Thin film mechanics

Stability and delamination

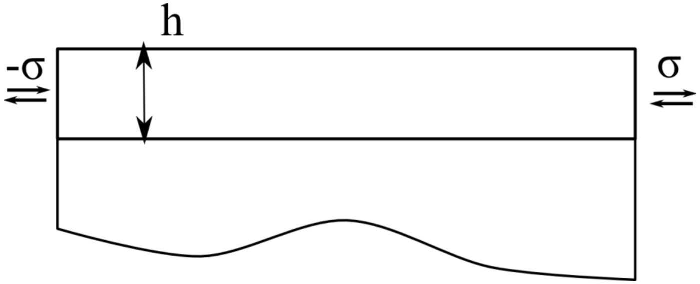
Etienne Barthel

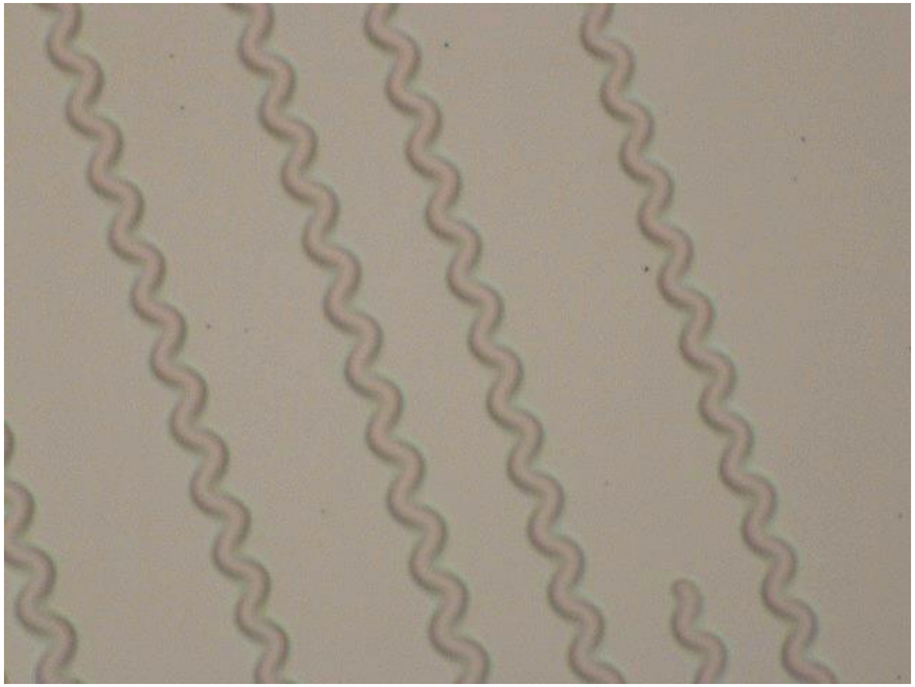
Thin film mechanics

Outline

- ☐ Residual stresses in thin films
- ☐ Thin film fracture and delamination
- ☐ Thin film indentation

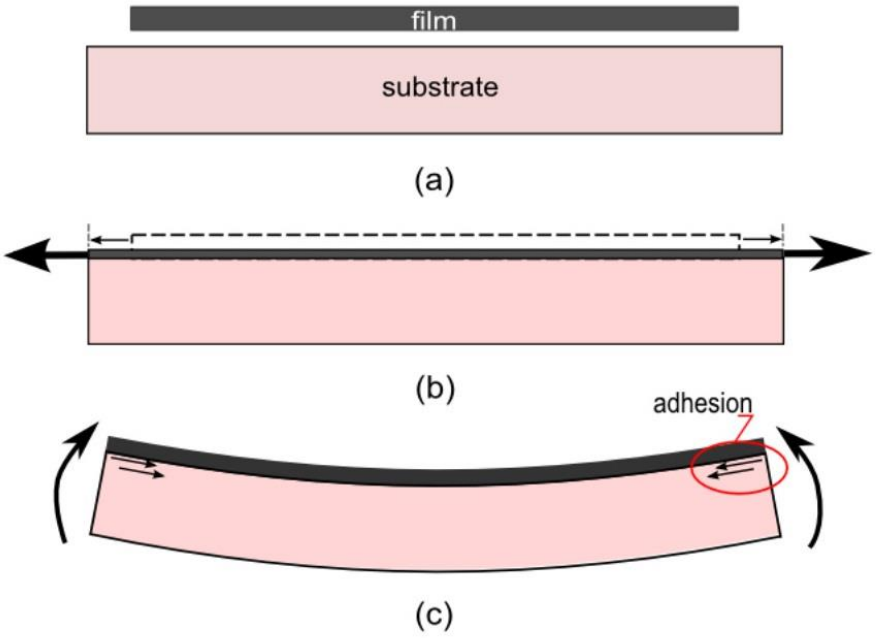
I – Residual stresses in thin films





Telephone cord delaminations

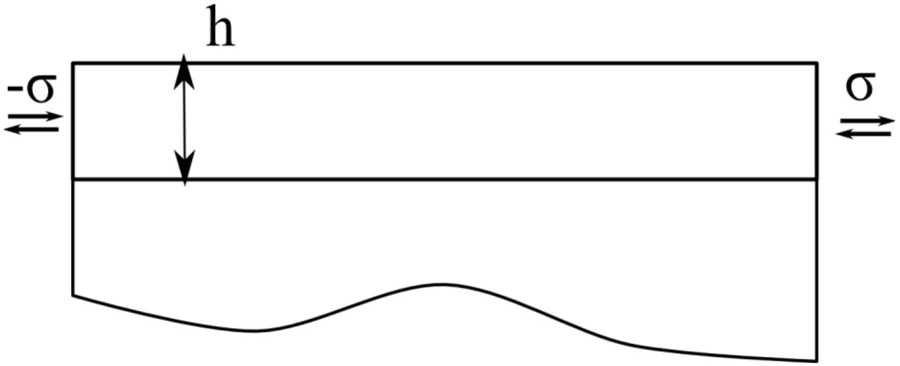
Misfit strains



Mechanisms : the misfit strains in thin films may be due to
- thermal contraction
- non equilibrium deposition (atomic bombardment) or phase transformations (solidification, resin curing, martensitic transformations)
- plastic deformation (eg shot peening)

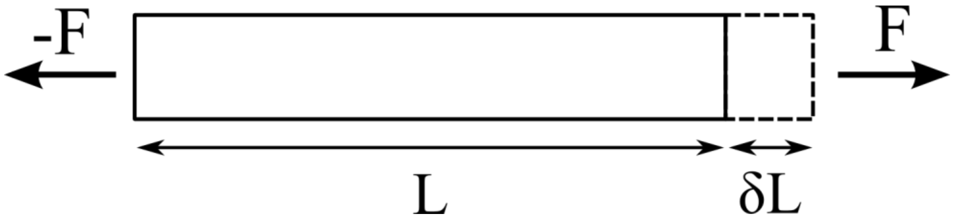
https://www.doitpoms.ac.uk/tlplib/coating_mechanics

Thin films & coatings



- Geometry: thin film = small h
- Mechanical responses: film/substrate contrast
- Loading: residual stresses

Elasticity



$$\begin{aligned}\sigma &= \frac{F}{A}\\ \epsilon &= \frac{\delta L}{L}\end{aligned}$$

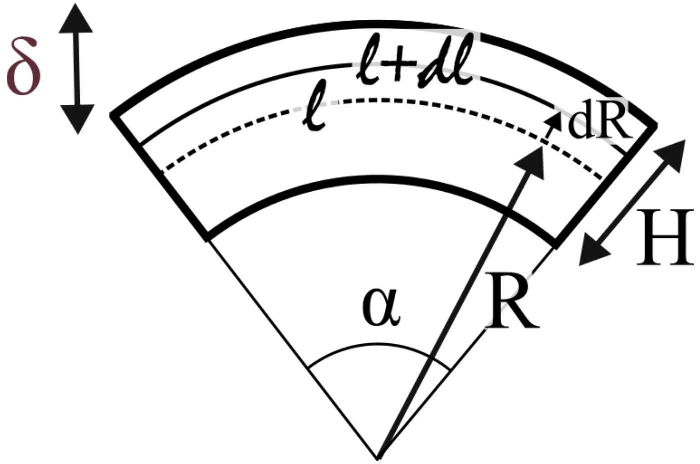
- Elastic response
  - stress is linear with deformation
  - E is elastic modulus

$$\sigma = E\epsilon$$

- Energy density
  - quadratic

$$\mathcal{E} = \frac{E\epsilon^2}{2} = \frac{\sigma^2}{2E}$$

Beam bending



$$\frac{l+dl}{l} = \frac{\alpha(R+dR)}{\alpha R} = 1+\epsilon$$

curvature $$\frac{1}{R} \simeq \frac{\delta}{l^2}$$

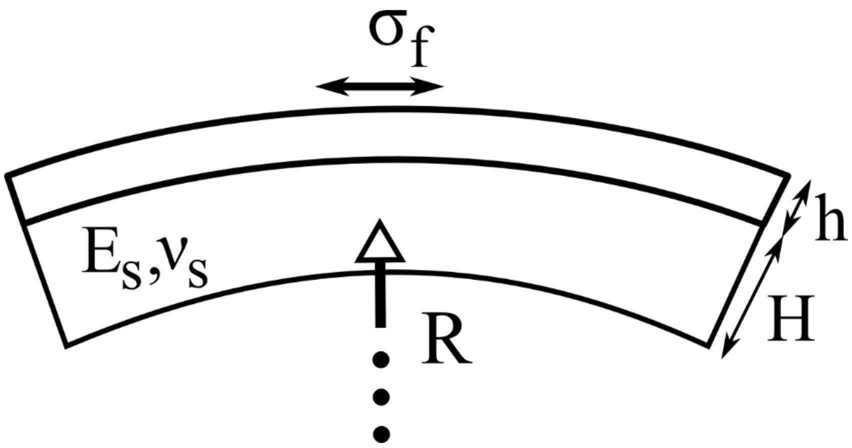
elastic energy density $$\mathcal{E} = \frac{1}{2}E\epsilon^2$$

$$\mathcal{E}_{\mathrm{tot}} \simeq ElH\epsilon^2 = ElH\left(\frac{H\delta}{l^2}\right)^2 = l\ EH^3\ \left(\frac{\delta}{l^2}\right)^2$$

curvature

bending stiffness

Residual stresses – Stoney



$$\mathcal{E} \simeq l\ E_s H^3 \left(\frac{\delta}{L^2}\right)^2$$

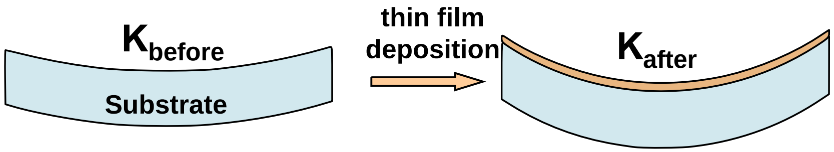
$$\mathcal{M} \simeq E_s H^3 \frac{\delta}{L^2} \simeq \frac{E_s H^3}{R}$$

$$\mathcal{M}_f \simeq \sigma_f h H$$

$$\boxed{\sigma_f \simeq \frac{E_s H^2}{hR}}$$

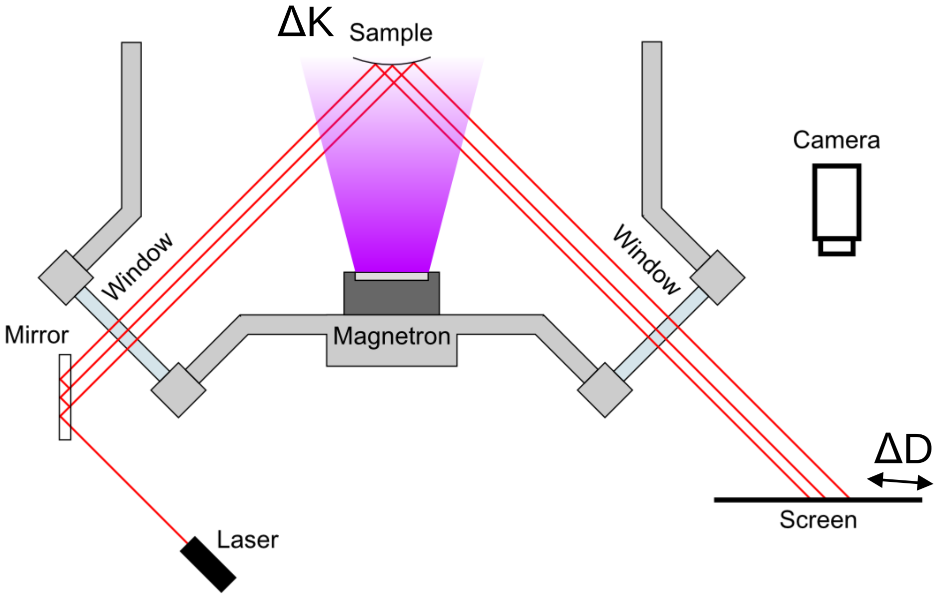
Stoney 1909

In-situ measurement of the stress



Stoney formula :

$$\Delta\sigma = \left(\kappa_{after} - \kappa_{before}\right) \frac{E_s}{6\left(1-\nu_s\right)} \frac{t_s^2}{t_f}$$

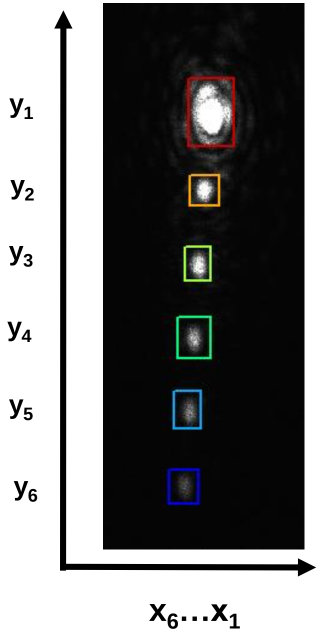


Relation between curvature of sample and distance between spots :

$$\kappa(t) = \frac{\cos(\alpha)}{2L}\left(1 - \frac{D(t)}{D_{ref}}\right)$$

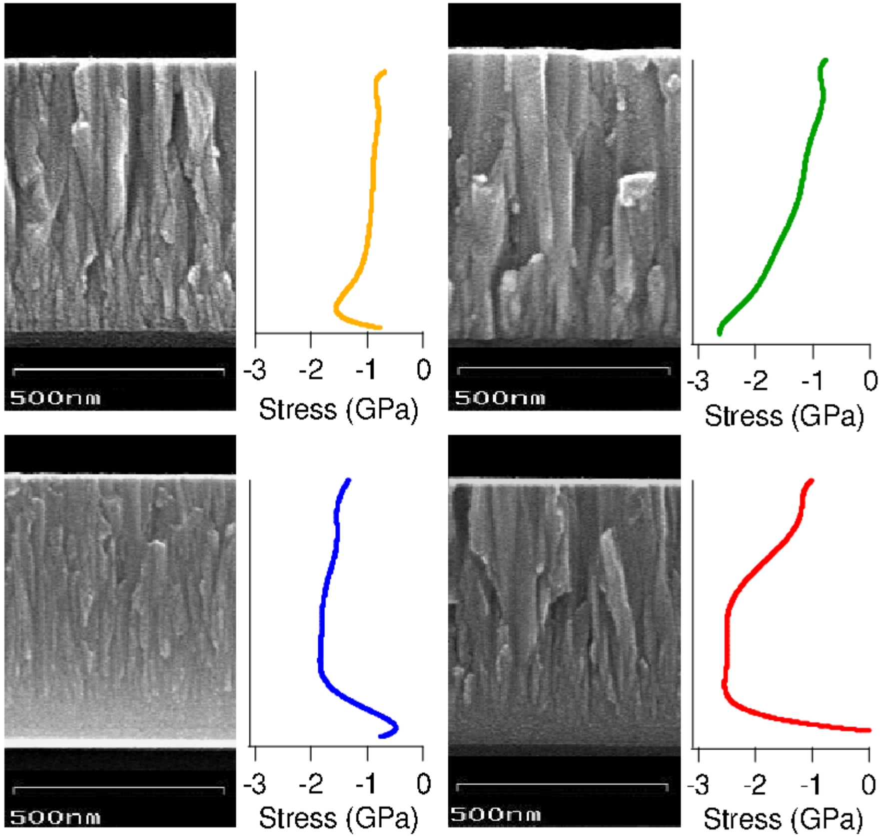
Displacement D(t) of the spots is found by image correlation :

$$y_{1\ldots6}(t+1) = \frac{D(t)}{D(t=0)}\, y_{1\ldots6}(t)$$



Precision on curvature is $K_{min}=6 \times 10^{-5}$ m^{-1}

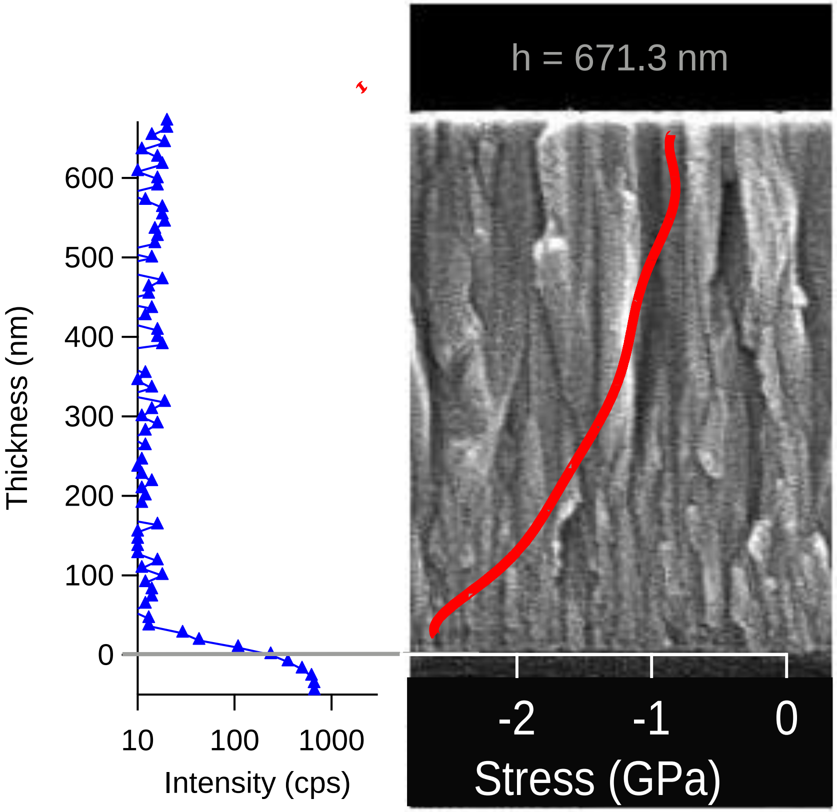
Experiments – stress in Mo layer



Faou et al. Thin Solid Films (2013) 222

Microstructure and residual stresses

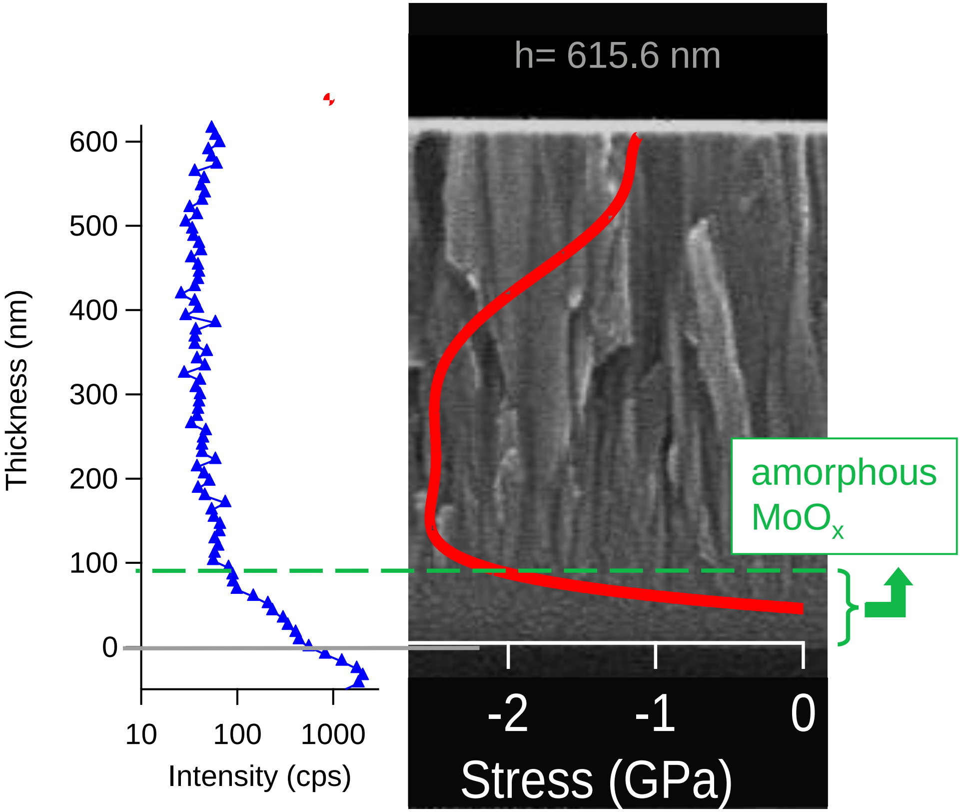
Mo, -75V



[O^-]

Local stress

MoO_x , -75V



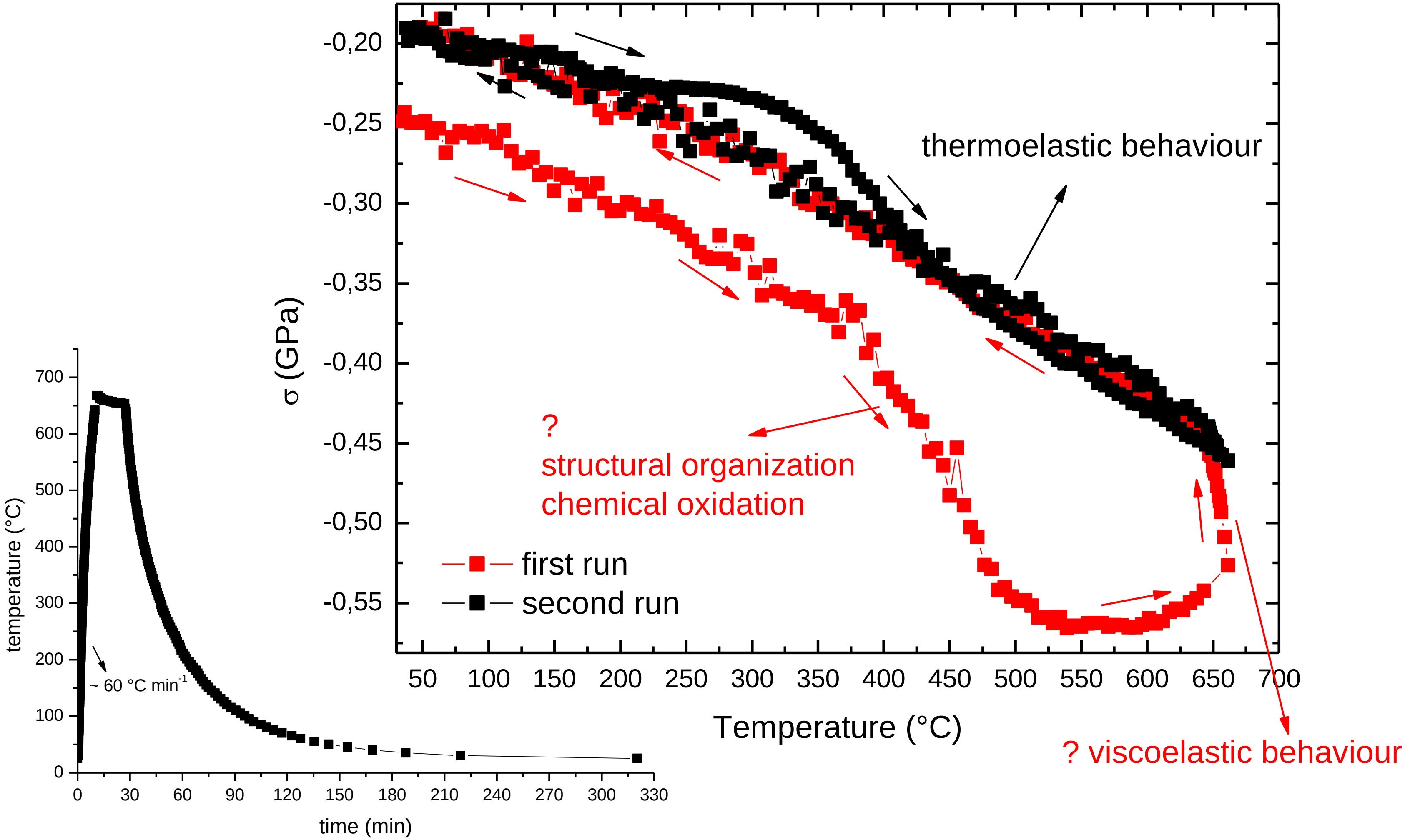
[O^-]

Local stress

Faou et al. Thin Solid Films (2013) 222

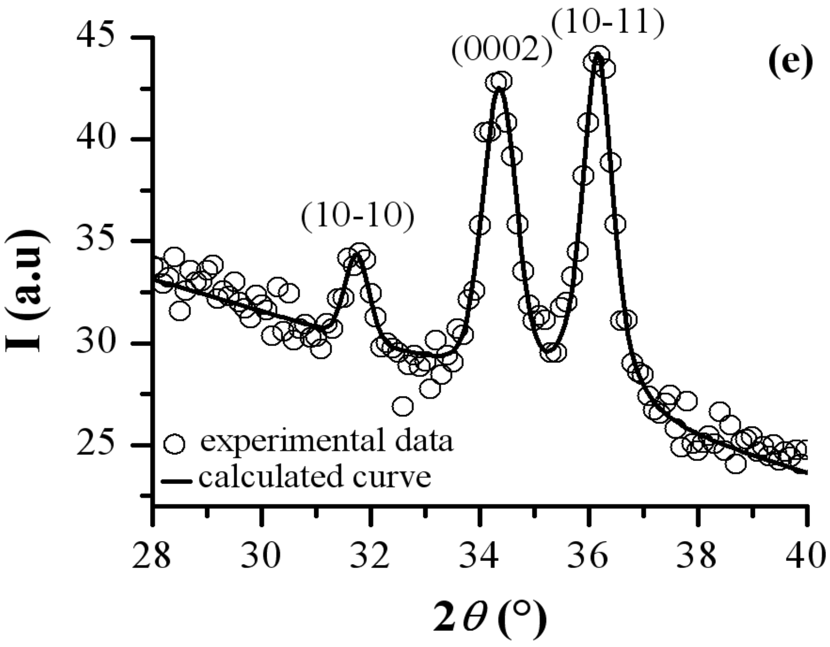
Stress measurements – Stoney method

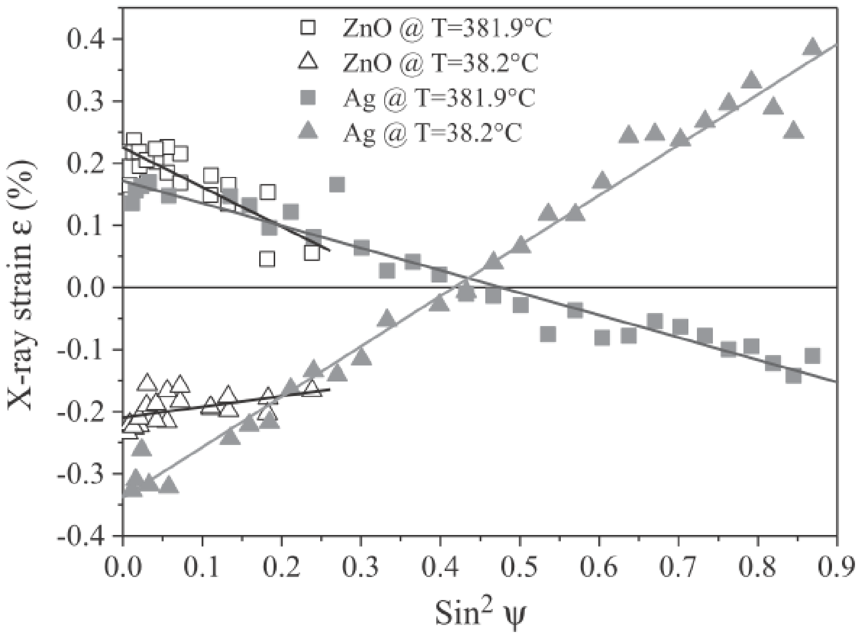
800 nm of ZnO:F on Si(100)

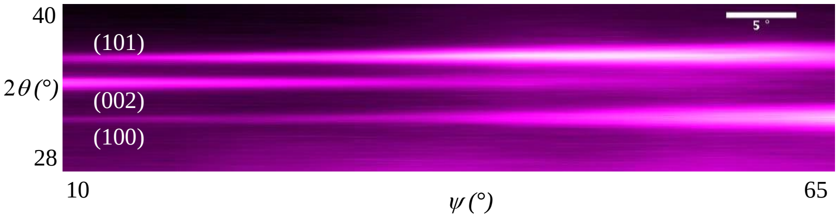


The stress “relaxation” on the first run is usually observed for magnetron films (Si_3N_4, $SnZnO_x$, …)

X Ray Determination of Residual Stresses



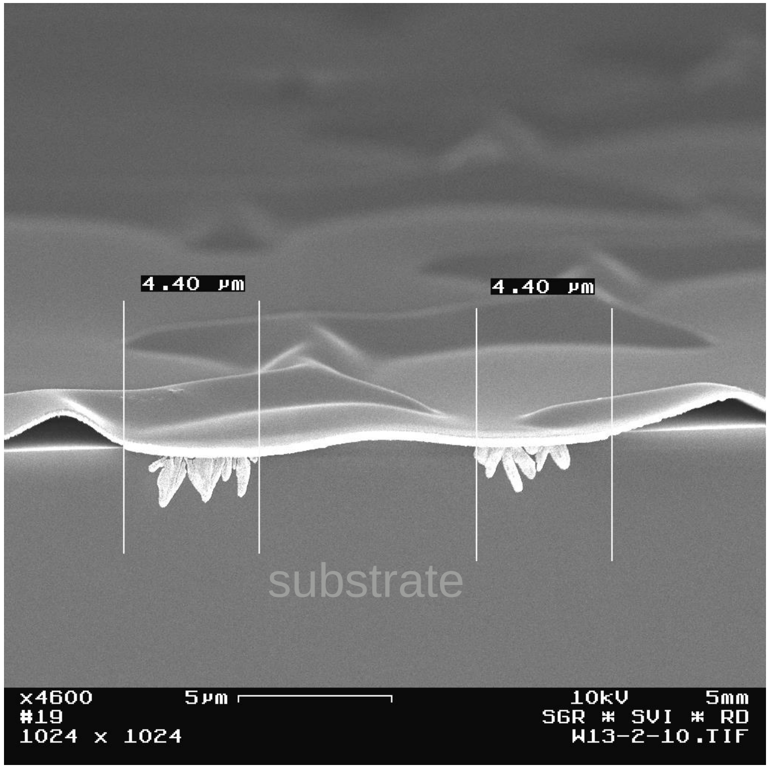


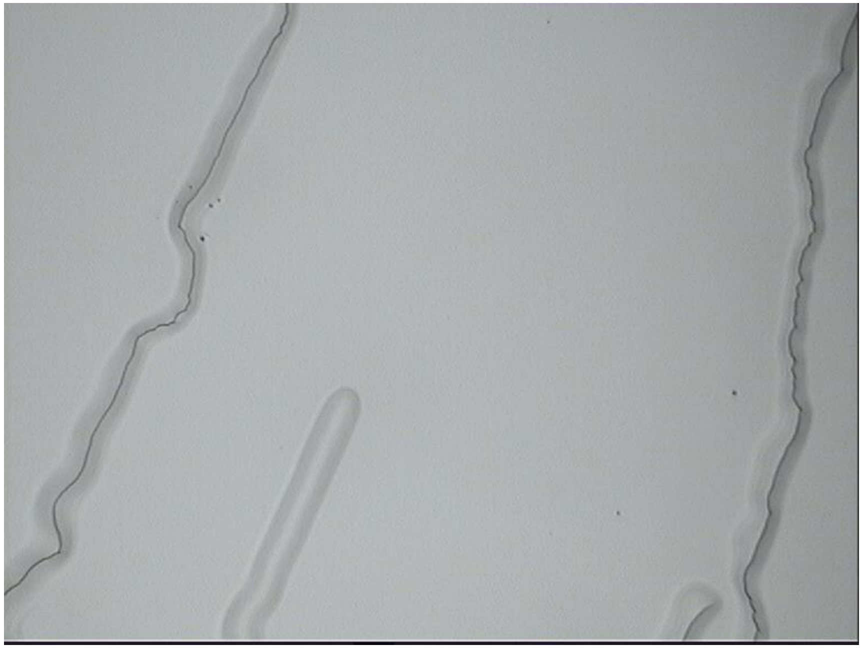


- **ZnO films (texture)**
- **Not possible on amorphous materials (SiO_2, SnO_2)**

F. Conchon, Pprime

II – Adhesion/fracture





Crack propagation – Energy release rate

- Energy release rate

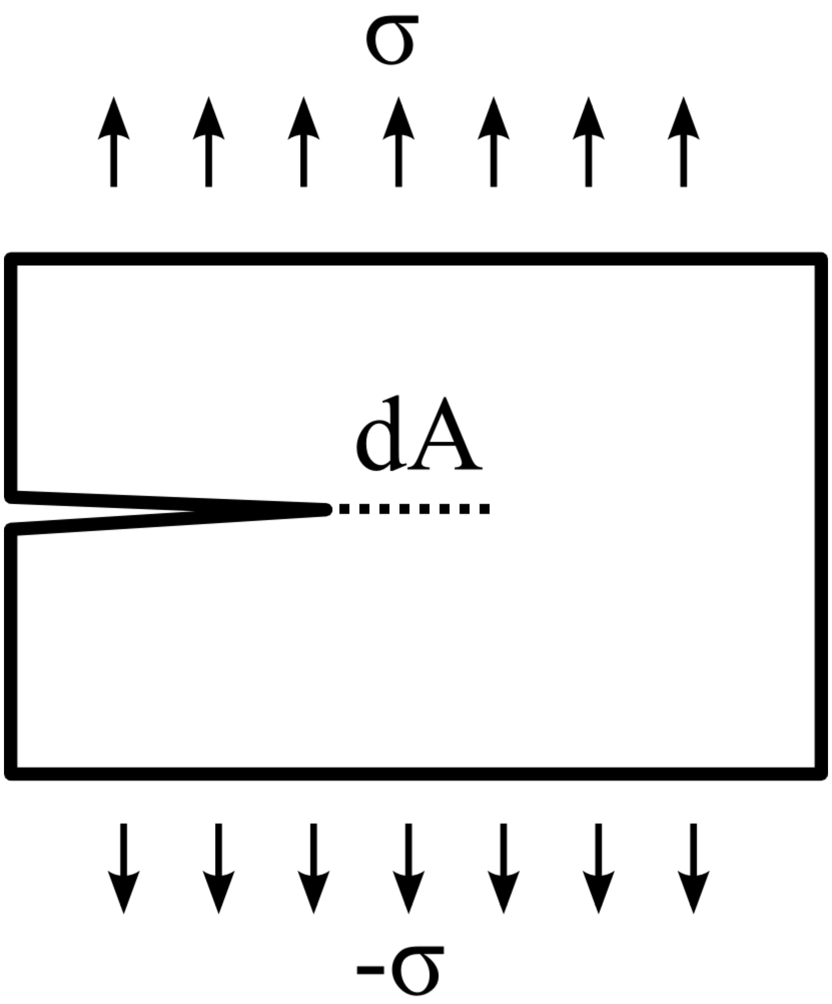
$$\mathcal{G} \equiv -\frac{d\mathcal{E}_{mech}}{dA}$$

A is the *fracture* area

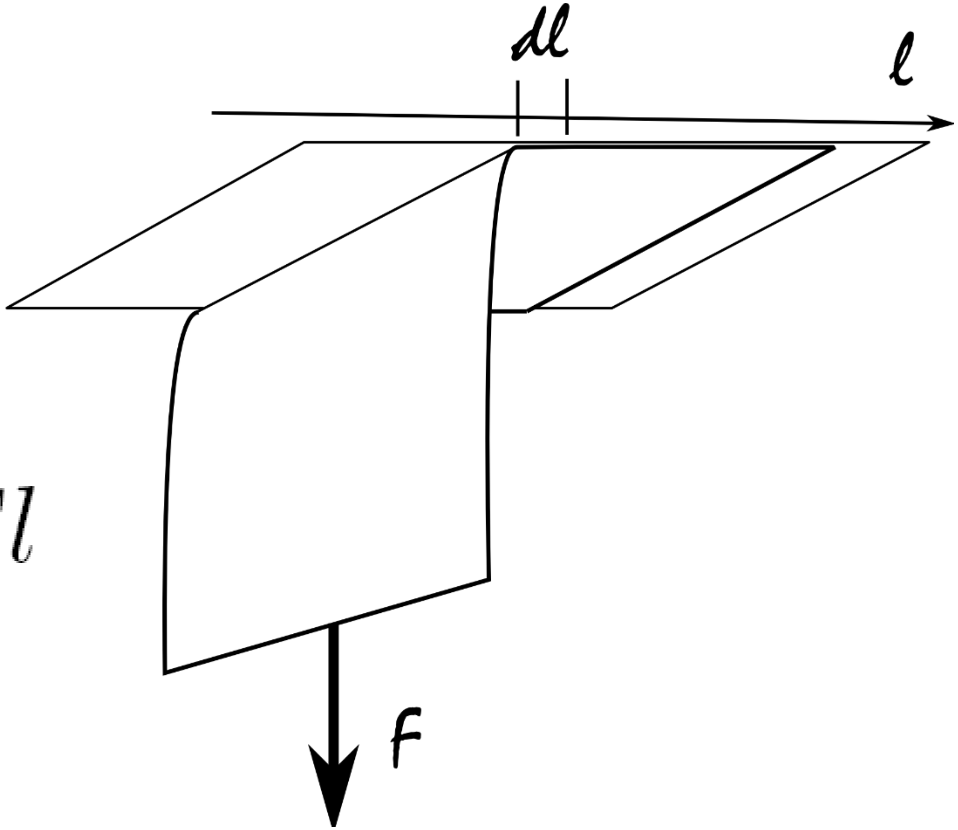
$$\mathcal{E}_{mech} = \mathcal{E}_{elas} + \mathcal{E}_{pot}$$

- Equilibrium

$$\mathcal{G} = w$$



90° Peel test

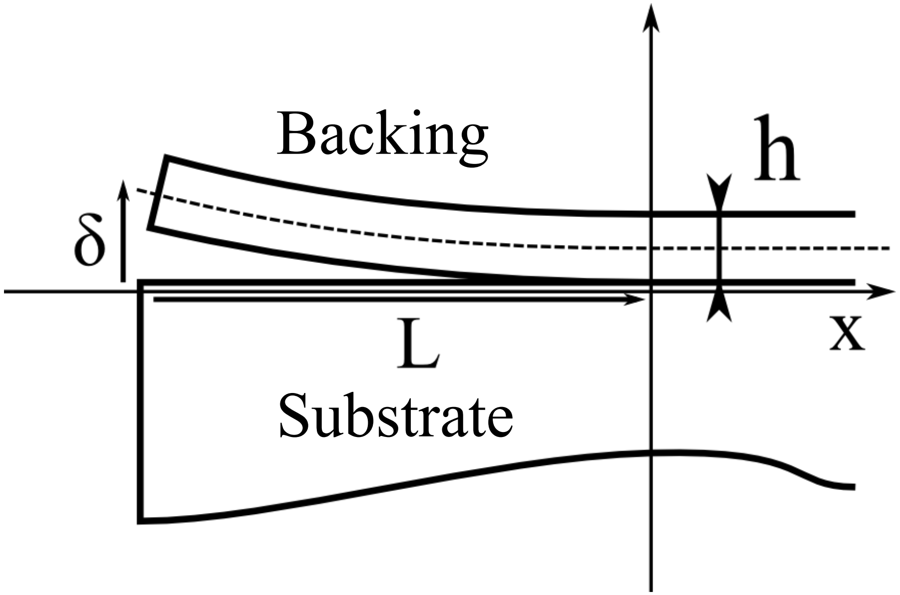


$$\mathcal{E}_{mech} = \mathcal{E}_{pot} = Fl$$

$$\mathcal{G} = -\frac{F}{b}$$

$$\boxed{|F| = wb}$$

The Double Cantilever Beam (DCB)



The Double Cantilever Beam (DCB)

- Linear system

$$F = \alpha\delta \quad \text{with} \quad \alpha = \frac{Eb}{4}\left(\frac{h}{L}\right)^3$$

- Energy release rate

$$\boxed{\mathcal{G} = \frac{3Eh^3}{8}\frac{\delta^2}{L^4}}$$

- or

$$\mathcal{G} = \frac{6}{Eh^3}L^2\left(\frac{F}{b}\right)^2$$

- Impacts the stability

(A)symmetric peel test – Elastic strip

- ethylene propylene rubber / PMMA + thin EPR film
- 10 cm wide / 12 mN / applied 60 mn
- a: no propagation / b: crack speed 2 µm/s

6. EXPERIMENTAL PROOF THAT STRESS DOES NOT DRIVE CRACKS

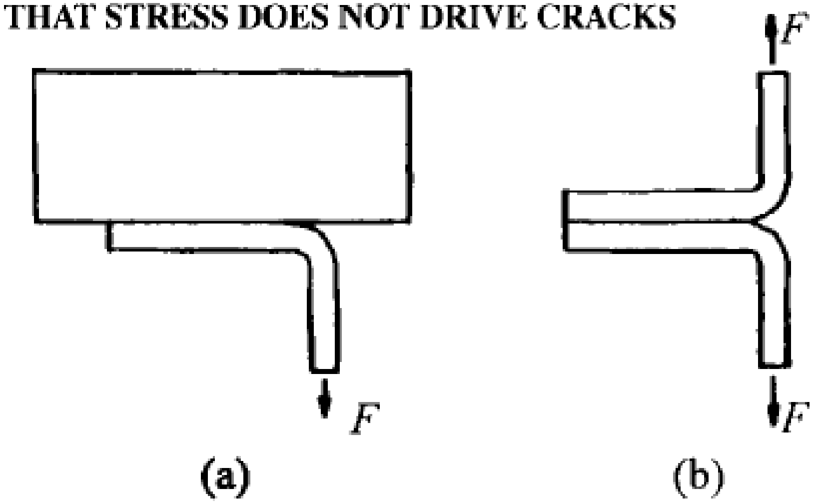
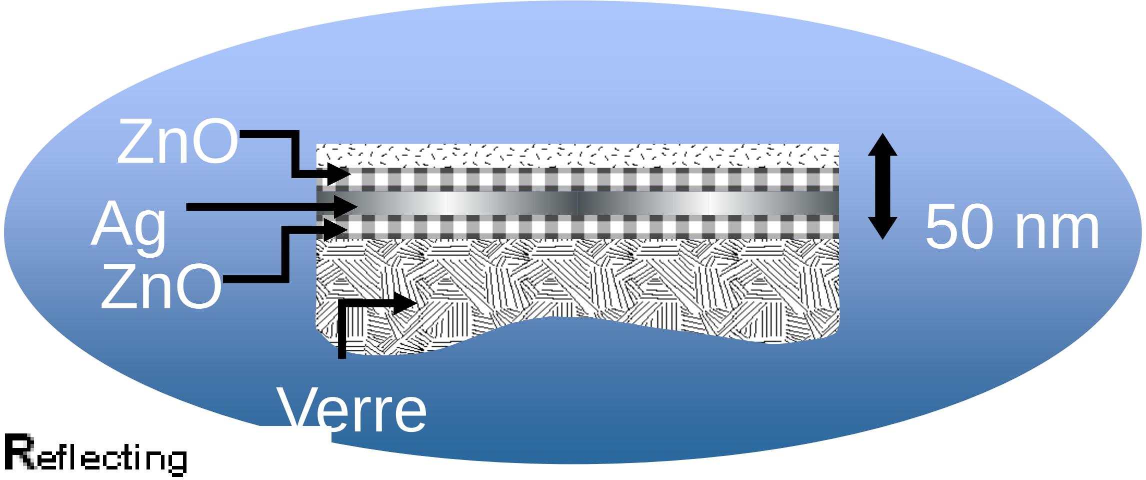


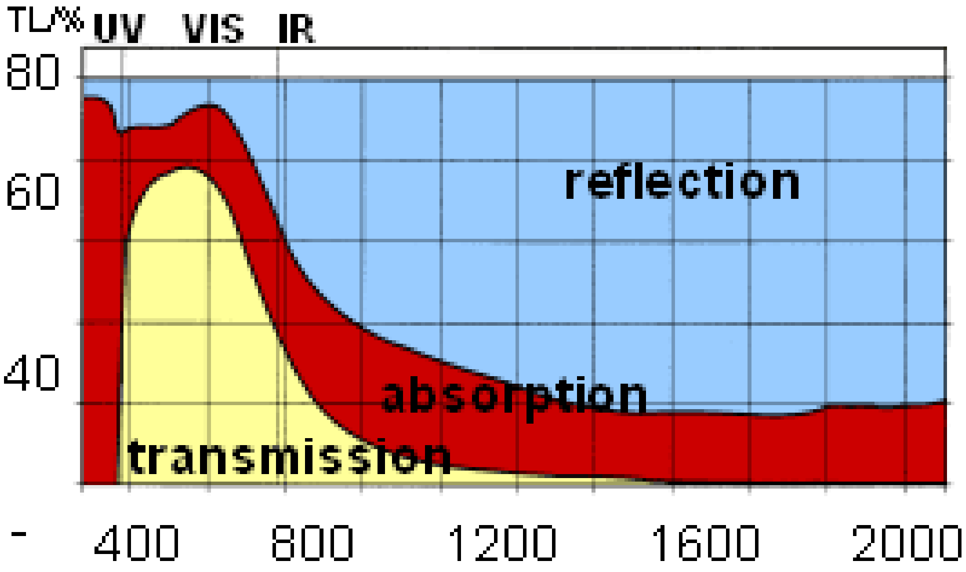
Figure 6. (a) Peel test with F just low enough to prevent cracking; (b) peel test at the same stress now fractures.

K. Kendall, J Adhes Sci Technol 8 (1994) 1271

Optical functionalisation

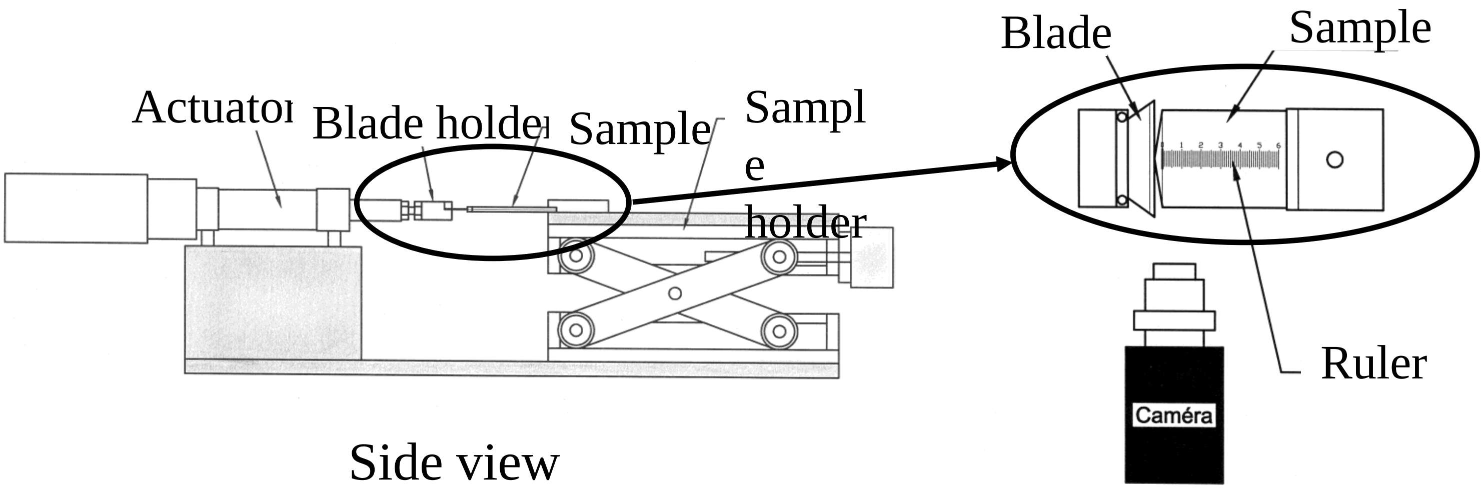


SGS THERMOCONTROL® Reflecting



DCB adhesion energy measurement

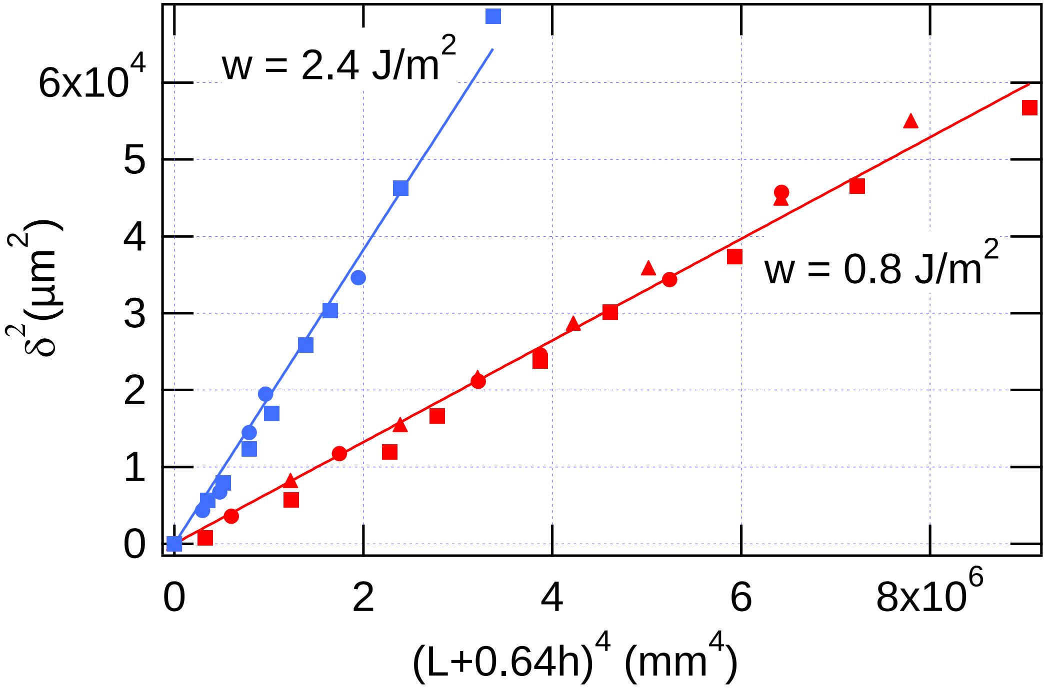
experimental set-up



Side view

Top view

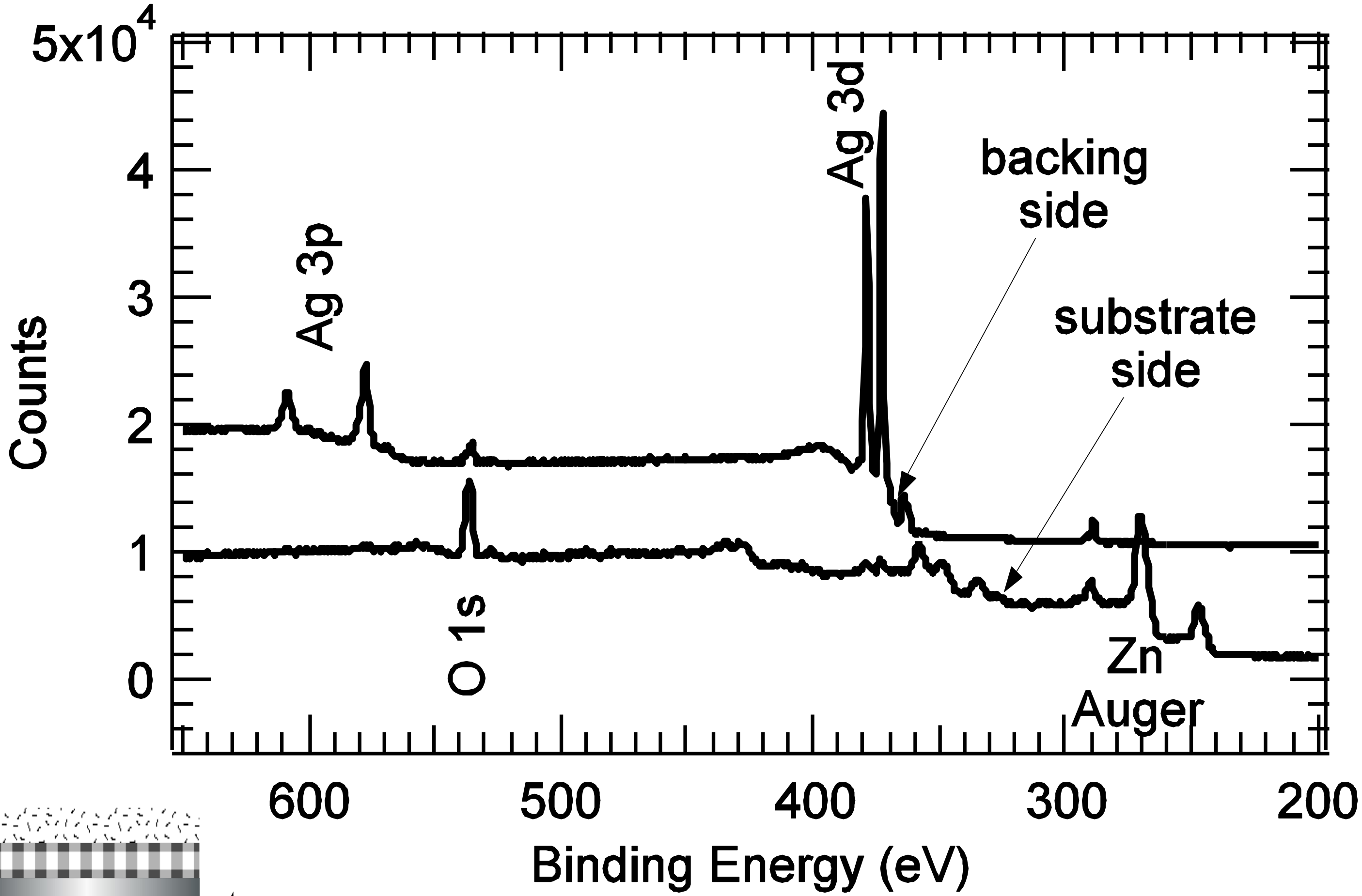
DCB – results

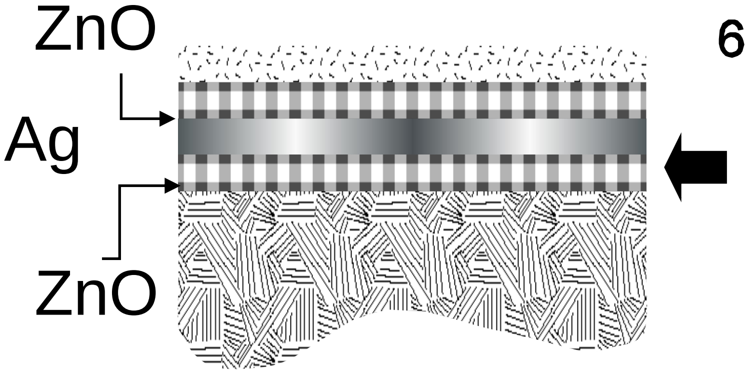


Obreimov, Kanninen

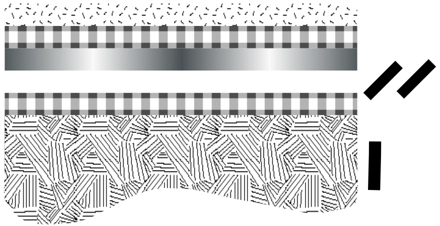
Barthel et al. Thin Solid Films, 2005

Indentification of the interfaces - XPS



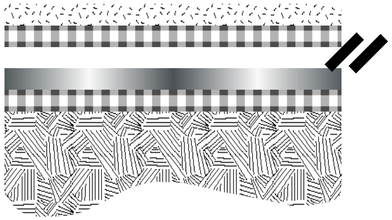


Impact of underlayer



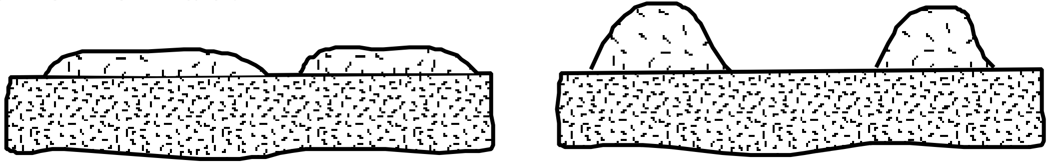
// is the locus of failure

1	Glass / Si_3N_4 // Ag / ZnO	**0,7 J/m^2** ± 0,2 (2 s.)
2	Glass / ZnO // Ag / ZnO	**1,5 J/m^2** ± 0,2 (2 s.)
3	Glass / TiO_2 / Ag // ZnO	**2,1 J/m^2** ± 0,7 (3 s.)
4	Glass / SnO_2 / Ag // ZnO	**2,4 J/m^2** ± 0,1 (3 s.)



Deposition is not at equilibrium

Silver on oxide:



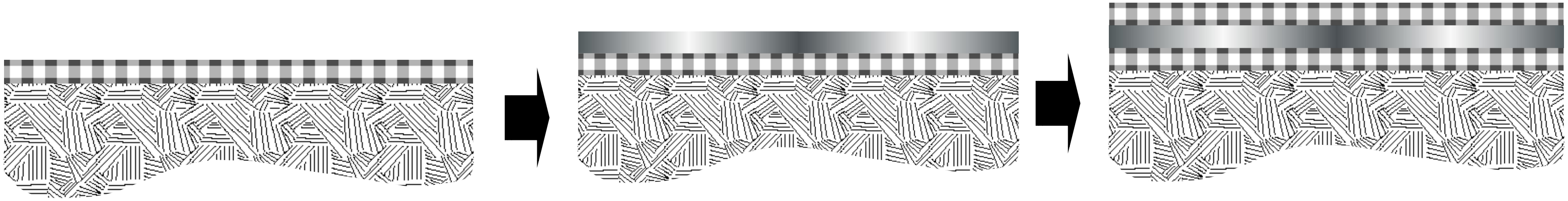
Ambiant Temperature

High

Temperature

Ag does not wet ZnO; ZnO wets Ag

History matters:



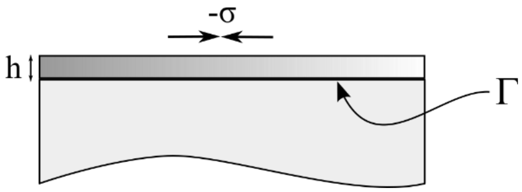
cf also Lin and Bristowe PRB 2007


PL1 = 52.927 µm
2010/06/18 14:28:02
Lentille MX(G)-5040Z : Normal : x350
Echelle 871.430 µm
Resolution 0.545 µm
200 µm

Elementary fracture mechanics

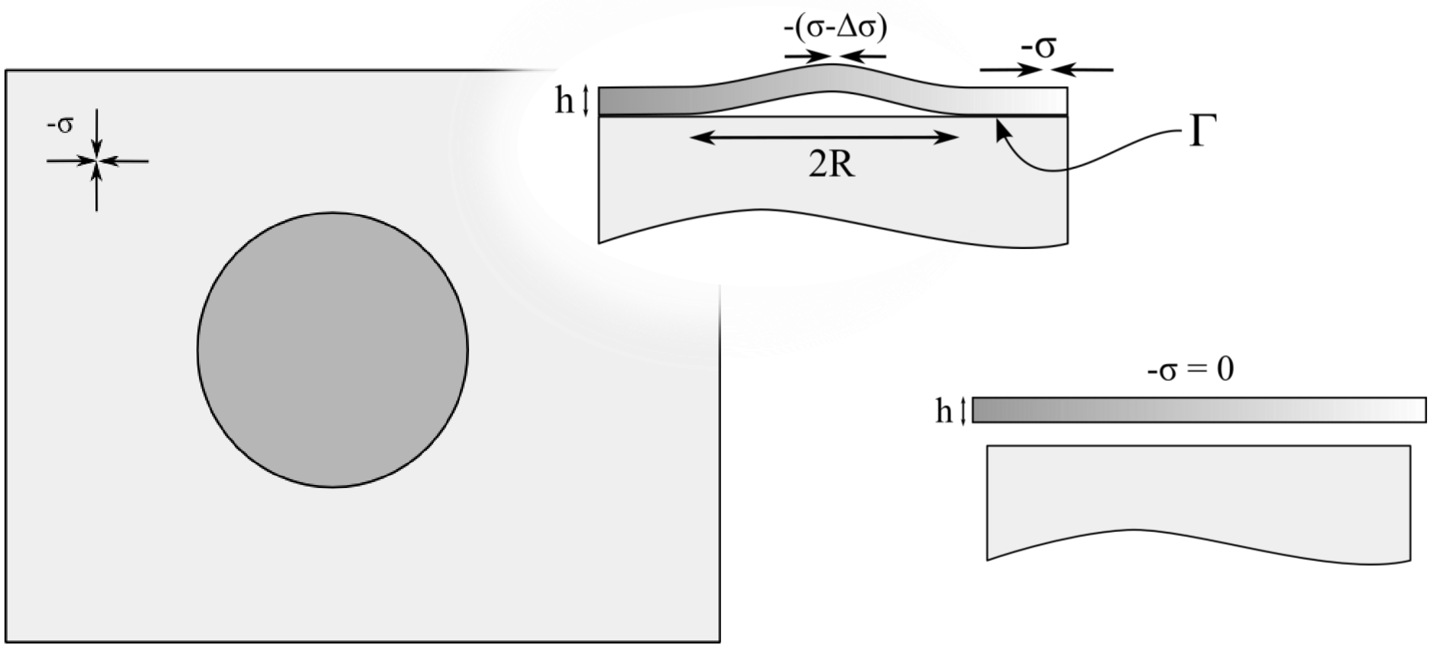
Available elastic energy:

$$G_0 = \frac{1}{2}h\frac{\sigma^2}{E}$$



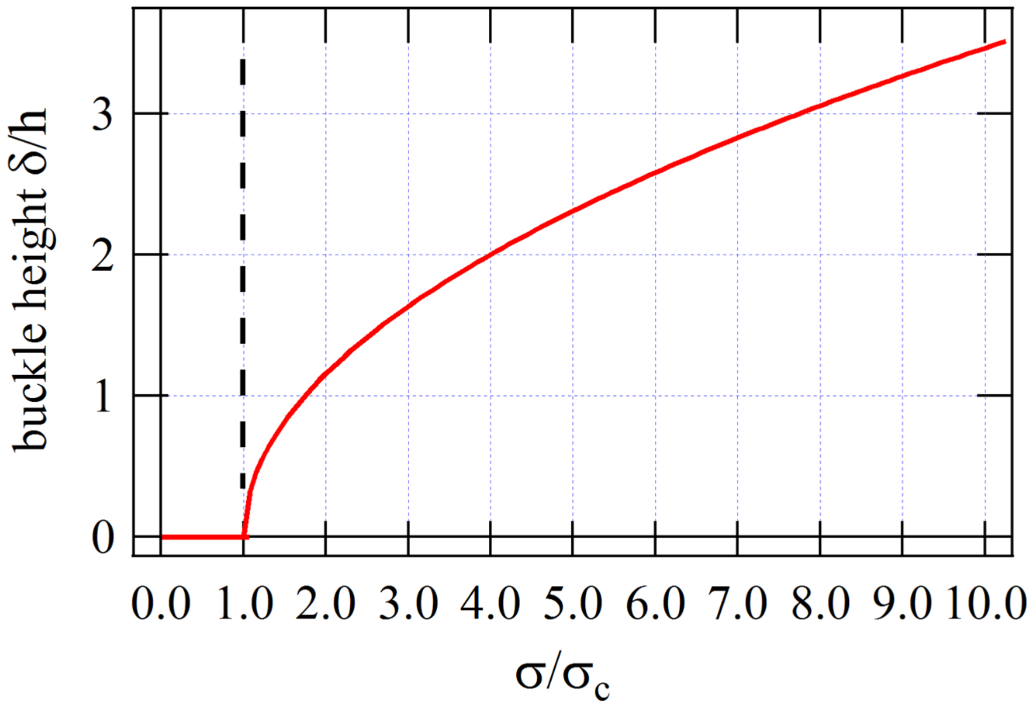
Adhesion energy:

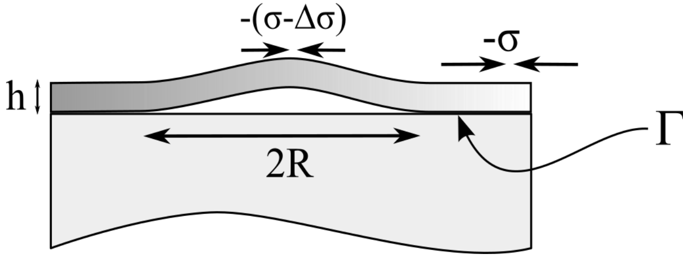
$$\Gamma$$



Equibiaxial film stress

Threshold – buckling stress – circular buckle



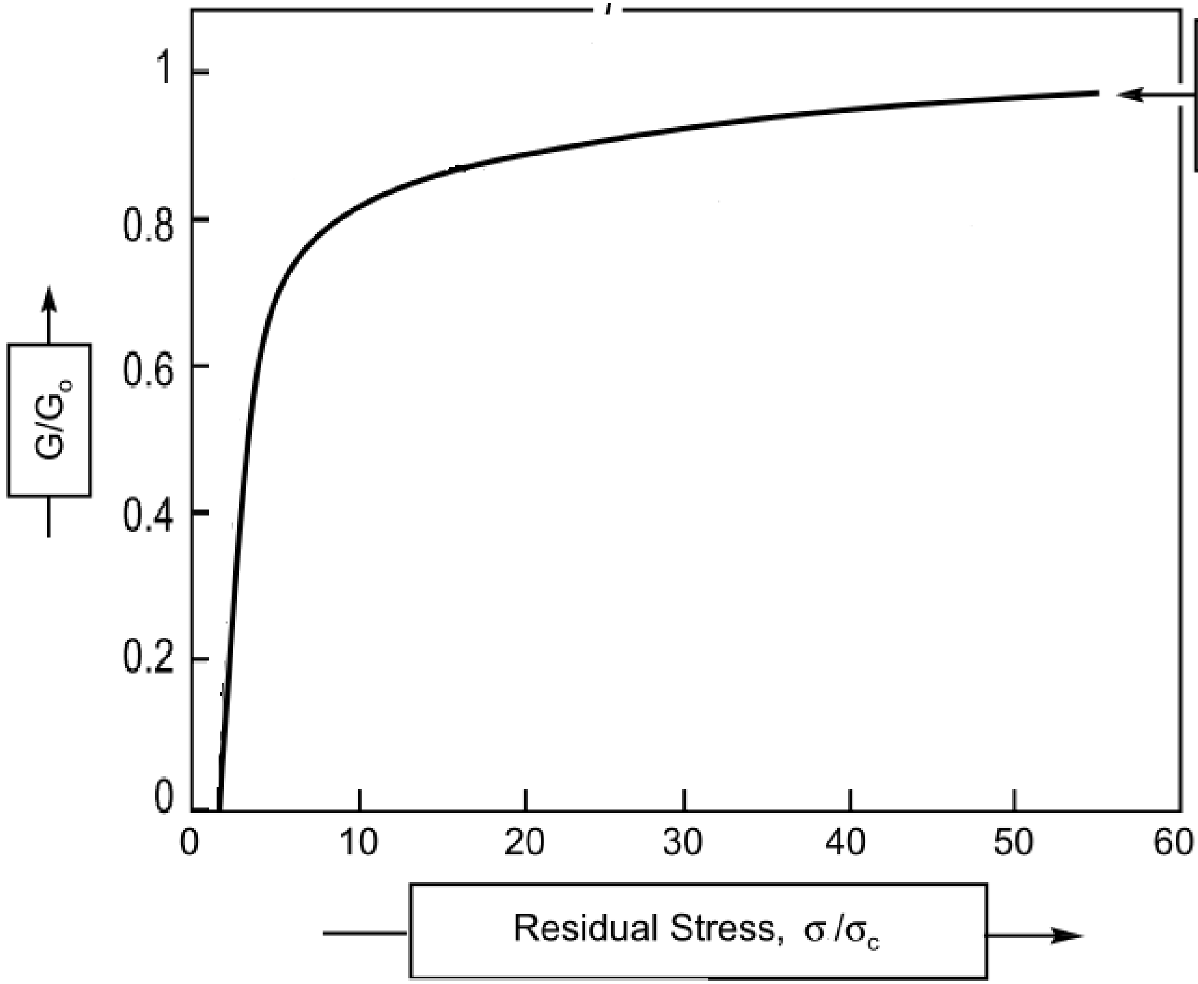


Buckling stress

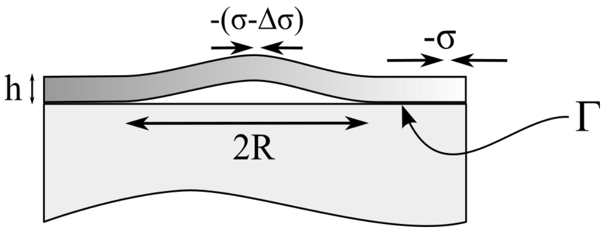
$$\sigma_c = \frac{\mu^2 D}{R^2 h}$$

$$D = \frac{Eh^3}{12(1-\nu^2)}$$

Circular blister – Energy release rate



Circular,
Radius R



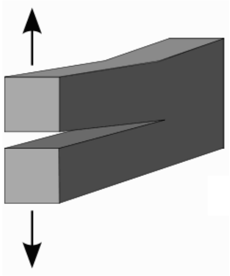
Buckling stress

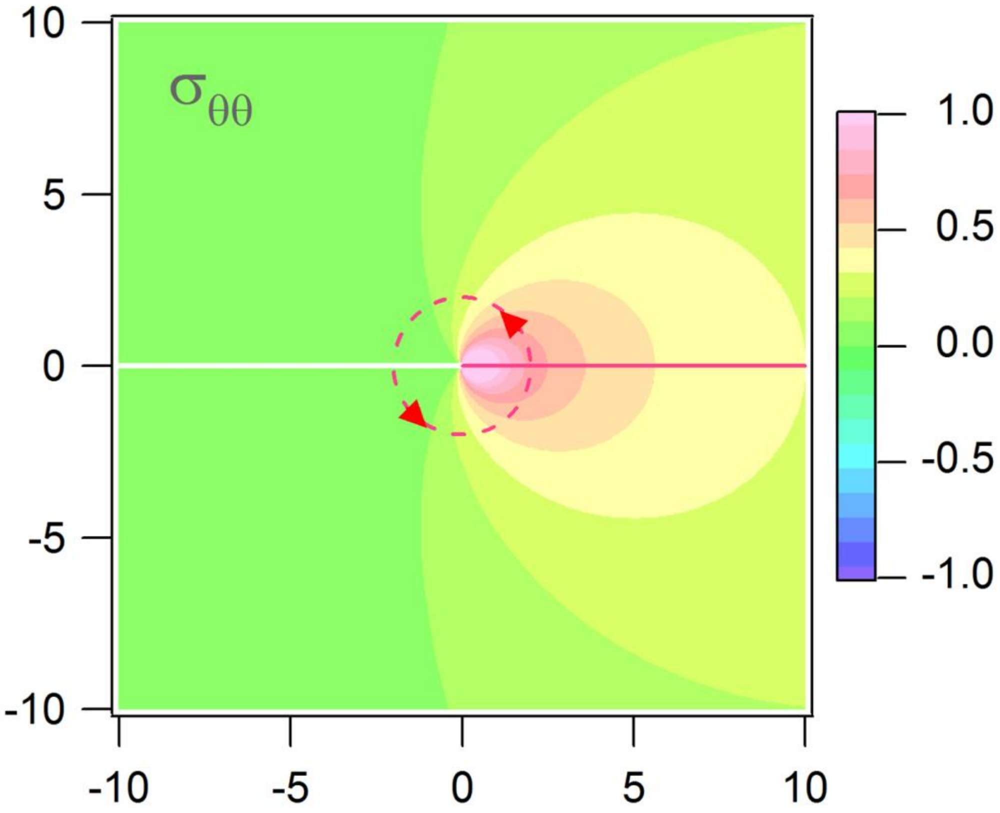
$$\sigma_c = \frac{\mu^2 D}{R^2 h}$$

$$D = \frac{Eh^3}{12(1-\nu^2)}$$

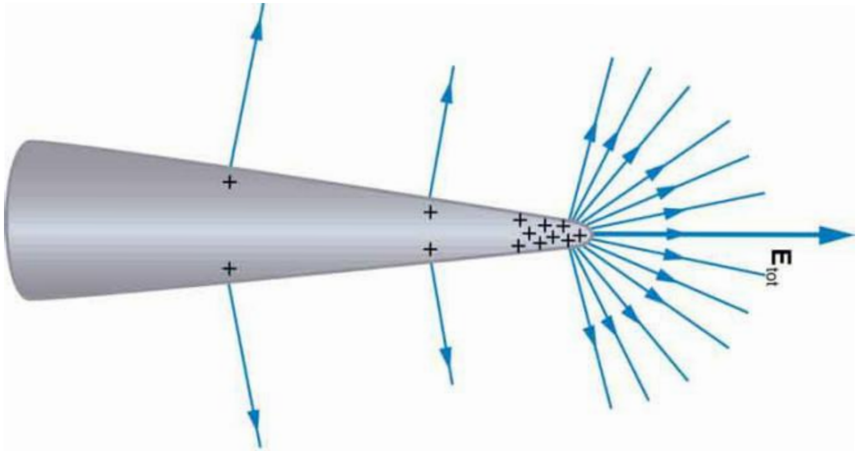
after Moon et al. JMPS (2002)

Fracture – mode I



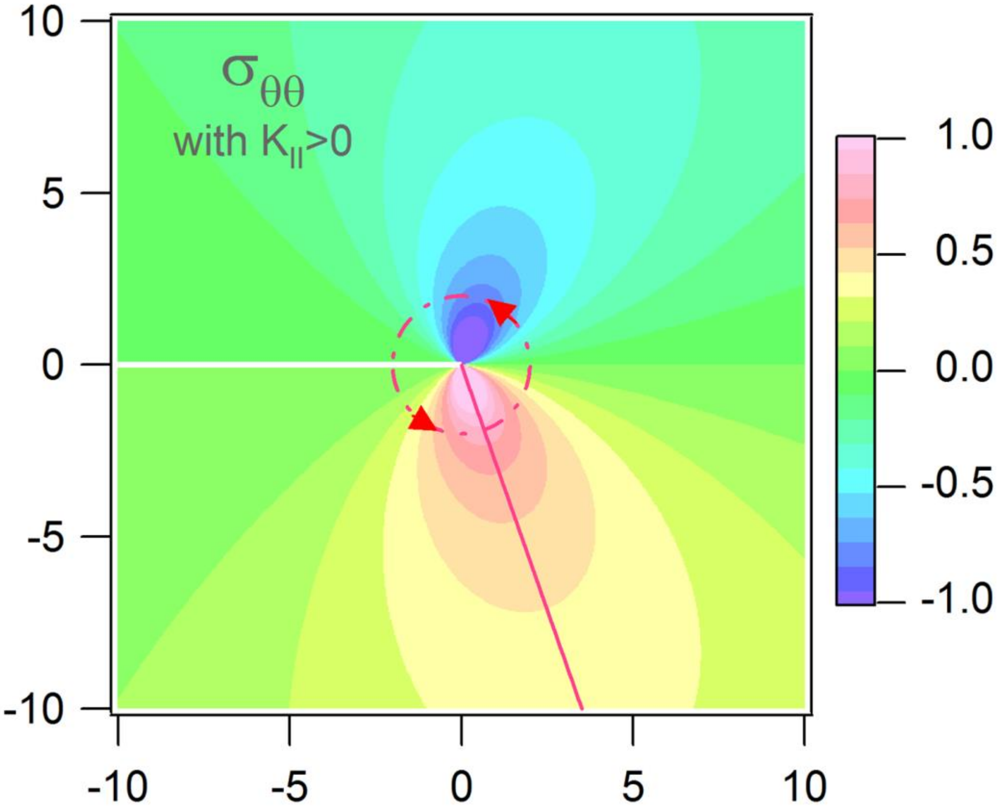


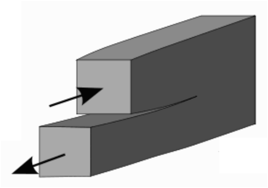
Electrostatic – curvature effect



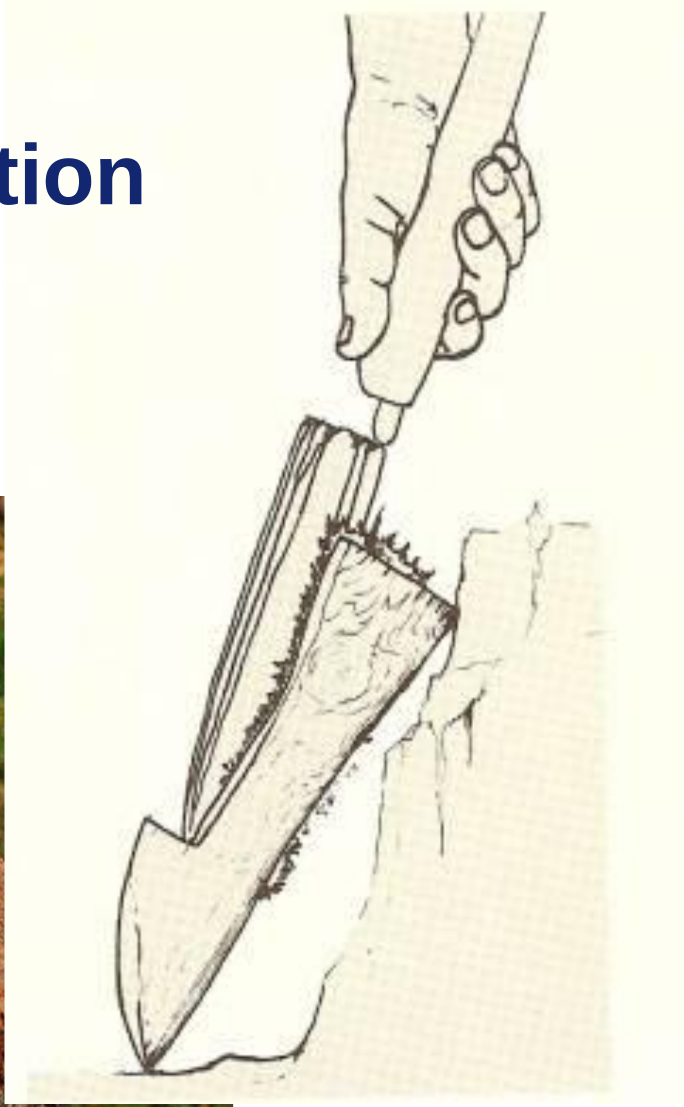
https://courses.lumenlearning.com/physics/chapter/18-7-conductors-and-electric-fields-in-static-equilibrium/

Fracture – mode II





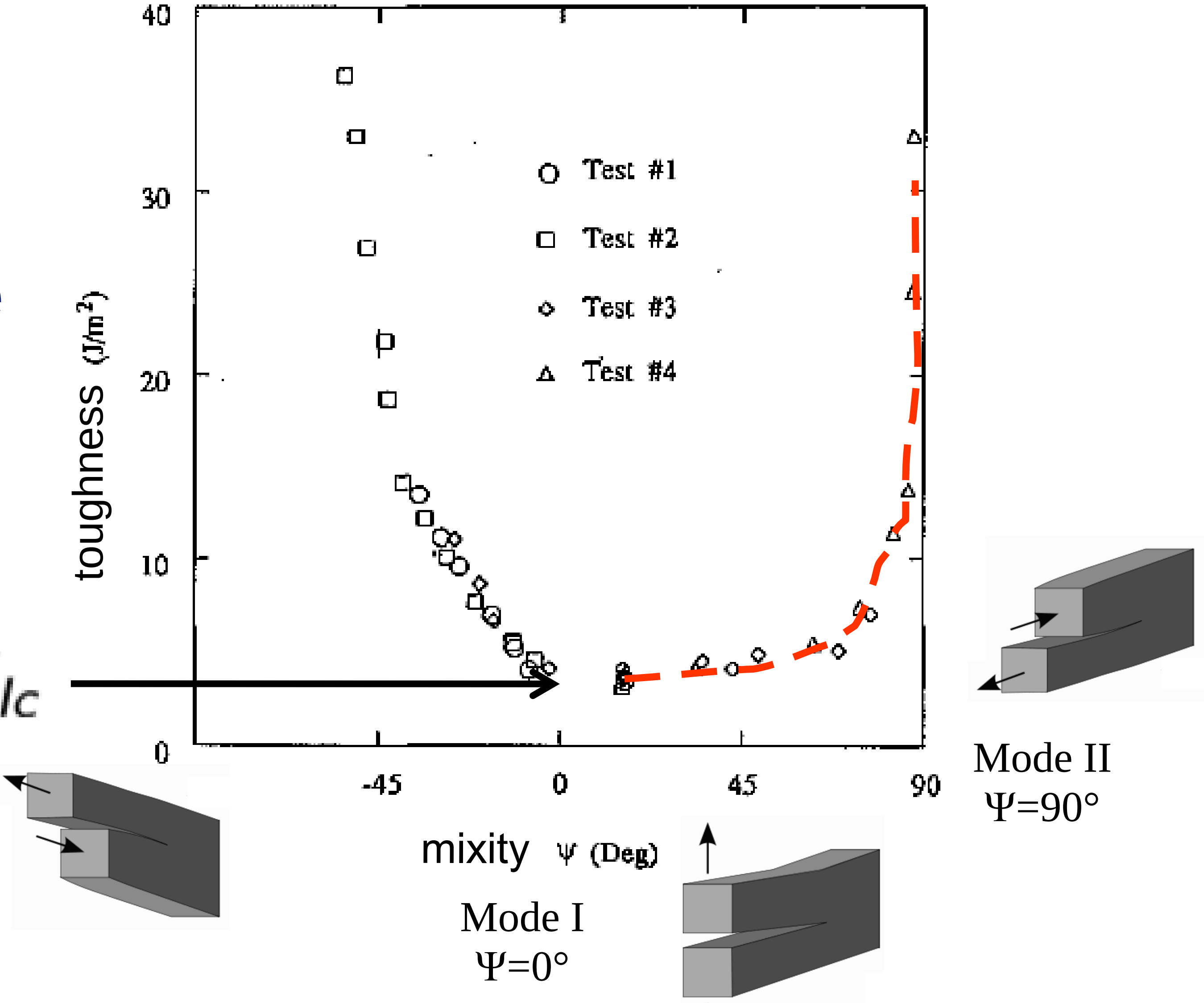
Fracture – crack path selection



https://exarc.net/ark:/88735/10543

J. Pelegrin (CNRS silver medal in 2017)

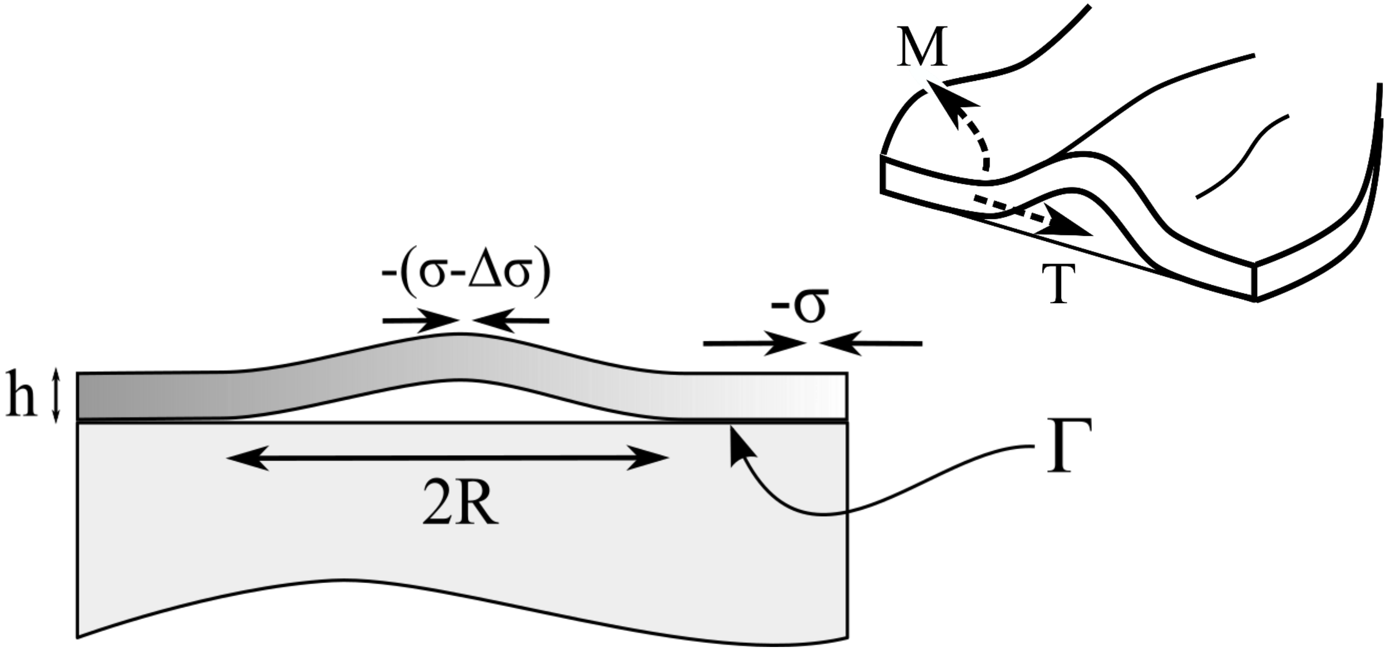
Interfacial toughness depends upon mode mixity



Interfacial toughness in mode I G_{Ic}

K.M. Liechti and Y. S. Chai, Journal of Applied Mechanics 59 ,295 (1992)
J.W. Hutchinson and Z. Suo, Adv. Appl. Mech. 29, 63 (1992)

Blister – impact of mode mixity



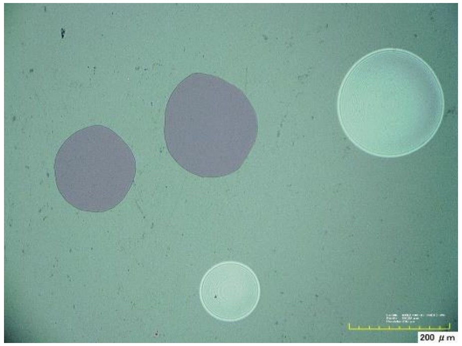
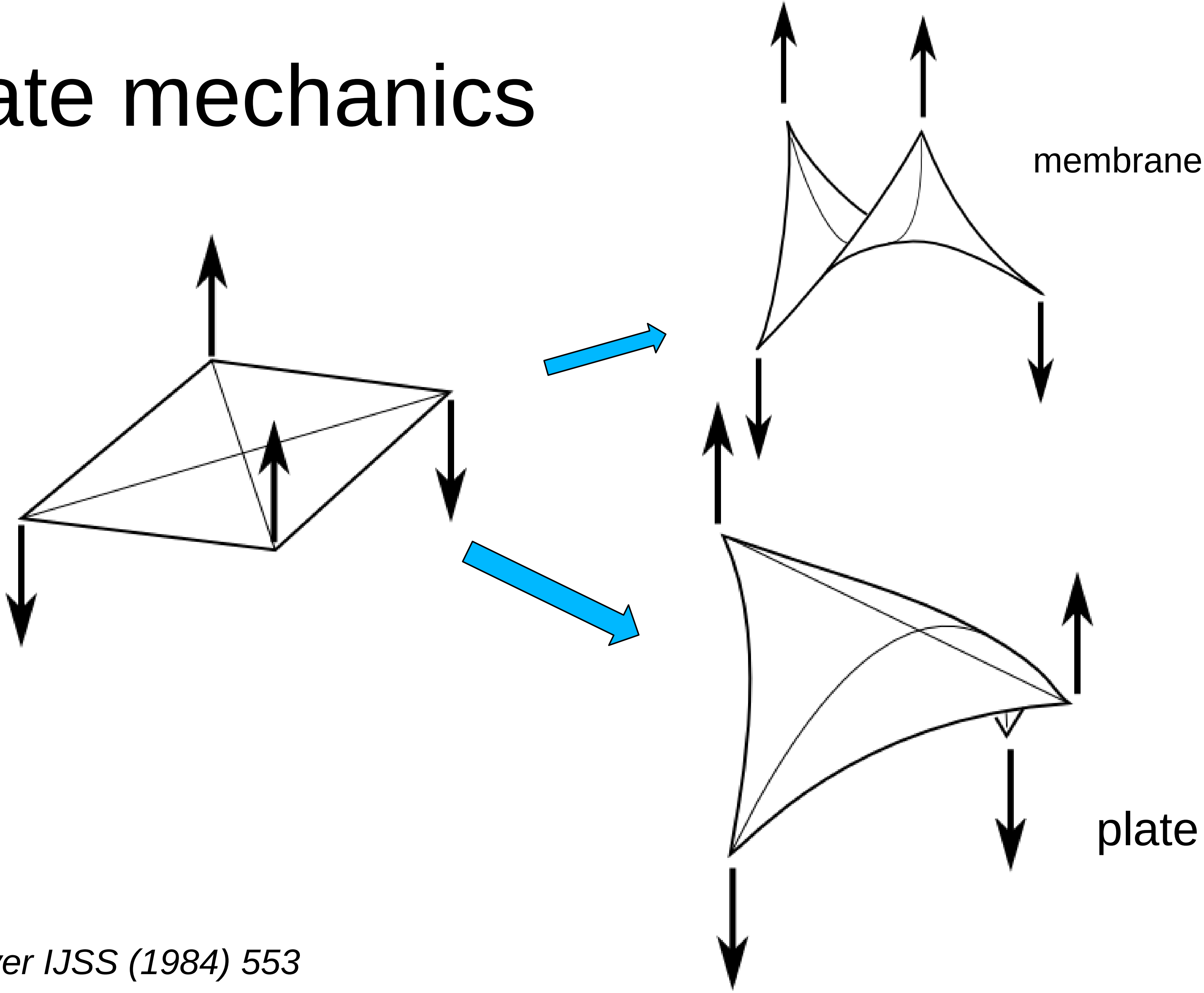
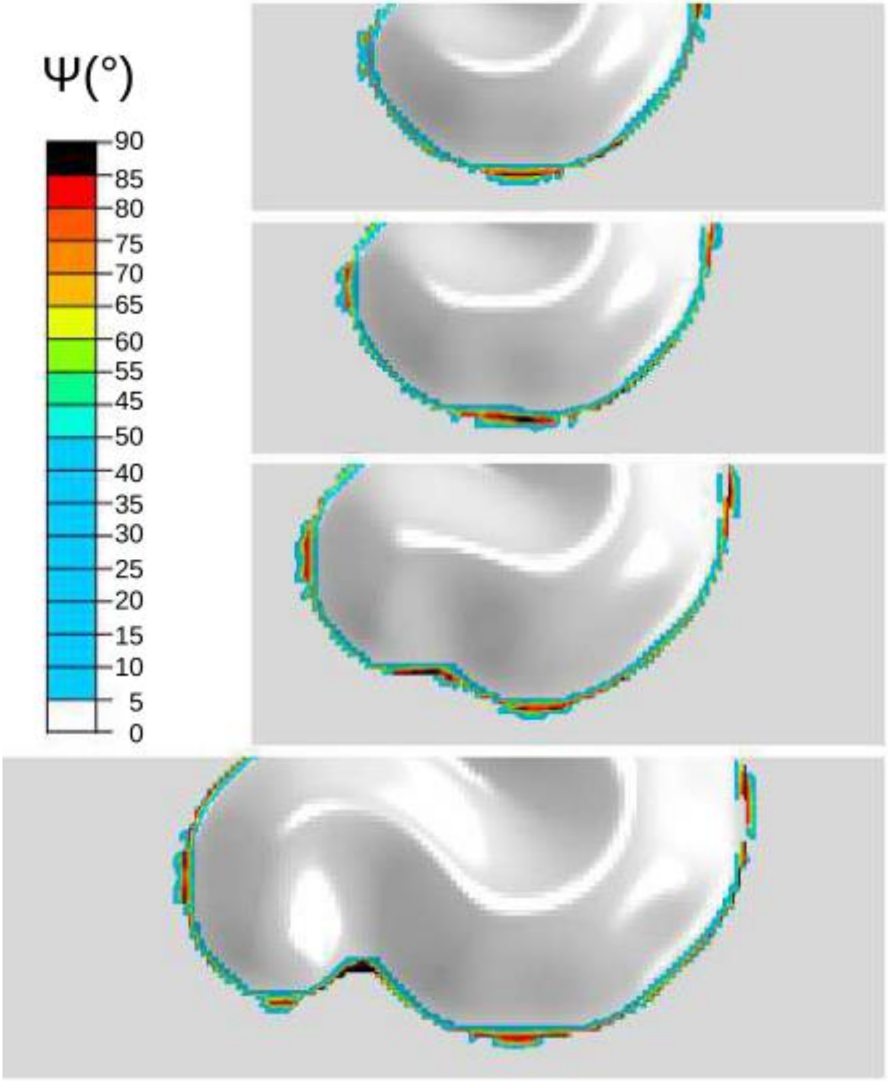


Plate mechanics



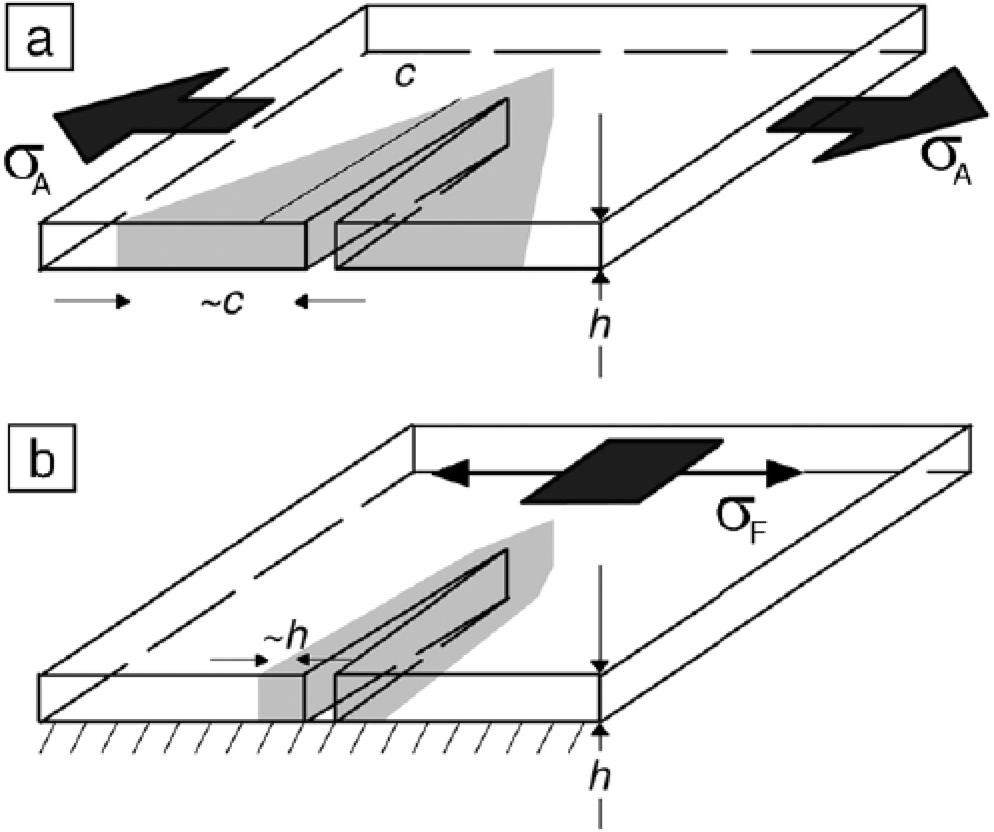
Hyer IJSS (1984) 553

Pinning by local conformation of the buckle



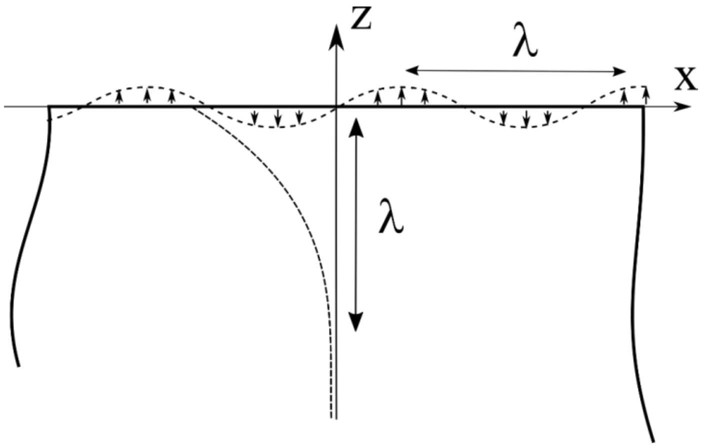
Faou et al. PRL (2012)

Channel cracks



Cook 2002

Saint-Venant principle



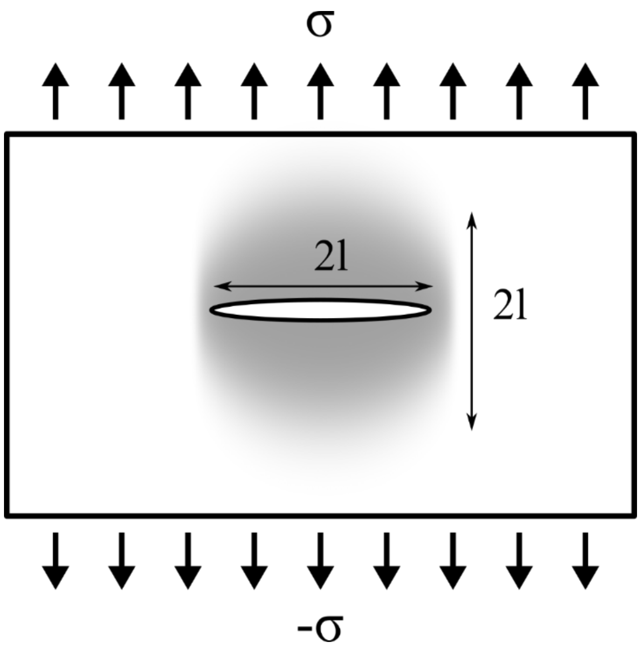
$$\Delta u = 0$$

$$u(x,z) = e^{ikx}\tilde{u}(z)$$

$$\Delta u(x,z) = \left(\frac{\partial}{\partial x^2} + \frac{\partial}{\partial z^2}\right) u(x,z) = e^{ikx}\left(-k^2 + \frac{\partial}{\partial z^2}\right)\tilde{u}(z) = 0$$

$$\tilde{u}(z) \propto e^{\pm kz}$$

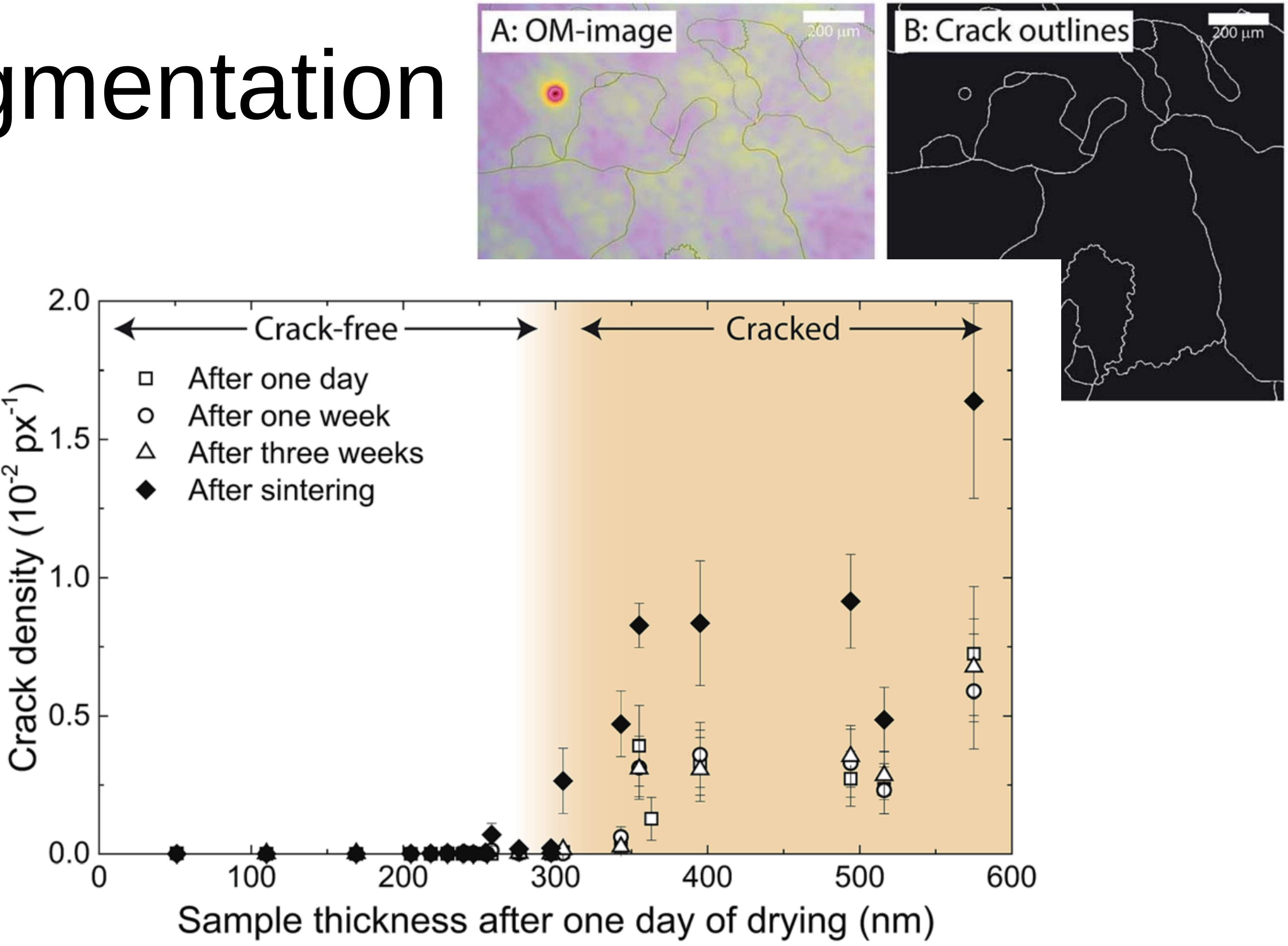
Energy release rate for a Griffith crack



$$\mathcal{E}_{\mathbf{tot}} \simeq\simeq l^2 b \left(\frac{\sigma^2}{E} \right)$$

$$G_{\mathrm{Griffith}} \simeq l \left(\frac{\sigma^2}{E} \right)$$

Fragmentation

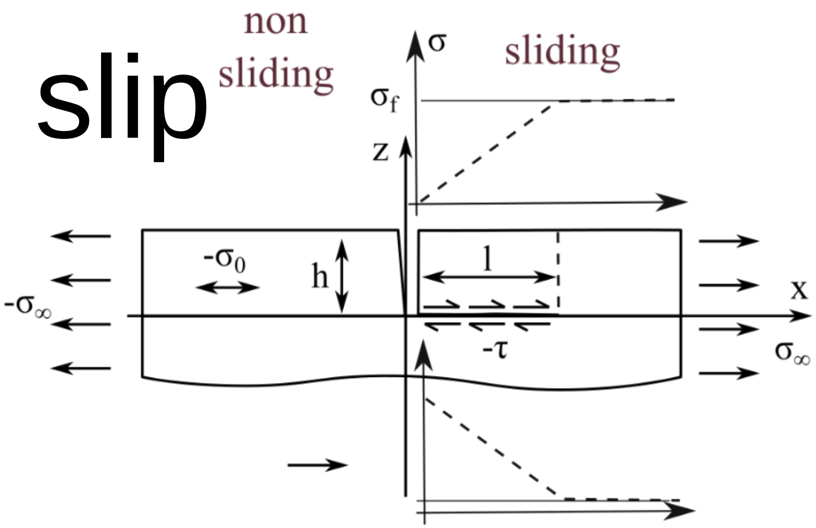


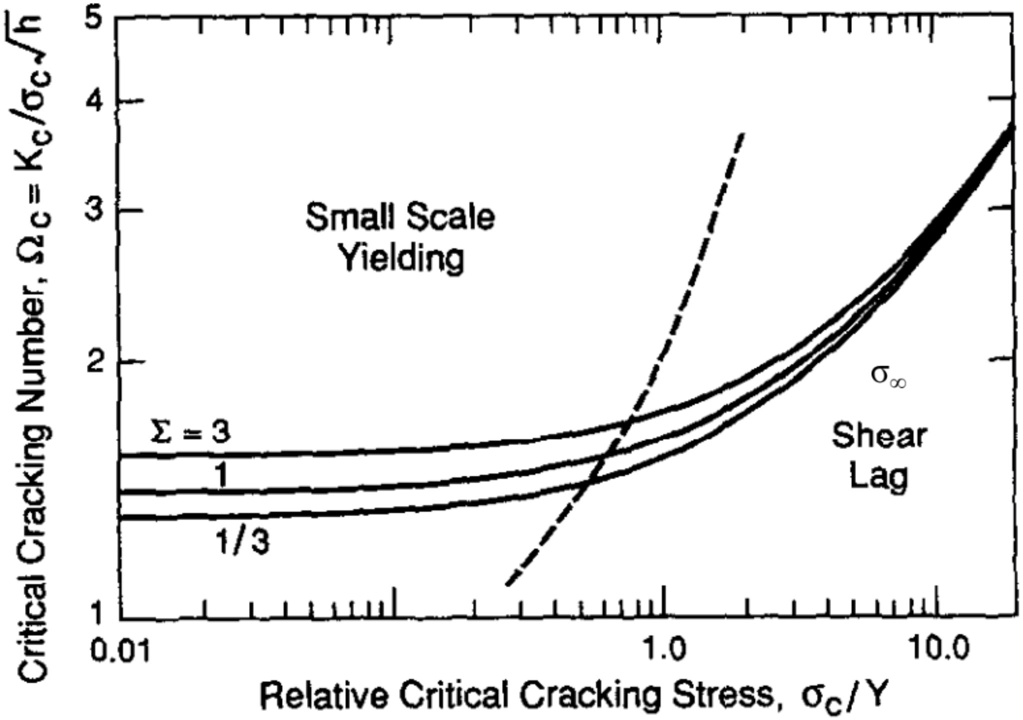
Sol-gel silica films

Kappert et al. Soft Matter 2015

Back to interfacial rupture

Relaxation through slip





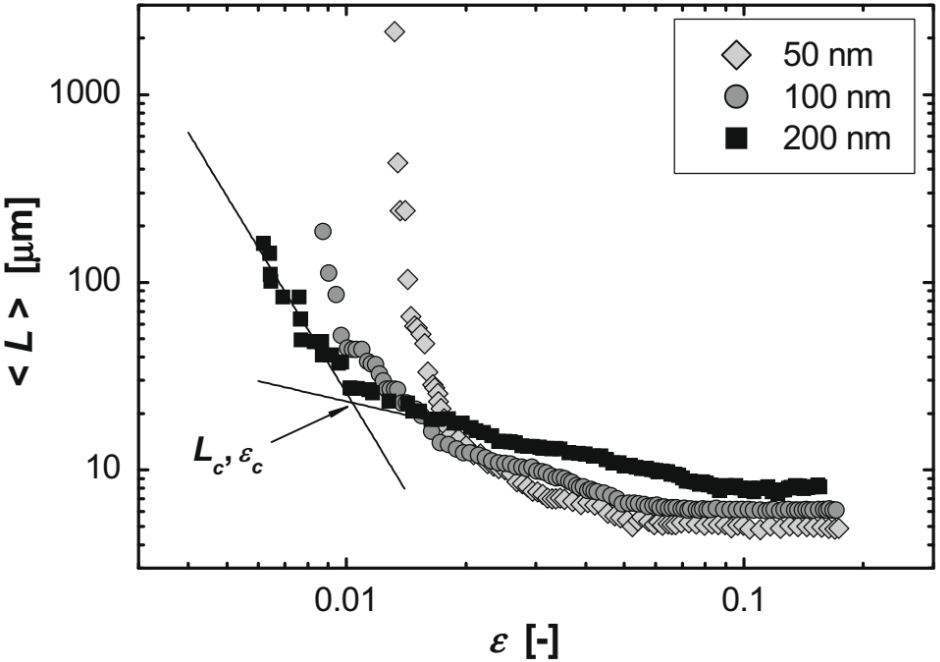
$$l = \frac{\sigma_f h}{\tau}$$

$$\Delta U \simeq -\frac{\sigma^2 h^2}{E^f}\left[\frac{\sigma}{3\tau} + \pi F(\Sigma)\right]$$

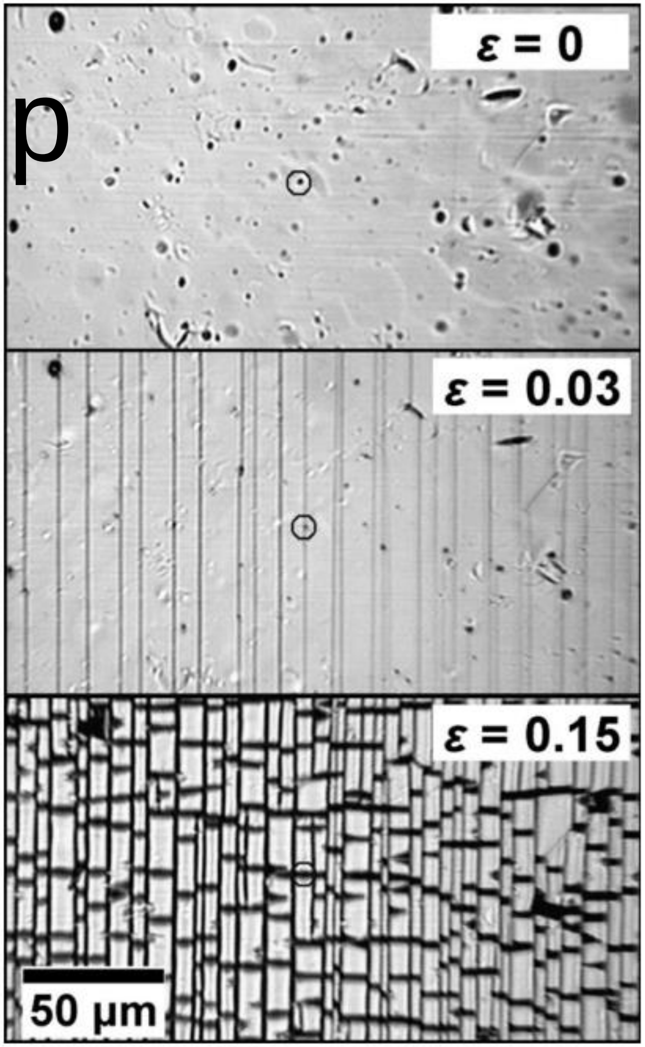
$$G \equiv -\frac{\Delta U}{h} \simeq -\frac{\sigma^2 h}{E^f}\left[\frac{\sigma}{3\tau} + \pi F(\Sigma)\right]$$

Hu & Evans Acta metall. 37 (1989) 917

Relaxation through slip

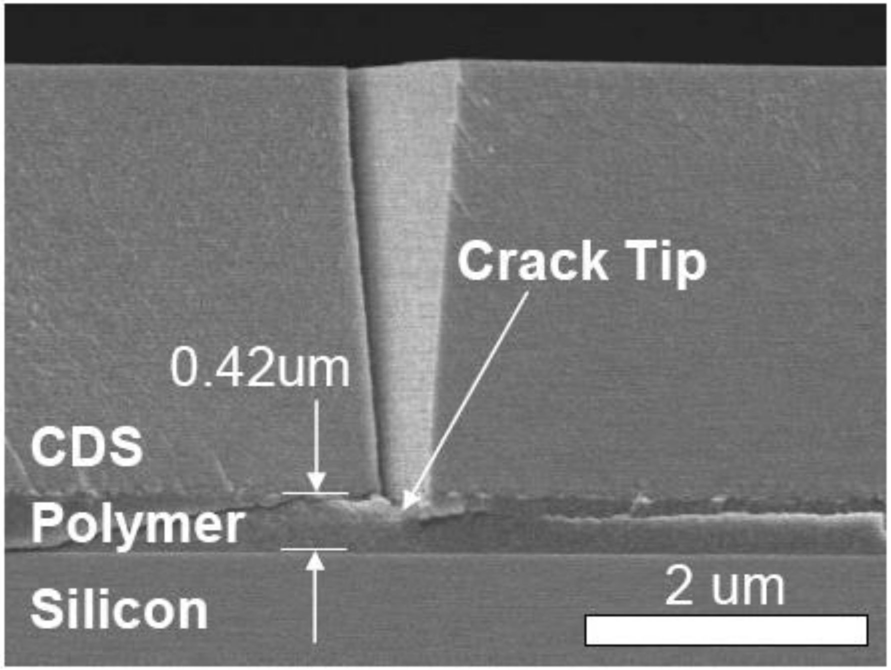


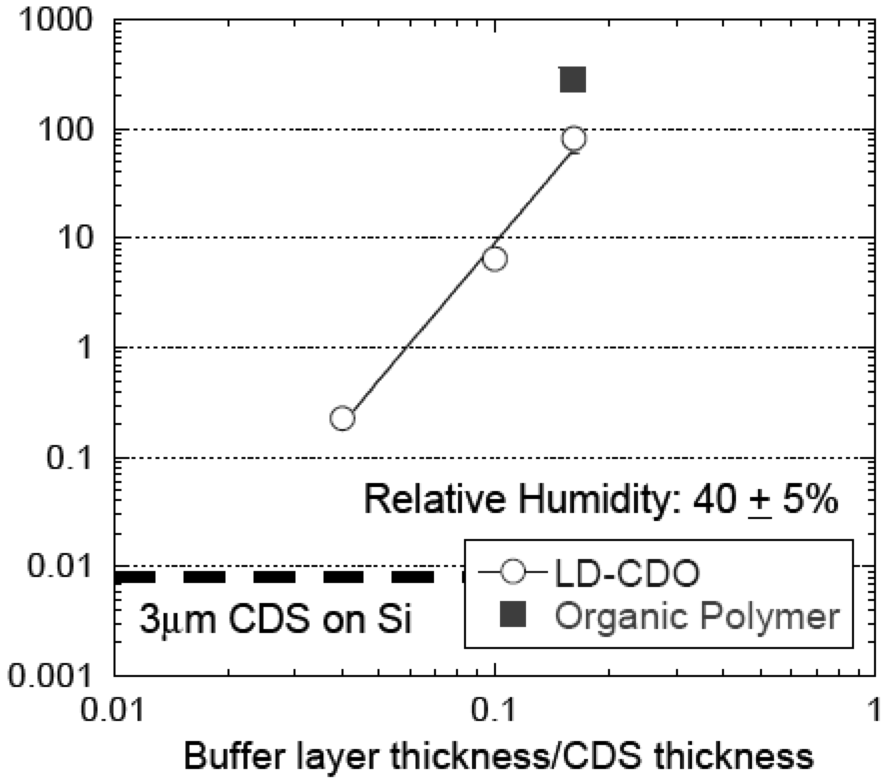
Mean fragment length



S. Frank et al. / Acta Materialia 57 (2009) 1442–1453

Coupling to the substrate





Tsui & Vlassack