Nanomechanical Testing in Materials Research and Development V

Proceedings

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About the plastic response of silicate glasses at the micronscale

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Authors

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(Perfect) plastic flow of silica glass ?

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1 : MINES Saint-Étienne

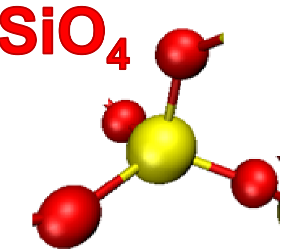
2 : EMPA Materials Science & Technology

3 : SAINT-GOBAIN

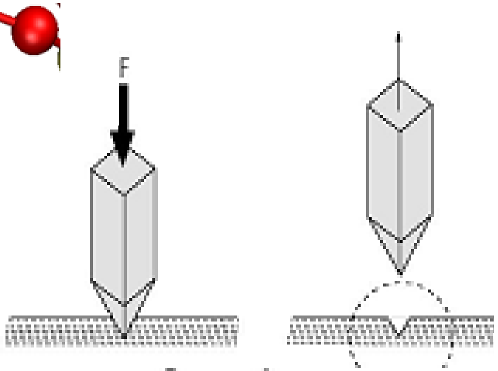
4 : ESPCI ParisTech

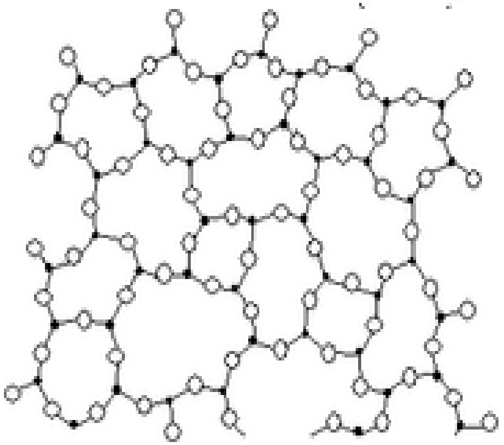
INSPIRING INNOVATION INNOVANTE PAR TRADITION

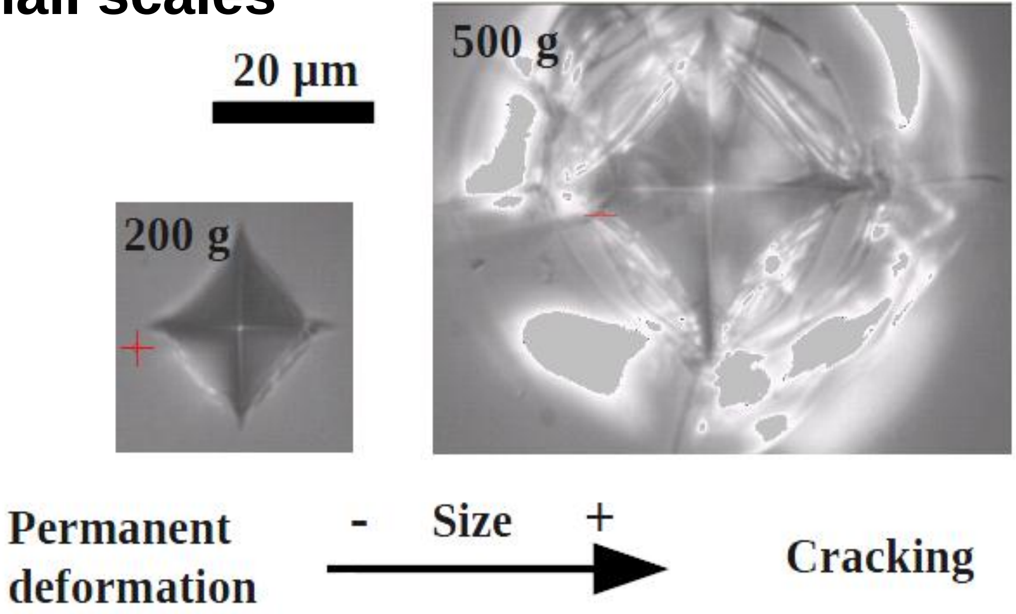
Plastic flow of what ???



- **Silicate glasses : Macroscopically brittle but … ductile at small scales**







An old story : first clues

- **1949 :** E.W. Taylor**,** Plastic deformation of optical glasses, ***Nature***.
- **1963 :** D.M. Marsh, Plastic flow in glass, ***Proc. R. Soc. Lond. A***

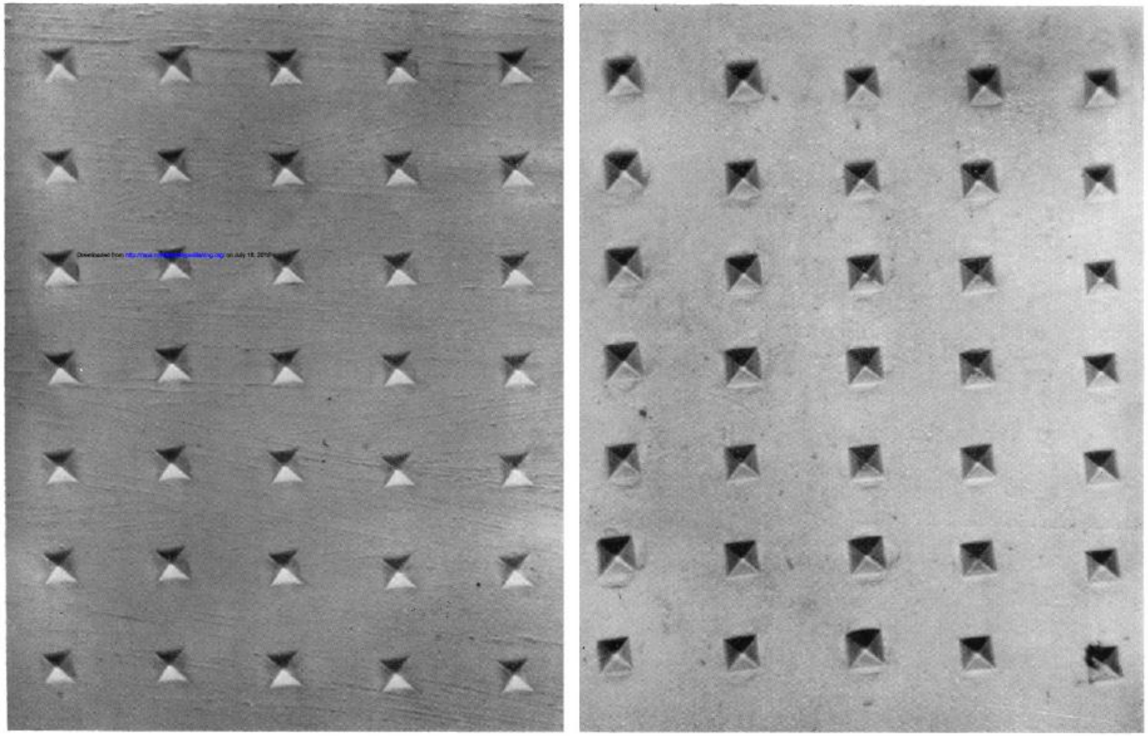


FIGURE 2. Comparison of Vickers hardness impressions in glasses and in metals. (*Left*) in soda glass. (*Right*) in a bearing steel.

An old story : densification of glass

- **1968 :** J. E Neely, J.D. Mackenzie, Hardness and low-temperature deformation of silica glass, **J. of Mat. Sci.**

  F.M. Ernsberger, Role of densification in deformation of glasses under point loading, ***J. Am. Ceram. Soc***.
- **1970 :** K.W. Peter. Densification and flow phenomena of glass in indentation experiments. **J. Non-Cryst. Sol.**

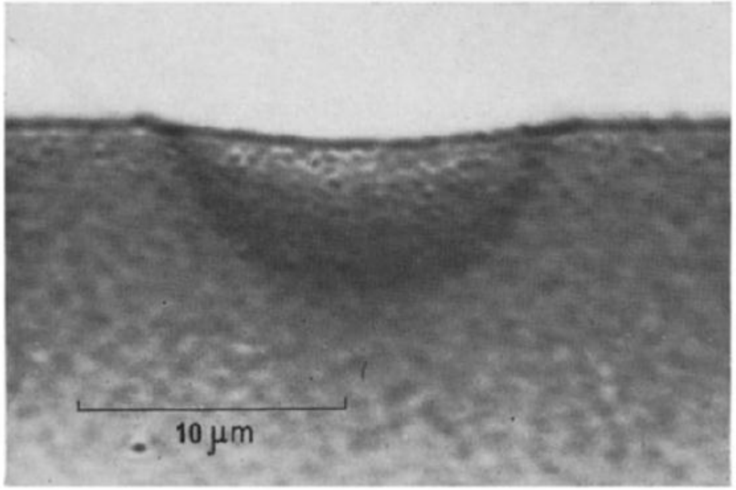


Fig. 1. Profile of a ball indentation on plate glass. Radius of curvature of the indenter $\rho = 20$ μm; load $L = 100$ g. Light microscope, top illumination.

→ "**Hardness of silica** glass (as determined by this method) is thus defined as a **resistance** of the material **to densification**"

An old story : glass normality

- **1979 :** A. Arora, D.B. Marshall, B.R. Lawn, and M.V. Swain, Indentation deformation/fracture of <u>normal and anomalous</u> glasses. **Journal of Non-Crystalline Solids**.

Soda-lime glass

<u>Normal Glasses :</u> pure **shear flow**

-> Shear bands

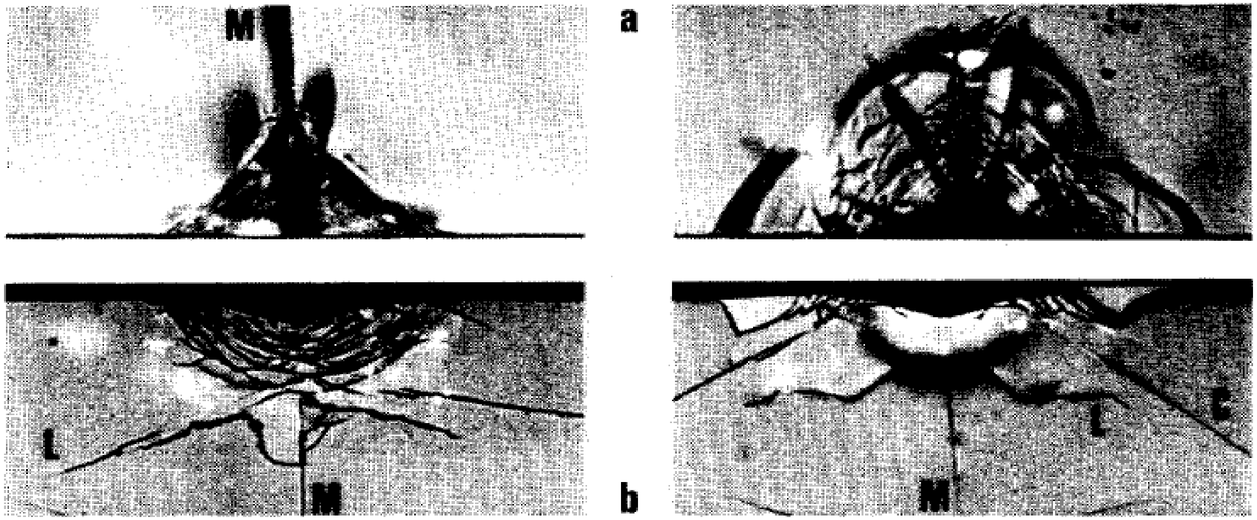


Fig. 2. Reflected light micrographs of Vickers indentations in soda-lime (left) and silica (right) glasses: (a) half-surface, (b) section, views. Indentation load 30 N. Width of field 220 μm. Crack labelling as in fig. 1. Note different deformation zone configurations (near-semi-circular region immediately below impression) in (b).

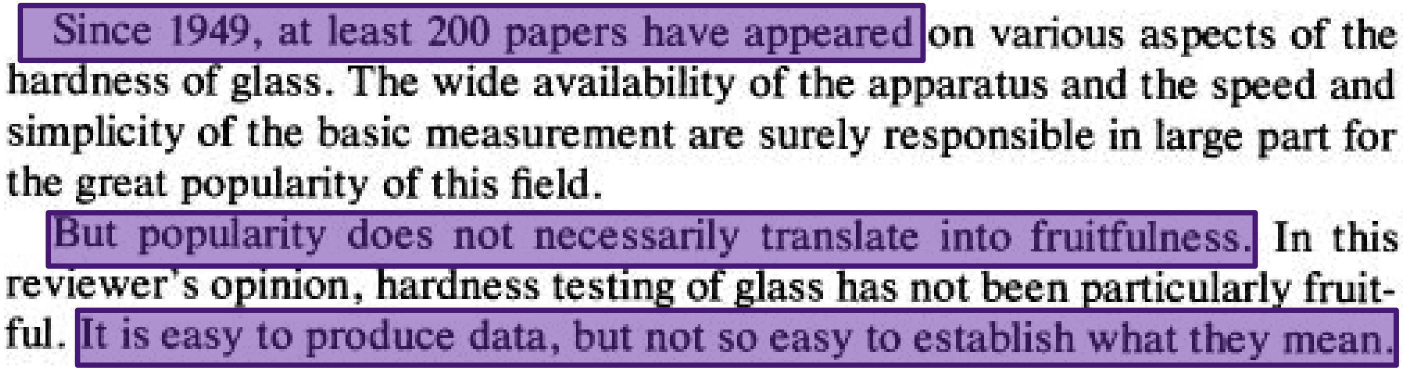
Silica

<u>Anomal Glasses :</u> **densification**

-> Hertz cone cracks

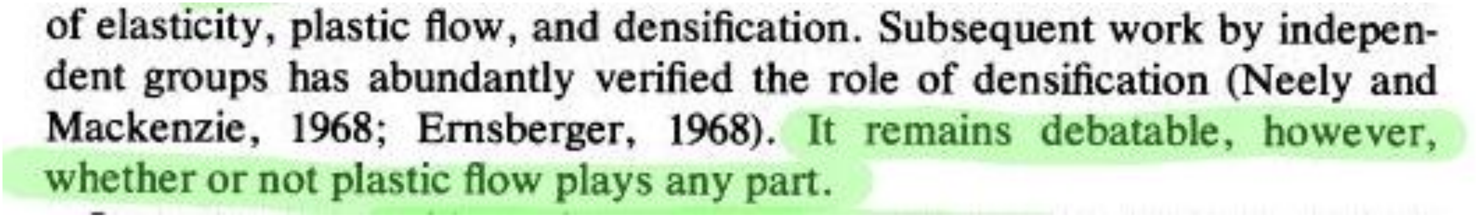
An old story : let's step back

- **1980 :** A.S. Argon, R.F. Bartholomew, F.M. Ernsberger, S.W. Freiman, and H.M. Gardon, **Glass Science and Technology, Elasticity and Strength in Glasses**



Since 1949, at least 200 papers have appeared on various aspects of the hardness of glass. The wide availability of the apparatus and the speed and simplicity of the basic measurement are surely responsible in large part for the great popularity of this field.

But popularity does not necessarily translate into fruitfulness. In this reviewer's opinion, hardness testing of glass has not been particularly fruitful. It is easy to produce data, but not so easy to establish what they mean.

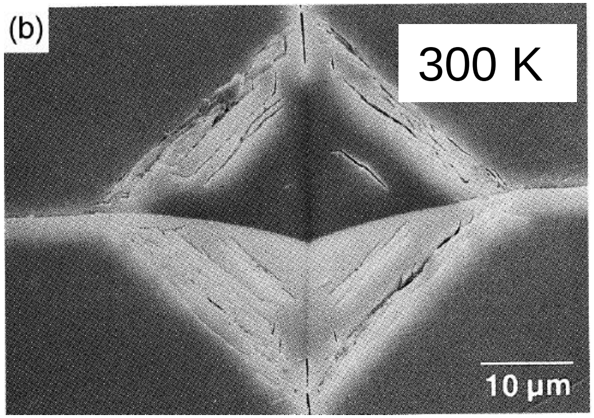


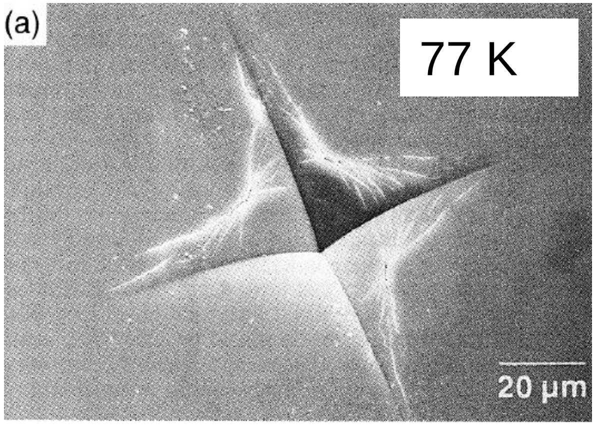
of elasticity, plastic flow, and densification. Subsequent work by independent groups has abundantly verified the role of densification (Neely and Mackenzie, 1968; Ernsberger, 1968). It remains debatable, however, whether or not plastic flow plays any part.

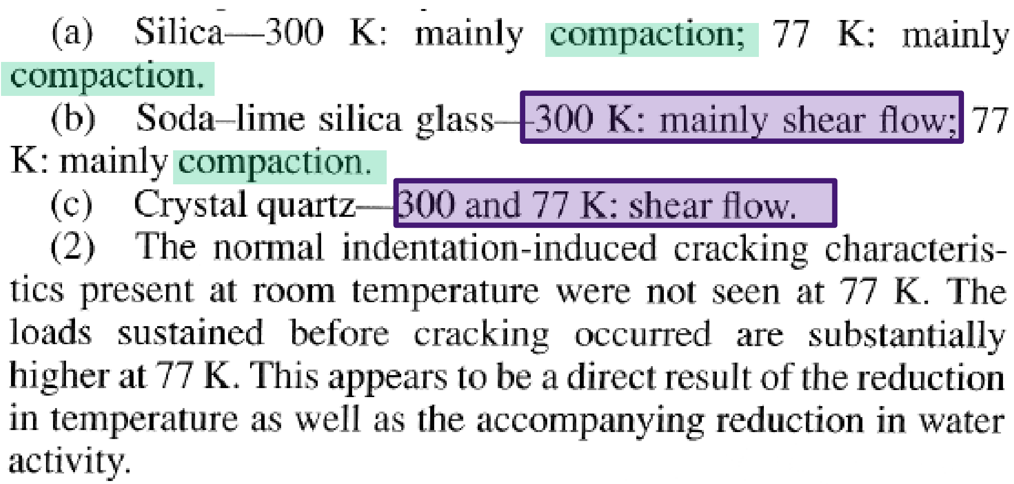
And after : shear flow vs densification

- **1995 :** C.R. Kurkjian, G.W. Kammlott, and M.M. Chaudhri, Indentation behavior of soda-lime silica glass, fused silica, and single-crystal quartz at <u>liquid nitrogen</u> temperature, **J. Am. Cer. Soc.**

Soda-Lime Glass







(a) Silica—300 K: mainly compaction; 77 K: mainly compaction.

(b) Soda–lime silica glass—300 K: mainly shear flow; 77 K: mainly compaction.

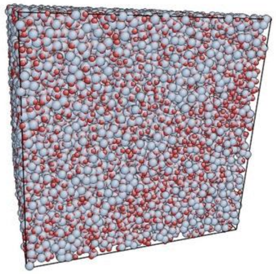
(c) Crystal quartz—300 and 77 K: shear flow.

(2) The normal indentation-induced cracking characteristics present at room temperature were not seen at 77 K. The loads sustained before cracking occurred are substantially higher at 77 K. This appears to be a direct result of the reduction in temperature as well as the accompanying reduction in water activity.

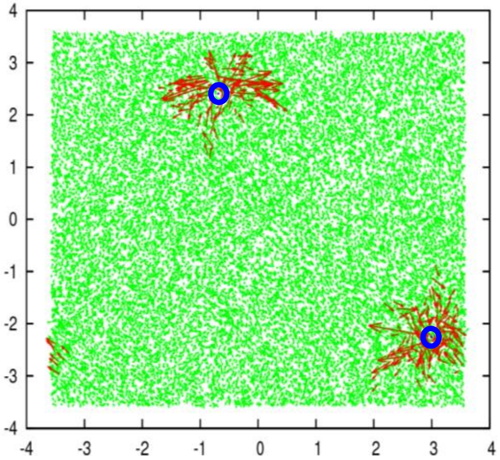
Shear Flow is a thermically activated process

Plastic flow by atomistic simulations

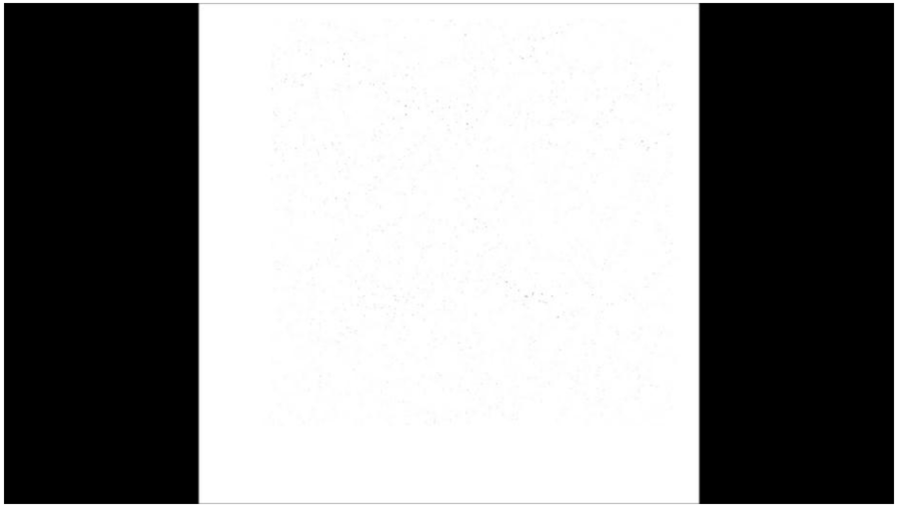
- **Pure shear (P = 0 GPa)**

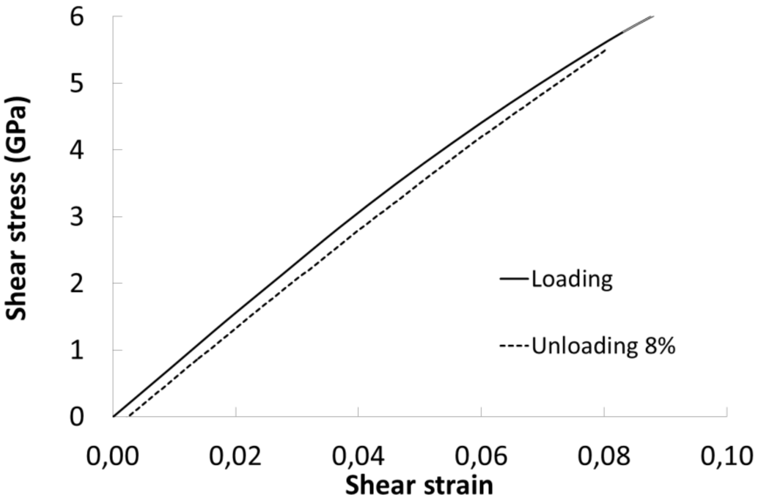


Results from group of A. Tanguy (ILM, Lyon)



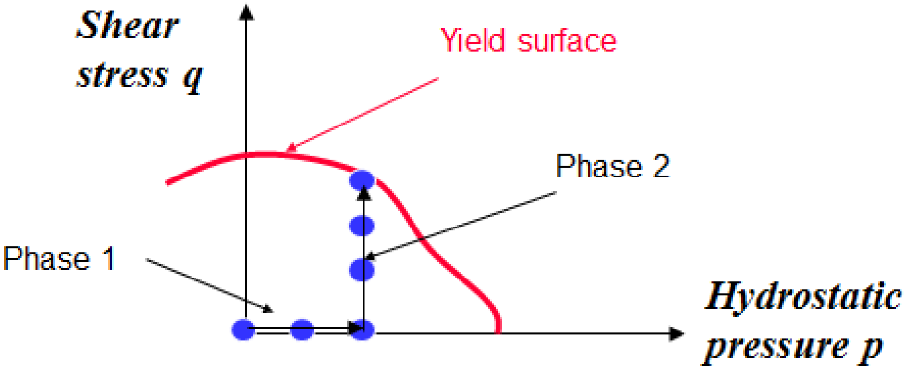
B. Mantisi *et al Eur. Phys. J. B* **85** (2012) 304

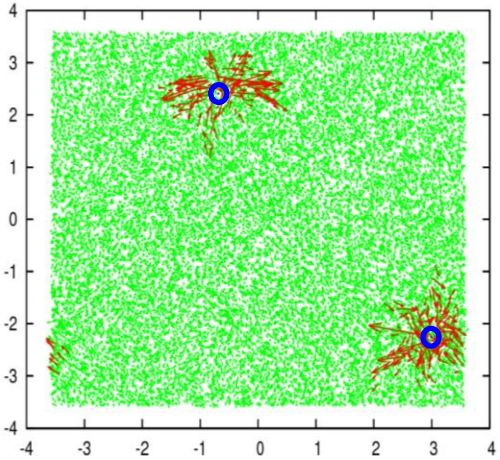




Plastic flow by atomistic simulations

- **High pressure (8 GPa)**





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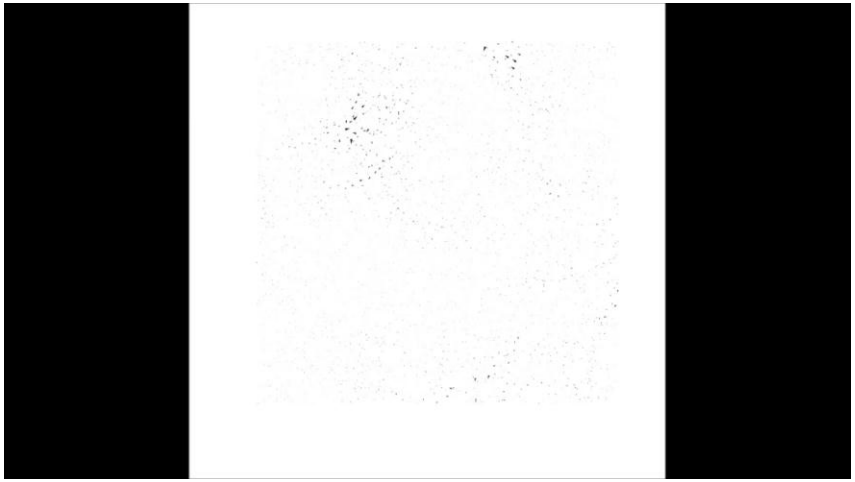
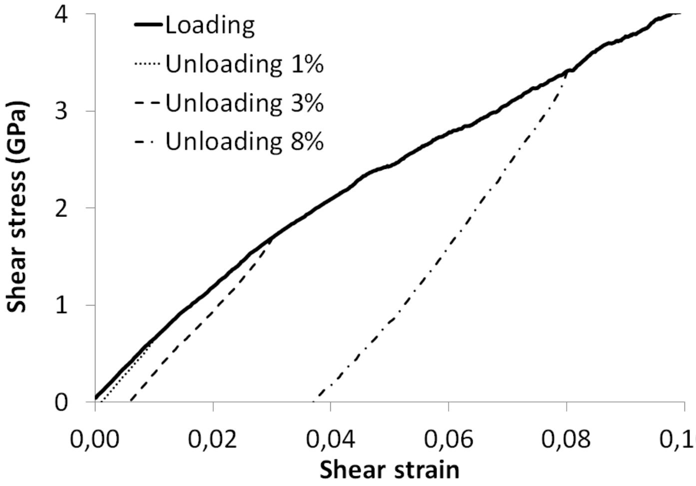




And after : Constitutive laws of fused silica

- **1996 :** J.C. Lambropoulos, S. Xu, and T. Fang. Constitutive law for the densification of fused silica, with applications in polishing and microgrinding. **J. Am. Ceram. Soc**.

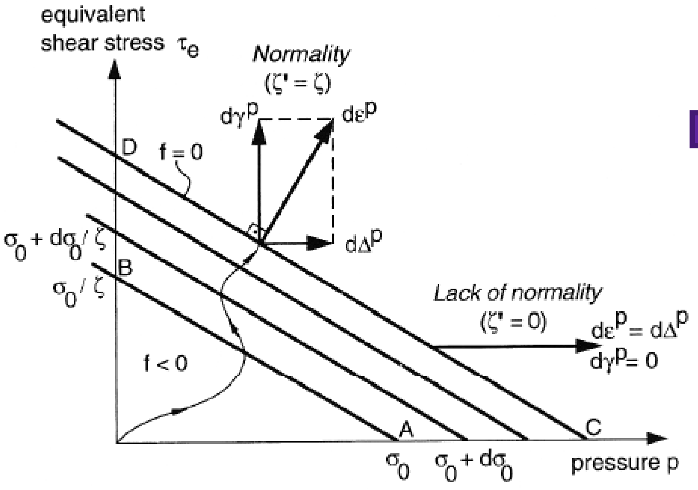
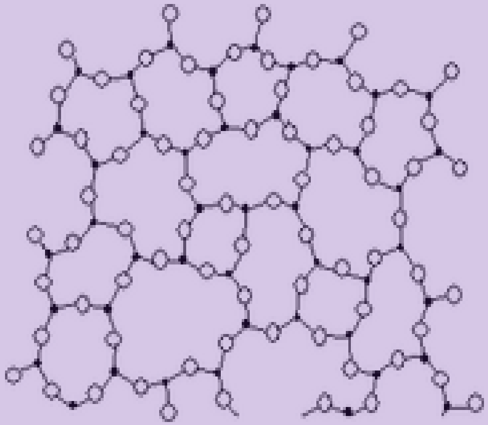


Fig. 4. Yield function describing the densification and flow (i.e., shear deformation) of silica in a stress space. The coordinates are the hydrostatic pressure $p = -\sigma_m$ and the equivalent shear stress τ_e. Shown are the initial yield surface (line AB), subsequent yield surfaces, the current yield surface CD, and a loading path. If $\zeta = \zeta'$, then normality is satisfied (the plastic strain increment is normal to the yield surface) and both permanent densification and hear flow result. If $\zeta' = 0$, then normality is not satisfied: although both hydrostatic pressure and shear stress are coupled in inducing permanent deformation, the permanent deformation consists exclusively of densification and no shear flow. The hardening modulus h relates increments in pressure to increments in permanent densification, i.e., $h = d\sigma_0/d\Delta^p$.

- Yield criterion coupling shear and pressure linearly
- Densification-only requires non associated flow rule

Plastic deformation = Structural buckling ?



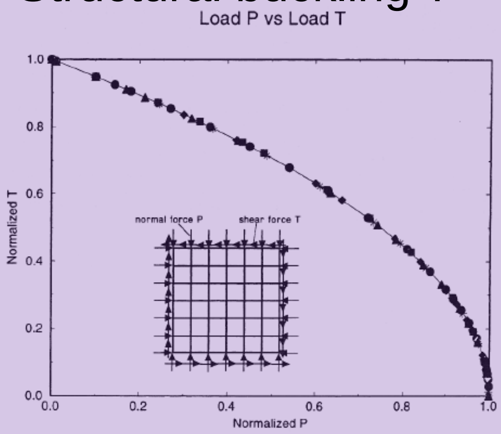


Fig. A1. Variation of shear and normal stress required by the collapse via elastic buckling of a 2-D elastic frame (see insert).

And after : Constitutive laws of fused silica

- **2008 :** G. Kermouche, E. Barthel, D. Vandembroucq, and Ph. Dubujet, Mechanical modelling of indentation-induced densification in amorphous silica, **Acta Materialia**

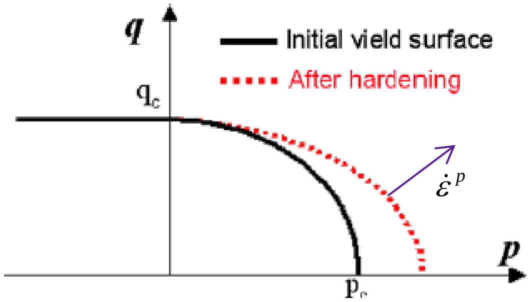
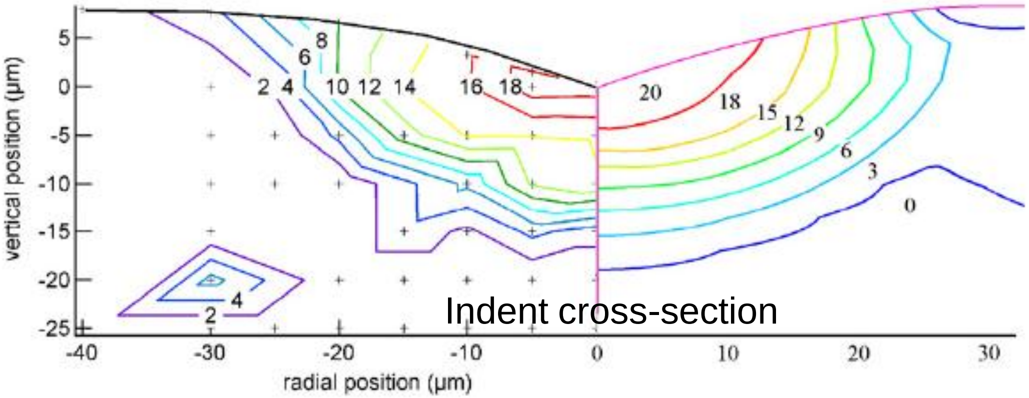


Fig. 1. The propc

- Based on mechanics of porous media
- Elliptic yield criterion coupling shear and pressure
- Associated flow rule
  - High pressure -> only densification
  - Low pressure -> dominated shear flow

Experimental densification map :

Micro-raman scattering



Computational densification map :

FEM

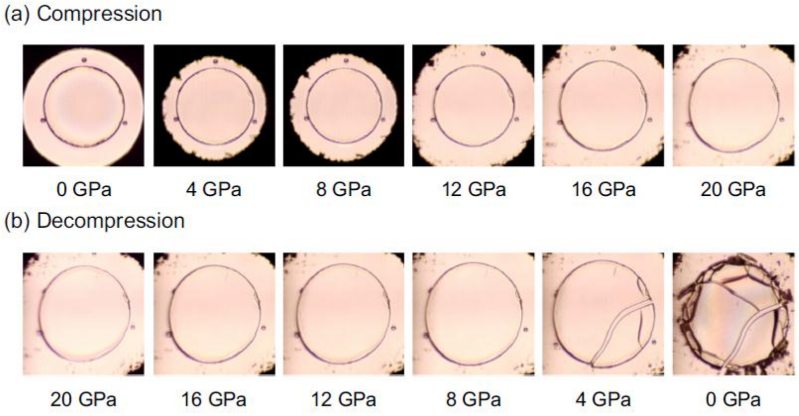
Fig. 7. Densification map: left, experimental results; right, finite element results.

And now : shear flow and/or densification ?

- **2010 :** T. Rouxel, H. Ji, J.P. Guin, F. Augereau, and B. Rufflé, Indentation deformation mechanism in glass: Densification versus shear flow. **Journal of Applied Physics**

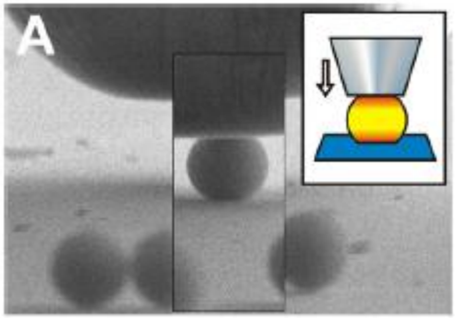
Shear flow in <u>silica</u> at room temperature

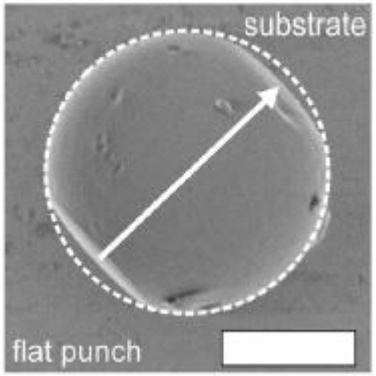
- **2012 :** Lacroix et al, **Acta Materialia**
- **2015 :** Wakabayashi et al, **Physical Review B**



<u>No</u> shear flow in <u>silica</u> at room temperature

- **2014 :** Keryvin et al, **Key Eng. Mat.**
- **2015 :** Romeis et al, **Scripta Materialia**





Why investigating silicate glasses plasticity

- **1971 :** S.M. Wiederhorn and H. Johnson, Effect of pressure on the fracture of glass, **Journal of Applied Physics**

→ "The <u>fracture surface energy </u>of glass is 2 to 4 times that expected from theoretical considerations."

- **2010 :** C.R. Kurkjian, P.K. Gupta, and R.K. Brow, The strength of silicate glasses: What do we know, what do we need to know?, **Int. J. of Applied Glass Science**

→ "<u>Residual stress </u>induced by densification is substantially lesser than induced by shear flow."

- **1992 :** W. C. Oliver et G. M. Pharr, An improved technique for determining hardness and elastic-modulus using load and displacement sensing indentation experiments, **J. Mater. Res**.

→ Fused silica became **the** <u>"calibration material" </u>for nanoindentation testing

Aims of this talk

- **Adress the issue of shear flow versus densification using In-Situ SEM Micro-Compression of silica micropillars**

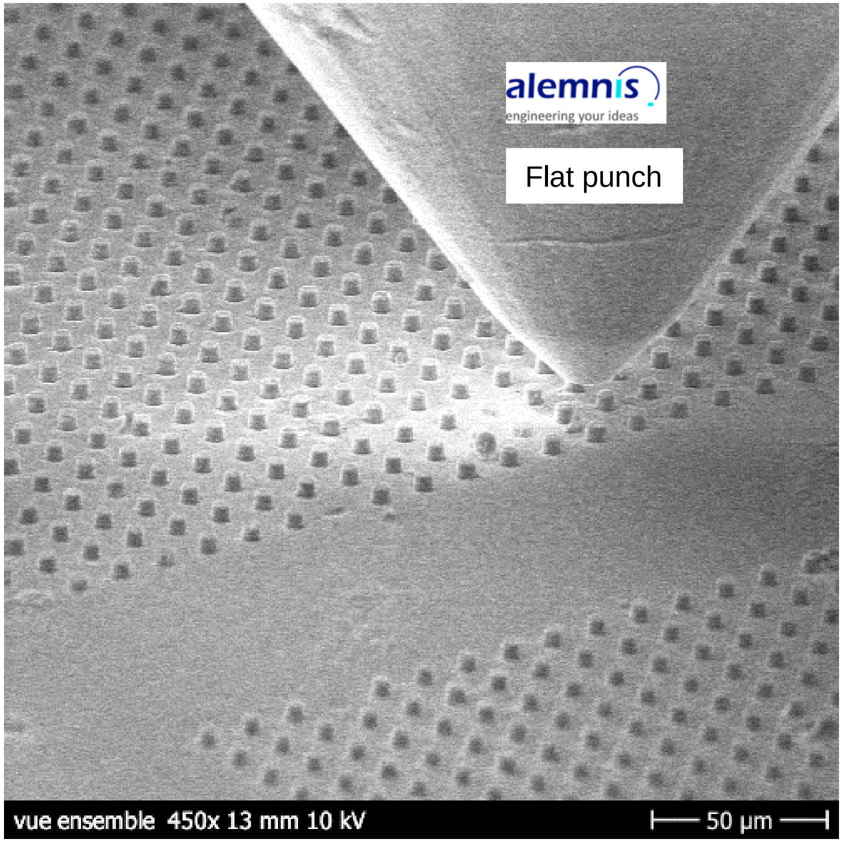
- **If there is shear flow, what about shear-hardening ?**
  - No dislocations and twins !

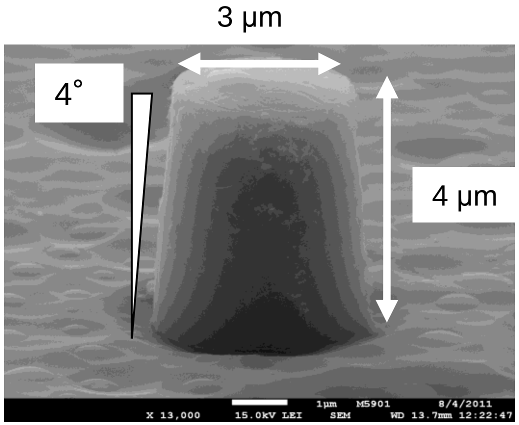
- **What about size effects in silica ?**

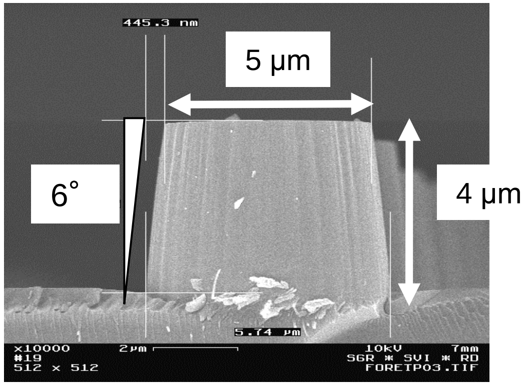
- **And fracture at small scale ?**

Materials & methods

- **Silica Pillars by RIE (d 5 µm & d 3 µm)**
- ***in situ* SEM displacement-contolled micro-compression**

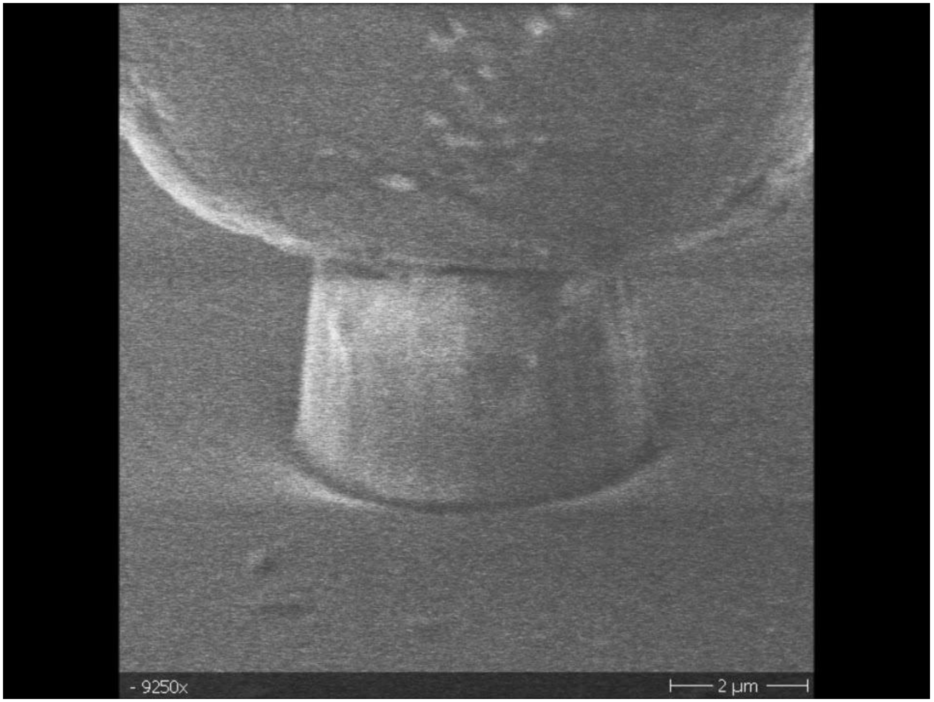




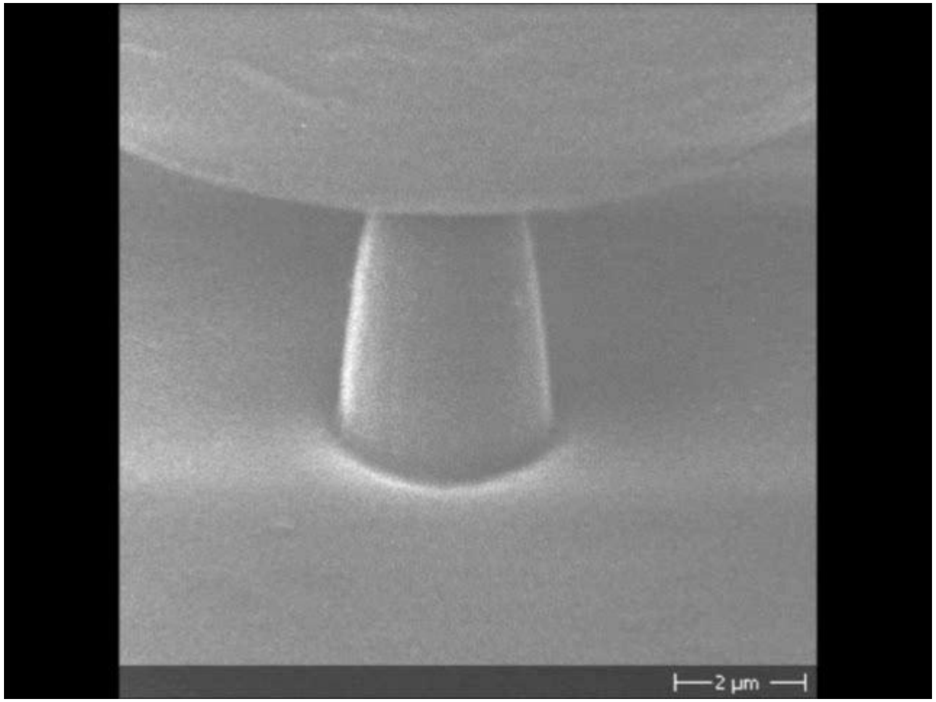


Results

- **D = 5 µm**



- **D = 3 µm**

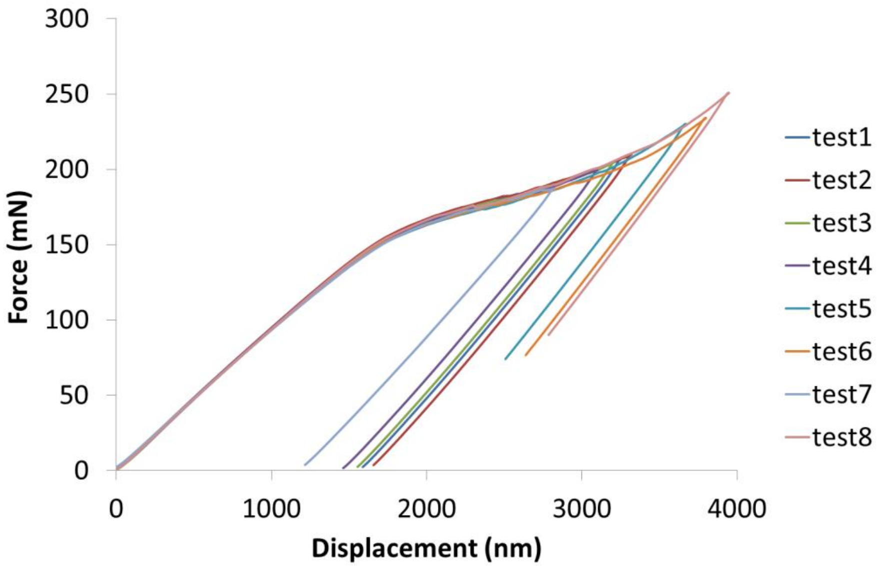


Homogeneous plastic flow of silica with radial expansion

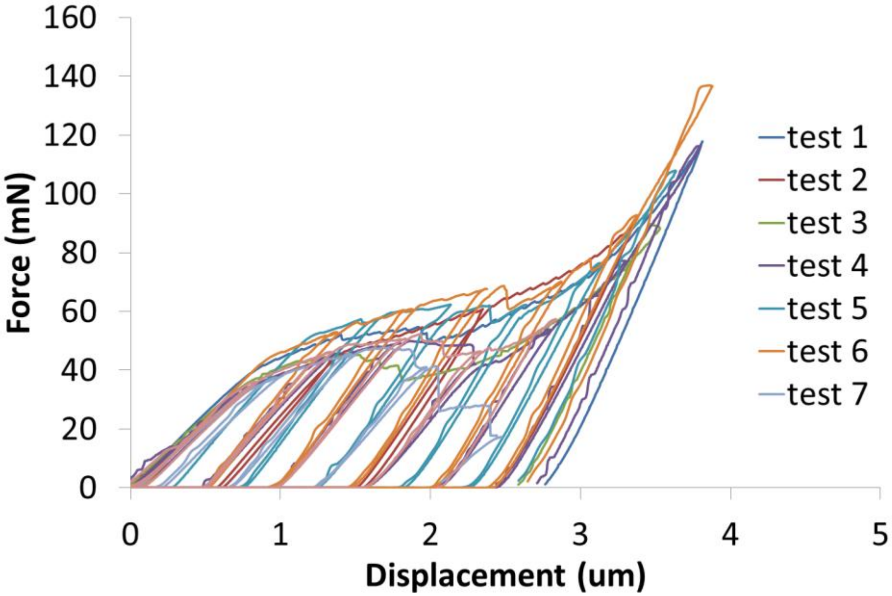
Crack nucleation and stable propagation during loading

Results

- **D = 5 µm**



- **D = 3 µm**



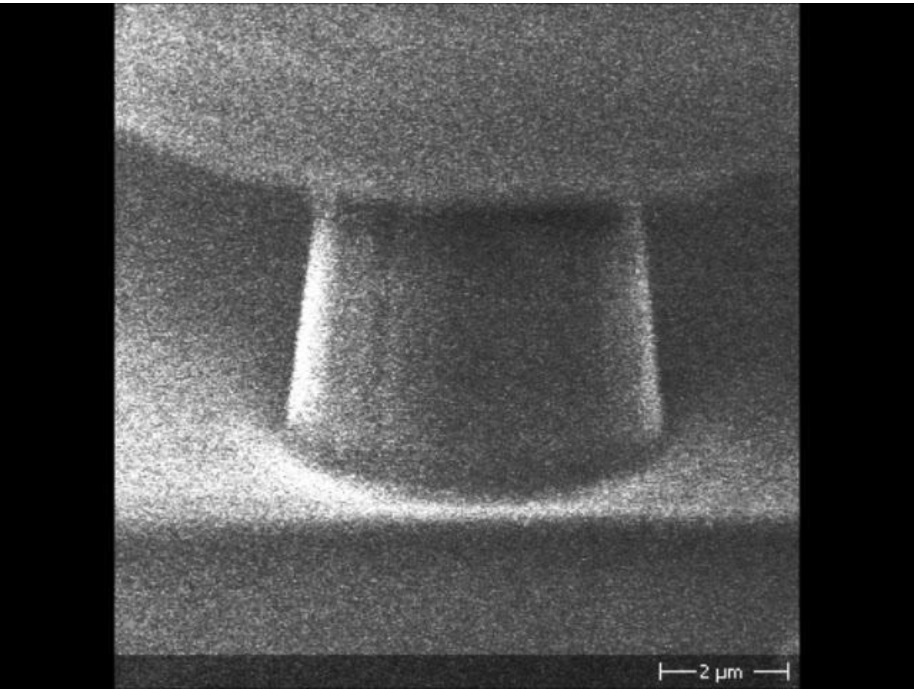
Very good repeatability for 5 µm pillars

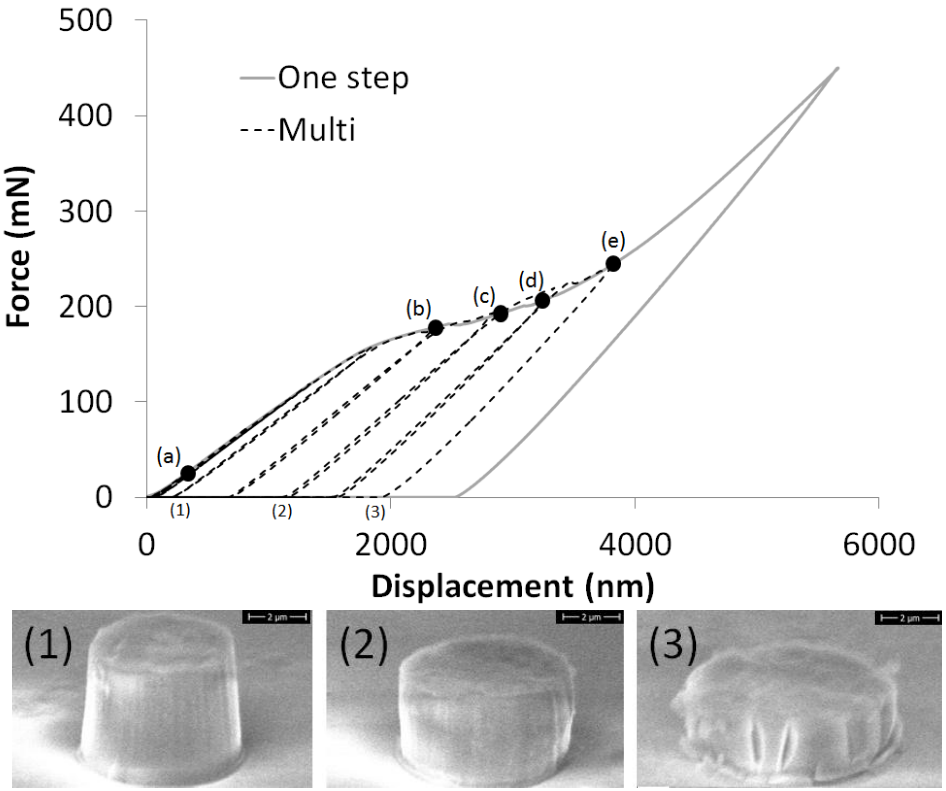
➡ Geometry of 3 µm pillars is much scattered

G.Kermouche et al, Perfect plastic flow in silica, submitted glass to Acta Mat., 2015

Results – cyclic tests

- **D = 5 µm**

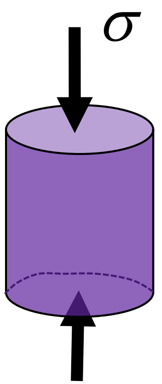




Cyclic tests and montonic loadings yield to same force-displacement curves.

Results

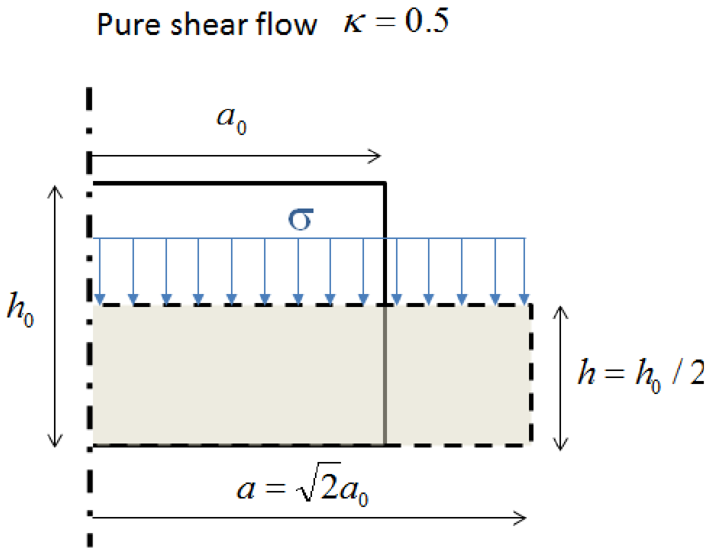
Pure densification or shear flow



Radial plastic expansion coefficient

$$\dot{\varepsilon}_r = -\left(\nu \dot{\varepsilon}_z^e + \kappa \dot{\varepsilon}_z^p\right)$$

Poisson coefficient



- Pure densification (as only plastic mechanism at stake) should lead to a decrease of pillars section during loading.
- ***Shear flow during uniaxial compression of silica pillars !***

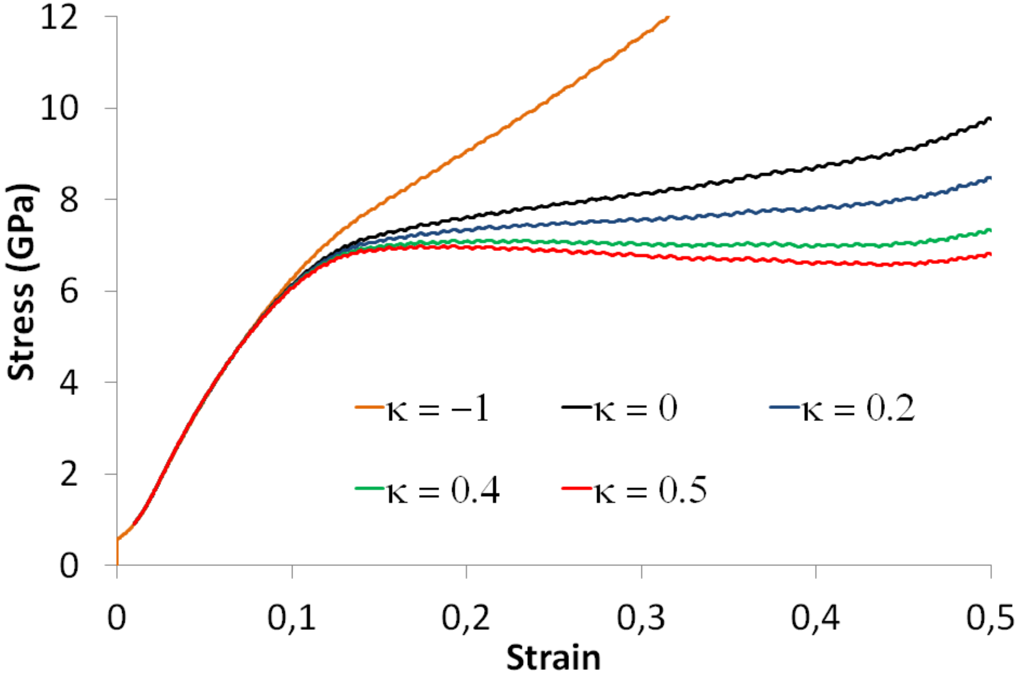
Results

- **Stress-strain curve ?**

Radial plastic expansion coefficien

$$\dot{\varepsilon}_r = -\left(\nu \dot{\varepsilon}_z^e + \kappa \dot{\varepsilon}_z^p\right)$$

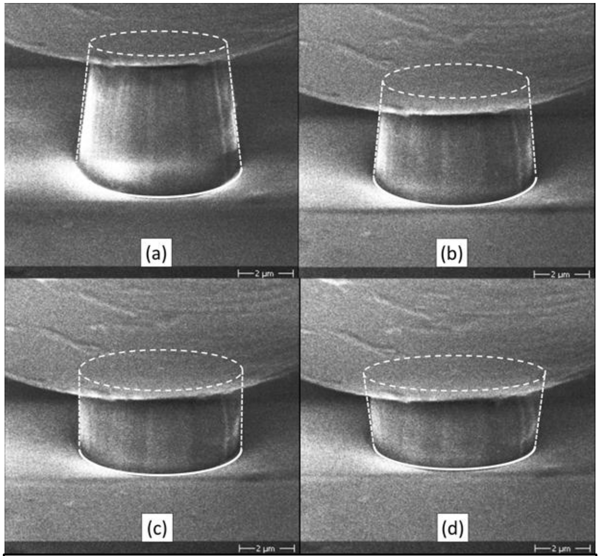
Poisson coefficient

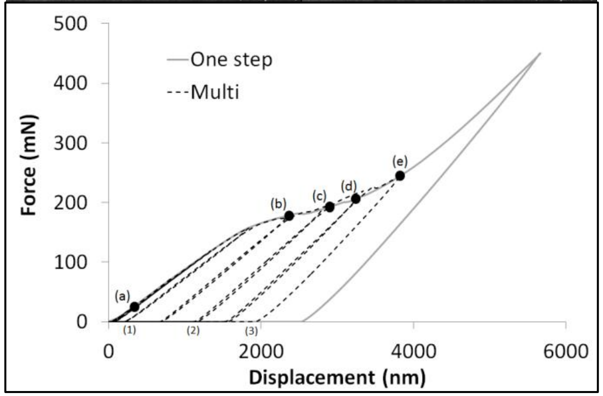


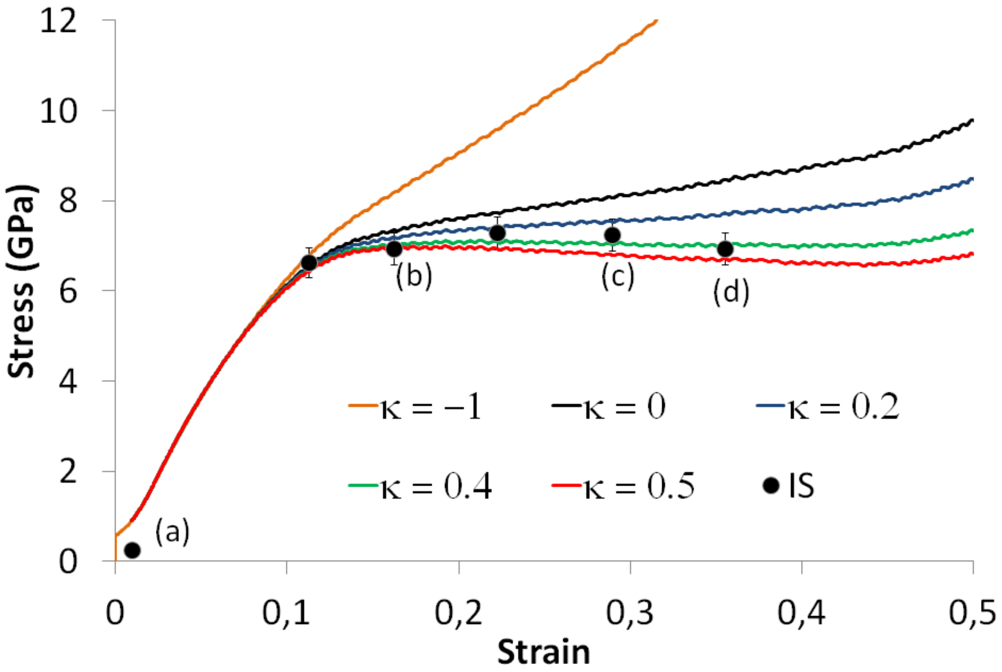
Depending of κ, absence or significant strain hardening slope would be deduced

Results

Stress-strain curve ?







Shear flow without shear-strain hardening ?

G.Kermouche et al, Perfect plastic flow in silica, submitted glass to Acta Mat., 2015

Discussion

- **Densification under uniaxial compression ?**

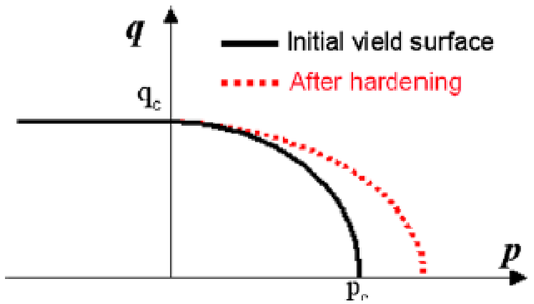
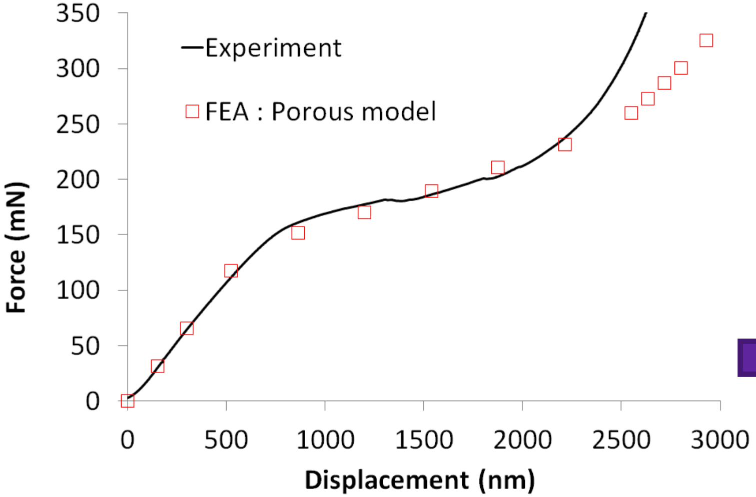
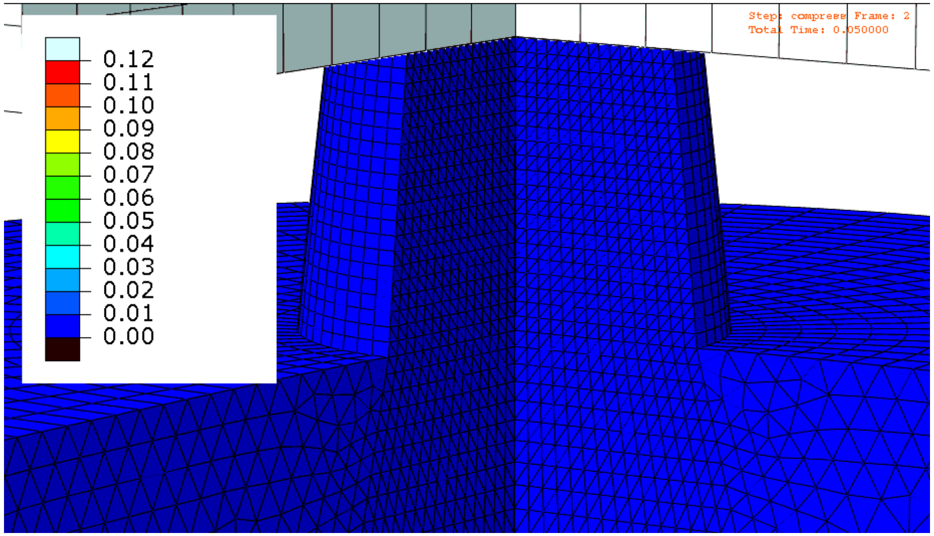


Fig. 1. The proposed yield criterion for amorphous silica.





The FEA load-displacement curve matches almost perfectly the experimental one

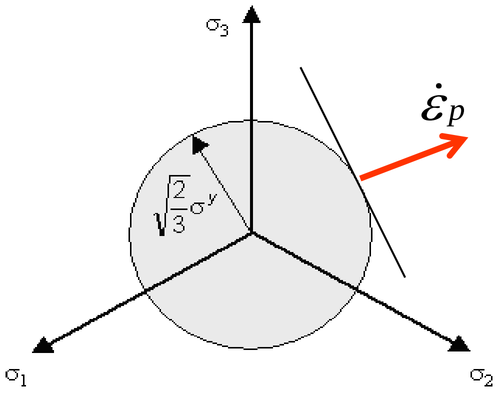
Densification is not homogeneously distributed

Lower densification compared to indentation experiments

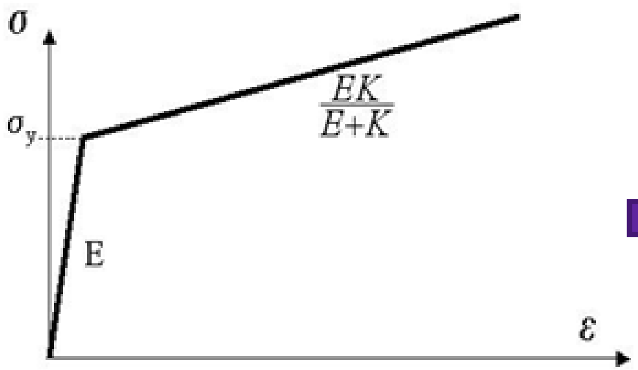
G.Kermouche et al, Perfect plastic flow in silica, submitted glass to Acta Mat., 2015

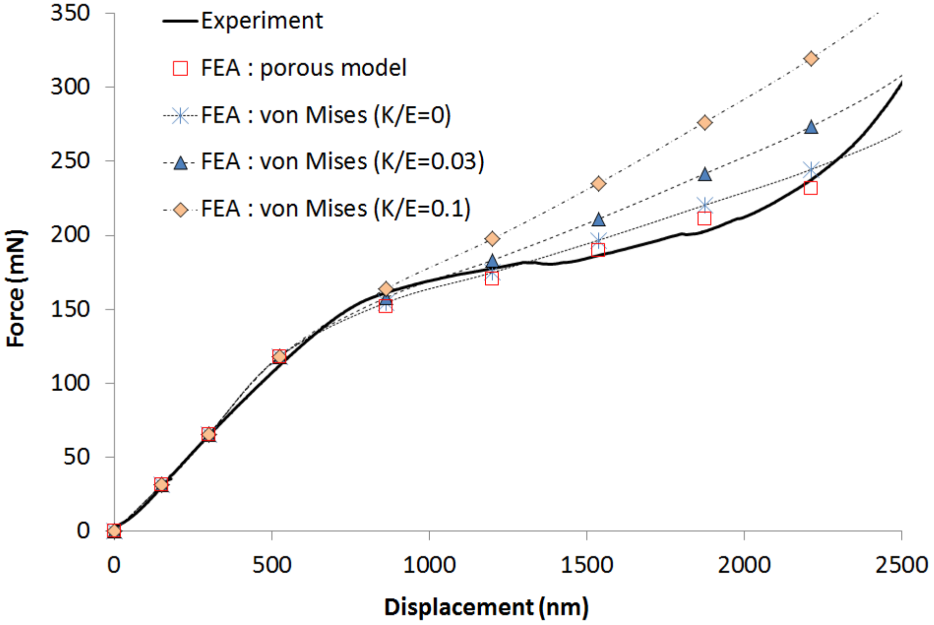
Discussion

- **Shear flow and shear-based strain hardening ?**



$$f(\boldsymbol{\sigma}) = q - (\sigma^y + K\varepsilon^p) \text{ with } q = \sqrt{\frac{3}{2} s_{ij} s_{ij}}$$





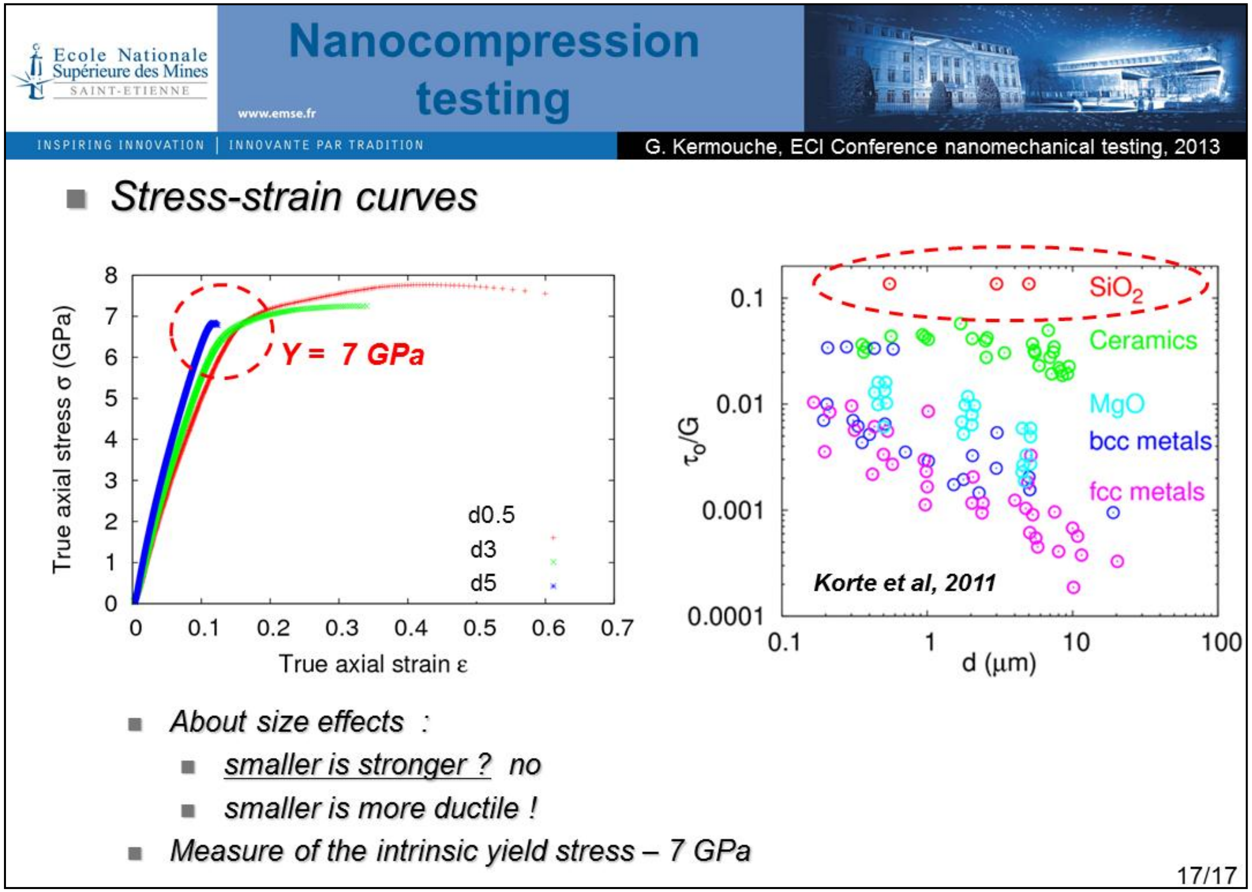
A simple shear-flow based model makes possible to reproduce quite well experimental data

Best results in considering no shear-based strain hardening (K = 0)

G.Kermouche et al, Perfect plastic flow in silica, submitted glass to Acta Mat., 2015

Discussion

- **Size effects in silica glass ?**



Ecole Nationale Supérieure des Mines SAINT-ETIENNE

Nanocompression testing

www.emse.fr

INSPIRING INNOVATION | INNOVANTE PAR TRADITION

G. Kermouche, ECI Conference nanomechanical testing, 2013

- *Stress-strain curves*

- *About size effects :*
  - *smaller is stronger ? no*
  - *smaller is more ductile !*
- *Measure of the intrinsic yield stress – 7 GPa*

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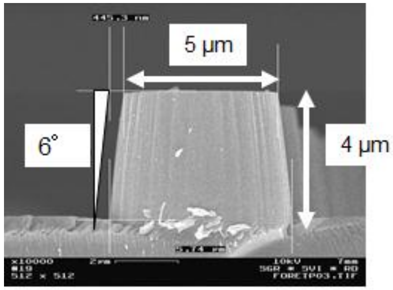
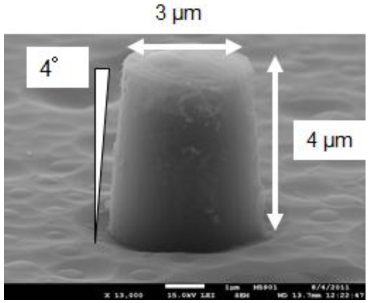
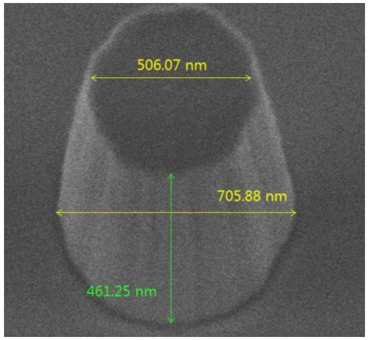
Discussion

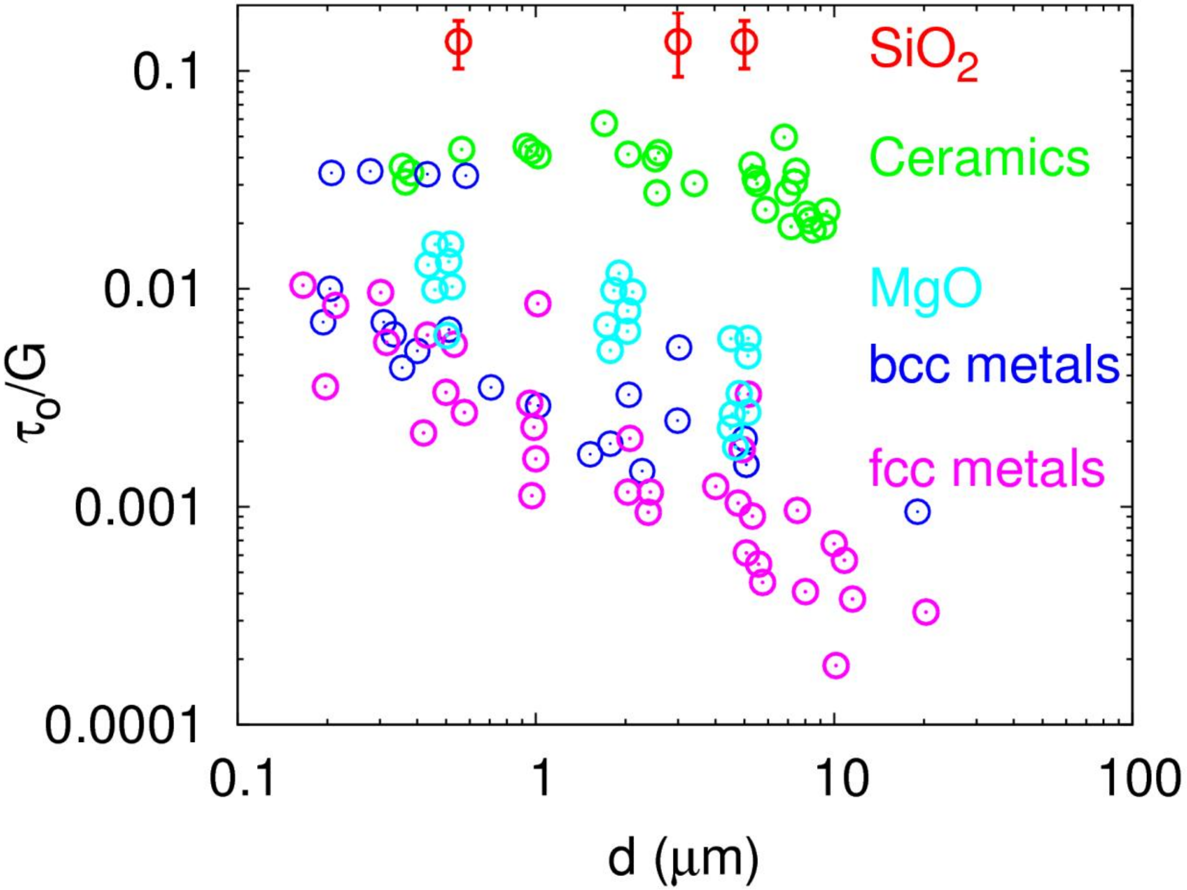
- **Size effects in silica glass ?**

Still not !



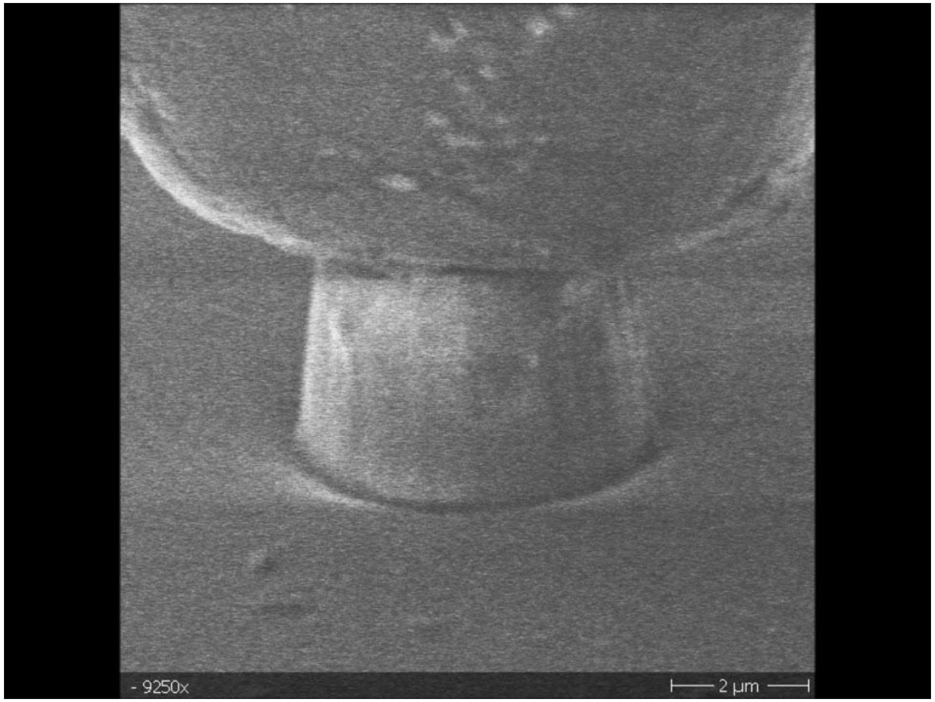




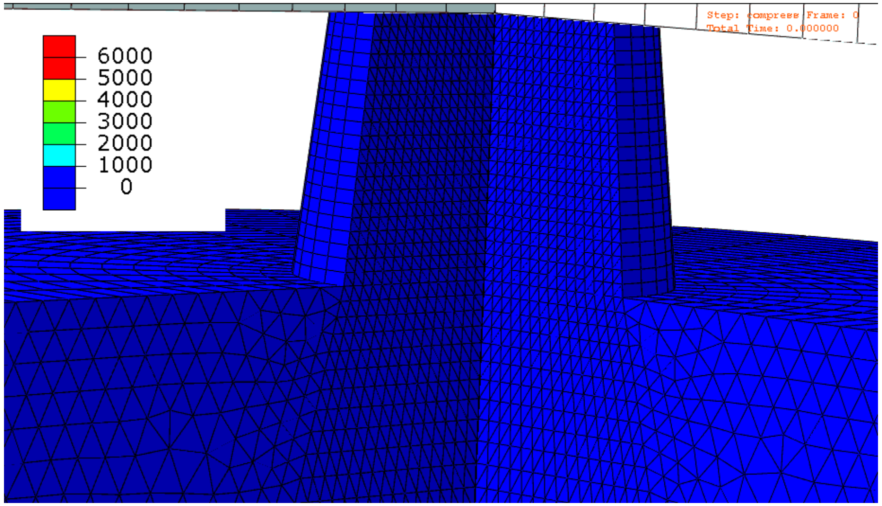


Discussion

- **And crack opening and propagation ?**



Orthoradial stress



→ 6 GPa is consistent with the known tensile strength of pristine fibers (Kurkjian, JNCS, 2003)

G.Kermouche et al, Perfect plastic flow in silica, submitted glass to Acta Mat., 2015

Conclusions

About experiments

- Make possible to deform silica pillars up to large strain without any unstable crack propagation.
- Make possible to measure a better stress-strain curve

About plastic flow of silica glass

- Mostly shear flow under uniaxial loading
- Almost no shear-based strain hardening
- No size effects

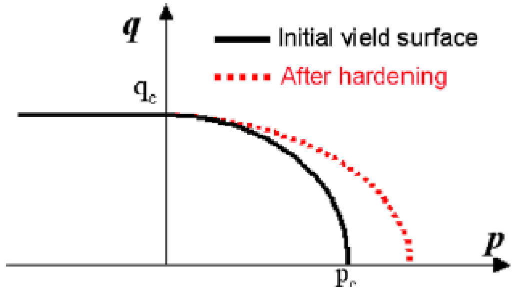


Fig. 1. The proposed yield criterion for amorphous silica.

- PERFECT PLASTIC FLOW IN SILICA GLASS

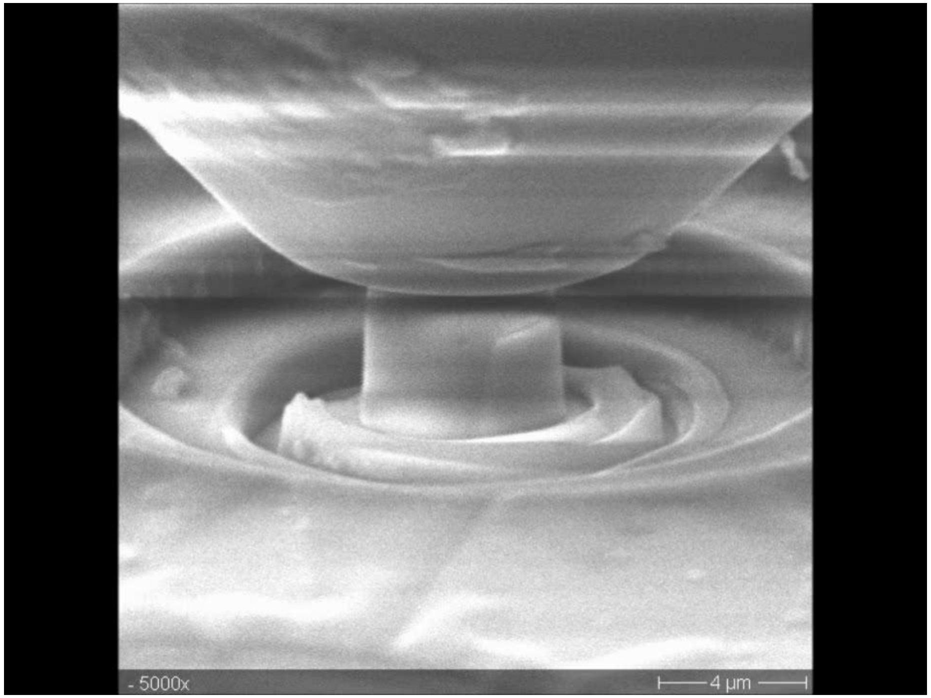
What I did not say !

- ~~Finally silica does not densify under contact loading !~~
- ~~Finally silica is not a brittle material~~

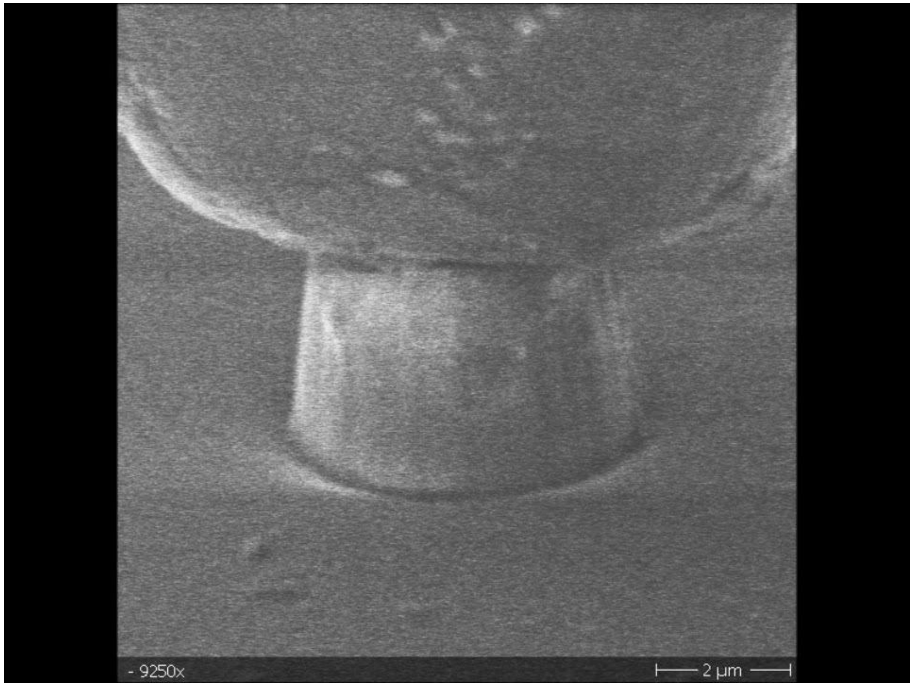
Thank you for your attention

- **Comparison with FIBed normal glasses ?**

Normal glass



Anomal glass



→ Do you see something different ?