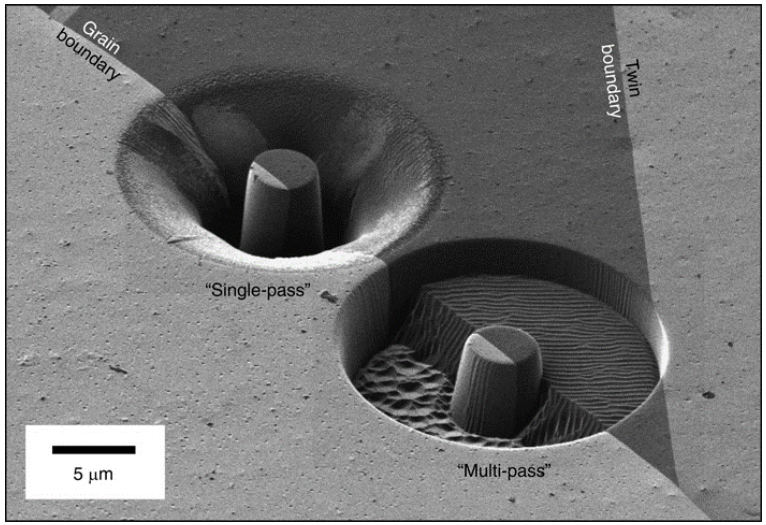
FIB micromachining



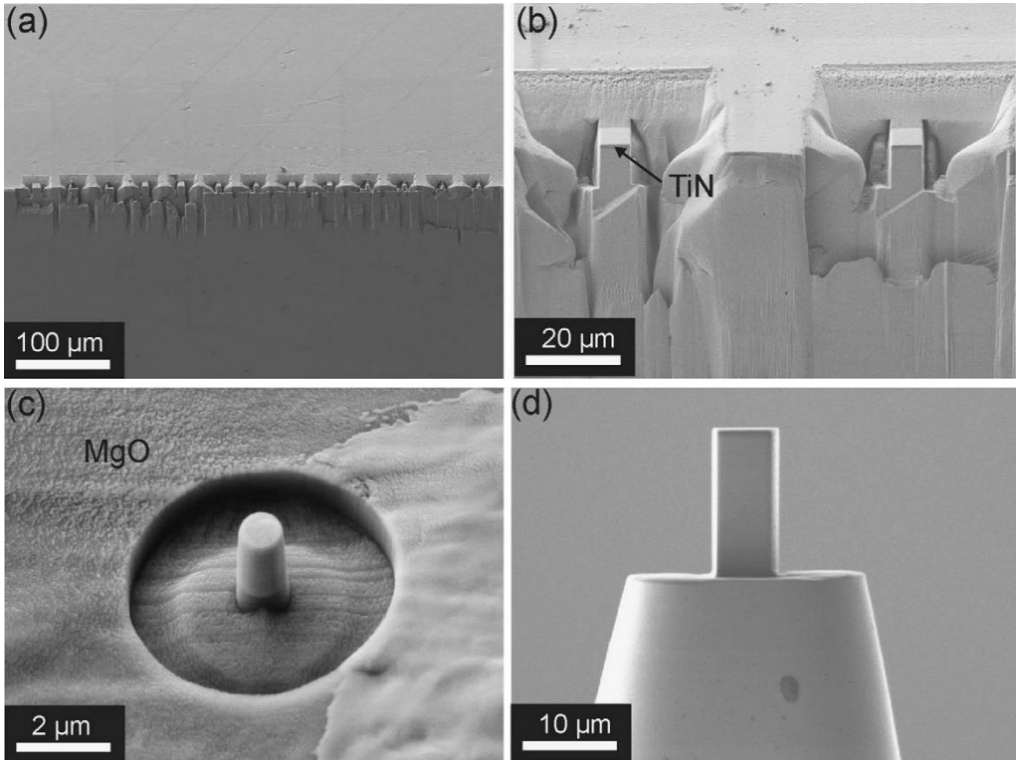


FIBtec.html

FIB micromachining: Site specific



Volkert and Minor MRS Bulletin 32 (2007) 389

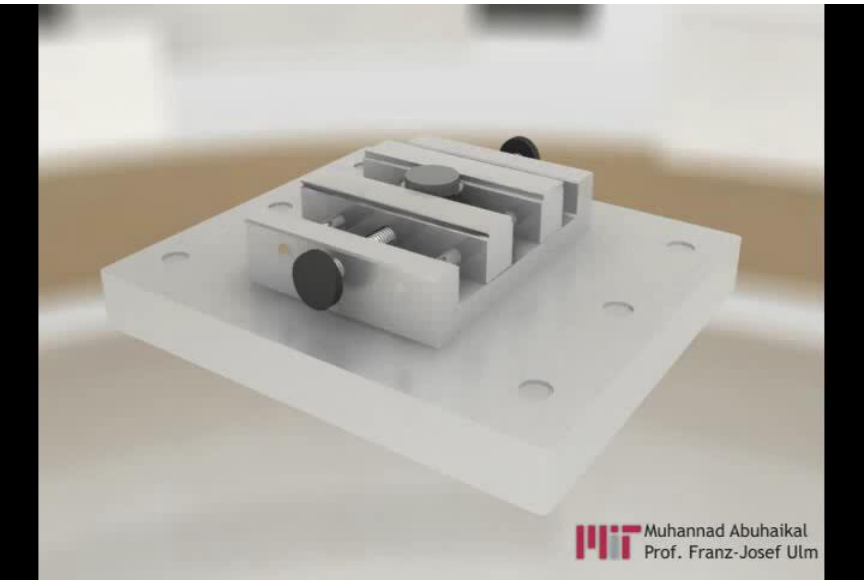


Kiener et al., Materials Science and Engineering A 505 (2009) 79–87

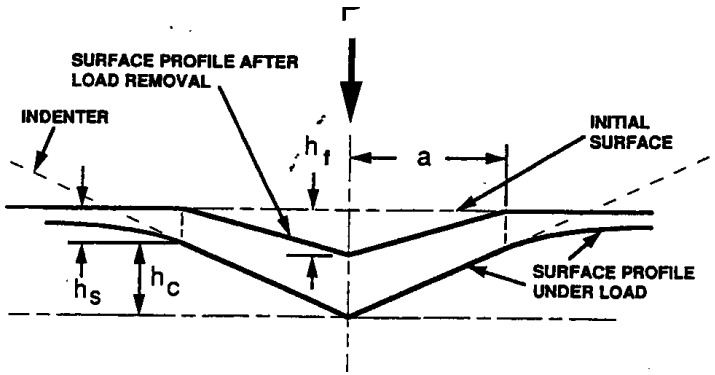
Methods: Testing and analysis

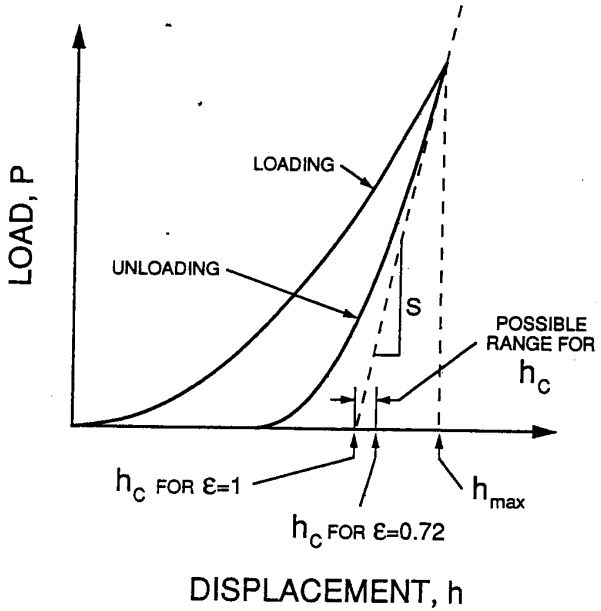
Nanoindentation

Mechanical probing of a material surface to nm-scale depths, while simultaneously monitoring LOAD and DEPTH.



https://www.youtube.com/watch?v=AEatCyb1t-A





- Flat specimen – polished

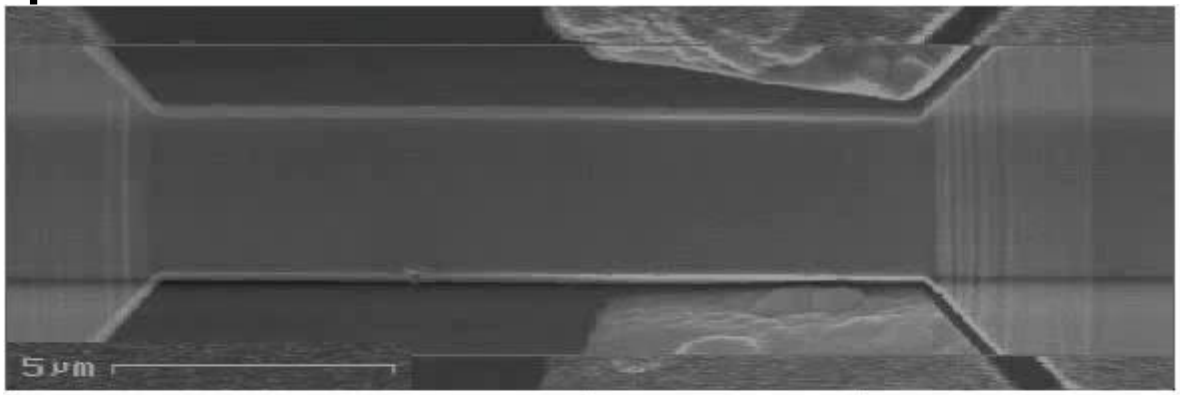
(Film – low surface roughness)

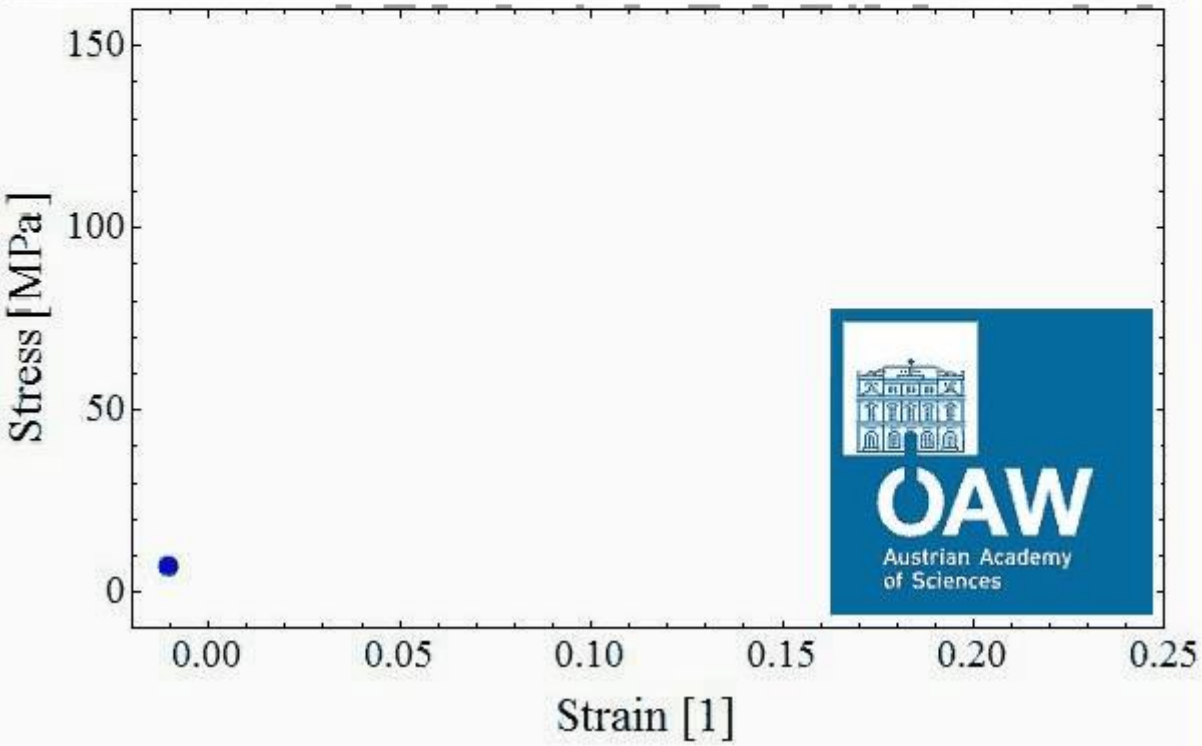
- Small volumes of material
- Large plastic strains can be applied

Oliver and Pharr, J. Mater. Res. 7 (1992) 1564

Methods: Testing and analysis

Micro compression-tension





Methods: Testing and analysis

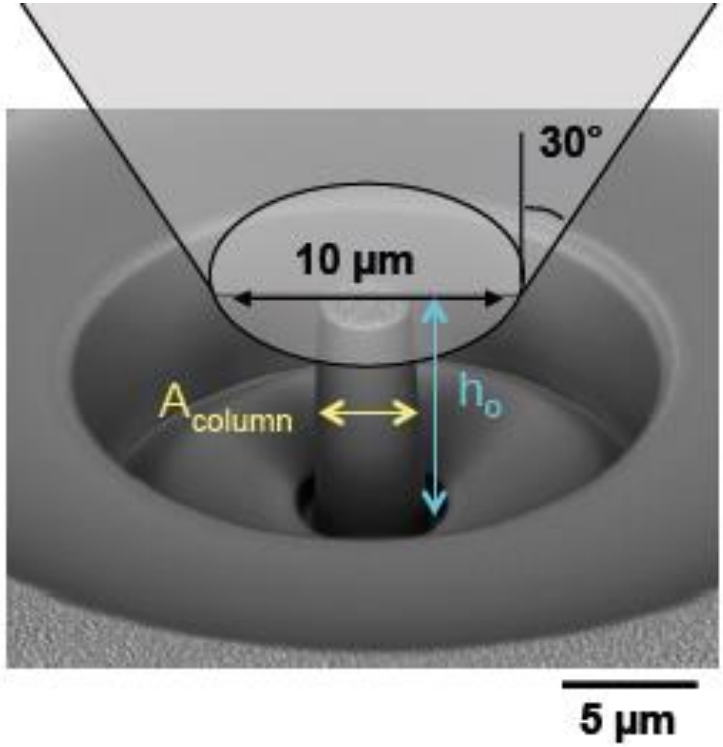
Micro compression-tension

- Uniaxial, uniform stress state
- Constant deformation volume

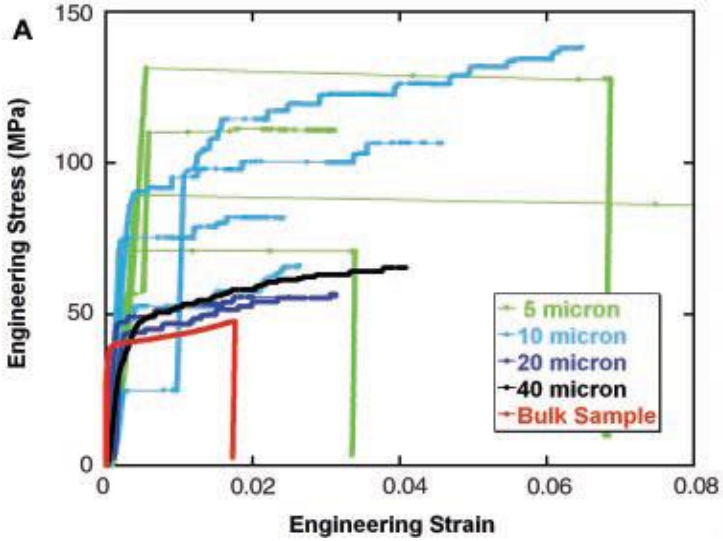
} As compared to indentation testing

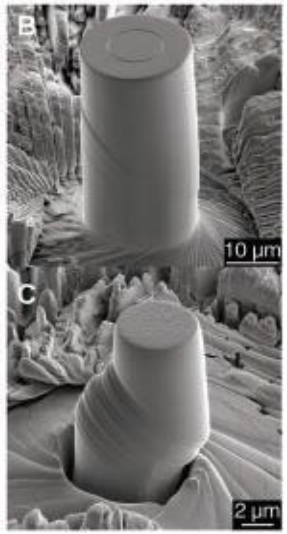
- Pre-defined volume before and after deformation, and in situ testing
- Straight forward stress-strain analysis:

$$\sigma = \frac{P}{A_{column}}, \varepsilon = \frac{\Delta h}{h_o}$$



Ni_3Al

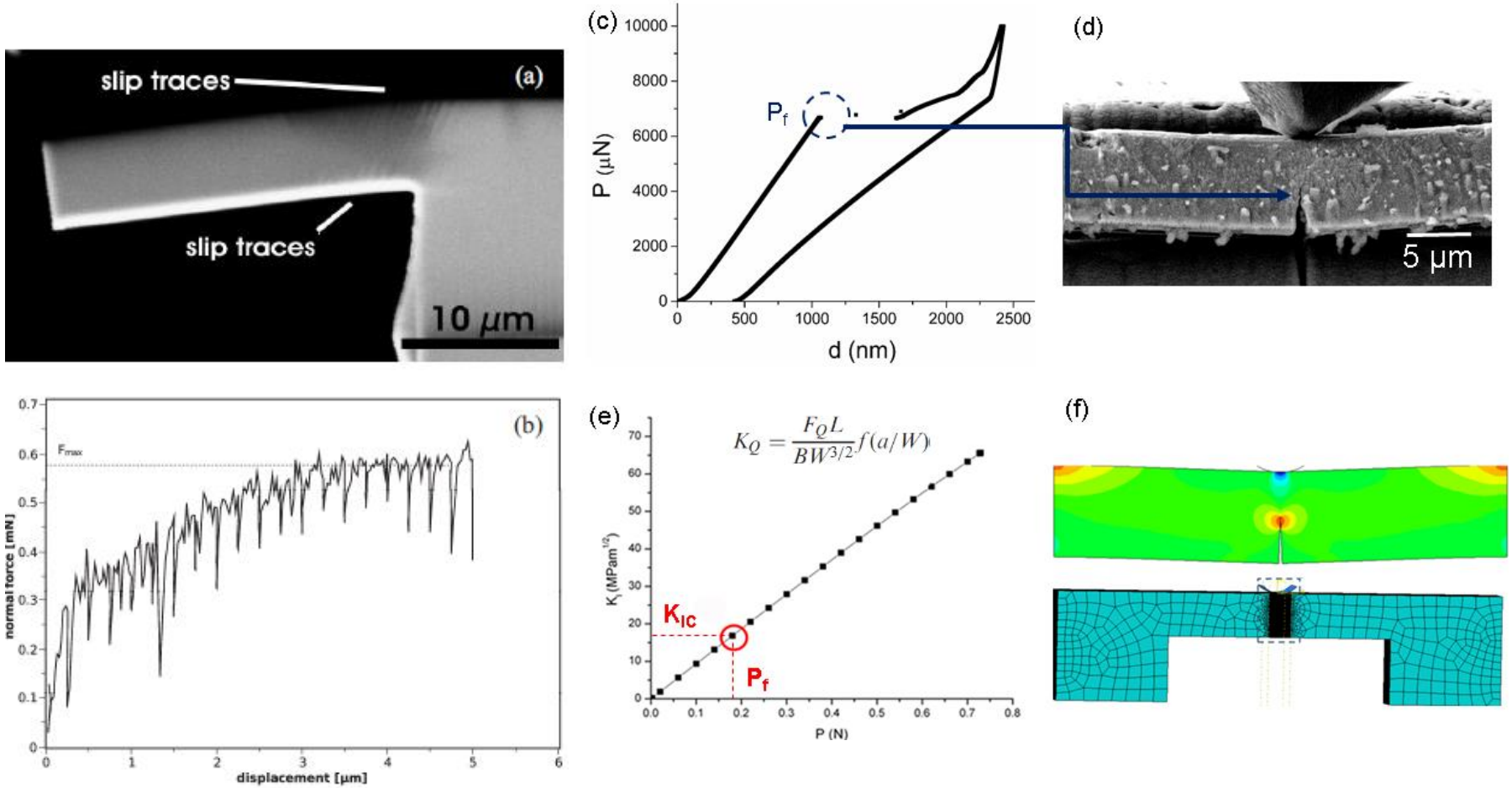




Uchic et al. Science 305 (2004) 986.

Methods: Testing and analysis

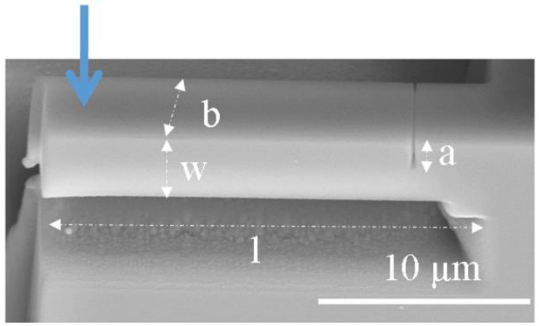
Microbeam bending and fracture:



Dehm et. al, Adv Eng Mater, 2006

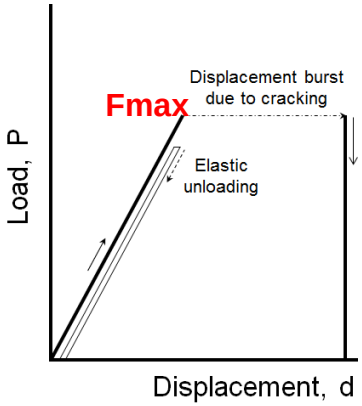
Jaya et. al, Phil Mag, 2012

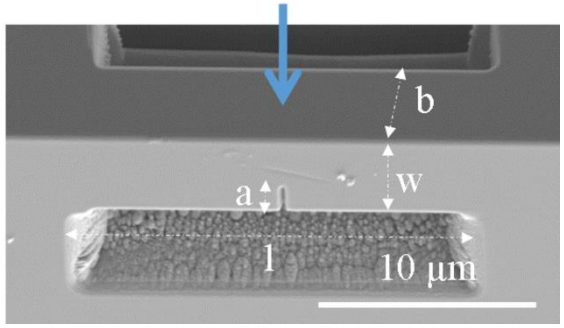
Methods: Effect of test geometry

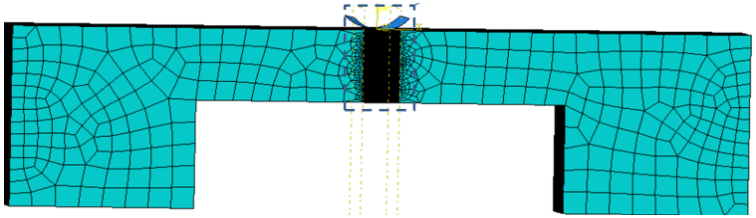


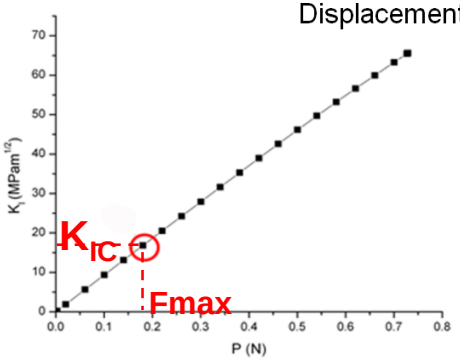
$$K_{IC} = \frac{F_{max} L}{B w^{\frac{3}{2}}} f\left(\frac{a}{w}\right).$$

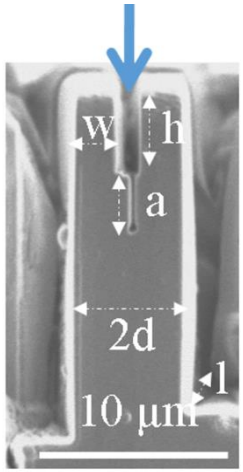
$$f\left(\frac{a}{w}\right) = 1.46 + 24.36\left(\frac{a}{w}\right) - 47.21\left(\frac{a}{w}\right)^2 + 75.18\left(\frac{a}{w}\right)^3.$$



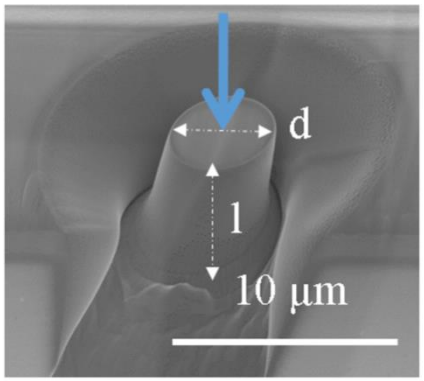








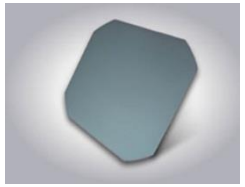
$$K_I = \sqrt{3}\,\frac{(e - \mu h)}{b d^{3/2}} F_{max}$$



$$K_c = \frac{\gamma F_{max}}{R^{3/2}}$$

Methods: Effect of test geometry

Si (100):



a) **Single cantilever bend**

K_{IC} ($MPam^{1/2}$): **0.76±0.08**

b) **Clamped beam bend**

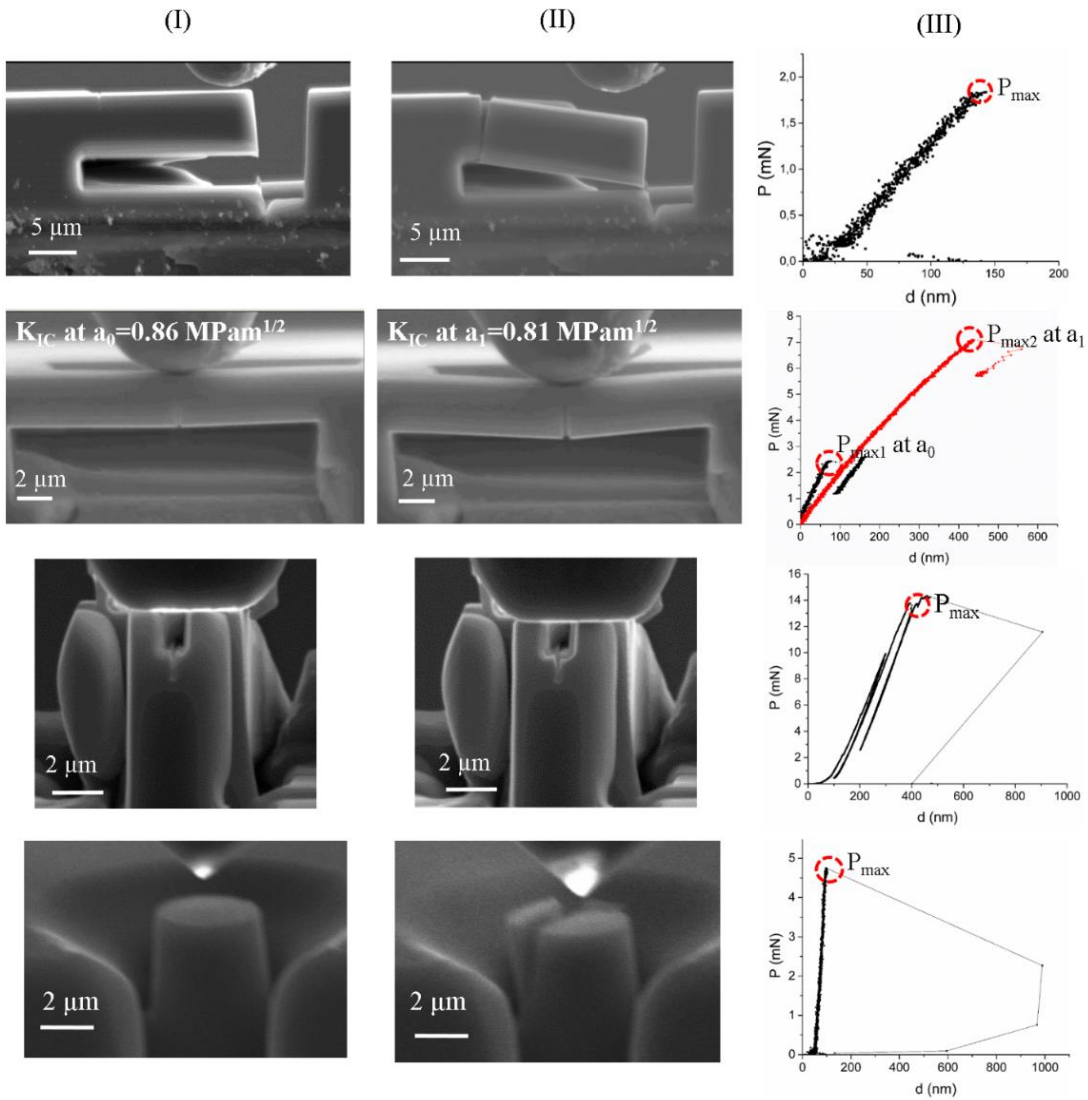
K_{IC} ($MPam^{1/2}$): **0.81±0.10**

c) **Double cantilever compression**

K_{IC} ($MPam^{1/2}$): **0.89±0.12**

d) **Pillar split**

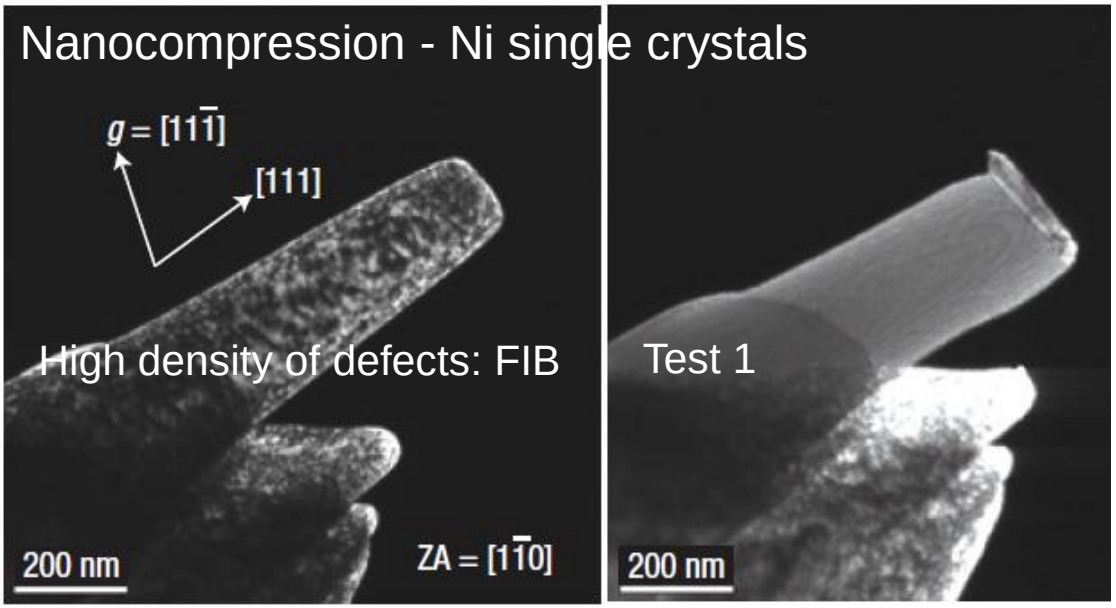
K_{IC} ($MPam^{1/2}$): **0.75±0.016***

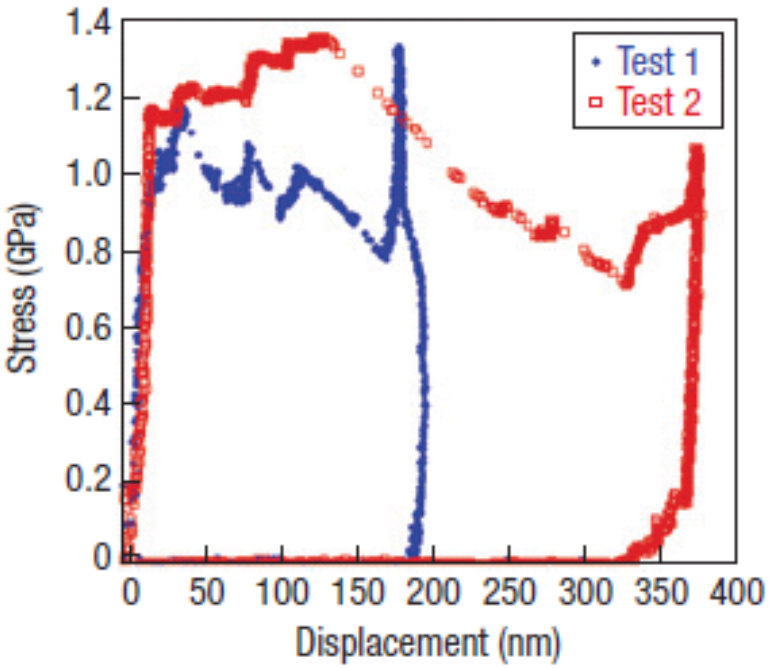


B. N. Jaya et al, JMR, 2015

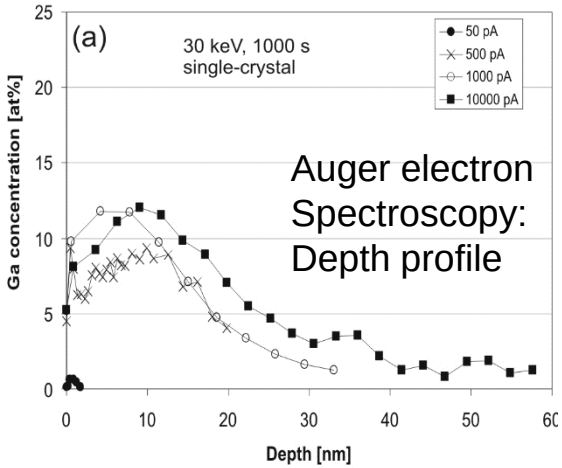
Methods: Errors and limitations

Sample preparation: Ion damage effects (FIB)





Shan et al. Nature Mater. 7 (2008) 115



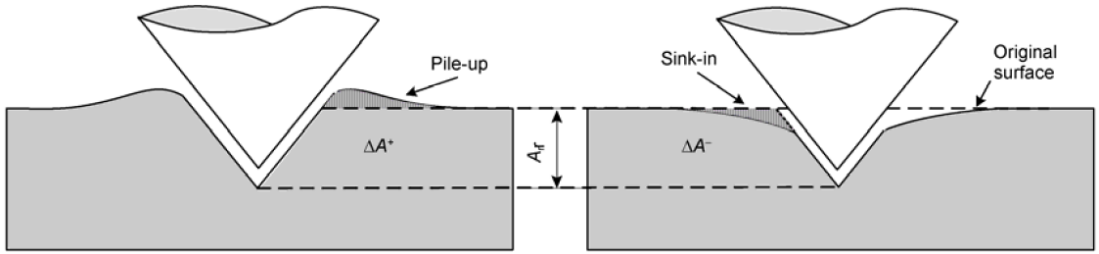
Kiener et al. Mater. Sci. Eng. A 459 (2007) 262.

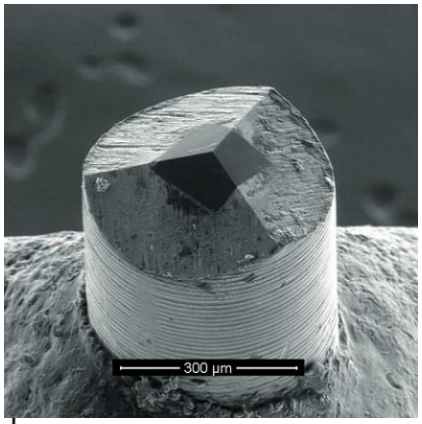
FIB damage may cause
- Back stress on sources
- Forest dislocations
- Precipitation hardening

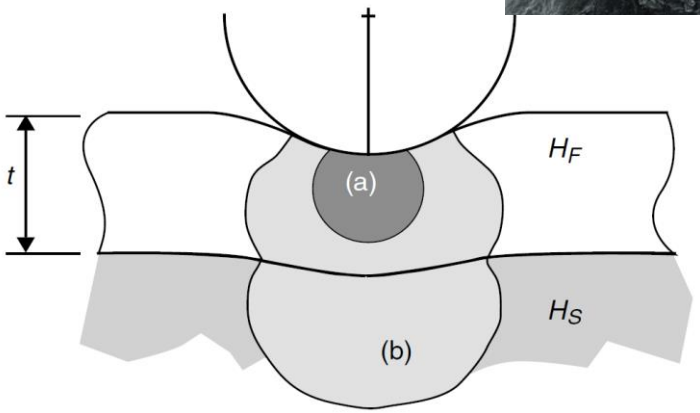
Methods: Errors and limitations

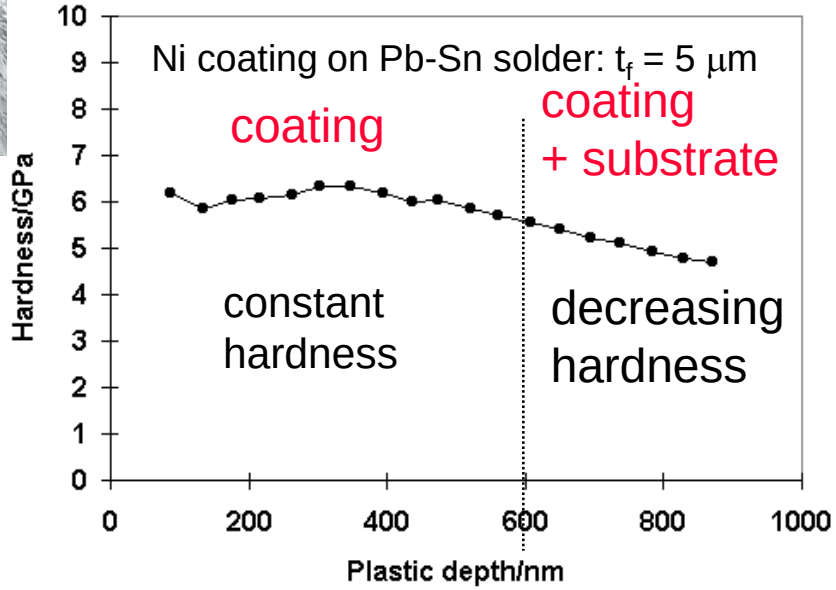
Sample testing: Indentation

- Pile-up, sink-in corrections
- Tip rounding and tip area calibration
- Substrate compliance
- Thermal drift
- Surface roughness







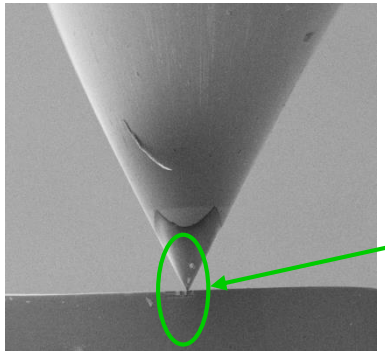


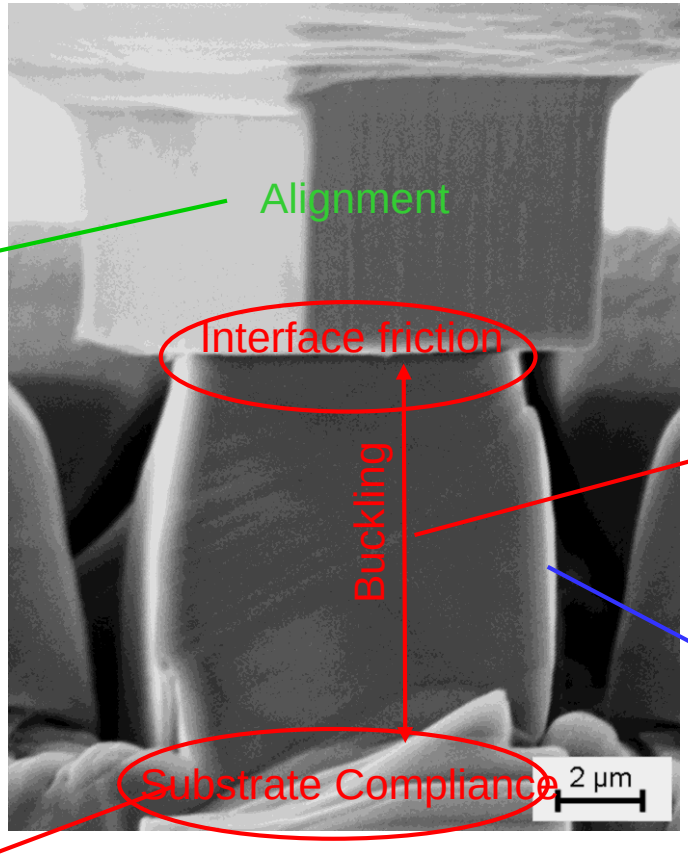
Pharr et al. Annu. Rev. Mater. Res. 40 (2010) 271

Methods: Errors and limitations

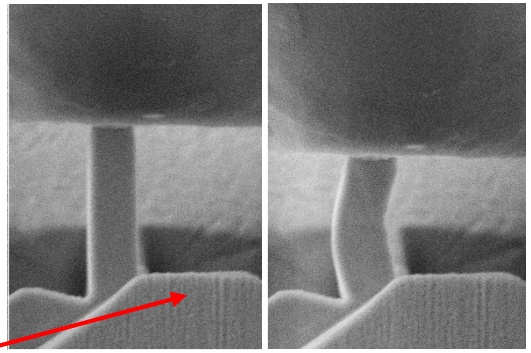
Sample testing: Micro-compression

Alignment

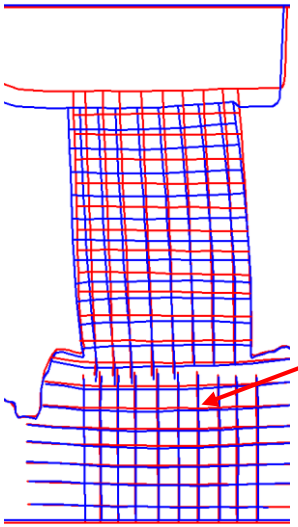




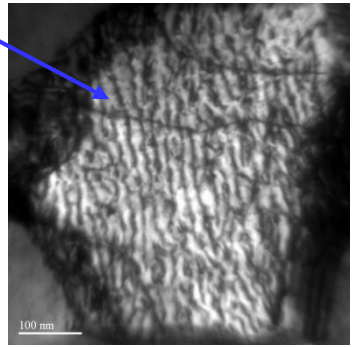
Buckling



Compliance

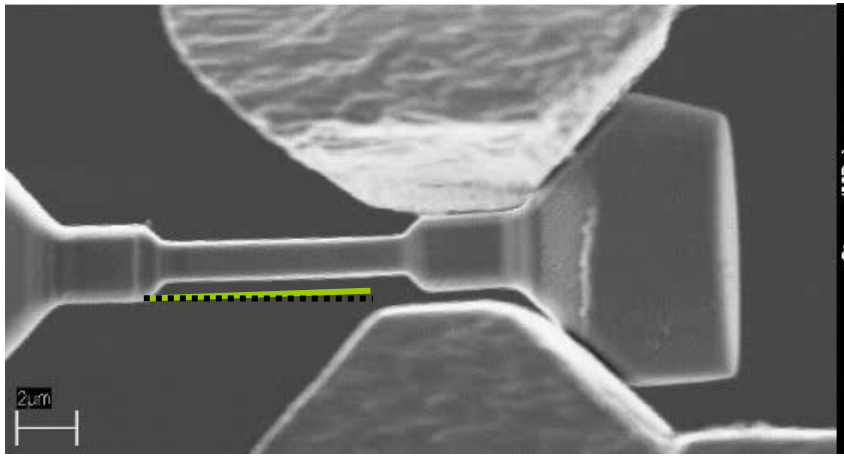


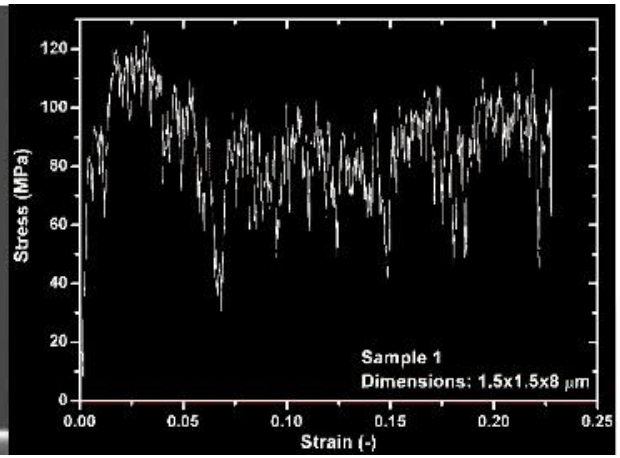
Ion damage

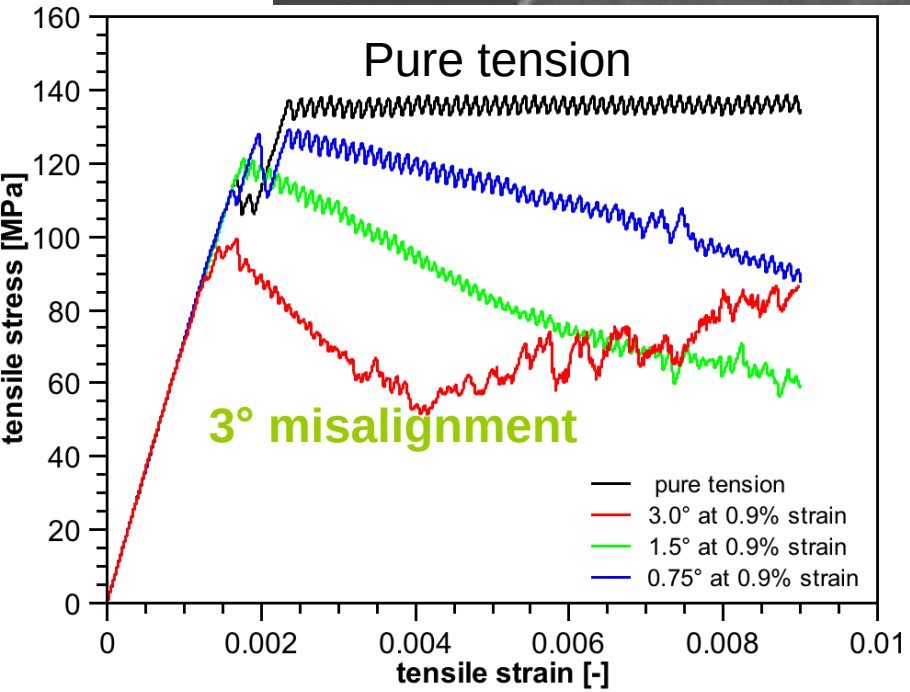


Methods: Errors and limitations

Sample testing: Alignment in tension







Other issues:

- Fit between the flanks of sample and gripper – imperfect
- Pre-test bending
- Sufficient stiffness of all involved parts (like copper needle or tungsten gripper)

Kiener et al., Acta Materialia 56 (2008) 580-592

Methods: Errors and limitations

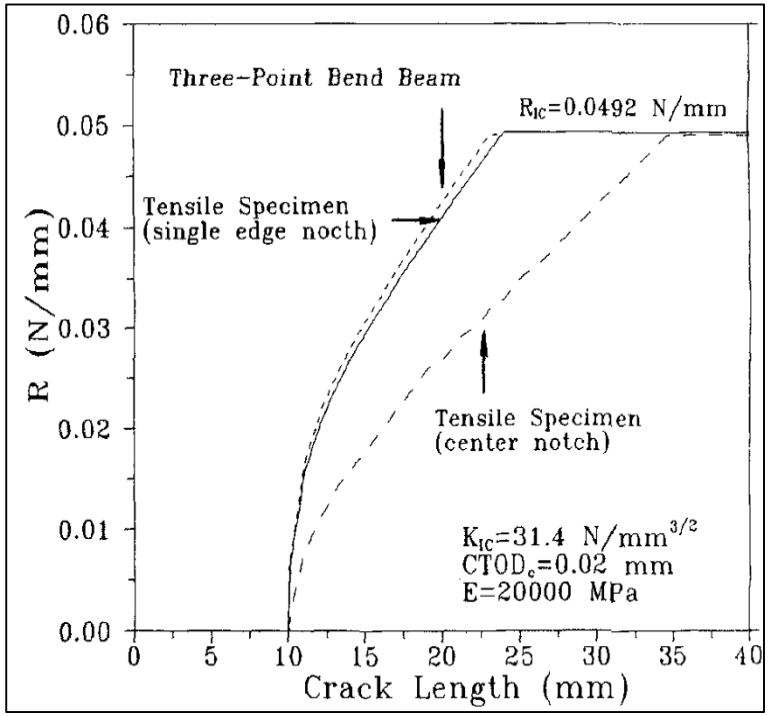
Effect of test geometry

Single and polycrystalline Si

Prediction (MPa m$^{1/2}$)	Measurement (MPa m$^{1/2}$)	Test method[a]	Reference
$T_{(111)} = 0.72$	0.65[b]	DCB, $E = 168$ GPa	34
	0.65[b]	DCB, $E = 168$ GPa	35
	0.937	T-DCB	36
	1.0	DT	38
	0.9	T-DCB	37
$0.73 < T_{(110)} < 0.80$	0.74[b]	DT, $M = 150$ GPa	39
	0.72	DCB	41
	0.65	SECB	43
	0.71	SECB	44
	0.95	SECB	45
	0.84	COD	42
$T_{(poly)} \approx 0.80$	1.1	SECB	46
	0.81	SECB	47
	1.0	SECB	48 and 49

DelRio et al., Appl Phy Rev, 2015

Cementitious material



C. Ouyang and S.P. Shah, J. Amer. Ceram. Soc., 1991

Results and Discussion:

Size effects

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- Inverse Hall
- Interface eff
- Smaller is s
- Toughening

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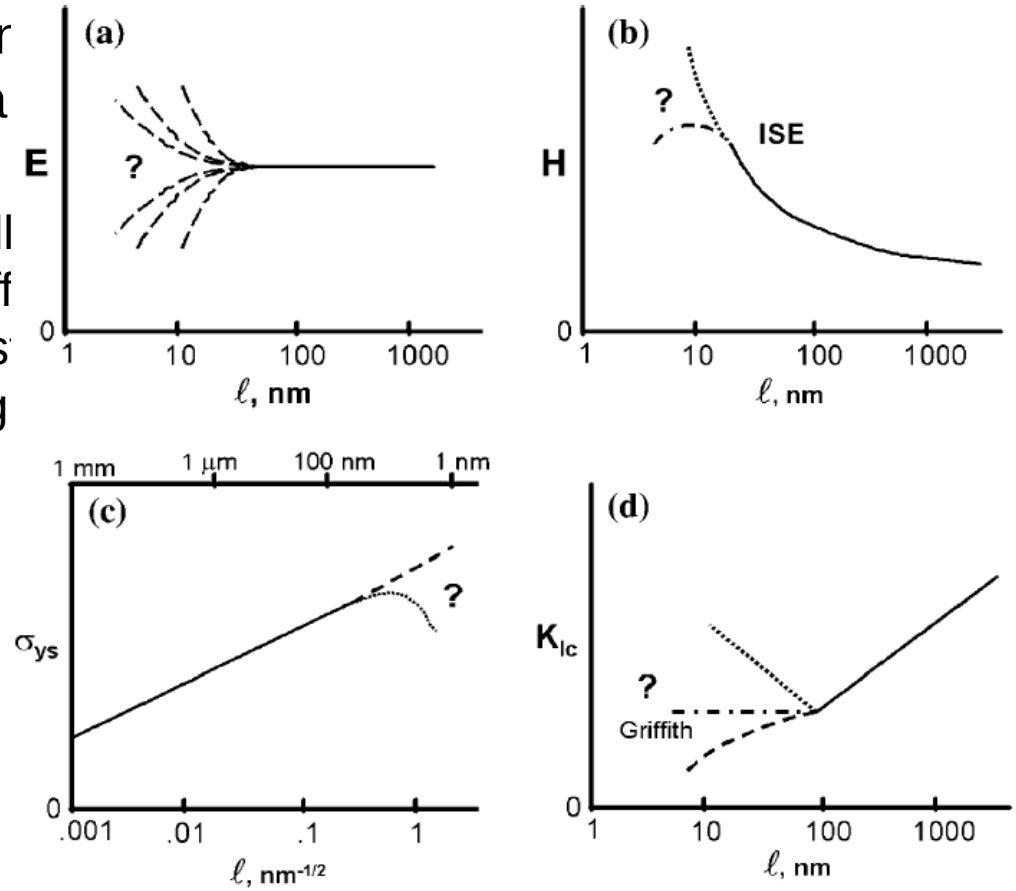
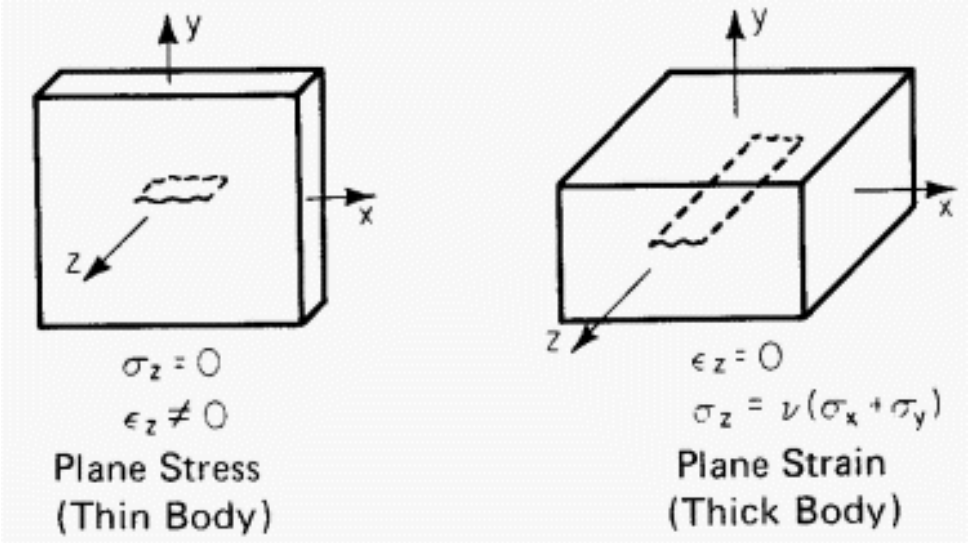
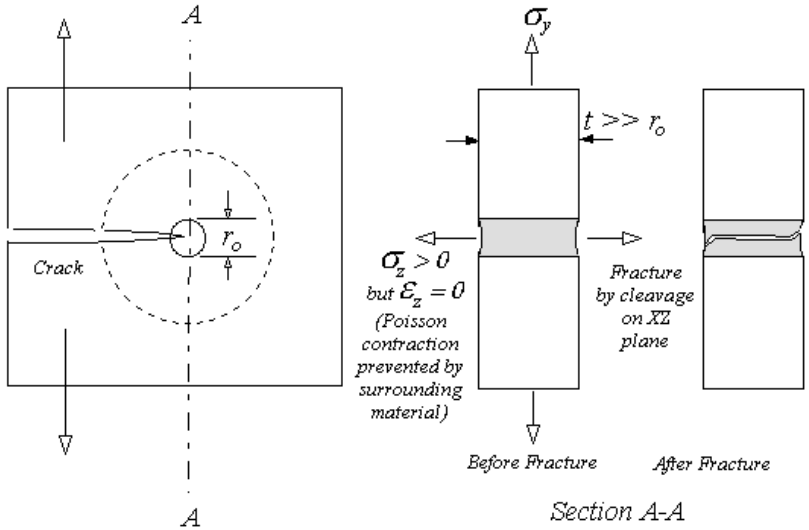
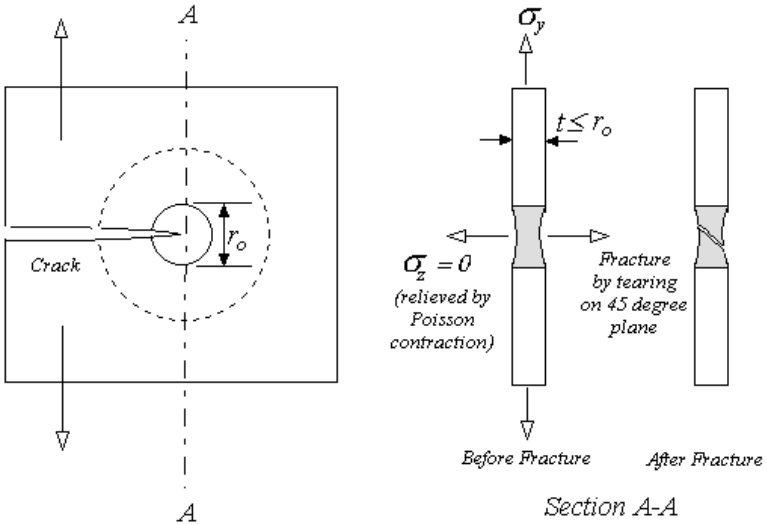


Single crystal Single phase Multi-phase/Multi-layered Complex architecture

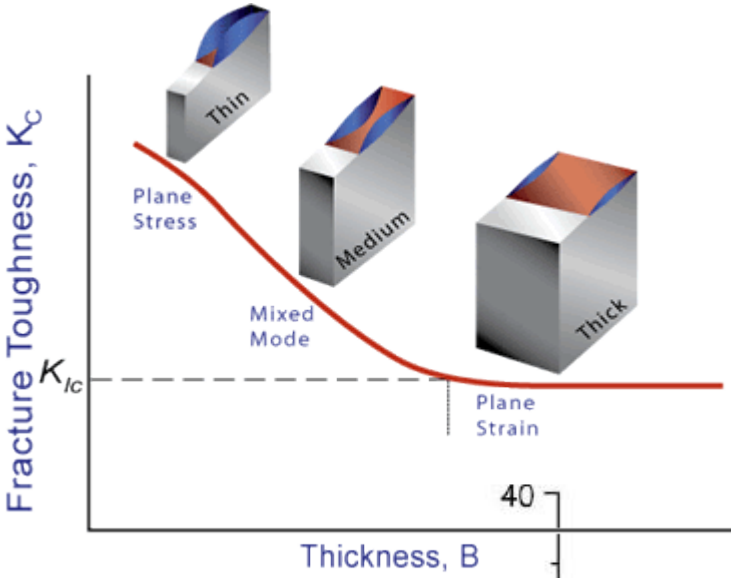
Extrinsic size effects: Plane stress vs strain

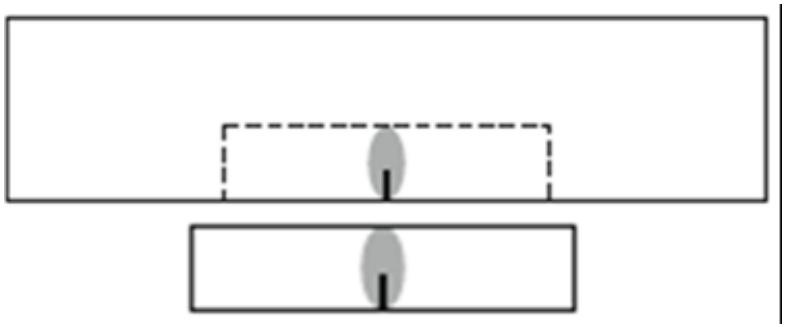


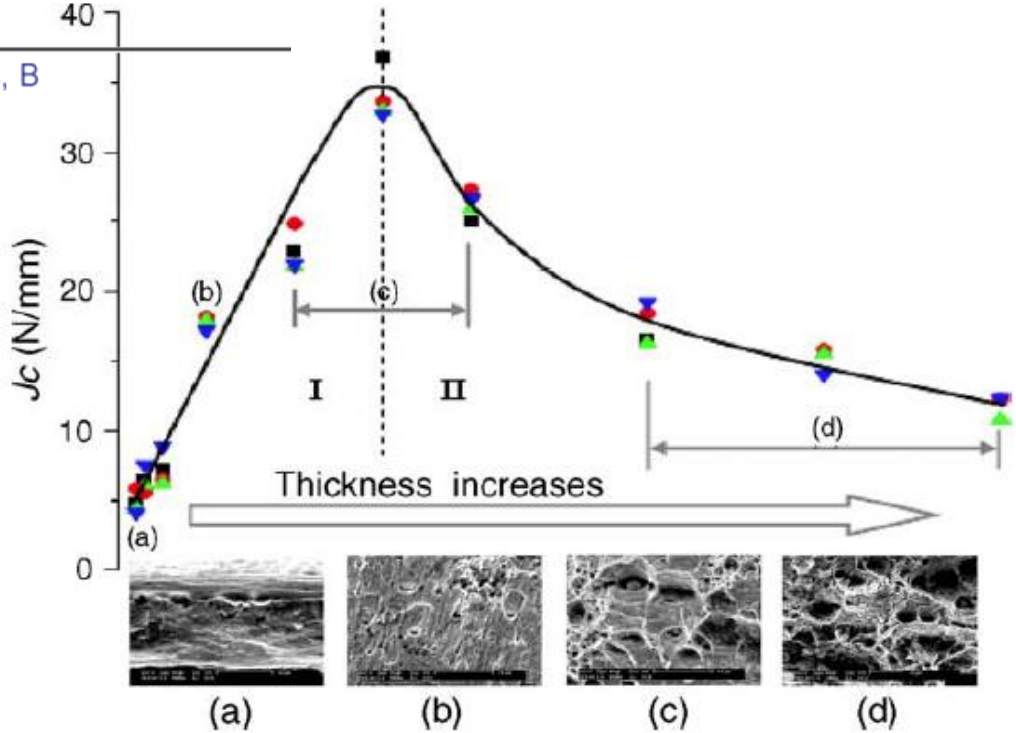




Extrinsic size effects: Plane stress vs strain





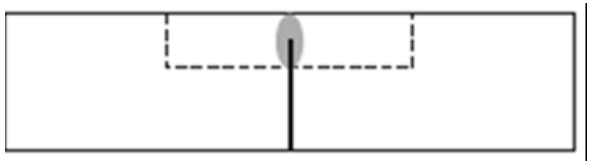


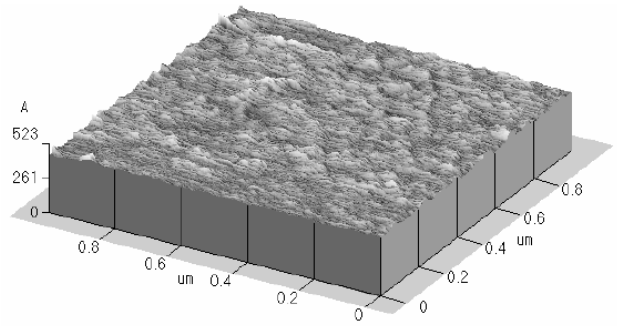
Kang et al, Exp Mech 2005

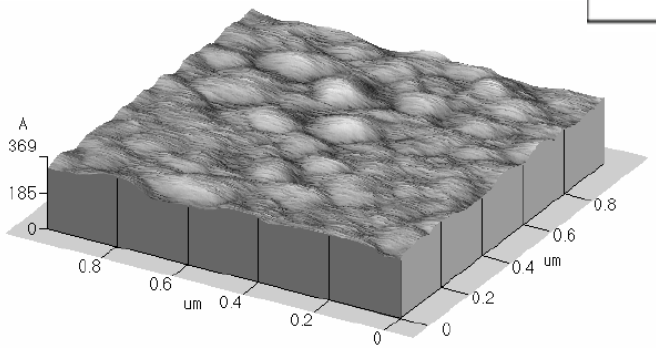
Extrinsic size effects: Effect of surface

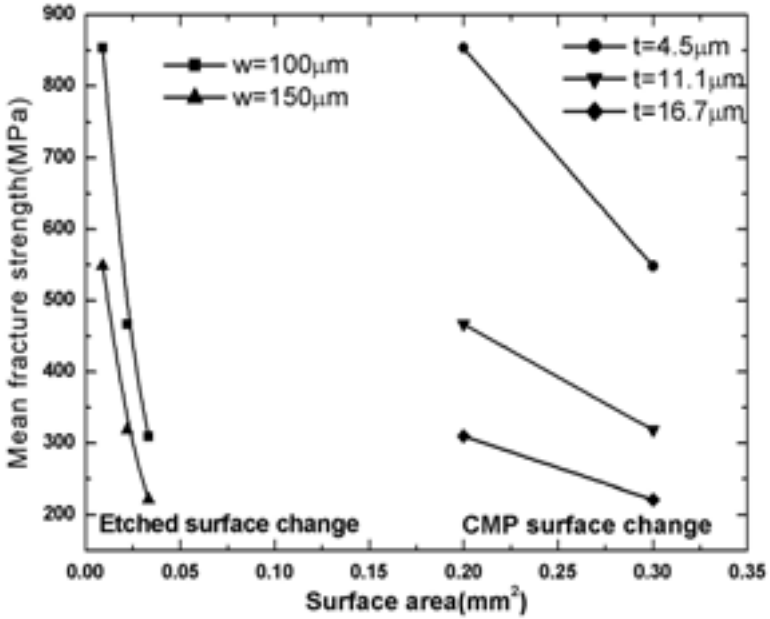
Chemical-Mechanical Polished

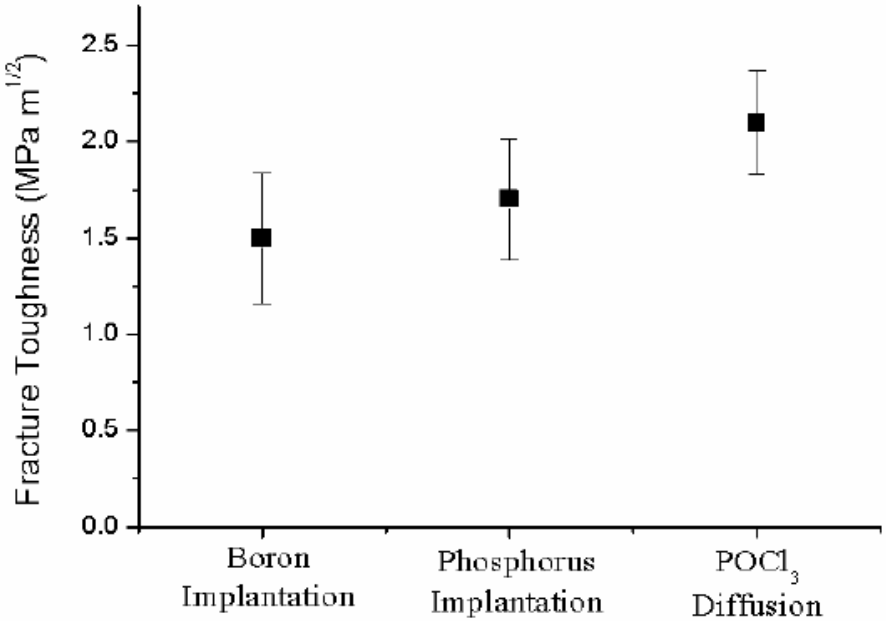
Dry-etched







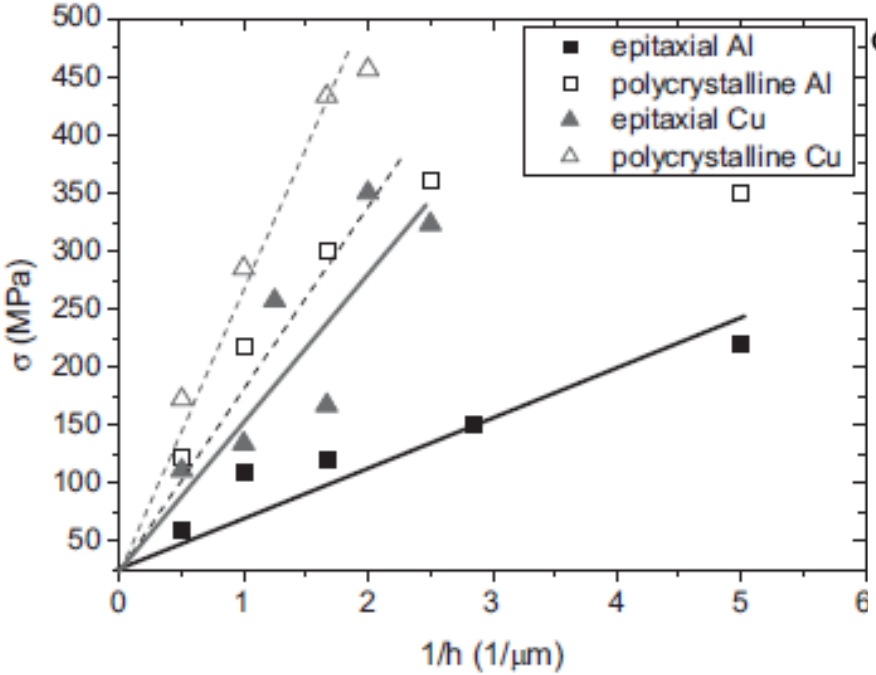




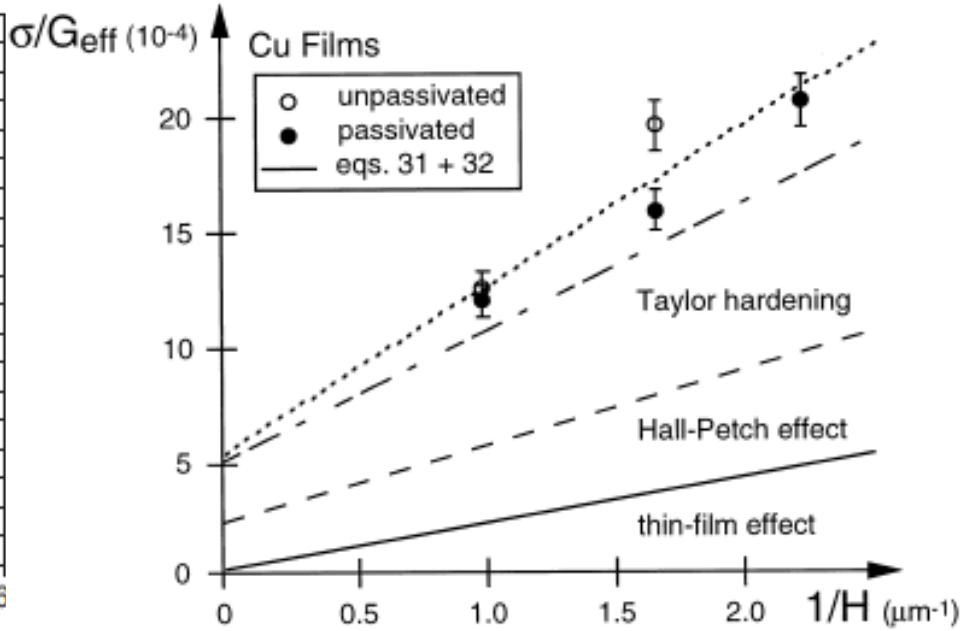
Dong Il Son, Key Eng Mater, 2005

Extrinsic size effects: Effect of surface

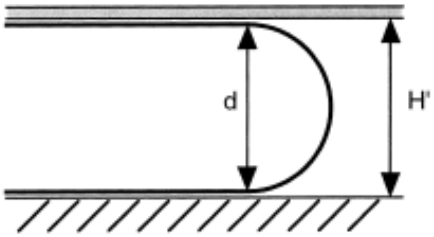
Effect of thin film thickness

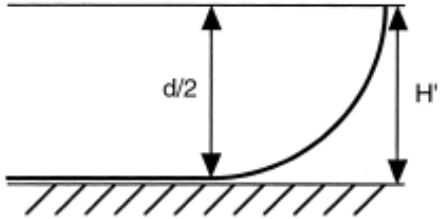


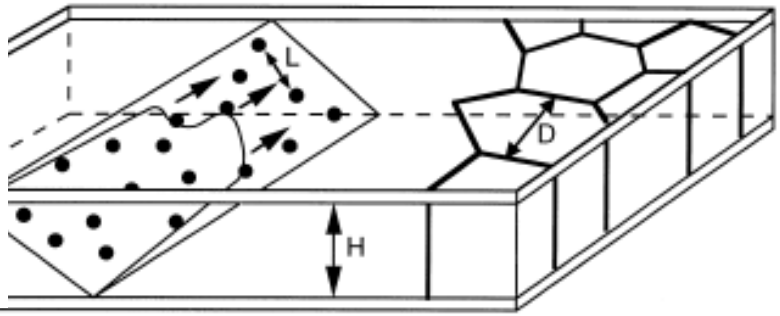
Dehm et al., Adv Eng Mater, 2006



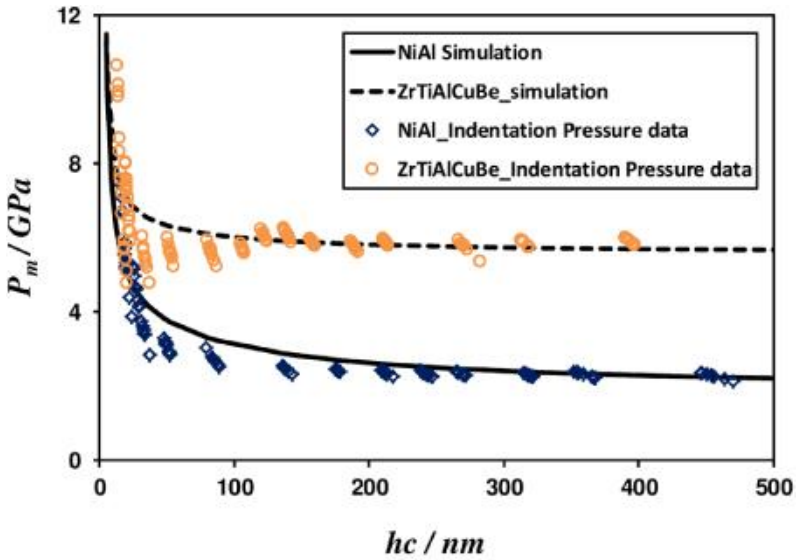
Arzt., Acta Mat, 1998

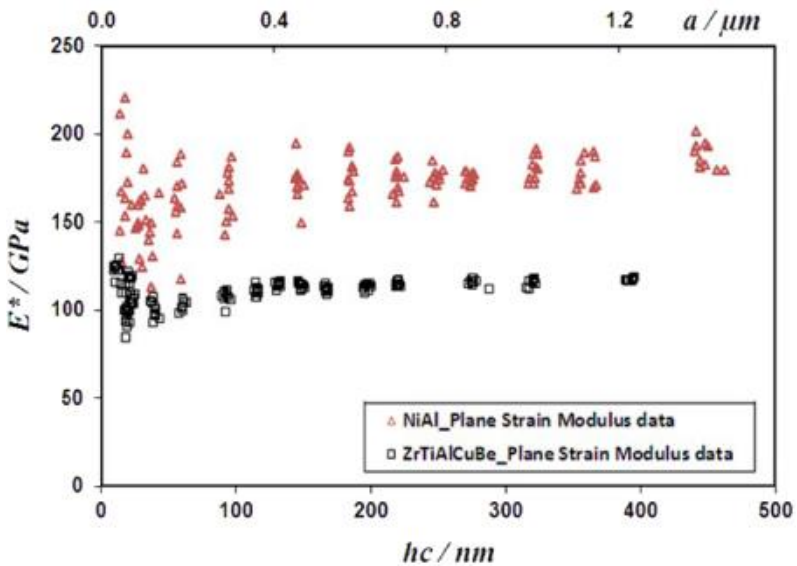




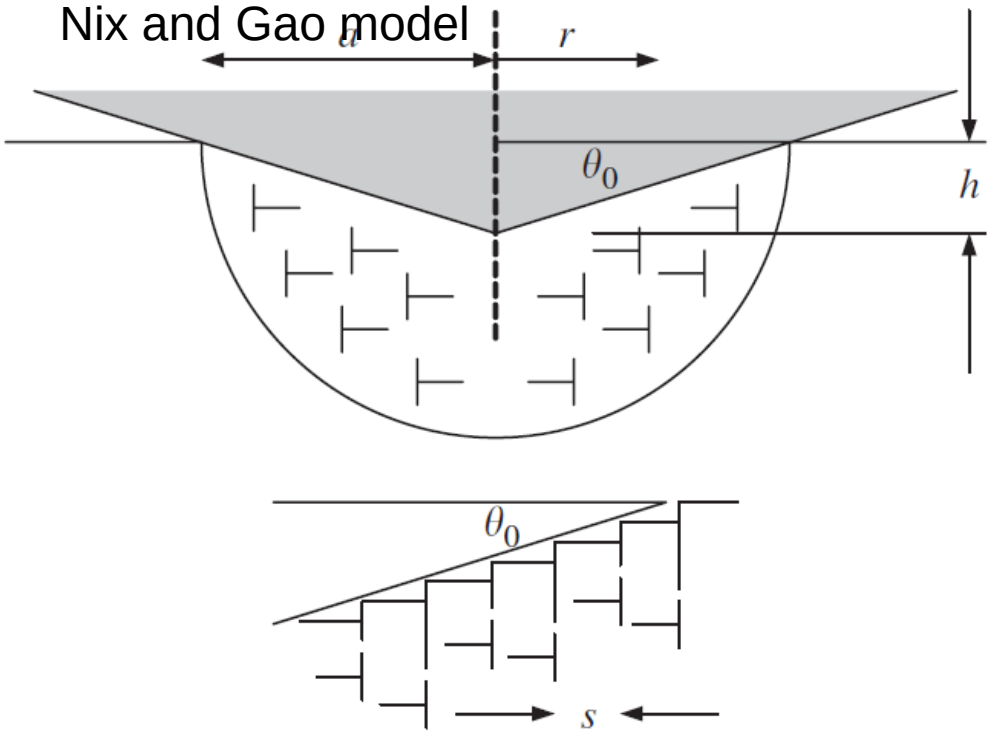


Extrinsic size effects: Indentation size effect





Nix and Gao model



Density of geometrically necessary dislocations:

$$\rho_g = \frac{3h}{2ba^2} = \frac{3}{2}\frac{\tan^2\theta}{bh}$$

X. Hou et al., J Appl Phy, 2013

Extrinsic size effects: GNDs and strain gradient plasticity

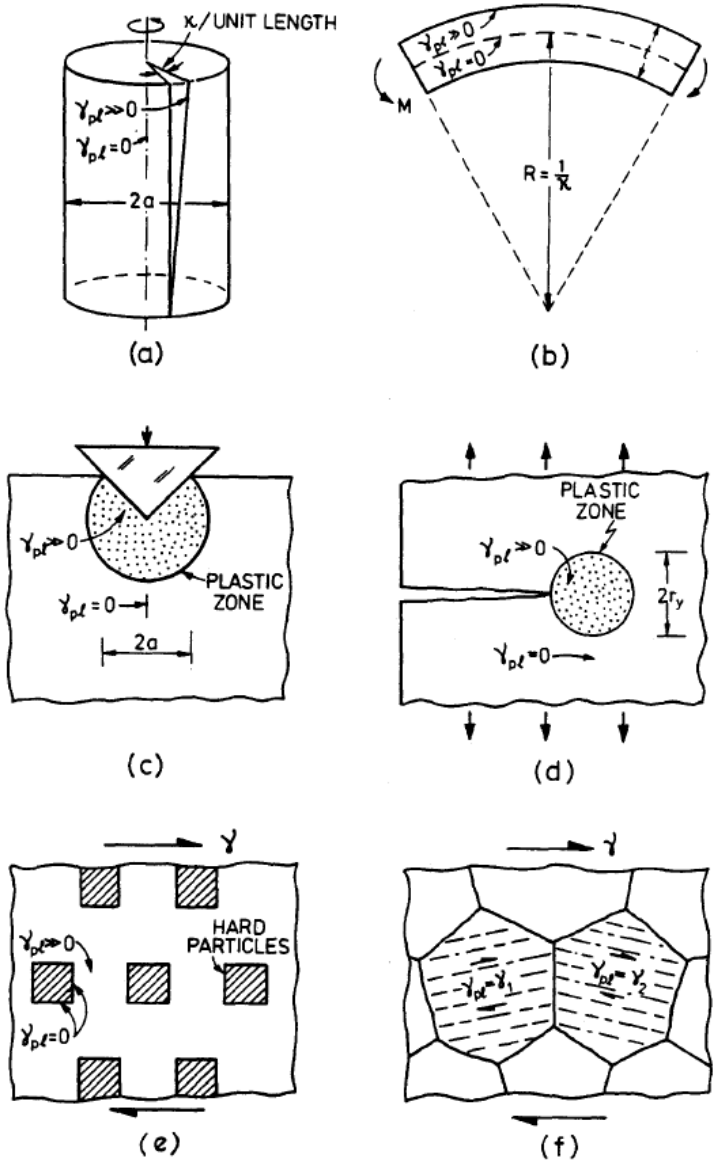
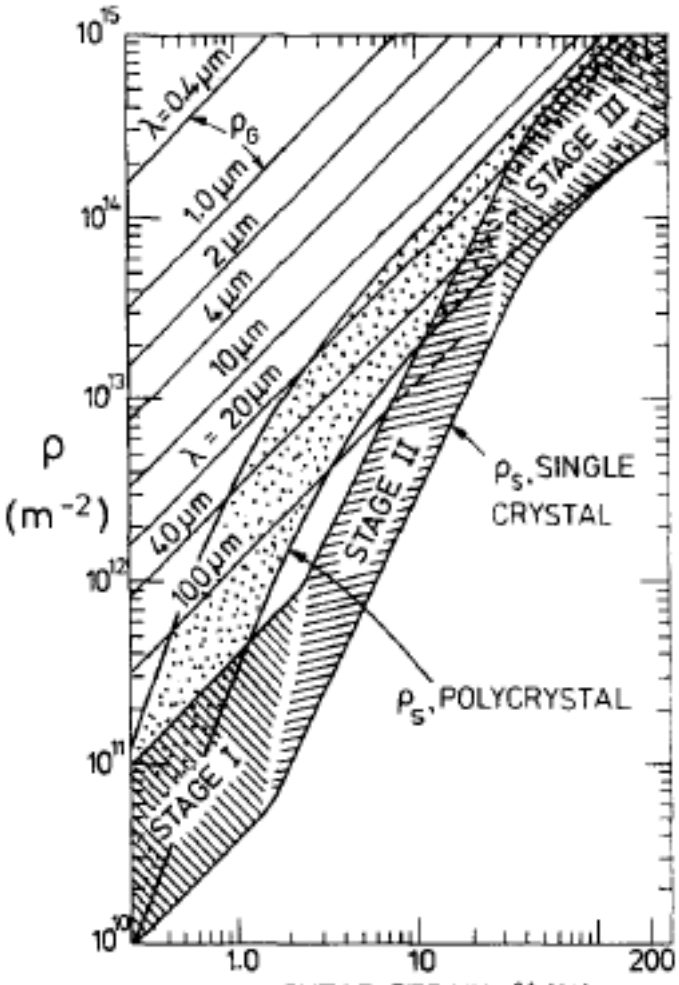
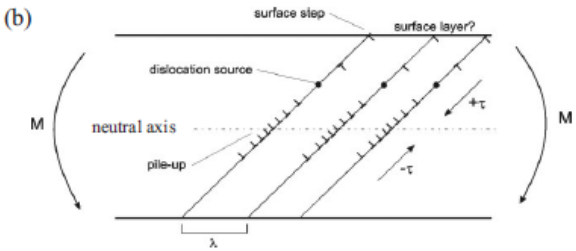


Fig. 1. Plastic strain gradients are caused by the geometry of deformation (a, b), by local boundary conditions (c, d) or by the microstructure itself (e, f).

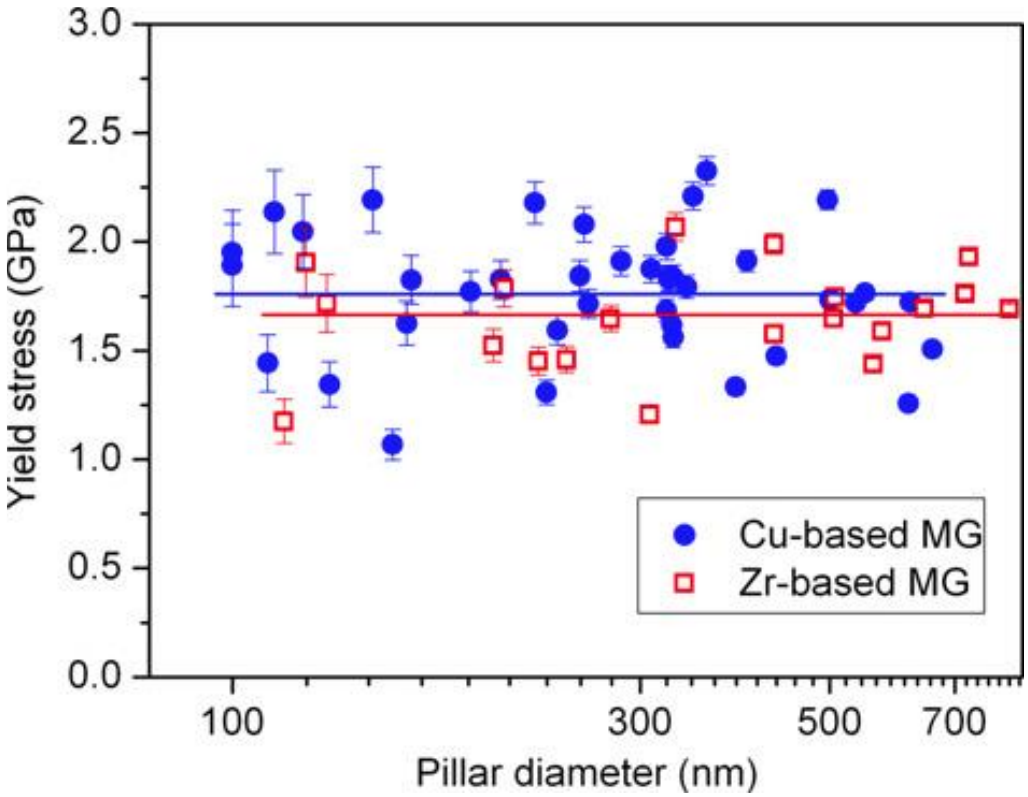




Fleck et al., Acta Mat, 1994

Extrinsic-Intrinsic borderline: No microstructure

Amorphous structures: Metallic glasses

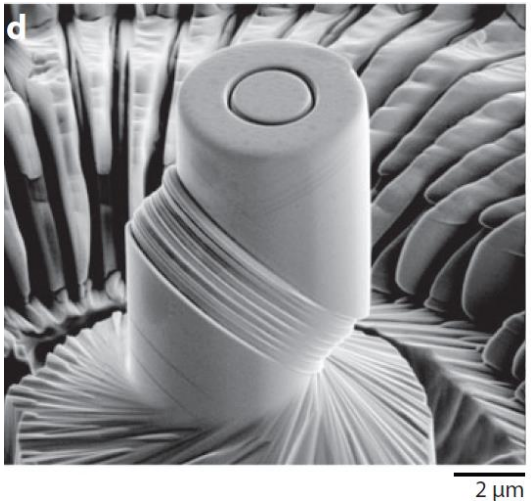


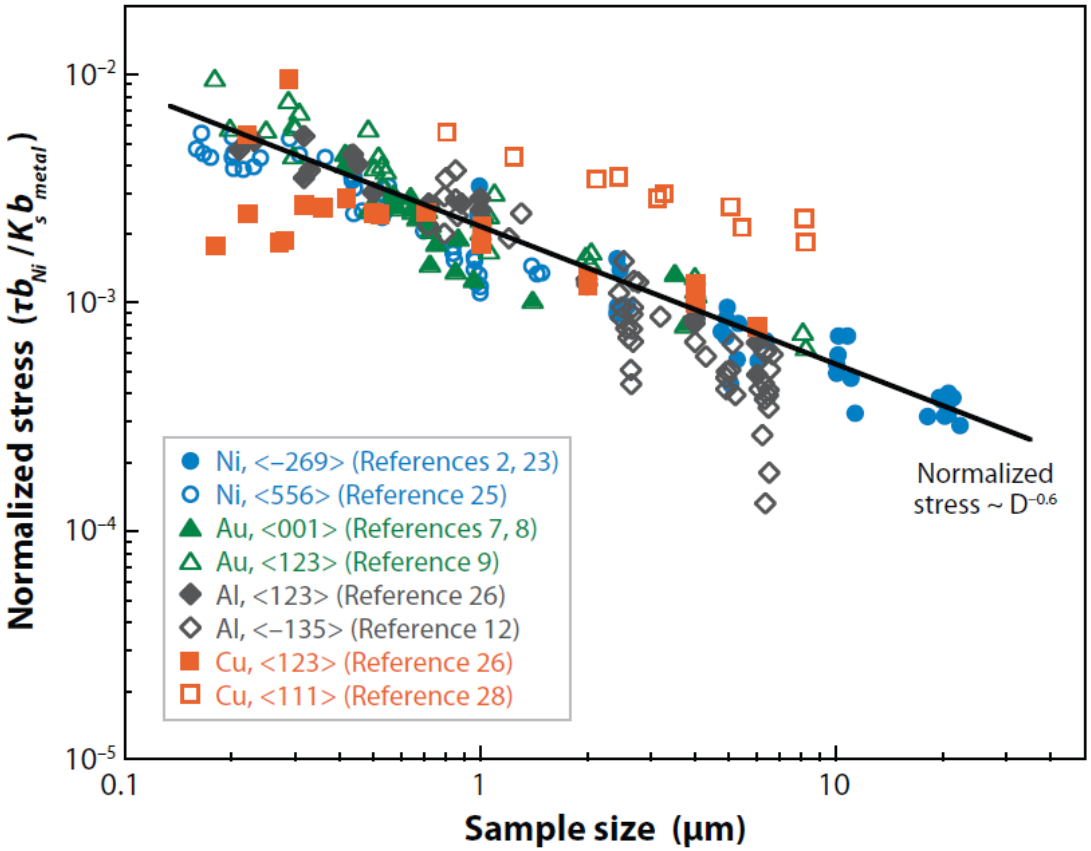
Add microstructre

Greer et al., Prog Mater Sci, 2011

External size effects: Single crystals, Effect of specimen size

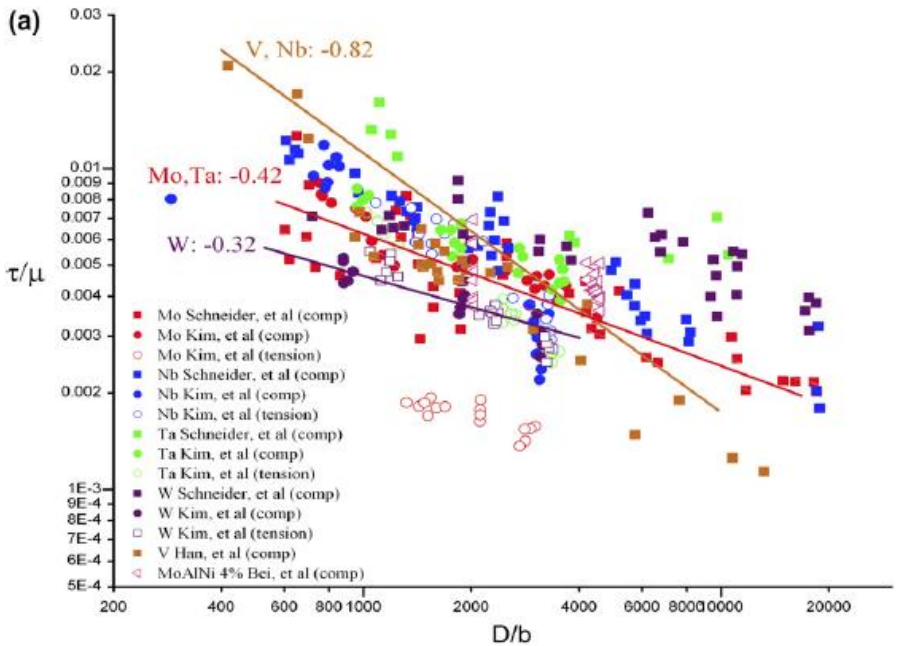
Ductile materials: FCC metals: Smaller is stronger, universal relationship

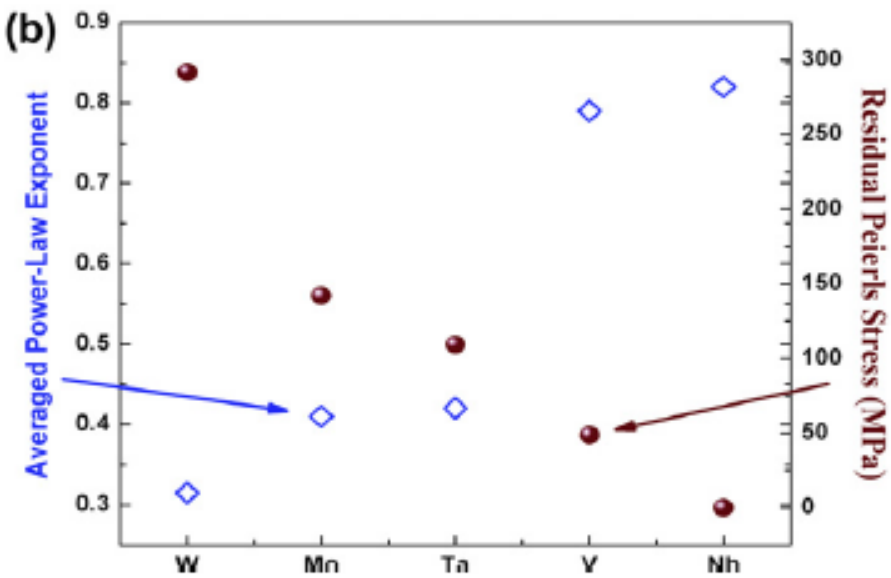




External size effects: Single crystals, Effect of specimen size

Semi-Ductile materials: BCC metals: Smaller is stronger but no universal relationship





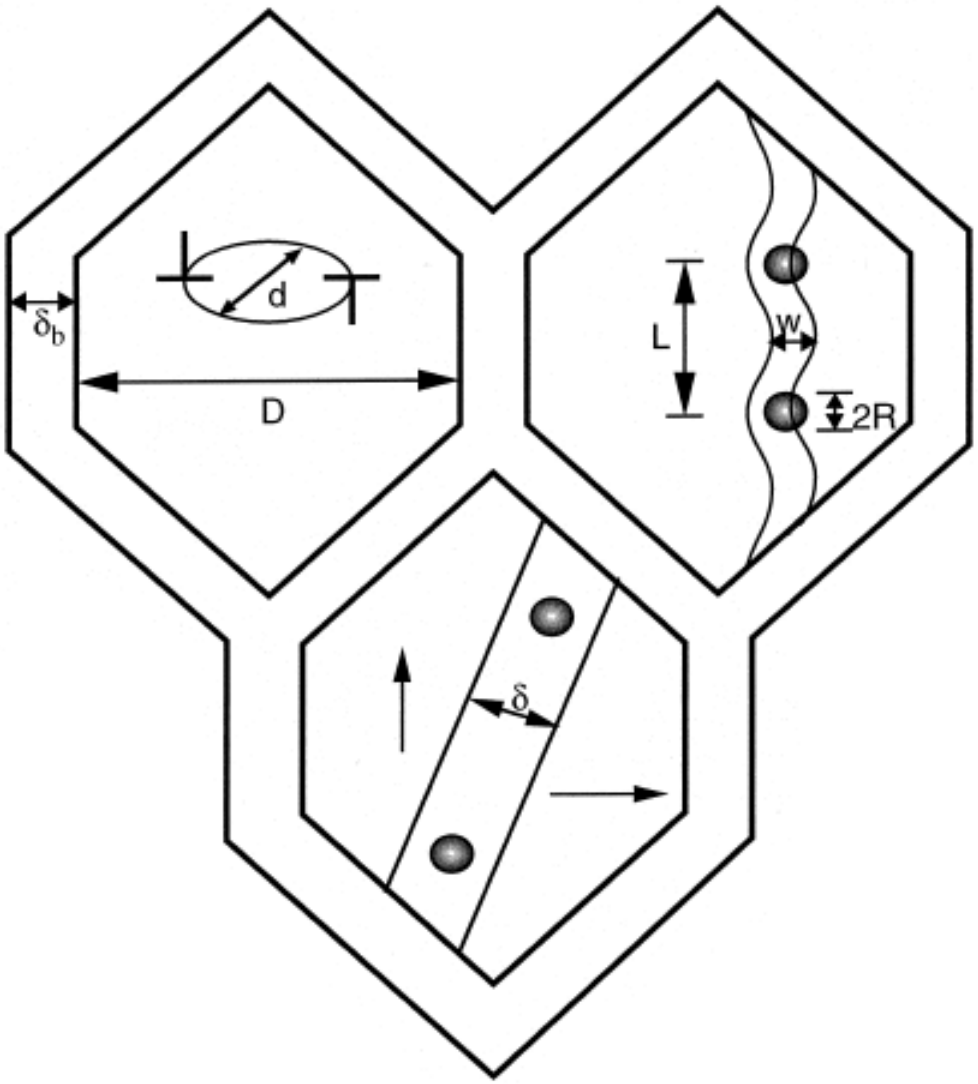
Greer et al., Prog Mater Sci, 2011

External size effects: Single crystals, Effect of specimen size

Dislocation starvation vs dislocation source truncation

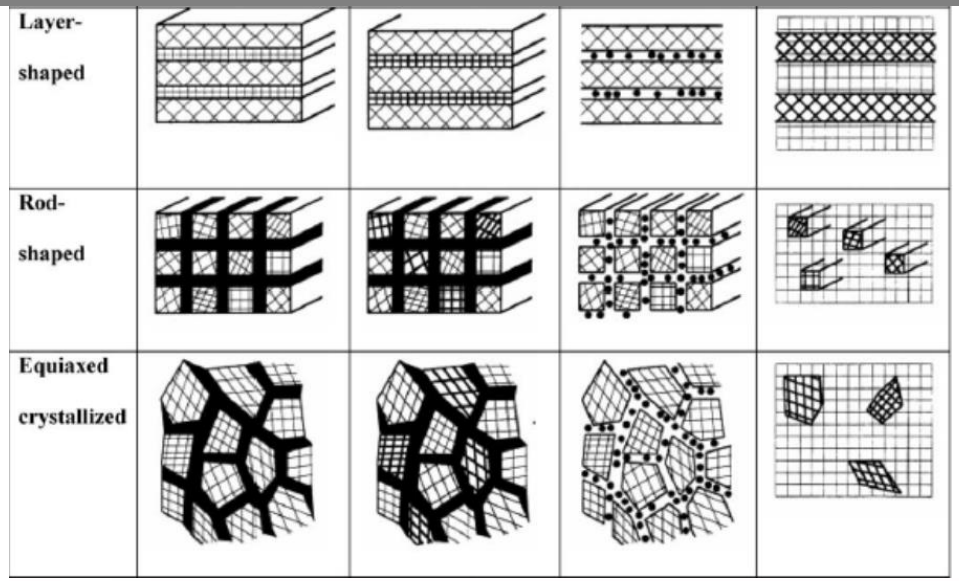
Greer and Nix, PRB, 2006

Intrinsic size effects

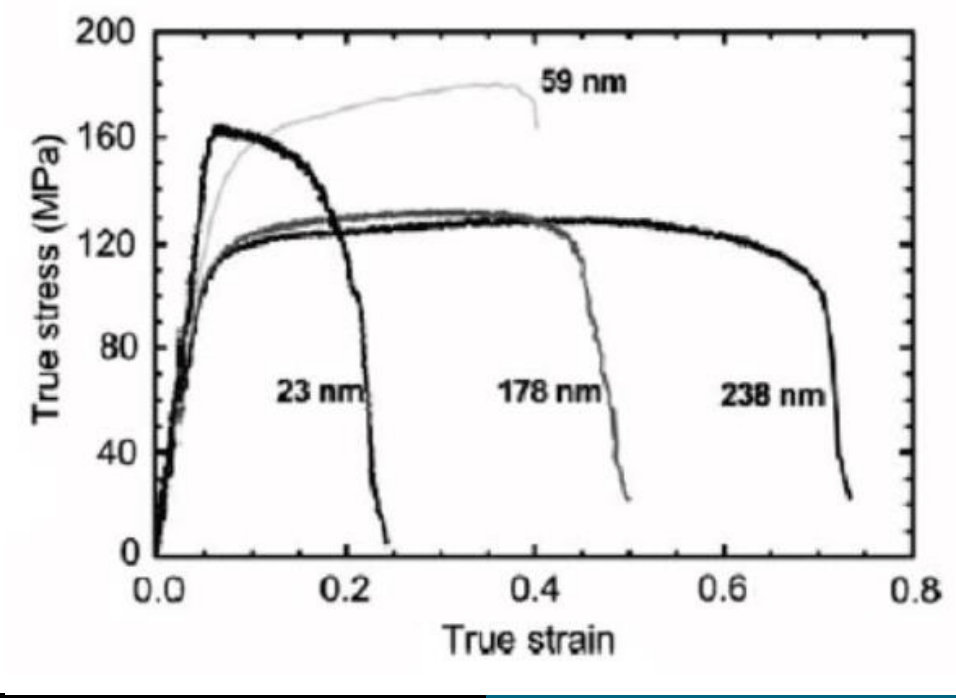


Arzt., Acta Mat, 1998

Intrinsic size effects: Effect of grain size

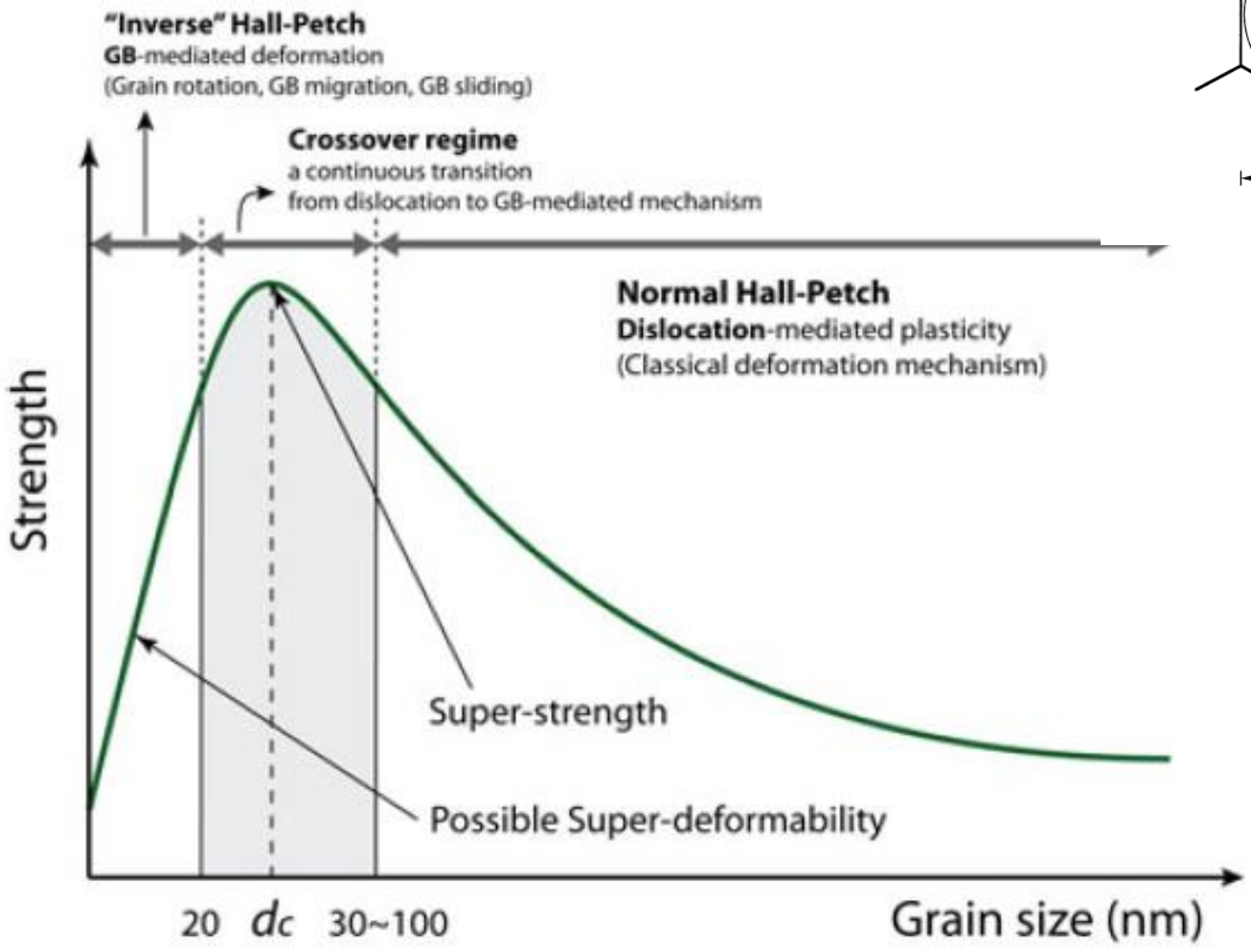


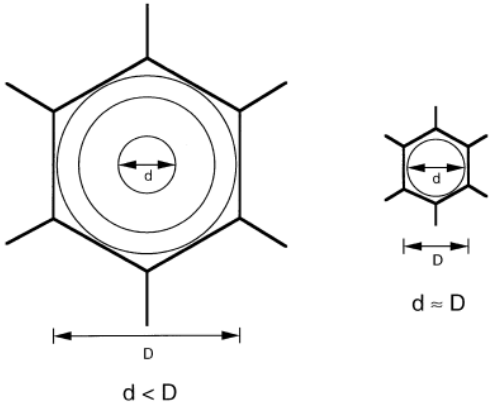
Zn: Tension test



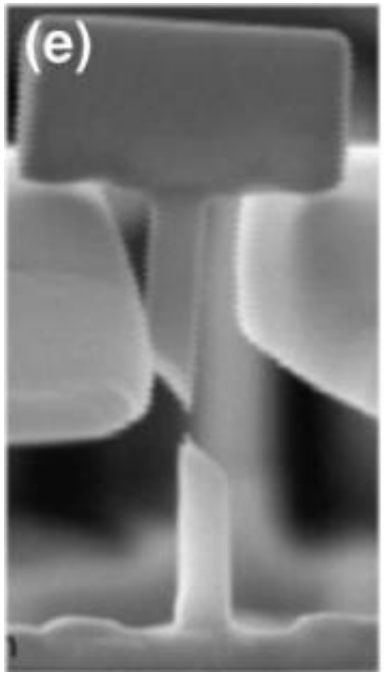
Meyers, Prog Mater Sci, 2006

Intrinsic size effects: Effect of grain size



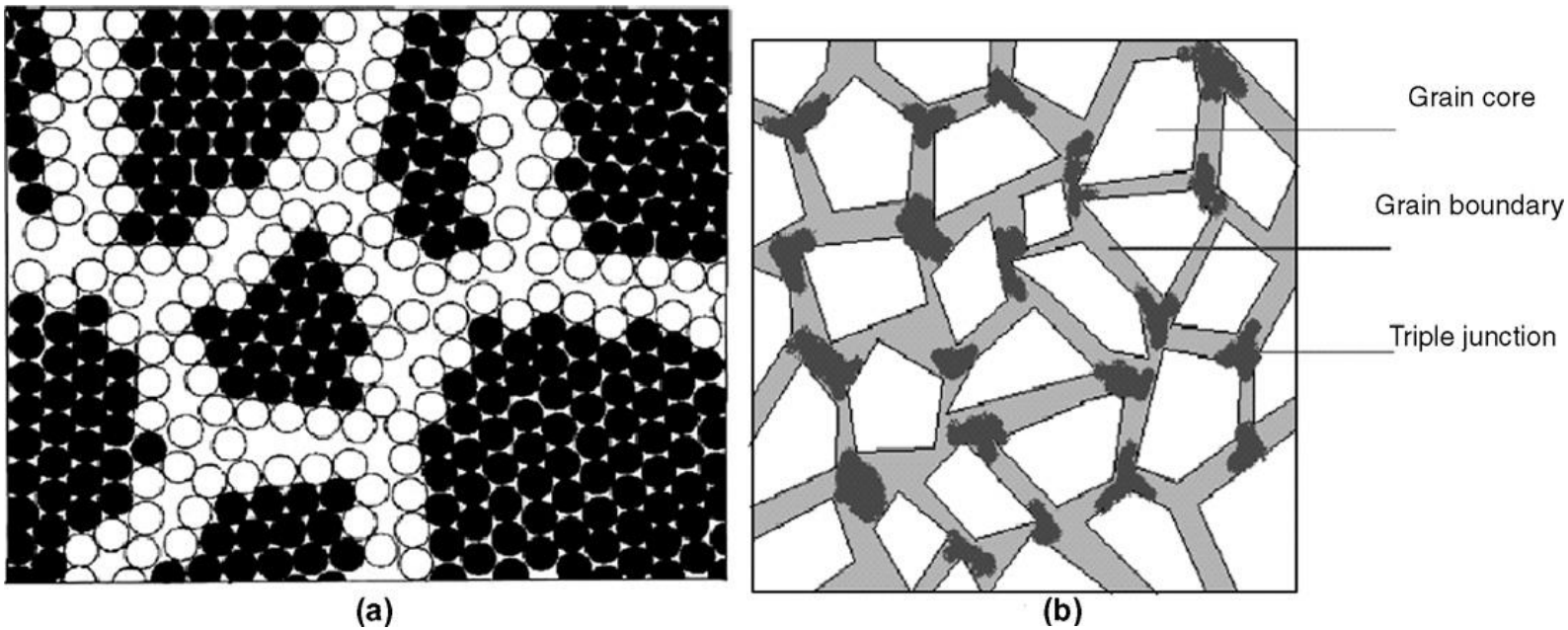


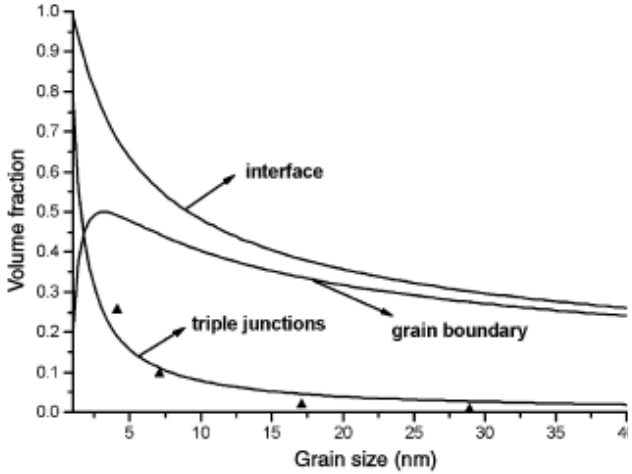
Ni-4%W

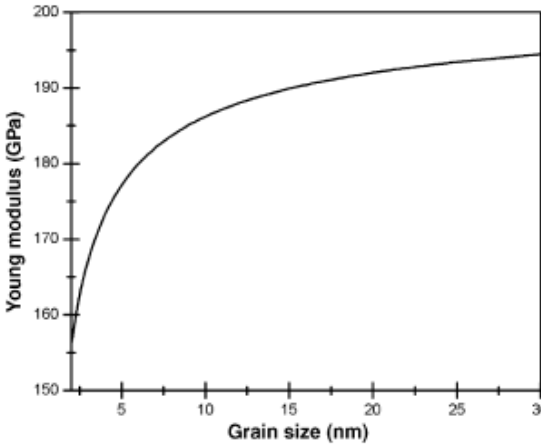


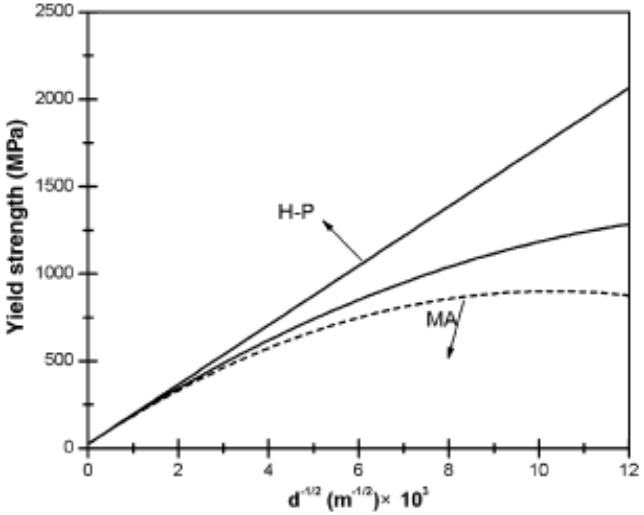
Greer et al., JOM, 2012

Intrinsic size effects: Effect of grain size







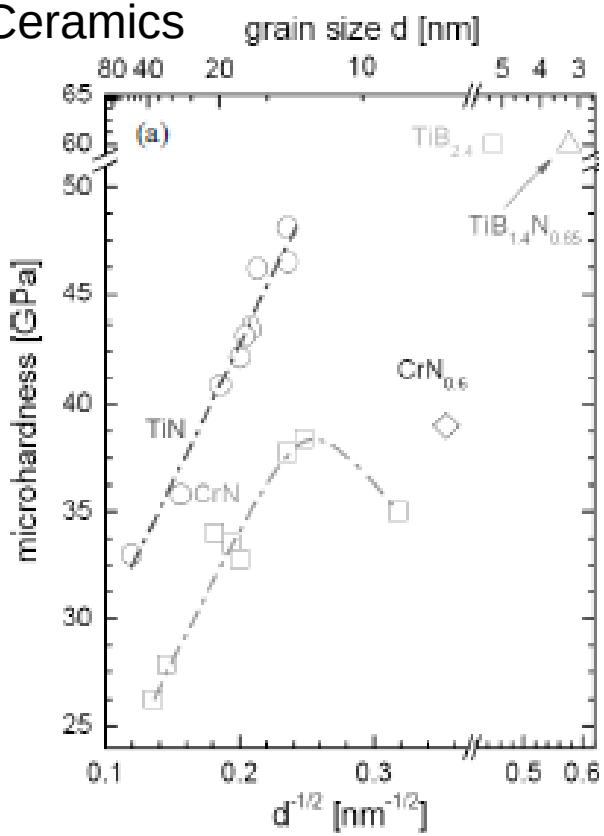


Qing, Int J Solids Struc, 2006

Intrinsic size effects: Poly-crystals

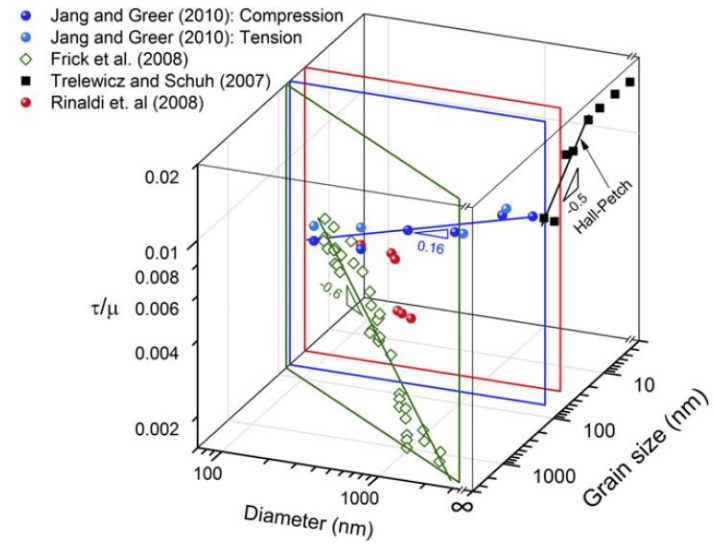
(Inverse) Hall-Petch

Ceramics



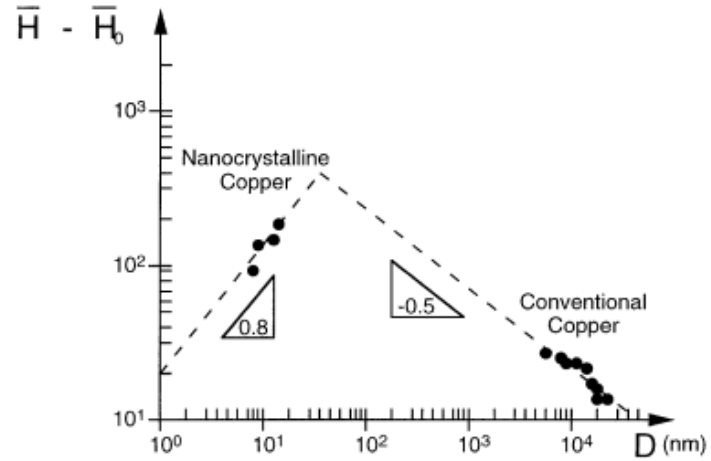
Dehm et al., Adv Eng Mater, 2006

Ni



Greer et al., Prog Mater Sci, 2011

Cu



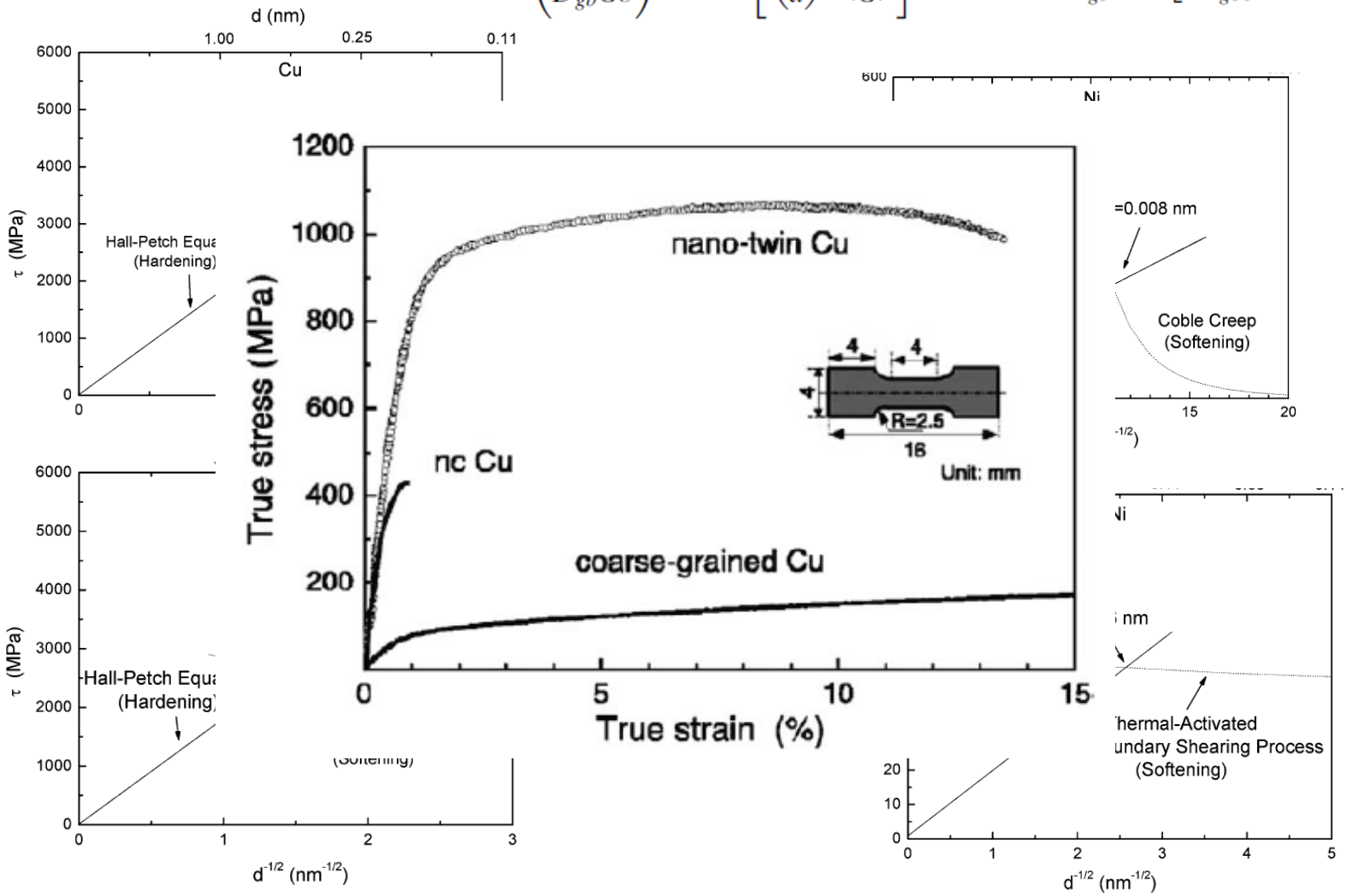
Arzt., Acta Mat, 1998

Intrinsic size effects: Poly-crystals

$$\tau = \tau_0 + c/\sqrt{d}$$

$$\left(\frac{\dot{\gamma}\,kT}{D_{gb}Gb}\right) = A_{Co}\left[\left(\frac{b}{d}\right)^3\left(\frac{\tau}{G}\right)\right]$$

$$\frac{\dot{\gamma}\,b^2}{D_{gb}} = 6\left[\frac{b^3 \upsilon_D}{d D_{gb0}}\sinh\left(\frac{\tau \upsilon}{kT}\right)\right]$$

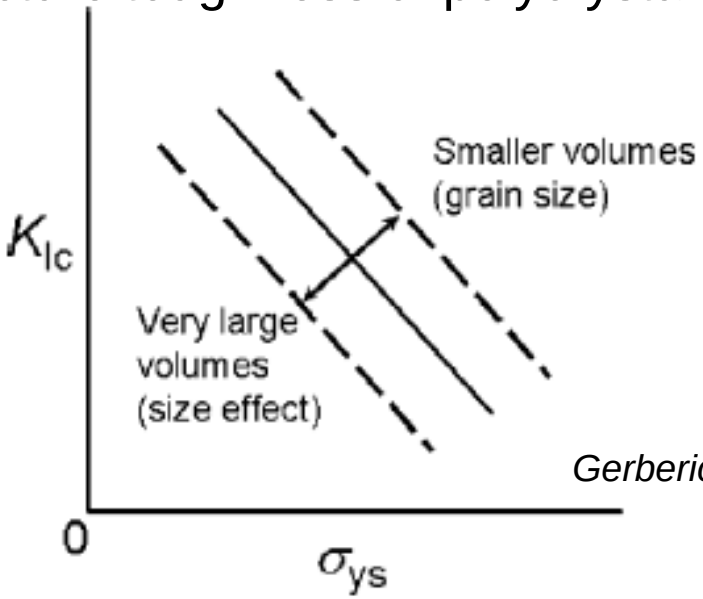


Dislocation vs Twinning vs Grain Boundary Sliding

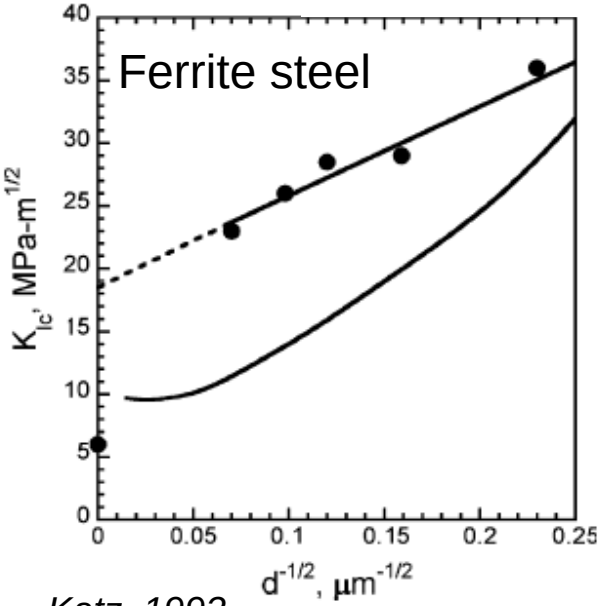
Mohamed et al., Met Trans, 2010

Intrinsic size effects: Effect of microstructure

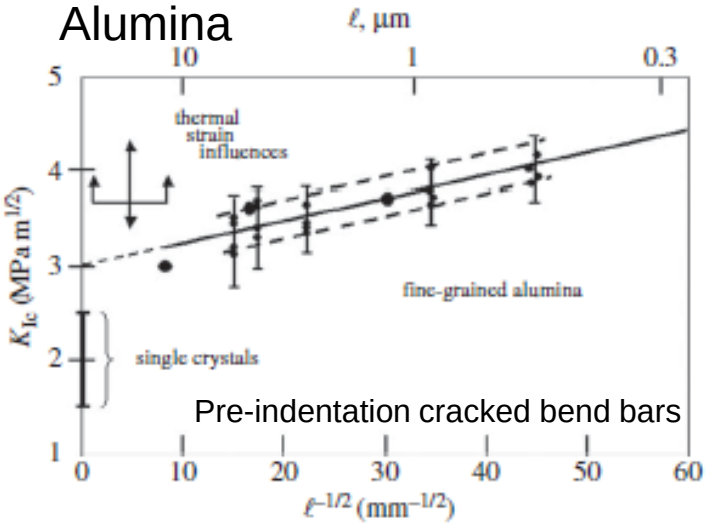
Fracture toughness of polycrystalline materials



Gerberich et al, IJF, 2006



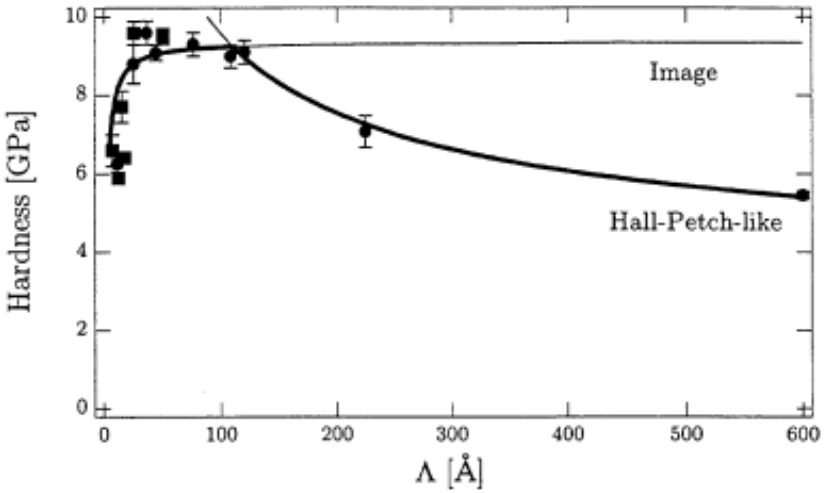
Katz, 1993



Armstrong, Phil Trans, 2015

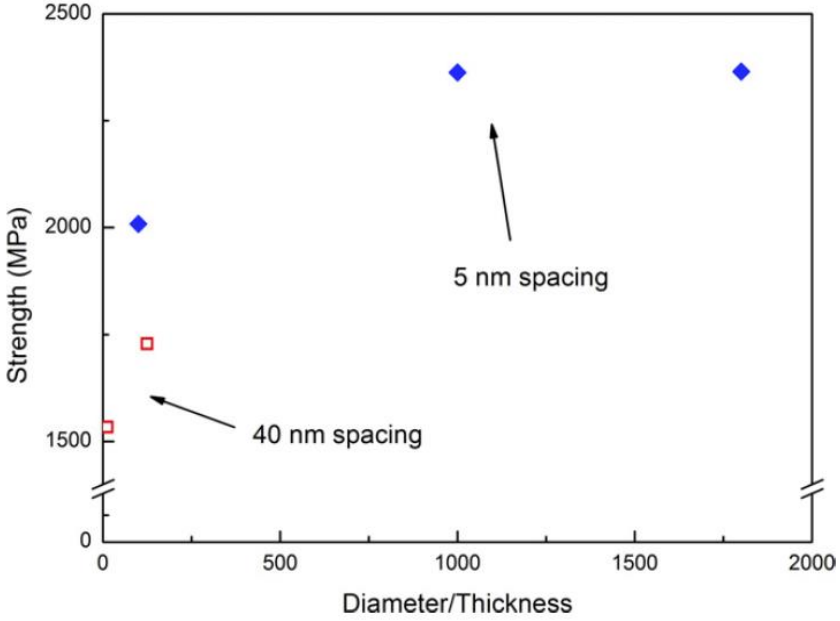
Intrinsic size effects: Multilayers-interface effects

Fe (001)/Pt (001) multilayers



Arzt., Acta Mat, 1998

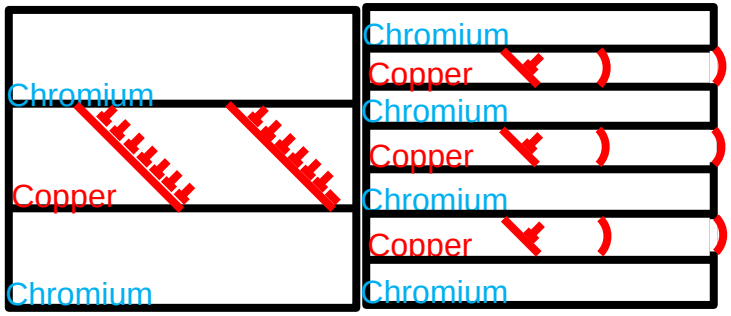
FCC/BCC: Cu-Nb nanolaminates

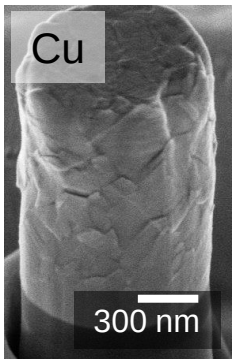


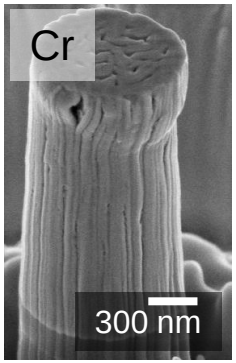
Greer et al., Prog Mater Sci, 2011

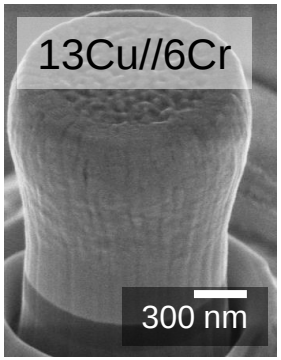
Intrinsic size effects: Multilayers-interface effects

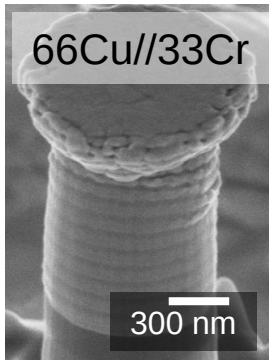
Confined layer slip

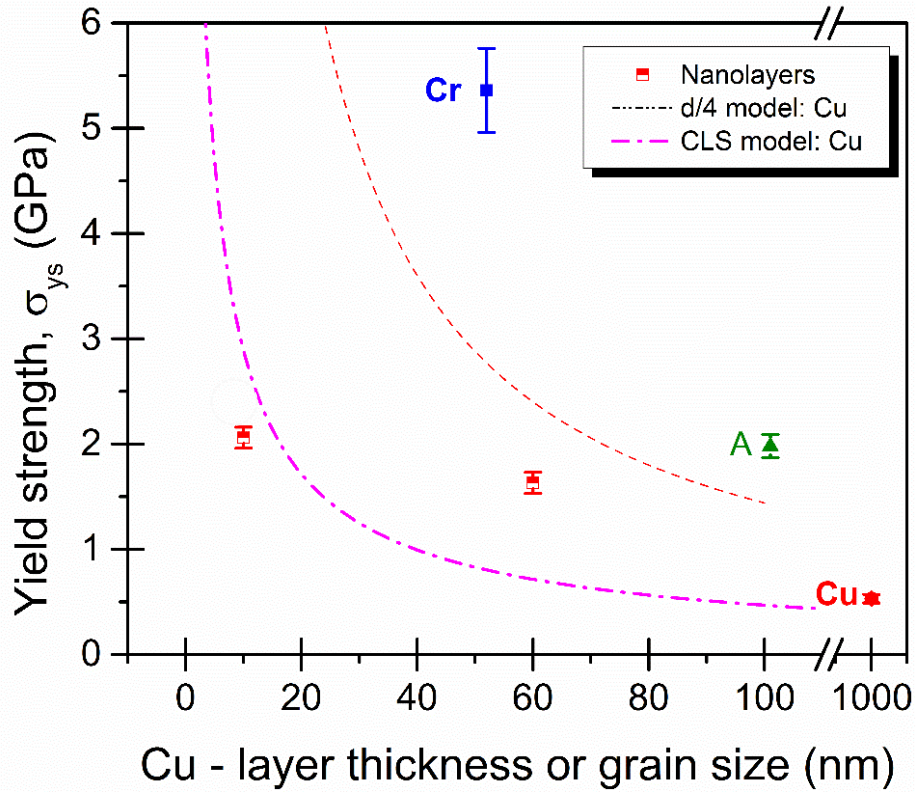






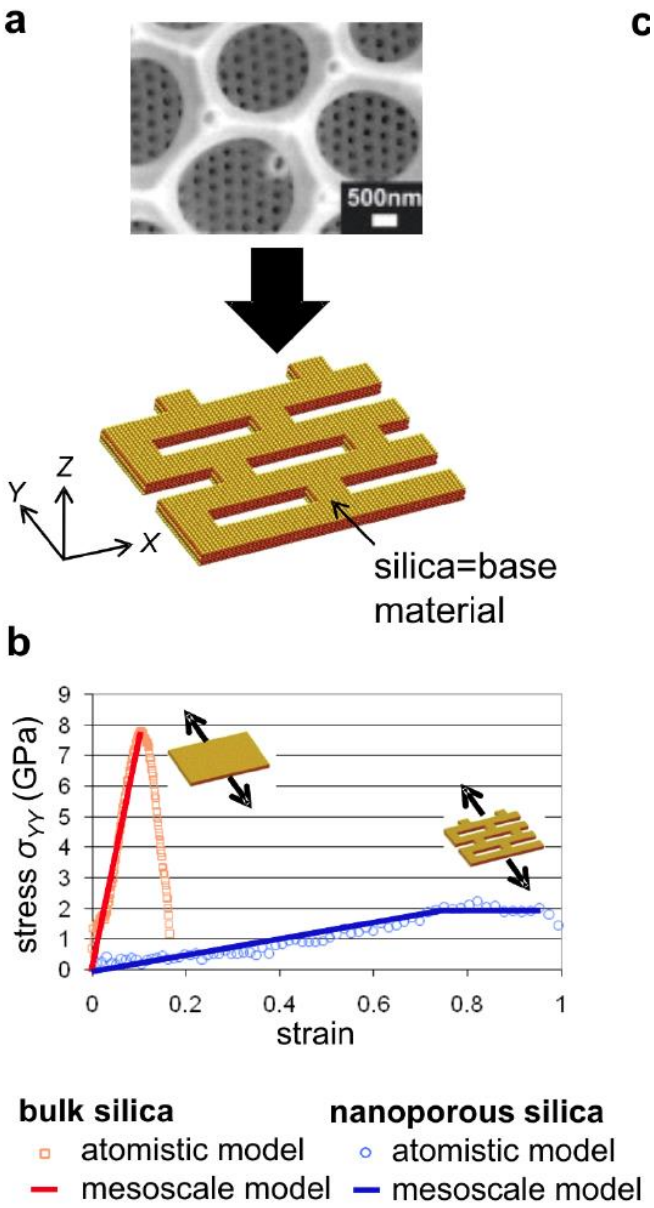


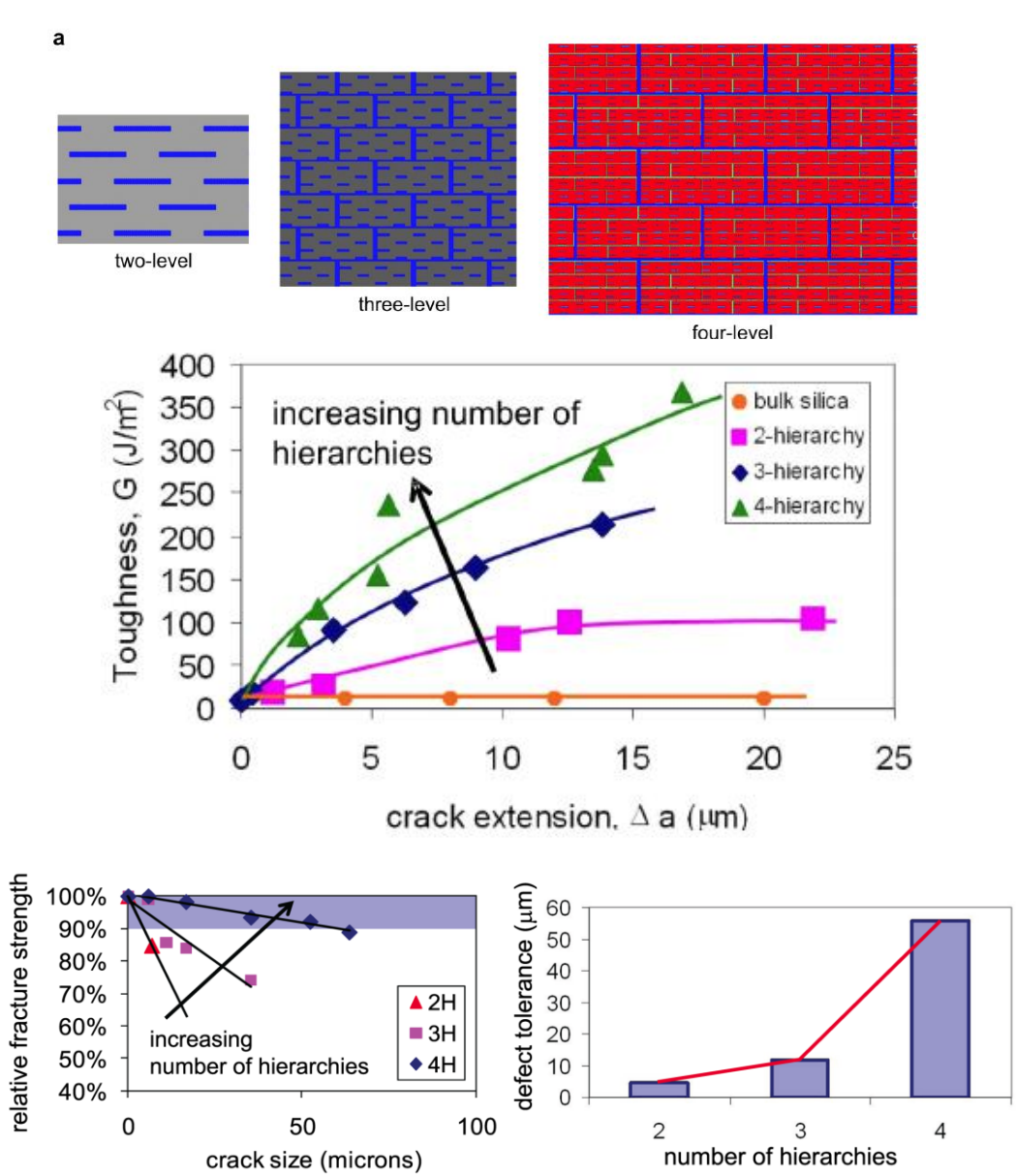




Raghavan et al., Acta Mat, 2015

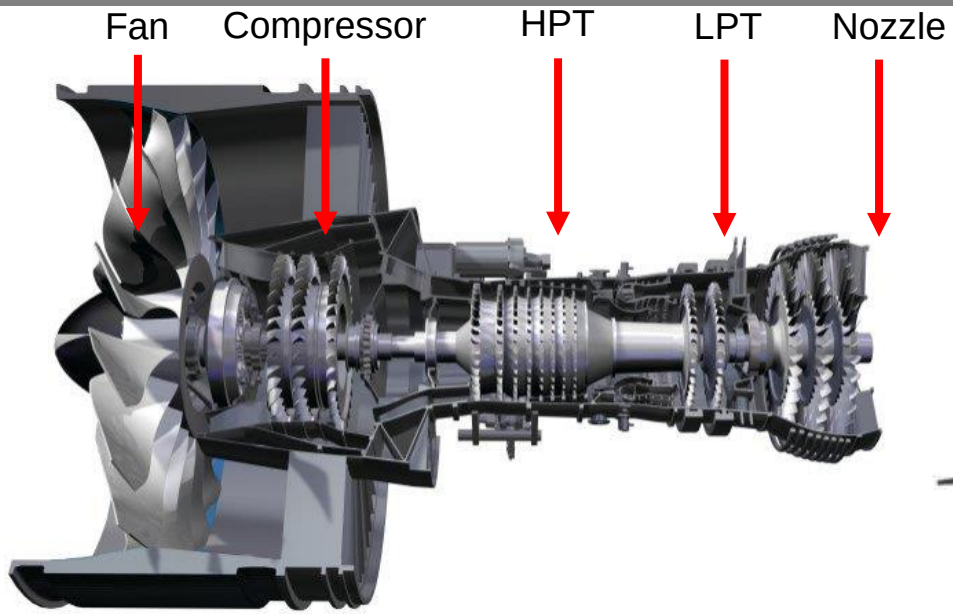
Intrinsic size effects: Architectured interface

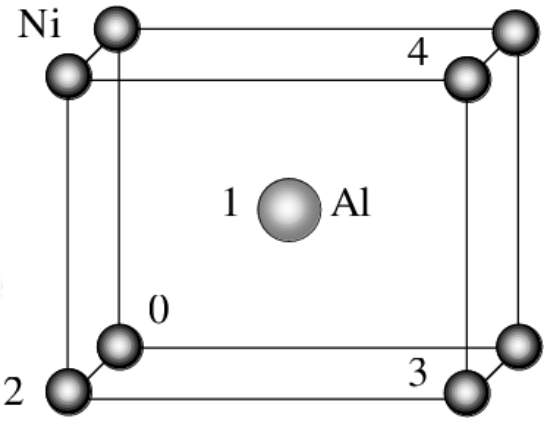


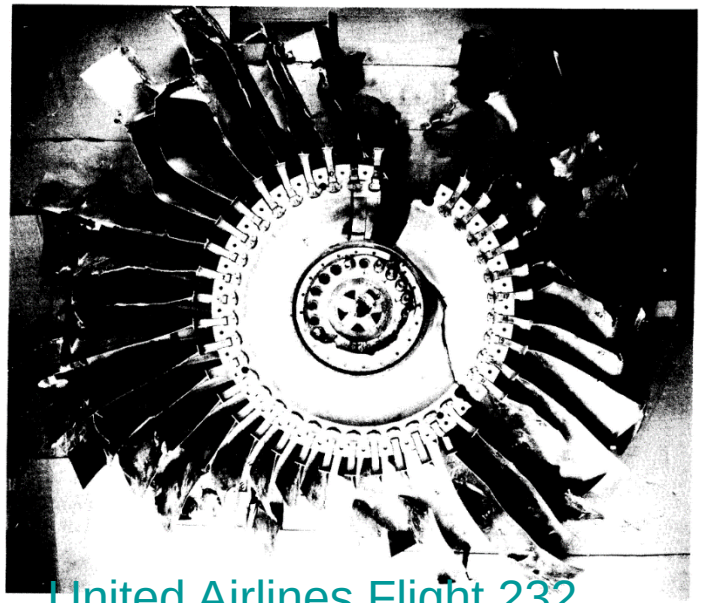


Sen and Buehler, Scientific reports, 2011

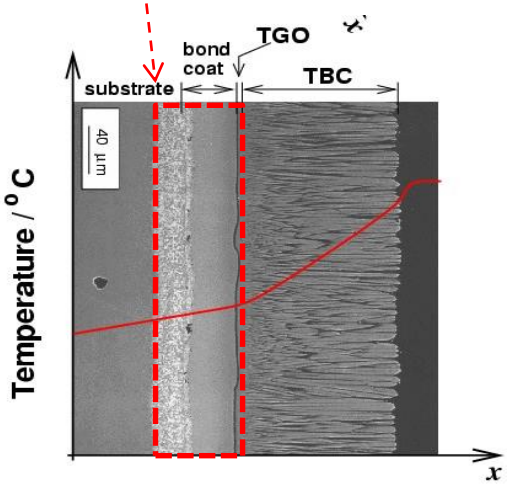
Case study I: NiAl bond coats





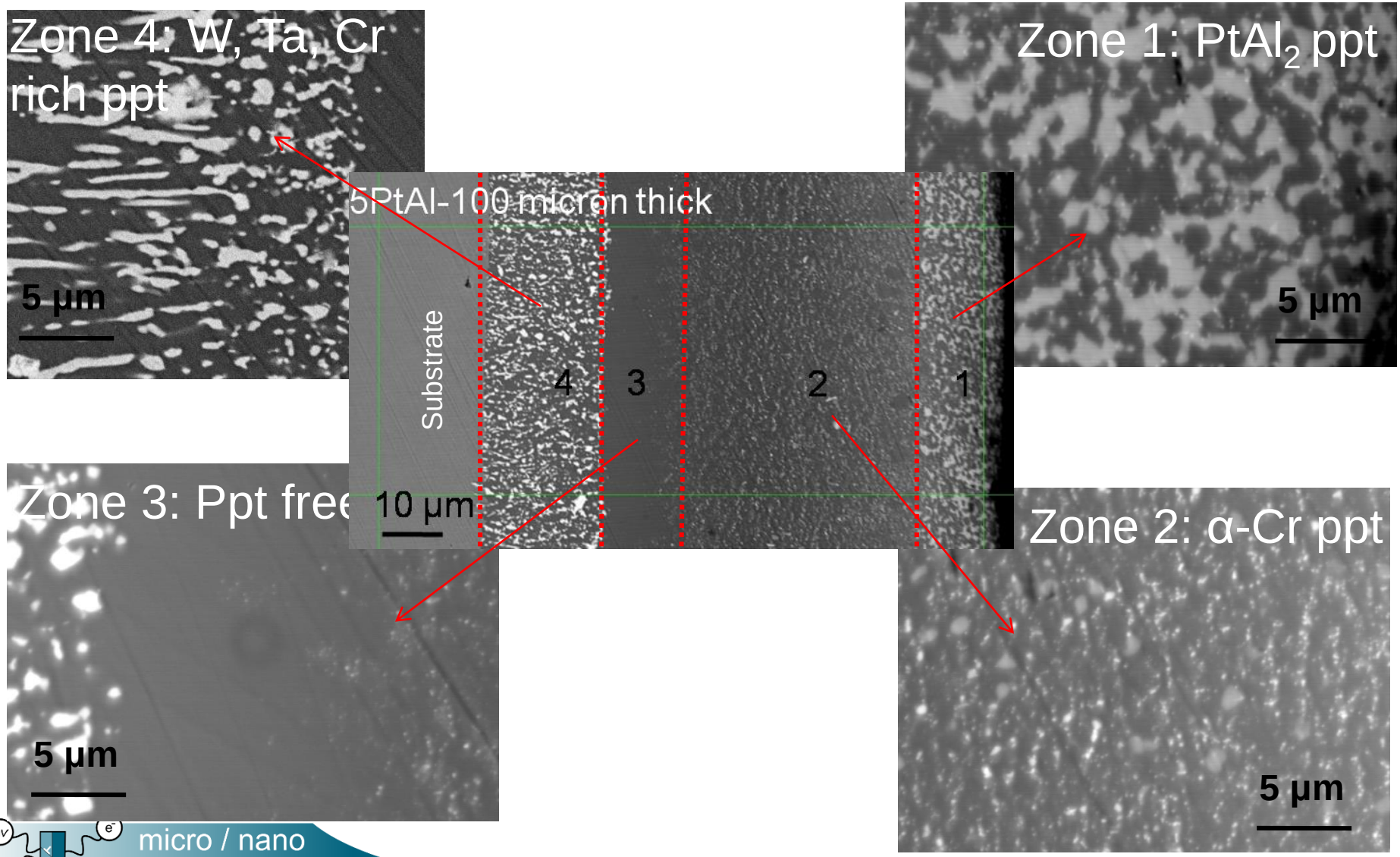


United Airlines Flight 232



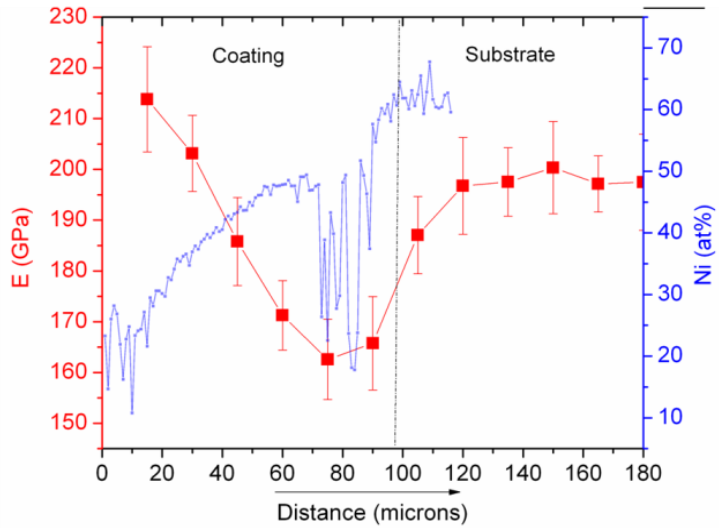
Micro-scale fracture behavior of NiAl bond coats

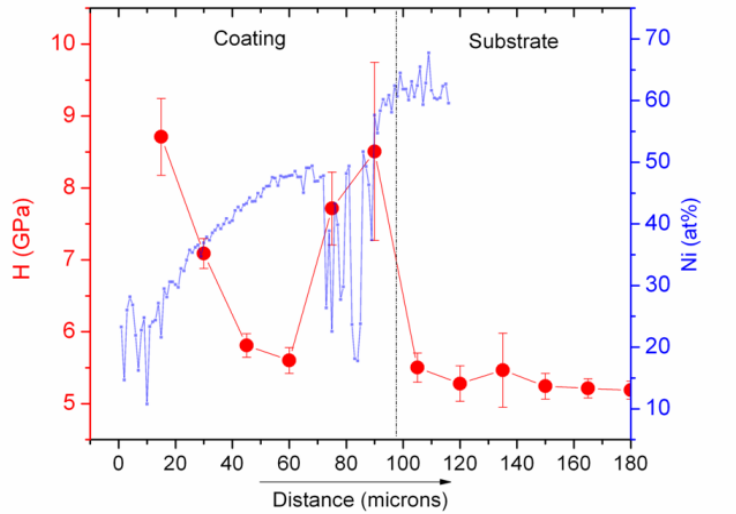
Complex, heterogeneous microstructure with 4 distinct zones



Jaya et. al, Phil Mag, 2012

Micro-scale fracture behavior of NiAl bond coats



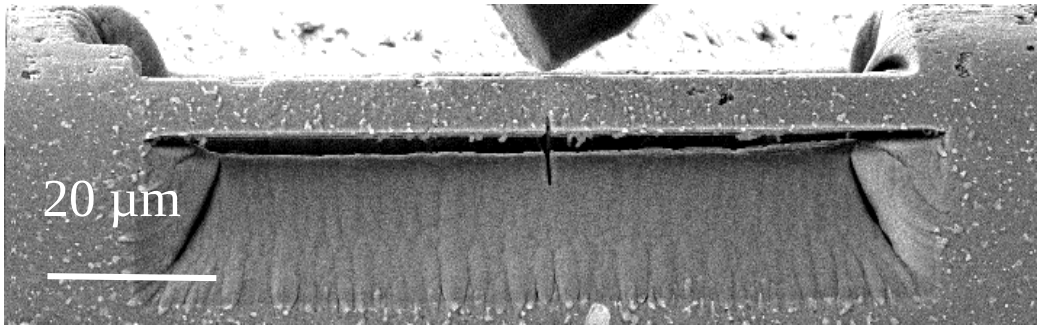


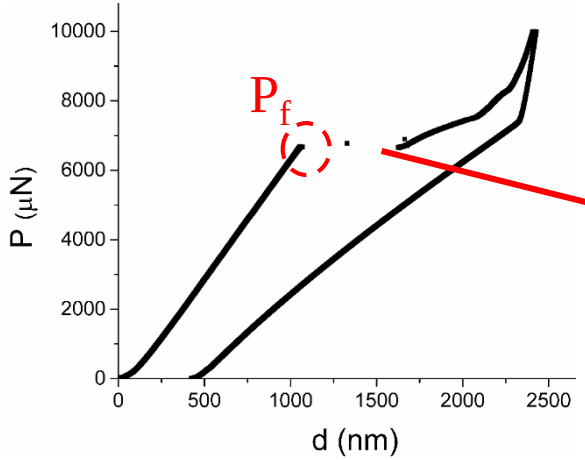
	Micro-scale fracture			K_{IC}/ bulk
	K_{IC}/ LEFM	K_{IC}/ EPFM	K_{IC}/ CTOD	
NiAl soft [110]	2.0[1]	3.5[2]	1.8[2]	1.8-4.5[3]
NiAl hard [100]	3.6[1]	8.6[2]	7.9[2]	6.9[3]
NiAl bond coat (low Al activity)	2.6[4]	N.A	N.A	N.A
NiAl bond coat (high Al activity)	8.5[5]	N.A	N.A	N.A

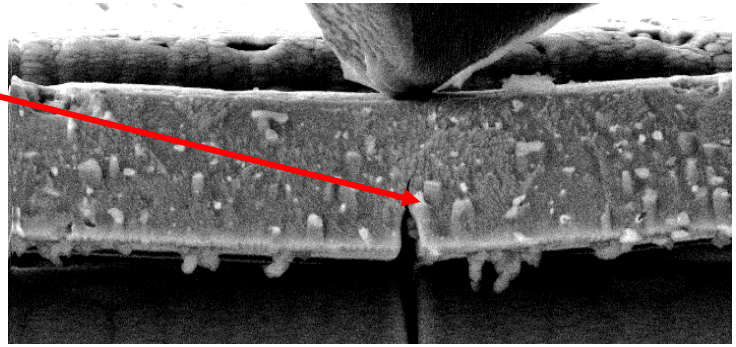
[1] F. Iqbal, J. Ast, M. Goeken and K. Durst, Acta. Mater. **60**, 1193 (2012).
[2] J. Ast, T. Przybilla, V. Maier, K Durst and M. Goeken, J. Mater Res. **29**, 2129 (2014).
[3] D. B. Miracle, Acta. Metall. Mater. **41**, 649, (1993) .
[4] R. Webler, M. Krottenthaler, S. Neumeier, K. Durst and M. Göken, Int. Symp. Superalloys, 93 (2012).
[5] [illegible] ayaram, Phil. Mag, (2015).

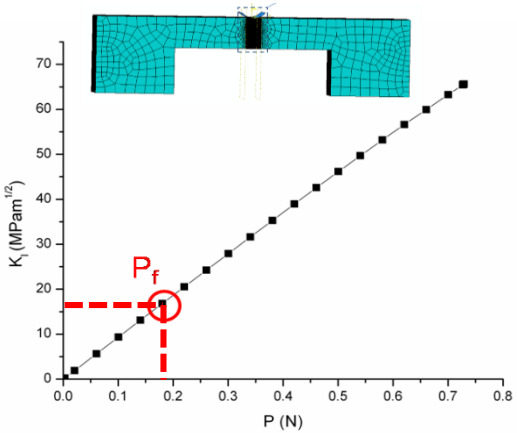
Micro-scale fracture behavior of NiAl bond coats

Pop-in load/change in compliance correlated with crack initiation or propagation event





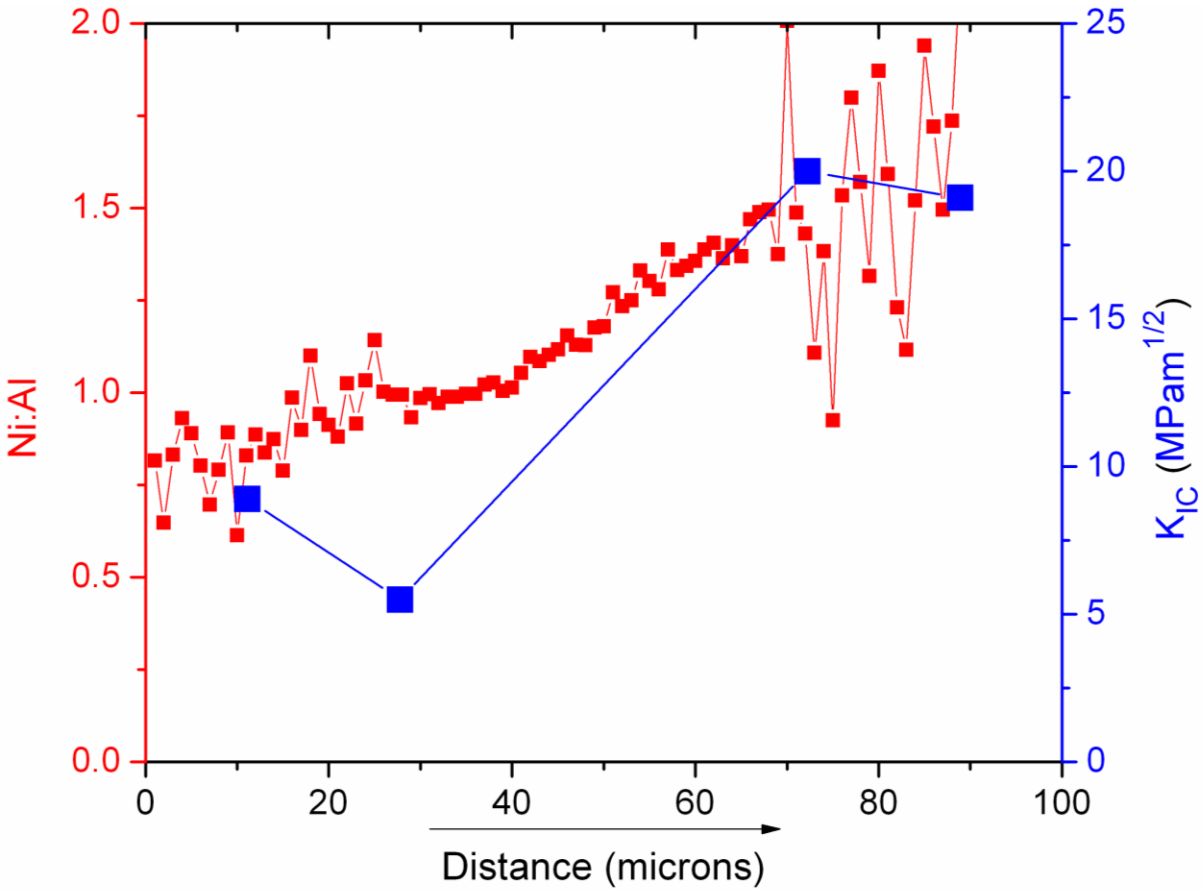


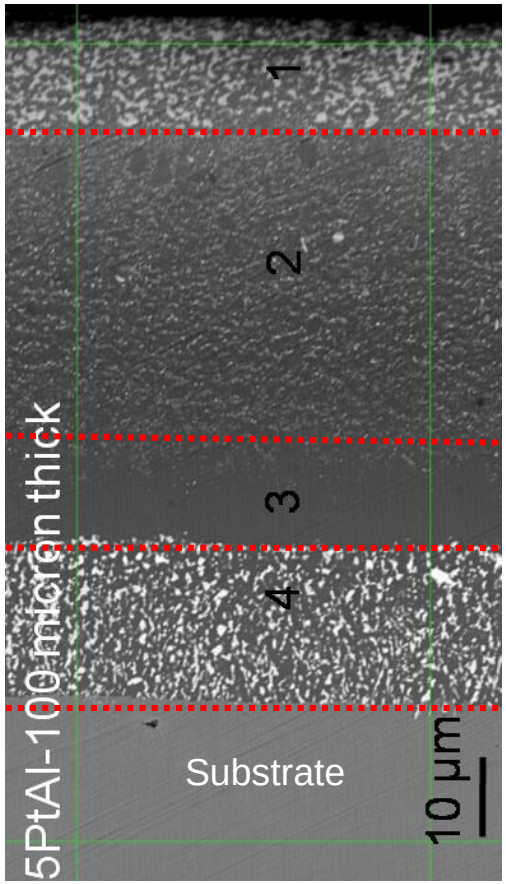


Jaya et. al, Phil Mag, 2012

Micro-scale fracture behavior of NiAl bond coats

Fracture toughness as a function of matrix stoichiometry

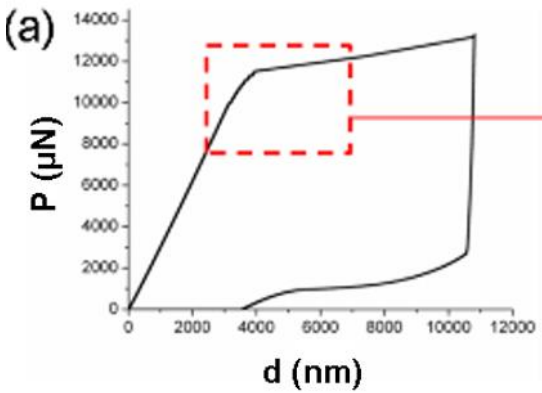


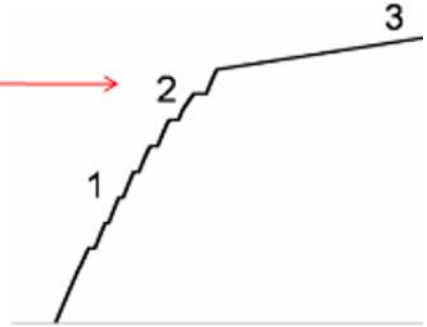


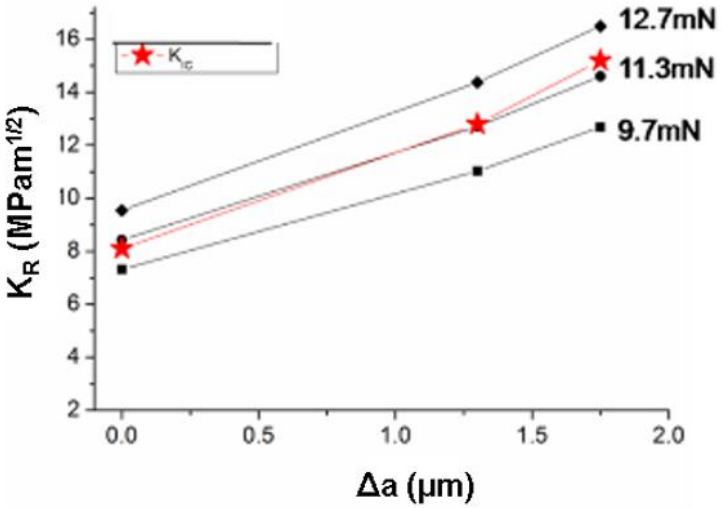
Jaya et. al, Phil Mag, 2012

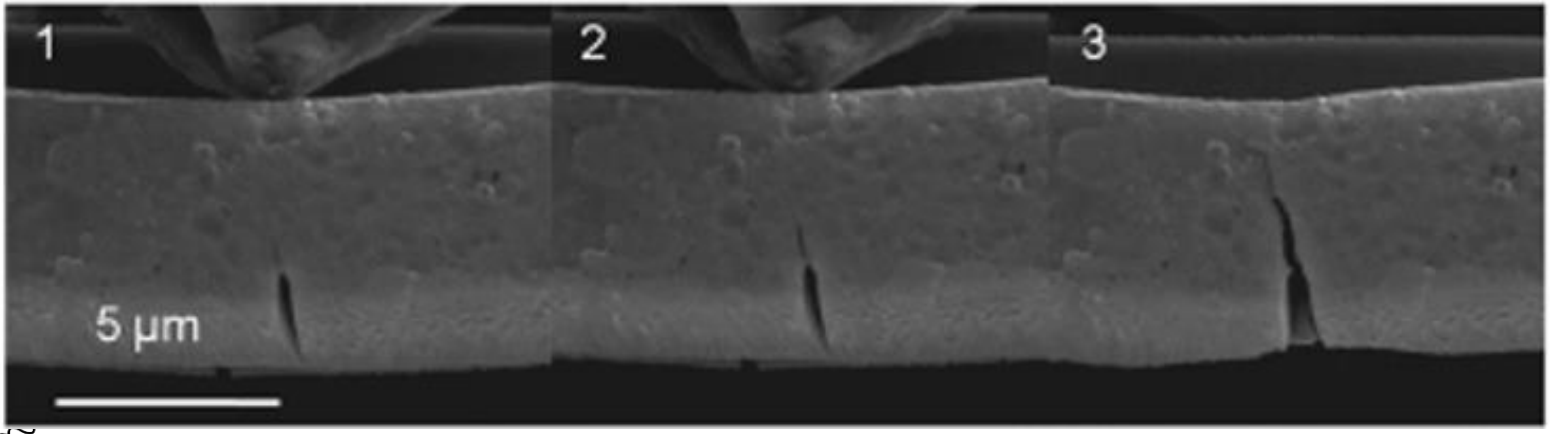
Micro-scale fracture behavior of NiAl bond coats

R-curve behavior







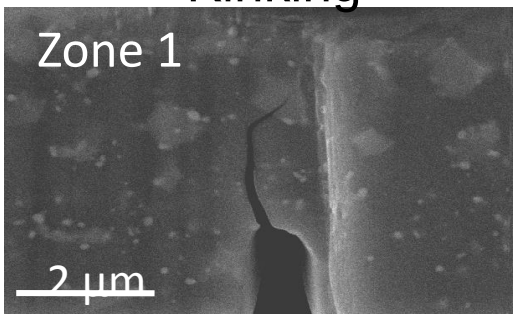


Jaya et. al, Phil Mag, 2015

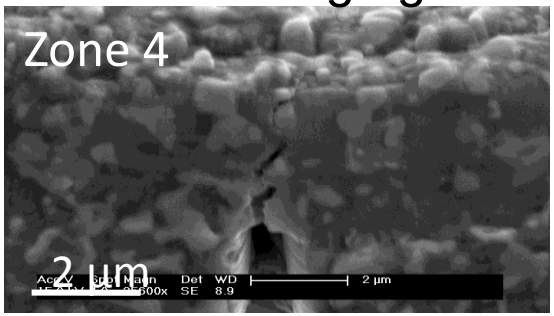
Micro-scale fracture behavior of NiAl bond coats

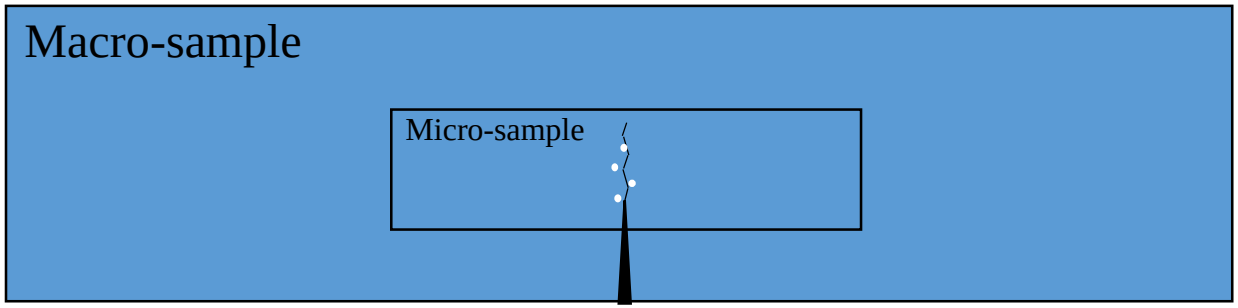
Toughening mechanisms in individual zones at the micron-scale

Kinking



Bridging





What did we learn? :

- Intrinsic fracture toughness K_{IC} increases with Ni:Al ratio across the bond coat
- Crack closure and rising fracture toughness seen at micron-length scales
- Toughening mechanisms vary depending on the zone.

Jaya et. al, Phil Mag, 2015

Summary

- There is no universal size effect across all length scales and all material systems
- Each material property responds differently to length scale effects
- There is a need to study each material system at the length scale regime at which it is applicable (designed for service)

Thank you for your attention!!