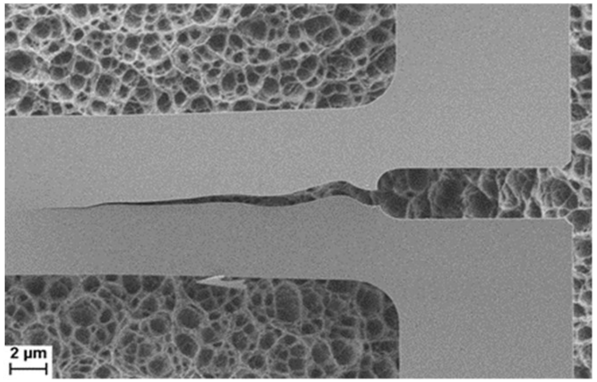
Institute of Mechanics, Materials and Civil Engineering
& Research Center on Architectured and Composite Materials (ARCOMAT)
& Research center in micro and nanoscopic materials and electronic devices (CERMIN)

Damage and fracture in thin films
and other nano-objects

T. Pardoen

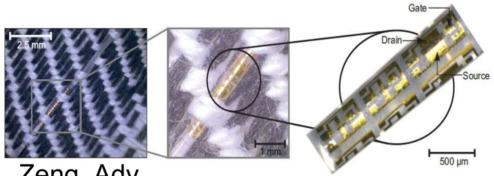


Workshop “Lancement ERC TimeMan”
Villeneuve-d’Ascq, 6 février 2019

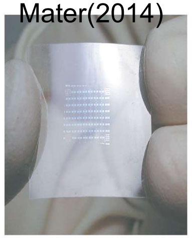
UCL Université catholique de Louvain

Fracture of thin films and coating dictates the reliability of a variety of modern technologies

Flexible electronics



Zeng. Adv Mater(2014)



Nomura, Kenji et al. *Nature* (2004).

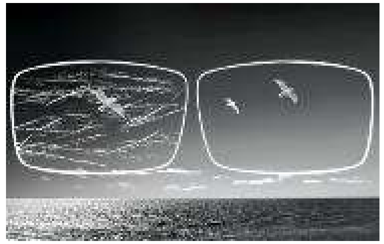
Philips' fluid' *smartphone*

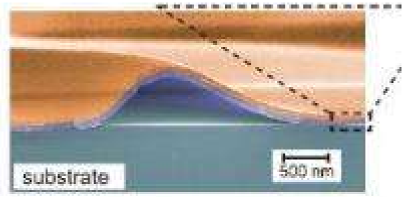
S. Coyle. *MRS Bull (2007)*

Thin functional coatings

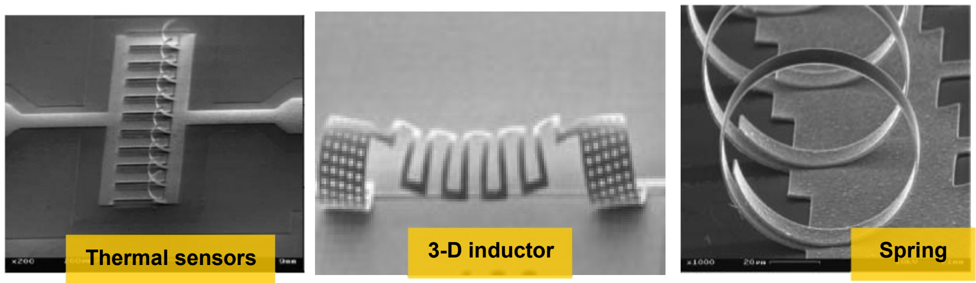
on glass, steel, Al, etc …
must resist :

- thermomech. loadings
- forming operations after deposition
- impact
- scratch and wear



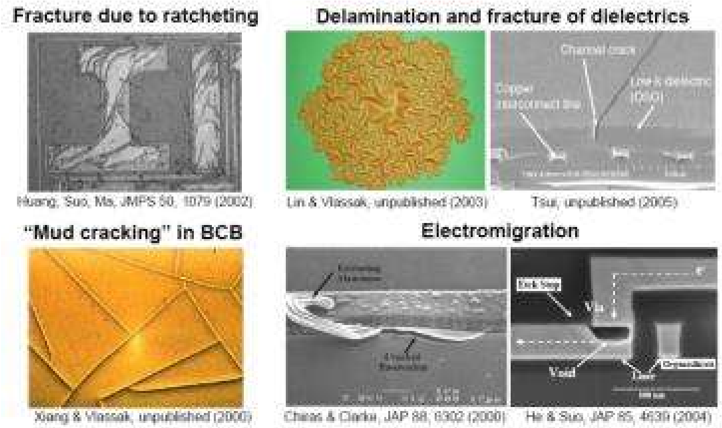


Mems and Nems



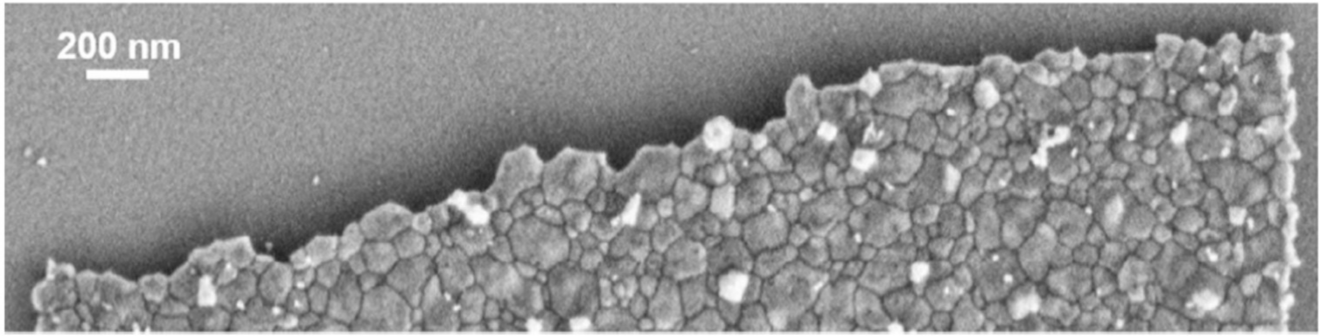
F. Iker, N. André, T. Pardoen, J.-P. Raskin, *JMEMS* (2006)

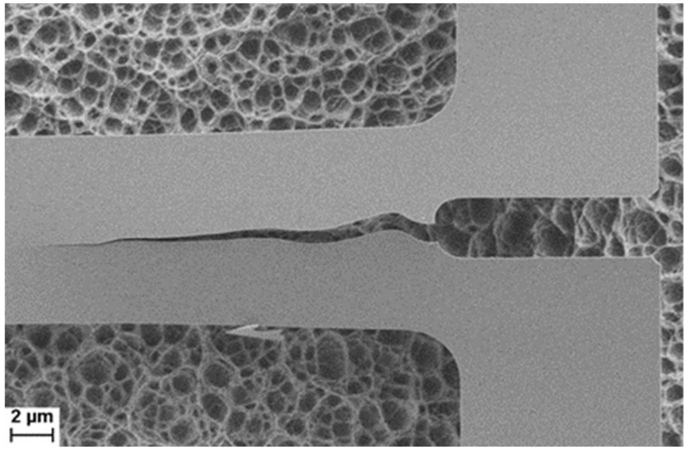
Micro and nano-electronics

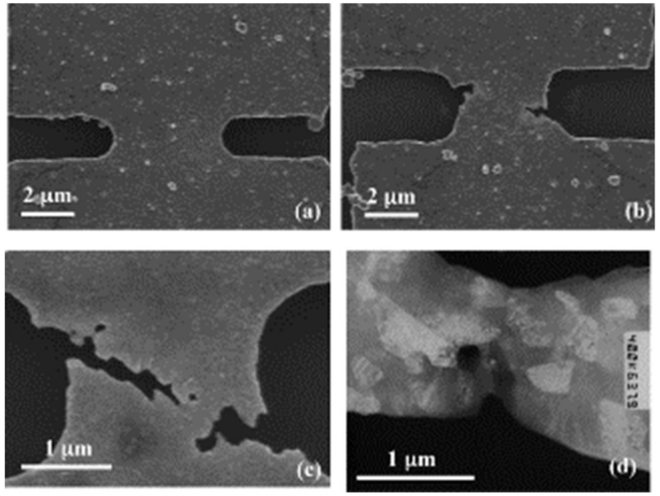


Courtesy of J. Vlassak

The analysis of the fracture of thin films allows unravelling fundamental elementary damage mechanisms – if possible in situ – direct interest for the preferate minerals of Patrick !







Outline

1. Introduction

2. Fracture of thin films on substrates

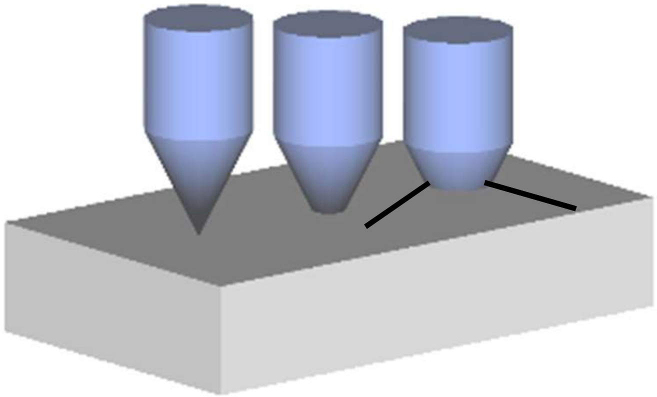
- test methods and extraction of *G*
- example 1 : CrN on polymer (indentation)
- example 2 : Au on polymer (for flexible electronics)

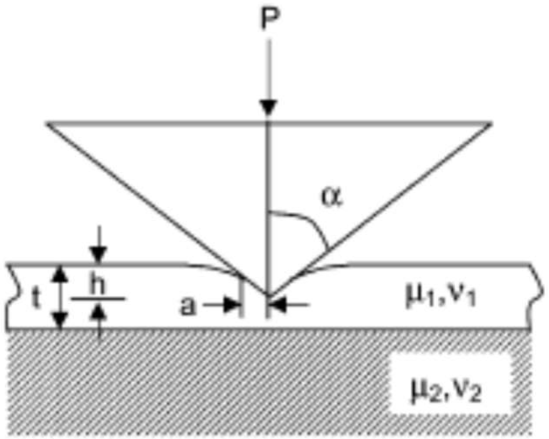
3. Fracture of freestanding films

- test methods for measuring the fracture strength & strain
- fracture strength of brittle films (case of PolySi)
- fracture strain of ductile films (case of Al)
- fracture toughness

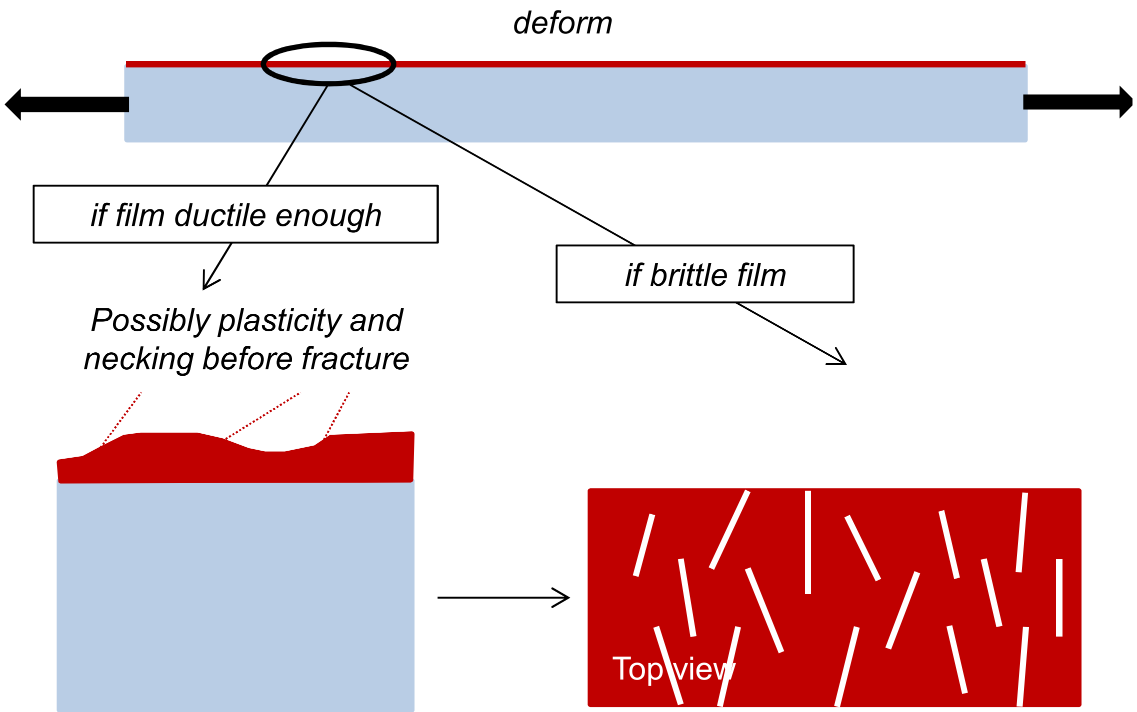
Approach 1 : Thin films on substrate

Nanoindentation

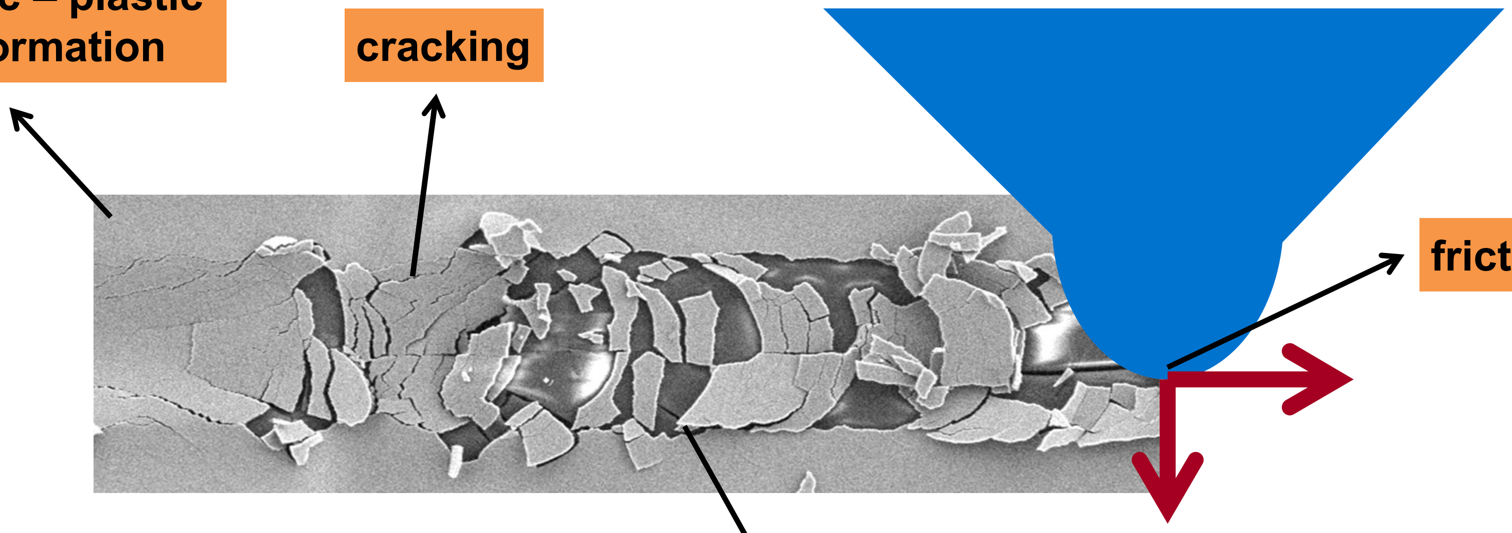




Tensile testing on elastomer



scratch



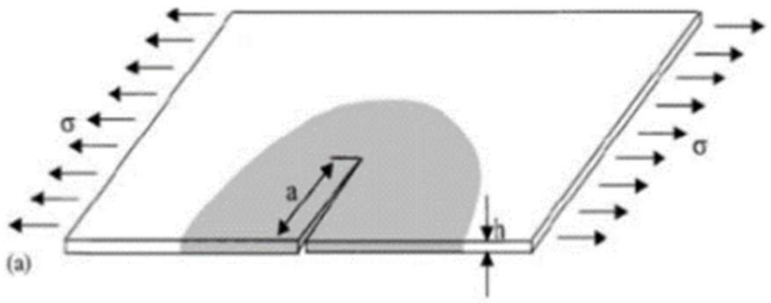
e.g. Favache *et al.* Wear 2015, TSF 2014

and many others: thermal loading, bending, etc

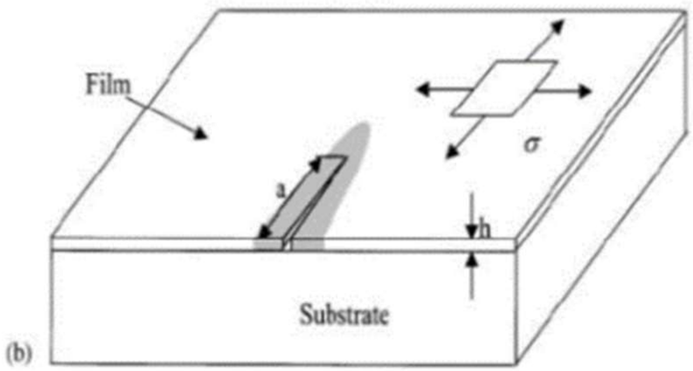
Basic expression of energy release rate for thin film (on substrate) fracture and delamination

Holding the plates at the loading grips fixed (du=0)

$$G = \frac{\partial W_{extForces}}{\partial A} - \frac{\partial W_e}{\partial A} = -\frac{\partial W_e}{\partial A}$$



$$\begin{cases} \Delta W_e = -Z\left(\begin{matrix}\alpha,\beta,\text{geometry,}\\ \text{loadingpatern}\end{matrix}\right)\dfrac{\sigma^2}{2E}a^2 h \\ G = -\dfrac{\partial W_e}{\partial A} = -\dfrac{1}{h}\dfrac{\Delta W_e}{\Delta a} = Z\dfrac{\sigma^2}{E}a \end{cases}$$



$$\begin{cases} \Delta W_e = -Z\left(\begin{matrix}\alpha,\beta,\text{geometry,}\\ \text{loadingpatern}\end{matrix}\right)\dfrac{\sigma^2}{2E}ah^2 \\ G = -\dfrac{\partial W_e}{\partial A} = -\dfrac{1}{h}\dfrac{\Delta W_e}{\Delta a} = Z\dfrac{\sigma^2}{E}h \end{cases}$$

G independent of *a* for films on substrate

$$G = Z\left(\begin{matrix}\alpha,\beta,\sigma_{Ys},\text{crack path, geometry}\\ \text{plasticity, viscoelasticity}\end{matrix}\right)\frac{\sigma_R^2 h}{E_f}$$

Z. Suo, in *Encyclopedia of Comprehensive Structural Integrity*, 2006

General relationship for thin film (on substrate) fracture and delamination under tensile loading

$$G = Z\left(\alpha, \beta, \text{crack path, geometry}\right)\frac{\sigma_R^2 h}{E}$$

Z here for no elastic mismatch and infinitely thick substrate (+ remember, G_c also depends on α and β through ψ)

Surface Crack

Z = 3.951

Channeling

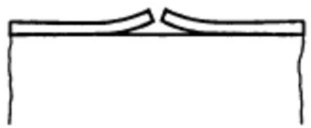
Z = 1.976

Substrate Damage

Z = 3.951

Spalling

Z = 0.343



Debond

$$Z = \begin{cases} 1.028 \text{ (initiation)} \\ 0.5 \text{ (steady - state)} \end{cases}$$

Hutchinson & Suo, Adv Appl Mech 1992

Example 1 : cracking resistance of CrN films on polymer

(as representative of many hard brittle coatings on softer substrates)

System of interest

Thin Solid Films 550 (2014) 464–471

Contents lists available at ScienceDirect

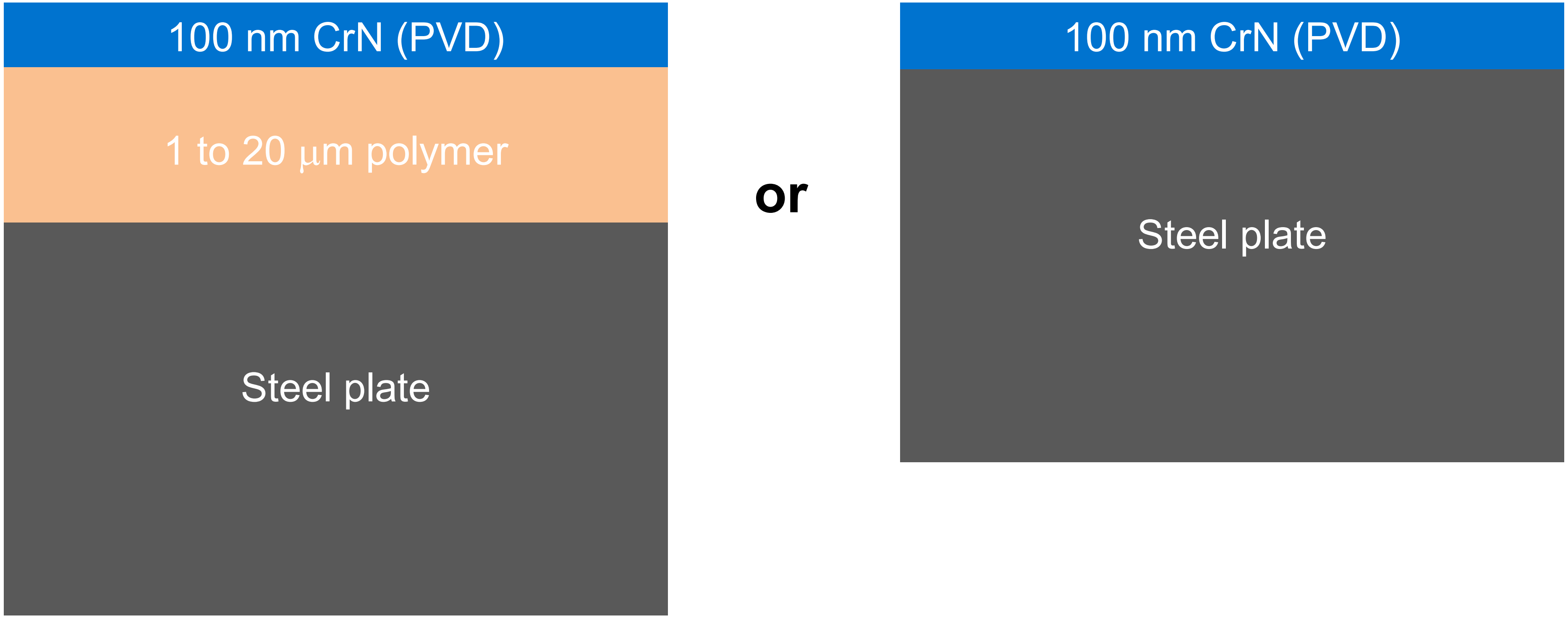
Thin Solid Films

journal homepage: www.elsevier.com/locate/tsf

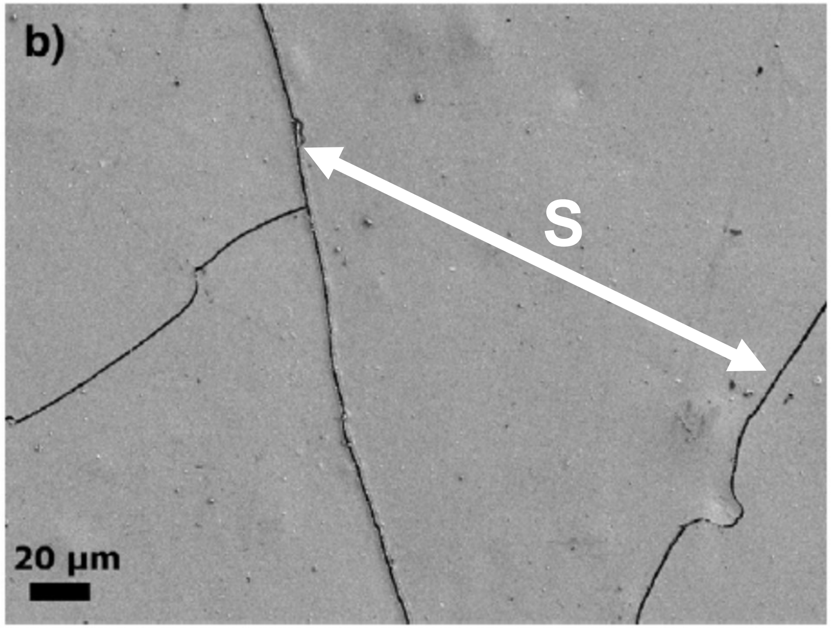
ELSEVIER

Fracture toughness measurement of ultra-thin hard films deposited on a polymer interlayer

Audrey Favache [a,*], Laure Libralesso [b], Pascal J. Jacques [a], Jean-Pierre Raskin [c], Christian Bailly [d], Bernard Nysten [d], Thomas Pardoen [a]



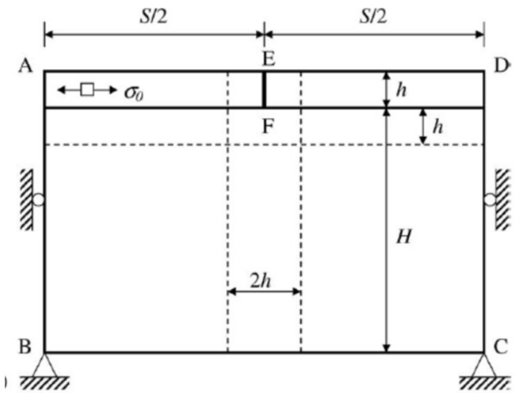
Observation of channel cracks upon deposition

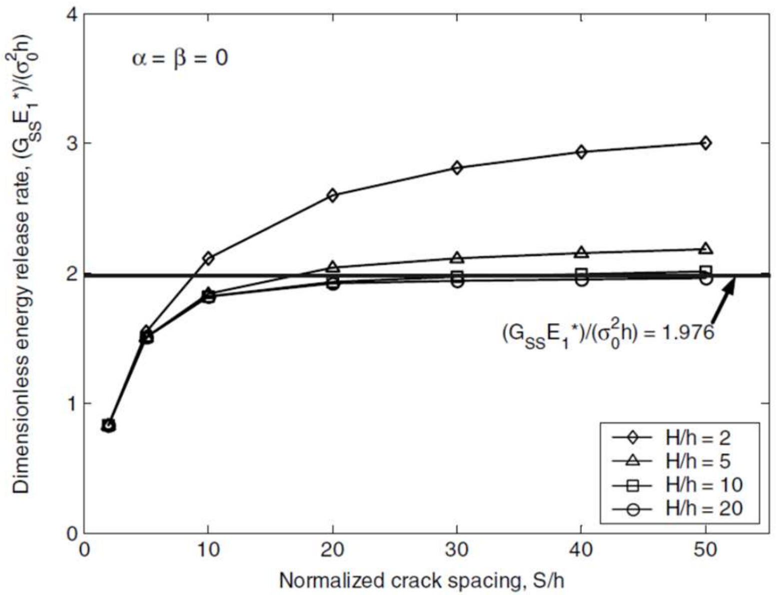


$$G = Z\left(\begin{array}{l}\alpha_{(polymer)}, \beta_{(polymer)}, \\ \text{channel crack spacing}\end{array}\right)\frac{\sigma_R^2 h}{E_f}$$

$$\alpha = \frac{E_f^* - E_s^*}{E_f^* + E_s^*} \text{ and } \beta = \frac{\mu_f(1-2\nu_s) - \mu_s(1-2\nu_f)}{2\mu_f(1-\nu_s) + 2\mu_s(1-\nu_f)}$$

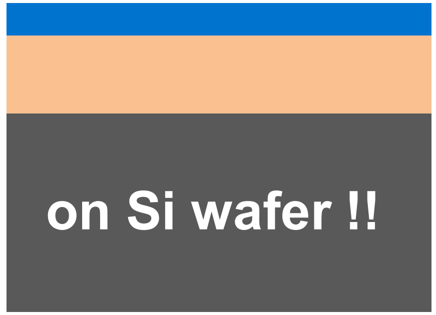
weak effect for channel cracks if $\alpha > 0$

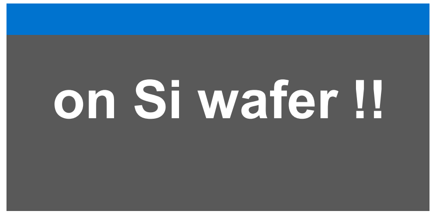


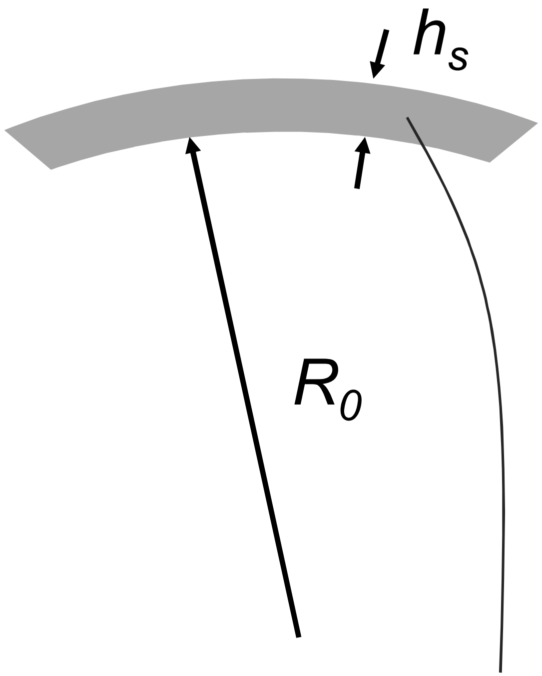


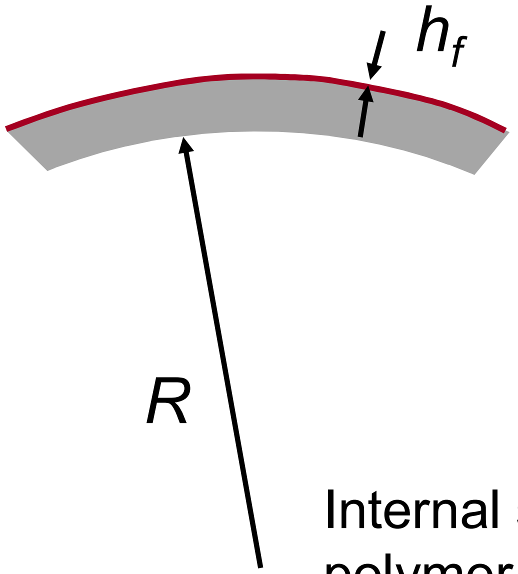
Huang et al., EFM 2003

Extraction of internal stress with Stoney method









$$\sigma_R \approx -\frac{1}{6}\frac{E_s}{(1-\nu_s)}\frac{h_s^{2}}{h_f}\left(\frac{1}{R}-\frac{1}{R_0}\right)$$

Internal stress slightly varies with polymer interlayer :
from 830 to 930 MPa

(we take the one from a system showing no cracking – CrN on steel : **910 MPa**)

Observation of channel cracks upon deposition

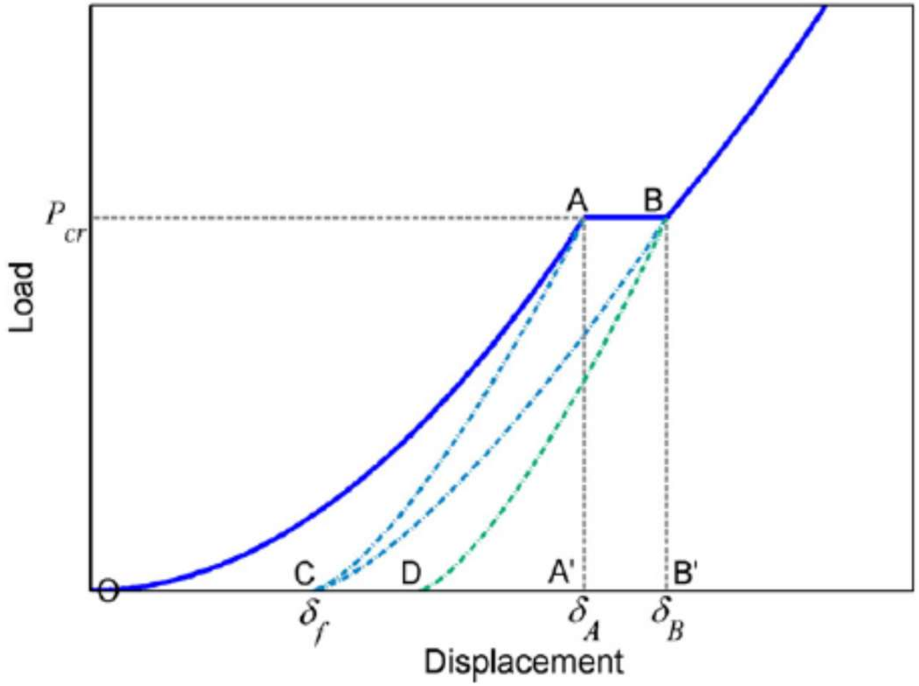
$$G = Z\left(\alpha_{polymer}, \beta_{polymer}, channelcrack\right)\frac{\sigma_R^2 h}{E_f}$$

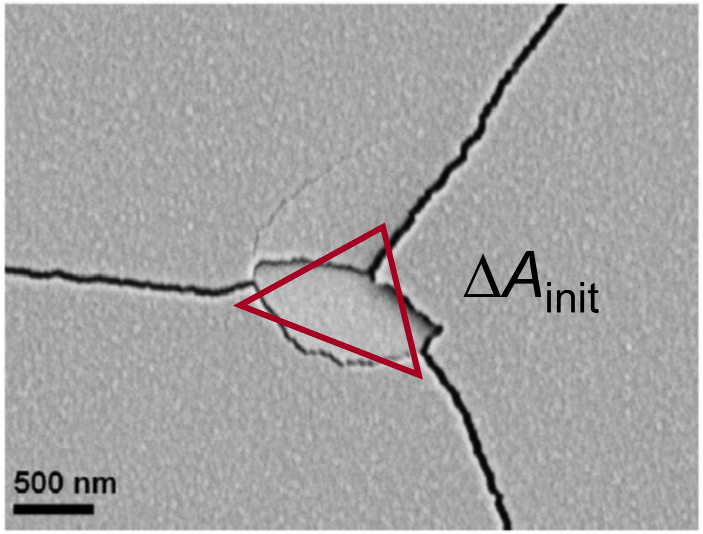
Crack propagation energy release rate calculated from initial cracking. For cracked samples $G = G_{Ic}$ (in italic). The 95% confidence interval given in brackets is calculated from the error on the internal stress and on the substrate modulus.

Sample	α	Crack spacing S [μm]	Z	G [J/m^2]
CrN–steel	0.01	Uncracked	2.0	0.7 [0.6, 0.8]
CrN–Si	0.14	Uncracked	2.2	0.8 [0.7, 0.9]
CrN–P1–steel	0.95	48 ± 10	14	*4.9 [4.4, 6.5]*
CrN–P1–Si	0.95	60 ± 15	14	*4.9 [4.4, 6.5]*
CrN–P2–steel	0.99	100 ± 20	39	*13.2 [11.8, 14.6]*
CrN–P3–steel	0.97	67 ± 10	22	*7.4 [6.6, 8.6]*
CrN–P4–steel	0.98	56 ± 10	29	*9.7 [8.5, 11.2]*
CrN–PI–Si	0.98	18000	28	*9.7 [8.7, 10.8]*

Note : polymer interlayer favours cracking !

Indentation based cracking (more complex than for bulk !)





Chen and Bull, TSF 2009

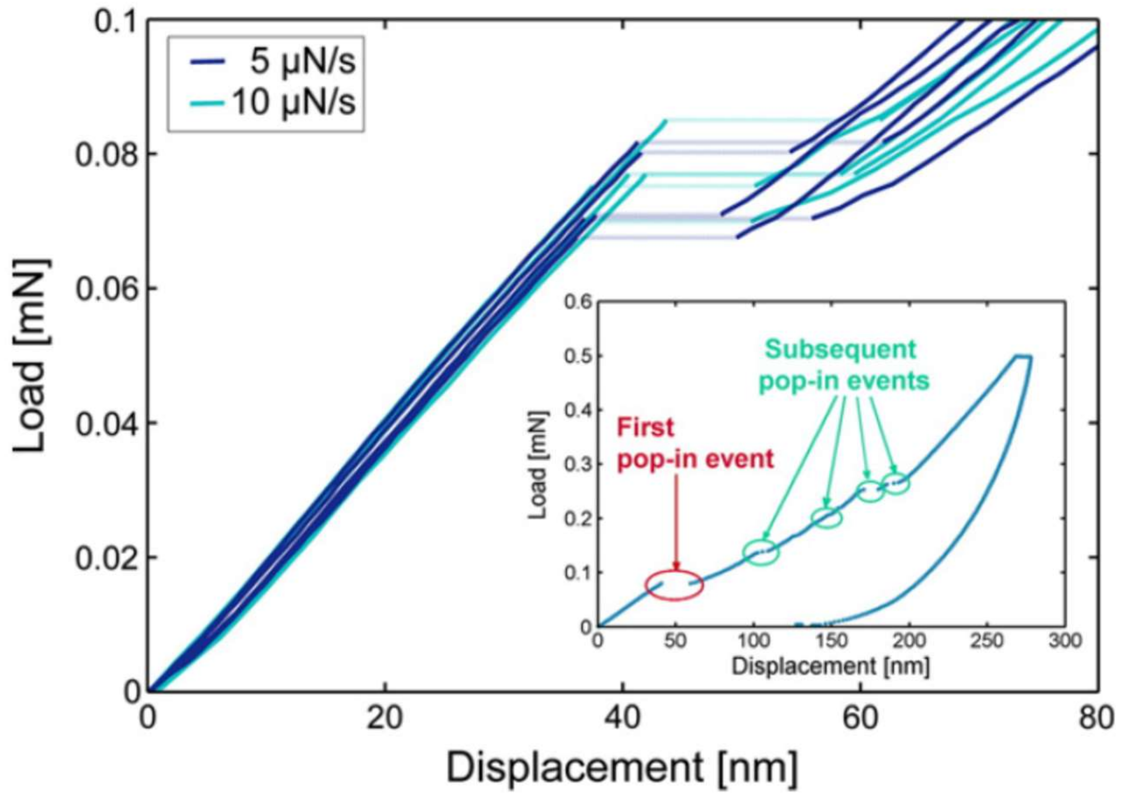
Lower bound Upper bound

$$U_1 = \int_{\delta_f}^{\delta_A} P_{cr} \left(\frac{x - \delta_f}{\delta_A - \delta_f} \right)^m dx + P_{cr}(\delta_B - \delta_A) \text{ and } U_2 = \int_{\delta_f}^{\delta_B} P_{cr} \left(\frac{x - \delta_f}{\delta_B - \delta_f} \right)^n dx.$$

$$G = \Delta U / \Delta A$$

Note : cracking observed only with polymer interlayer !

Indentation based cracking



Sample	α	Crack spacing S [μm]	Z	G [J/m^2]	G (J/m^2) from indent
CrN–steel	0.01	Uncracked	2.0	0.7 [0.6, 0.8]	
CrN–Si	0.14	Uncracked	2.2	0.8 [0.7, 0.9]	
CrN–P1–steel	0.95	48 ± 10	14	*4.9 [4.4, 6.5]*	
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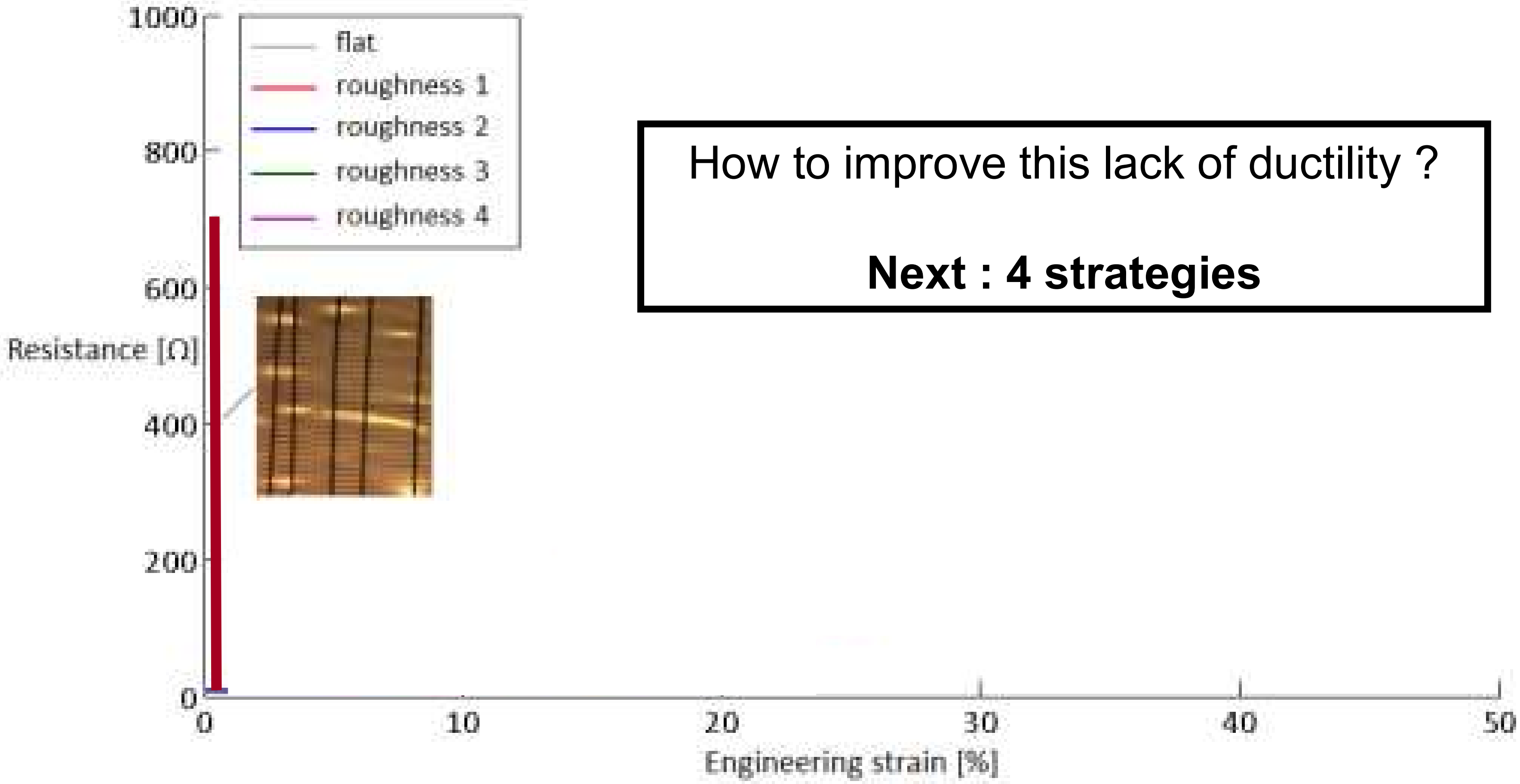
CrN–P2–steel	0.99	100 ± 20	39	*13.2 [11.8, 14.6]*	
CrN–P3–steel	0.97	67 ± 10	22	*7.4 [6.6, 8.6]*	7.1 ± 5.7
CrN–P4–steel	0.98	56 ± 10	29	*9.7 [8.5, 11.2]*	
CrN–PI–Si	0.98	18000	28	*9.7 [8.7, 10.8]*	14.7 ± 10

Example 2 : cracking resistance of Au films on polymer

(as representative of metal on polymer flexible electronics type devices)

Thin Au films are not ductile (fracture strain below 1 or 2 %)

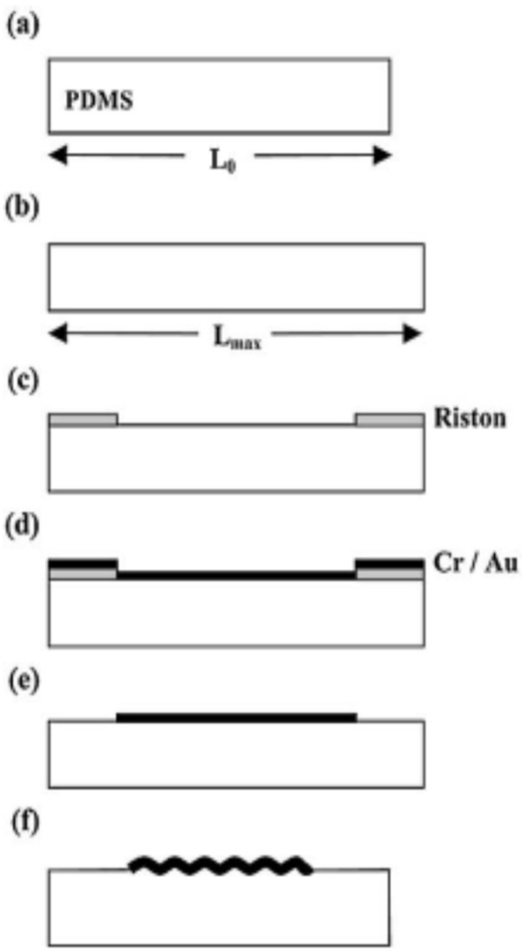


How to improve this lack of ductility ?

Next : 4 strategies

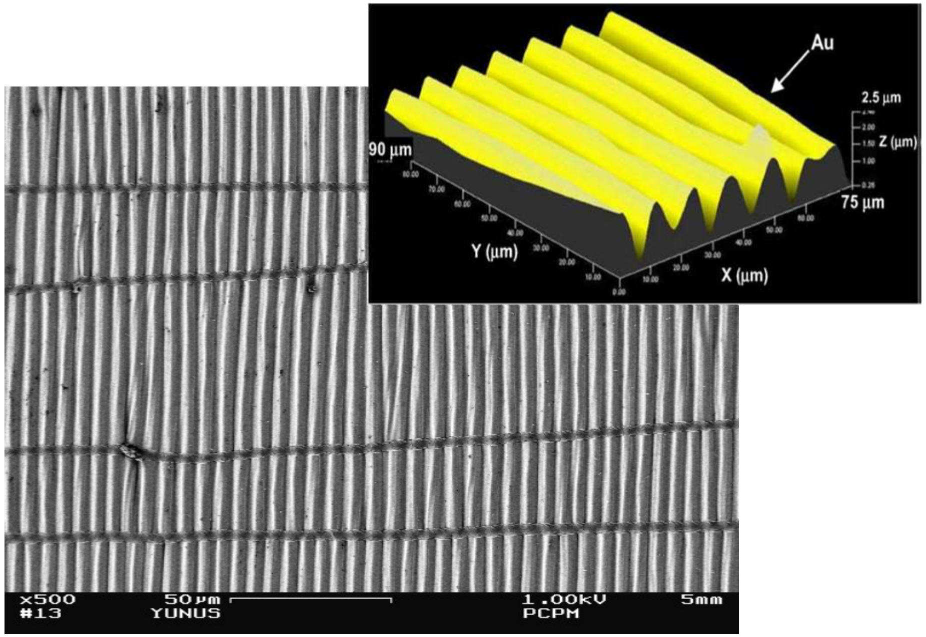
First ductilization principle : wrinkling patterns

Basic concept

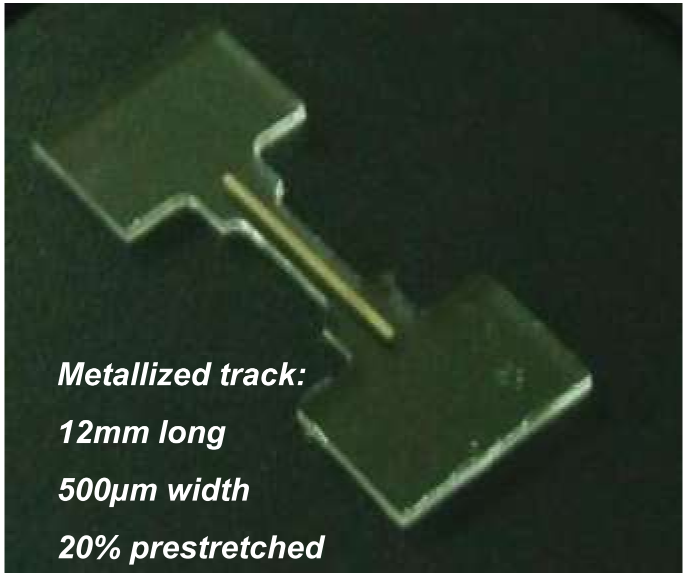


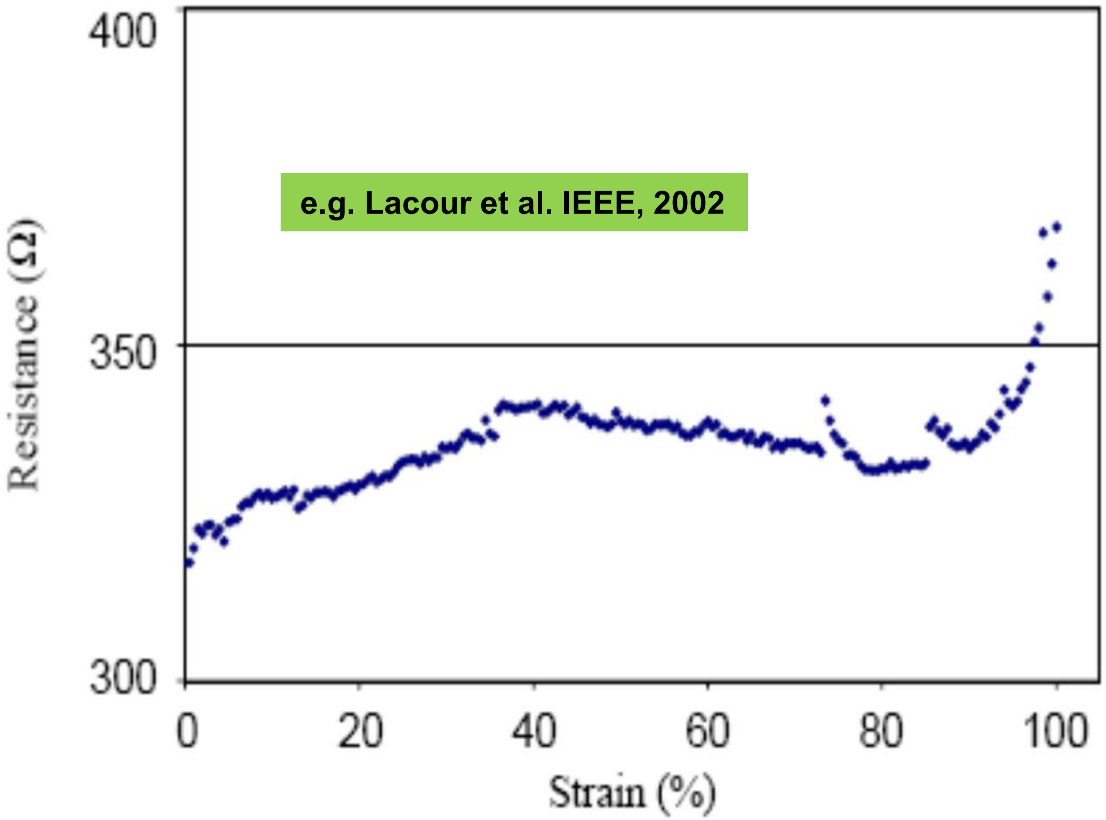
5 to 30% of prestretch
5 nm of Cr adhesion layer
100 nm gold evaporated
Upon release, wavelet morphology

e.g. Lacour et al. IEEE, 2002



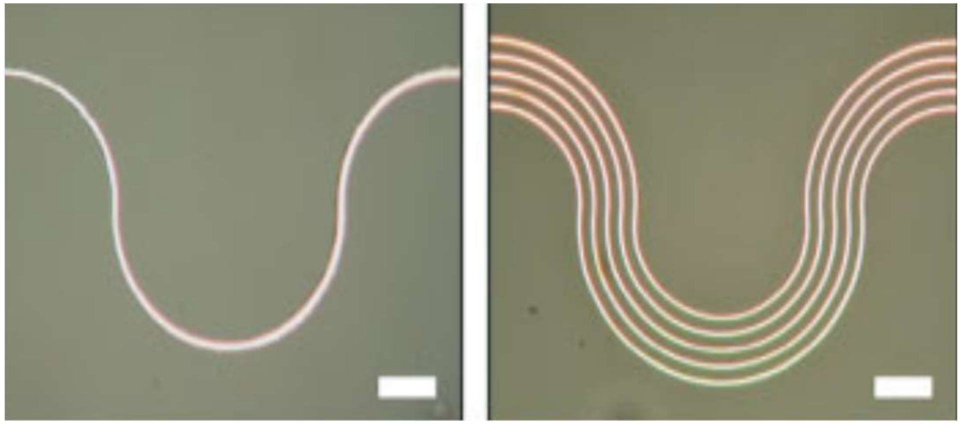
High stretchability without loss of electrical conductivity under monotonous and cyclic loadings





Second ductilization principles : 2D in plane or 3D out of plane structures

2D Serpentine pattern

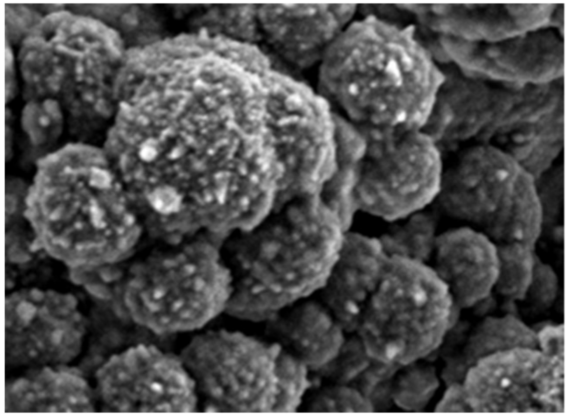


Low Scale

No adhesion

Expensive technology

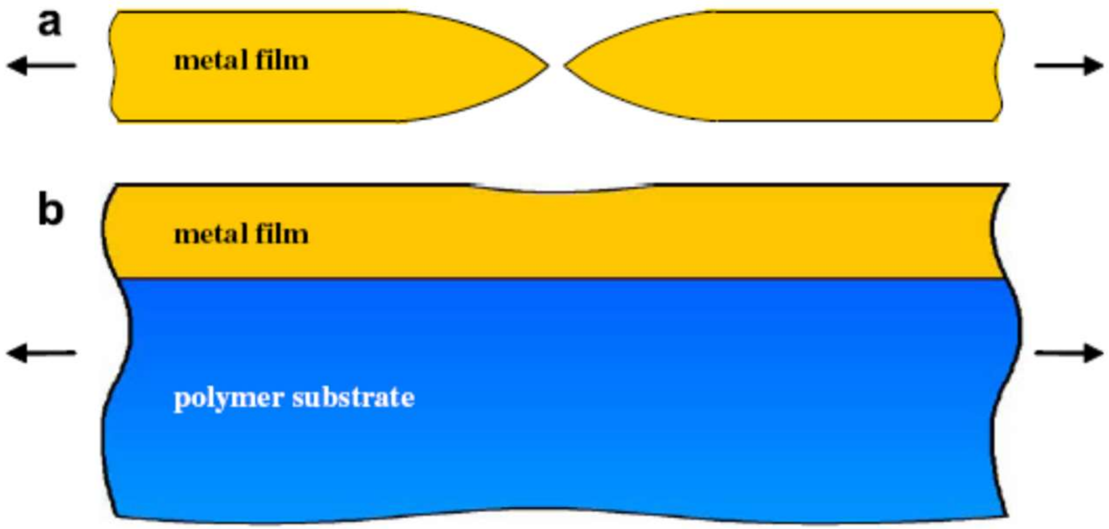
3D structure of mushrooms

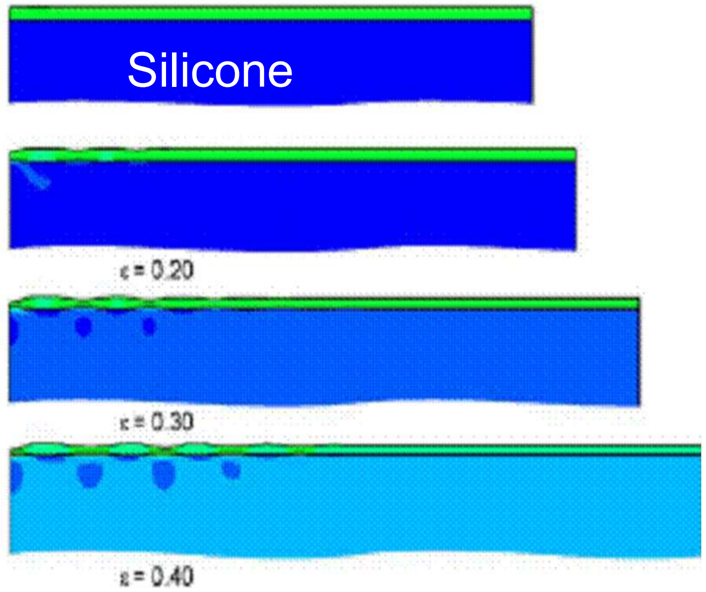


Time consuming process

++ Very low resistivity
+ Contact and integration
- Elasticity

Third ductilization principle : retard or multiply necking





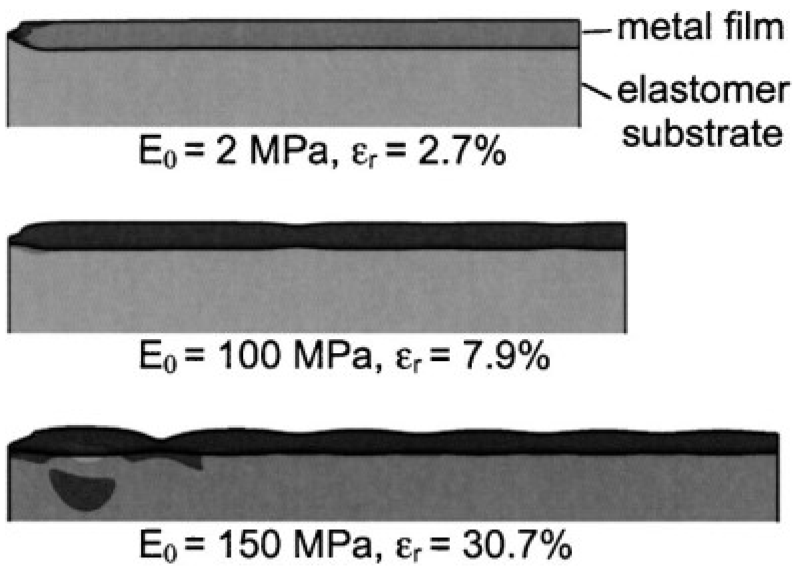
From Suo's group

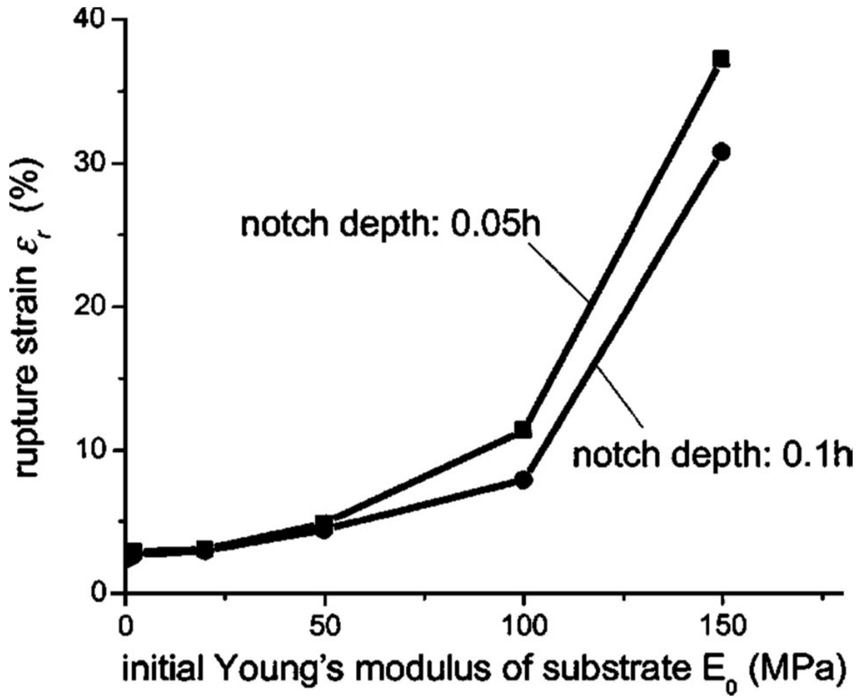
Li et al., Mech Mater 2005

Li & Suo, IJSS 2006

This requires playing with materials characteristics, e.g. strain hardening capacity and rate dependency (see next section of freestanding films)

Third ductilization principle : retard or multiply necking





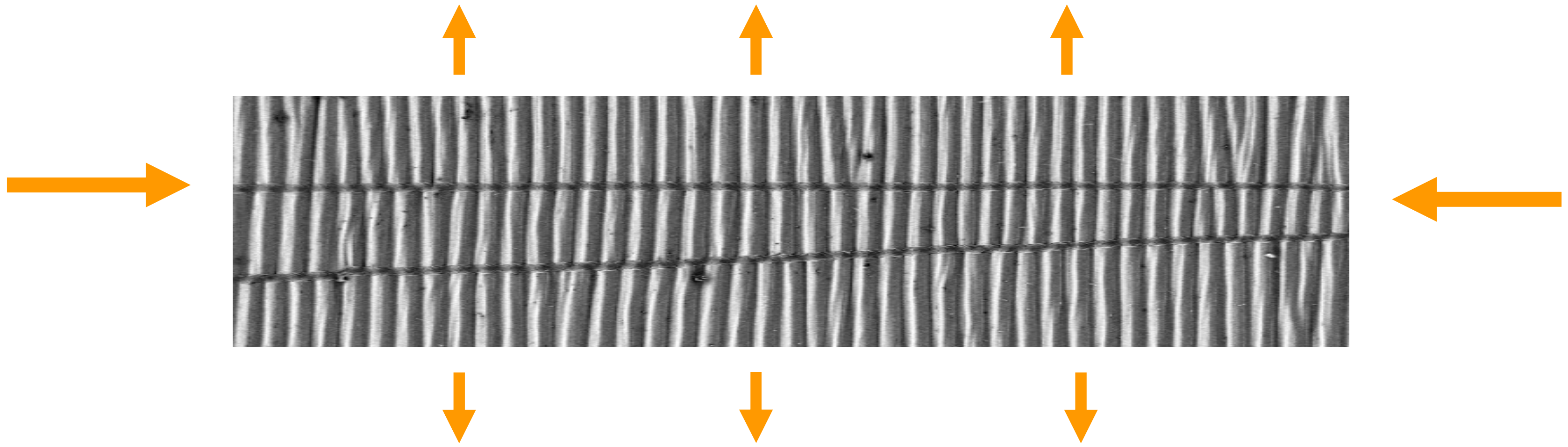
The delocalization process optimisation depends also on stiffness mismatch

Li et al., APL 2004

Li & Suo, IJSS 2006

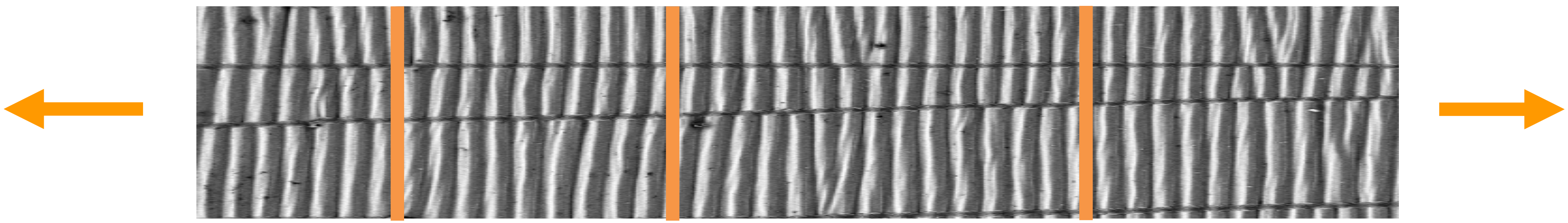
Fourth ductilization principle : favour non percolating crack path

Starting point – why longitudinal cracks ?
Large tensile stresses build up in the film in the transverse direction due to the transverse extension upon unloading after deposition



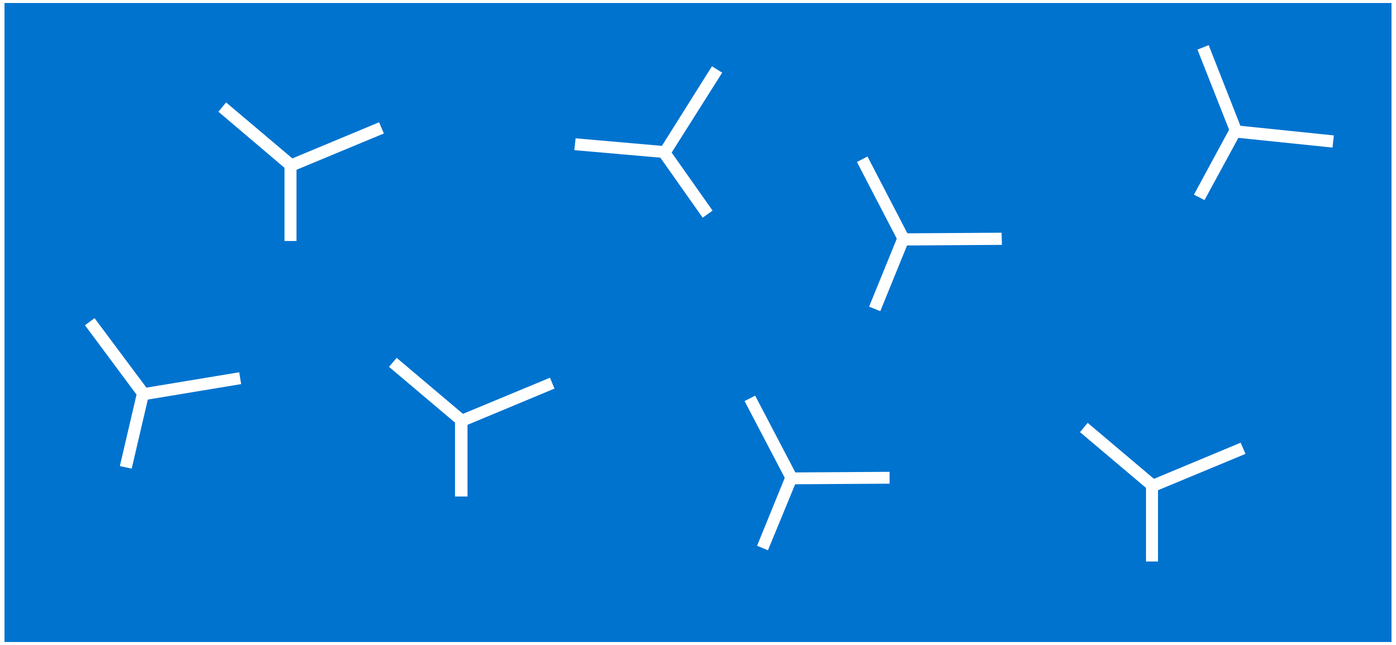
Fourth ductilization principle : favour non percolating crack path

If pulling next in longitudinal direction, wrinkles flatten and, then, long transverse cracks develop (depending on film fracture strain and possible – delayed – necking) interrupting electrical conduction



Fourth ductilization principle : favour non percolating crack path

How to avoid long percolating cracks ?
One example : tri-branched pre-cracks



Example 1 of combination of strategies : *Stretchable helical gold conductor*

APPLIED PHYSICS LETTERS **91**, 141911 (2007)

Stretchable helical gold conductor on silicone rubber microwire

S. Béfahy,[a] S. Yunus, T. Pardoen, and P. Bertrand
MAPR, Université catholique de Louvain, Croix du Sud 1, 1348 Louvain-la-Neuve, Belgium

M. Troosters
Neurotech SA, Chemin du Cyclotron 6, 1348 Louvain-la-Neuve, Belgium

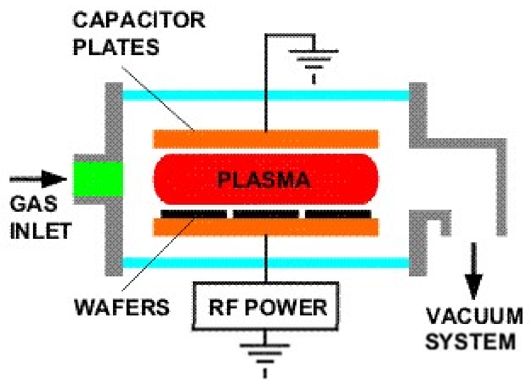
Befahy *et al.*, APL 2007

Process

Ph. D. of S. Befahy at UCL, 2006

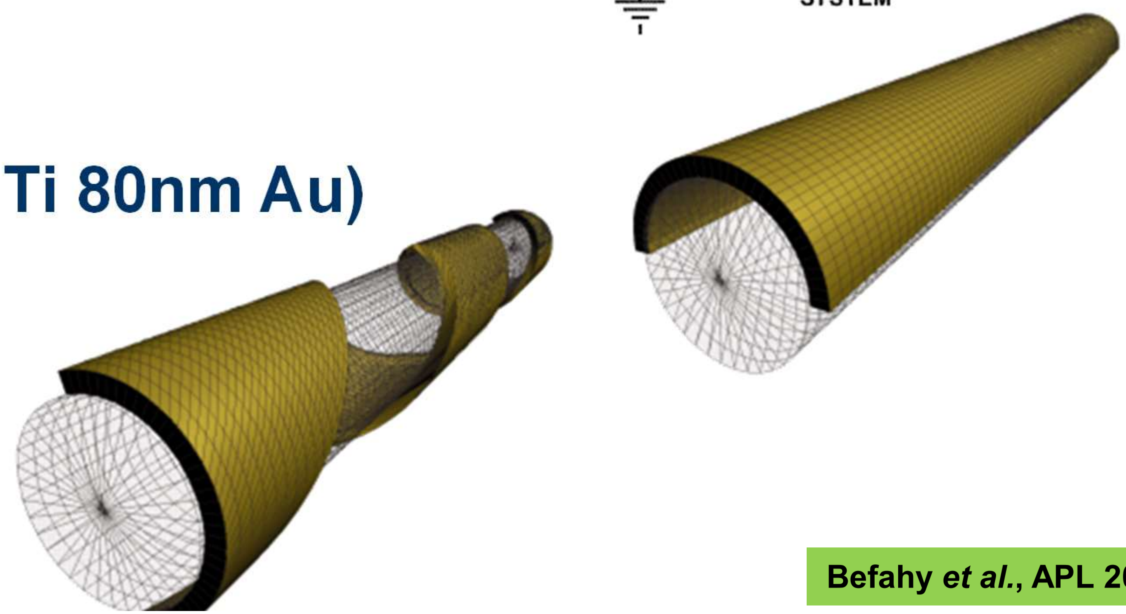
1. Pre-stretch and pre-twist a silicone wire to favor wrinkles and

2. Oxygen RF cold plasma on prestrained wires



3. Metallization (5nm Ti 80nm Au)

4. Release



Befahy *et al.*, APL 2007

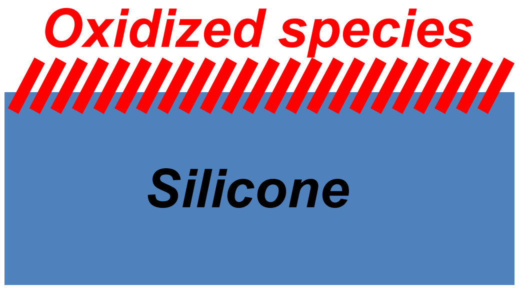
Details of Step 2 of process : oxygen RF cold plasma on prestrained wires

to avoid delamination improve adhesion of PDMS

Link with lecture 1

- Challenges
  - Presence of free siloxanes
  - Low surface energy (21-22 mJ/m2)

- Solutions
  - Solvent extraction
  - Surface activation (oxidation)
    - Low pressure plasma
    - UV (atmospheric pressure)
    - Ozone (atmospheric pressure)

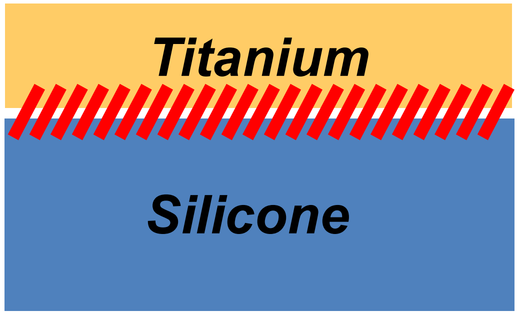


Ph. D. of S. Befahy at UCL, 2006

Details of Step 2 of process : oxygen RF cold plasma on prestrained wires

Link with lecture 1

- Challenges
  - Presence of free siloxanes
  - Low surface energy (21-22 mJ/m2)

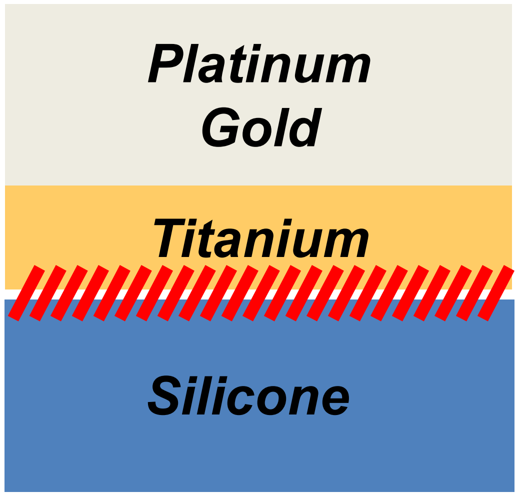


- Solutions
  - Solvent extraction
  - Surface activation (oxidation)
    - Low pressure plasma
    - UV (atmospheric pressure)
    - Ozone (atmospheric pressure)
  - Titanium or Chromium intermediate thin layer (~5nm)

Ph. D. of S. Befahy at UCL, 2006

Details of Step 3 of process : deposition

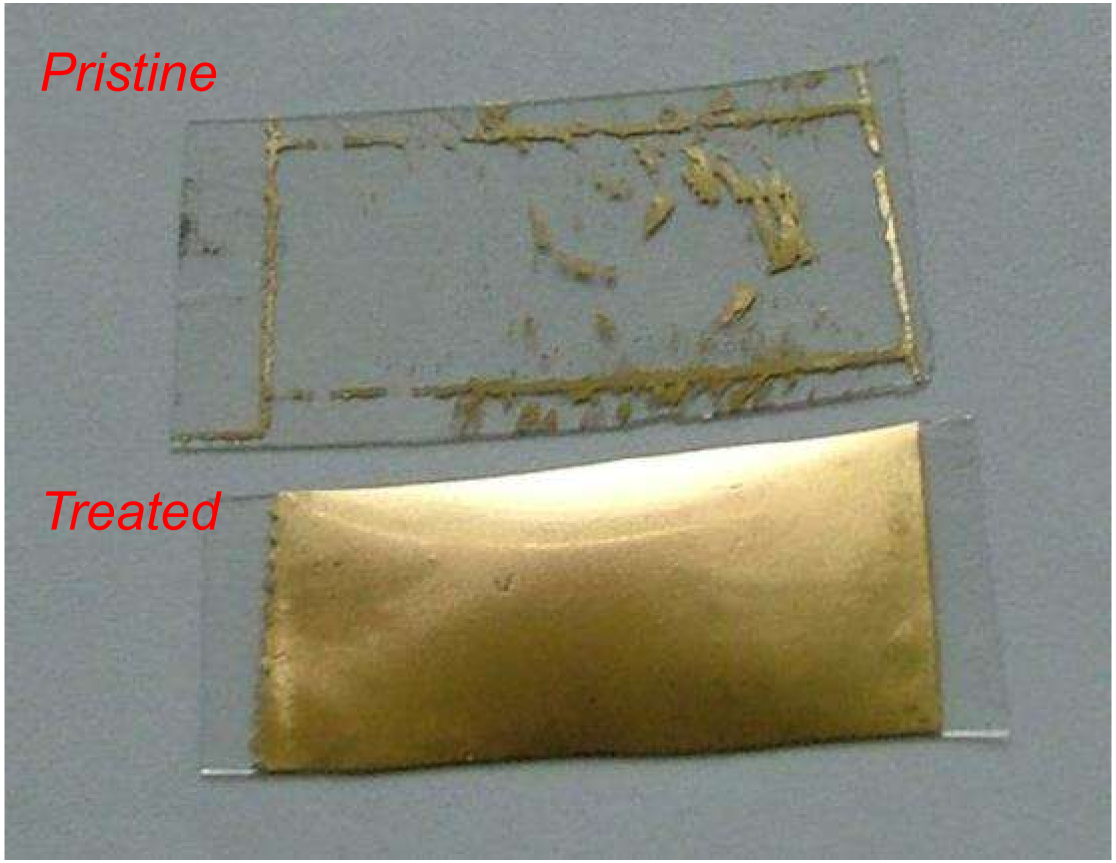
- Metallization by *Physical Vapor Deposition*
- ~5nm of titanium
- ~100nm of platinum or gold



Ph. D. of S. Befahy at UCL, 2006

Good adhesion !

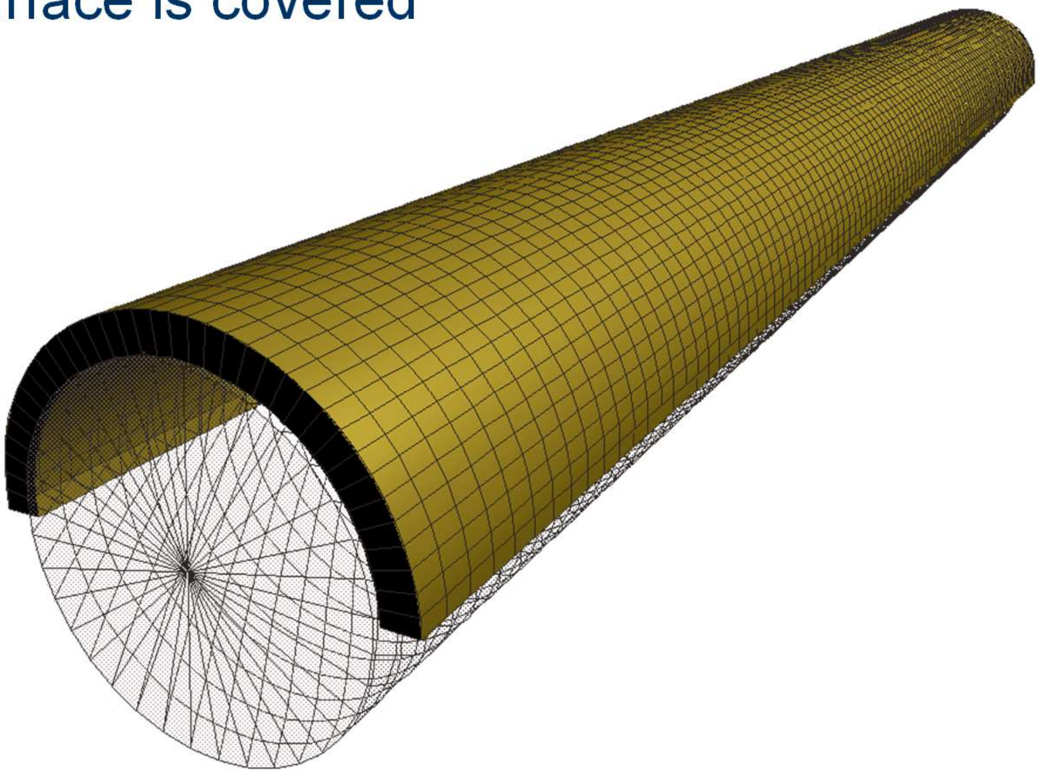
Peel Scotch test



Ph. D. of S. Befahy at UCL, 2006

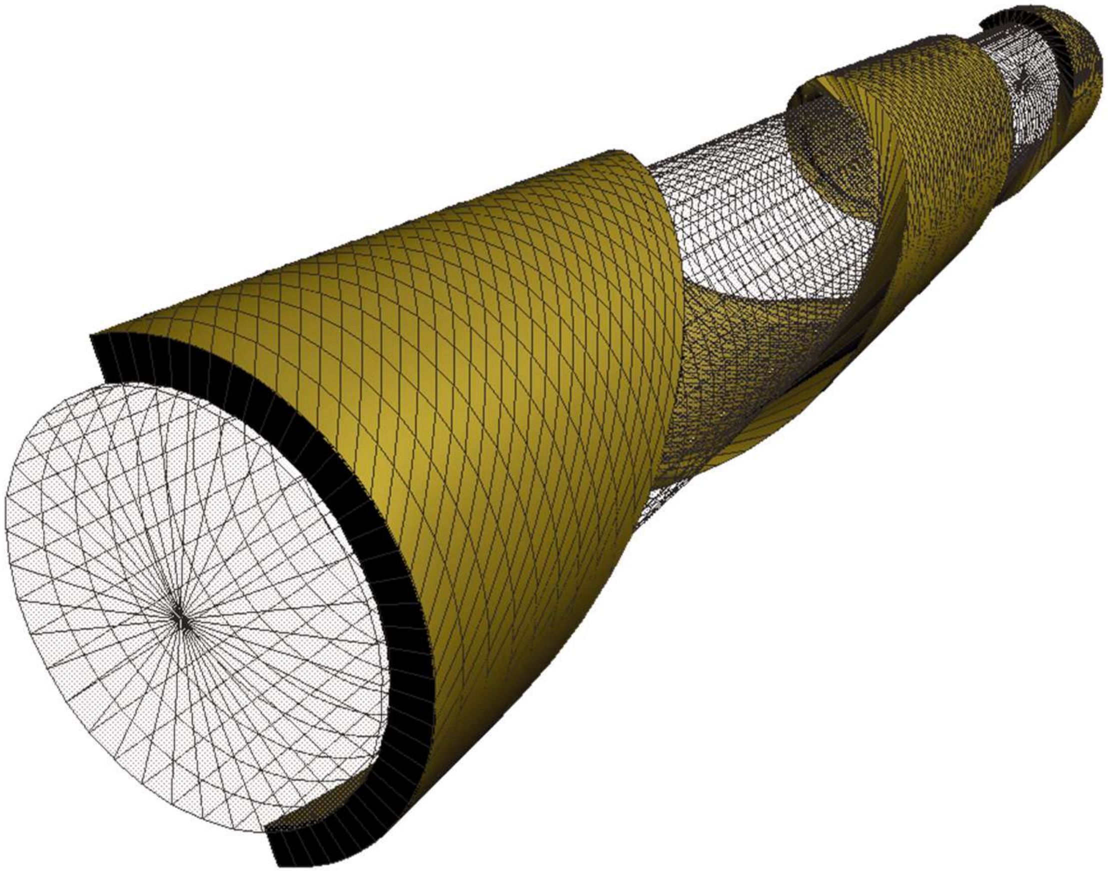
Details of Step 3 of process : deposition

- 5nm Ti and 80nm Au
- Half the surface is covered



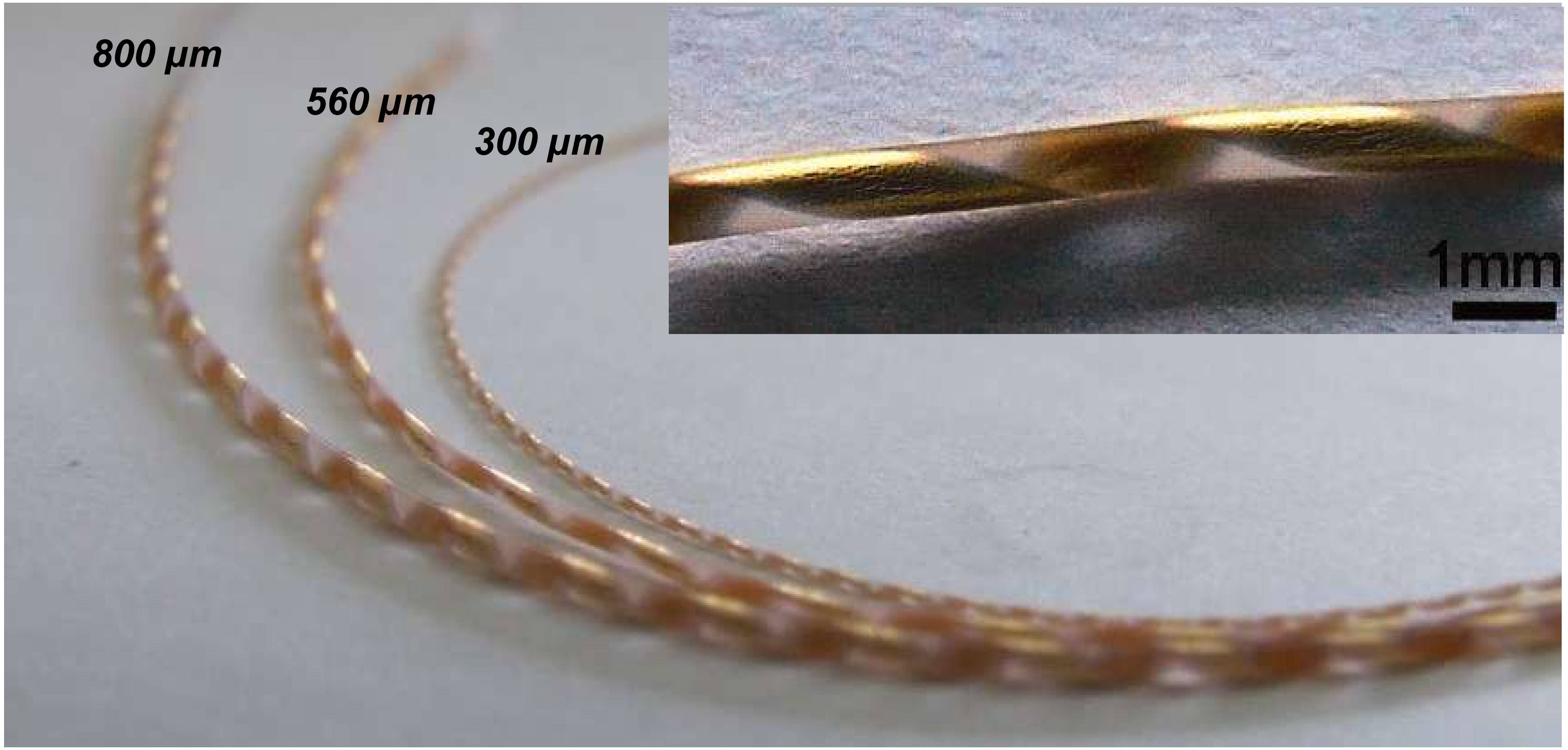
Ph. D. of S. Befahy at UCL, 2006

Details of Step 4 of process : release



Ph. D. of S. Befahy at UCL, 2006

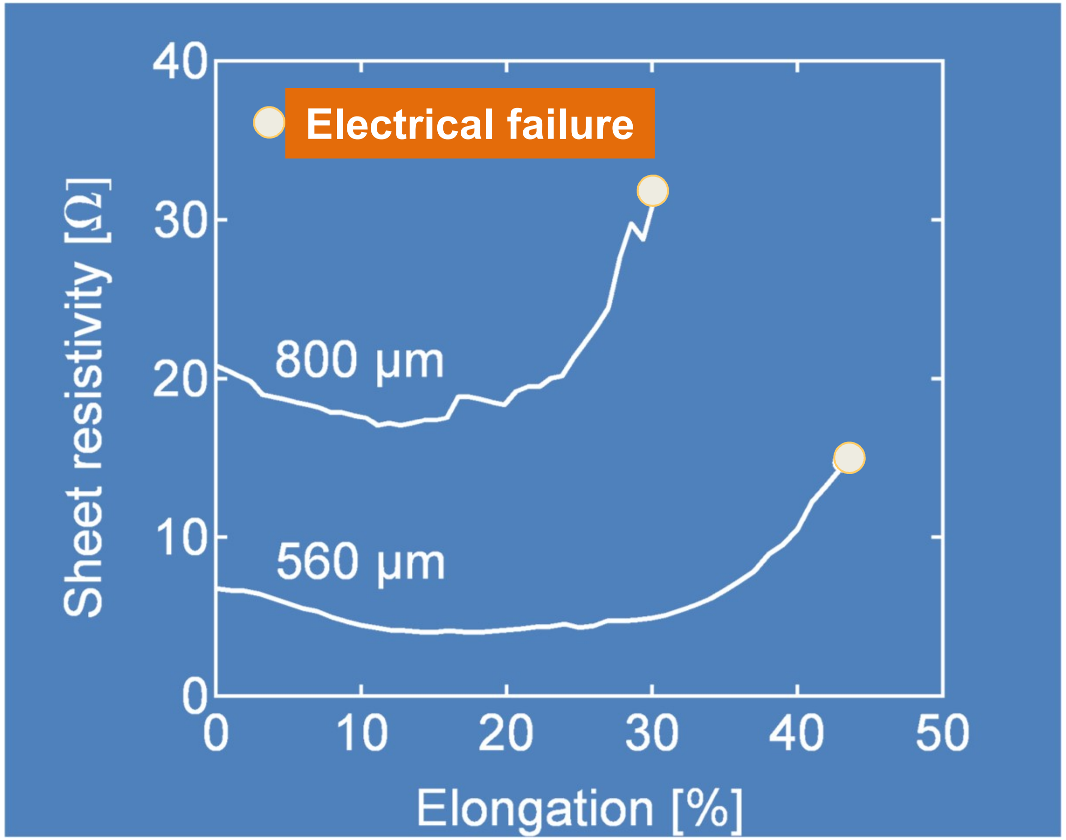
In real



Patent PCT/EP2007/053159

Ph. D. of S. Befahy at UCL, 2006

Performances of the wires



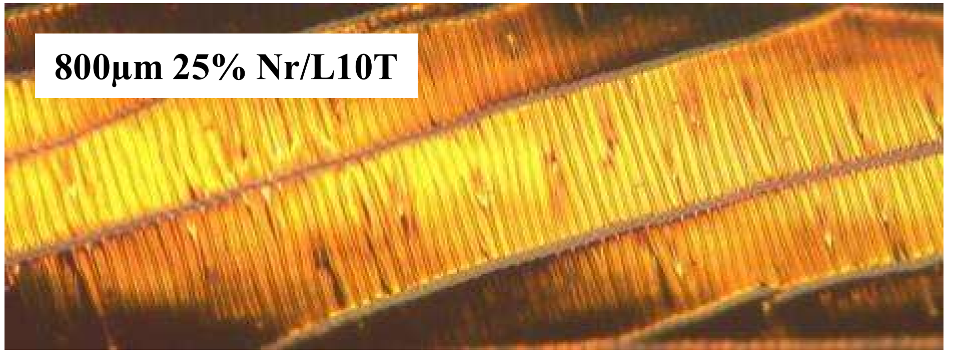
9mm long
20 full rotations
25% of stretch
Two different diameters

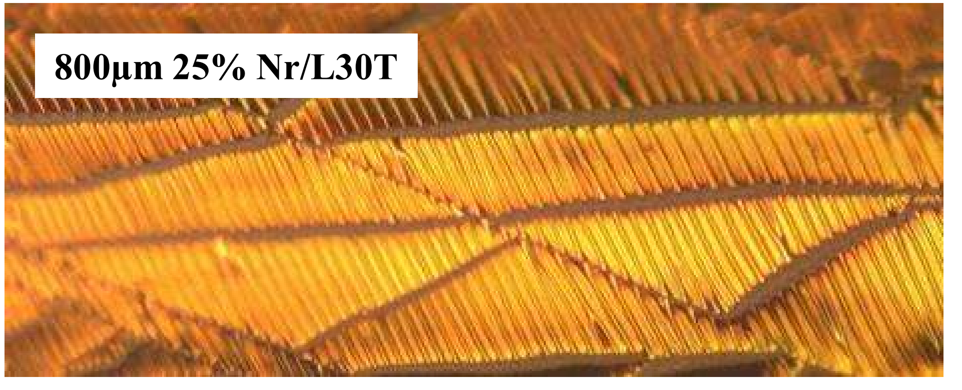
- 800µm diameter more stretchable
- at least 30% stretchability
- a minimum in the evolution of the resistance
- No sharp increase in resistance

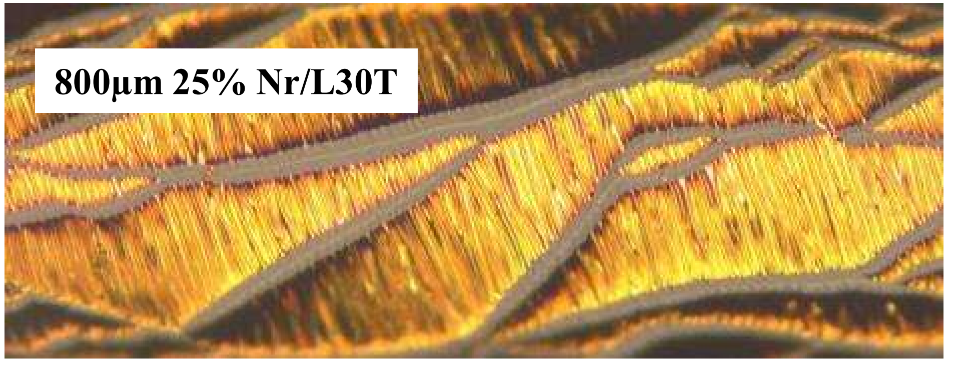
Befahy *et al.*, APL 2007

Ph. D. of S. Befahy at UCL, 2006

Quantitative characterization of cracking pattern

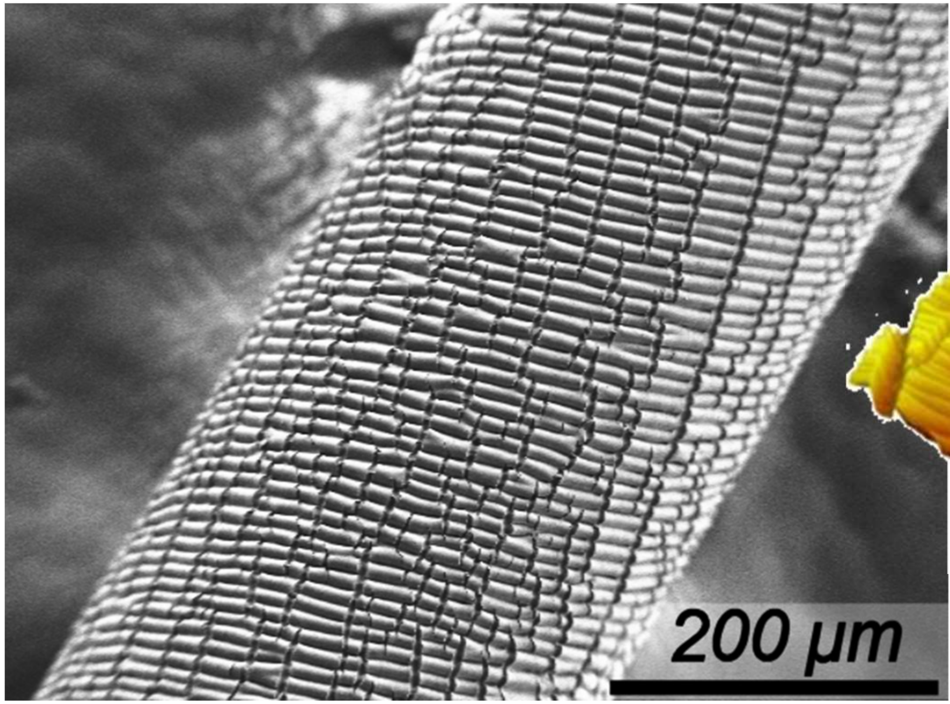


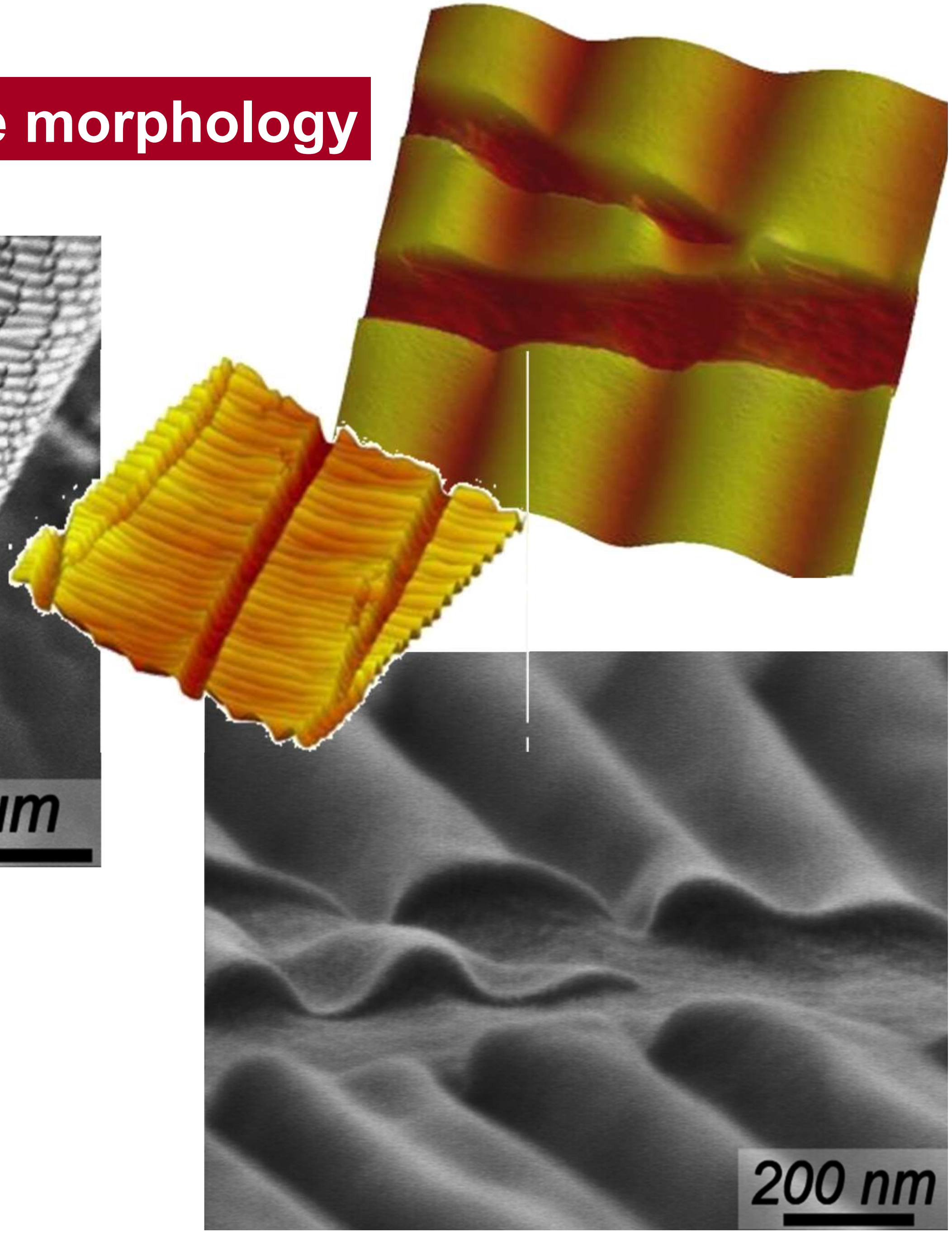




Befahy *et al.*, APL 2007

Surface morphology



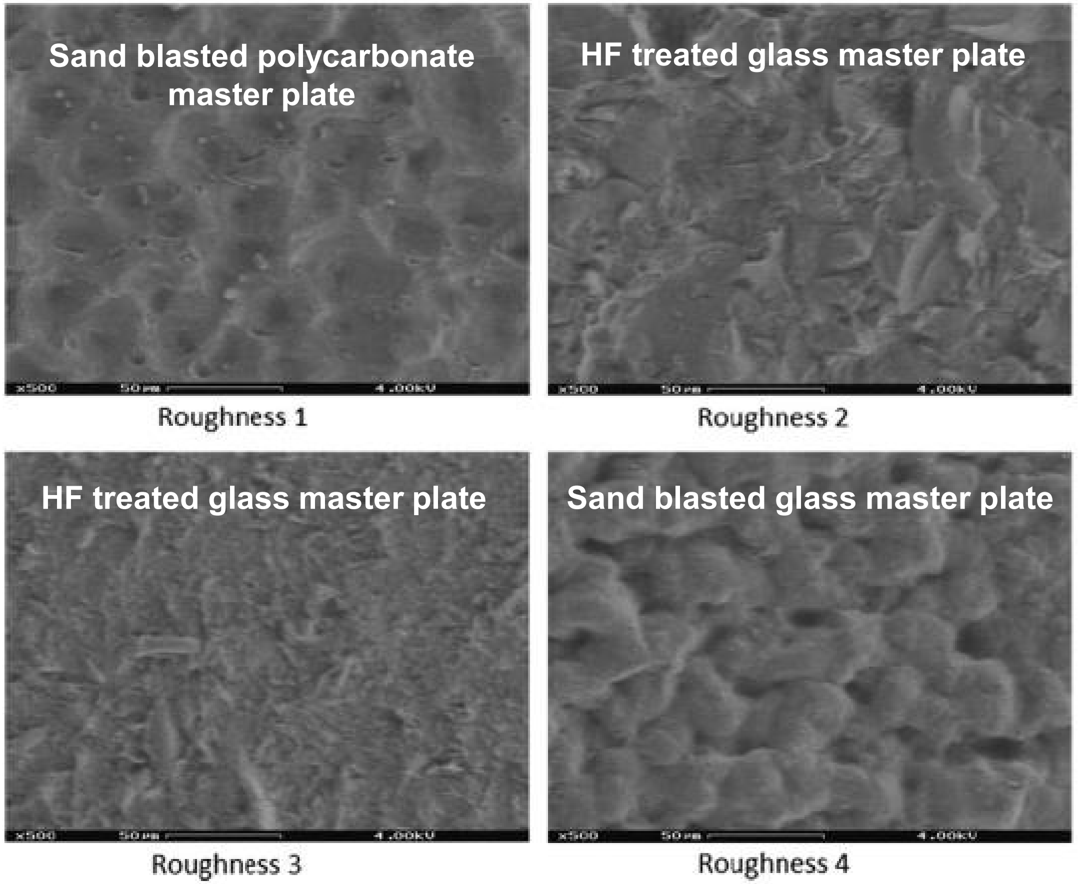


Adhesion OK
Non percolating cracks OK
Wrinkles OK

Befahy *et al.*, APL 2007

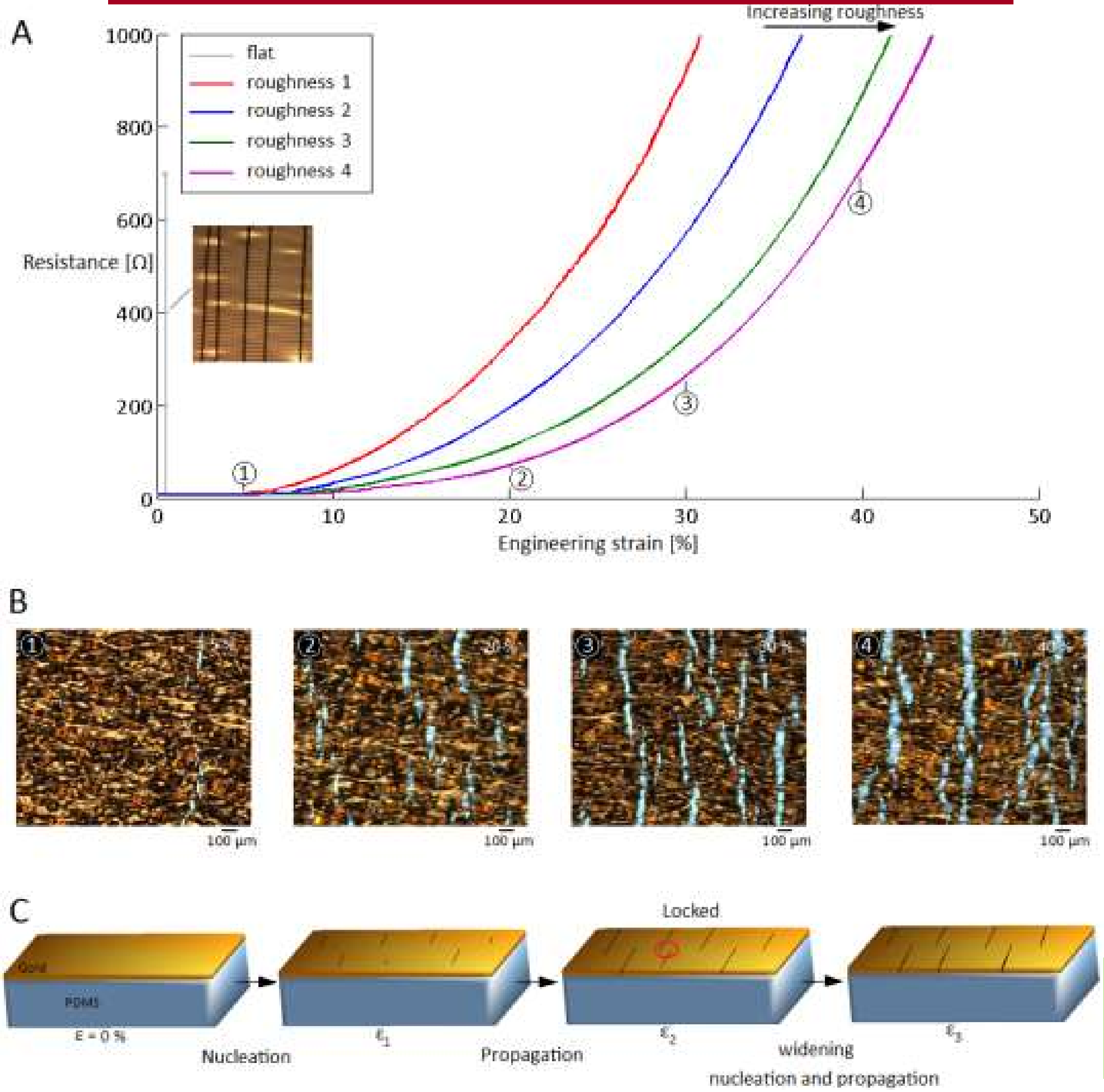
Example of combination of strategies

Idea : play with substrate roughness to randomize crack pattern



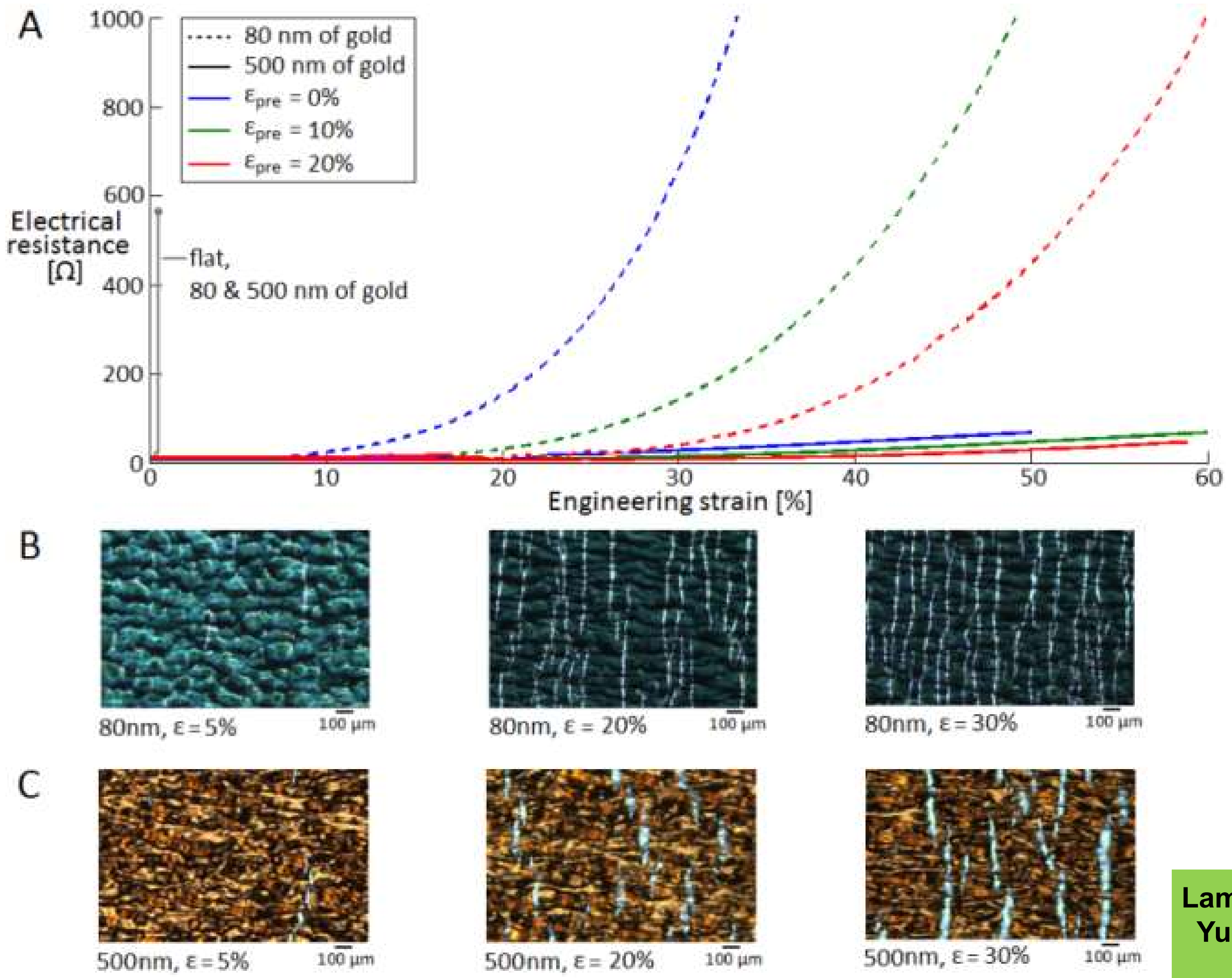
Lambricht, Pardoen, Yunus, Acta Mater 2013

Example of combination of strategies



Lambricht, Pardoen, Yunus, Acta Mater 2013

Example of combination of strategies



Lambricht, Pardoen, Yunus, Acta Mater 2013

Approach 1 : Thin films on substrate Conclusion

Pro and cons

Easy to manipulate at macro level

Adapted to macro testing devices

Closer to a system property – to explore extrinsic effects

Difficulties to deconvolute substrate effects to estimate e.g. hardness or fracture toughness

Difficult to extract stress level

Careful with internal stress

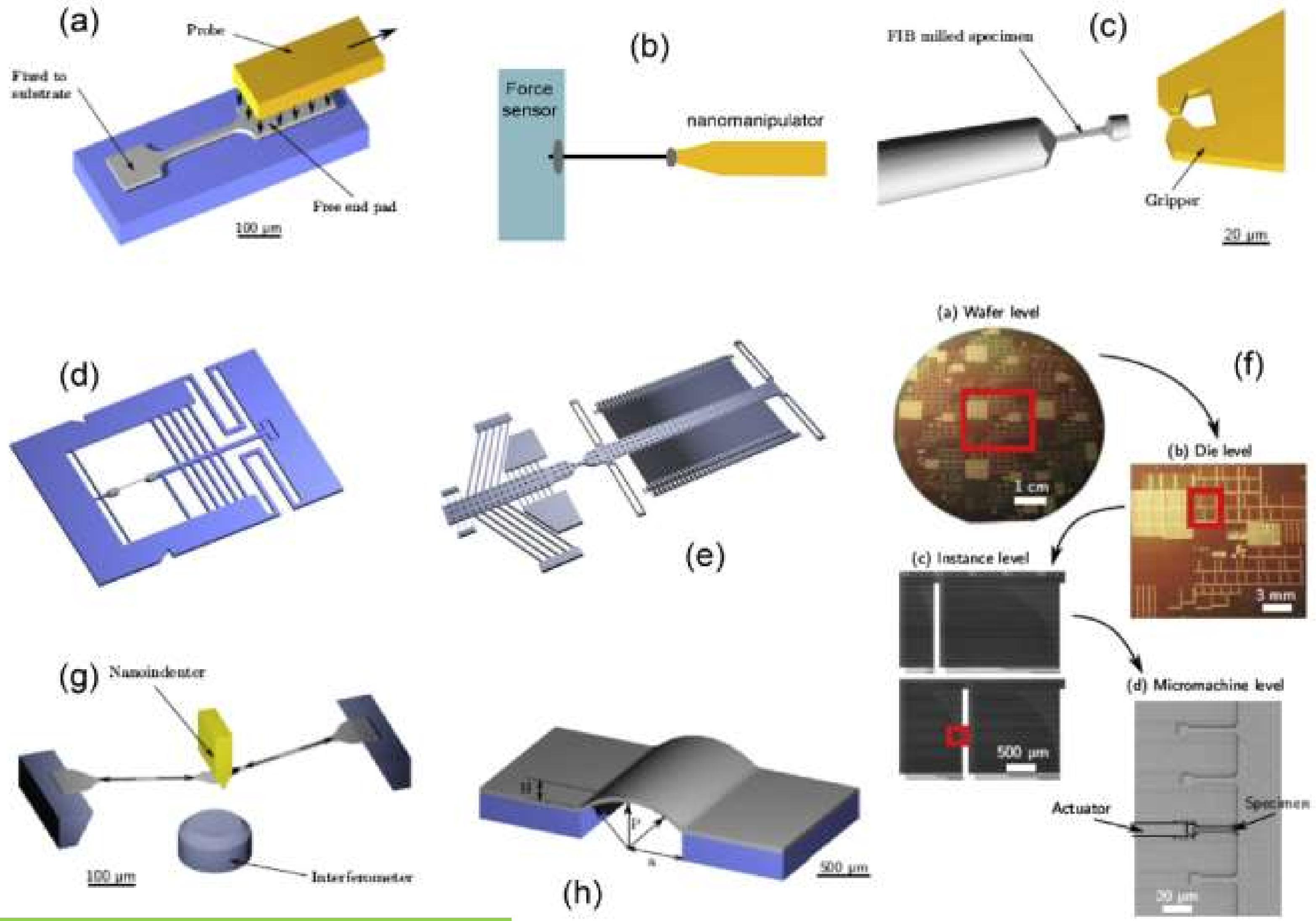
Outline

1. **Introduction**

2. **Fracture of films on substrates**
   - test methods and extraction of *G*
   - example 1 : CrN on polymer (indentation)
   - example 2 : Au on polymer (for flexible electronics)

3. **Fracture of freestanding films**
   - Test methods for measuring the fracture strength - strain
   - fracture strength of brittle films
   - fracture strain of ductile films
   - fracture toughness

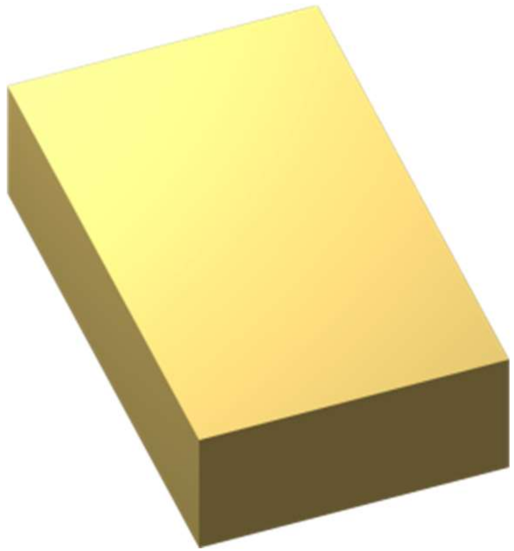
Approach 2 : Mechanical testing of freestanding small scale objects



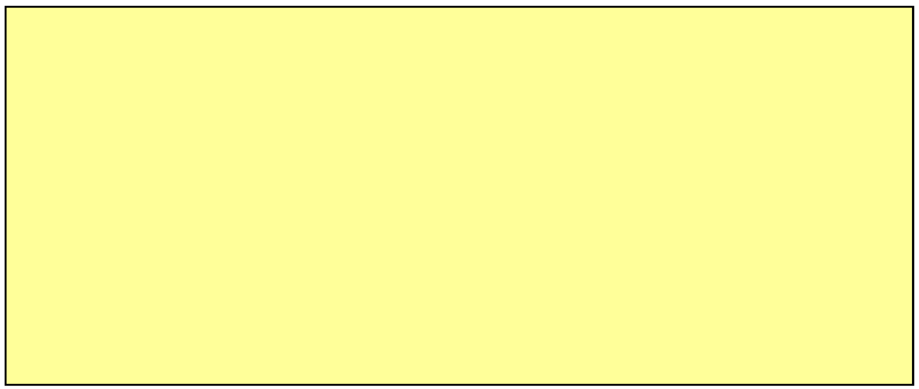
See ref in Pineau, Benzerga, Pardoen, Acta Mater 2016

UCLouvain method : Fabrication of an elementary on chip micro- or nano- test structure

Start with Si wafer



Top view

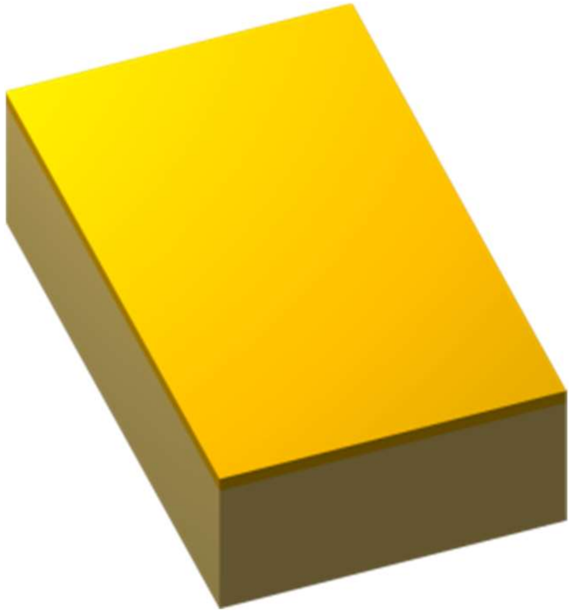


Cross section view

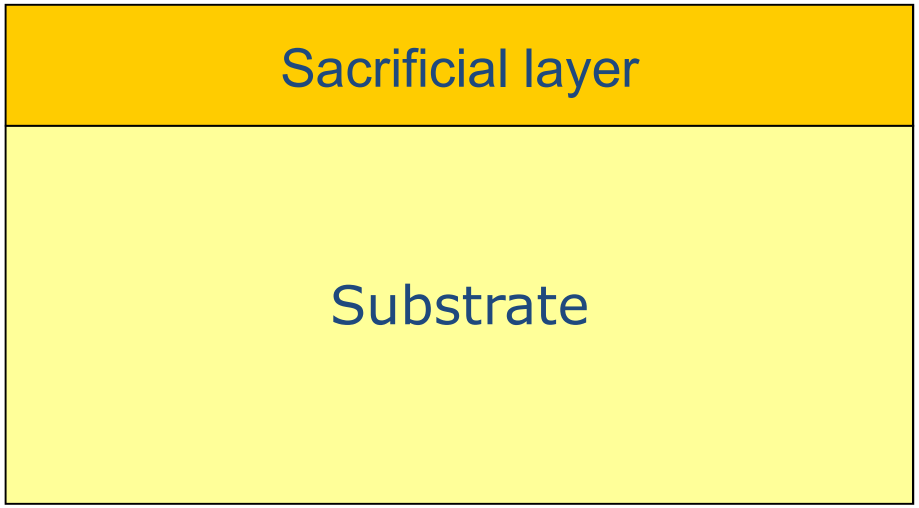
S. Gravier et *al.*, *JMEMS*, vol. 18 (2009) 555

Fabrication steps

Deposition of sacrificial layer
(e.g. SiO_2)



Top view

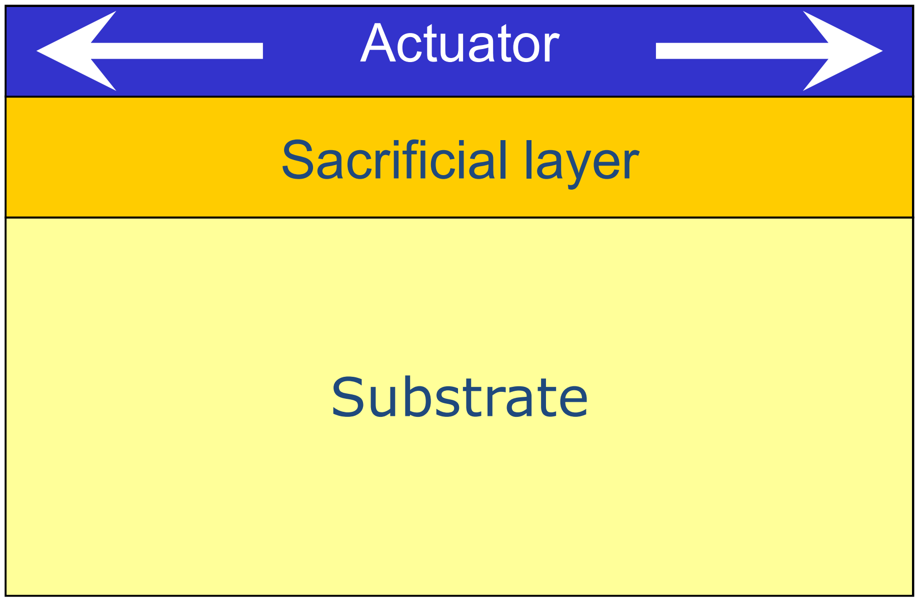


Cross section view

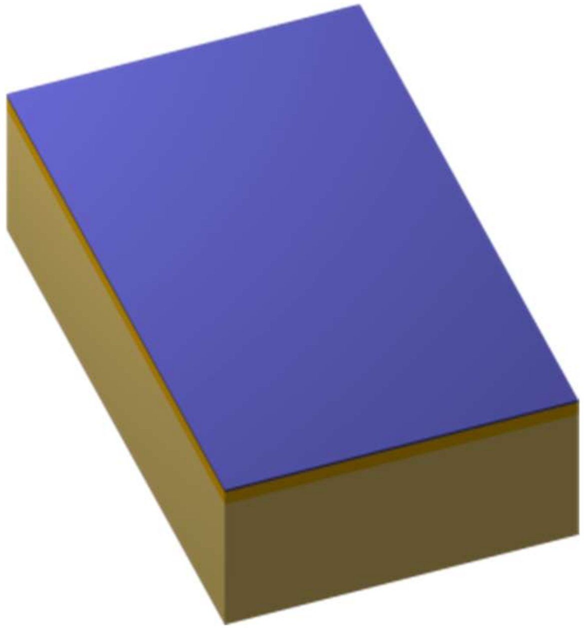
S. Gravier et *al.*, *JMEMS*, vol. 18 (2009) 555

Fabrication steps

Deposition of the actuator layer involving large internal tensile stress
(e.g. Si_3N_4)



Cross section view



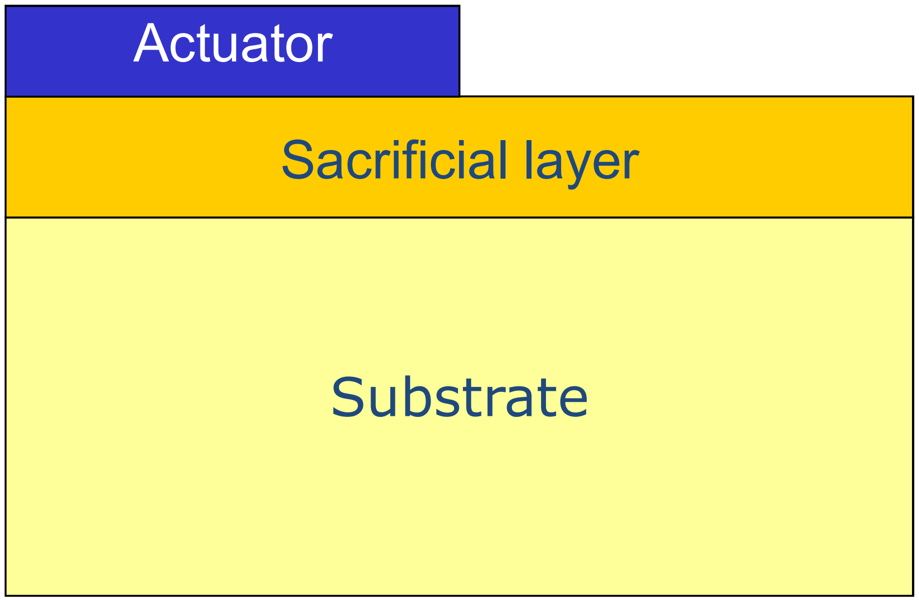
Top view

Stoney method
to measure $\sigma^{internal}$

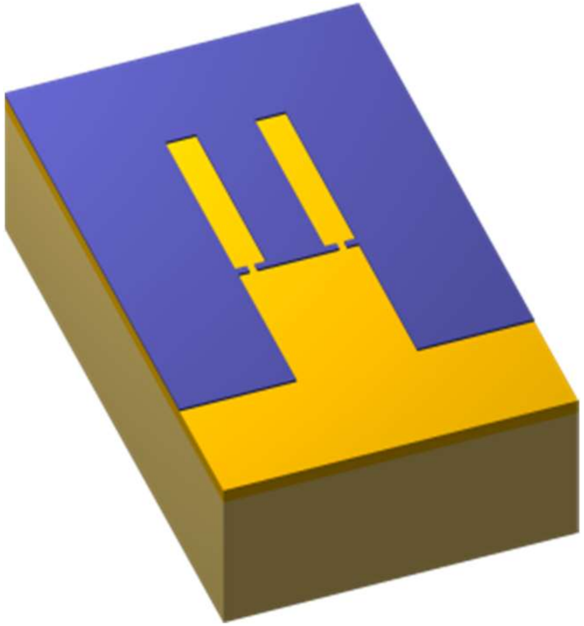
S. Gravier et *al.*, *JMEMS*, vol. 18 (2009) 555

Fabrication steps

First photolithography



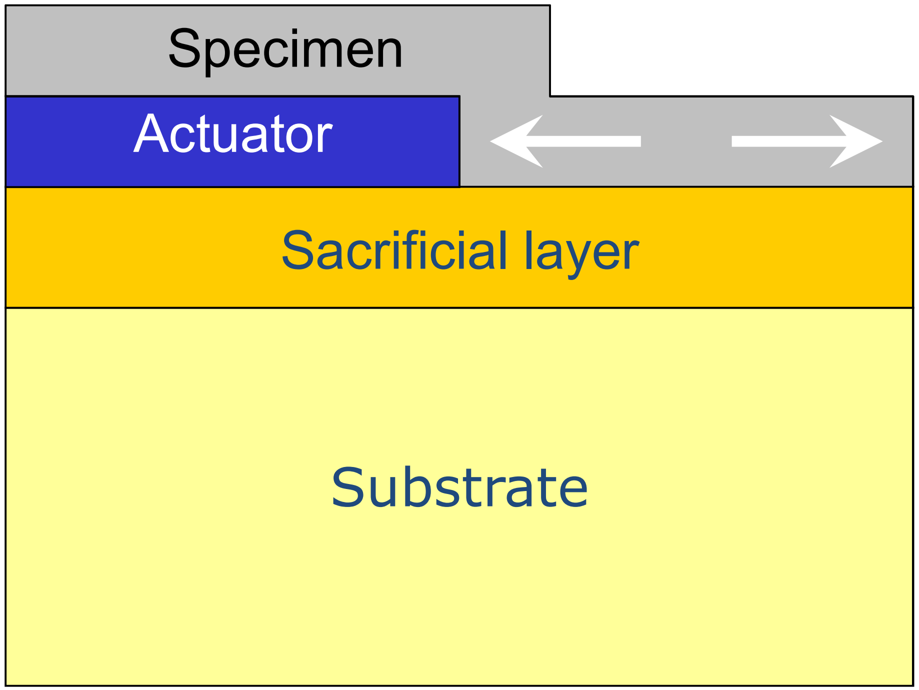
Cross section view



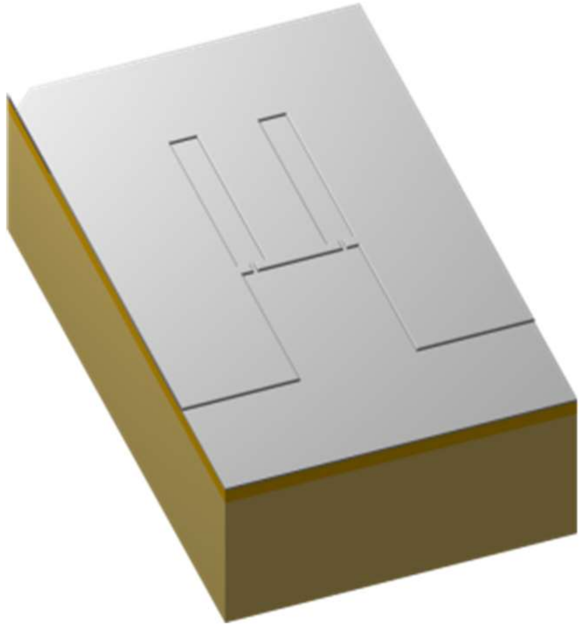
Top view

Fabrication steps

Deposition of test material



Cross section view

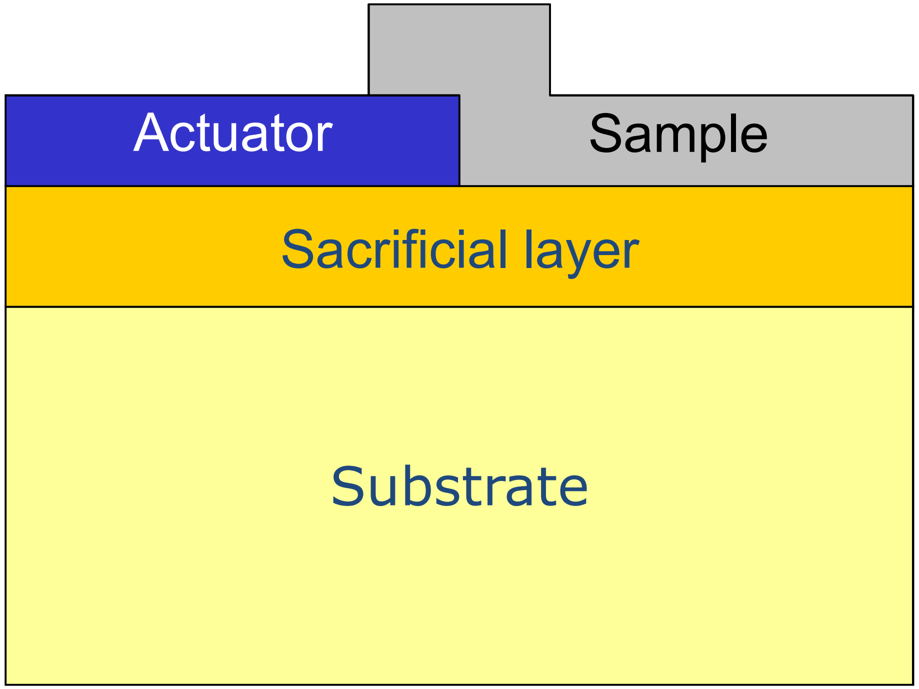


Top view

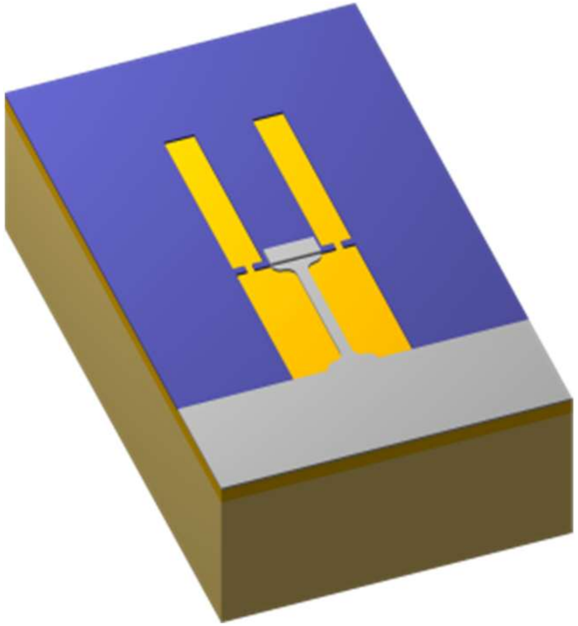
Stoney method
to measure $\sigma^{internal}$

Fabrication steps

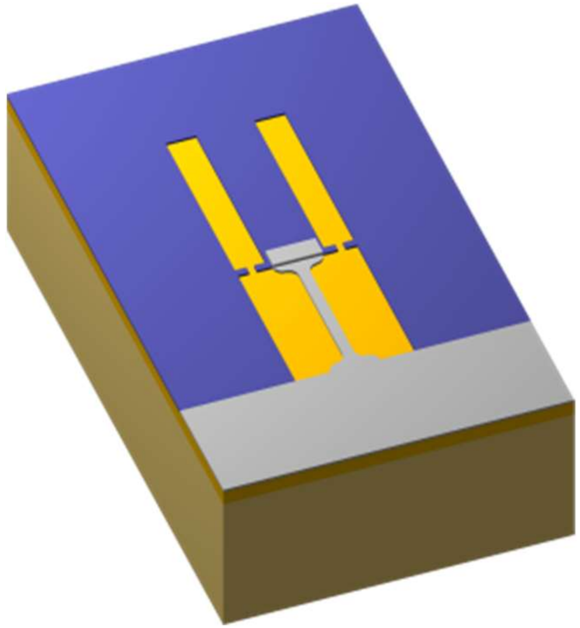
Second photolithography



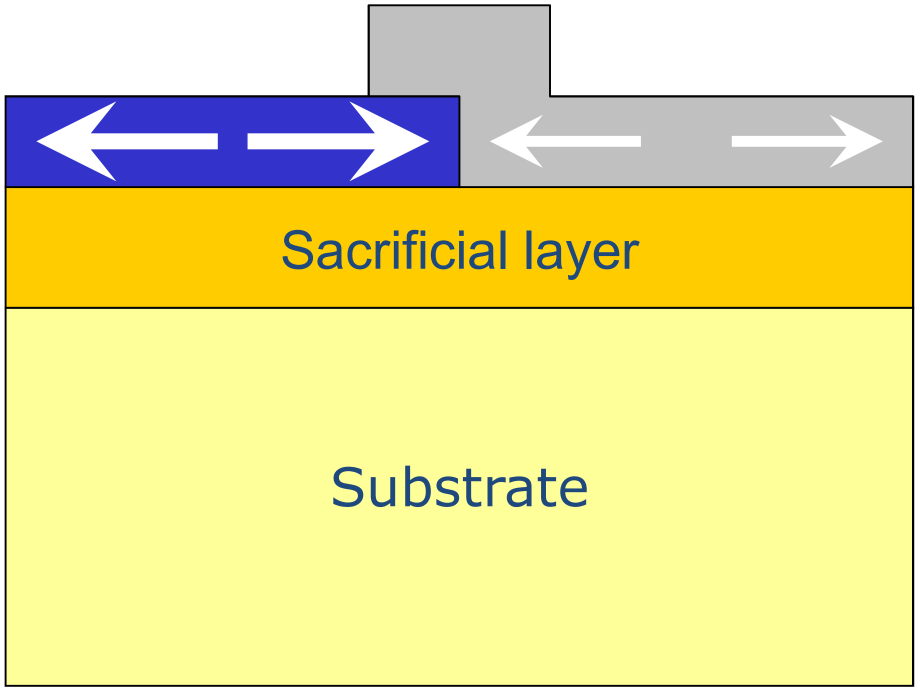
Cross section view


Top view

Fabrication steps



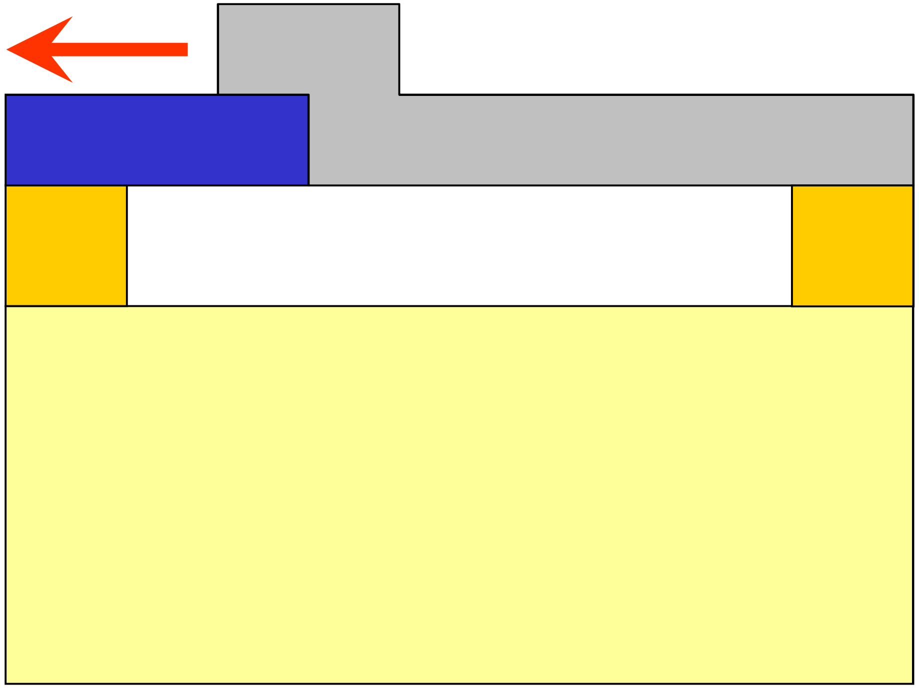
Starting point of the tensile test

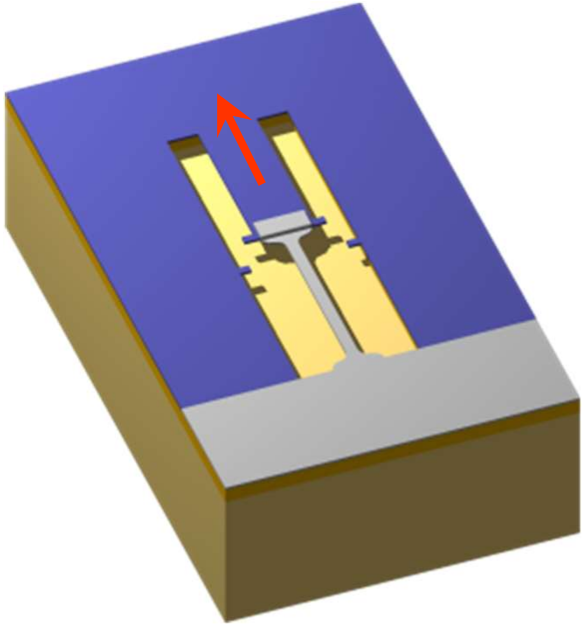


Fabrication steps

Release of the structures

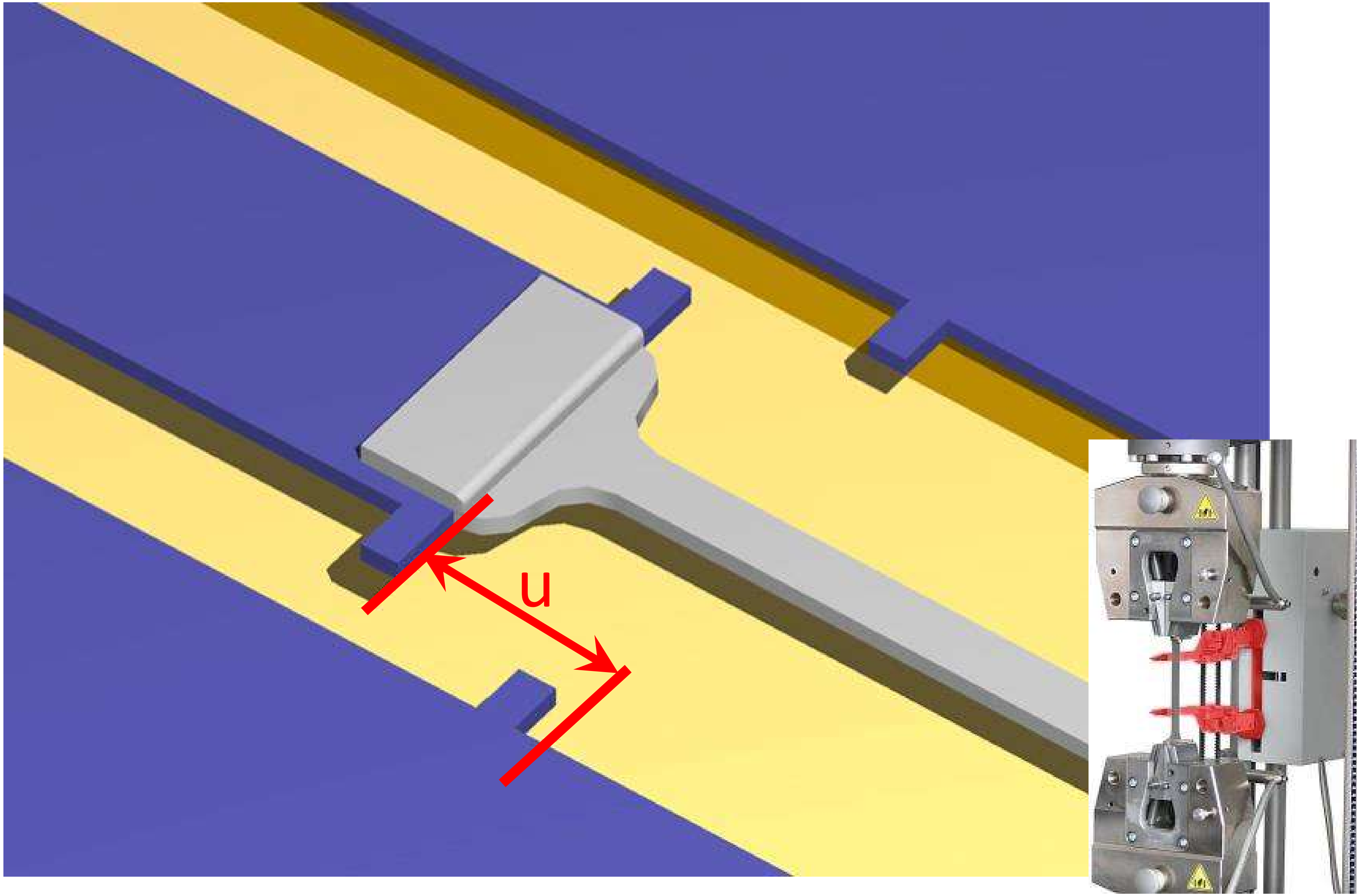
(e.g. *HF wet etching)*



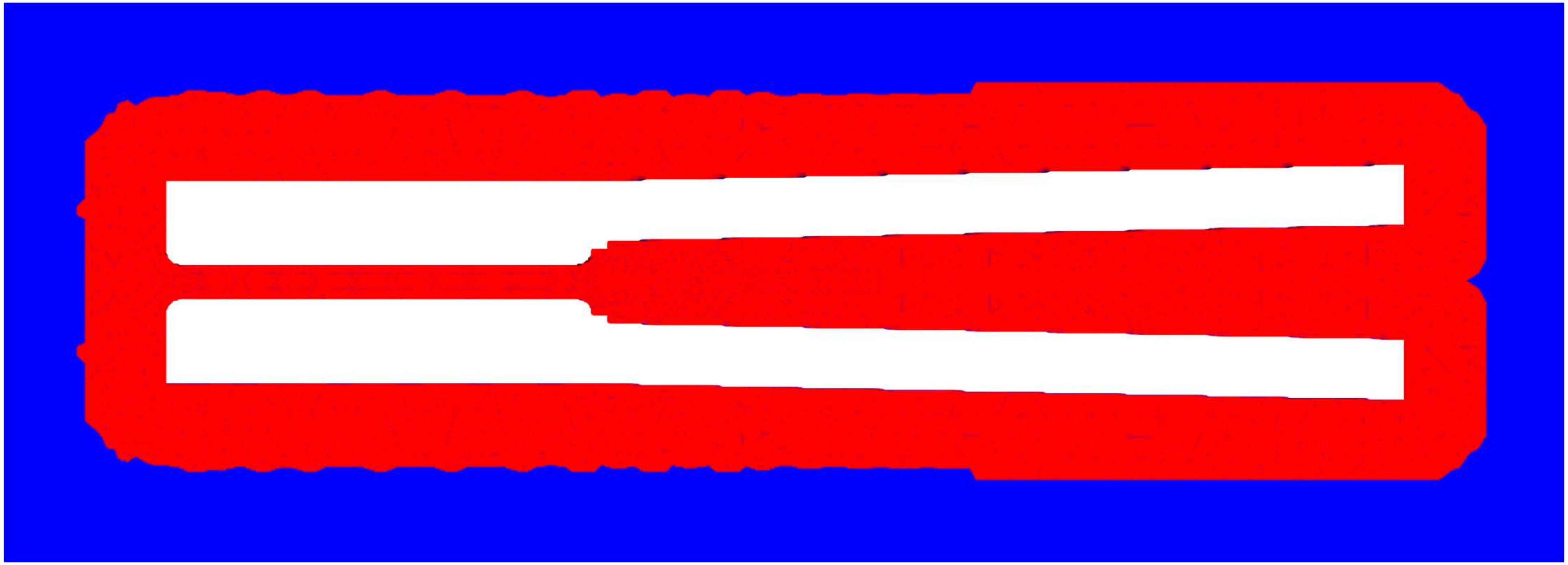


- Critical : etching selectivity
- Actuator is wider and specimen is thus released first
- Strain rate is not controlled

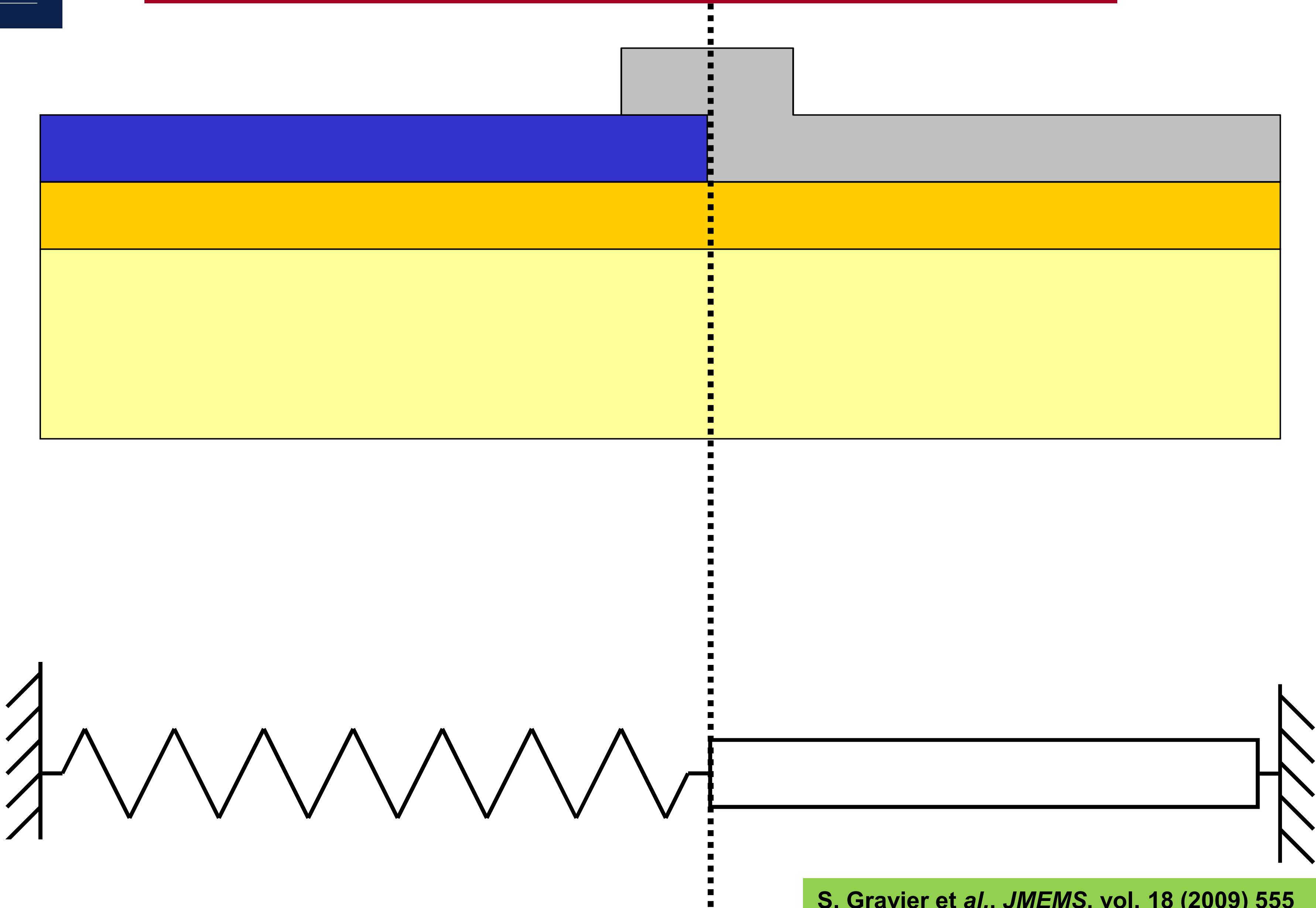
Measurement of displacement



Simulations of the release process

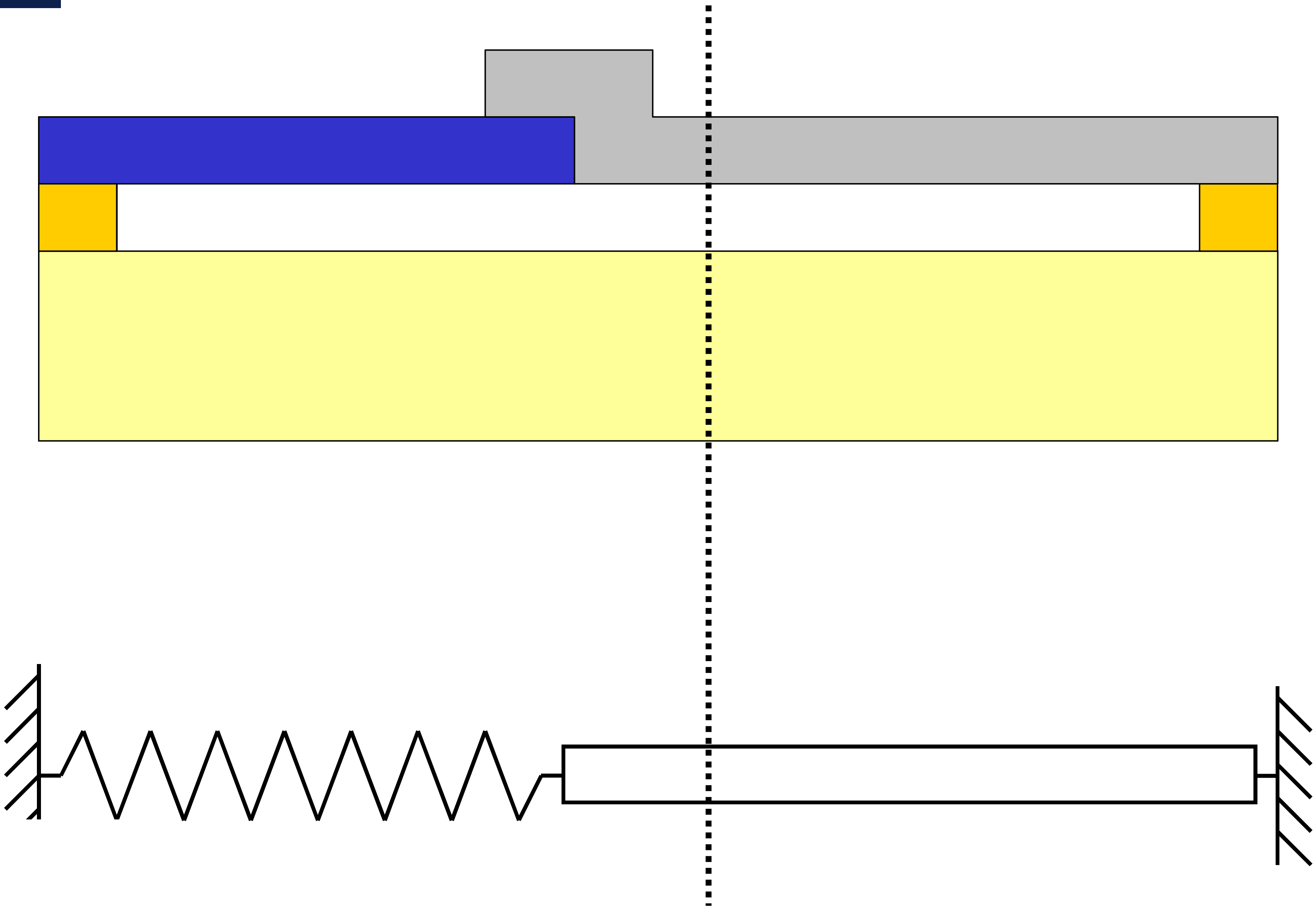


Principle of the force measurement

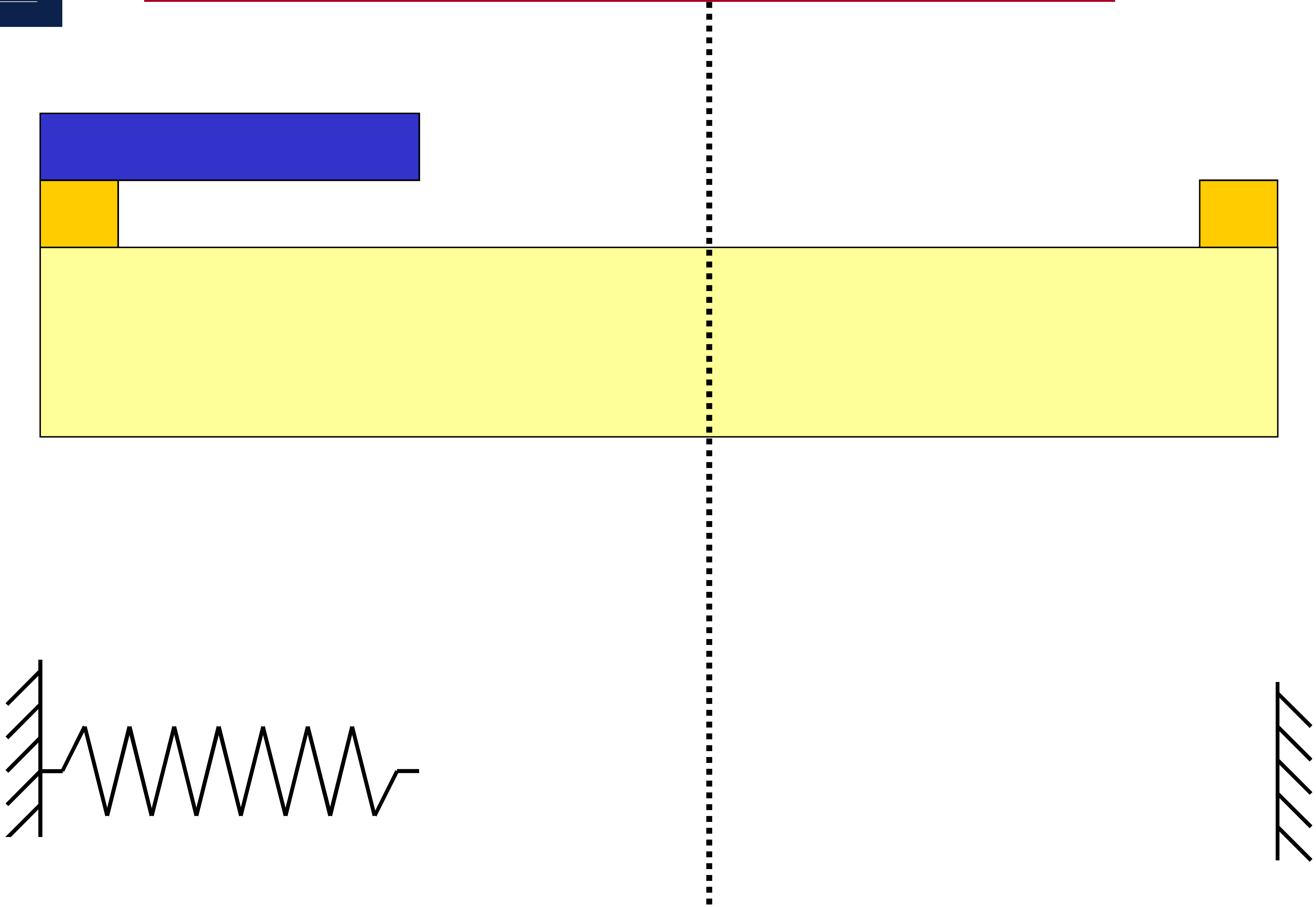


S. Gravier et *al.*, *JMEMS*, vol. 18 (2009) 555

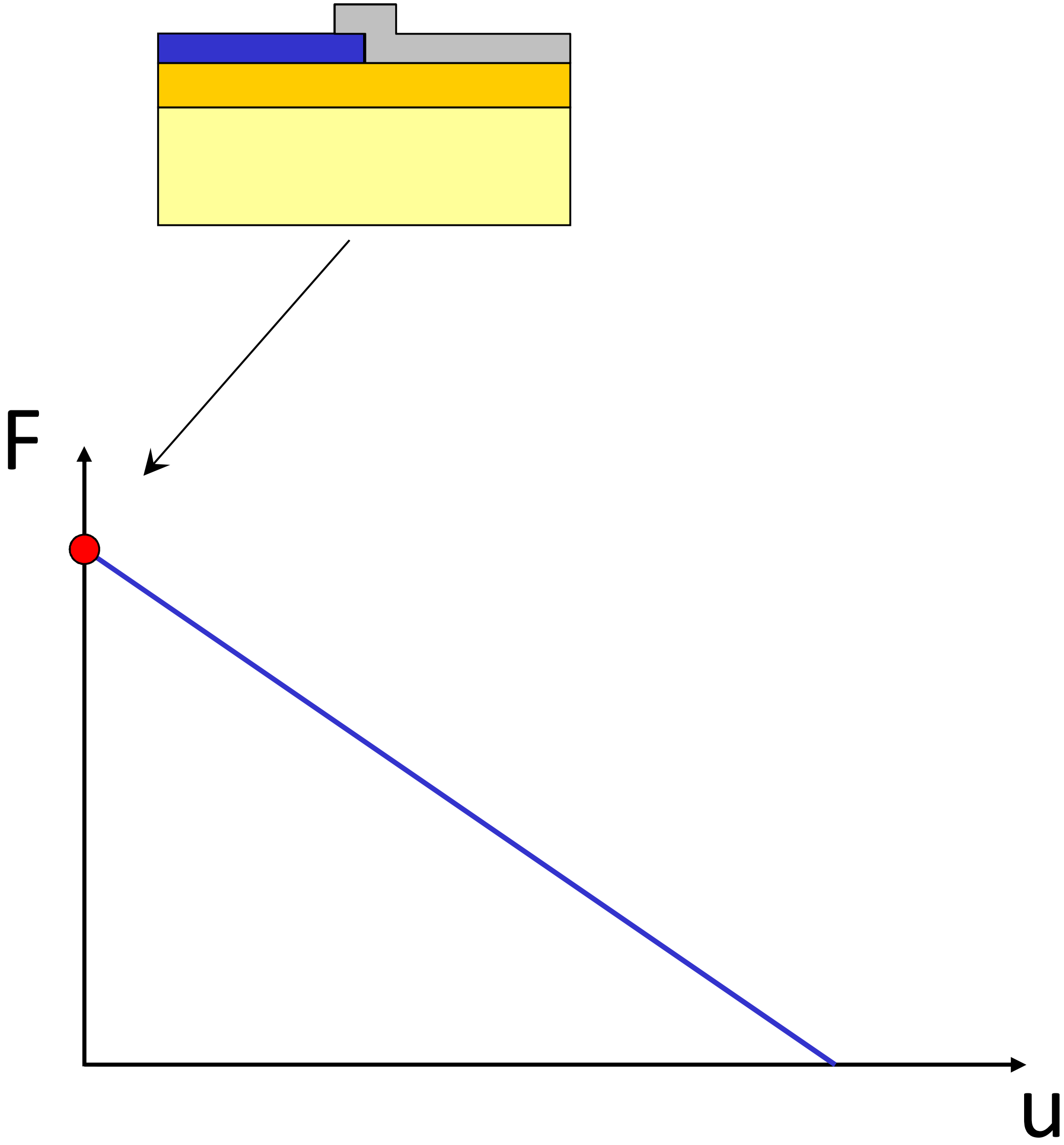
Principle of the force measurement



Principle of the force measurement

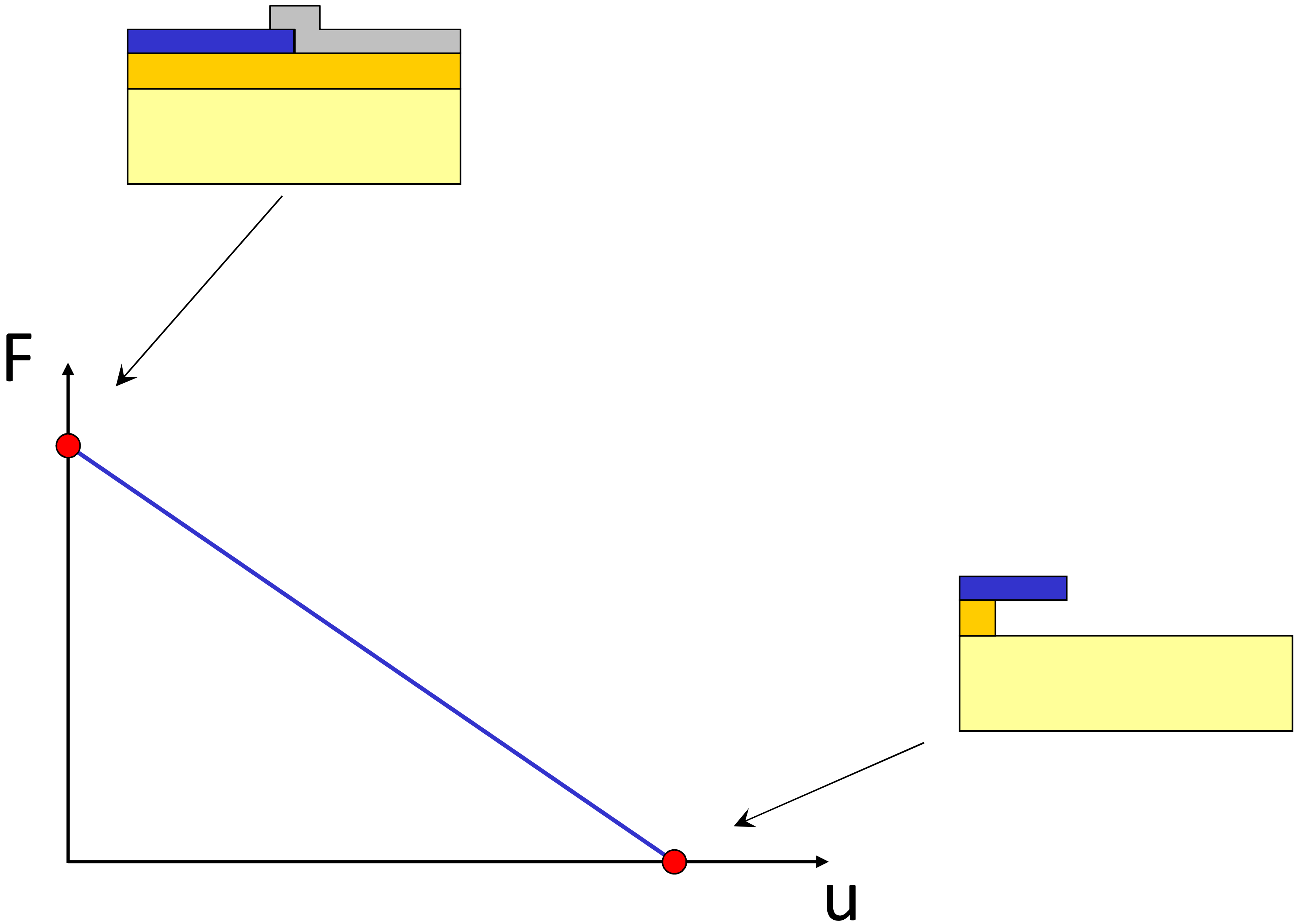


Principle of the force measurement

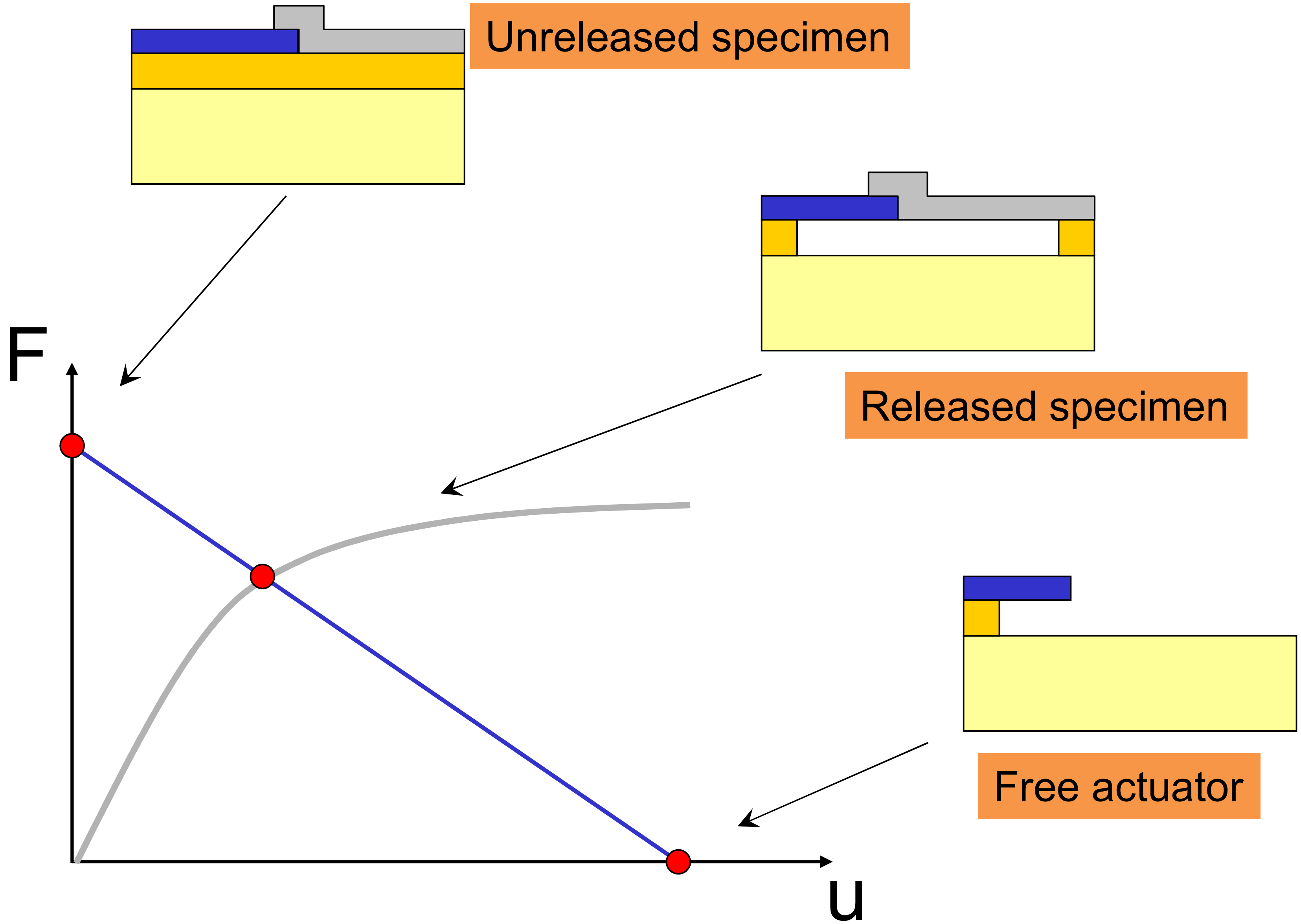


UCL Université catholique de Louvain

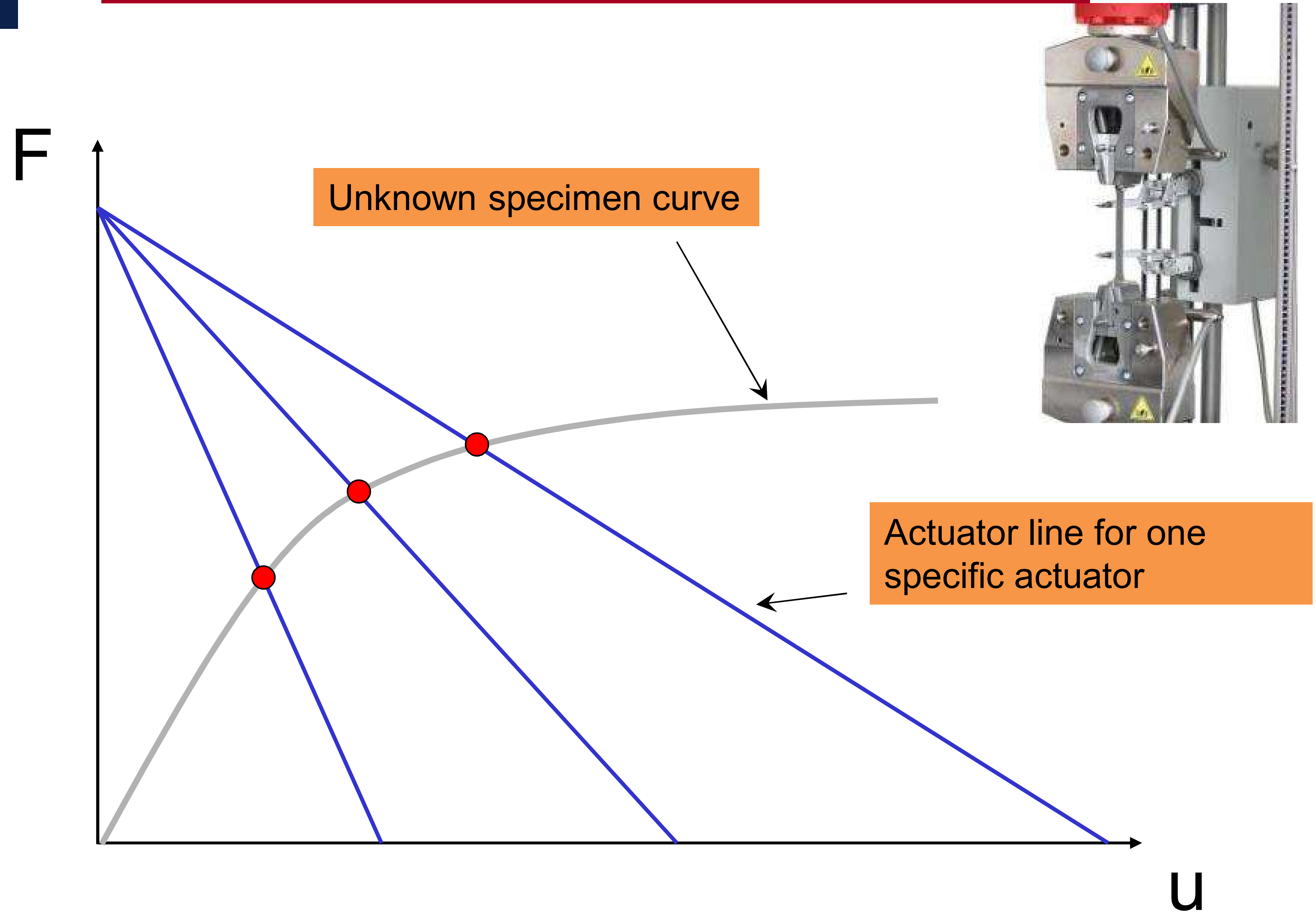
Principle of the force measurement



Principle of the force measurement

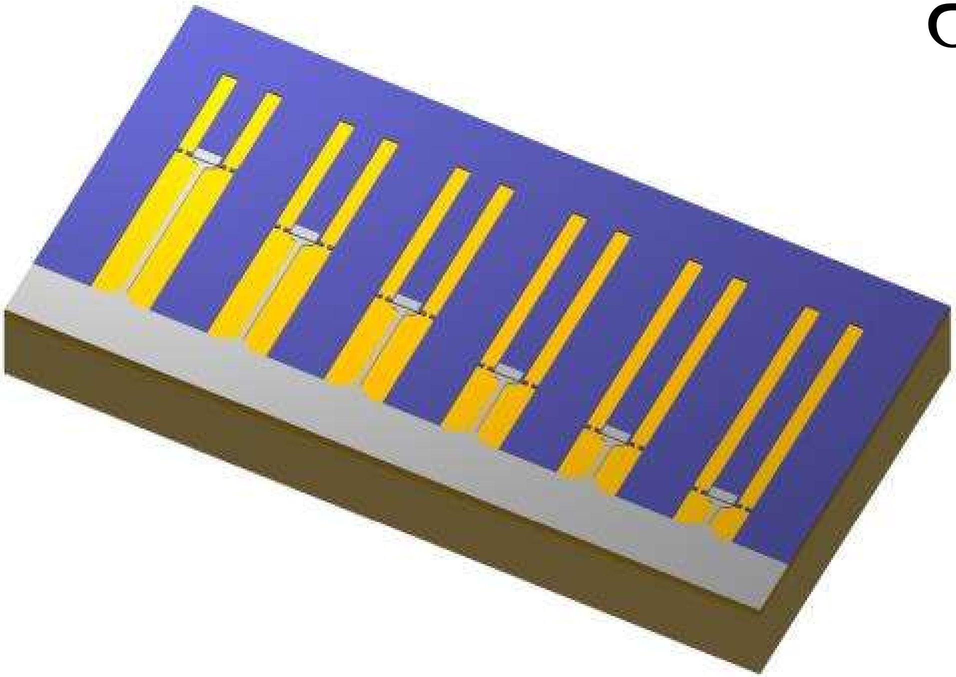


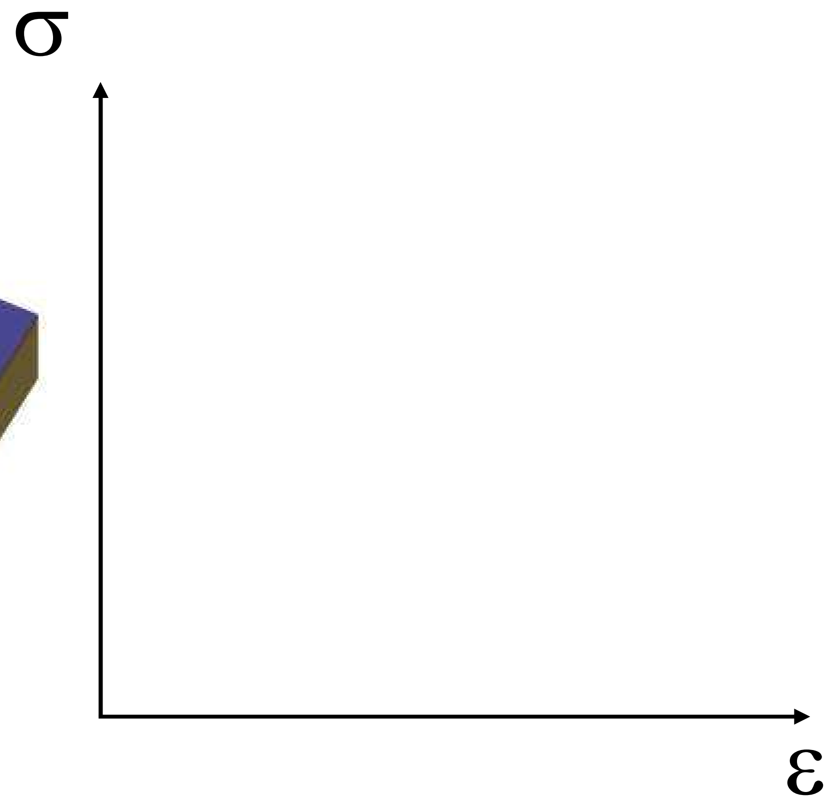
Principle of the force measurement



From single tensile stage to full stress strain curve determination

Both actuator and sample length can be varied.





S. Gravier et *al.*, *JMEMS*, vol. 18 (2009) 555

From single tensile stage to full stress strain curve determination

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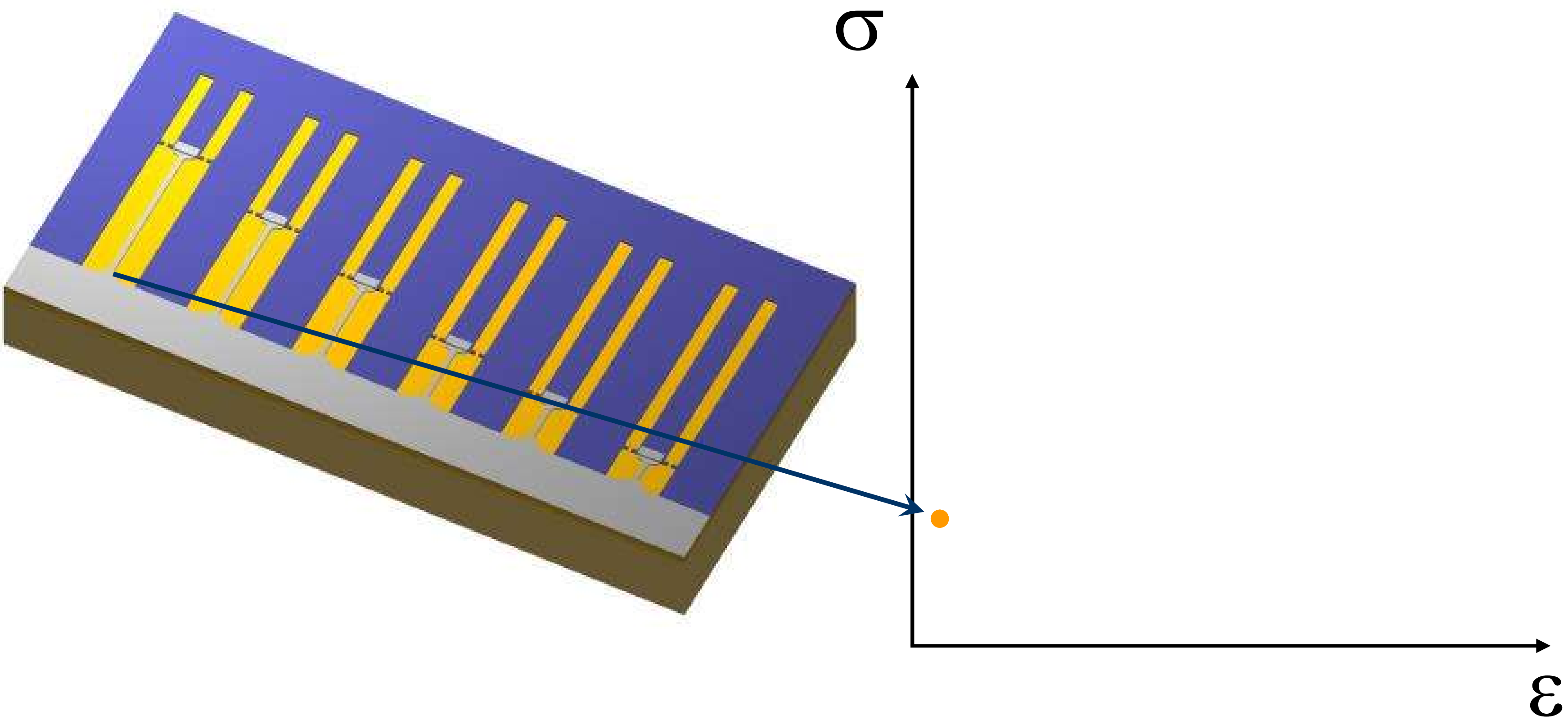

From single tensile stage to full stress strain curve determination

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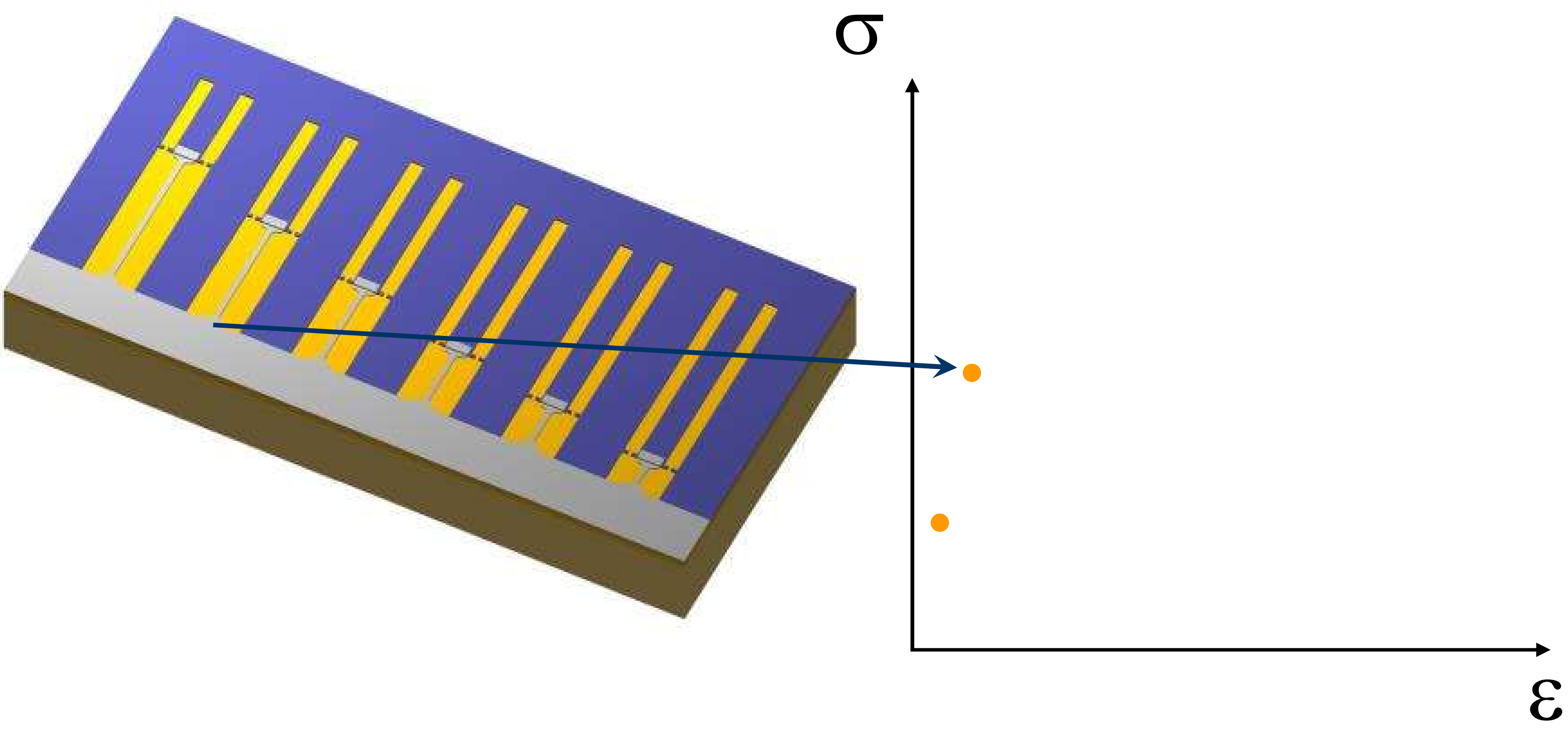

From single tensile stage to full stress strain curve determination

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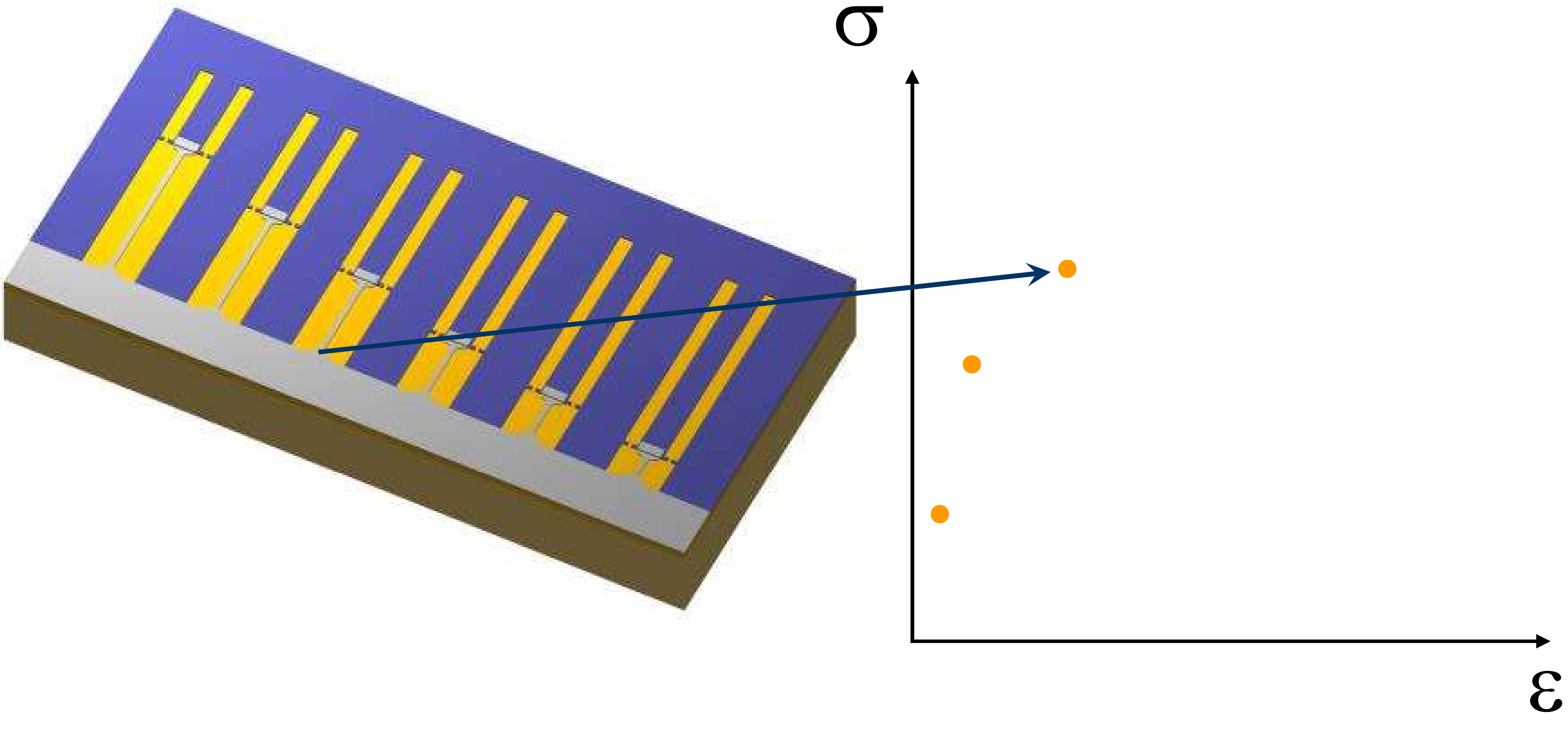

From single tensile stage to full stress strain curve determination

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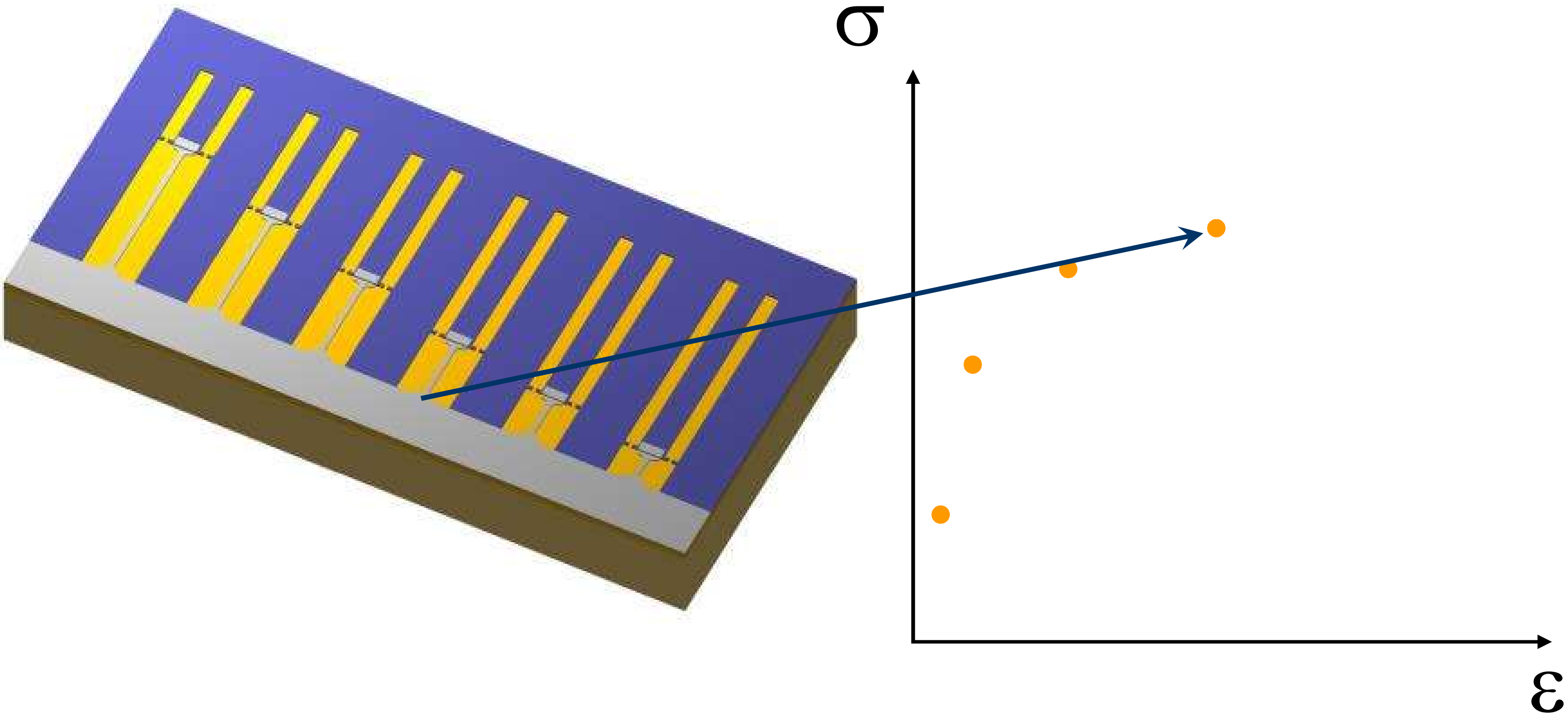

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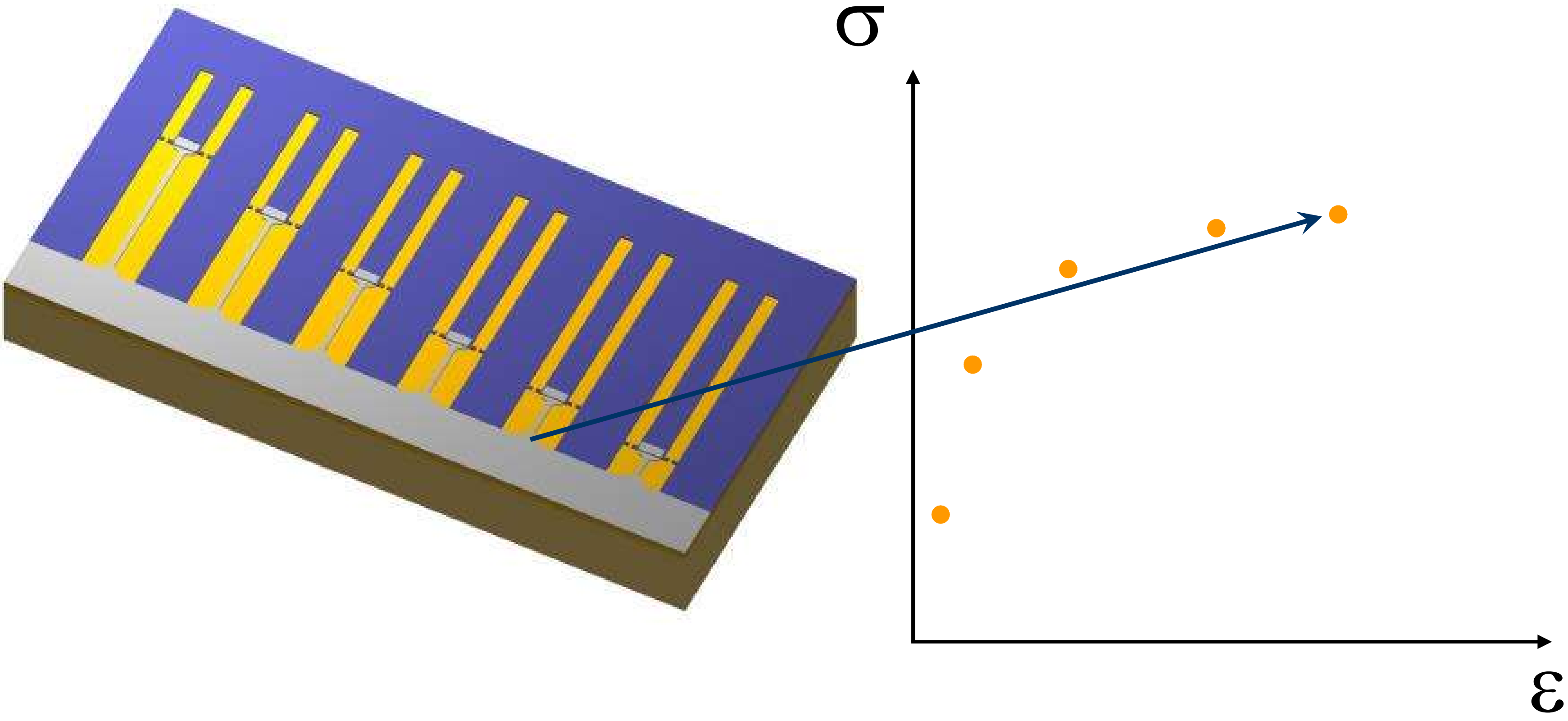
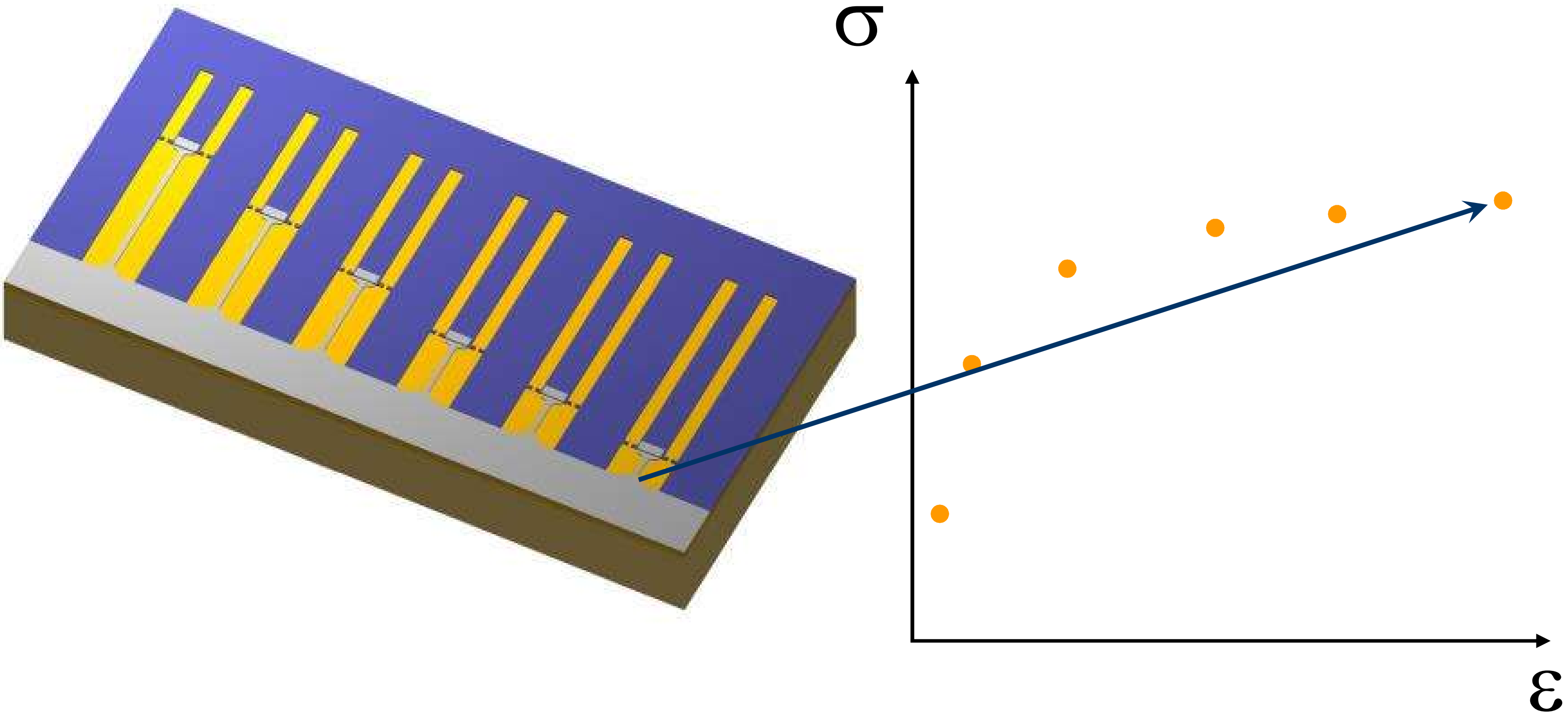


From single tensile stage to full stress strain curve determination

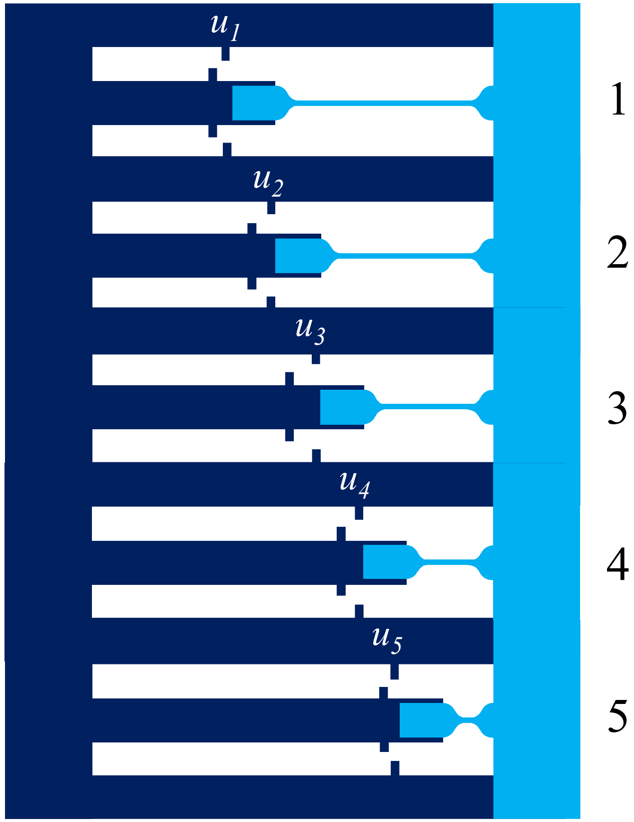
Both actuator and sample length can be varied.



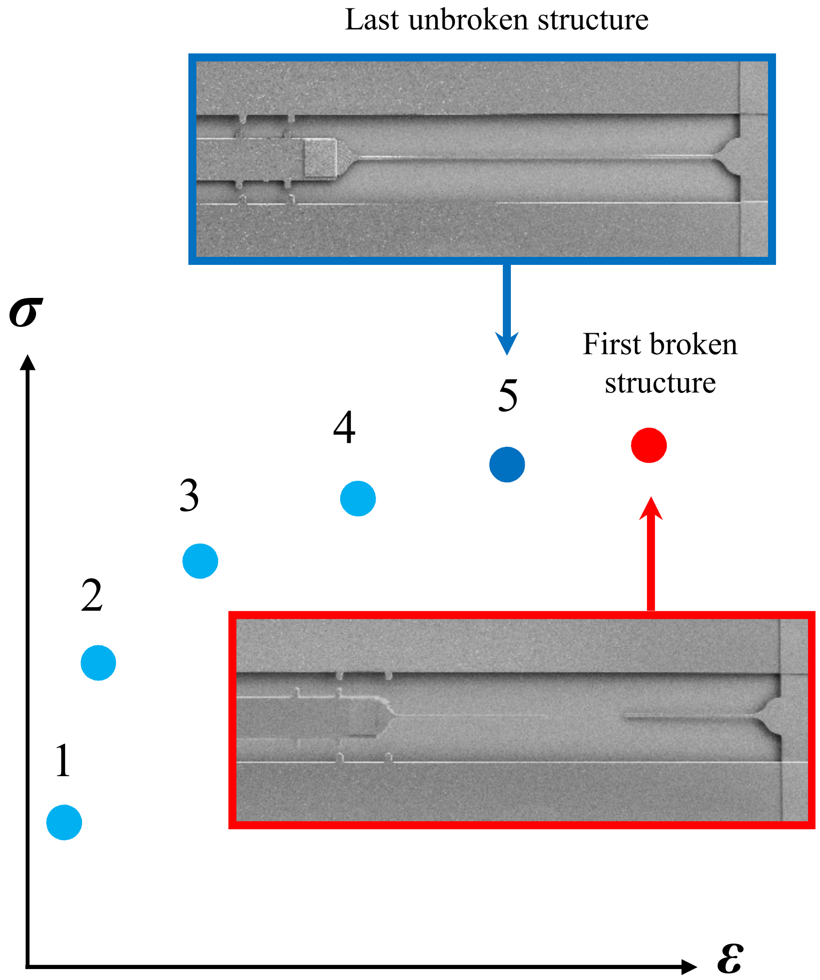
Determination of fracture strain

Actuator material

Specimen material



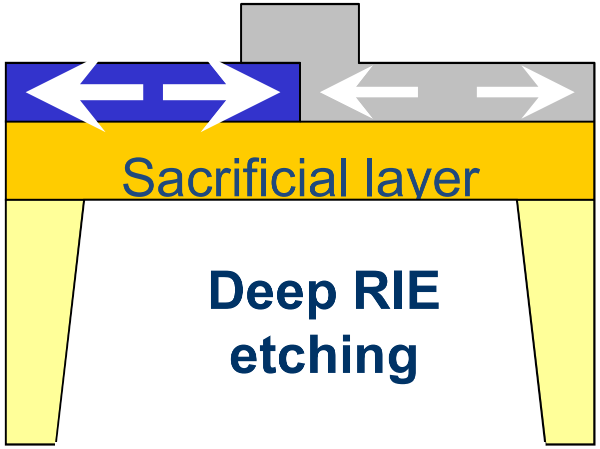
u_1, u_2, ...

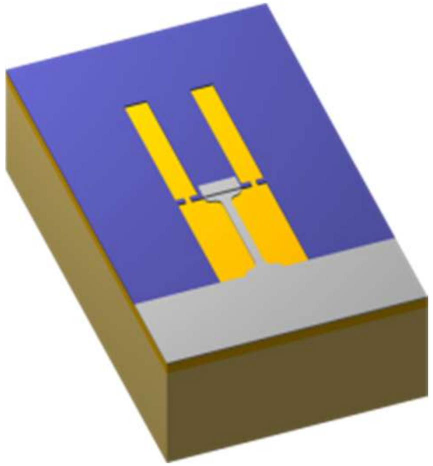


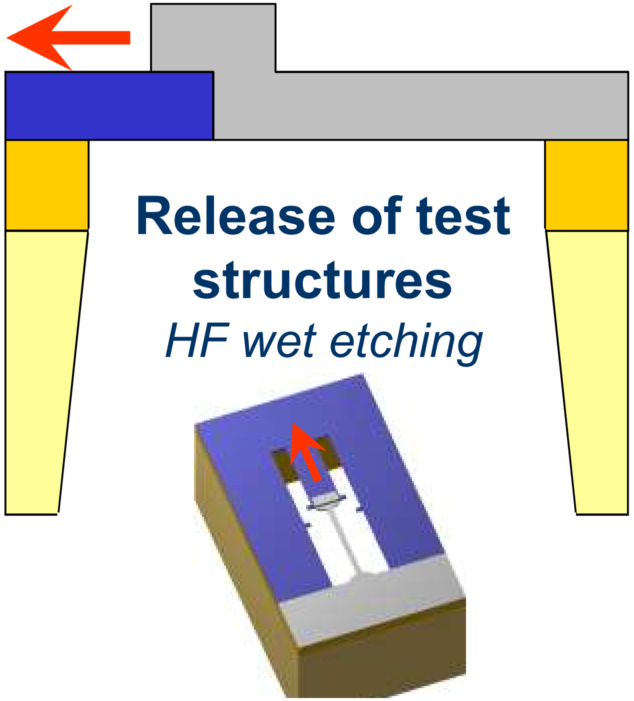
Discrete stress - strain curve

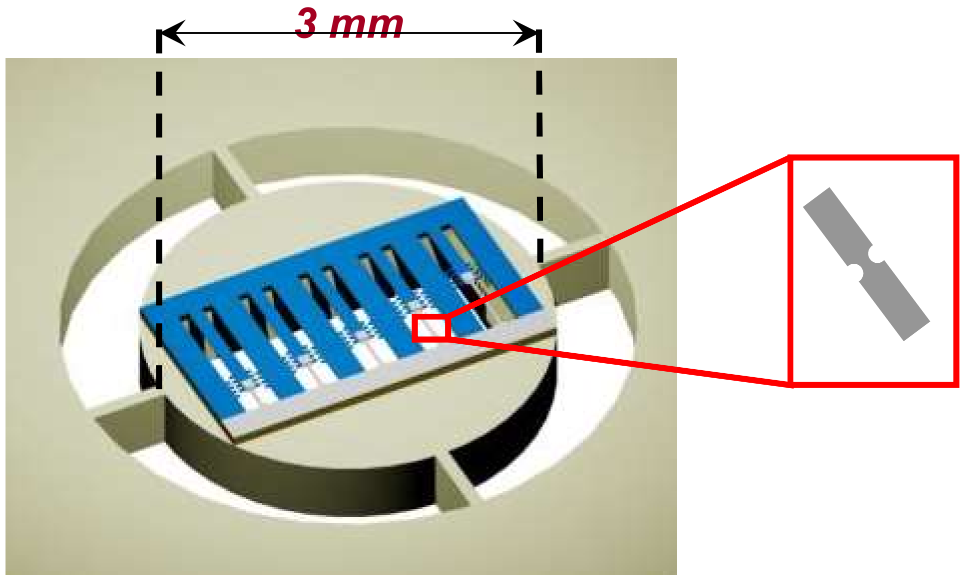
S. Gravier et *al.*, *JMEMS*, vol. 18 (2009) 555

Extension to in- or ex- situ TEM analysis









Colla *et al.*, *Nature Comm* 2015

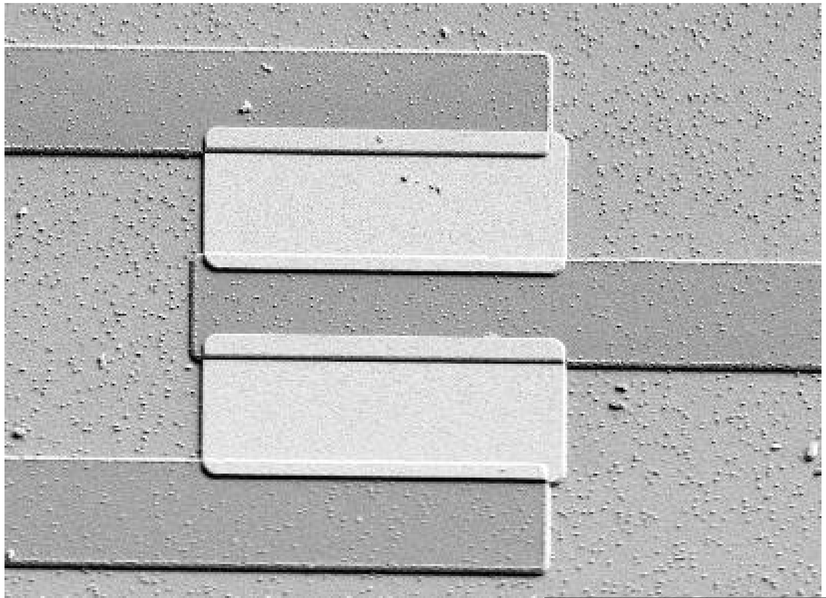
Nanomechanical lab on chip

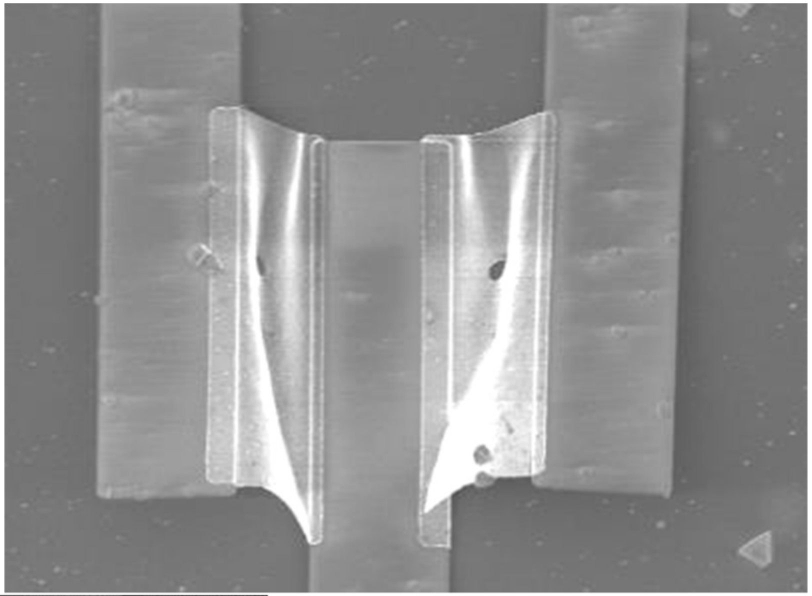
1 wafer

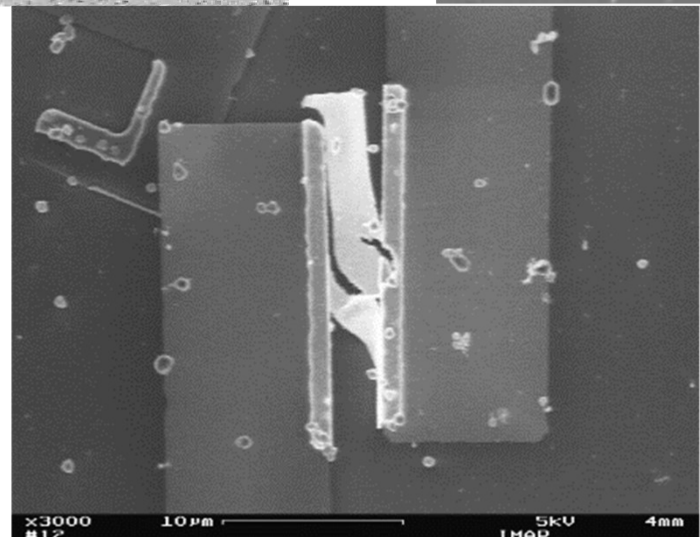
2 weeks of processing

~ 10.000 test structures

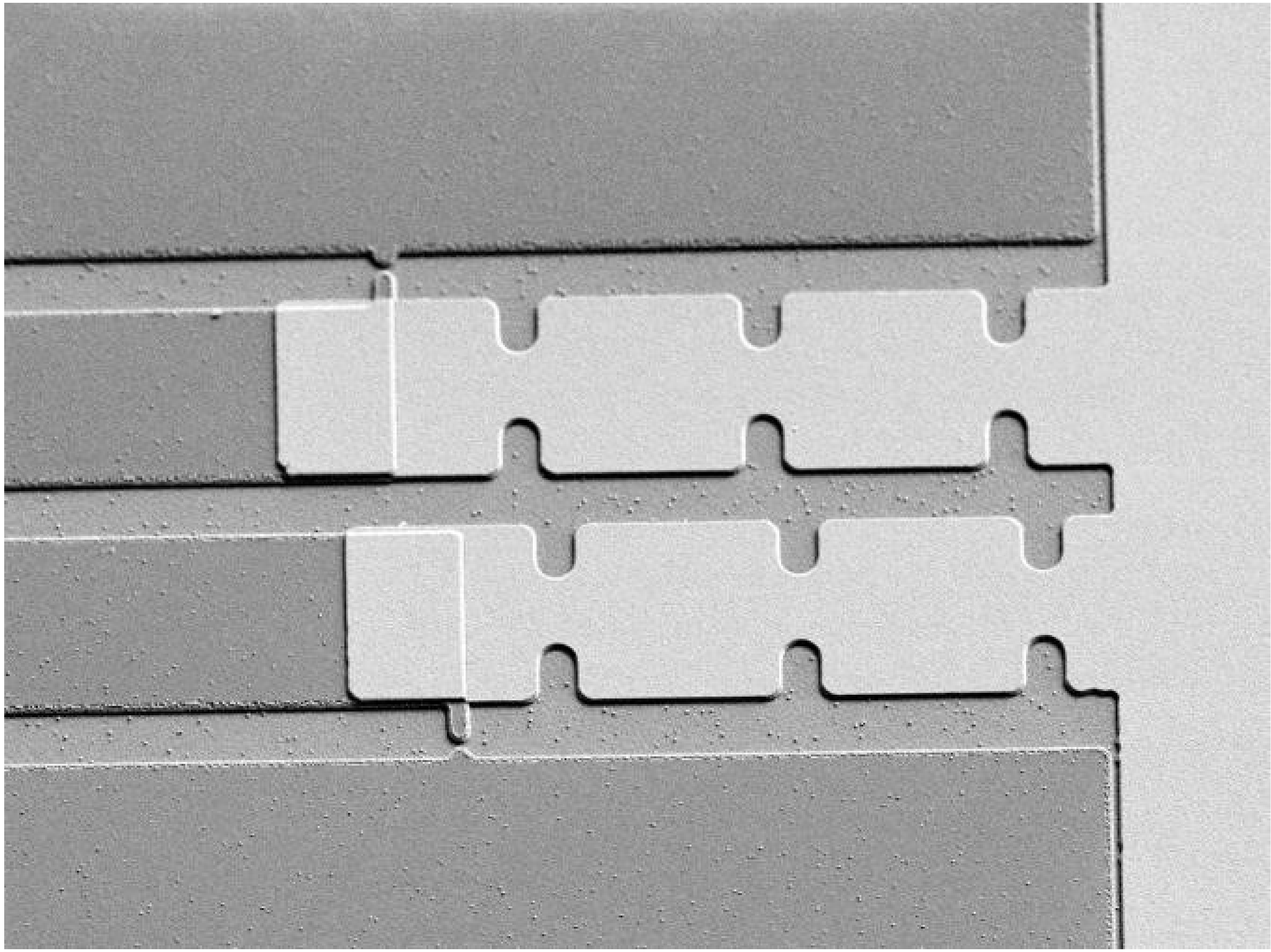
Shear tests



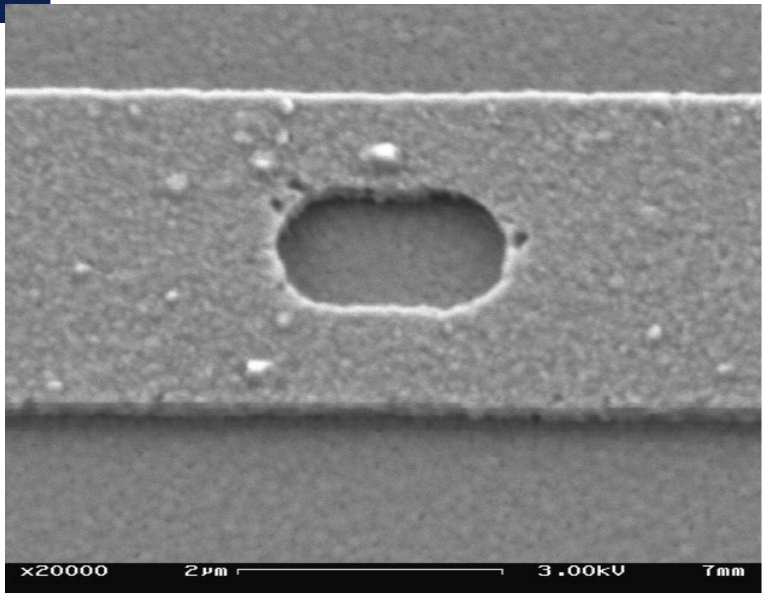


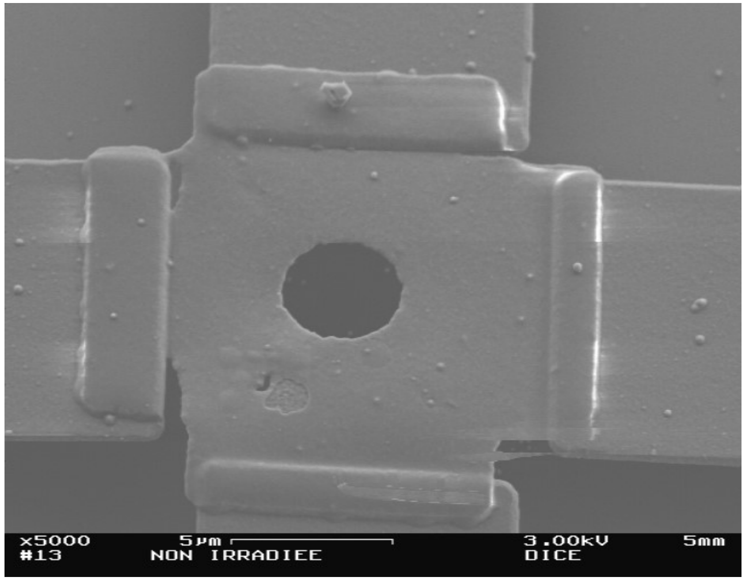


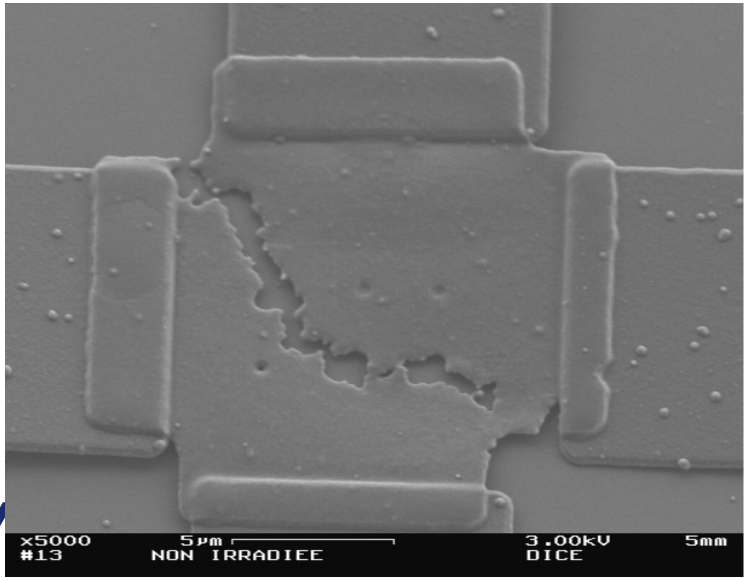
Notched specimens

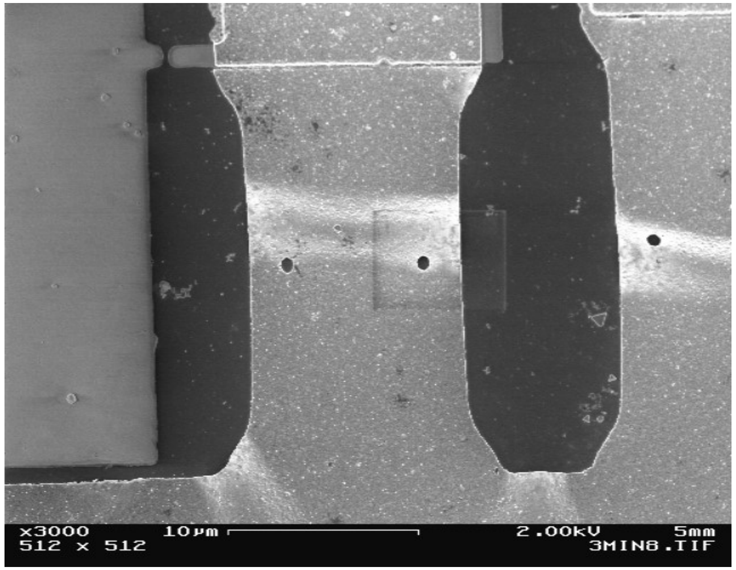


- and many others … -

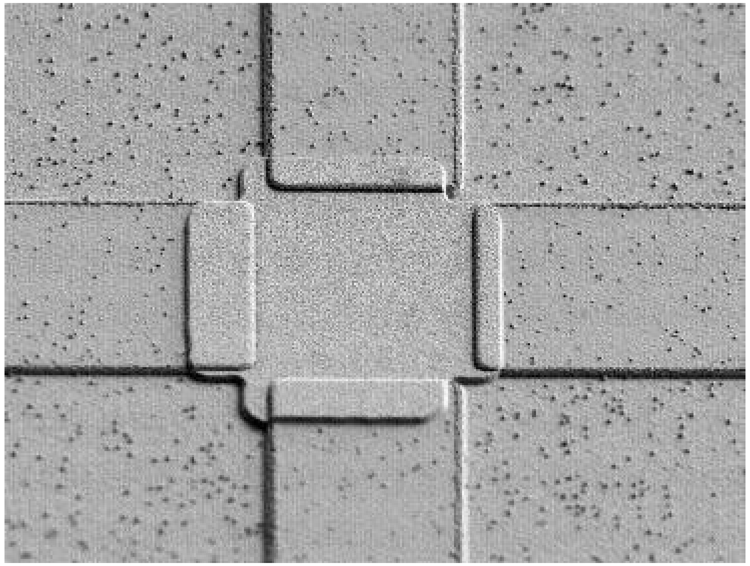


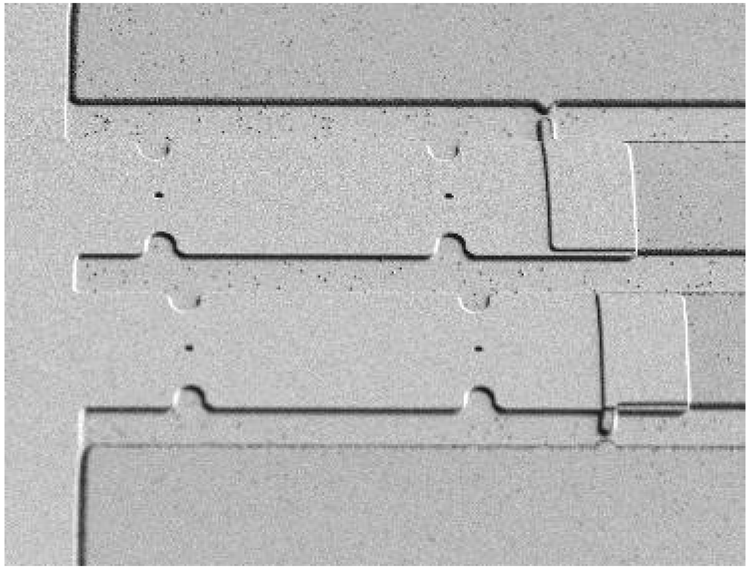


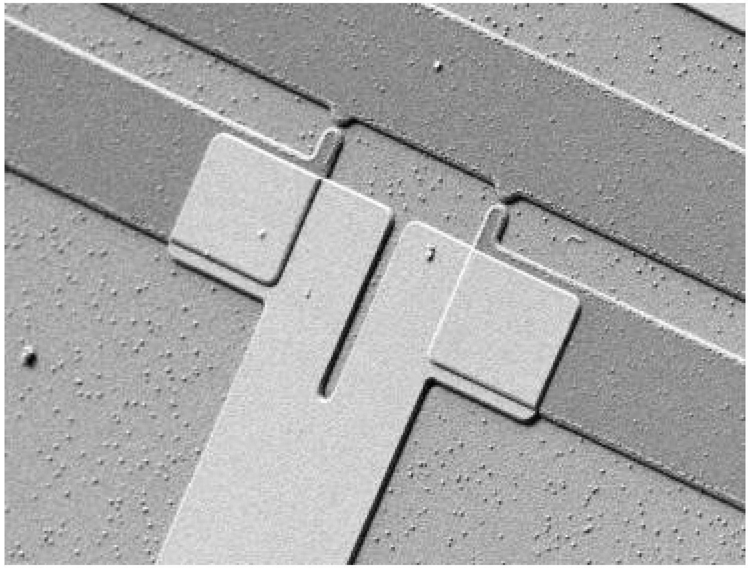


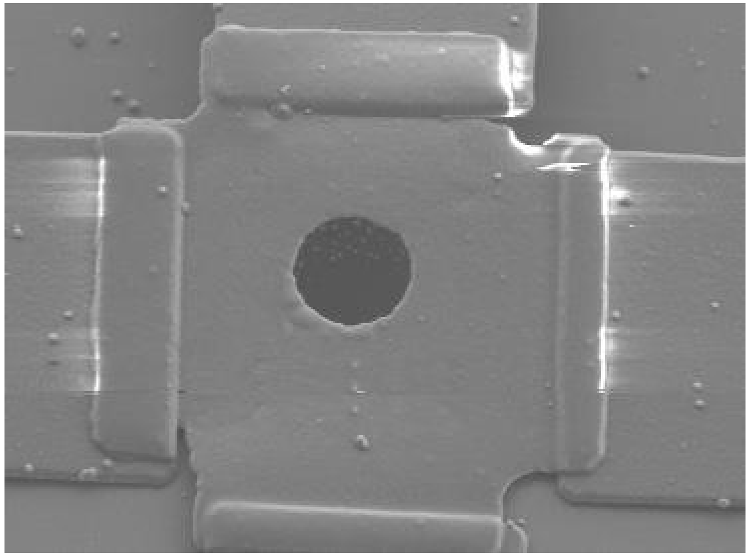


and many others…









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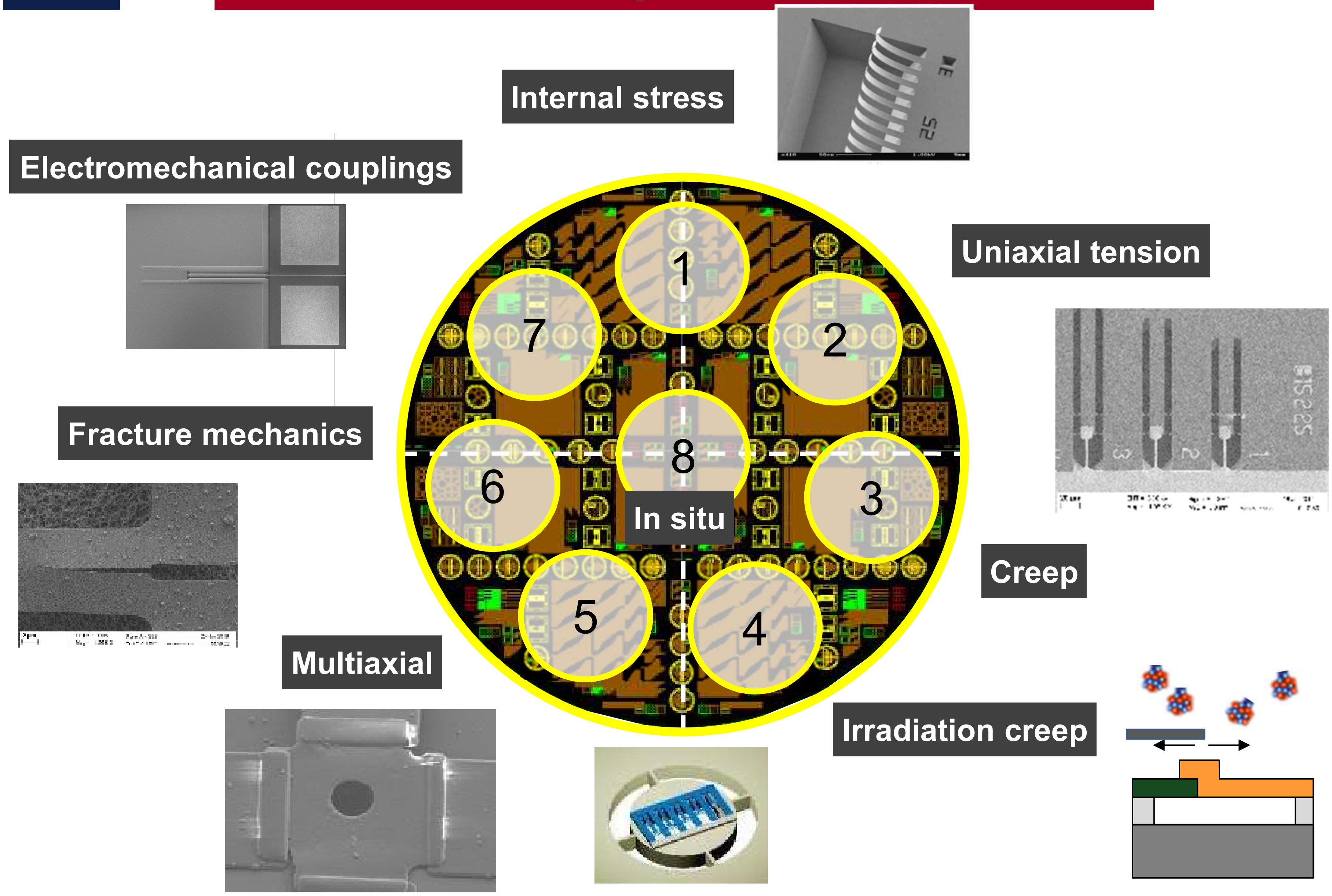
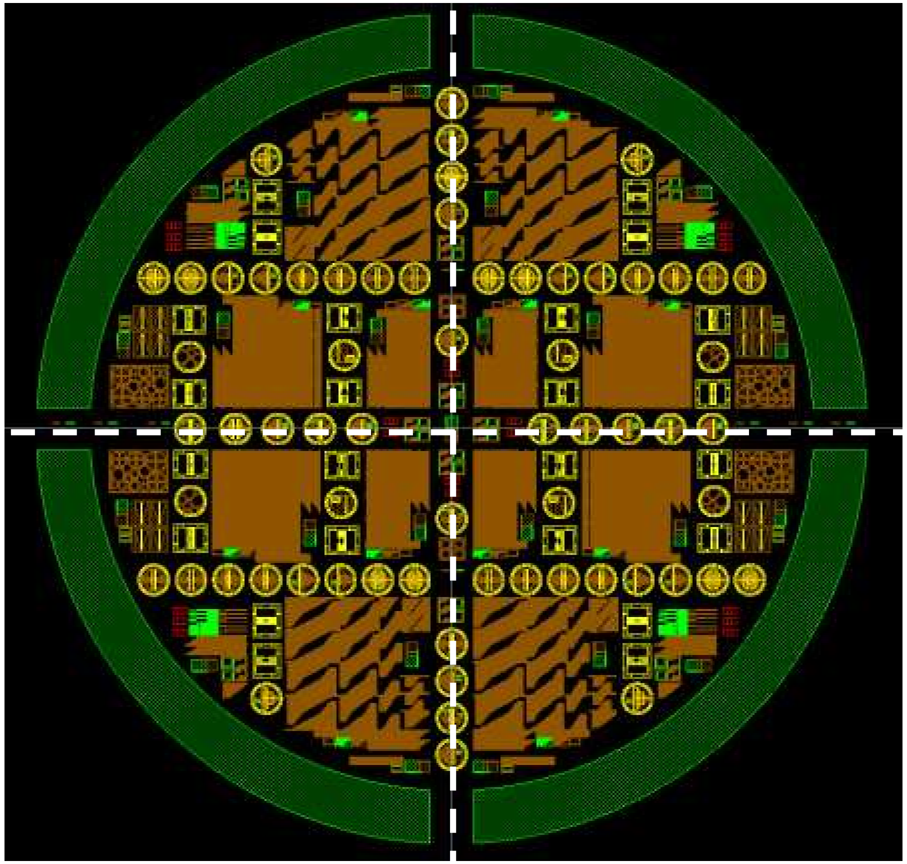
UCL On chip nanomechanical testing platform



Lab-on-chip platform – *Last generation (#8)*

Global top view of the last generation masks
3 inches wafer

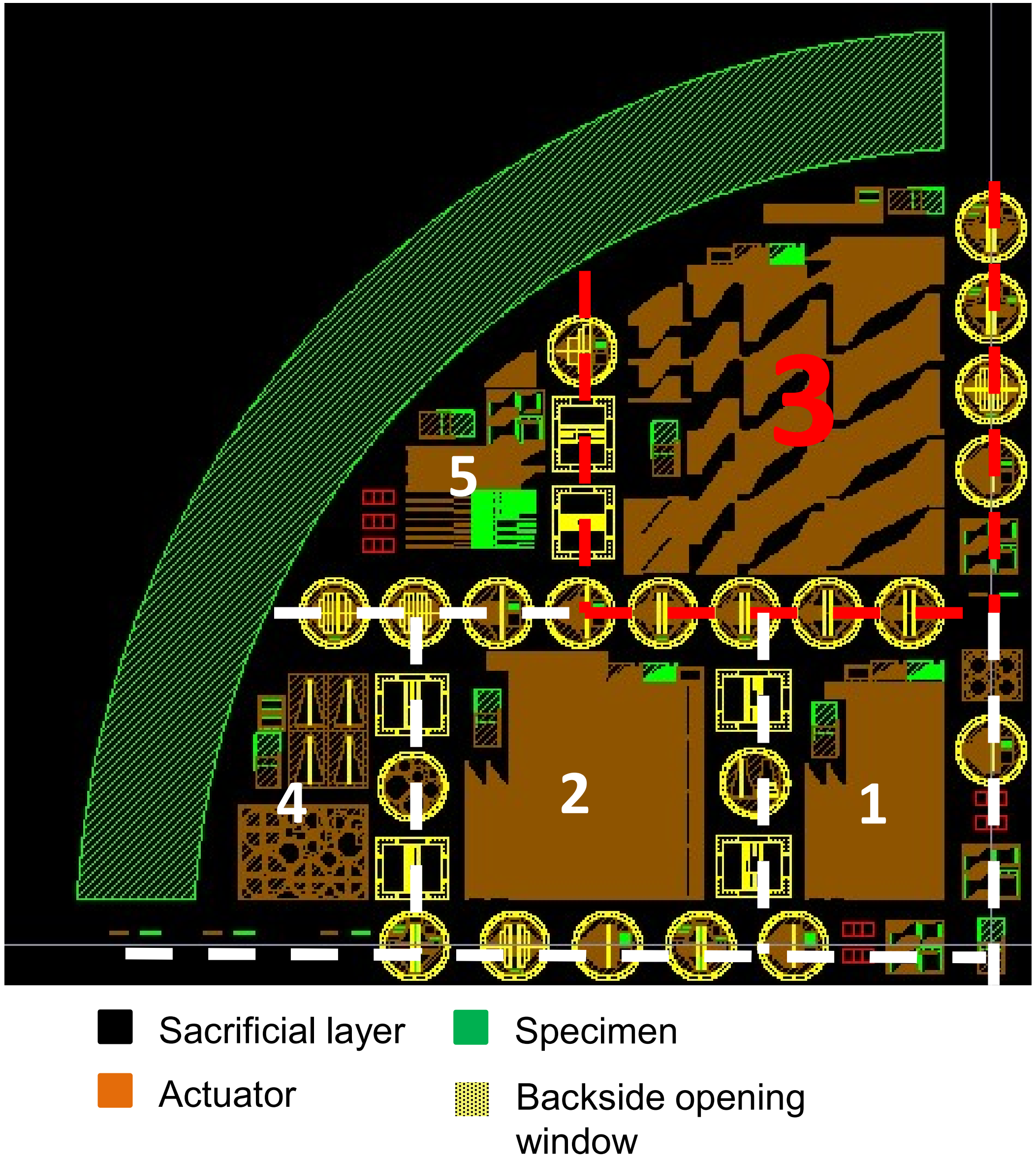


4 equivalent areas

All the structures are repeated 4 times

4*22 TEM compatible sets on 1 wafer

Lab-on-chip platform – *Last generation (#8)*



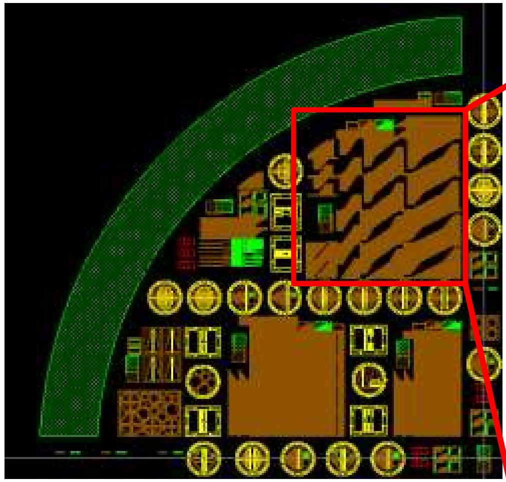
Platform 1, 2 and 3: uniaxial tensile testing for brittle and ductile materials

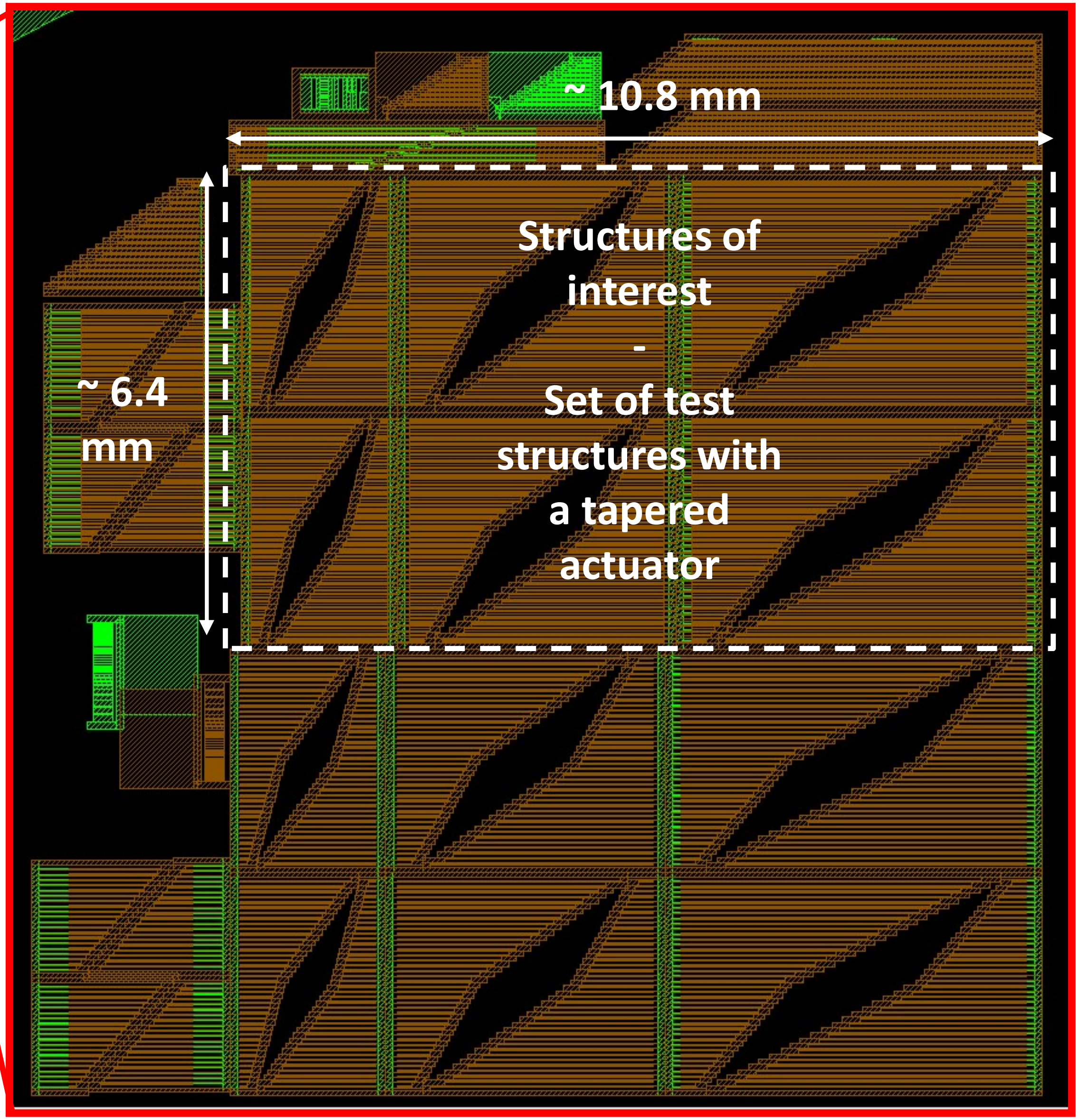
- Large PAD dedicated to measure the thickness
- Structures to extract the mismatch strain and the Young's modulus

Platform 4: Shear and biaxial tensile testings

Platform 5: Structures to extract the mismatch strain, pillars, single and double clamped beams

Lab-on-chip platform – *Last generation (#8)*





- Sacrificial layer
- Actuator
- Specimen
- Backside opening window

Structures of interest

12 sets of 40 micromachines

→ 3 specimen lengths : 25, 50 and 100 µm

→ 4 specimen widths : 1, 2, 4. 6 µm

Outline

1. Introduction

2. Fracture of films on substrates

- test methods and extraction of *G*
- example 1 : CrN on polymer (indentation)
- example 2 : SiN on polymer (subcritical crack growth)
- example 3 : Au on polymer (for flexible electronics)

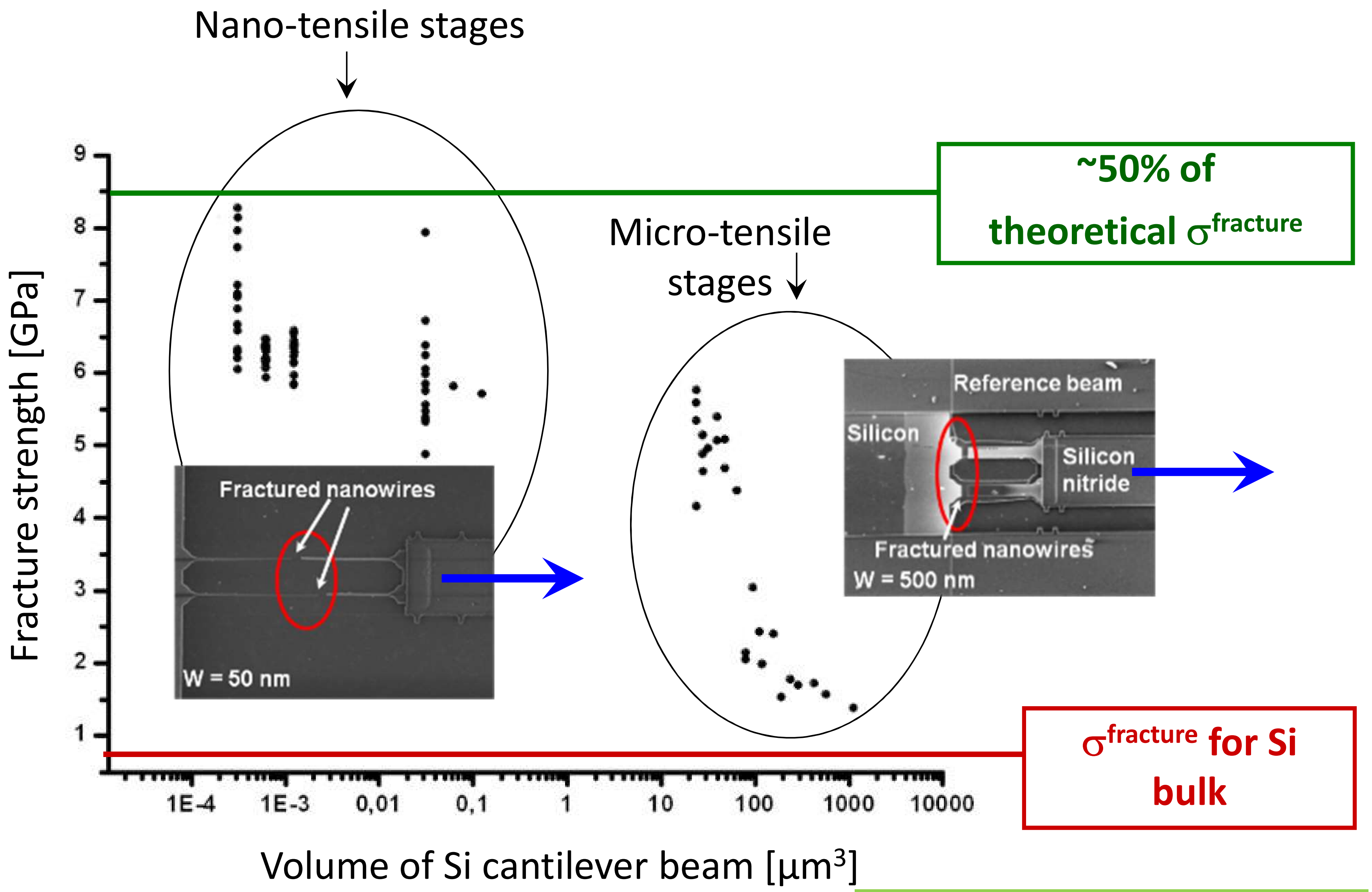
3. Fracture of freestanding films

- Test methods for measuring the fracture strength - strain
- fracture strength of brittle films
- fracture strain of ductile films
- fracture toughness

Example 1 : Fracture strength of PolySi

(PolySi is THE enabling structural material for MEMS devices)

Start with single crystal Si micro and nanowires



V. Passi *et al.*, Rev Sci Inst (2011), JMEMS (2012)

Engineering Fracture Mechanics 168 (2016) 190–203

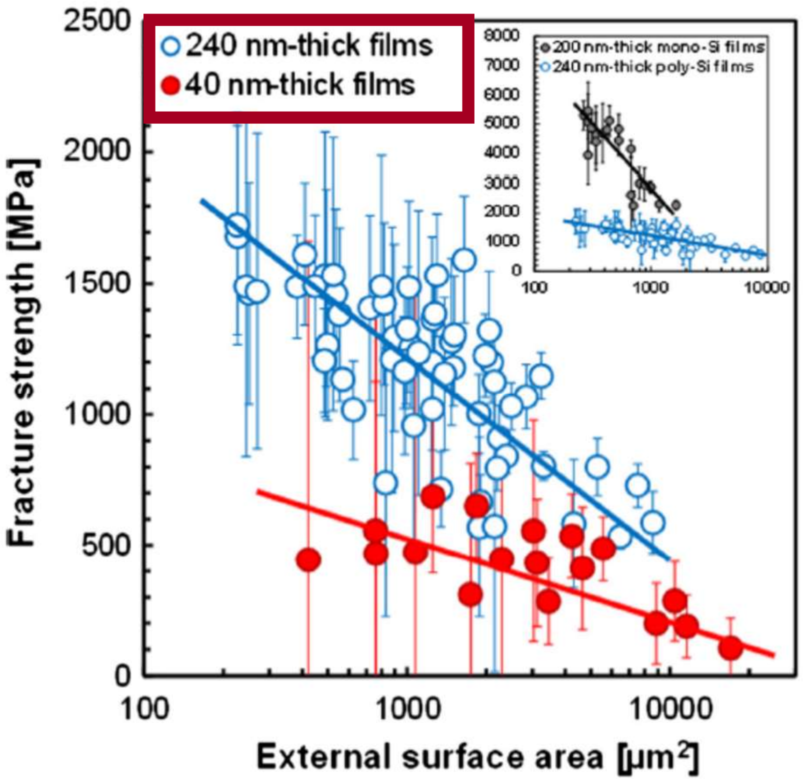
Contents lists available at ScienceDirect

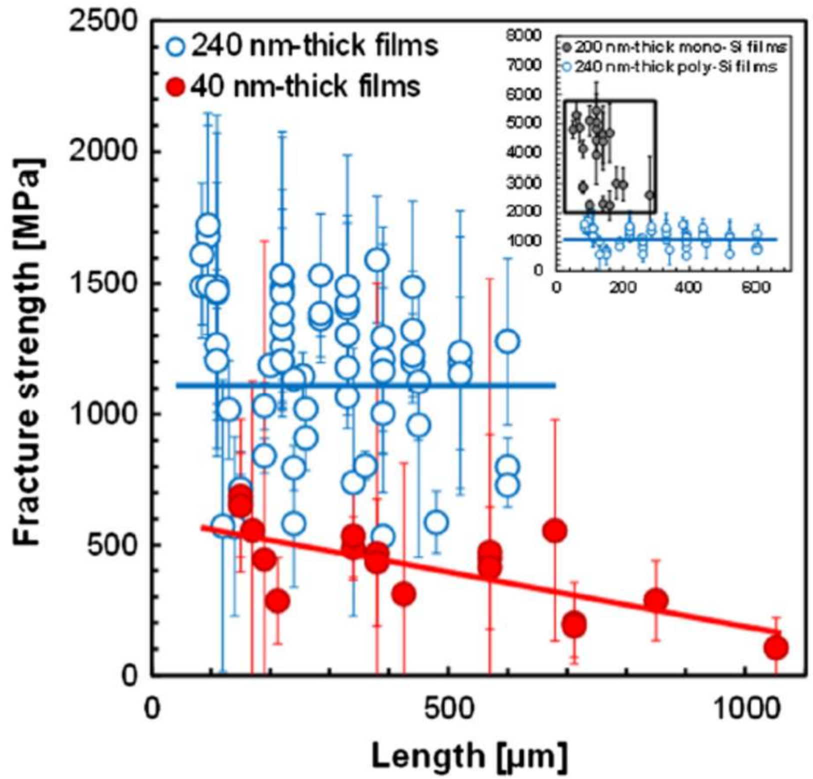
Engineering Fracture Mechanics

journal homepage: www.elsevier.com/locate/engfracmech

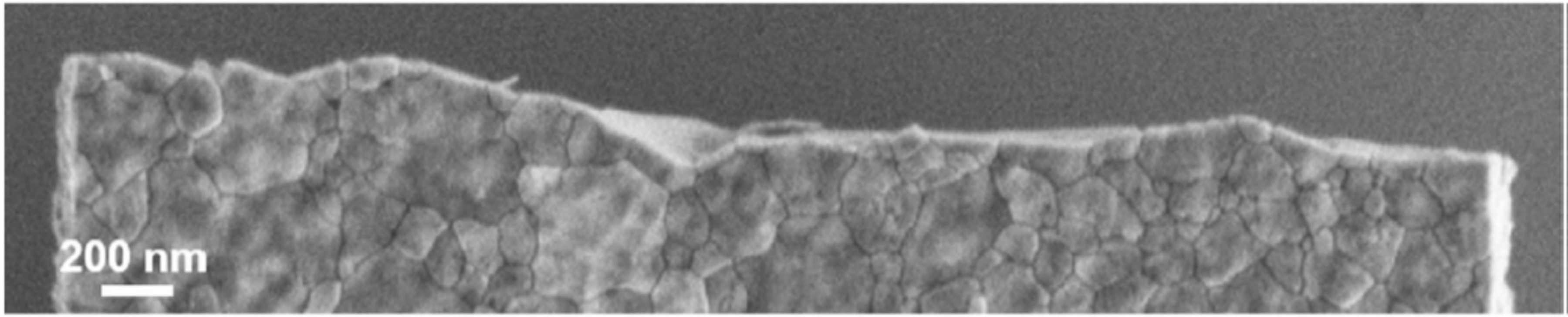
Size dependent fracture strength and cracking mechanisms in freestanding polycrystalline silicon films with nanoscale thickness

R. Vayrette [a,b,*], M. Galceran [c,d], M. Coulombier [a], S. Godet [d], J.-P. Raskin [b,e], T. Pardoen [a,e]

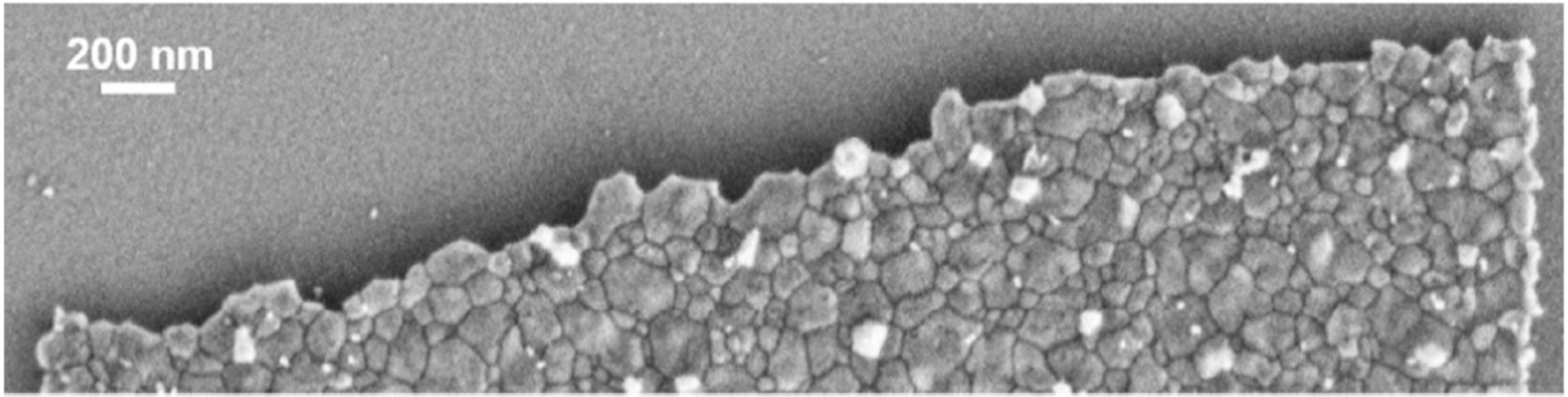




transgranular fracture in 240nm thick film



intergranular fracture in 40nm thick film

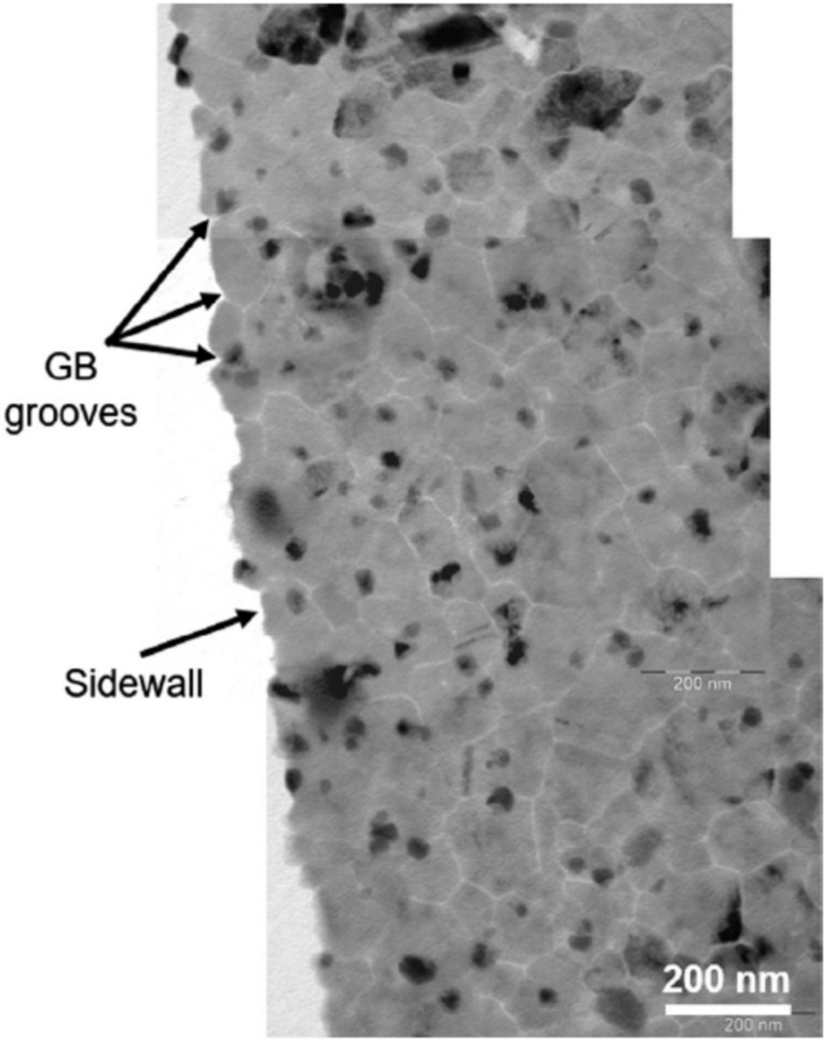


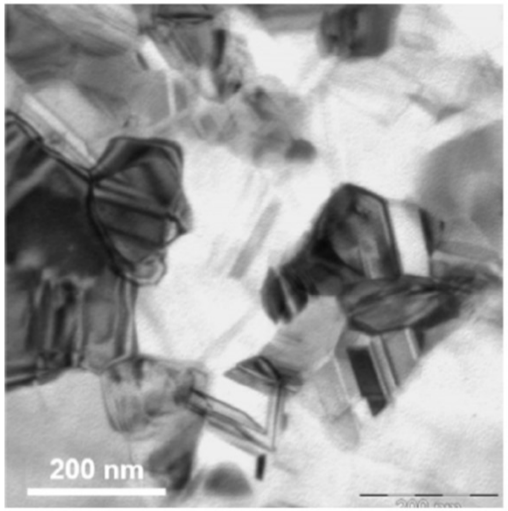
Why trans- versus inter- ?

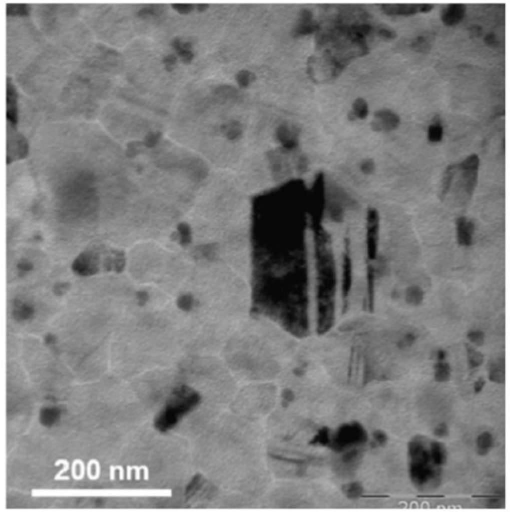
by ACOM-TEM

Thickness (nm)	HAGB (%)	CSLB (%)	Σ3 (%)	LAGB (%)
240	64.2	30.2	14.5	5.6
40	70.4	23.7	9.8	5.9

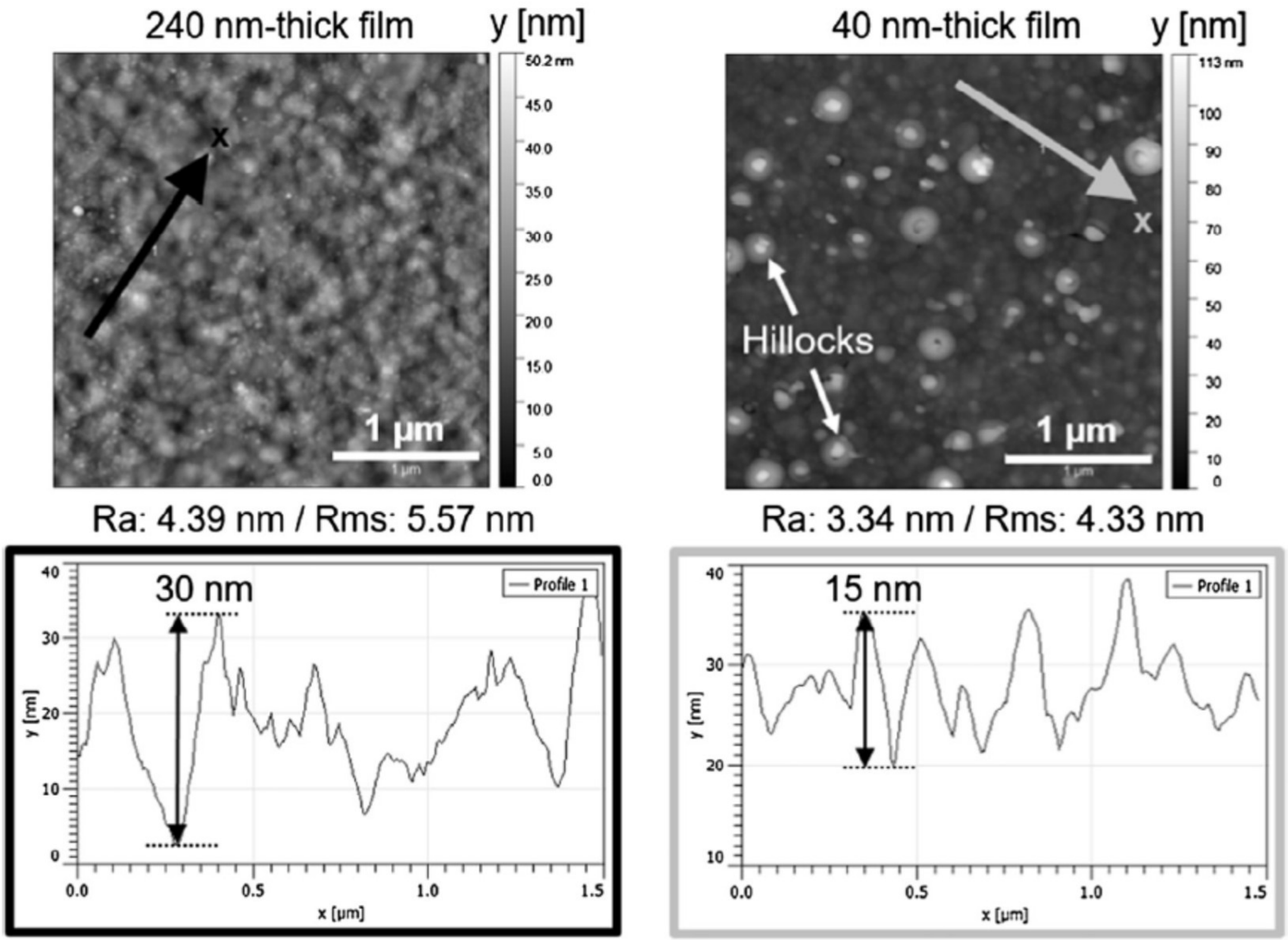
- Same distribution of GB character
- Similar crystallite size
- More twin lamellae in 240 nm thick
- GB grooves on both types of films







Why trans- versus inter- ?



We believe (!) that the larger relative amplitude of GB grooving in the 40nm thick film is the reason for the transition to intergranular fracture

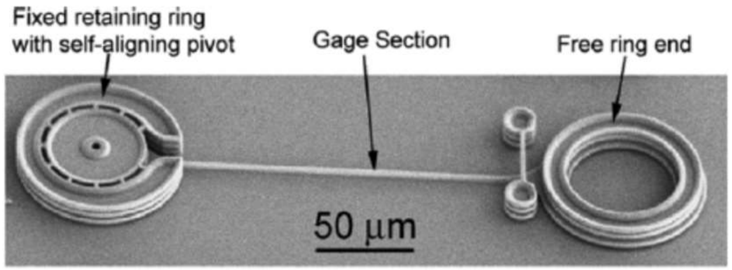
To go deeper on PolySi fracture, the advise is to consult the excellent studies performed at Sandia Laboratory

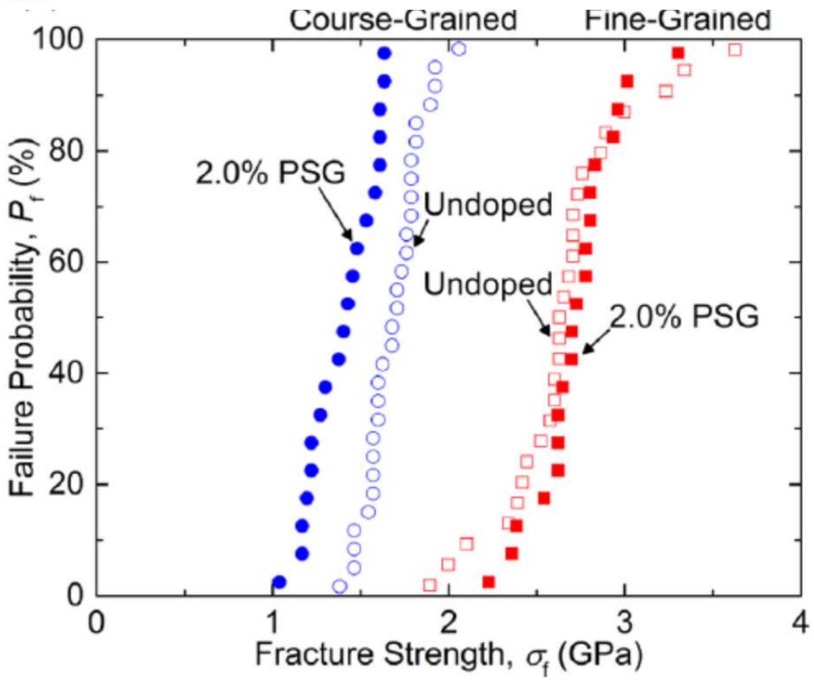
APPLIED PHYSICS REVIEWS **2**, 021303 (2015)

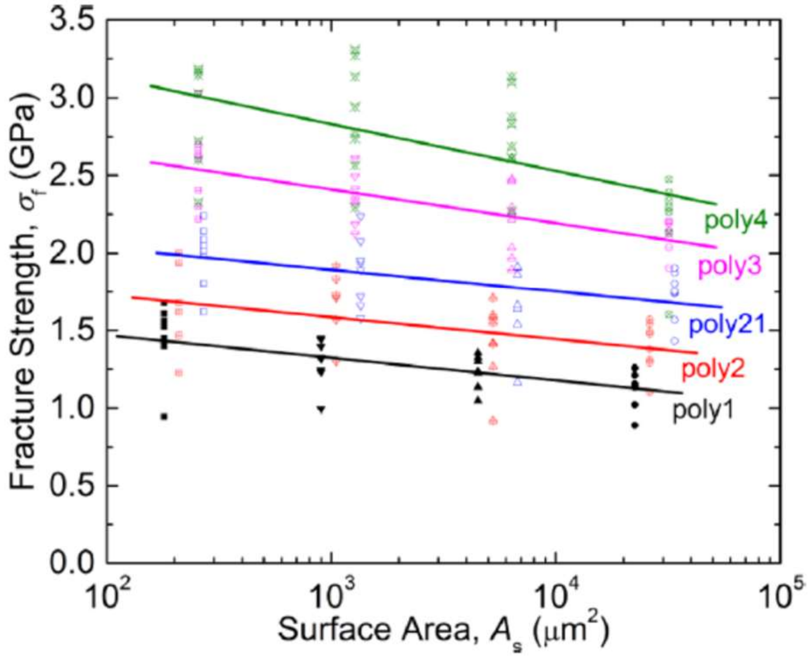
APPLIED PHYSICS REVIEWS

Fracture strength of micro- and nano-scale silicon components

Frank W. DelRio,[1,a)] Robert F. Cook,[2,b)] and Brad L. Boyce[3,c)]
[1]*Applied Chemicals and Materials Division, Material Measurement Laboratory, National Institute of Standards and Technology, Boulder, Colorado 80305, USA*
[2]*Materials Measurement Science Division, Material Measurement Laboratory, National Institute of Standards and Technology, Gaithersburg, Maryland 20899, USA*
[3]*Materials Science and Engineering Center, Sandia National Laboratories, Albuquerque, New Mexico 87185, USA*





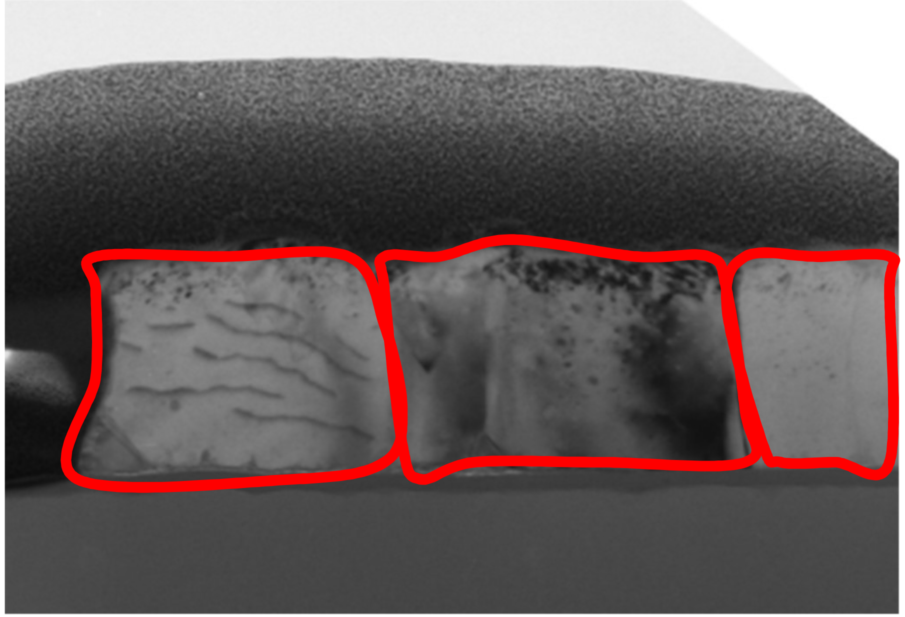


Example 2 : fracture strain of Al thin films

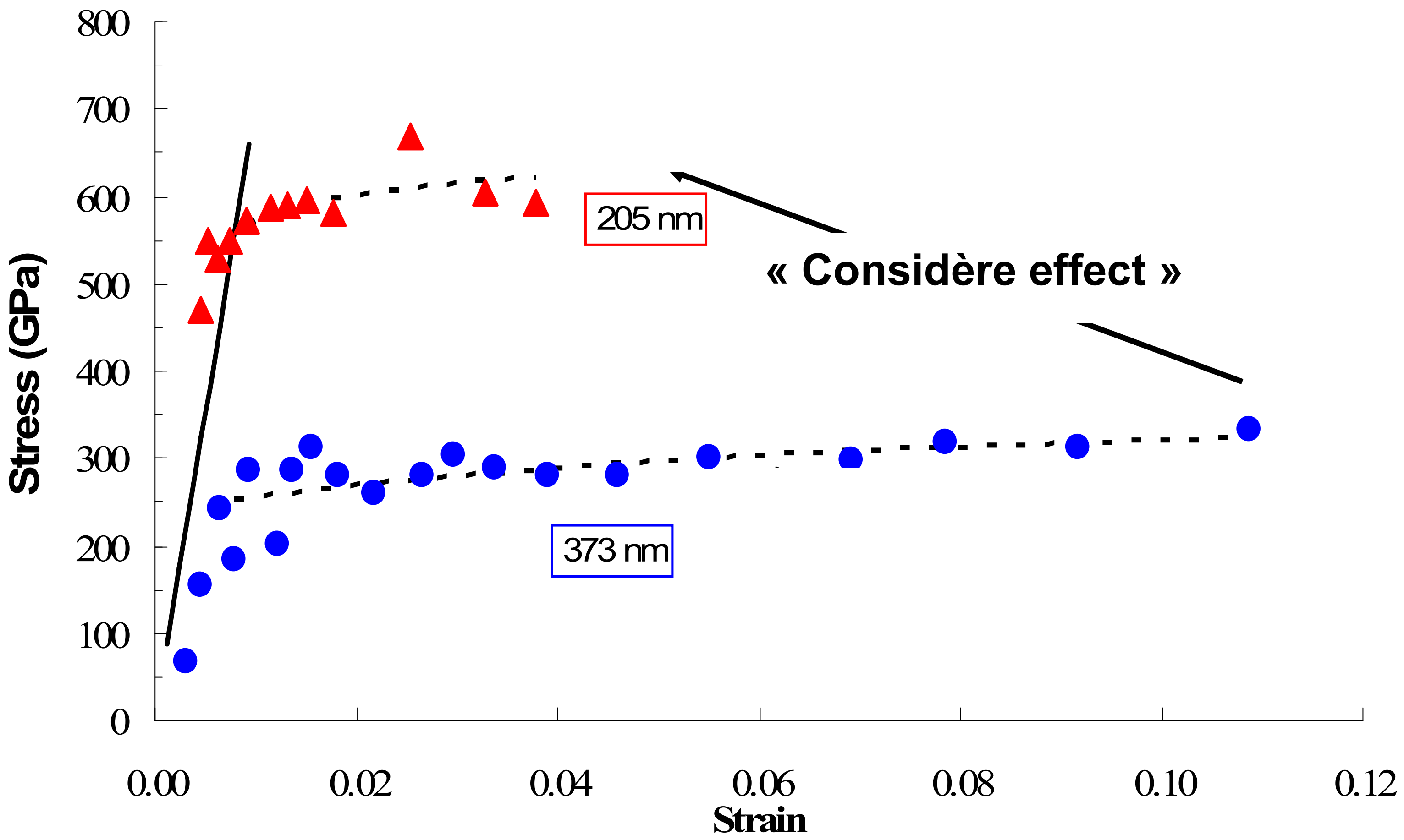
Example of Al films

Pure Al evaporated films
2 thicknesses = 205 and 373 nm, grain size ≈ 180 and 230 nm

AlSi1% evaporated films
thickness = 200 nm, grain size ≈ 200 nm

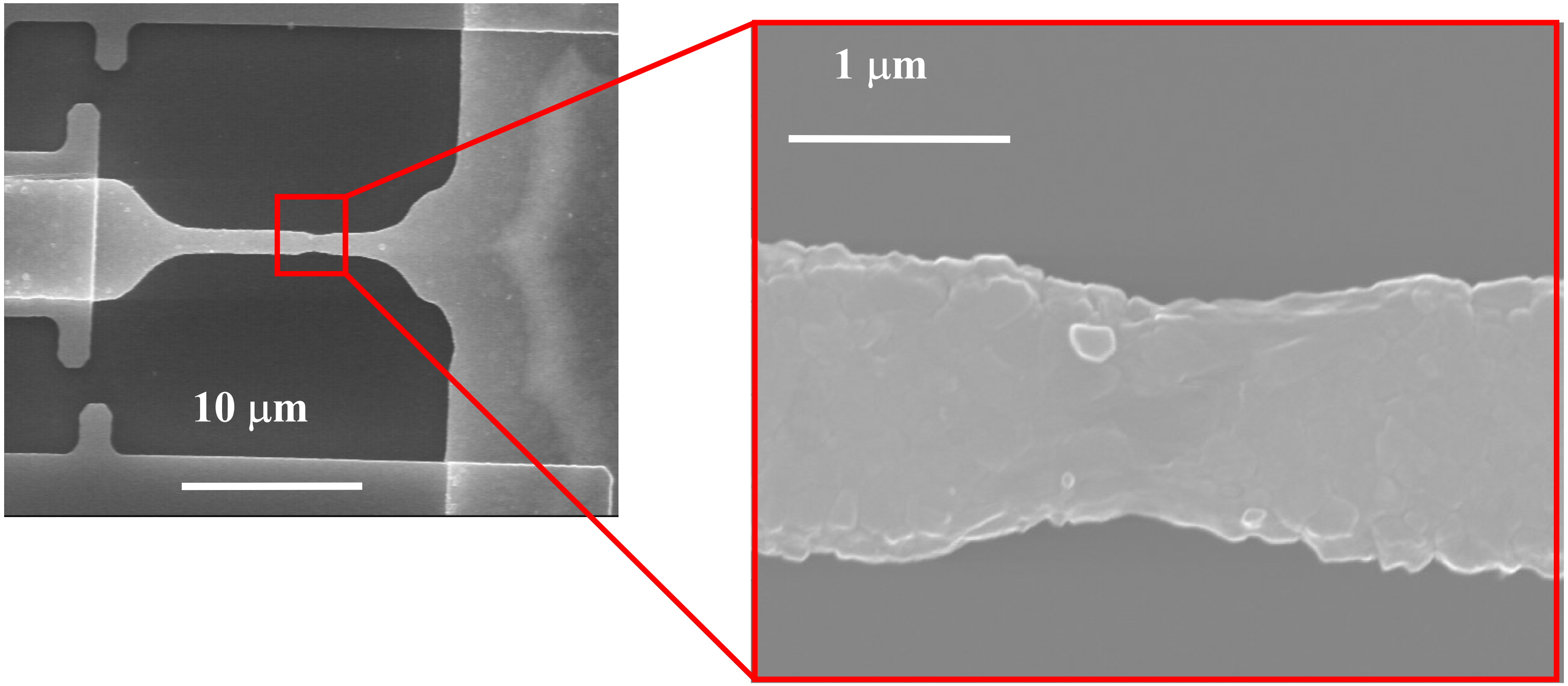


Example of Al films

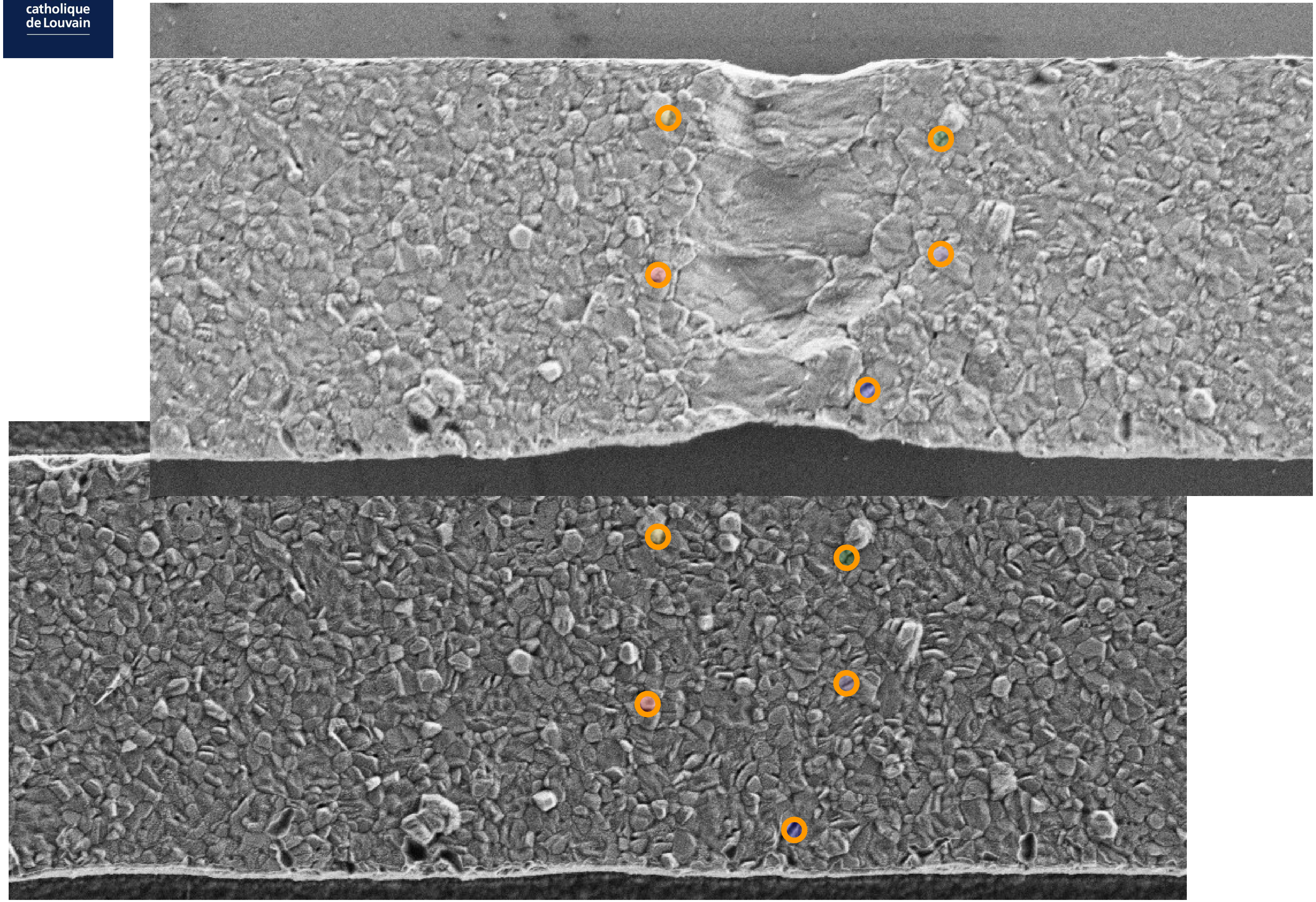


Clear evidences of stable necking

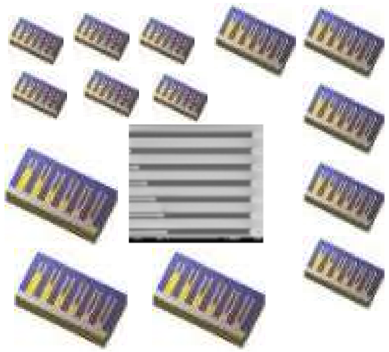
elongated neck !

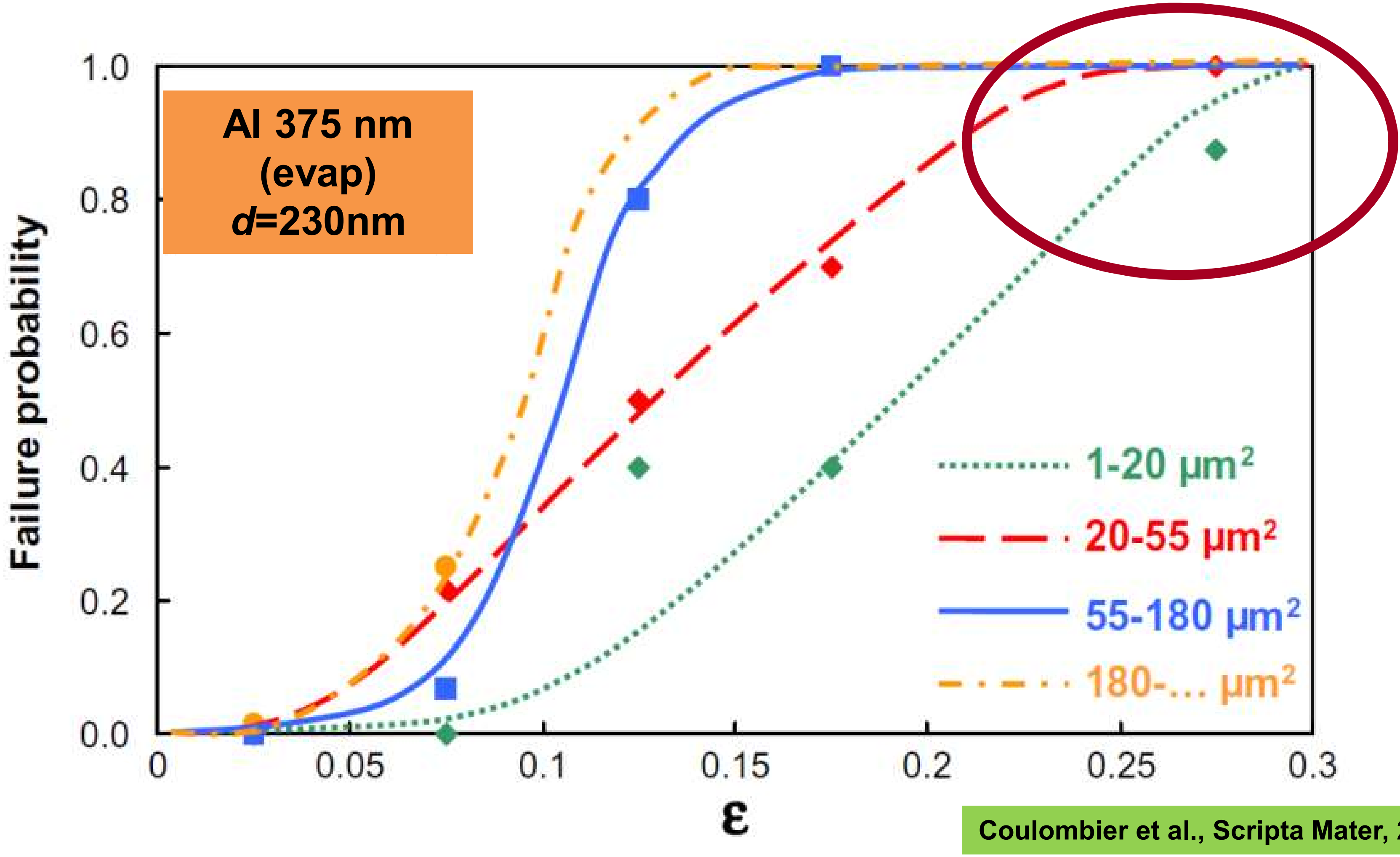


Large post necking ductility



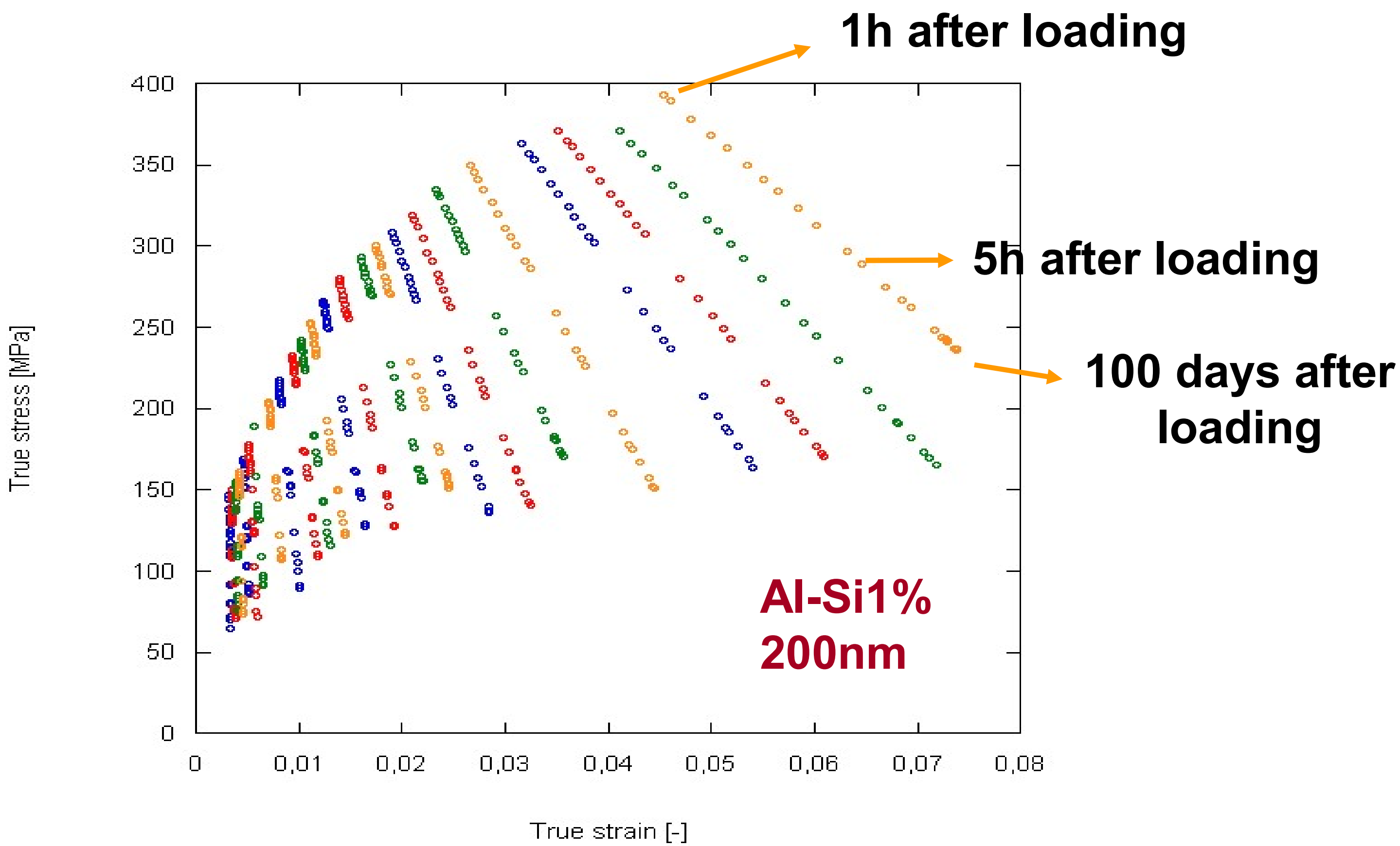
In some specimens, fracture strain near 30%





Coulombier et al., Scripta Mater, 2010

Relaxation tests on AlSi 1%



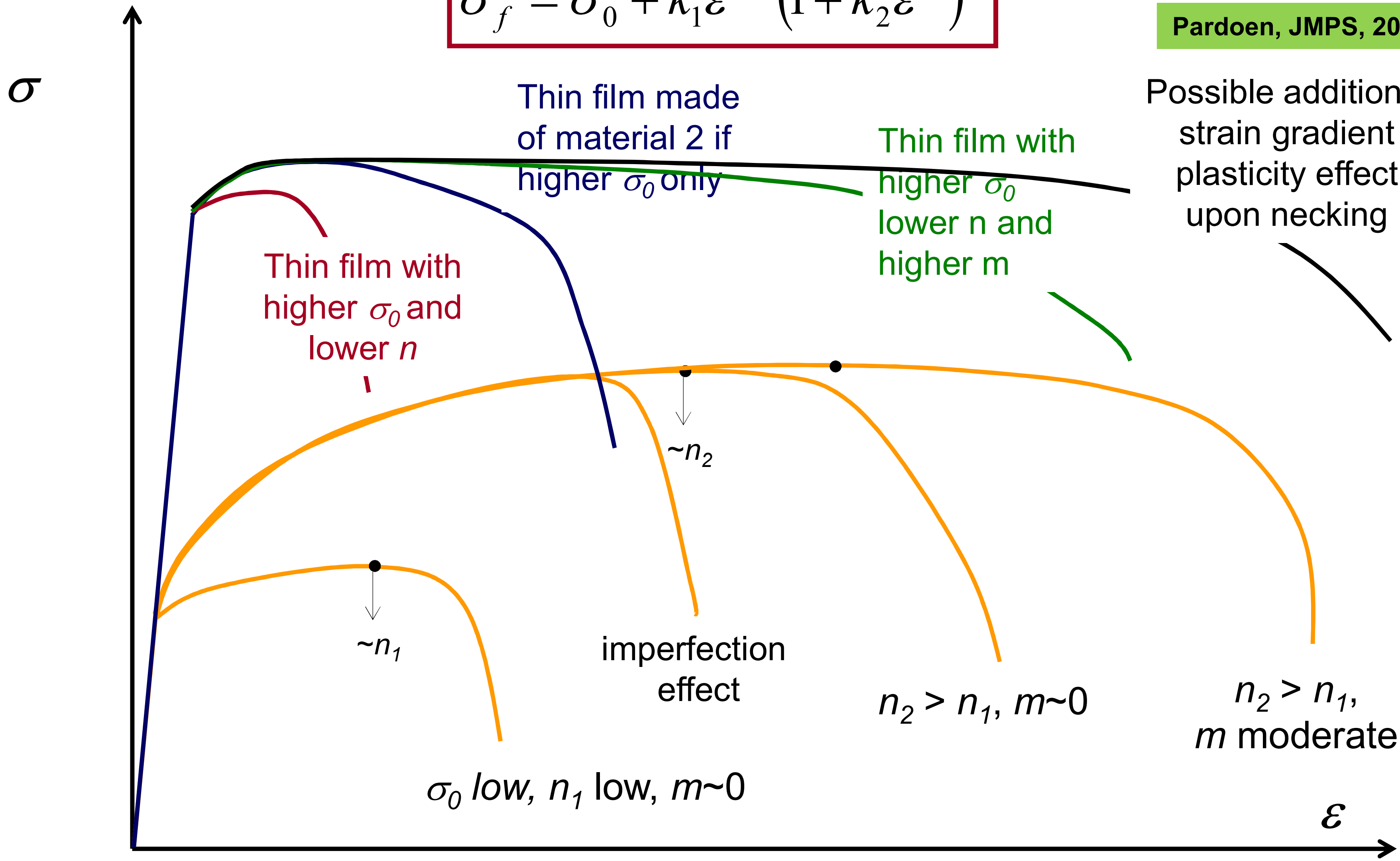
m ≈ 0.1 to 0.15
Even larger in pure Al (too fast to be measured)
(as explained by thermally activated deformation mechanisms, involving grain growth)

Mompiou et al. Acta Mater 2013

The big picture on necking

$$\sigma_f = \sigma_0 + k_1 {\varepsilon^p}^n \left(1 + k_2 \dot{\varepsilon}^p\right)^m$$

Pardoen, JMPS, 2014



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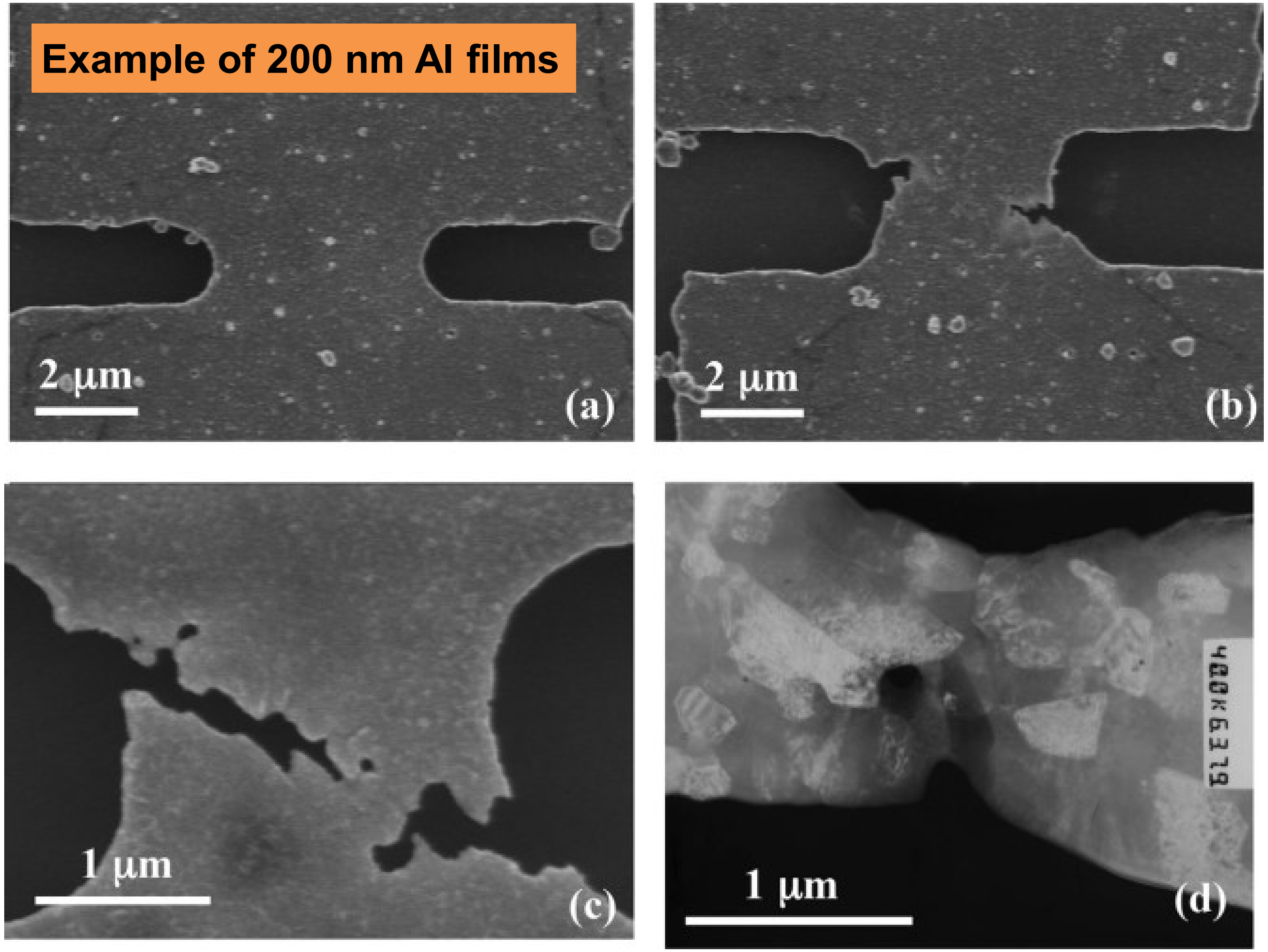
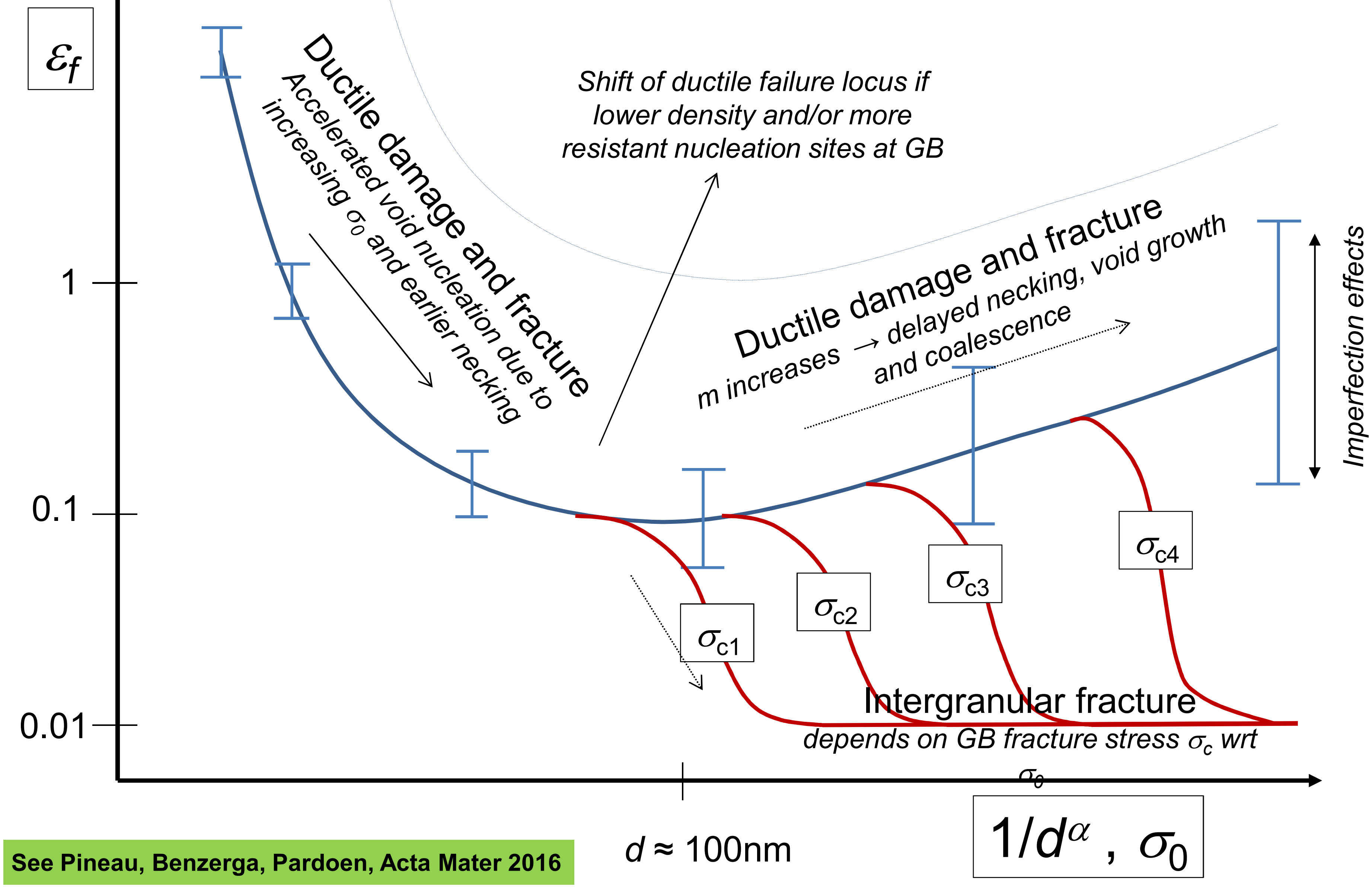
Metallic films fracture and nanowires by damage at GB



Big picture on the fracture metallic films



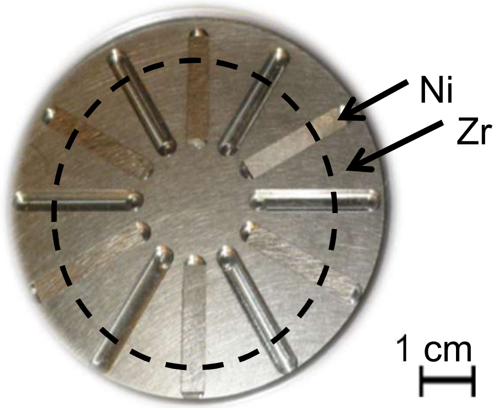
See Pineau, Benzerga, Pardoen, Acta Mater 2016

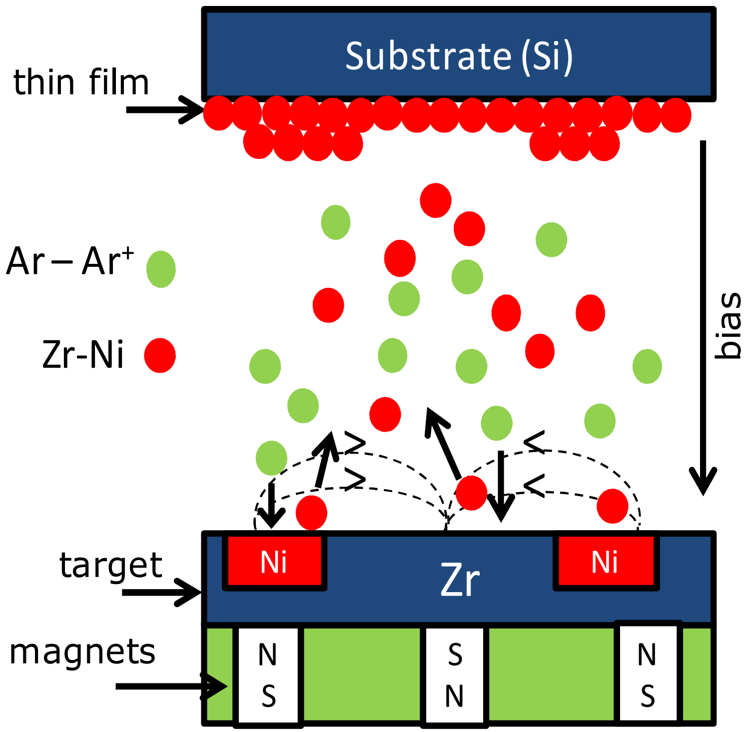
Example 3 : fracture of ZrNi metallic glass films

$Zr_{65}Ni_{35}$ films

DC-magnetron sputtering

at Plateforme Technologique Amont (PTA), Grenoble





Composition control

(Electron Problem Micro Analysis, EPMA)

- No impurities
- Uniform composition along the substrate

$Zr_{65}Ni_{35}$ (%at.) composition

Thickness control

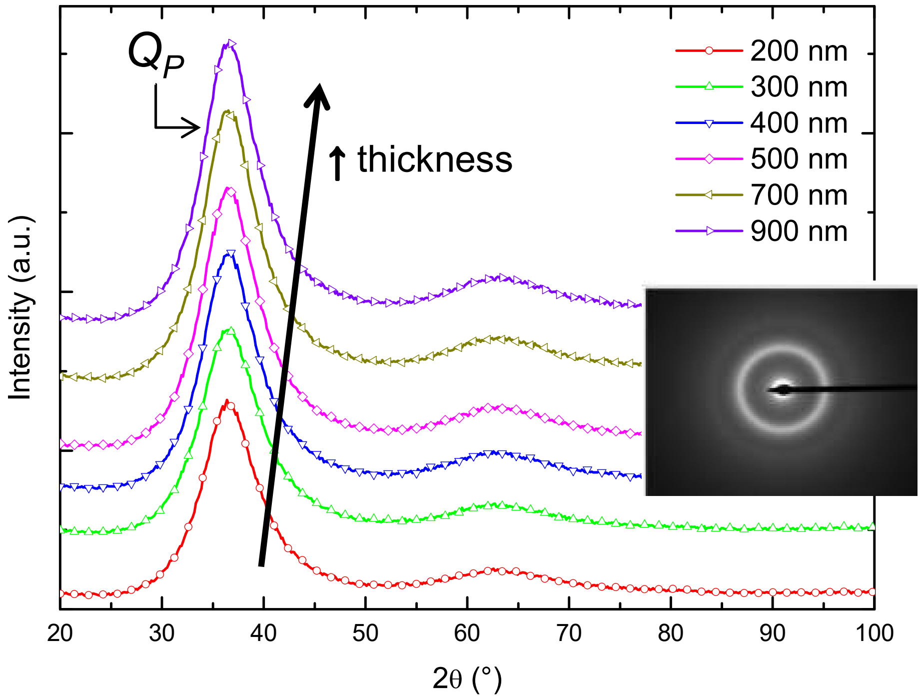
(cross-section SEM + mechanical profilometer)

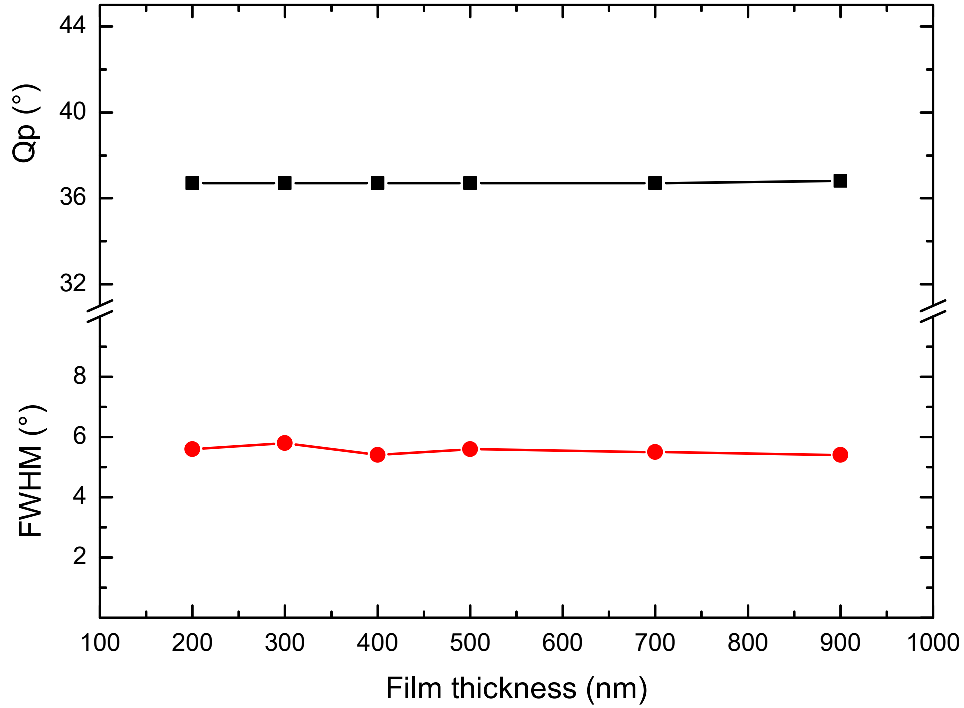
Linear growth rate ~ 1 nm/s

Thickness ranges from 200 to 900 nm

$Zr_{65}Ni_{35}$ films

DC-magnetron sputtered with thickness between 200 and 900 nm



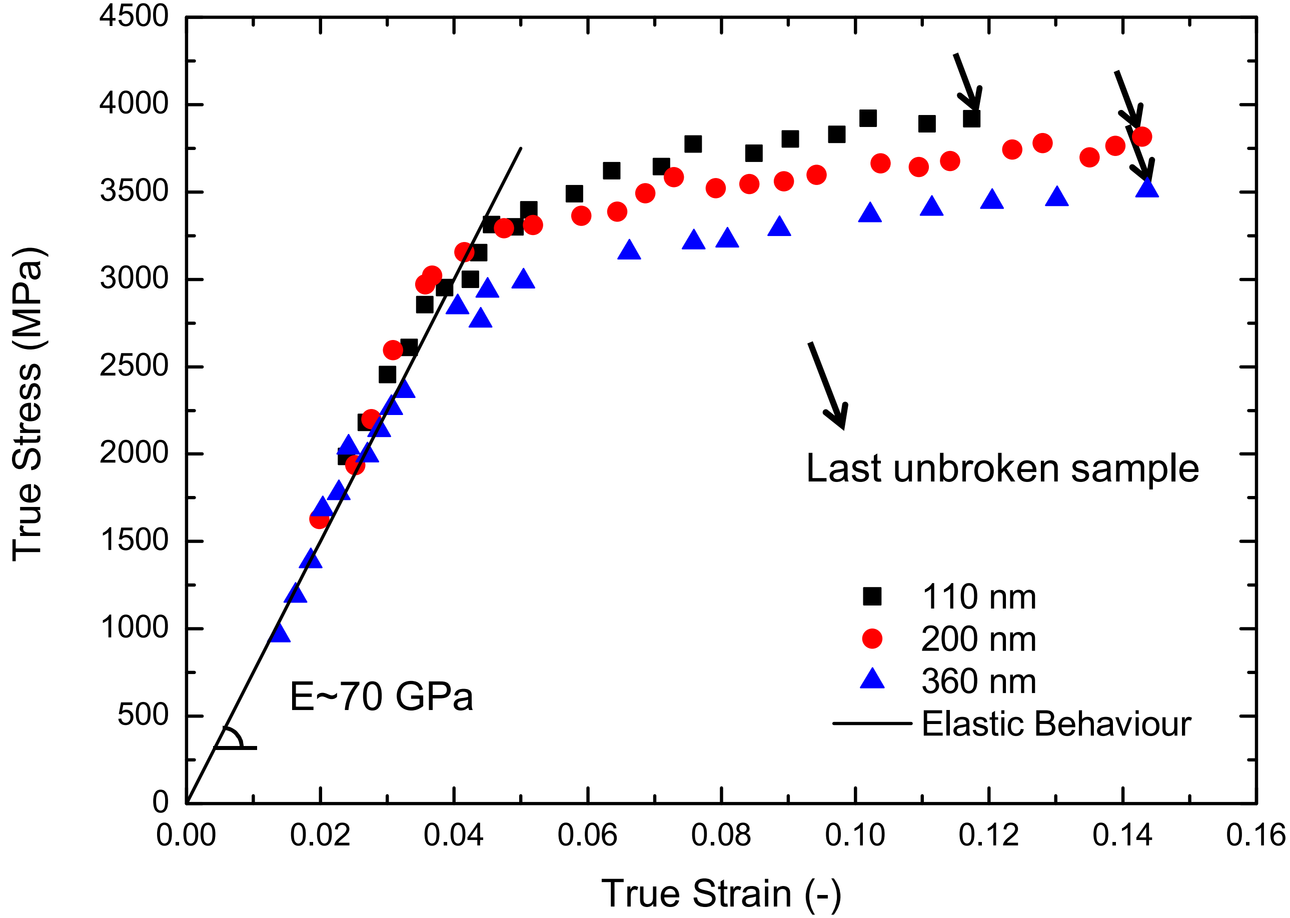


Amorphous structure (presence of diffuse halos)

No peak shift (Q_P) and same FWHM for different thicknesses
➔ **atomic structure independent of thickness**

M. Ghidelli *et al.*, J. Alloys Compd., 615, 348-351 (2014)

Uniaxial tension response of 360 nm-thick $Zr_{65}Ni_{35}$ film



Actuator thickness
160 nm

Actuator length
variable

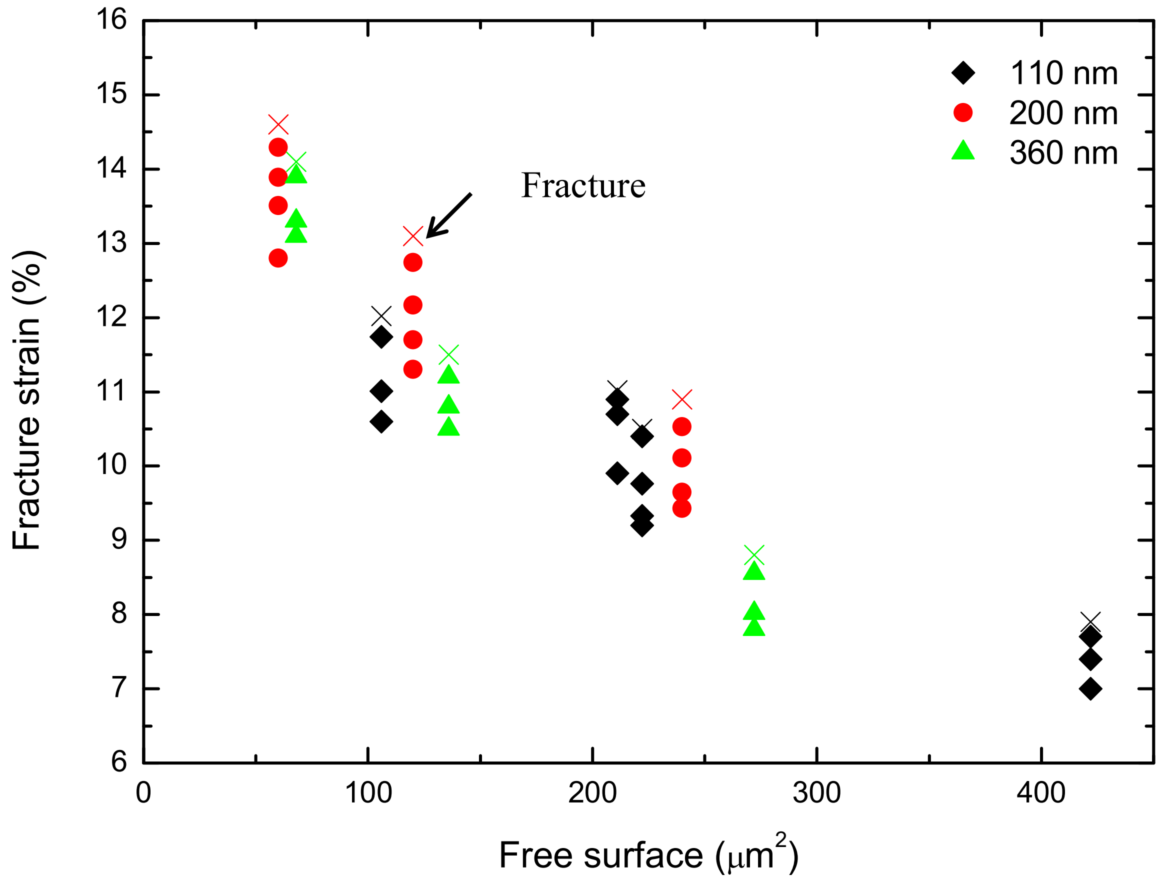
Actuator width
15 µm

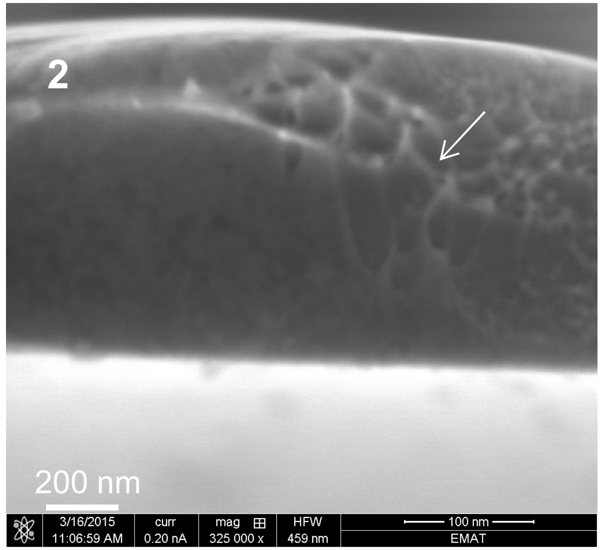
Specimen width
1 or 2 µm

Specimen length
25 µm

Elastic behavior up to 4% with E ~ 70 GPa (OK Brillouin spectro)
Large fracture strain up to 15% (decreasing with increasing length)
Yield stress around 2900 MPa

Fracture strain decreases with increasing specimen free surface

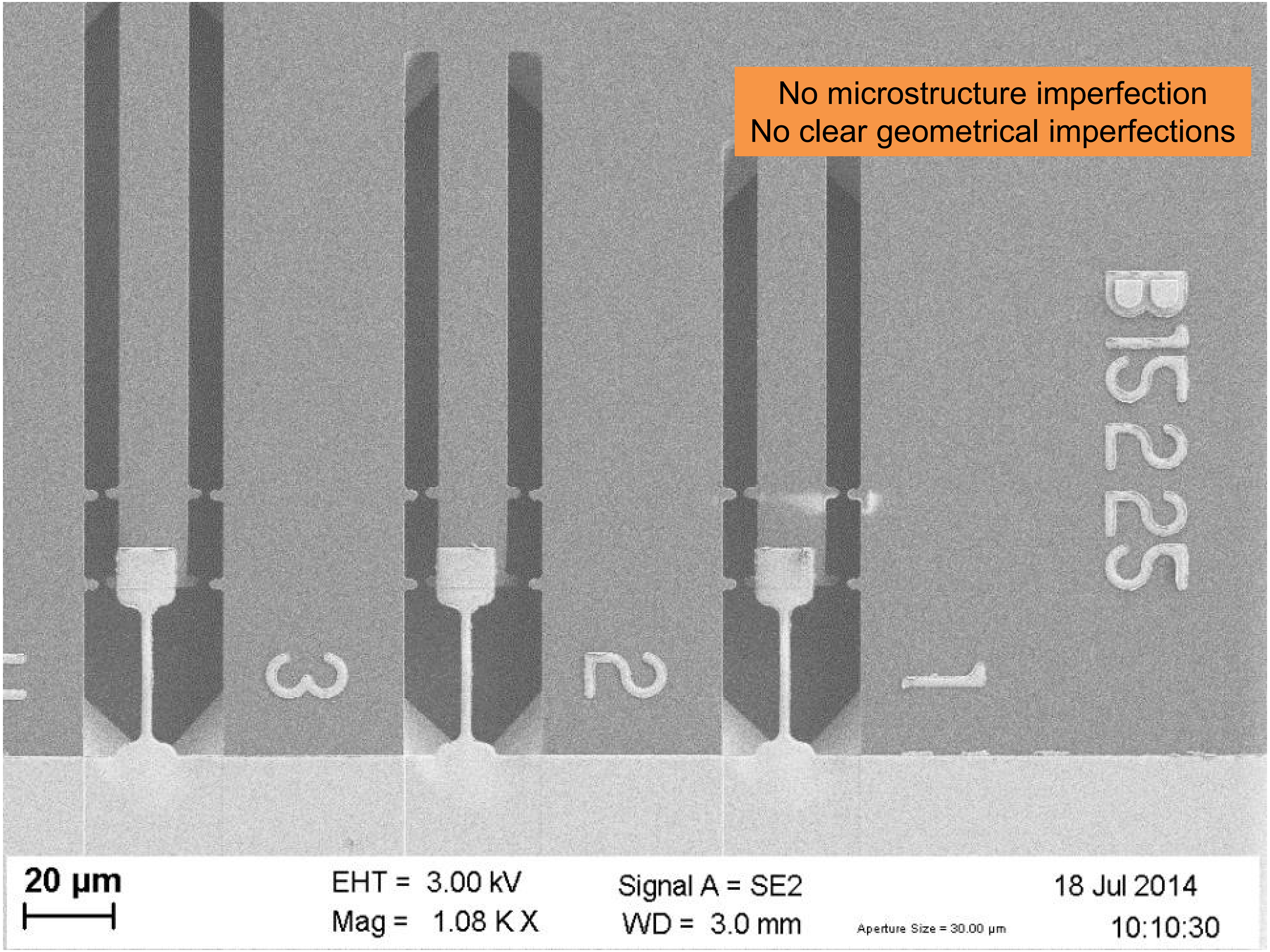


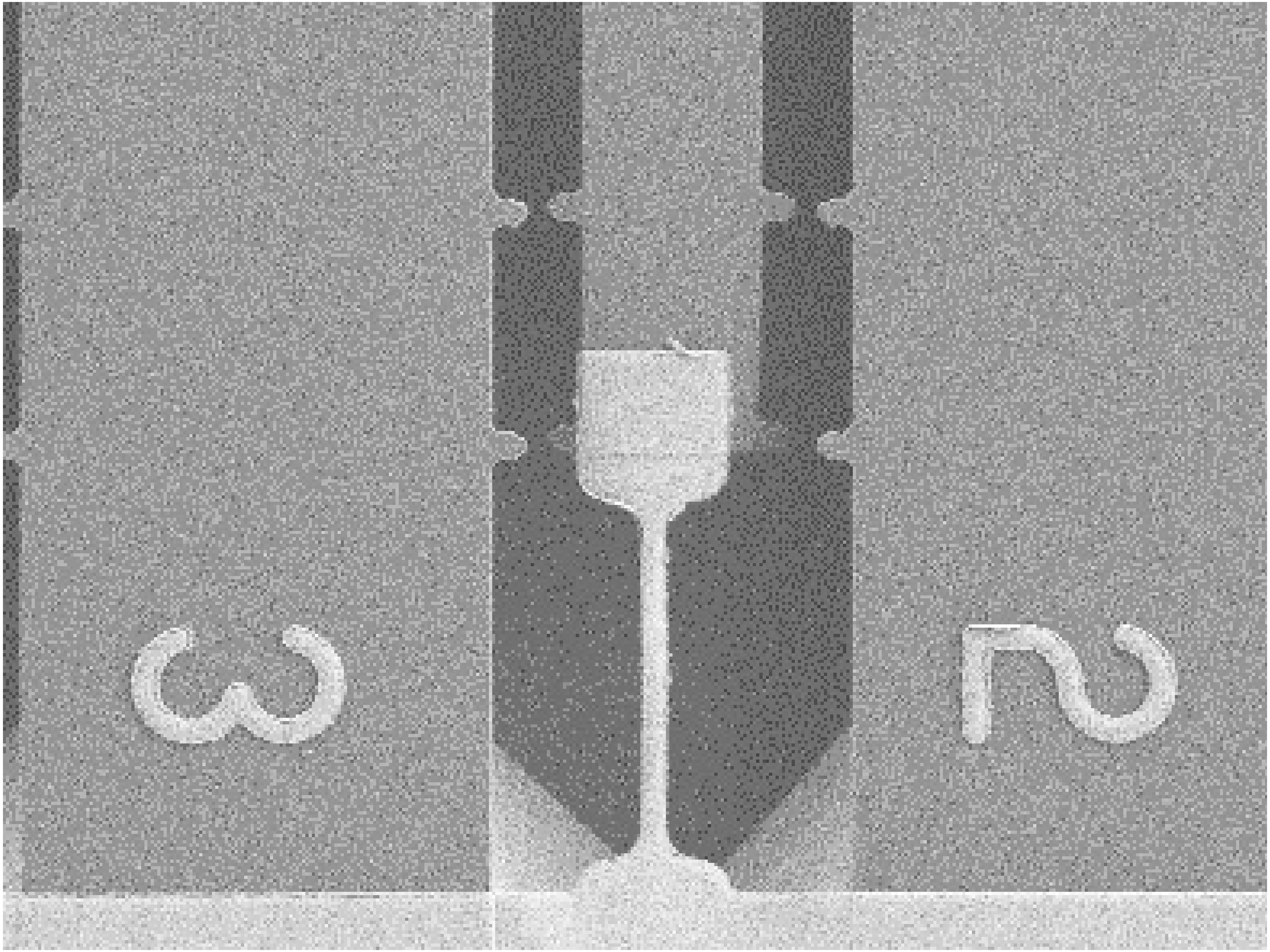


TEM by dr H. Idrissi

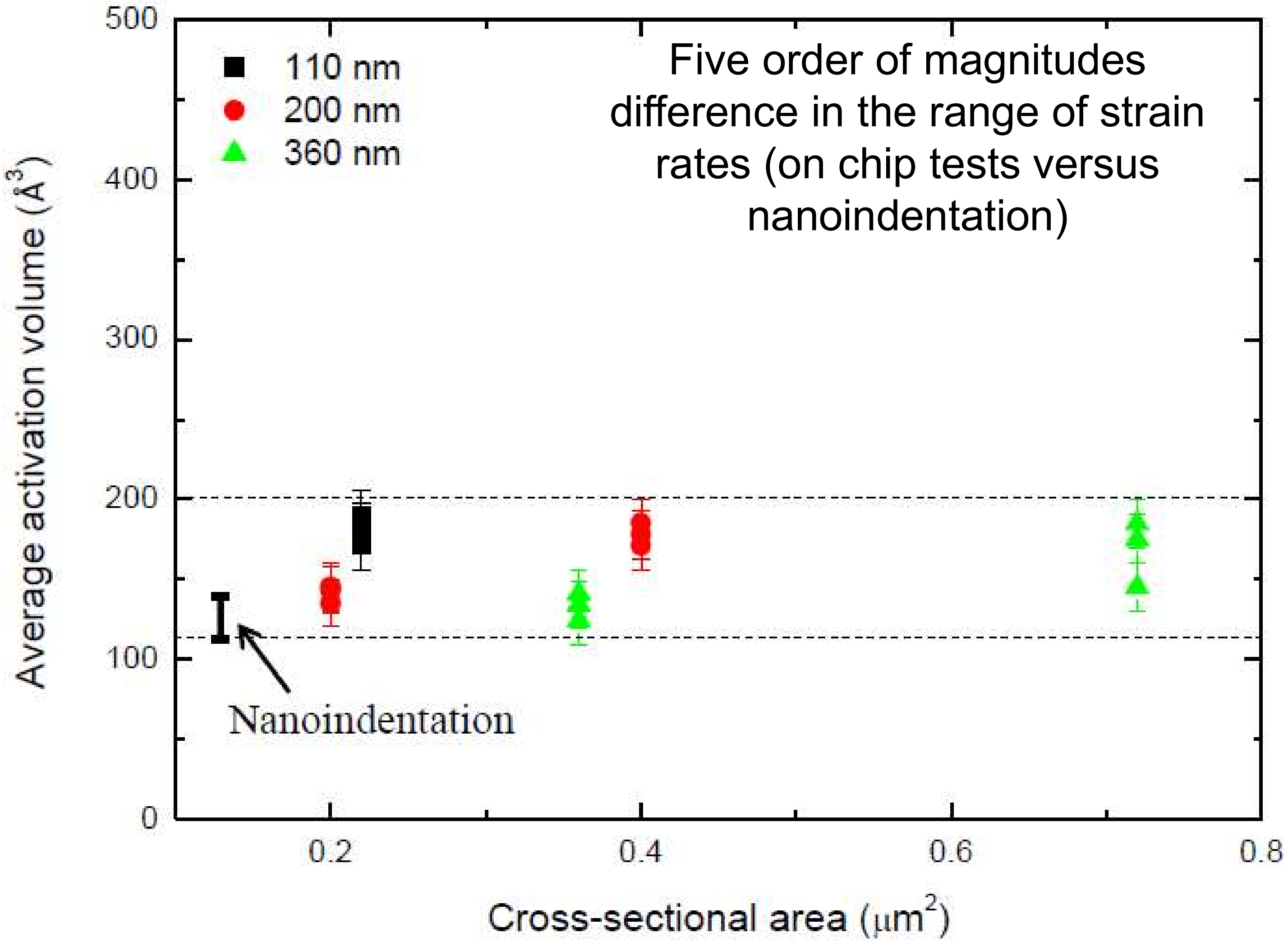
TEM shows no evidence of shear bands
Fracture surface involve flat regions and corrugations (dimples)

No microstructure imperfection
No clear geometrical imperfections



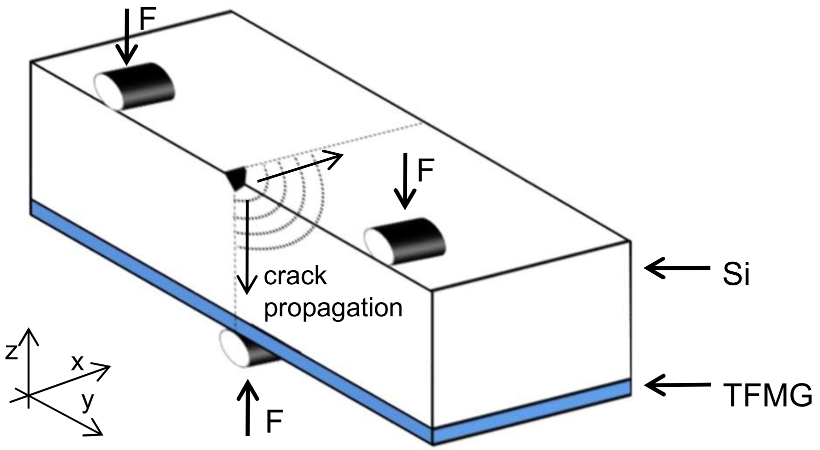


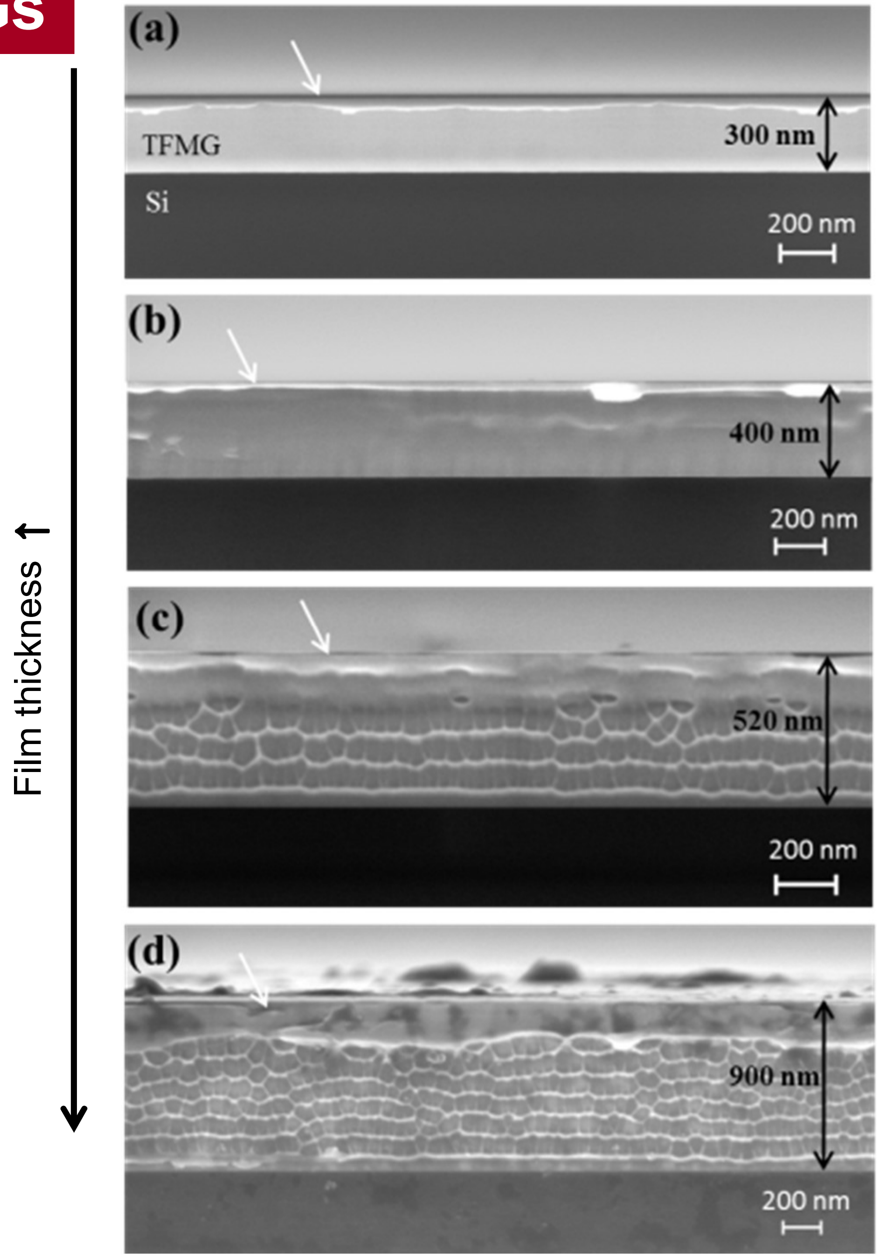
Confirmation of the high rate sensitivity measured by nanoindentation



Fracture of $Zr_{65}Ni_{35}$ TFMGs

Method: crack propagation from substrate + SEM observation



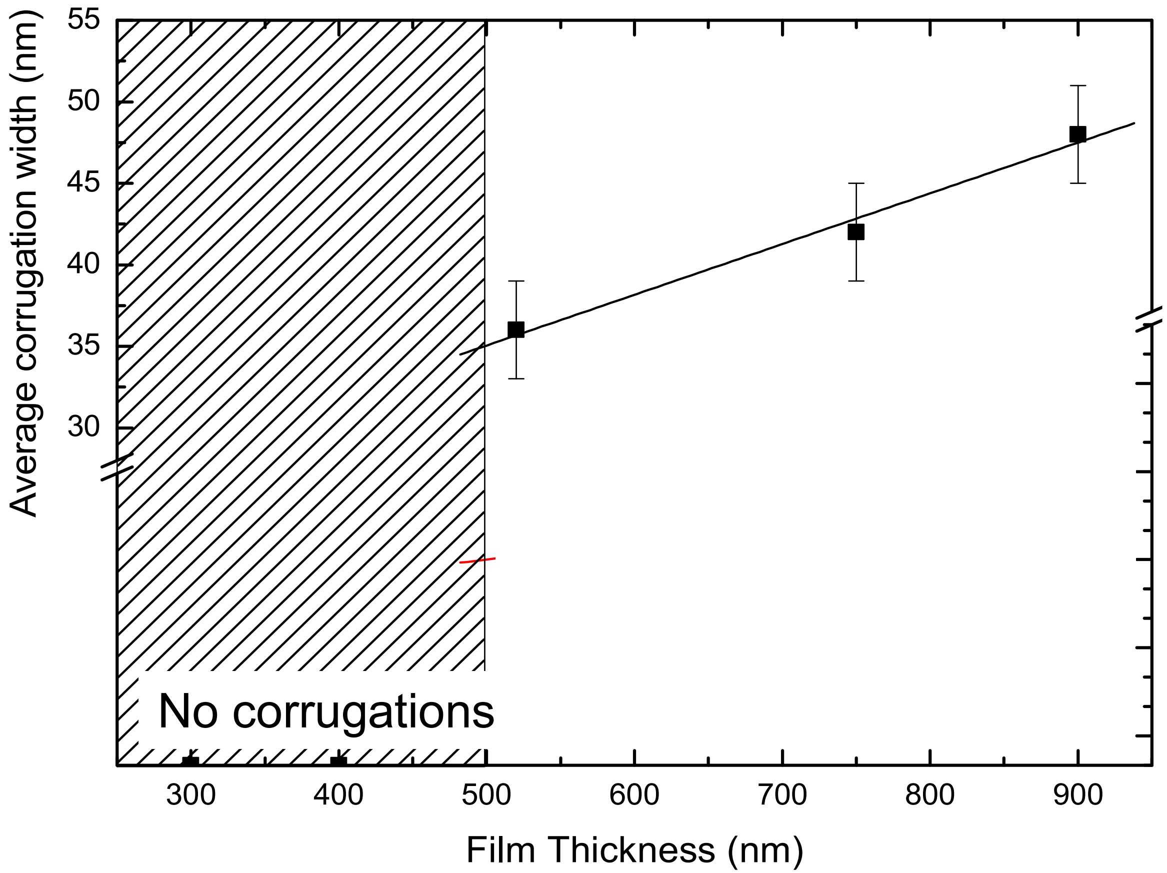


Corrugation pattern formation for thickness ≥ 500 nm

Presence of a folded layer for all thicknesses

M. Ghidelli *et al.*, Scripta Materialia, 89 (2014) 9

Fracture of $Zr_{65}Ni_{35}$ TFMGs



Fracture toughness estimated by

$$K_c = \sigma_y \sqrt{40w}$$

K_c → Fracture toughness

σ_y → Yield strength

w → corrugation width

Xi et al., Phys. Rev. Lett. *94*, 125510 (2005)

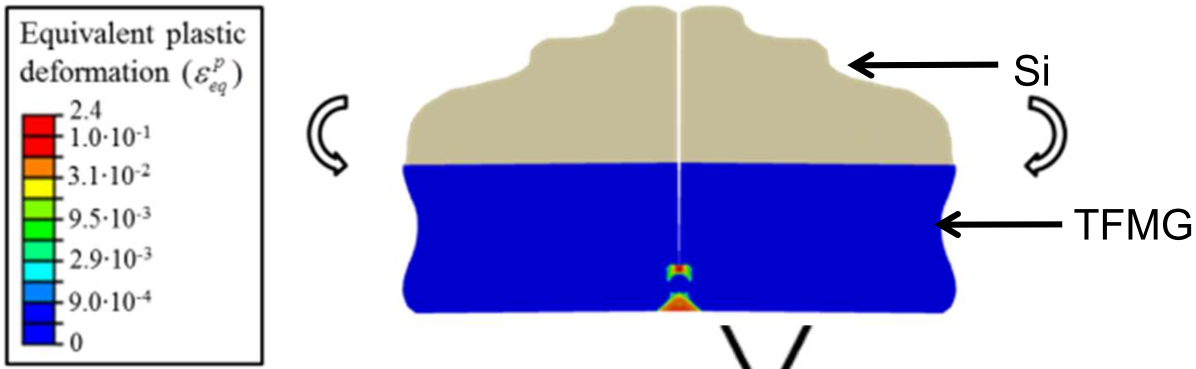
Corrugation width ↑ when thickness ↑

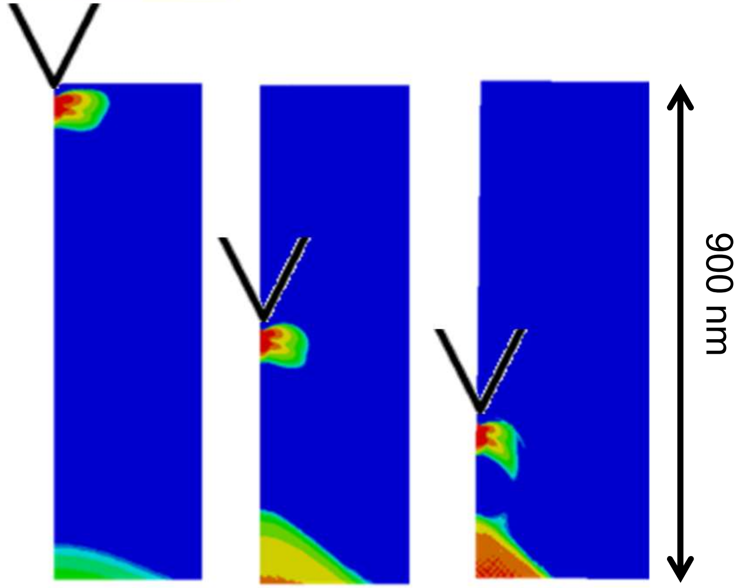
Corrugation size << bulk values ~ mm (Xi *et al.* PRL 2005)

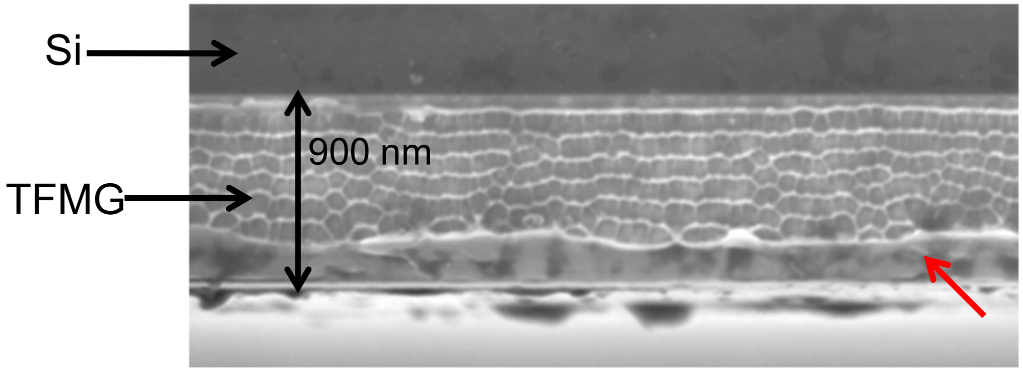
Fracture toughness (2 to 4 MPa m$^{1/2}$ << bulk values (K_c ~ 50 MPa m$^{1/2}$)

Fracture of $Zr_{65}Ni_{35}$ TFMGs

Finite element simulations of static crack @ 900 nm film





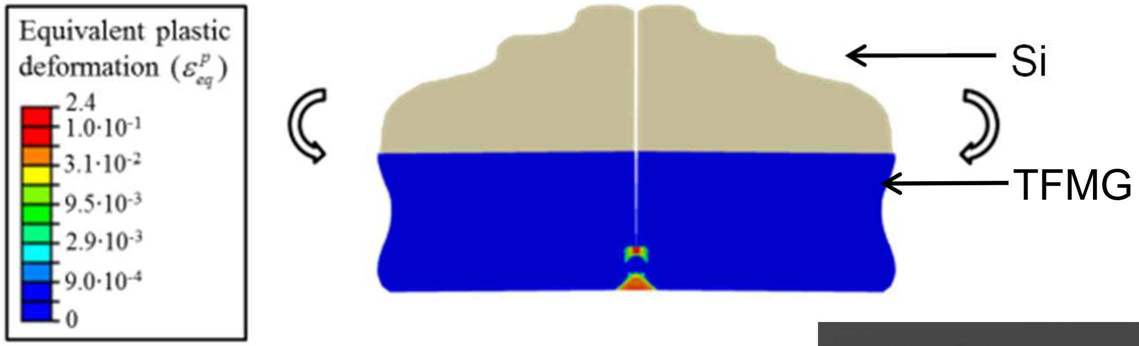


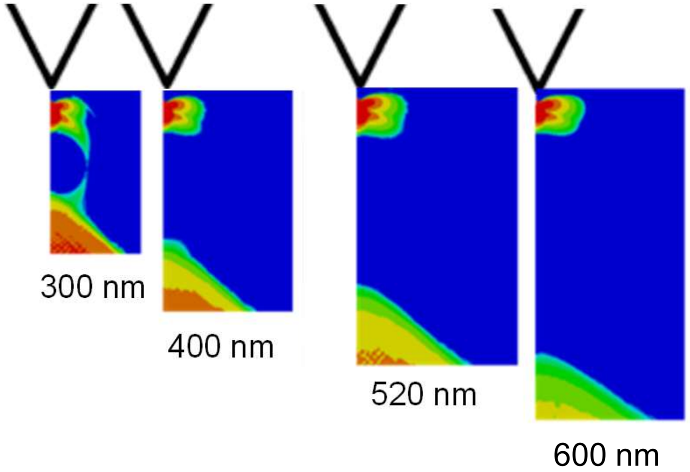
Corrugation size corresponding to finite strain zone

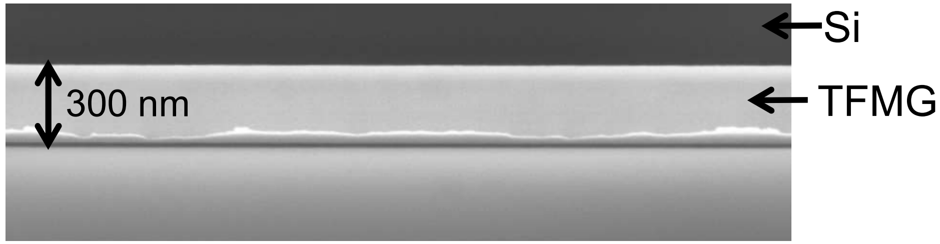
Plastic collapse **➔ folded layer**

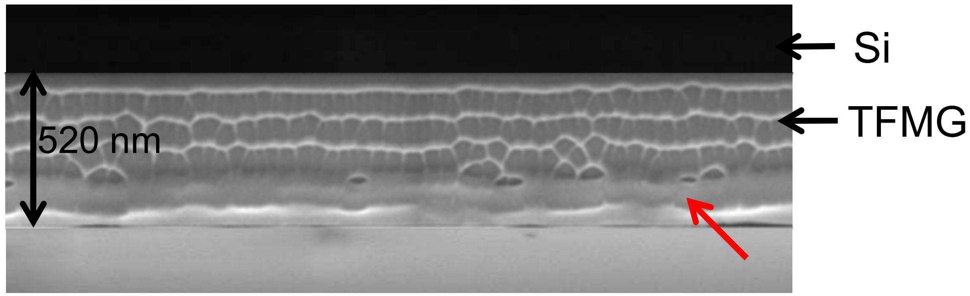
Small toughness (K_c = 2.2 MPa $m^{1/2}$) ➔ confinement effect

Fracture of $Zr_{65}Ni_{35}$ TFMGs







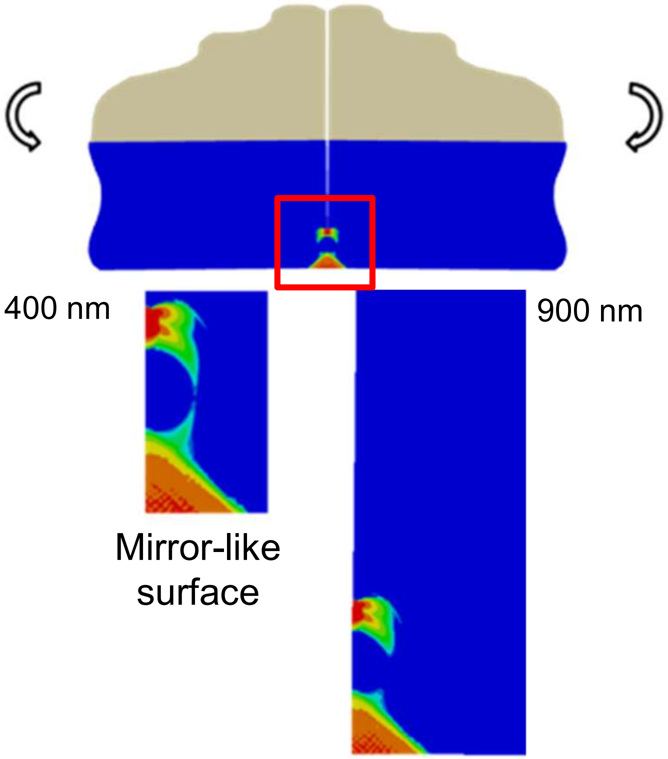


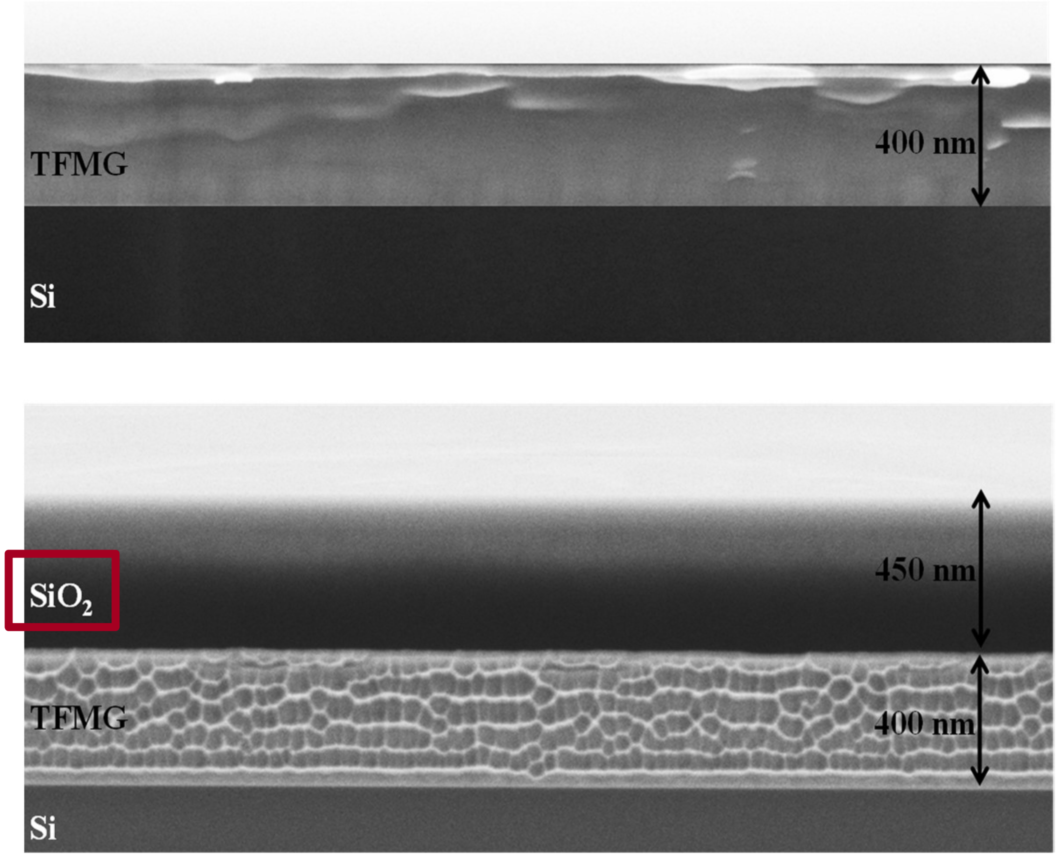
Plastic collapse **for 300 and 400 nm-thick film**
➔ mirror-like surface

M. Ghidelli *et al.*, Acta Materialia (2015)

Fracture of $Zr_{65}Ni_{35}$ TFMGs

Is it possible to avoid the *plastic collapse* for thicknesses < 500 nm?
…. Add a cap layer





Compressive plastic zone shifting into the SiO_2 layer and no folded layer

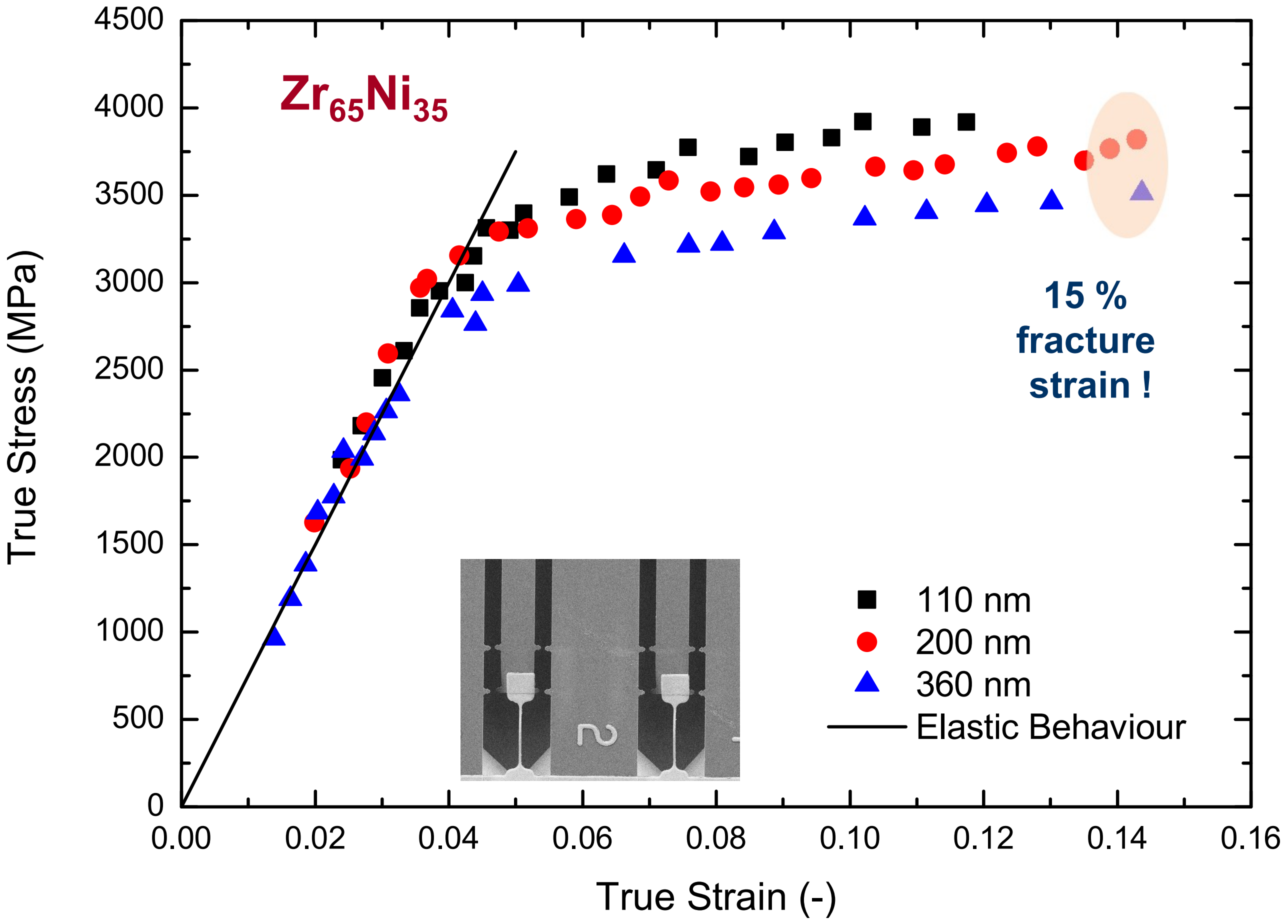
M. Ghidelli *et al.*, Acta Materialia (2015)

UCL Université catholique de Louvain

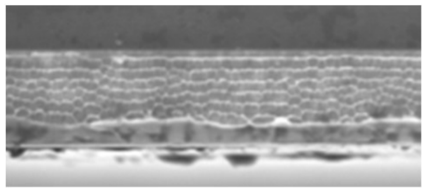
Ultra-tough metallic glasses

with UCL on-chip test laboratory

Metallic glasses are wonderful materials except for their brittleness



Can we learn from this discovery to make ductile-tough metallic glasses ?



Ph. D. thesis M. Ghidelli, 2015
INPG + UCL
e.g. Ghidelli *et al.,* Acta Mater 2015

UCL Université catholique de Louvain

Outline

1. **Introduction**

2. **Fracture of films on substrates**
   - test methods and extraction of *G*
   - example 1 : CrN on polymer (indentation)
   - example 2 : SiN on polymer (subcritical crack growth)
   - example 3 : Au on polymer (for flexible electronics)

3. **Fracture of freestanding films**
   - Test methods for measuring the fracture strength - strain
   - fracture strength of brittle films
   - fracture strain of ductile films
   - fracture toughness

How to characterize the fracture resistance of thin freestanding films?

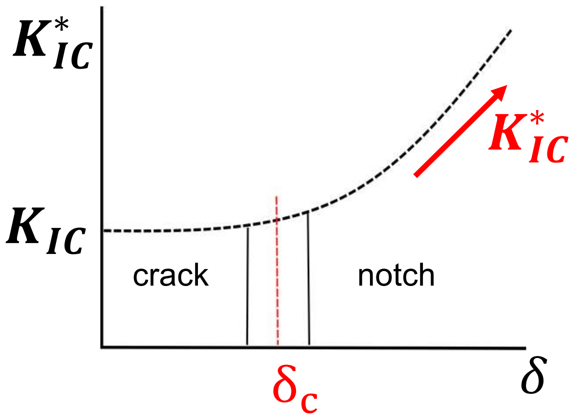
Freestanding configurations - challenges

- **Initial crack tip opening displacement** must be smaller than the critical crack tip opening displacement for valid fracture mechanics test

$$\delta_c \approx \frac{G_c}{\sigma_0}$$

for G_c = 1J/m^2 & σ_0 = 5 GPa, $\delta_c \approx$ 0.2 nm

for G_c = 10J/m^2 & σ_0 = 0.2 GPa, $\delta_c \approx$ 50 nm



- **Transfer of films without damaging**

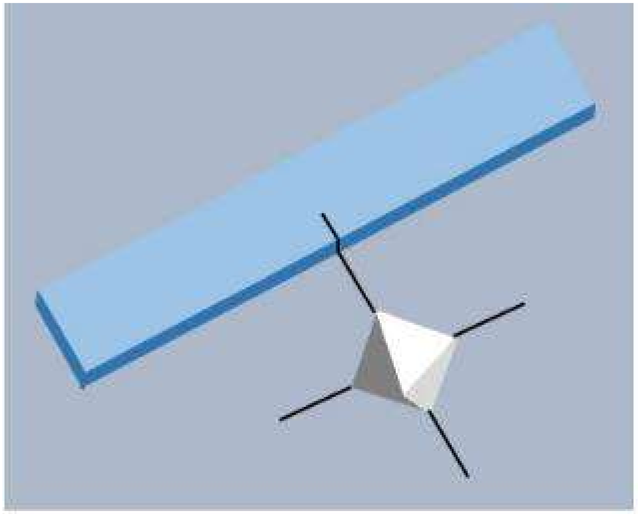
- **Clamping**

- **Detecting cracking** initiation and crack growth

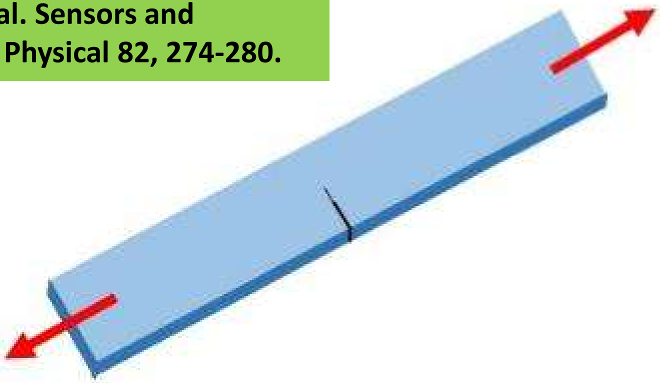
- **Measure extremely small loads**

- **Generate statistically representative data**

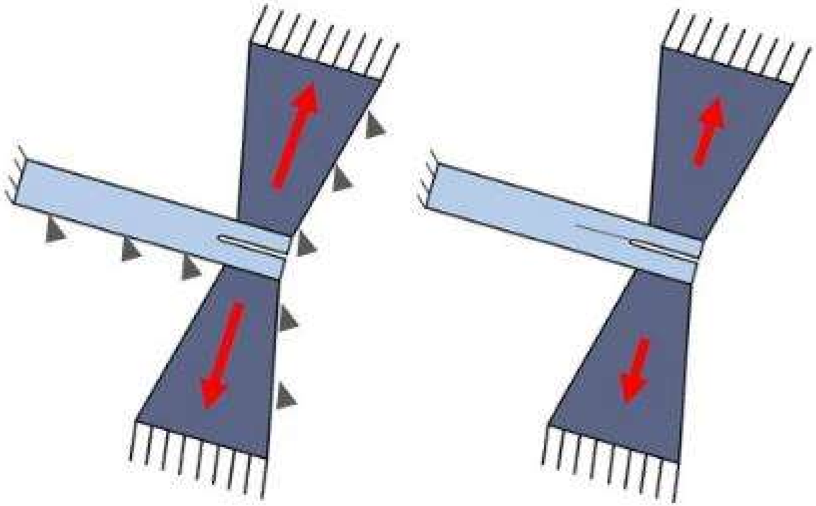
Two methods with valid cracks



(1) pre-crack by nanoindentation, release, pull in tension with microdevice, determine cracking initiation



Kahn, H., et al. Sensors and Actuators A: Physical 82, 274-280.



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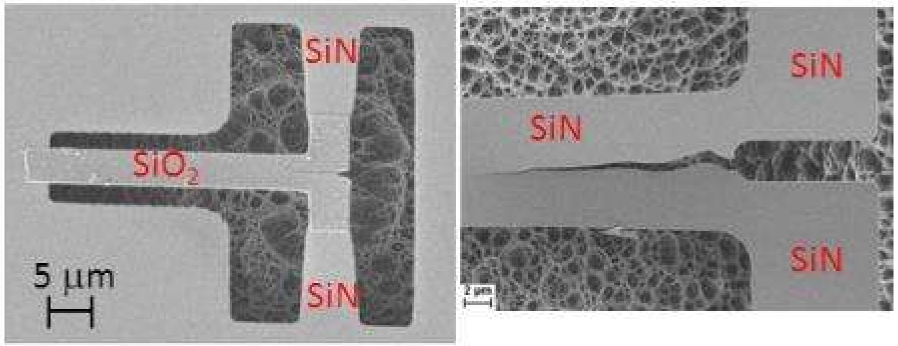
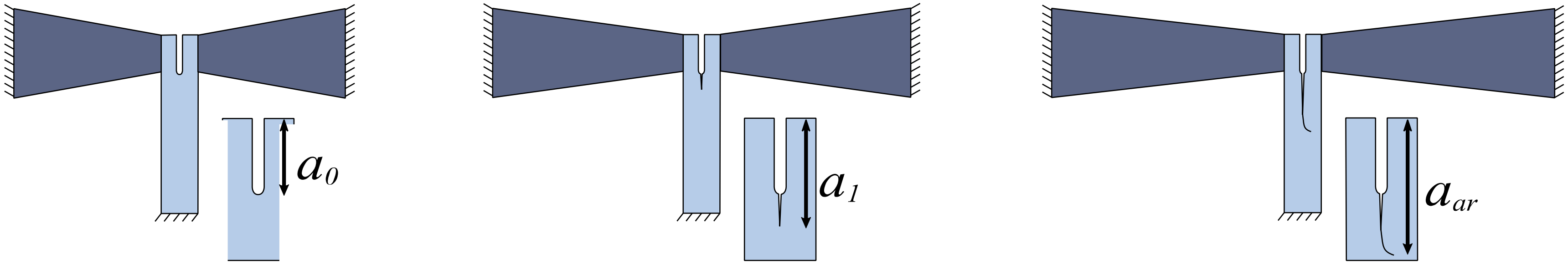
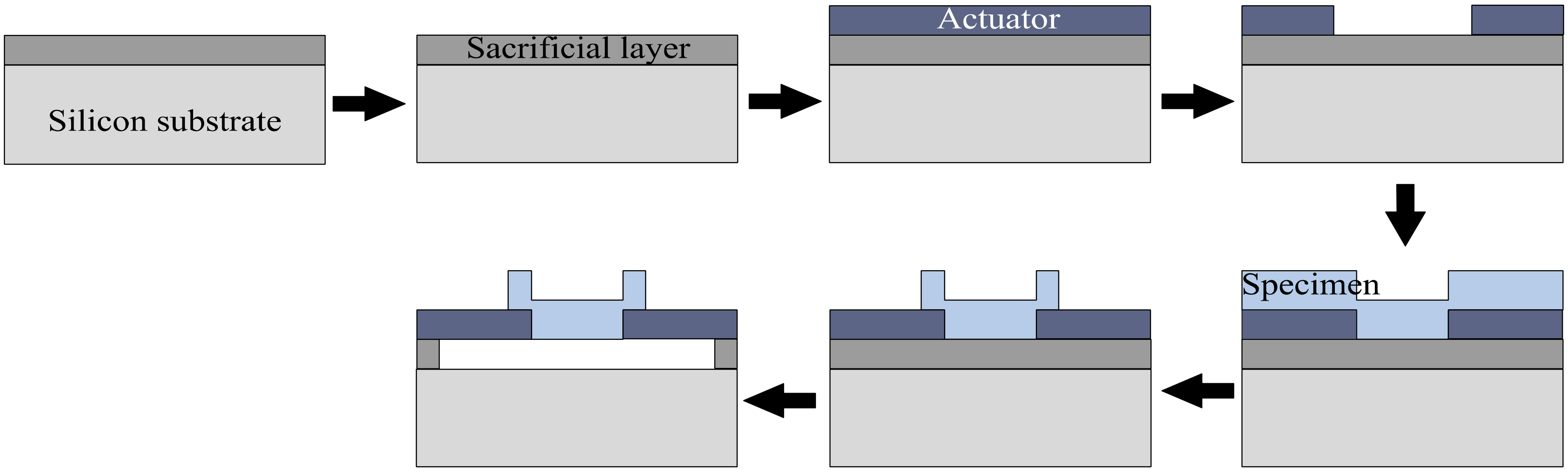
(2) notched specimen, internal stressed actuator, release, cracking and arrest, measure final crack length



Extension to crack on chip configuration

Crack arrest





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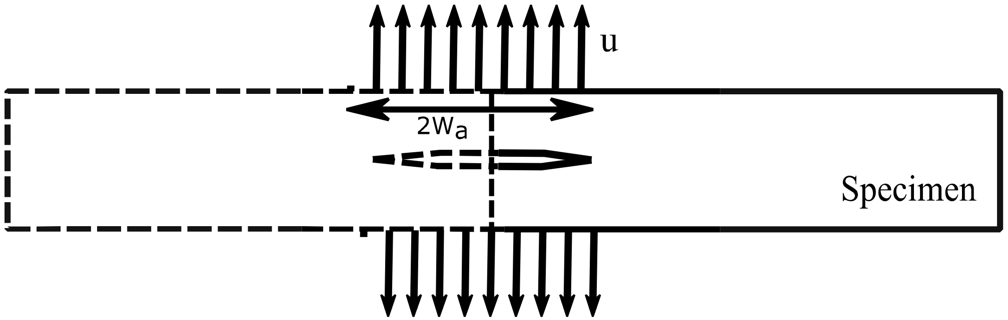
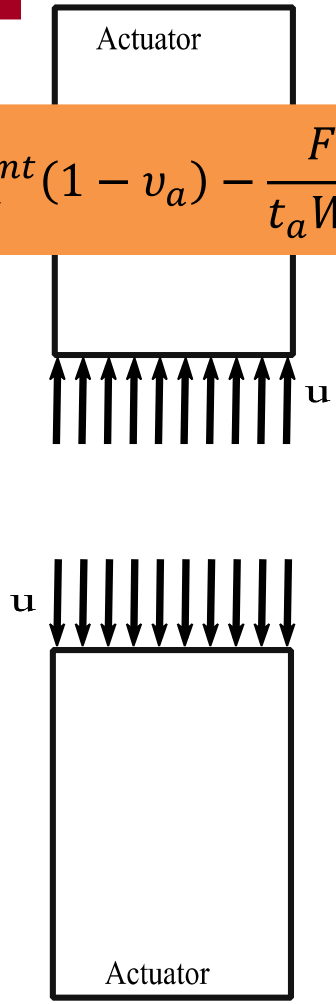
Theoretical analysis

$$\frac{2u}{F} = C(E, a, W_a, W, L)$$

$$u = \frac{L_a}{E_a}\left(\sigma_a^{int}(1-\upsilon_a) - \frac{F}{t_a W_a^*}\right)$$



+

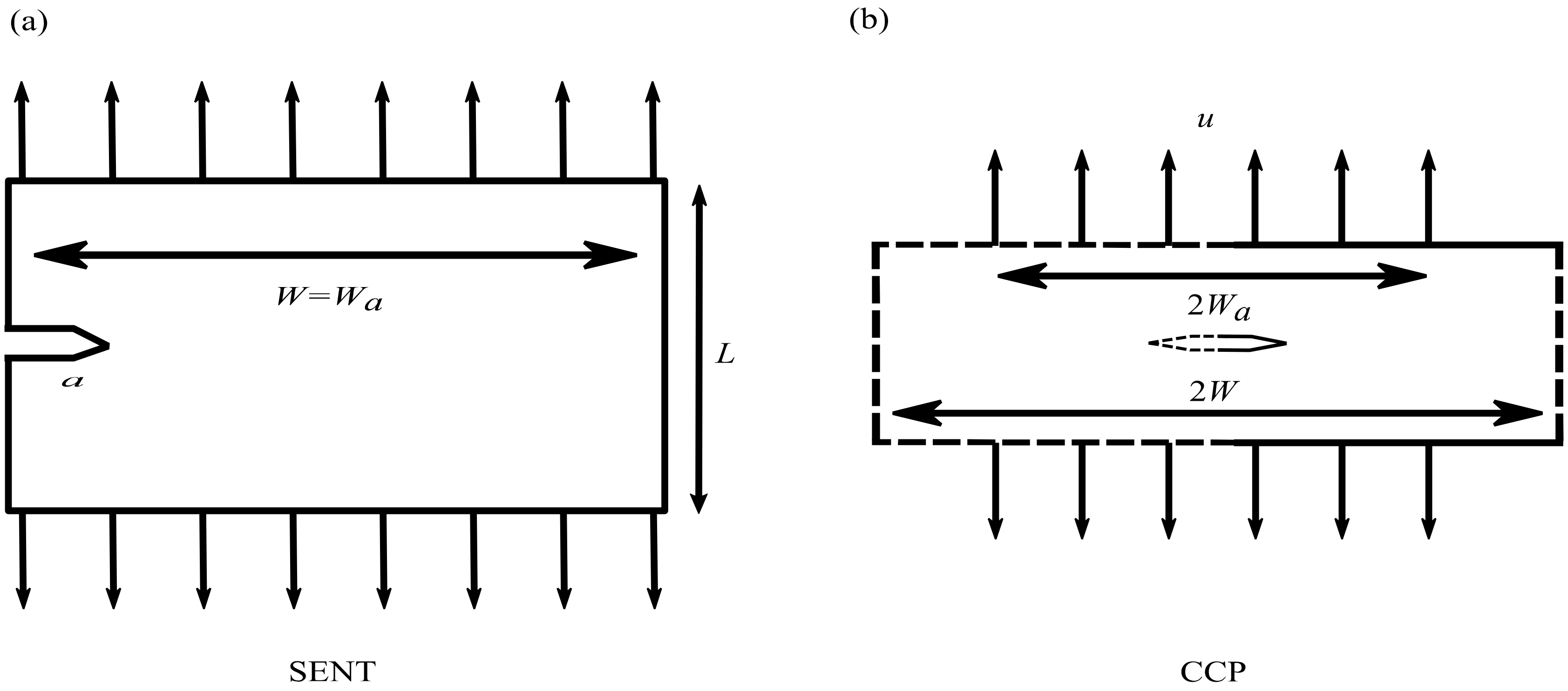


Assuming linear elastic overall behaviour, use superposition principle

$$F = \left(\frac{(1-\upsilon_a)L_a}{\frac{L_a}{t_a W_a^*} + \frac{E_a}{E}\frac{C^*\left(a, \frac{W_a}{W}, \frac{L}{W}\right)}{2}}\right)\sigma_a^{int}$$

Theoretical analysis

With short crack length, the test structures ressemble Center Cracked Panels (CCP) or Single Edge Notched Tension (SENT)



Limit 1: $L \approx W$ & $L < W_a$

Theoretical analysis SENT and CCP panels

$$K = \frac{F}{W^* t} Y\left(\frac{a}{W}\right)\sqrt{\pi a}$$

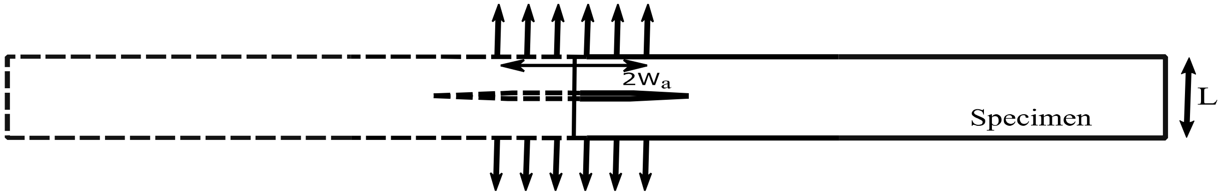
with $G = \frac{F^2}{2t}\frac{\partial C}{\partial a}$ and $G = \frac{K^2}{E^*}$

$$K_{SENTapprox} = (1-\nu_a)\sigma_a^{int}\sqrt{L_a}\sqrt{\frac{L_a}{L}}\frac{1.12\sqrt{\pi\frac{a}{w^*}}\sqrt{\frac{W^*}{L}}}{\frac{L_a}{L}\frac{W}{W_a}\frac{t}{t_a}+\frac{E_a}{2E}\left(\alpha_2 1.12^2\pi\left(\frac{a}{W^*}\right)^2\frac{W^*}{L}+\alpha_3\right)}$$

$$K_{CCPapprox} = (1-\nu_a)\sigma_a^{int}\sqrt{L_a}\sqrt{\frac{L_a}{L}}\frac{\sqrt{\pi\frac{a}{w^*}}\sqrt{\frac{W^*}{L}}}{\frac{L_a}{L}\frac{W}{W_a}\frac{t}{t_a}+\frac{E_a}{2E}\left(\alpha_2\pi\left(\frac{a}{W^*}\right)^2\frac{W^*}{L}+\alpha_3\right)}$$

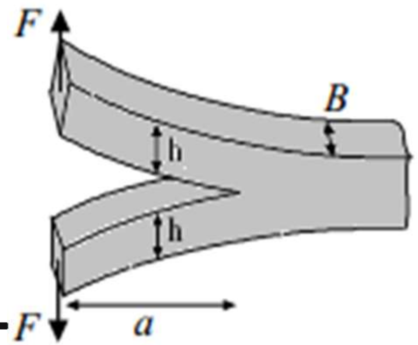
Theoretical analysis

With longer crack lengths, the test structures ressemble Double Cantilever Beam geometry (DCB)

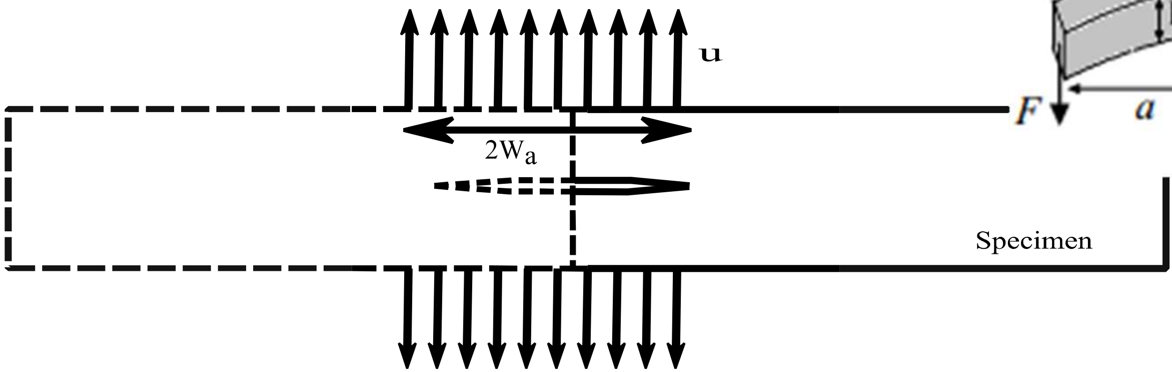


DCB

Limit 2: $W_a << W$ & $L << W$



(d)



In between

Limit 3: $Wa < W$ & $L \approx W$

Theoretical analysis

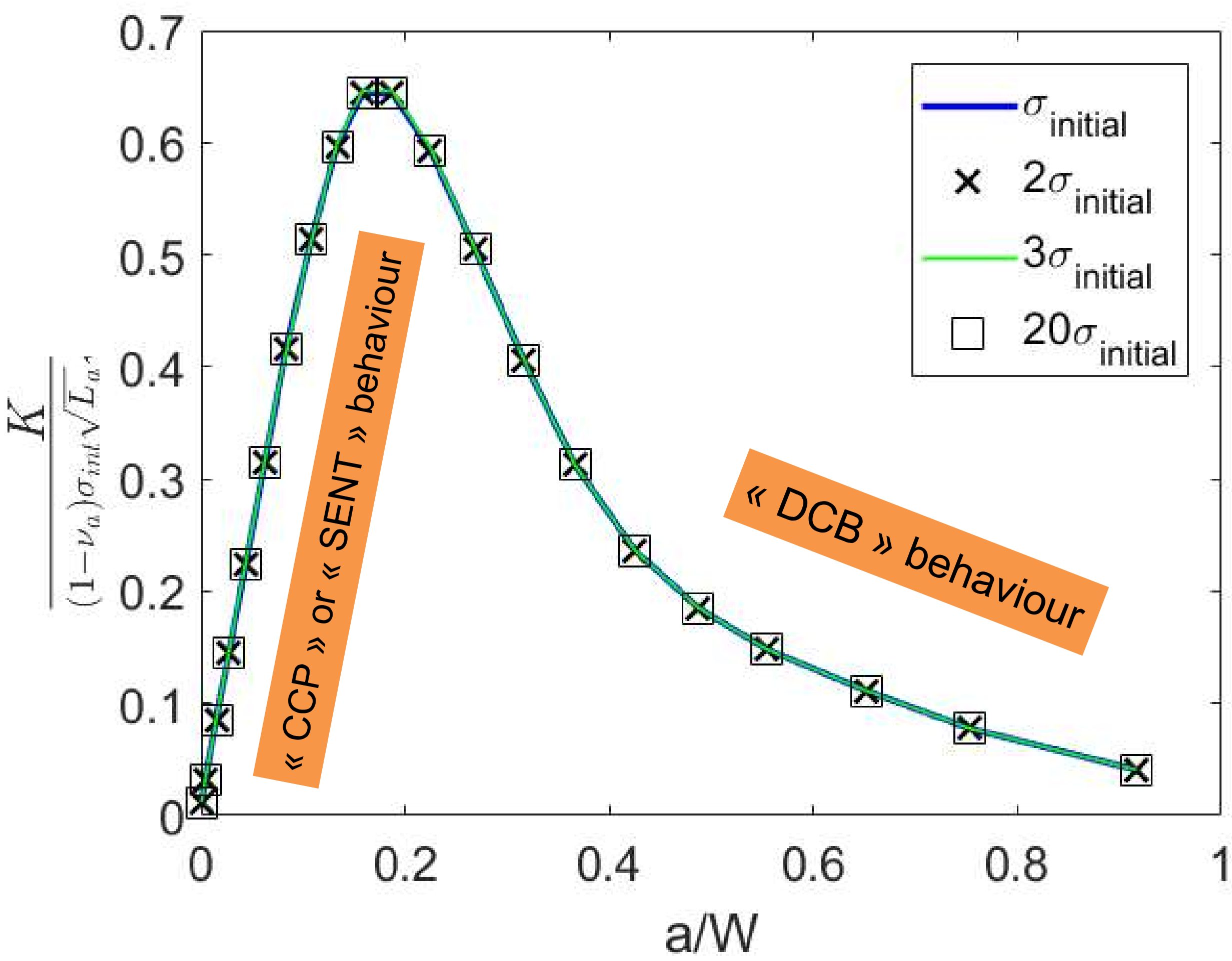
with $G = \frac{F^2}{2t}\frac{\partial C}{\partial a}$ and $G = \frac{K^2}{E^*}$

$$K_{DCBasym} = 4\sqrt{\frac{6}{\alpha_2}}(1-\nu_a)\sigma_a^{int}\sqrt{L_a}\,\frac{\frac{a}{W}\frac{L^2}{W^2}\sqrt{\frac{L_a}{L}}}{4\frac{E_a}{E}\frac{a^3}{W^3}+\frac{L^3}{W^3}\frac{L_a}{W_a^*}\frac{t}{t_a}}$$

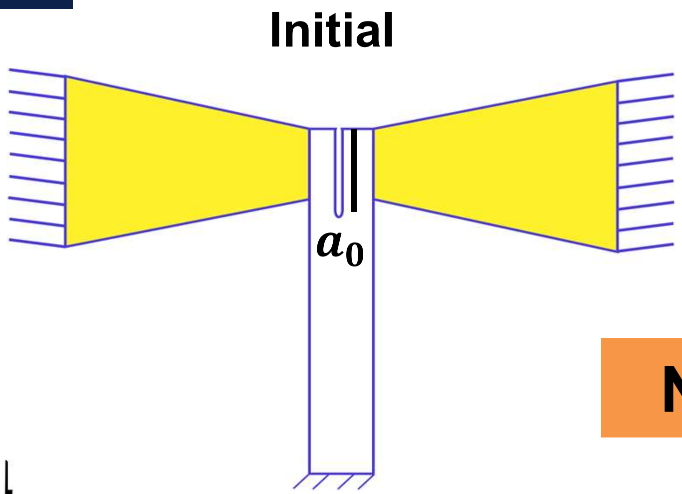
$$K_{DCBsym} = 2\sqrt{\frac{6}{\alpha_2}}(1-\nu_a)\sigma_a^{int}\sqrt{L_a}\,\frac{\frac{a}{W}\frac{L^2}{W^2}\sqrt{\frac{L_a}{L}}}{4\frac{E_a}{E}\frac{a^3}{W^3}+\frac{L^3}{W^3}\frac{L_a}{W_a^*}\frac{t}{t_a}}$$

Finite element analysis

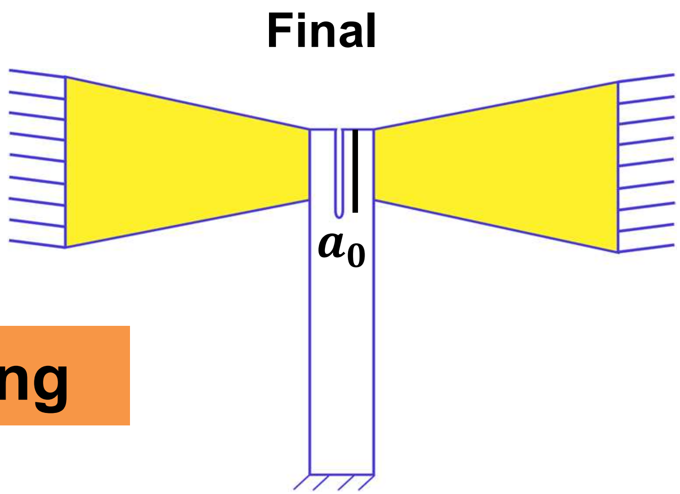
Verification : K_I scales linearly with internal stress in actuator



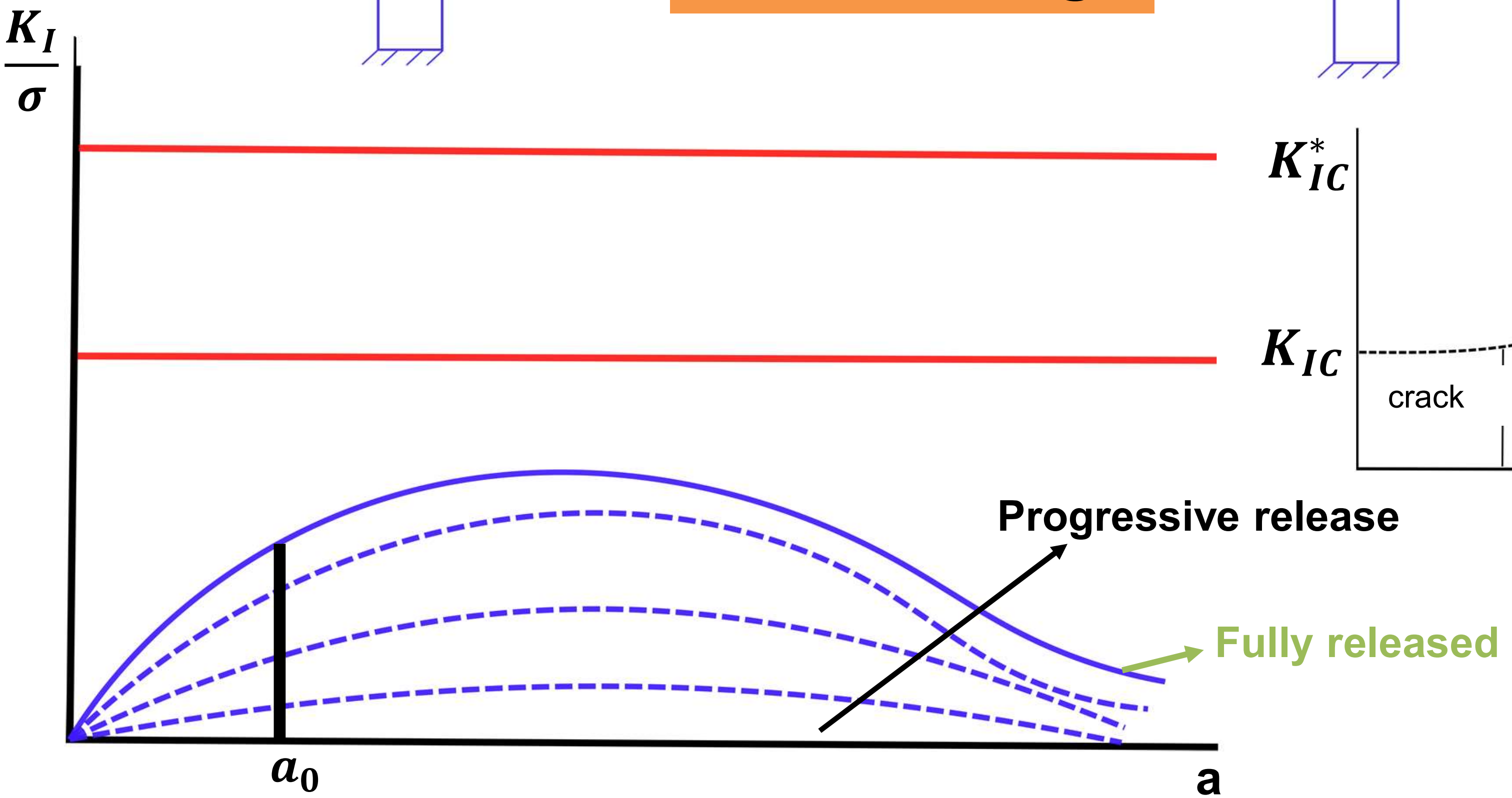
Cracking process in practice

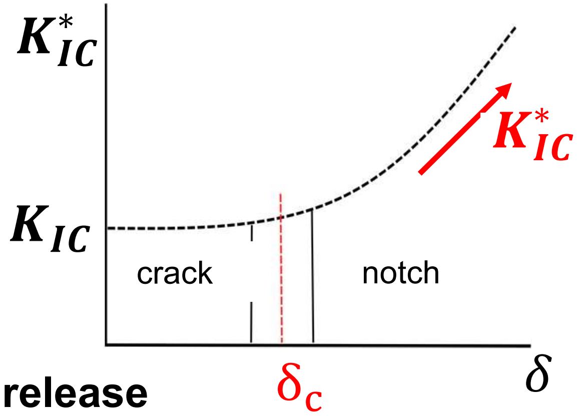


Short L_a

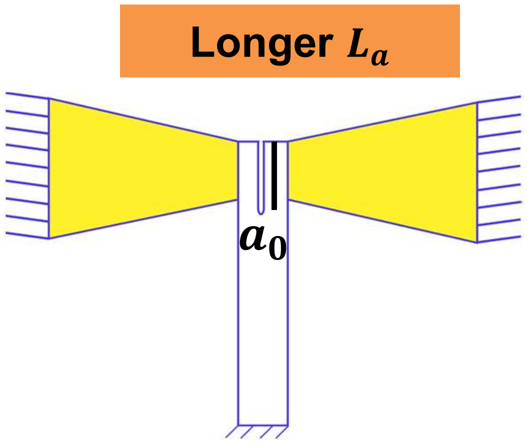


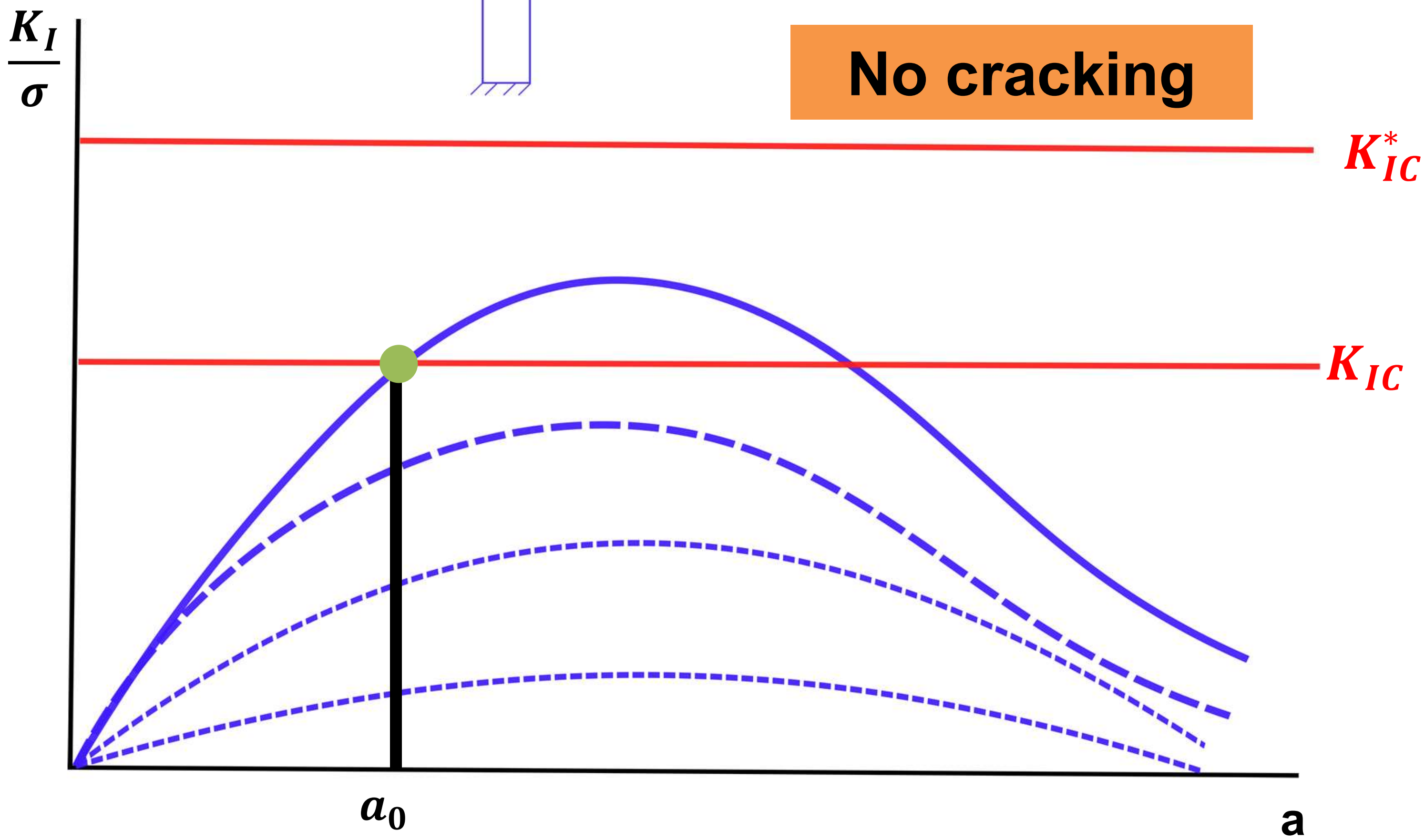
No cracking





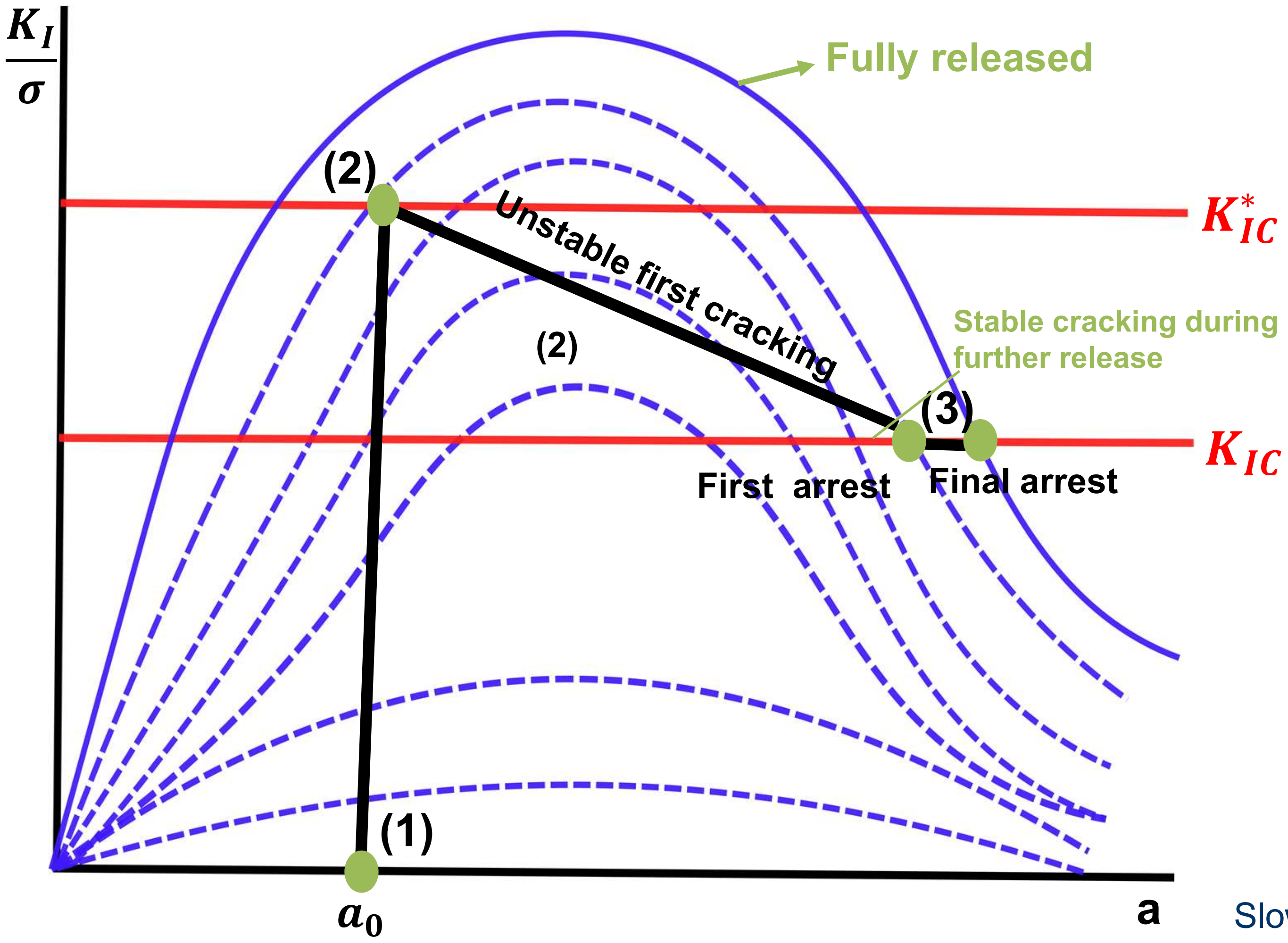
Cracking process in practice



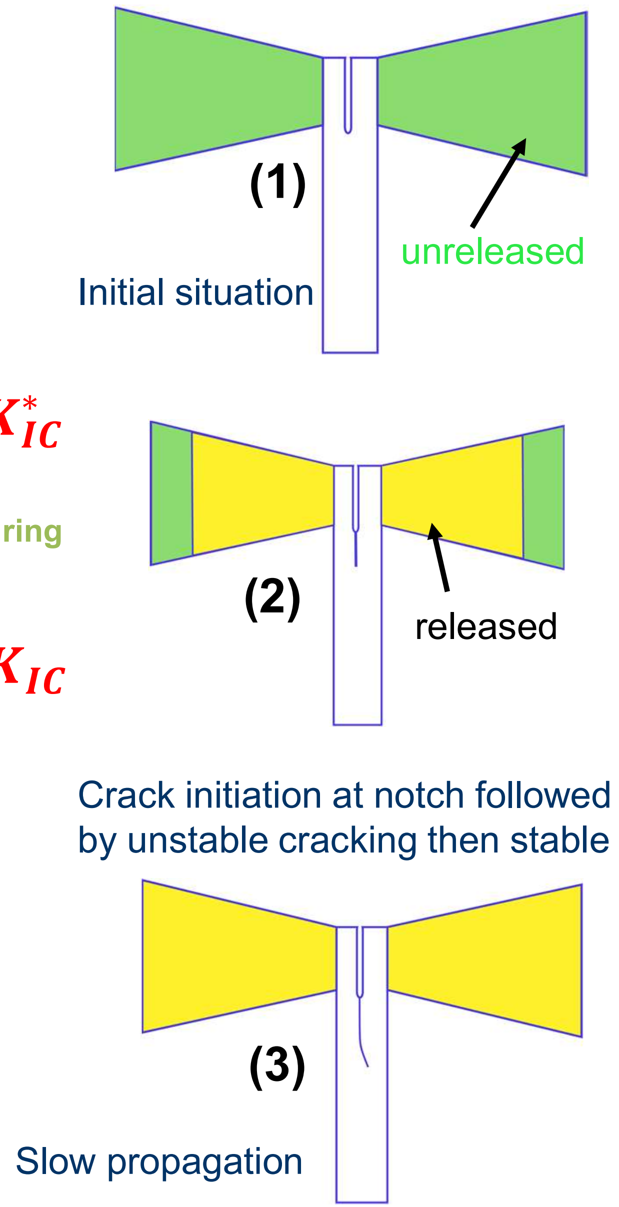


Cracking process in practice

Cracking

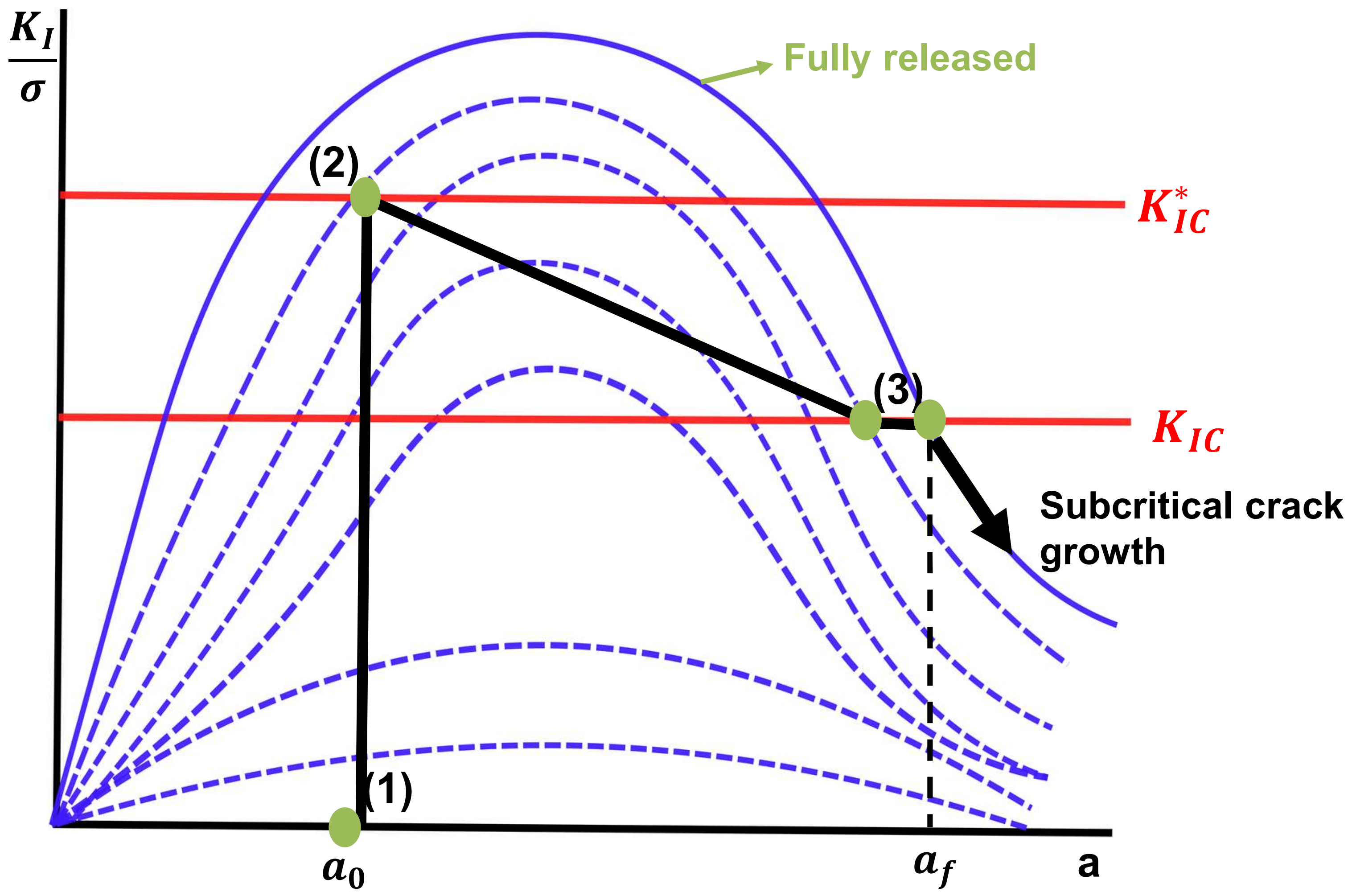


Long L_a

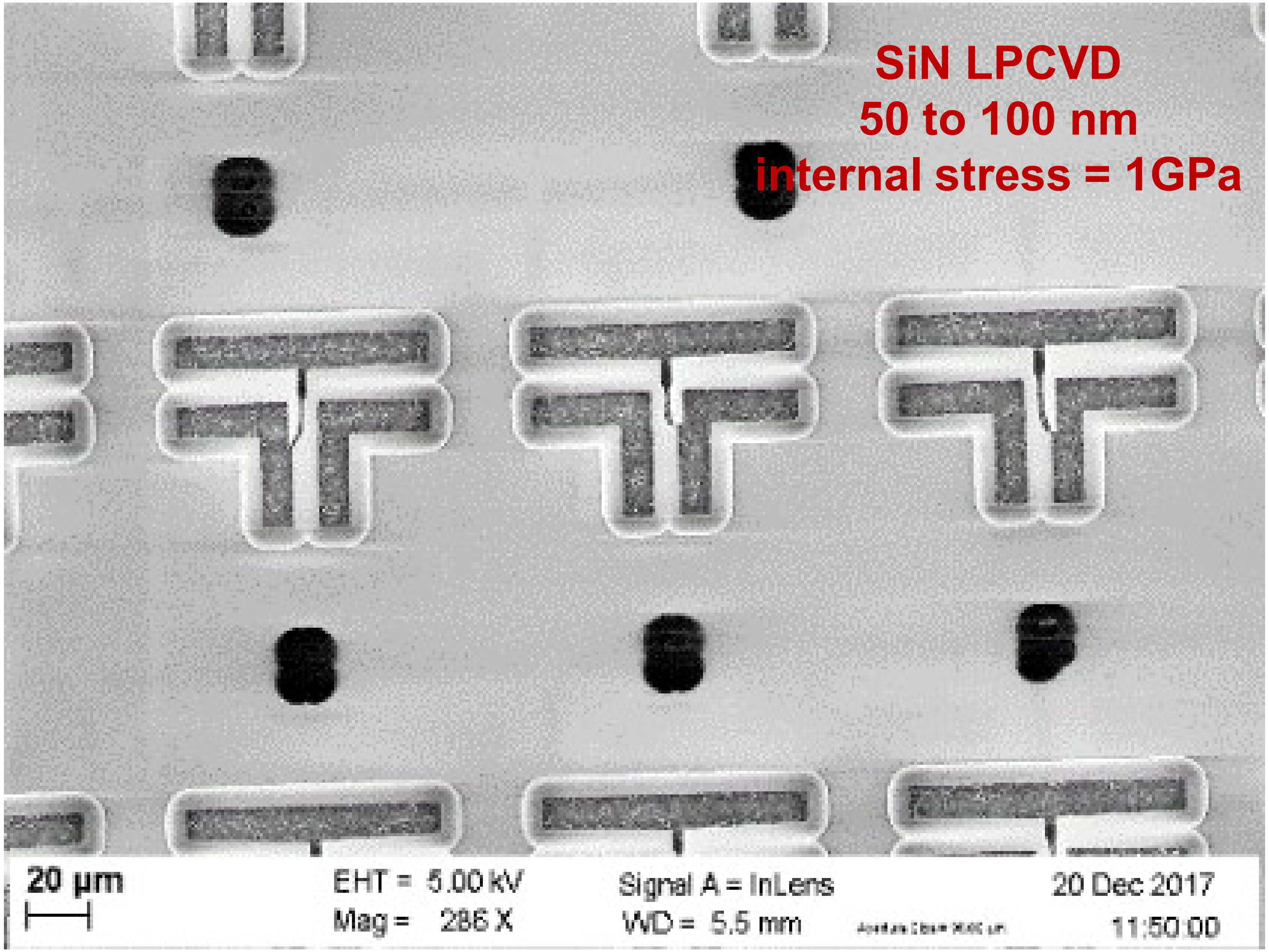


Cracking process in practice

Possible subcritical crack growth: environmental, creep…

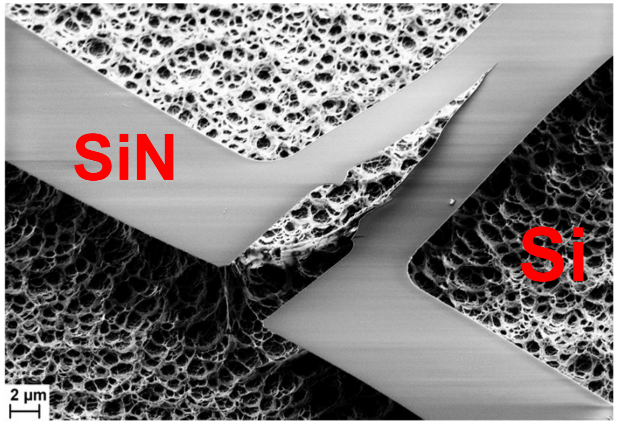


Experimental results

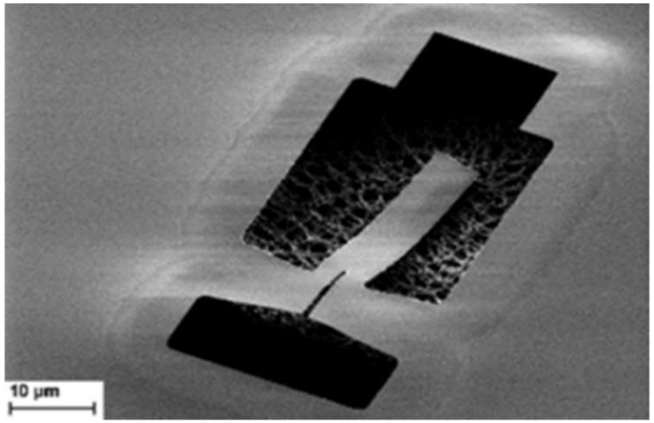


Experimental problems

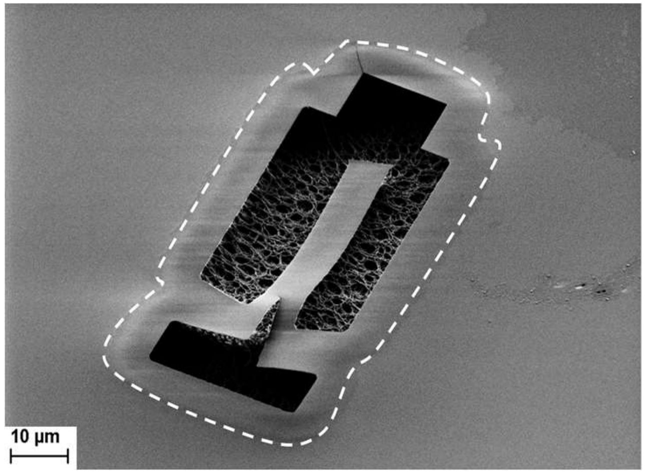
Mode III



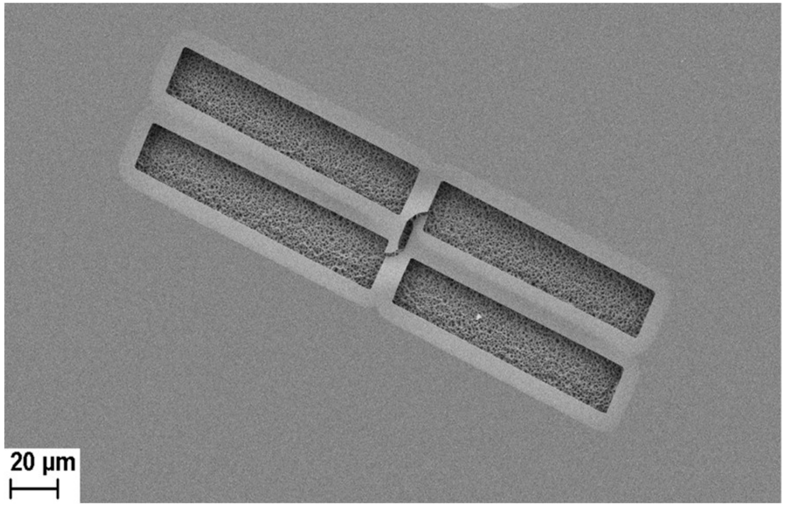
Stiction



Underetching

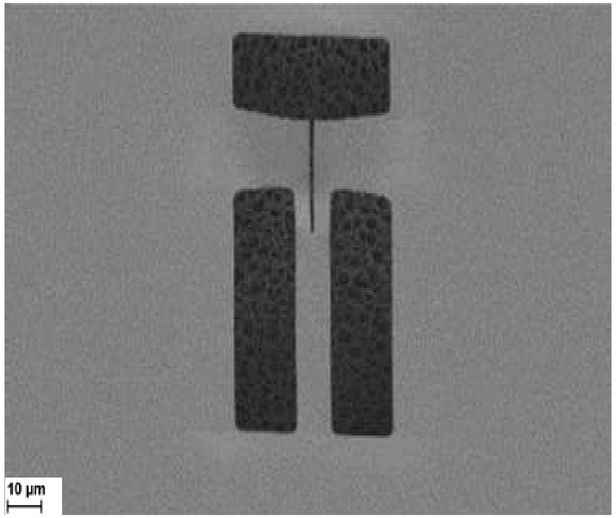


Kinking out

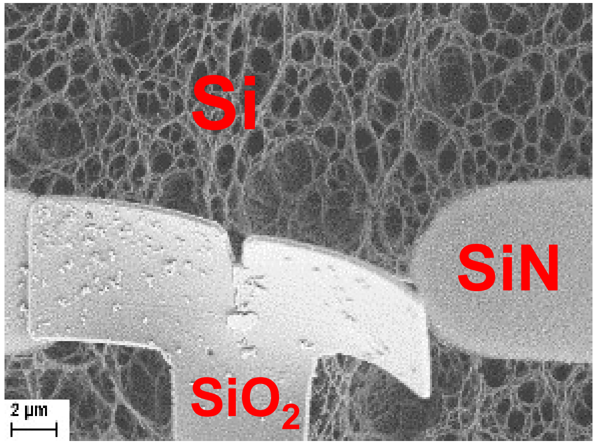


Experimental problems

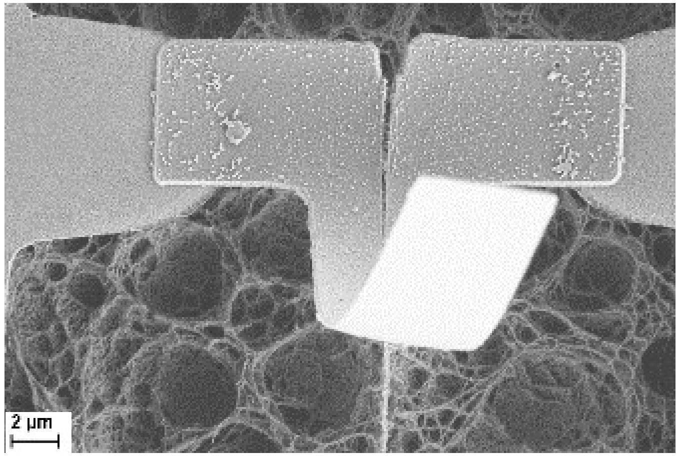
No cracking



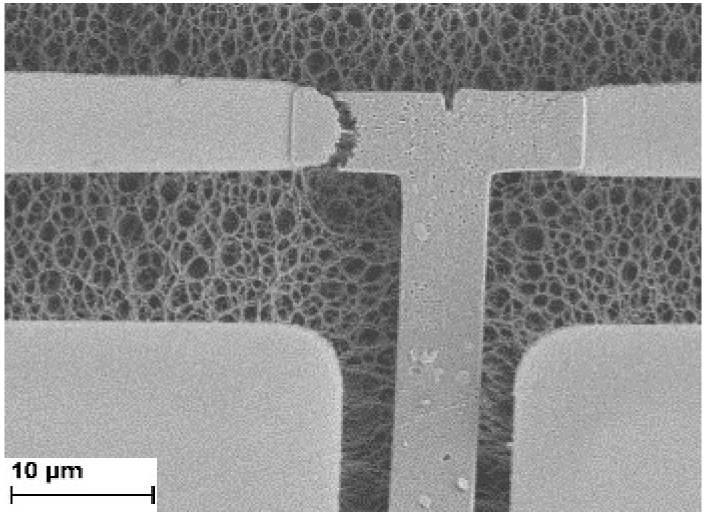
No attachment



Out of plane

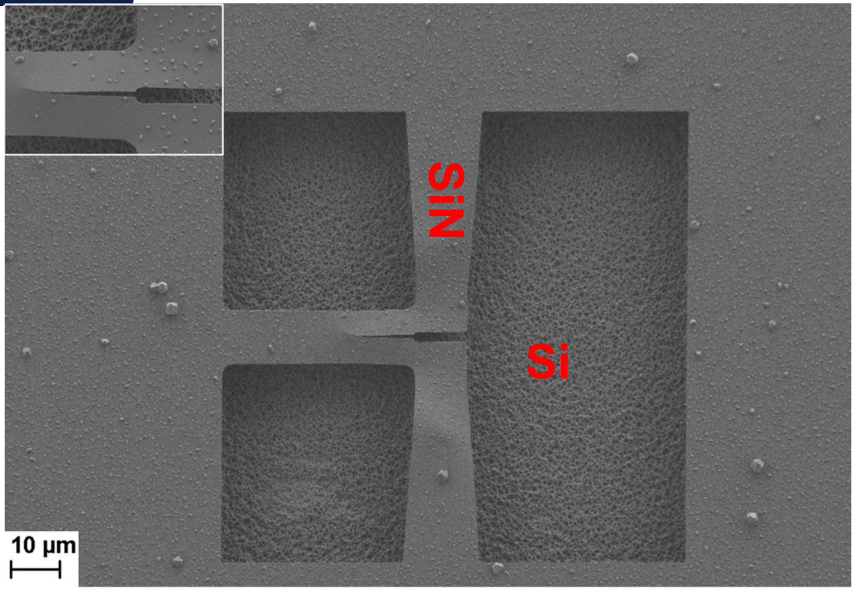


Undesired fracture

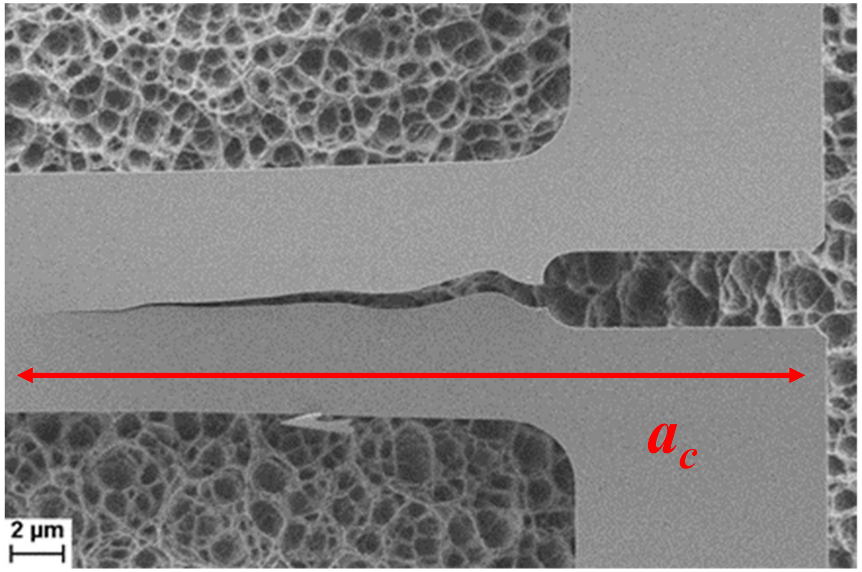


Application to 55 and 93 nm thick SiN

SiN of 55 nm

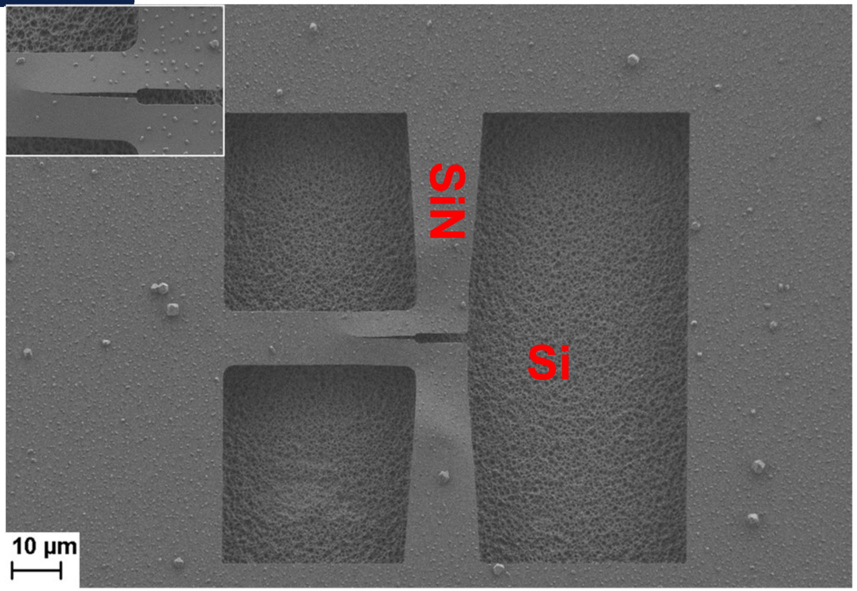


SiN of 93 nm

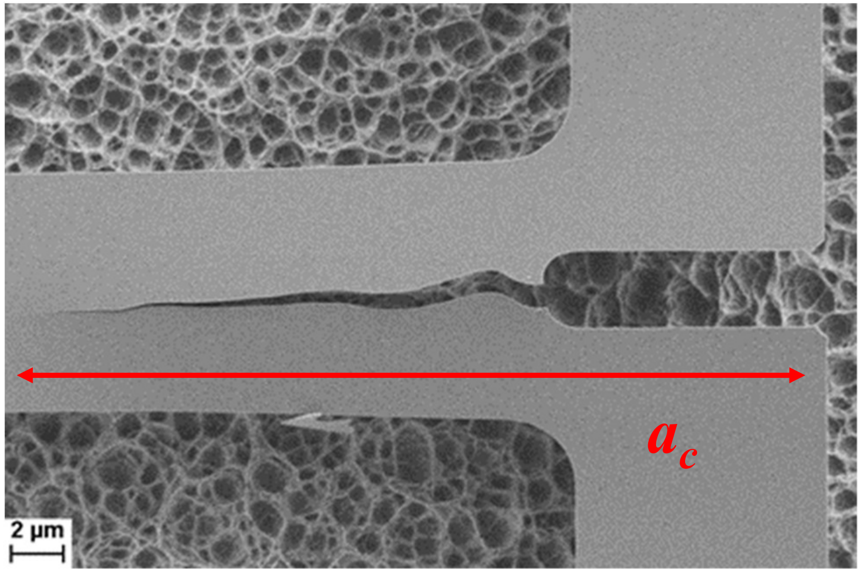


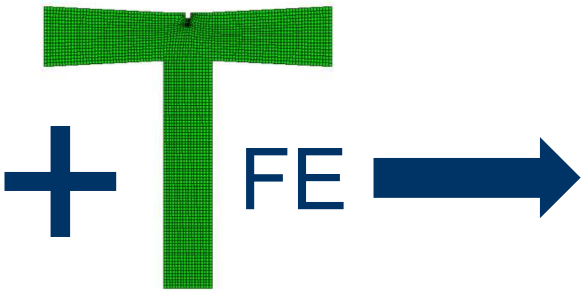
Application to 55 and 93 nm thick SiN

SiN of 55 nm



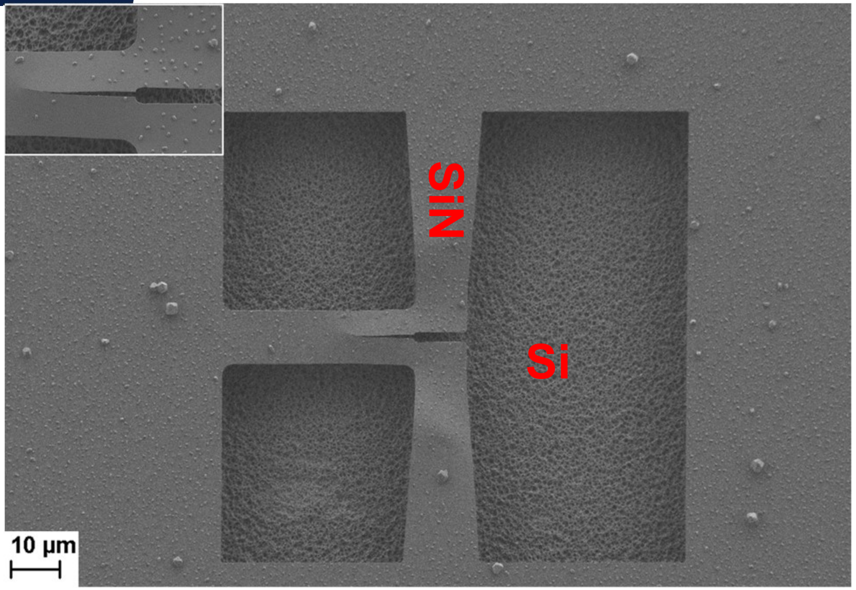
SiN of 93 nm



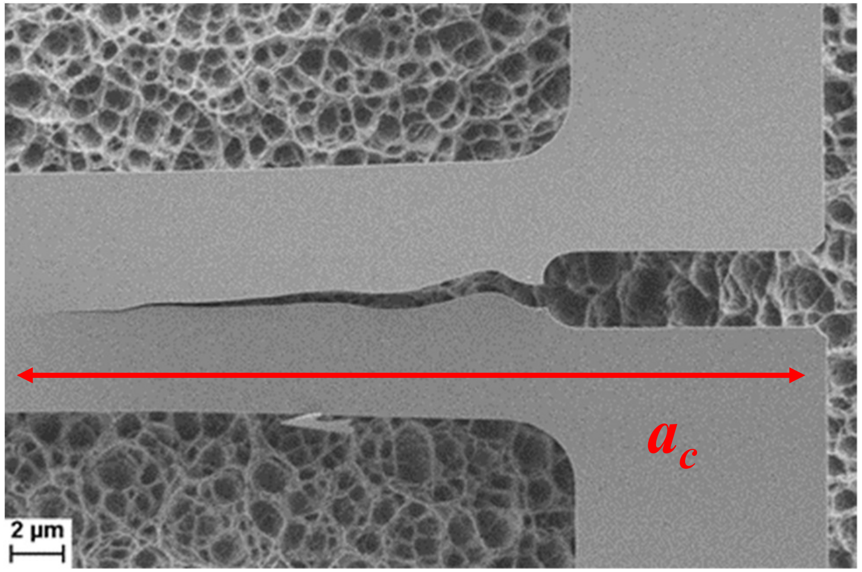


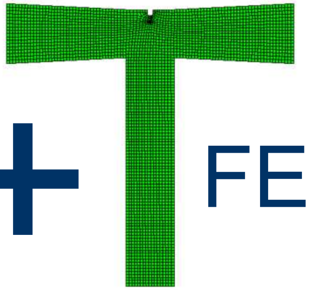
Application to 55 and 93 nm thick SiN

SiN of 55 nm



SiN of 93 nm





FE

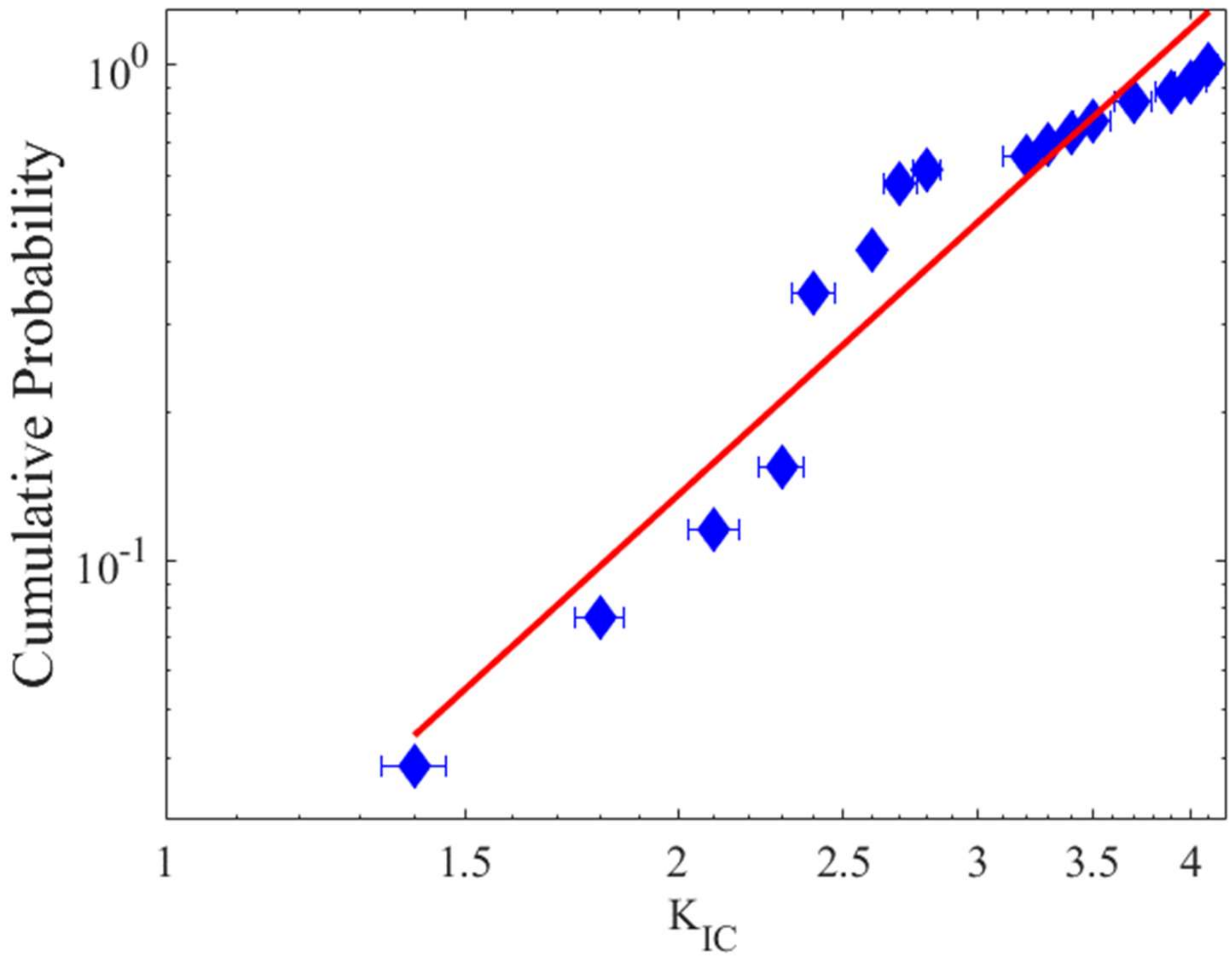
Ref. number	L_a^* [µm]	L [µm]	W [µm]	W_a^* [µm]	$t=t_a$ [nm]	a_{c_arrest} [µm]	K_{Ic} [MPa$\sqrt{\text{m}}$]
I	15±1	10±1	50±2	11±0.2	55±1	25.5 ±0.2	1.2±0.1
II	10±1	10±1	30±2	11±0.2	55±1	19±0.2	1.4±0.2
III	10±0.3	8±0.1	50±1	9±0.1	55 ±1	14.5±0.7	1.8±0.1
IV	85±4	8.8±1	48±3	10.25±0.3	93±1	26.9±0.1	1.7±0.3
V	62.4±4	8.8±1	48.3±2	10.05±0.3	93±1	27±0.2	1.4±0.2
VI	75.5±2	9.1±1	48.5±1	10.25±0.1	93±1	27.7±0.1	1.5±0.2
VII	85.9±4	9±1	48.2±4	10.25±0.3	93±1	28.2 ±0.1	1.6±0.3
VIII	53±5	8.6±1.5	48.5±3	10.6±0.7	93±1	18±1	2.9±0.1
IX	50±6	8.6±1.5	48±3	10.5±0.3	93±1	20±1.2	2.1±0.3
X	53.5±5	9.4±1	48±1	9.8±0.3	93 ±1	24.4±0.2	1.6±0.2
XI	46.1±1	9±1	44±1	9.5±0.4	93±1	23±0.2	1.5±0.2
XII	65.2±7	9±1	35±1	9.6±0.5	93±1	16.5±0.3	3.4±0.4
XIII	54±2	9±1	37±2	9.55±0.3	93±1	17.2±0.4	2.9±0.3
XIV	52.7±4	9±1	42±2	10.3±0.3	93±1	20.7±0.2	2.1±0.4
XV	62.5±2	9±1	39.2±1	10.3±0.5	93±1	21±1.5	2.4±0.05

$K_{Ic\_mean} \sim 2$ MPa$\sqrt{m}$

K_c of 1.82 ± 0.03 MPa.m$^{1/2}$

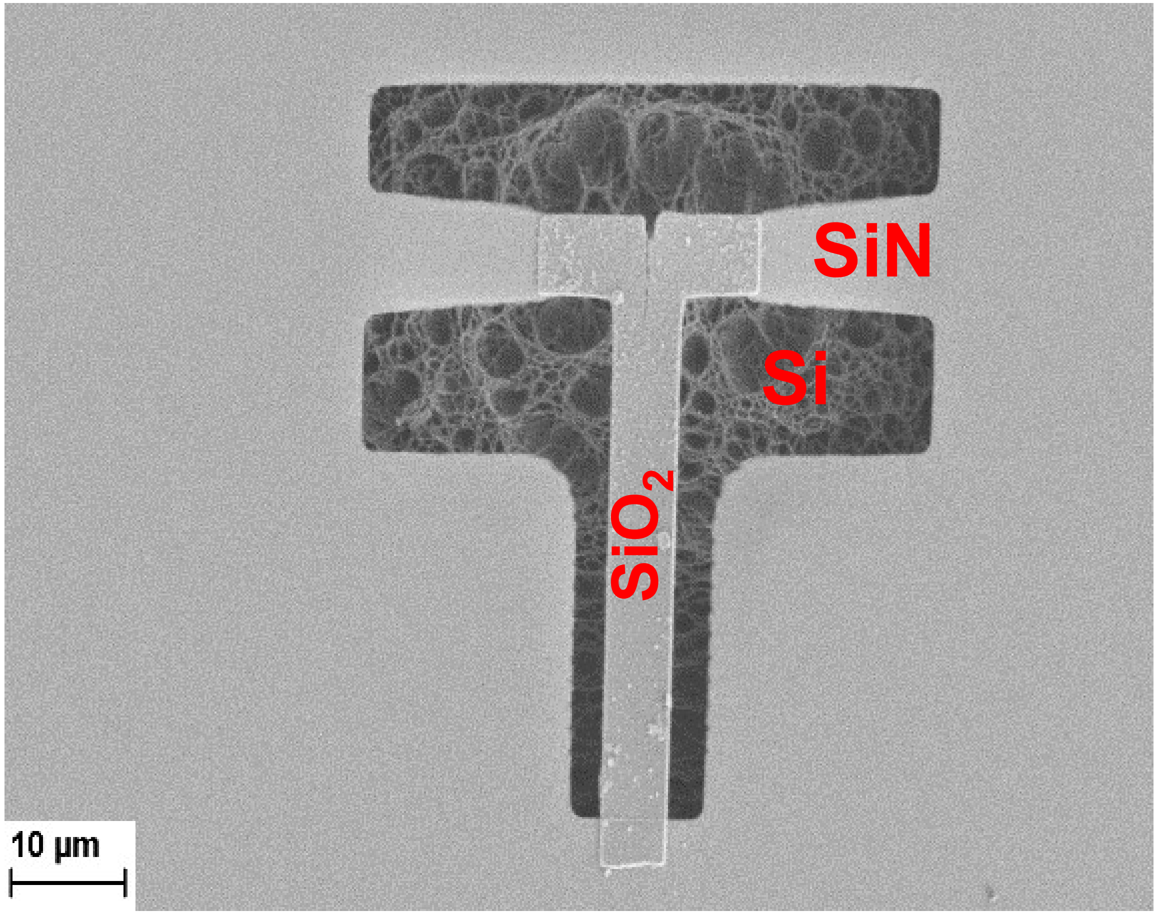
Pierron Group (2016) ACS Appl. Mater. Interfaces

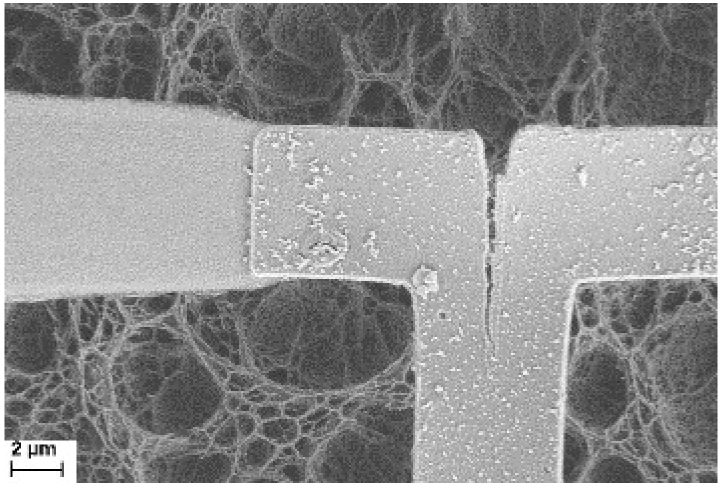
Application to 55 and 93 nm thick SiN



Median= 2.7 MPa$\sqrt{m}$
$R^2 = 92\%$
Mean=2.9 MPa$\sqrt{m}$

Application to 150 nm thick SiO_2





Approach 2 : Freestanding thin films Conclusion

Pro and cons

Generate true intrinsic properties (but …) – no artifact from substrate

Allow in situ TEM testing

Testing is complex – MEMS types devices help

Points of attention

- Importance of the state of the surface (oxide, roughness, …)
- Higher strength at small scale but also higher rate sensitivity
- Huge effect of imperfections : statistical treatment essential
- Fracture toughness often not valid except if sufficiently brittle

Outline

1. Introduction

2. Fracture of films on substrates

- test methods and extraction of *G*
- example 1 : CrN on polymer (indentation)
- example 2 : Au on polymer (for flexible electronics)

3. Fracture of freestanding films

- test methods for measuring the fracture strength & strain
- fracture strength of brittle films
- fracture strain of ductile films
- fracture toughness