In situ deformations in X-ray goniometers

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Why are we combining in situ tests with XRD ?

→application of a macroscopic stress: tensile test or compressive test, hydrostatic pressure or not, 4 points bending tests….

→ temperature change (mainly heating…)

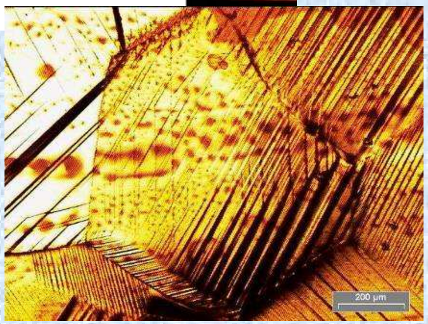
→ microstructural analysis :

- phase analysis, texture analysis, crystallite size, microstrains (→density of dislocations or single defects), …

→measurement of the deformation at the microscopic level

phase changes induced by deformation studying in situ by XRD

MSA : CuAlBe structure $L1_0$ → martensite structure 18R



Malard, Geandier, plasticité (2007)

differences between neutron – X-ray diffraction

-neutrons:

-light elements

-difference between Cu-Ni

-Studies of Magnetic properties

-bulk samples

-photons:

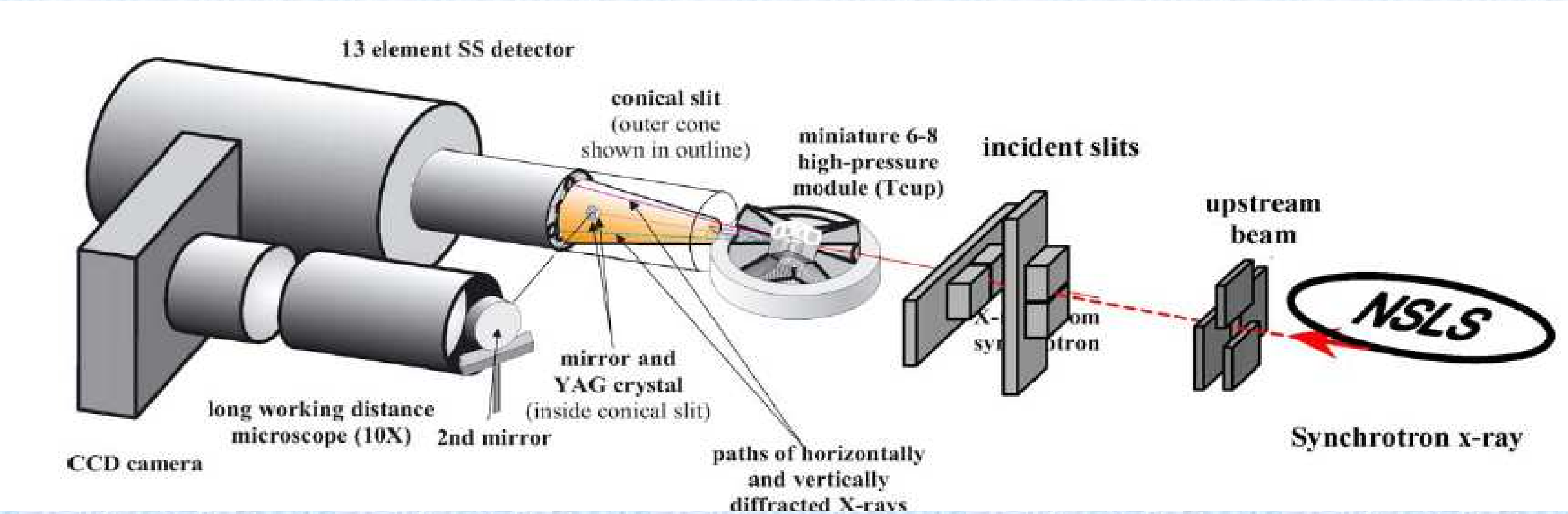
-Flux

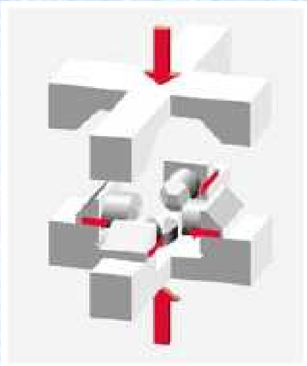
-Resolution

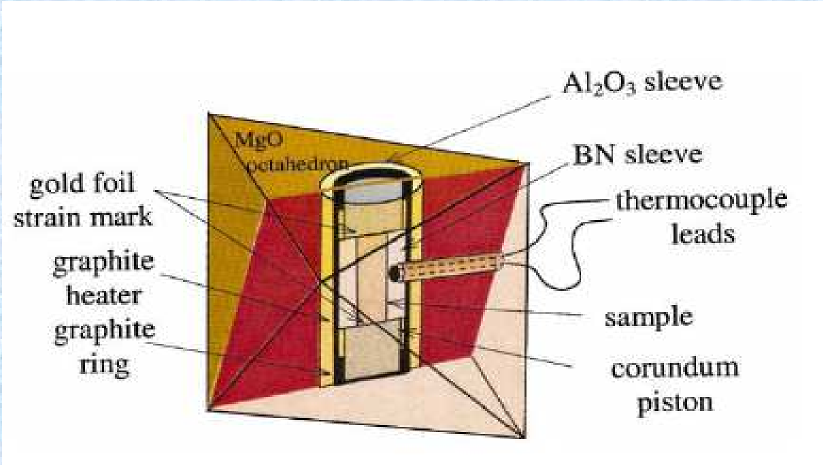
-Thin Films – bulk (SR)

-Gradients of composition

in situ deformation studies

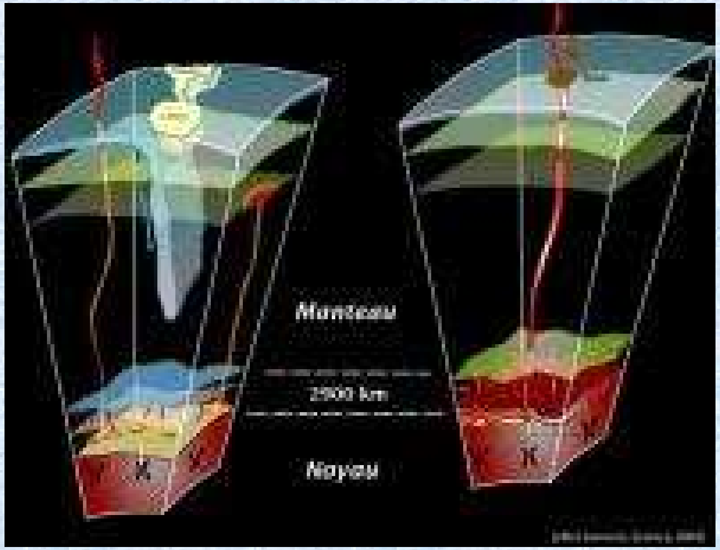




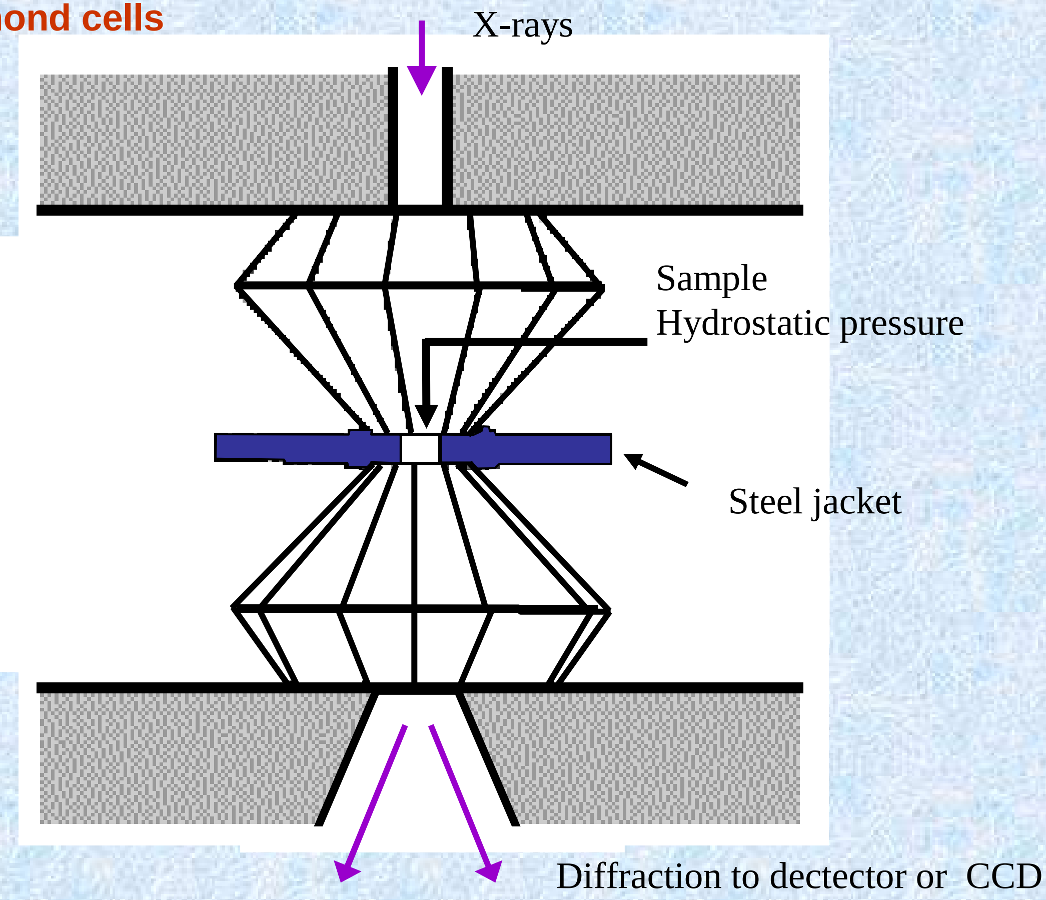


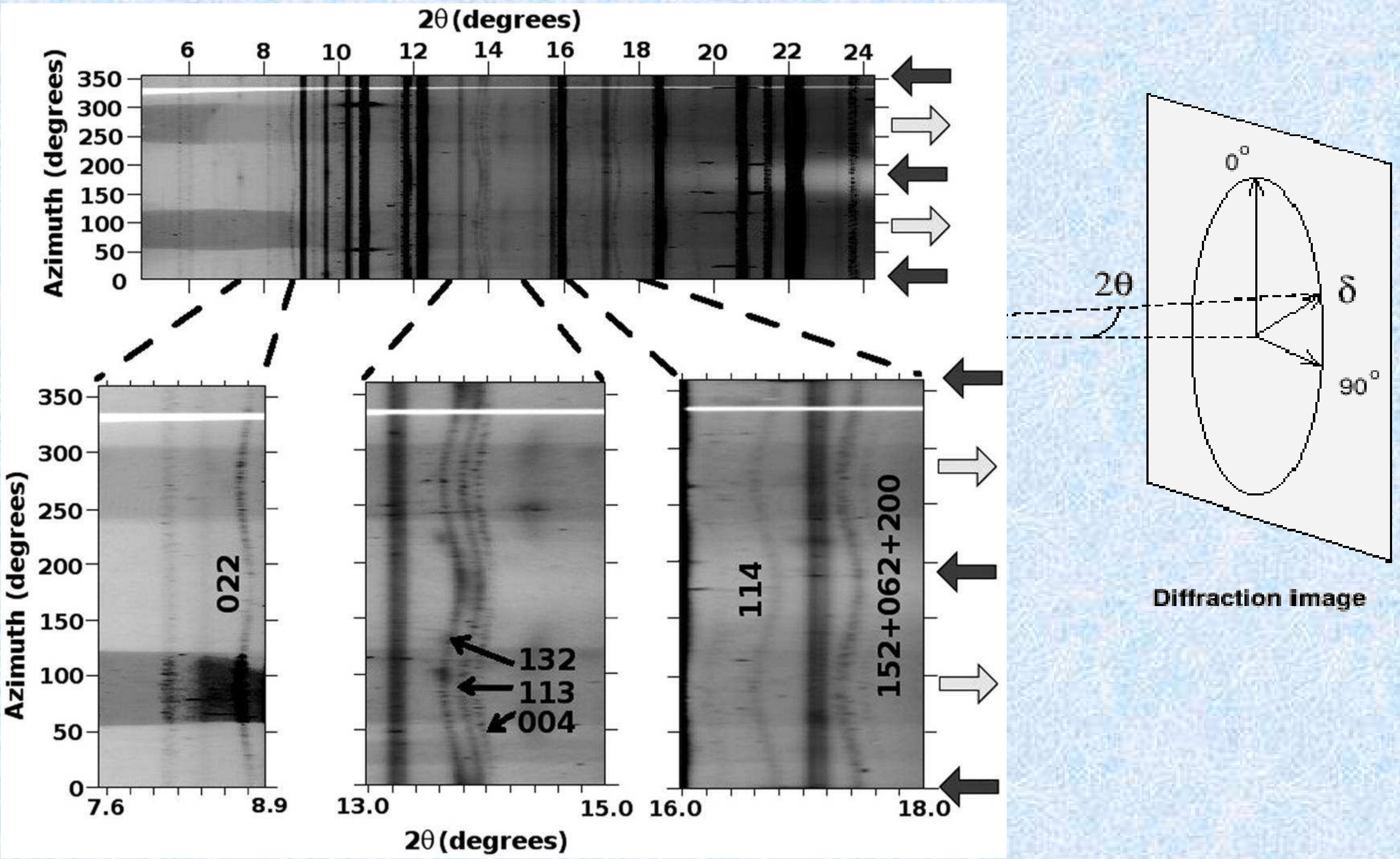
J. Rabier, plasticité 2007

in situ deformation studies



diamond cells

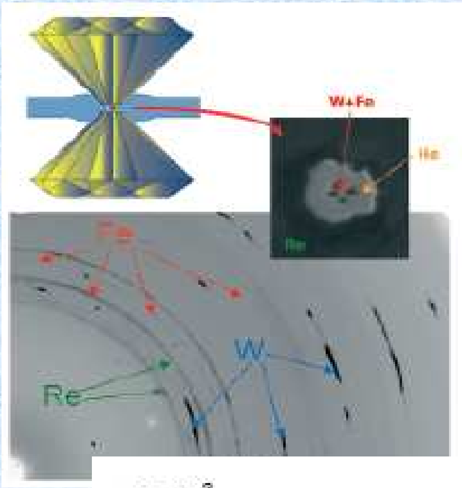


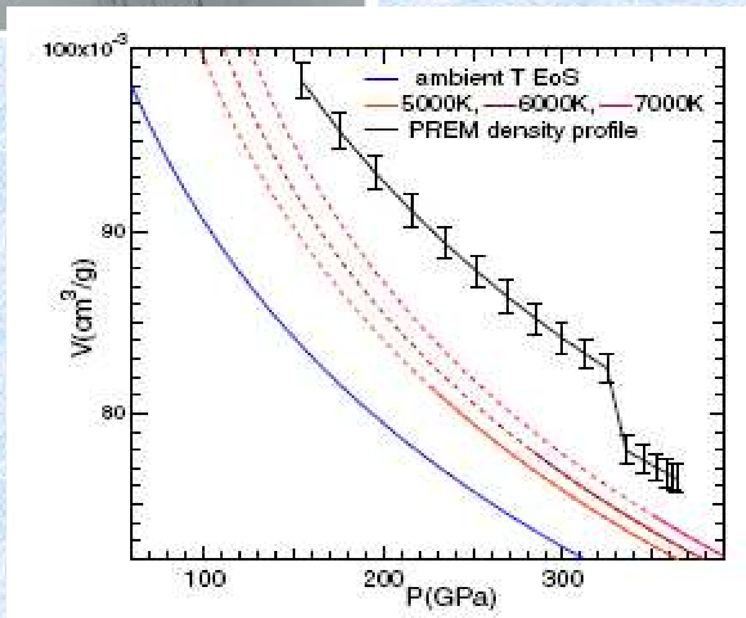


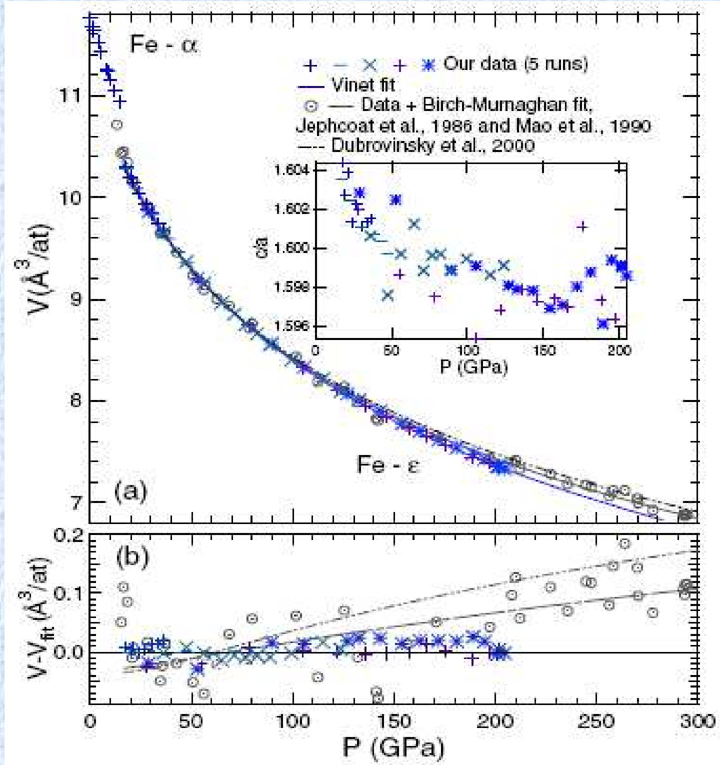
S. Merkel et al., Science 316, 1729 -1732 (2007)

in situ deformation studies

→equation of state of iron





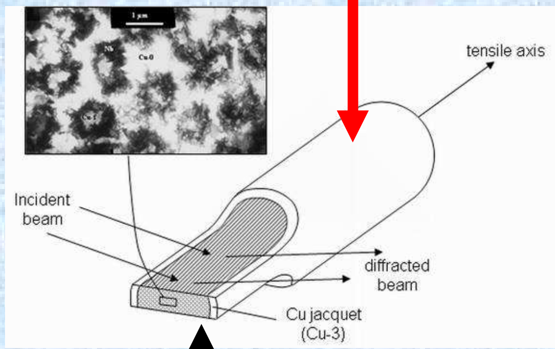


Dewaele et al., PRL (2006)

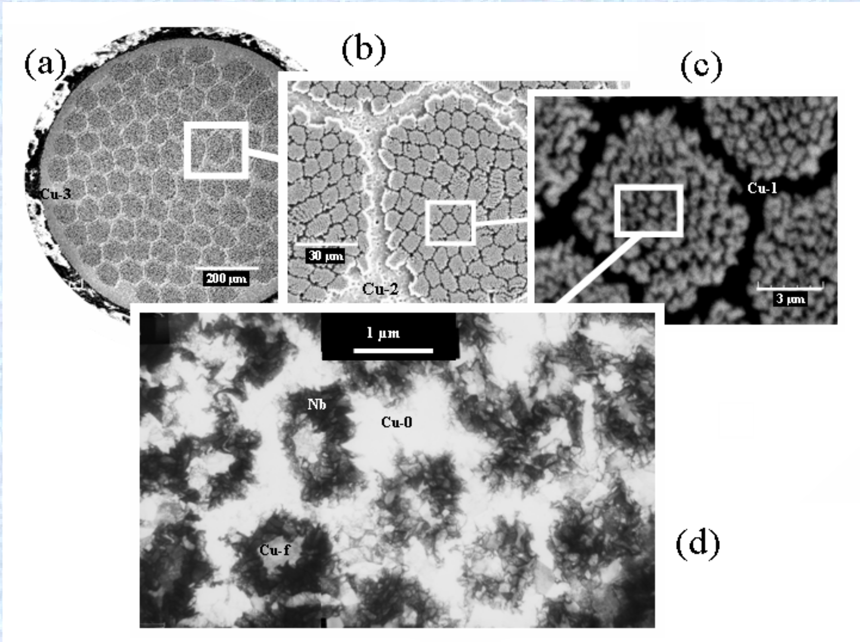
Study of deformations in situ by XRD et ND

to understand the mechanism of deformation into composites Cu-Nb

Neutrons POLDI

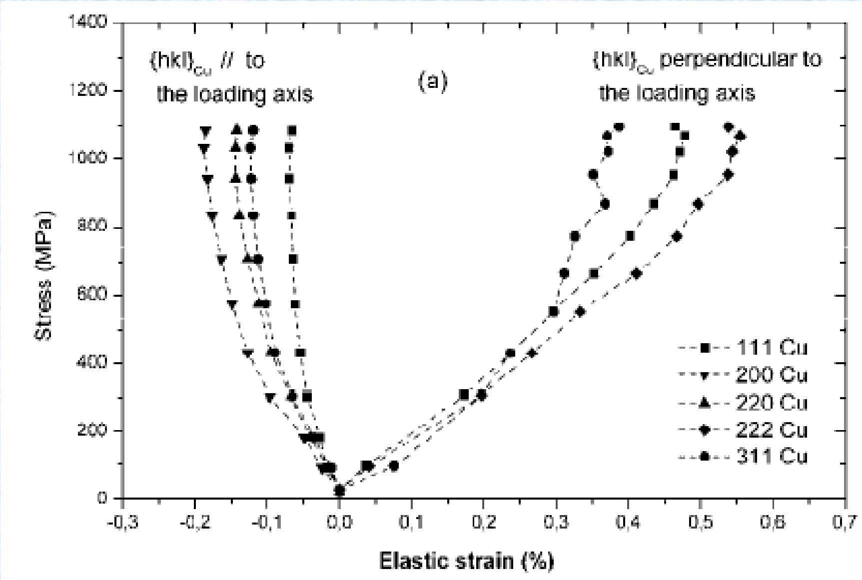


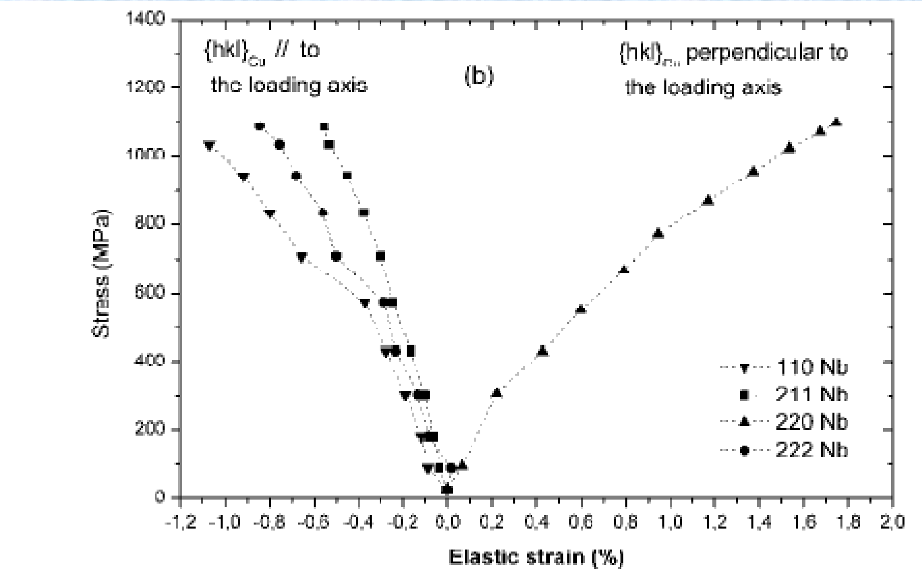
XRD SLS

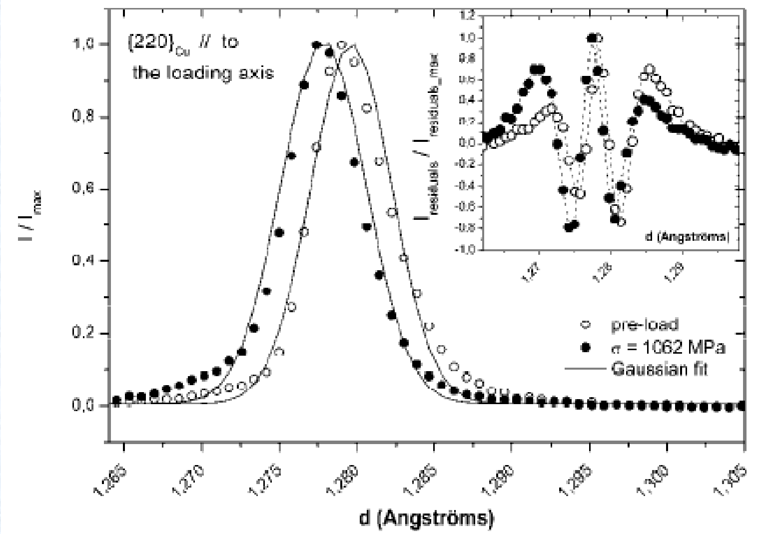


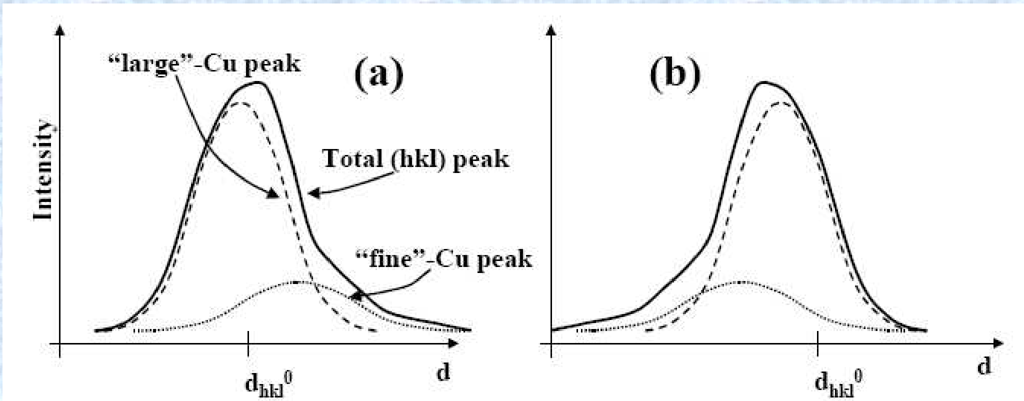
L. Thilly, APL (2006)

Informations on the 2 (or 3) phases !!!









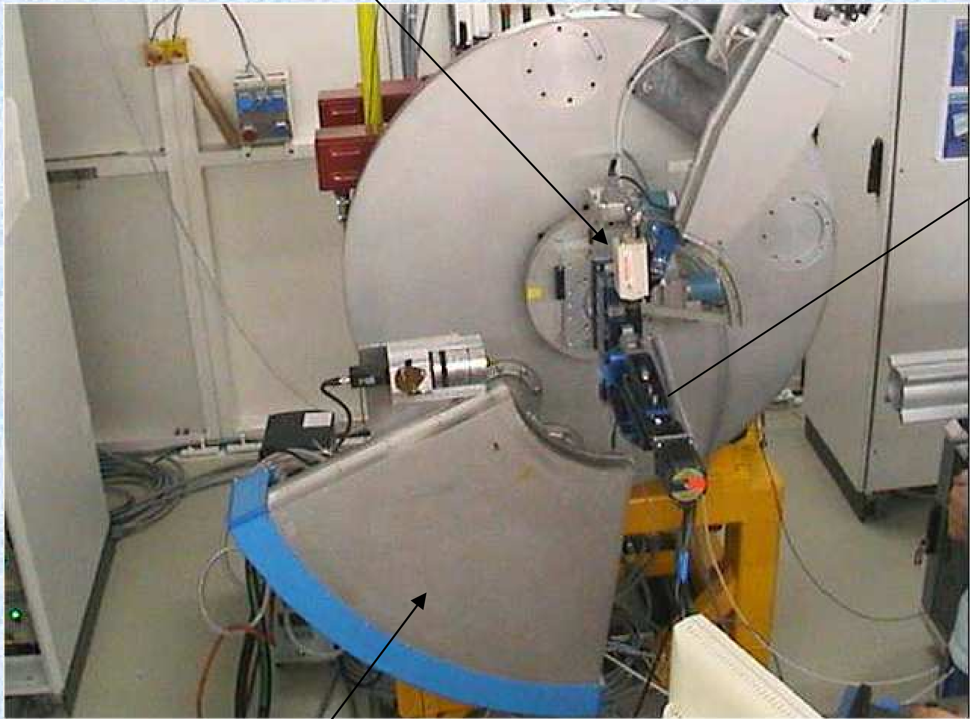
Initial state

Loading state

L. Thilly, APL (2006)

L. Thilly, APL (2007)

CCD Camera



Tensile tester

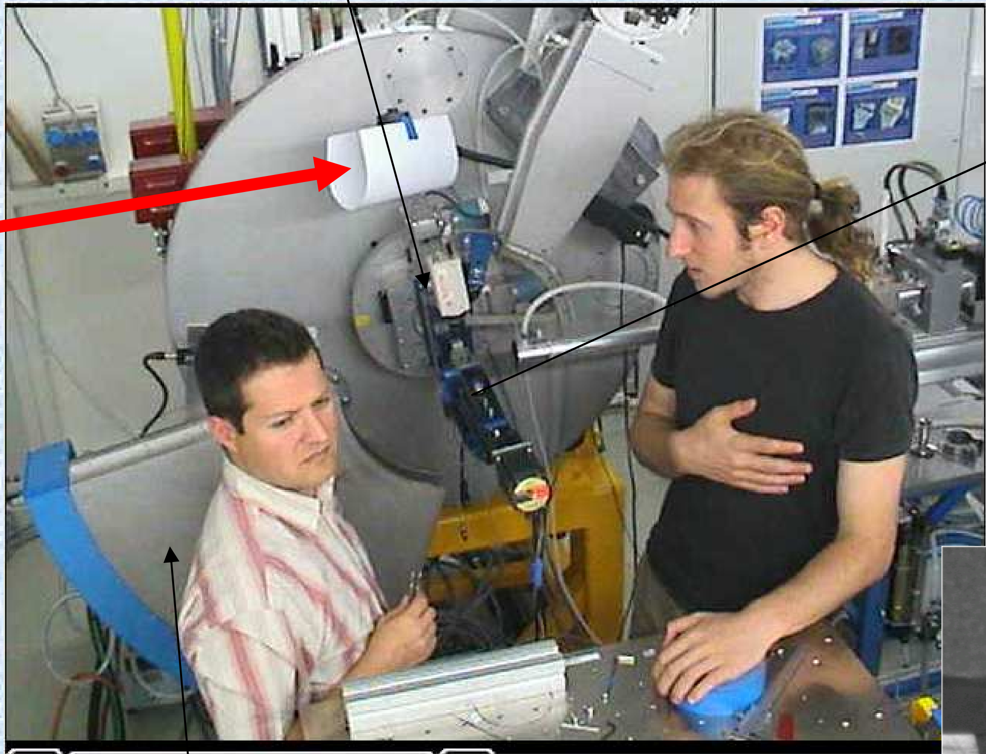
Curved Detector 60°

Materials Sciences - X04SA-Beamline

SLS

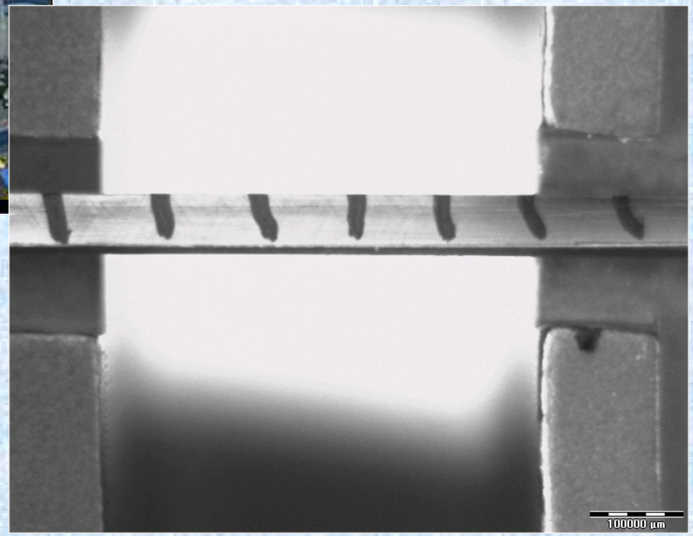
L. Thilly, APL (2007?)

CCD Camera



Tensile tester

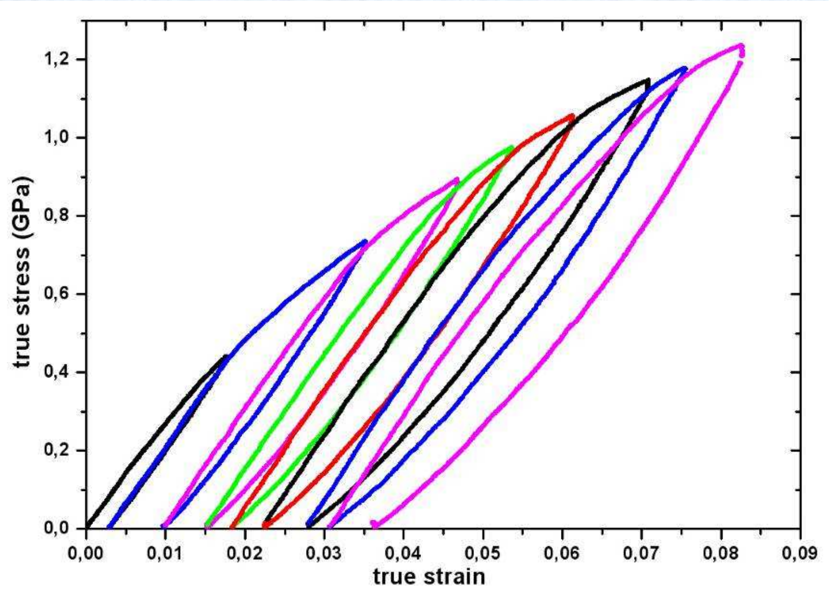
CCD Camera
↓
macroscopic deformation

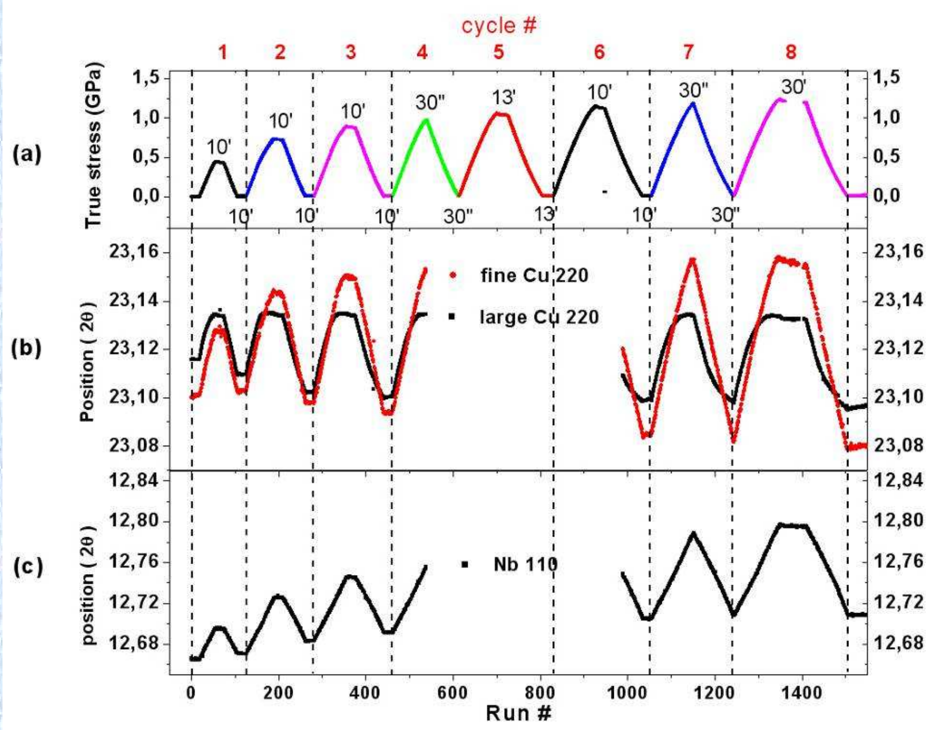


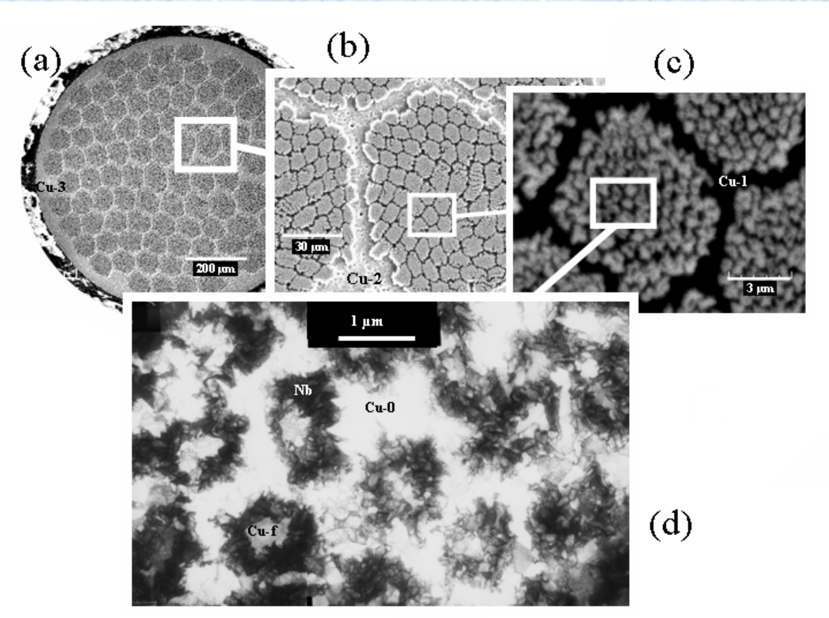
Curved Detector 60°

XRD
↓
microscopic deformation

L. Thilly, APL (2007?)

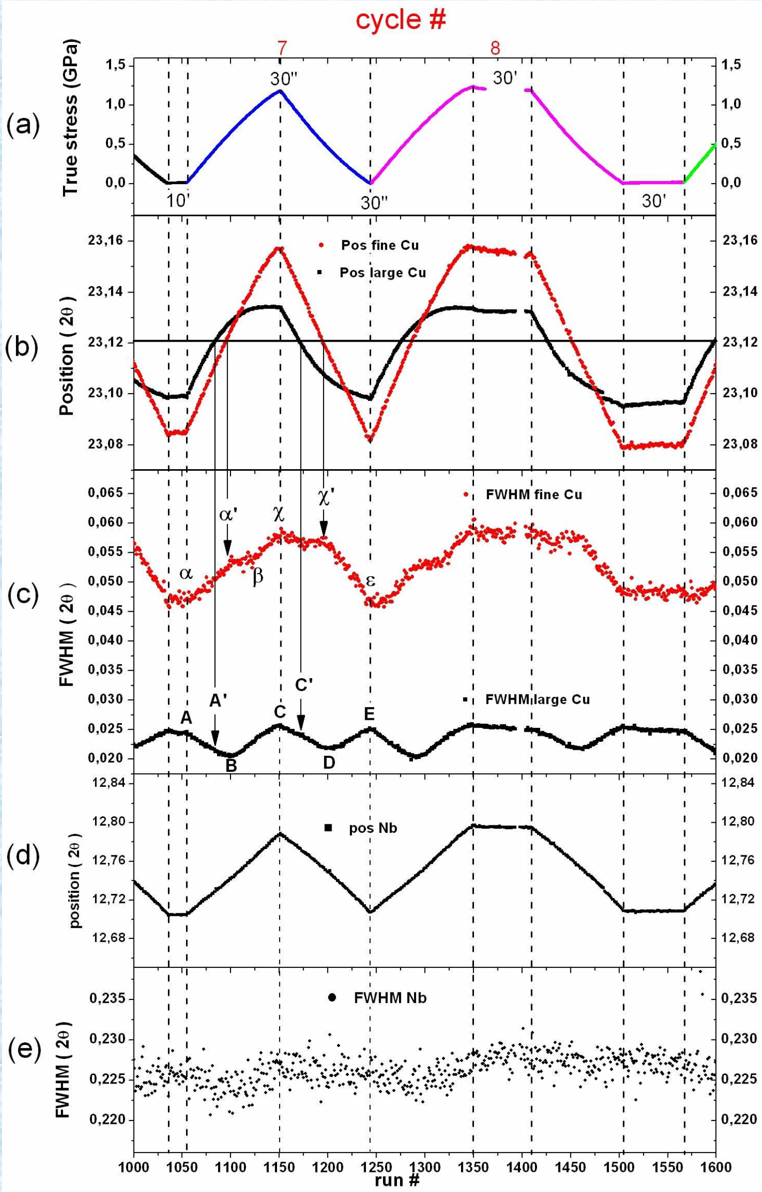






Thanks to XR we can « see » all the phases (2 (or 3))

L. Thilly, APL (2007?)



L. Thilly, APL (2007?)

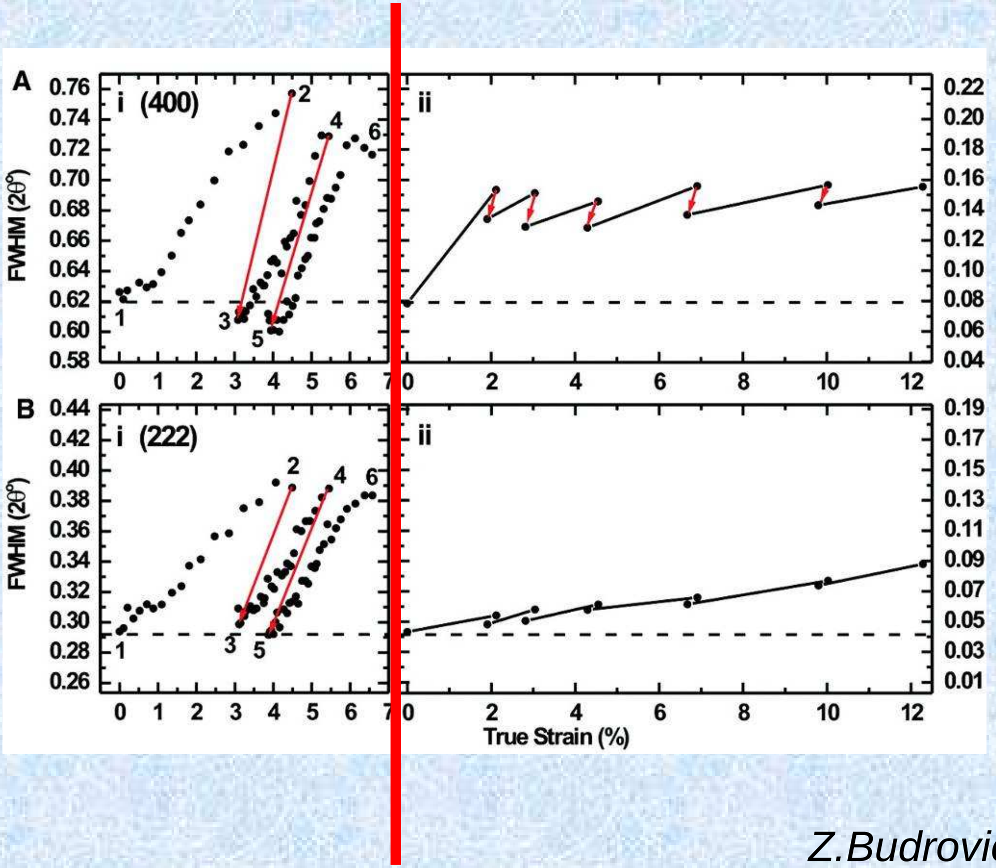
in situ deformation studies

→deformation mechanism of nanocrystalline Ni

In material with coarse grains → irreversible broadening of Bragg peaks

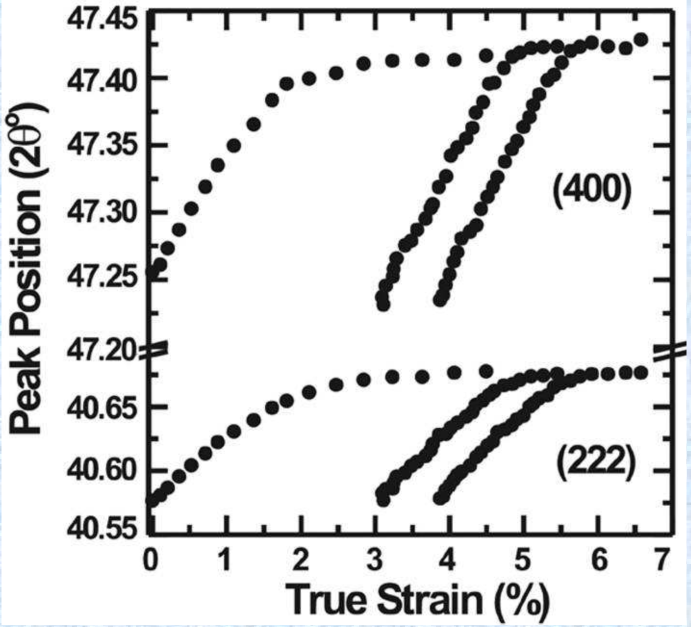
In material with nanocrystalline grains → reversible broadening of Bragg peaks

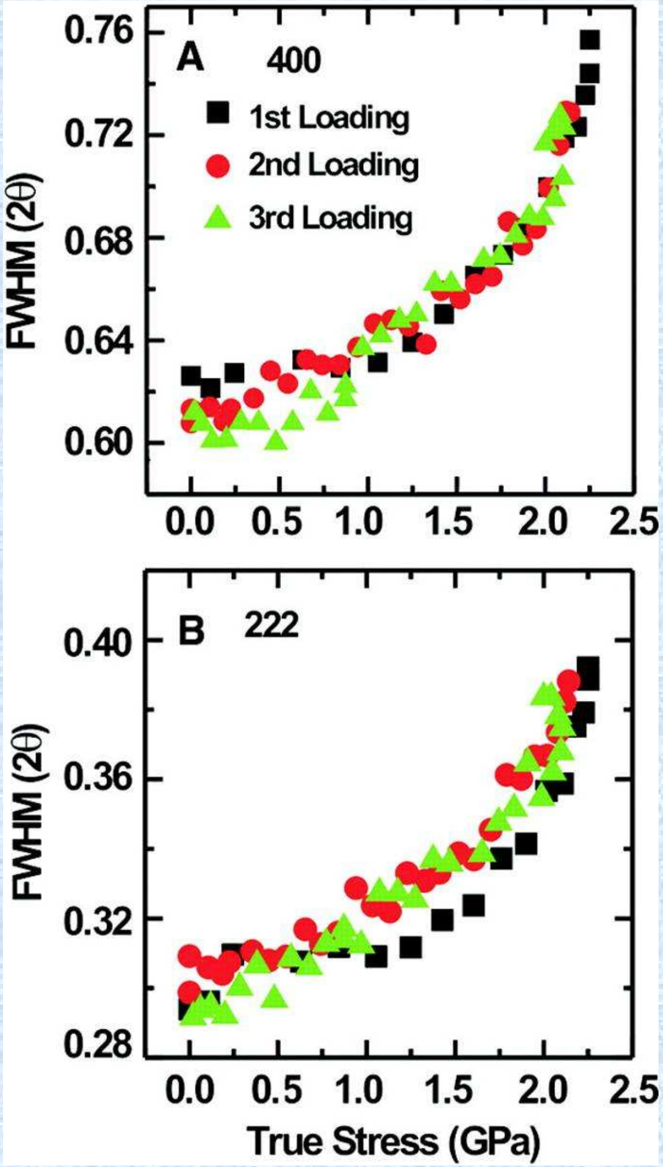
Nanocrystalline Ni



Microcrystalline Ni

Z.Budrovic et al. , science (2004)

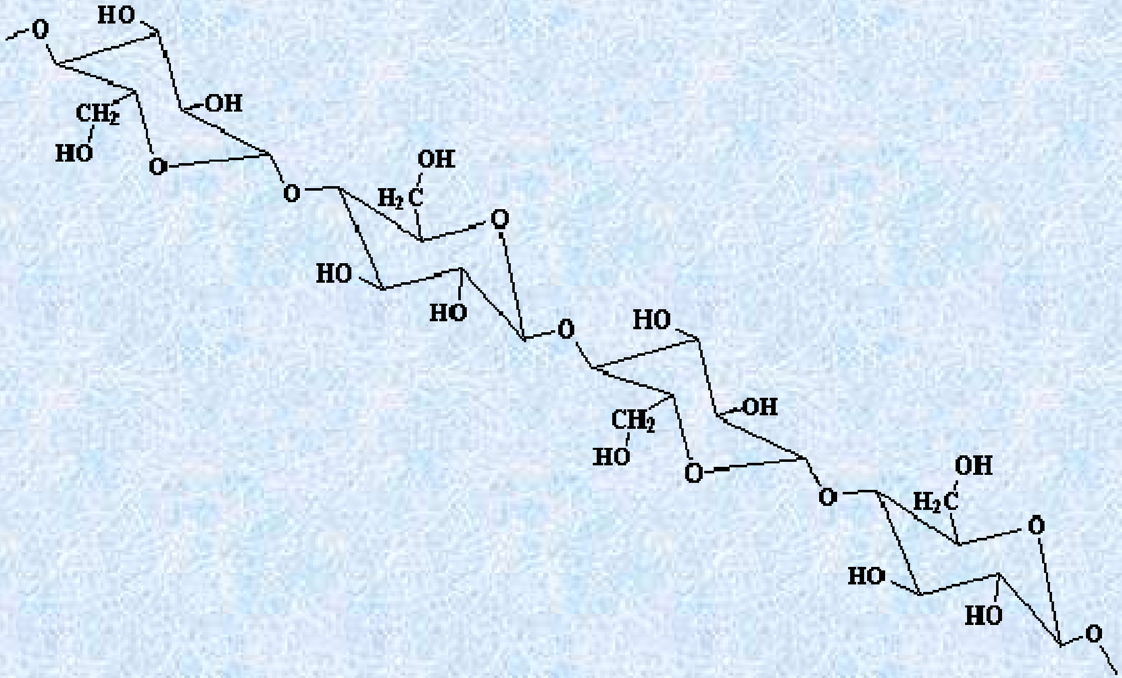




Z.Budrovic et al. , science (2004)

in situ deformation studies

→deformation mechanism of polymers

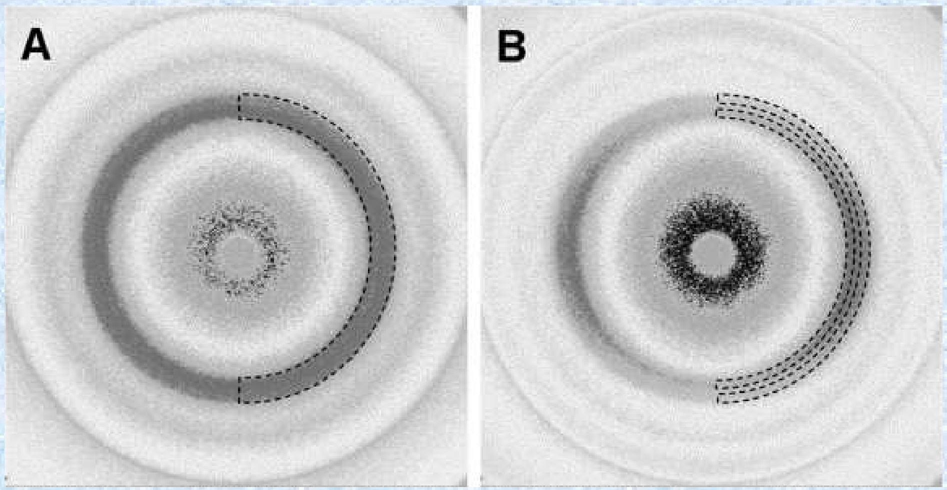


Cellulose is the most abundant biopolymer; it gives strength and stiffness to plant fibres (woods, cotton, paper…).

in situ deformation studies

→deformation mechanism of polymers

beginning (A) and at the end (B) of a tensile experiment

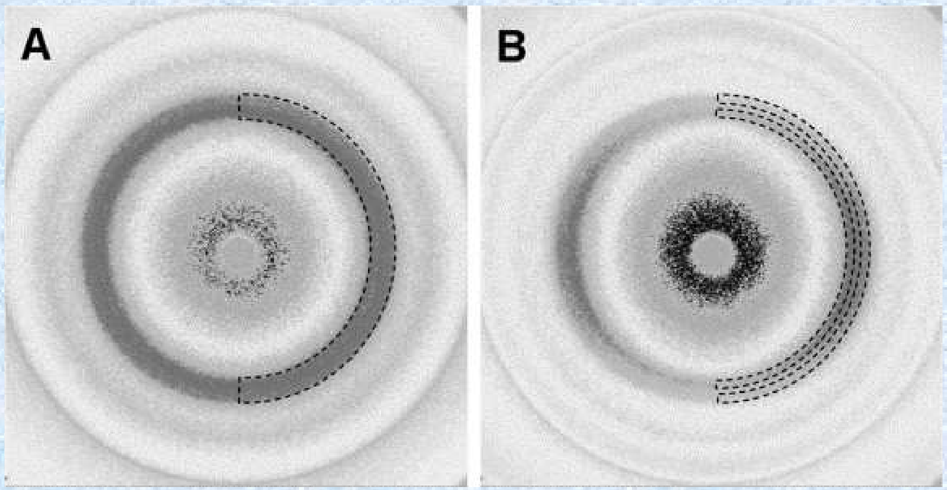


2D images allow analysing the texture of the cellulose fibers, i.e. to quantify the degree of orientation of the fiber in the cellulose as a function of the applied stress for example.

Wolfgang Gindl et al, 2006

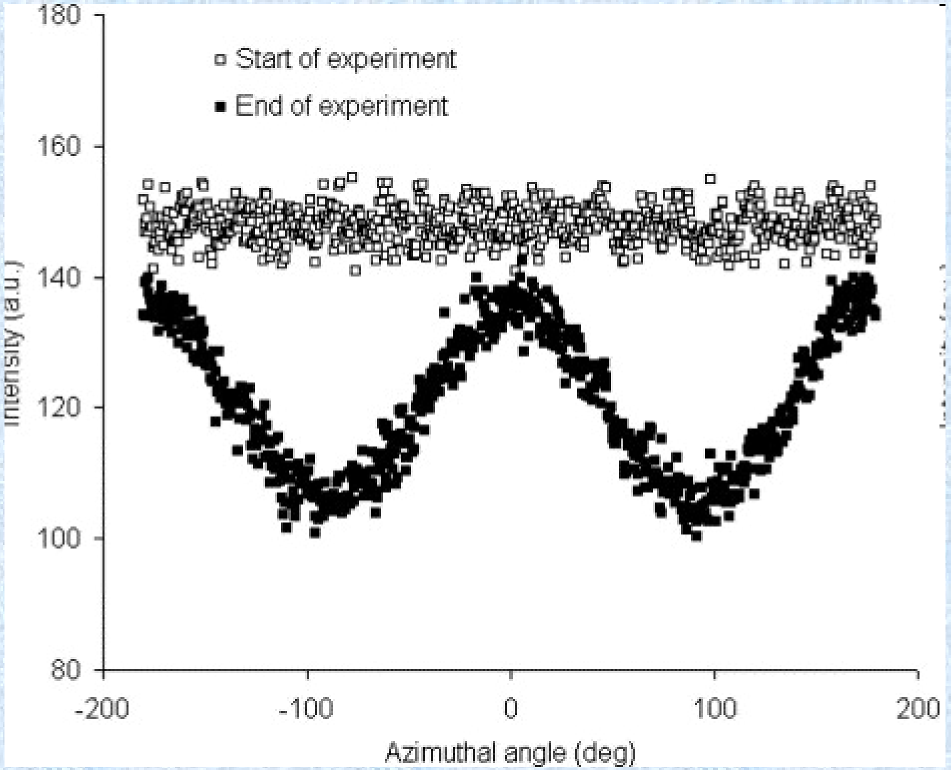
in situ deformation studies

→deformation mechanism of polymers

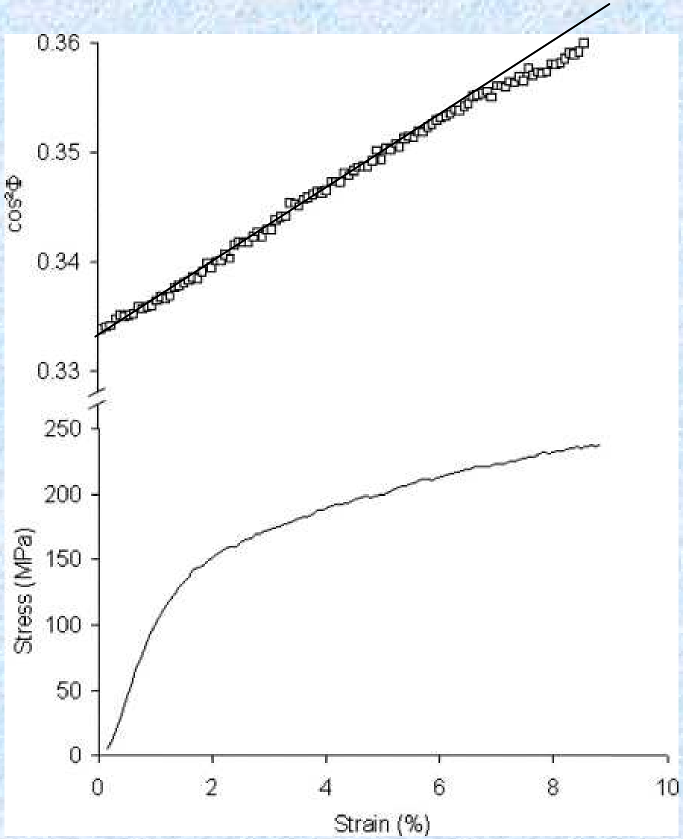


The inset arcs show half of the integrated area for the determination of the orientation factor. (A) The arc covers the cellulose II (1−10/200) reflections and the cellulose I (200) reflection. (B) For the separate determination of the orientation factor of cellulose I and cellulose II, the inner arc covers the cellulose II (1−10/200) reflections and the outer arc covers the cellulose I (200) reflection, respectively.

Wolfgang Gindl et al, 2006



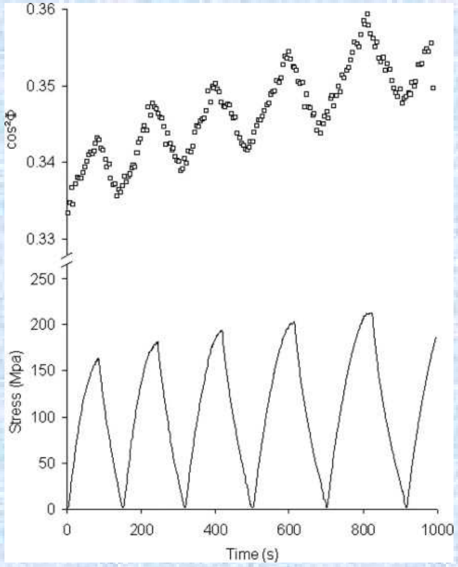
Example of integrated intensity distribution (combined cellulose II 1−10/200 and cellulose I 200 reflection) at the start and at the end of an experiment

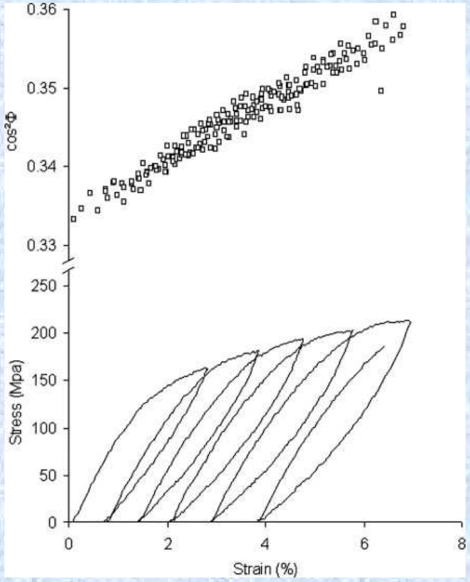


Evolution of the orientation factor as a function of deformation

Stress-strain curve

The correlation between the 2 curves allows seeing that the orientation of fibers depends linearly of the applied deformation.





Evolution of the orientation factor as a function of deformation

Stress-strain curve

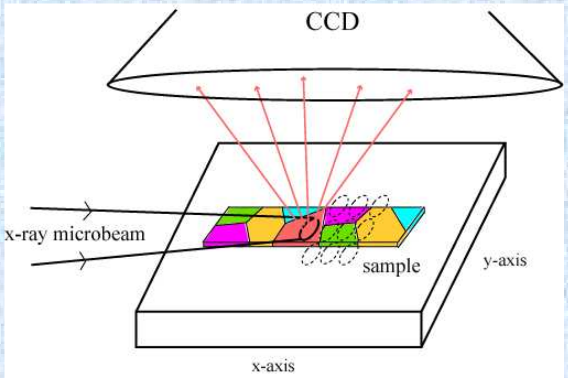
Conclusion:

Re-orientation of crystallite fibers into cellulose during deformation. The orientation factor is linear versus the deformation. After complete unloading, a given degree of orientation remains.

in situ deformation studies

→deformation mechanism at small scales

Principle: Scan the surface with a monochromatic beam. The beam size is smaller than 1x1 µm² to study heterogeneities of strain or composition in materials.



NOTE: Rotation of sample are forbidden *(Confusion Sphere larger than beam size): 2D detectors*.

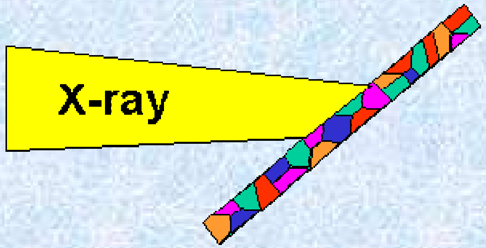
Applications: Micro electronic, Metallurgy, Biology, Soft matter, geology, art and archeology.

4-15 keV / 0.7 x 0.8 µm

Piezo X-Y stage

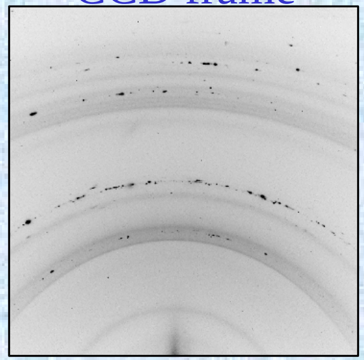
Ph. Goudeau, plasticité (2007)

Using 2D detectors



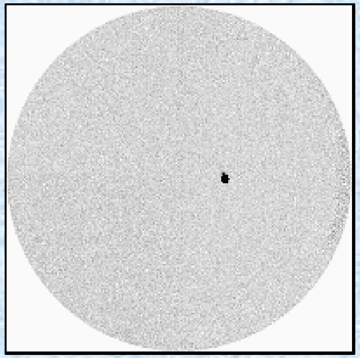
Beam size >> grain size

CCD frame

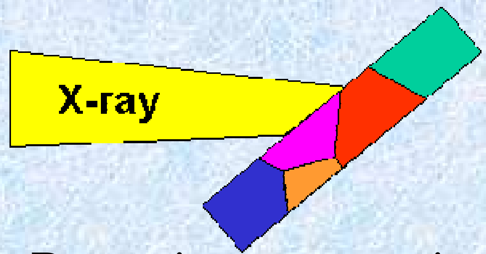


- Powder Diffraction
- Monochromatic beam
- rotation of sample is not necessary (except for texture analysis)

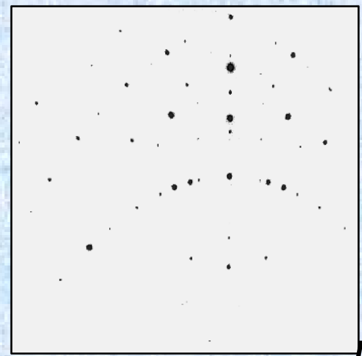
Beam size > grain size



- Single crystal diffraction with monochromatic beam
- Sample Rotation necessary
- confusion sphere can be a huge problem!



Beam size < or ~ grain size

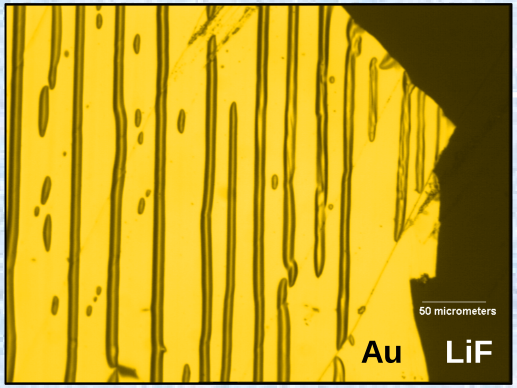


- Single crystal diffraction with polychromatic beam
- No sample rotation

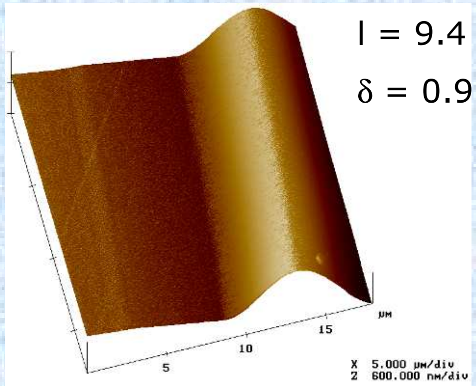
Ph. Goudeau, plasticité (2007)

in situ deformation studies

→deformation mechanism at small scales



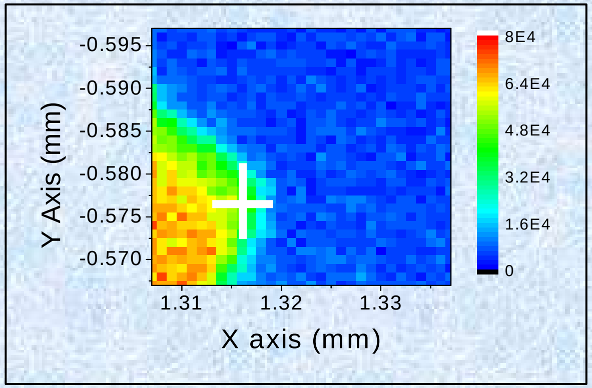
Plastic Def. = 1.7 %



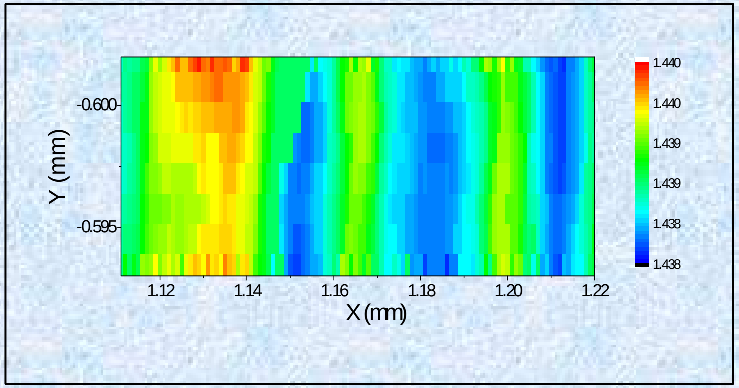
l = 9.4 μm

δ = 0.9 μm

Mapping with Fluorescence



$d_{(220)}$ carto.



→in situ measurement during compression

Ph. Goudeau, plasticité (2007)