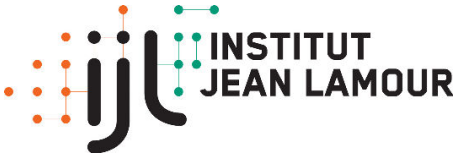


Stress determination by in-situ diffraction: laboratory, synchrotron, neutrons

Guillaume Geandier

Instrumentation & Méthodes d'analyses pour étudier le couplage Mécanique/Diffusion/Oxydation
05-06 June 2019

Context

Stresses are everywhere !

sources of residual / internal stresses :

- fabrication process
- microstructures modification (thermal treatment - phase transformation)
- mechanical process (cutting, formability, . . .)
- ageing
- oxidation
- . . .

Stresses (residual or internal) can be beneficial or catastrophic !

Context

Stresses are everywhere !

sources of residual / internal stresses :

- fabrication process
- microstructures modification (thermal treatment - phase transformation)
- mechanical process (cutting, formability, . . .)
- ageing
- **oxidation**
- . . .

Stresses (residual or internal) can be beneficial or catastrophic !

Stress determination

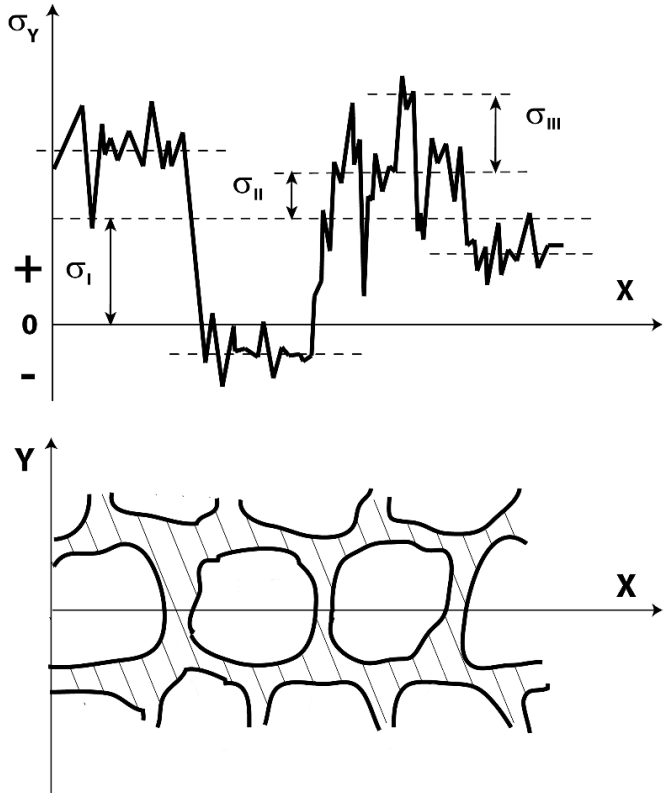
Different levels of stress in materials

- sample scale
- grain scale
- intra granular scale

using diffraction methods

- phase scale

- laboratory scale
- large scale facilities : synchrotron sources and neutrons



Overview

Stress determination by in-situ diffraction: laboratory, synchrotron, neutrons

Introduction

Laboratory
- Common analysis
- Stress determination

on synchrotron
- low energy - reflexion mode
- Laue method
- High energy diffraction

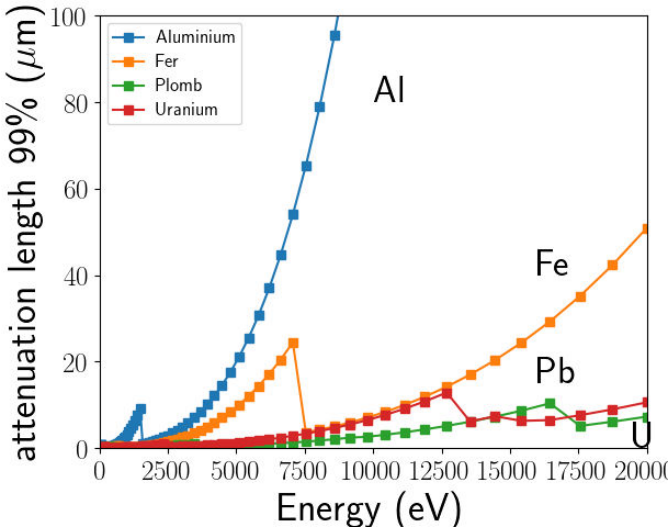
using neutrons

Conclusions

Aknowledgments

Common analysis

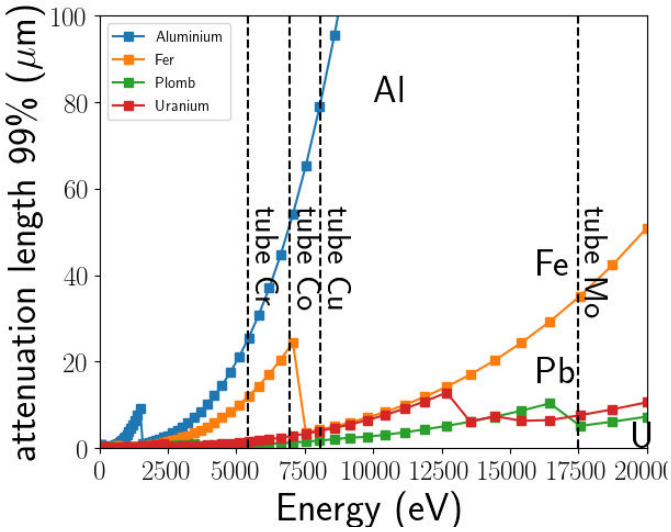
in laboratory



tubes : Cr, Co, Cu, W, ...

Common analysis

in laboratory

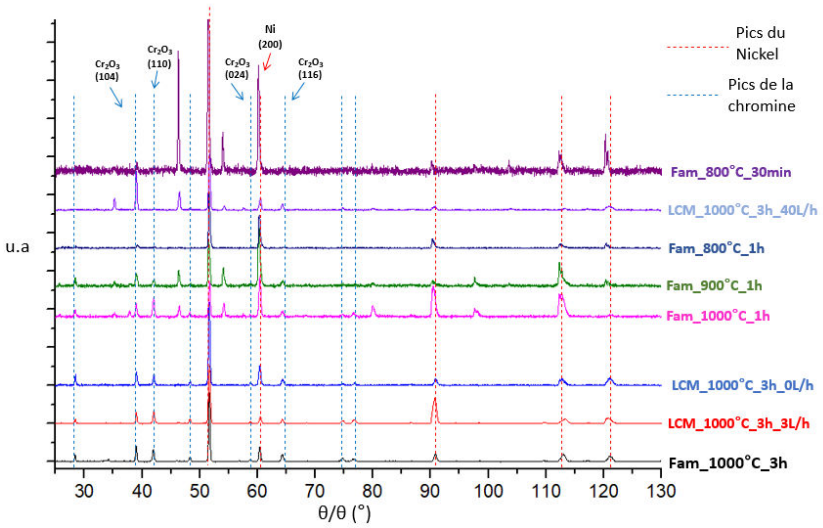


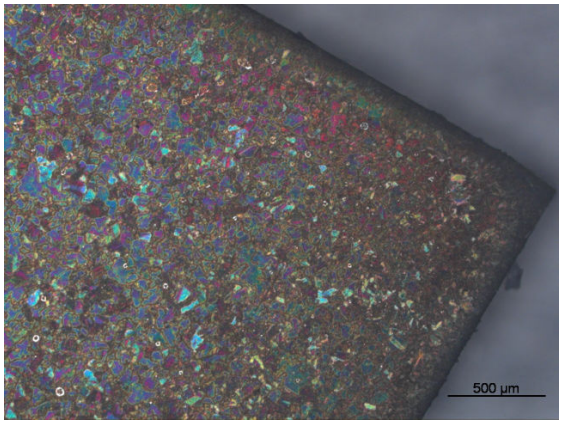
tubes : Cr, Co, Cu, W, ...

Common analysis

in laboratory

1^{rst} step : phase identification, ex : Ni38Cr.





→ X-ray are phase selective !

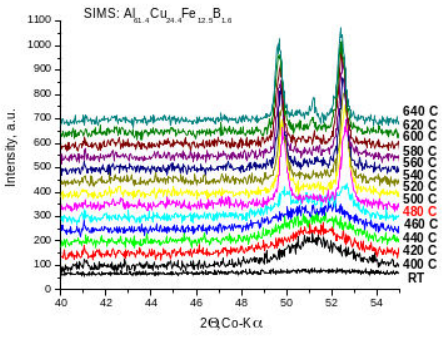
Common analysis

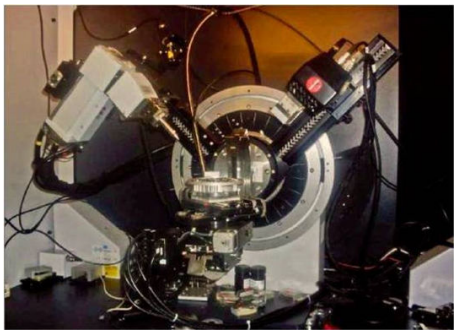
in laboratory

Temperature analysis :

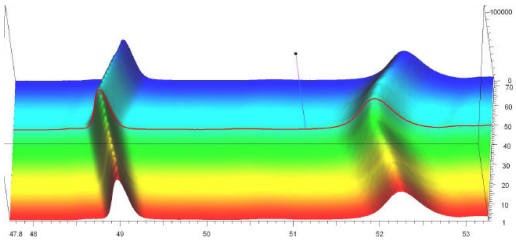
- TTK450 : N2liq – 450 K
- HTK1200 : Tamb – 1200 ° C
- XRK900 : Tamb – 900 ° C
- DHS1100 : Tamb – 1100 ° C

PVD Characterization as a function of temperature



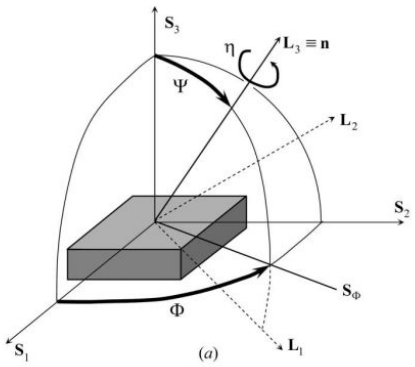


thermal expansion characterization of epitaxial layer

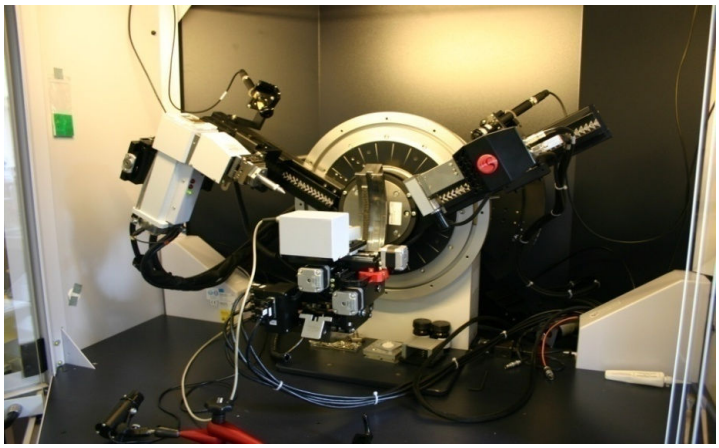


Stress determination

in laboratory



Stress reference system in sample or laboratory space.



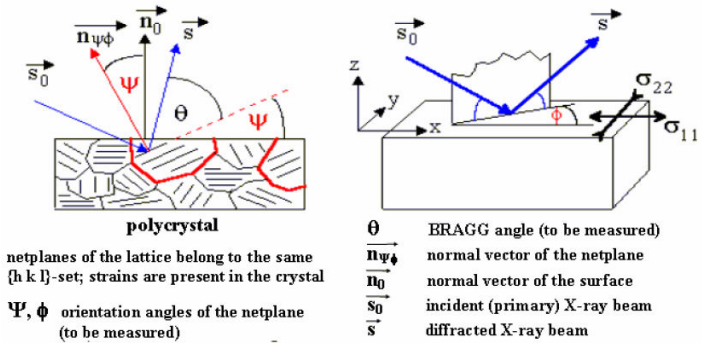
4 circles goniometer : ω, θ, ϕ, χ (ou ψ).

M. François J. Appl. Cryst. (2008). 41, 44–55

Stress determination

Diffraction case

1^{er} order (sample scale) - $\sin^2\psi$ method



Hypothesys

- homogeneous and isotropic material
- elastic strain

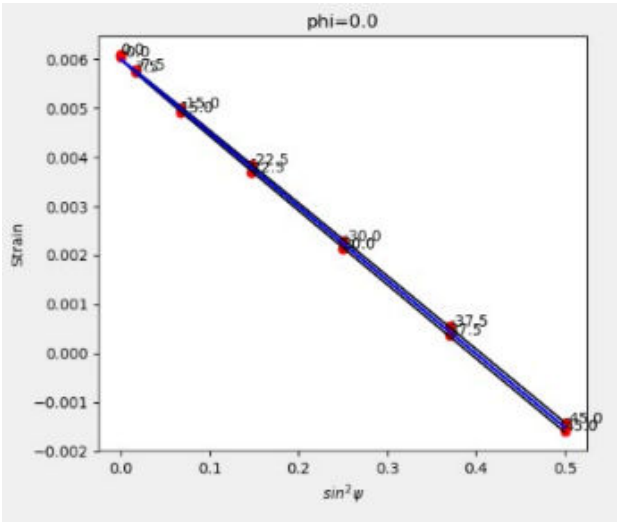
σ_ϕ : Stress component along ϕ direction
d_0 : reticular distance without stress
$d_{\phi,\psi}$: reticular distance under stress
ψ : declinaison angle
E: Young's modulus
ν : Poisson's ratio

Strain in along (ϕ, ψ) direction is related to the elements σ_{ij} of stress tensor using:

$$\epsilon_{\phi\psi} = \frac{1}{2}S_2[(\sigma_\phi - \sigma_{33})sin^2\psi + \sigma_{33} + (\sigma_{13}cos\phi + \sigma_{23}sin\phi)sin2\psi] + S_1(\sigma_{11} + \sigma_{22} + \sigma_{33}) \quad (1)$$

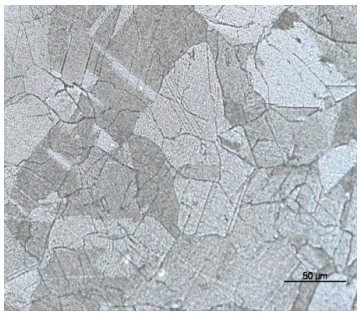
Stress determination

in laboratory

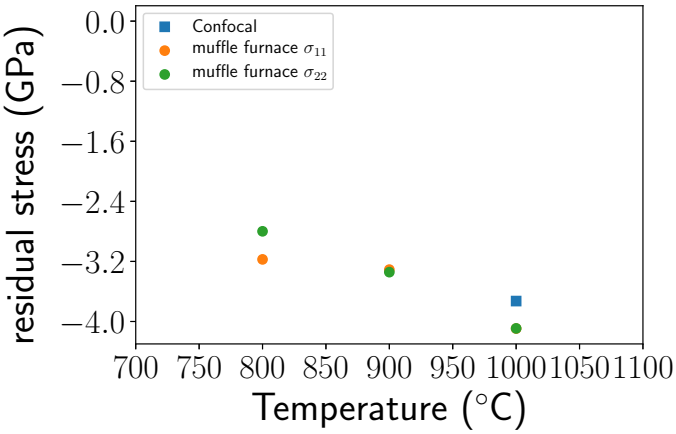


residual stress : -3977 ± 50 MPa

X-Light software
(https://github.com/sangpham171/X-Light)



Ni38Cr



Stress determination

in laboratory

Stresses determination using laboratory devices.

- residual stresses determination
- different kinds of characterization methods
- flexible

- time consuming
- limited energy range (tubes)
- reflection mode for bulk samples
- limited penetration depth (normal stress undetermined)

Stress determination

in laboratory / on synchrotron(s)

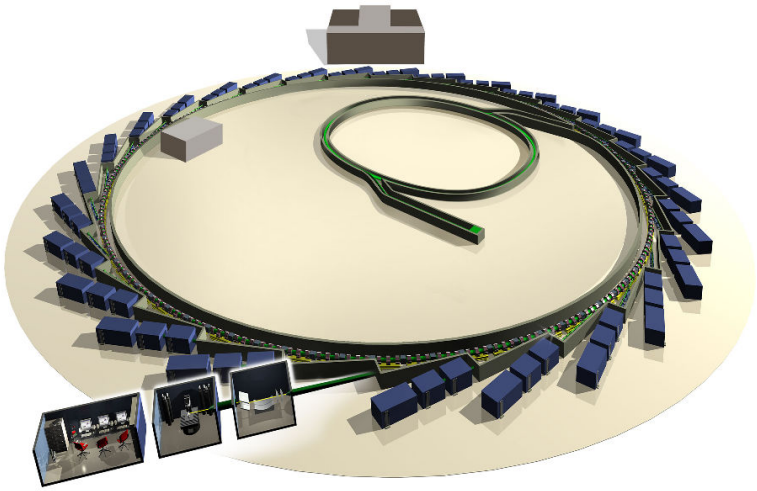
Stresses determination using laboratory or synchrotron devices.

- residual stresses determination + internal stresses
- different kinds of characterization methods + coupled techniques
- flexible

- time consuming → experiments planned over 1 year + lot of data and data analysis
- limited energy range (tubes) almost no limit
- reflection mode for bulk samples almost no limit
- limited penetration depth (normal stress undetermined) almost no limit and full tensor determination

Stress determination

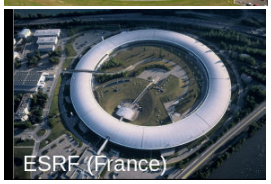
in laboratory / on synchrotron(s)



Schematic of Diamond Light Source, showing the components that make up the synchrotron.

→ more than 50 light sources worldwide (synchrotron, XFEL) lightsouces.org

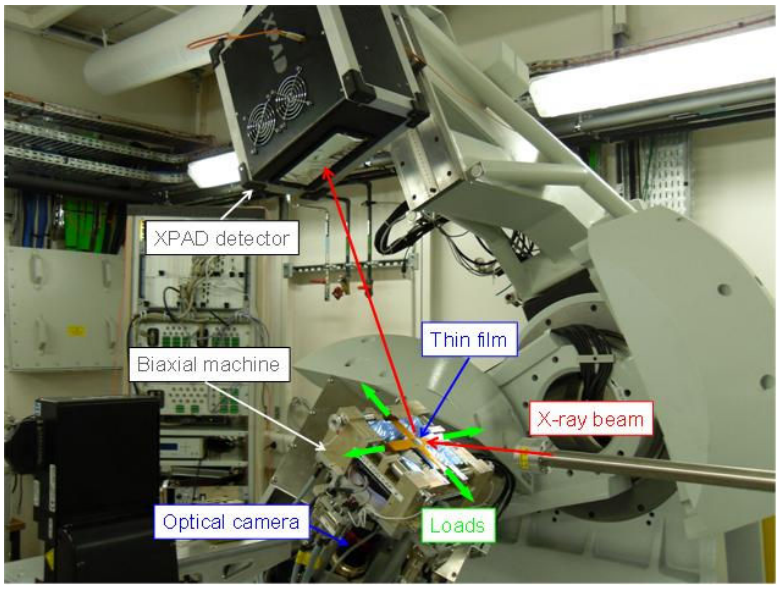
Energy : 1meV to 750 keV —- beam size : 50nm – cm



Internal stress determination

on synchrotron(s)

Experimental set-up at DiffAbs beamline at SOLEIL.

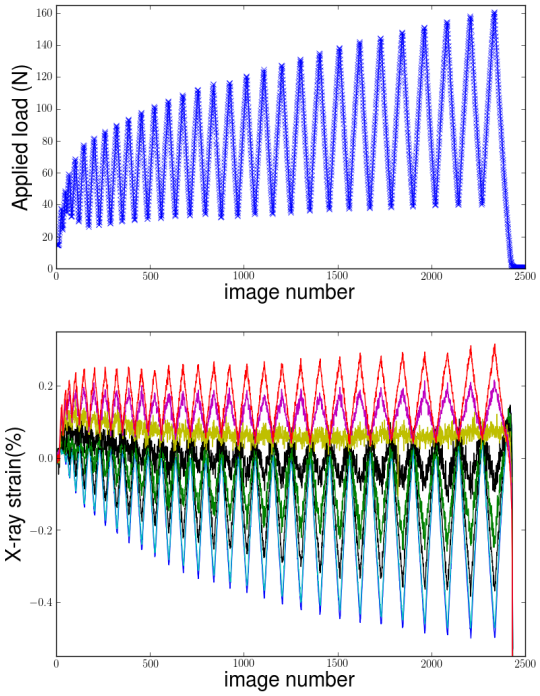


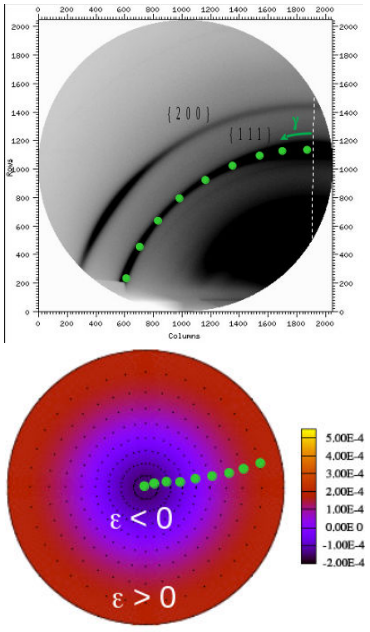
In-situ biaxial tensile test combined with X-rays diffraction and image correlation techniques.

XRD lattice strains
DIC true strains

Internal stress determination

on synchrotron(s)



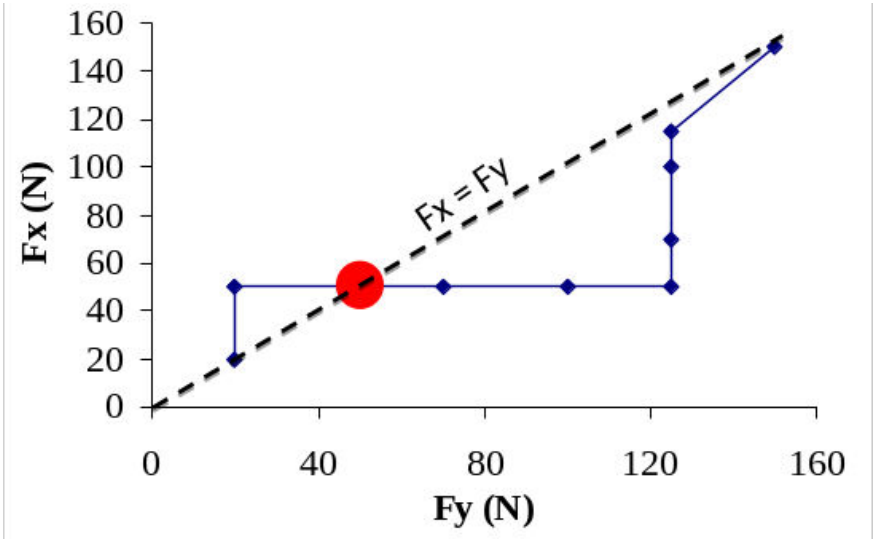


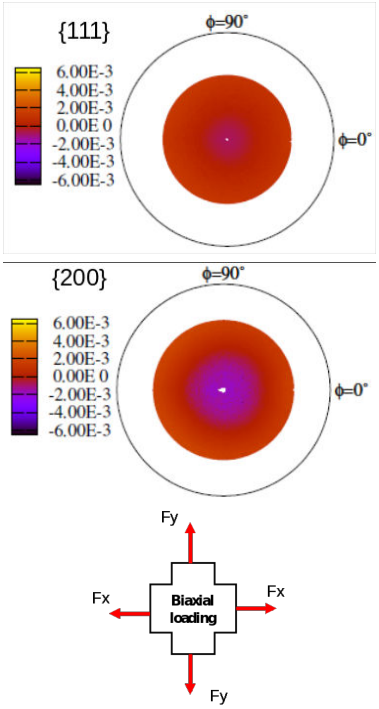
{111} Strain Pole Figure

Internal stress determination

on synchrotron(s)

Equi-biaxial loading

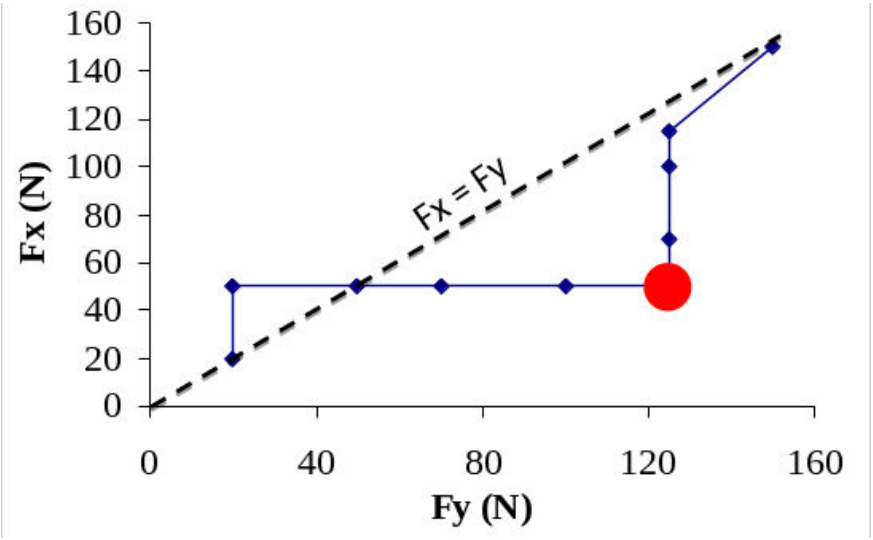


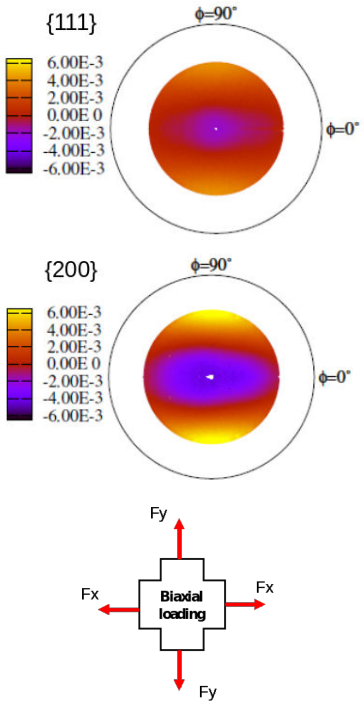


Internal stress determination

on synchrotron(s)

Non equi-biaxial loading

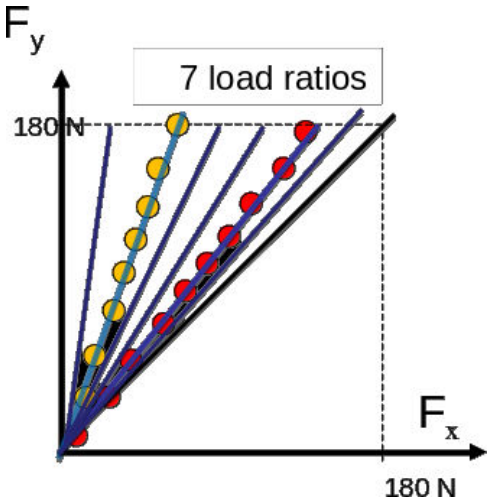


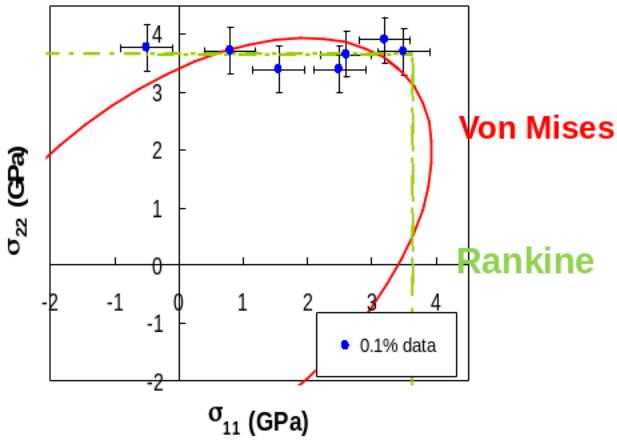


Internal stress determination

on synchrotron(s)

In situ biaxial tensile test combining XRD and DIC
Mechanical behaviour of thin film:
Yield surface: Brittle behaviour





Djaziri et al., Acta Mater. 61 2013 // Renault et al., ICMCTF May, 2013

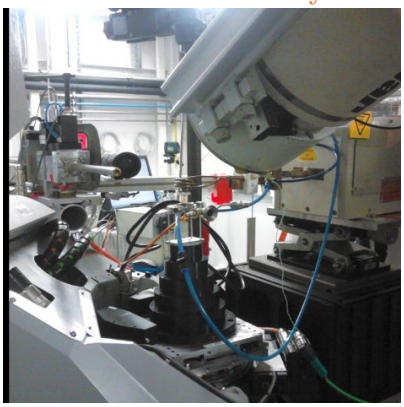
Internal stress determination

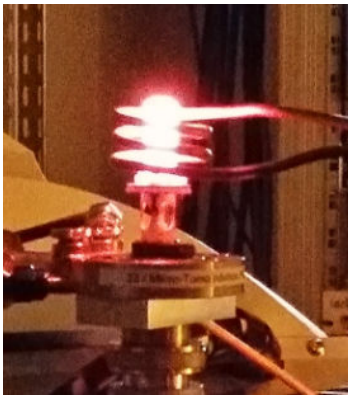
on synchrotron(s)

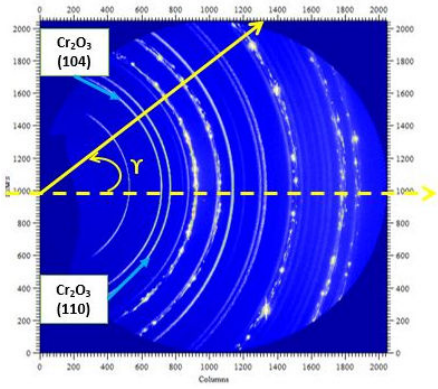
Ni-30Cr (non-textured alloy): nickel containing chromium alloy

	% Ni	% Cr	% Si	% Mn	C	P	S
Ni-30Cr	69.65	30.22	inf0.01	¡ 0.01	230 ppm	30 ppm	40 ppm

Synchrotron radiation and in situ measurements



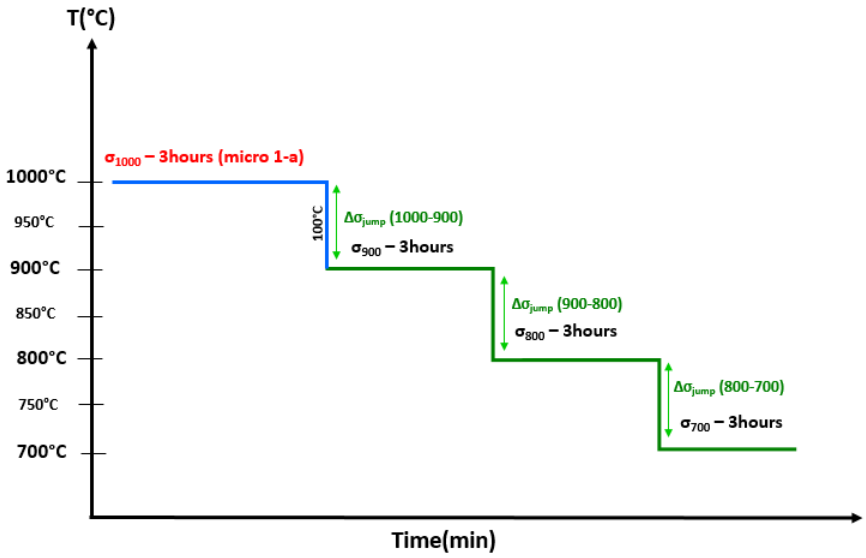




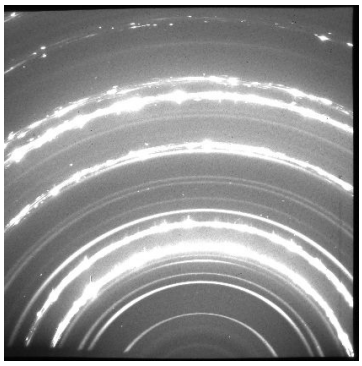
Beamline BM02, ESRF Grenoble

Internal stress determination

on synchrotron(s)



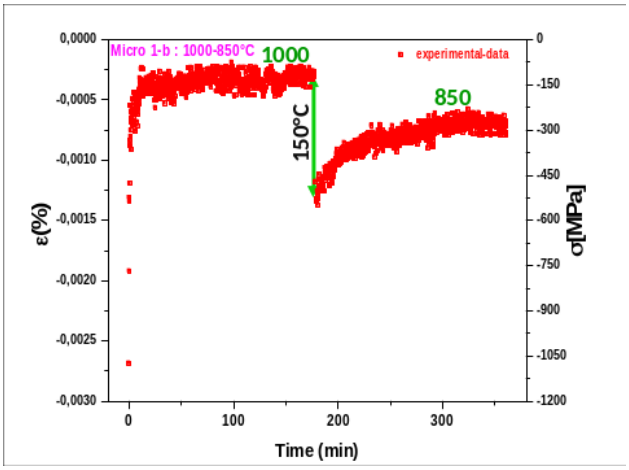
During thermal treatment : continuous recording of 2D images
1 test == 3700 images

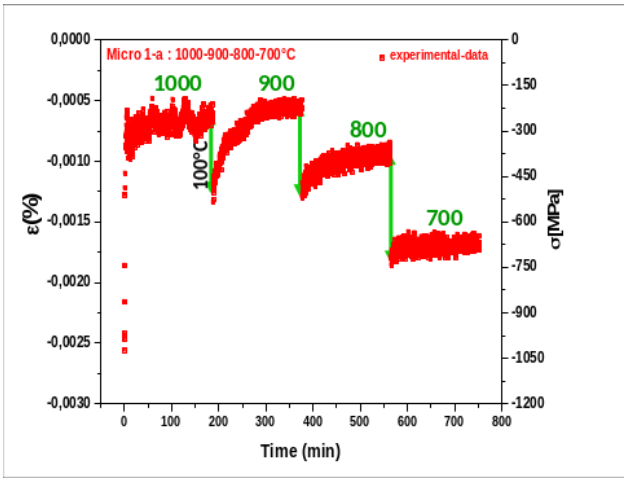


Rakotovao F. PhD thesis, Université de la Rochelle, 2016

Internal stress determination

on synchrotron(s)

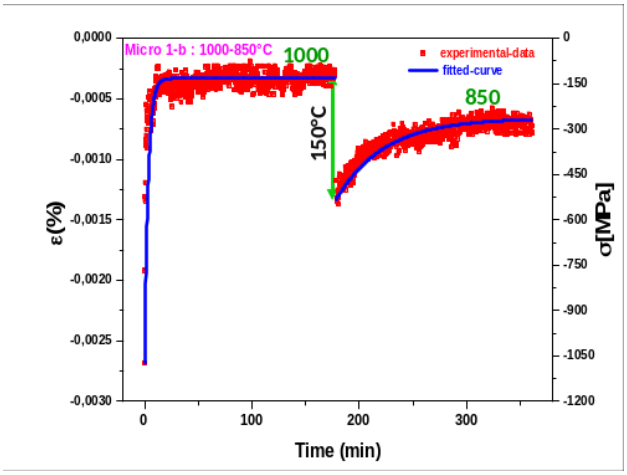


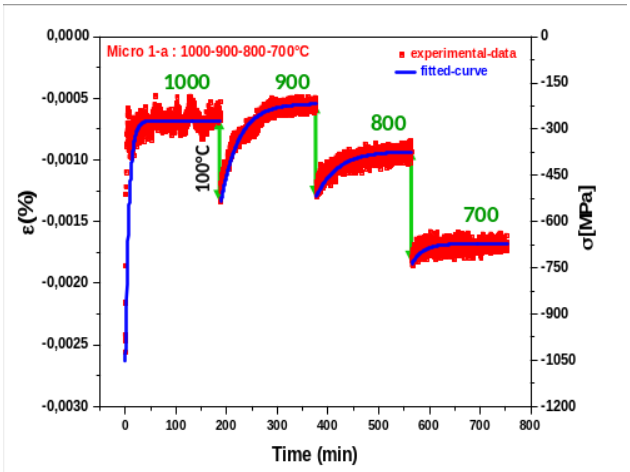


Rakotovao F. PhD thesis, Université de la Rochelle, 2016

Internal stress determination

on synchrotron(s)



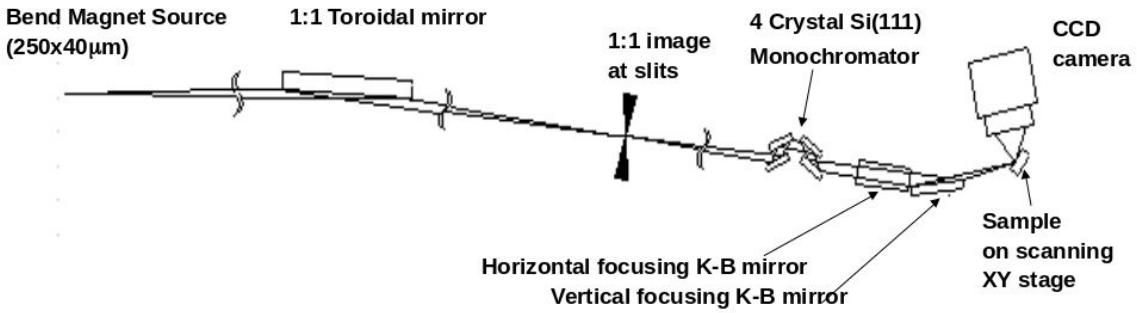


With n=1, fitted curves well describe experimental behaviour
→ evidence of diffusion-creep relaxation in Cr_2O_3

Rakotovao F. PhD thesis, Université de la Rochelle, 2016

Internal stress determination

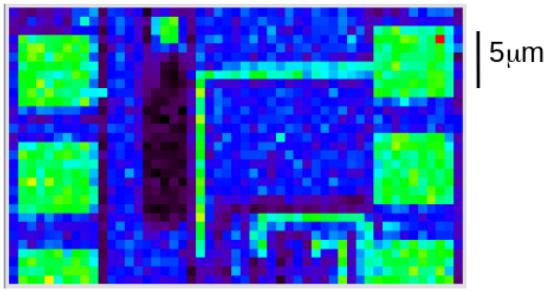
sur synchrotron(s) : Laue method



Beam size on sample: 0.8 x 0.8 μm^2

Photon energy range : 5-25 keV (pink beam)
monochromatic beam – method available

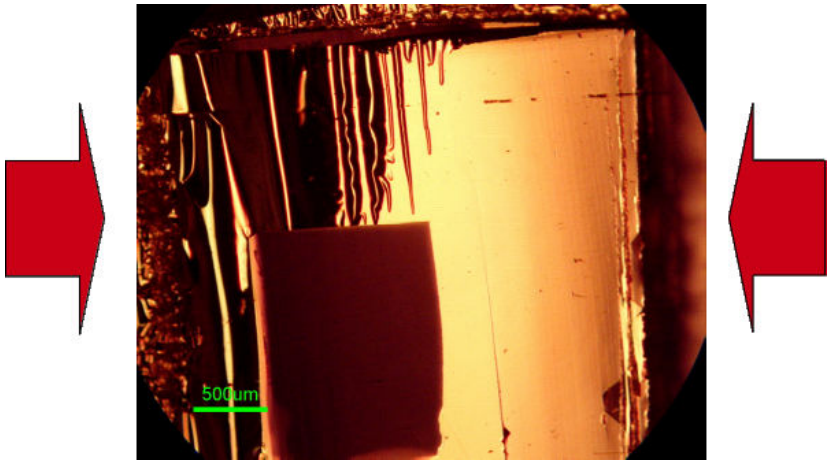
fluorescence



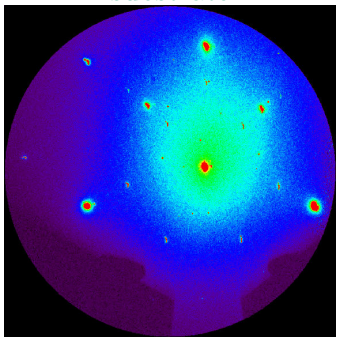
Internal stress determination

sur synchrotron(s): Laue method

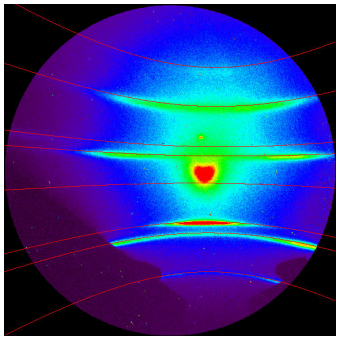
Compression of thin film deposited on monocrystalline substrate
combining Laue microdiffraction / monochromatic microdiffraction



substrate

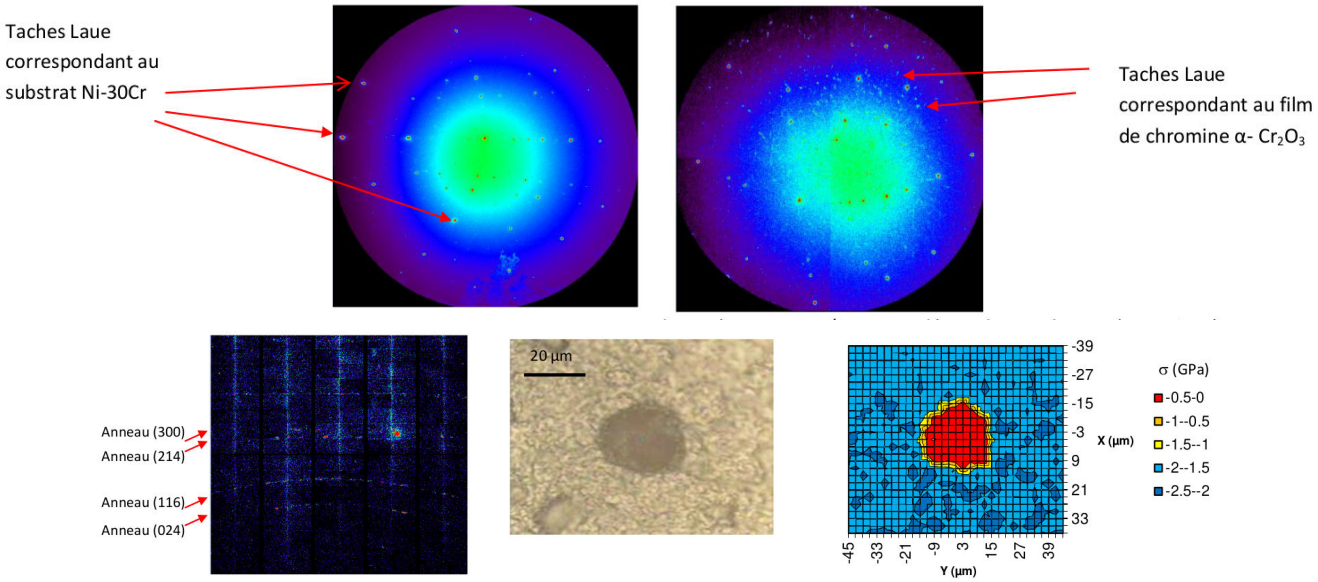


thin film



Internal stress determination

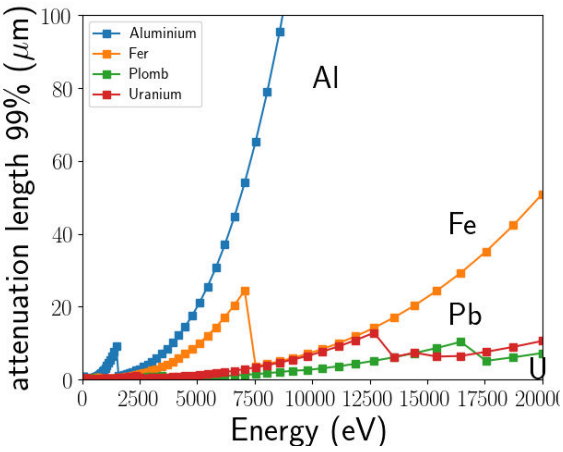
sur synchrotron(s): Laue method



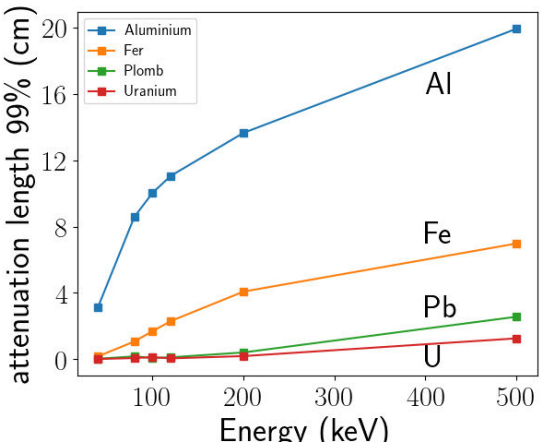
Guérain M. PhD thesis, Université de la Rochelle, 2012

Internal stress determination

on synchrotron(s) : high energy



Laboratory / low energy beamlines

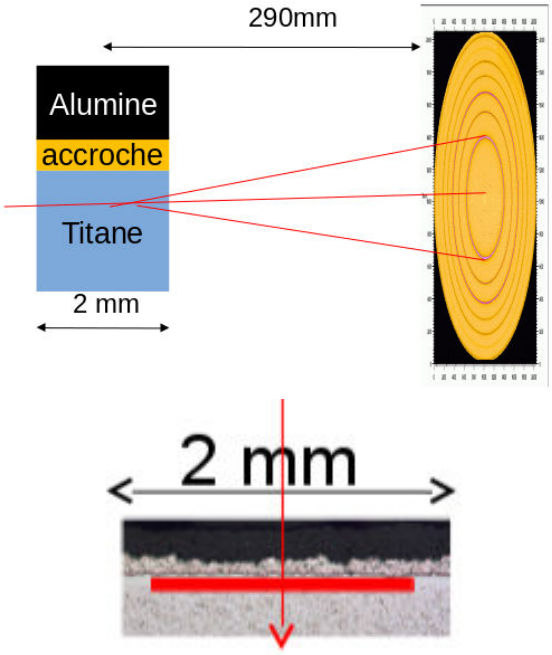


High energy beamlines

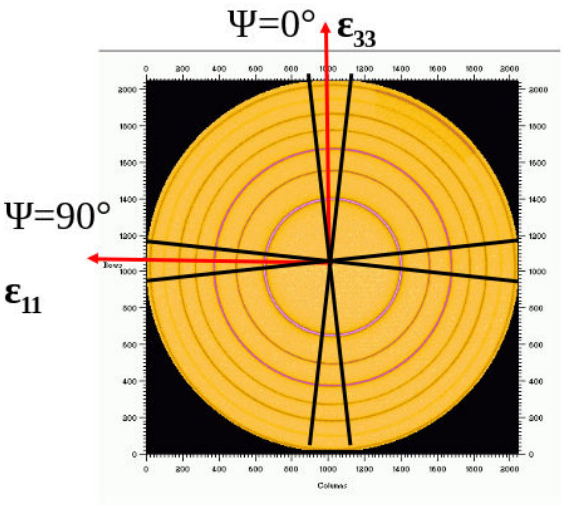
Internal stress determination

on synchrotron(s) : high energy

ID11 : Transmission, E= 80 keV



Beam size: h: 5 μm v: 300μm



Sample rotation (0°, 90°)

Internal stress determination

on synchrotron(s) : high energy

- multilayer material
- multiphase material

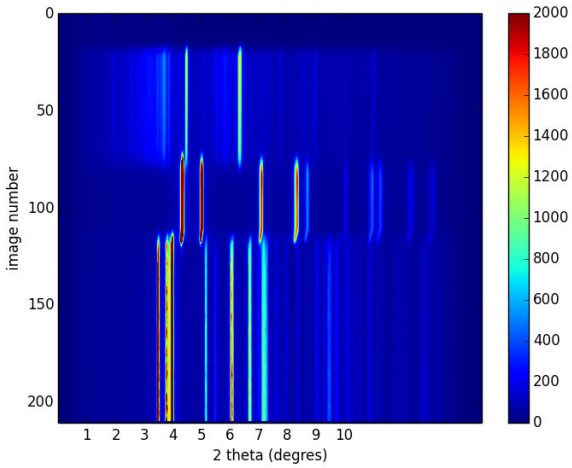
Stress determination in each phase
Stress determination along each direction
→ Full tensor determination along depth

One complete analysis:

- 100 2D images (scan over 500μm) for each profile
  → 15 for Alumina
  → 10 for hanging layer
  → 80 for Titanium
- 2 directions: ϕ=0° and 90°
- 4 phases : 1 in alumina, 1 in hanging layer and 2 in titanium alloys

→ **130000 peaks** to analyze

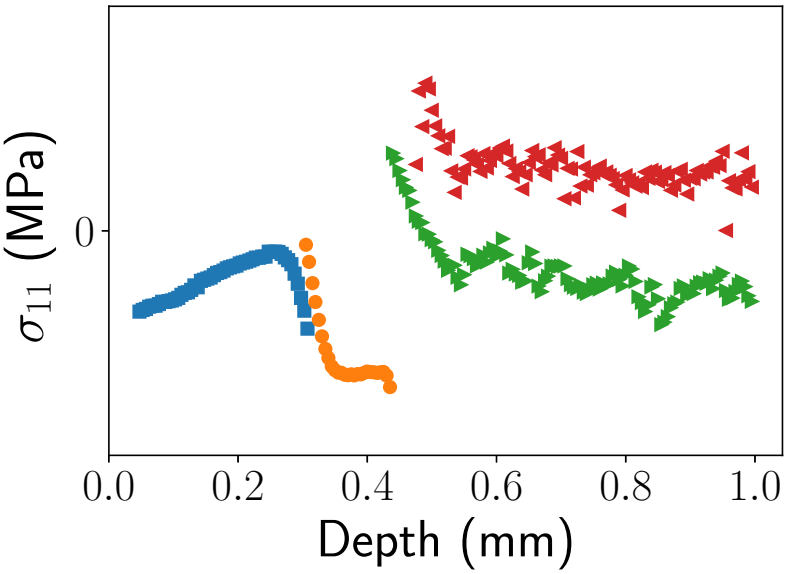
Phase mapping along depth

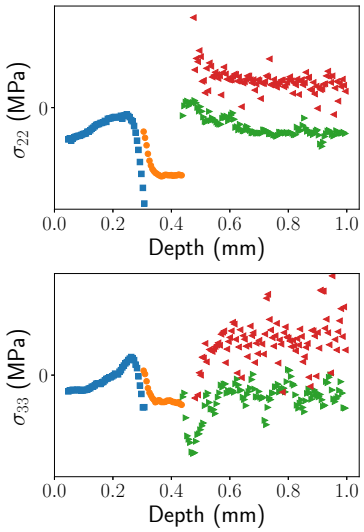


Internal stress determination

on synchrotron(s) : high energy

residual stress profils at a function of depth:



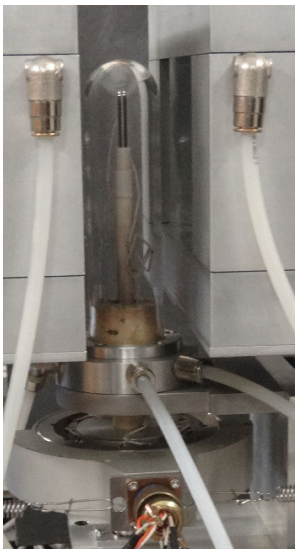


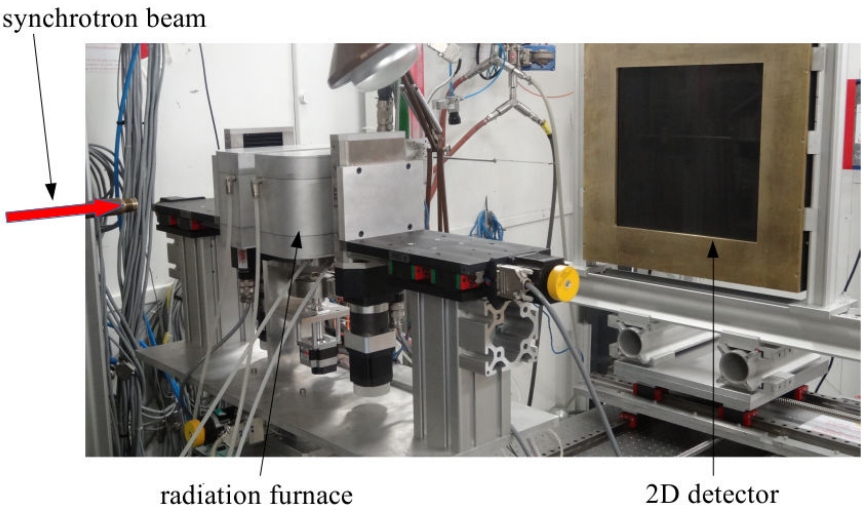
Internal stress determination

on synchrotron(s) : high energy

- Radiation furnace
- Welded thermocouple
- Argon atmosphere
- Sample rotation
- ID15B (ESRF)

- High energy beam (87 keV)
- Transmission geometry
- 2D detector
- Acquisition rate : 10 frames / s
- Beam size : 400 x 400 μm2

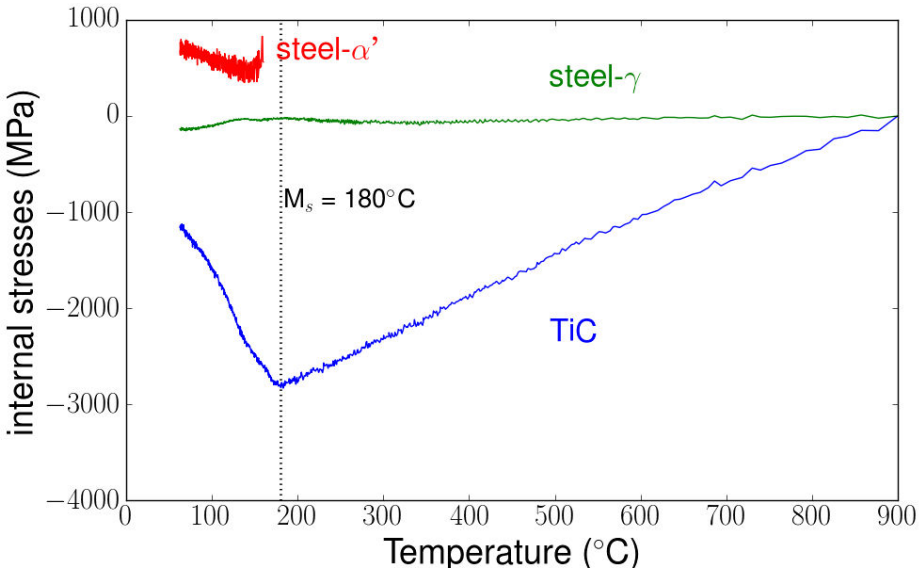




Internal stress determination

on synchrotron(s) : high energy

Mean stresses in MMC versus temperature



stresses
$\sigma_{11} = \sigma_{22} = \sigma_{33}$
σ_{12}, σ_{13}, σ_{23}
negligible

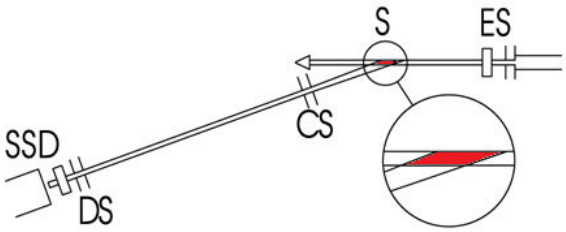
Geandier et al, Materials 2018, 11, 1415; doi:10.3390/ma11081415

Internal stress determination

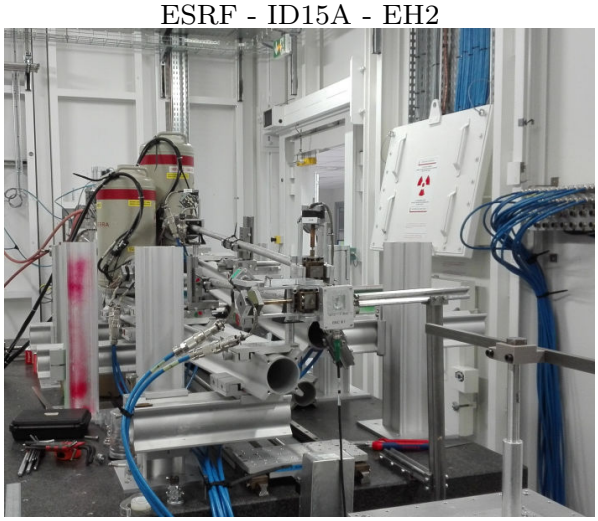
on synchrotron(s) : high energy

Energy dispersive setup

- fixed angle
- white beam: 50 - 300 keV
- 3D scans
- Energy dispersive detectors



ESRF - ID15A - EH2



Internal stress determination

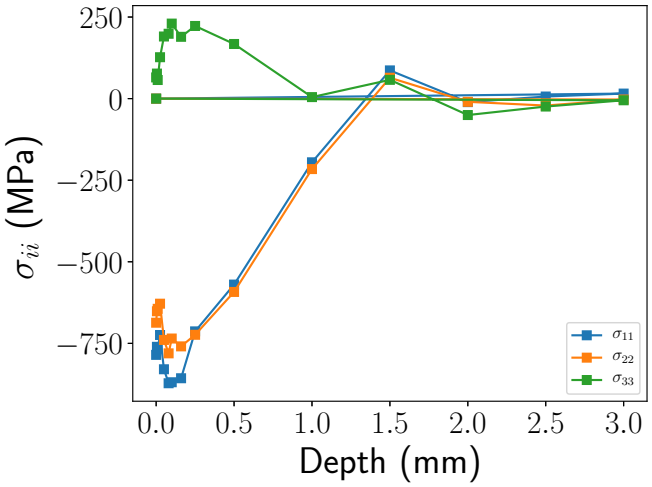
on synchrotron(s) : high energy

Energy dispersive setup

GFAC samples

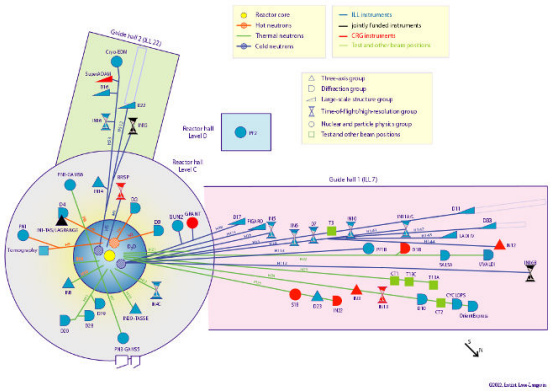
- Full stress tensor determination
- shot-peened samples
- mono phase sample : inconel
- no material removal

→ good agreement between synchrotron and laboratory (with material removal) measuremnts



Stress determination

with neutrons



ILL experimental hall

→ 43 neutron sources worldwide neutronsouces.org

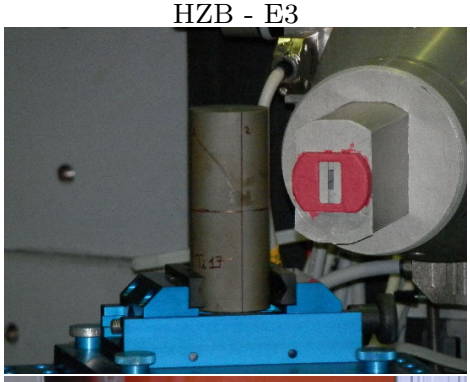
Stress determination

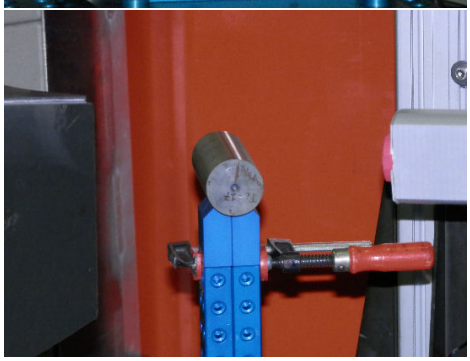
with neutrons

- large penetration depth
- large gauge volume
- heavy samples
- full stress tensor

Example:
Titanium alloys
Cylinder (h 200mm, 40mm diameter)

HZB - E3



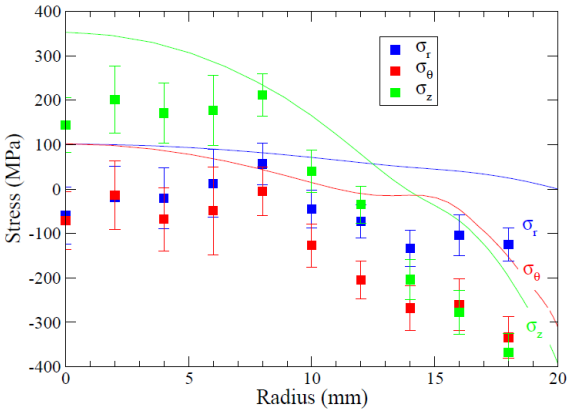


Residual stress determination

with neutrons

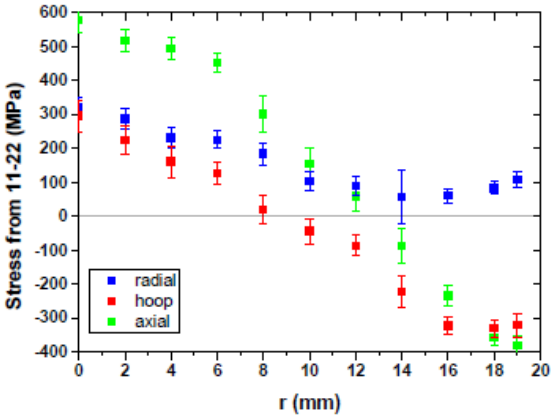
Experimental study and simulation of stress genesis during titanium alloys quench: phase transformation effects ?

Ti17 alloy (without phase transformation)



Higher Stress : phase transformations effects ?

Ti17 alloy (with phase transformation)



D. Maréchal et al., colloque GFAC 2016

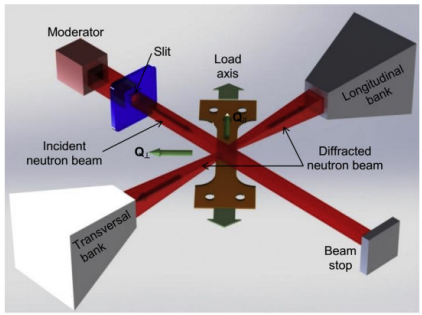
Internal stress determination

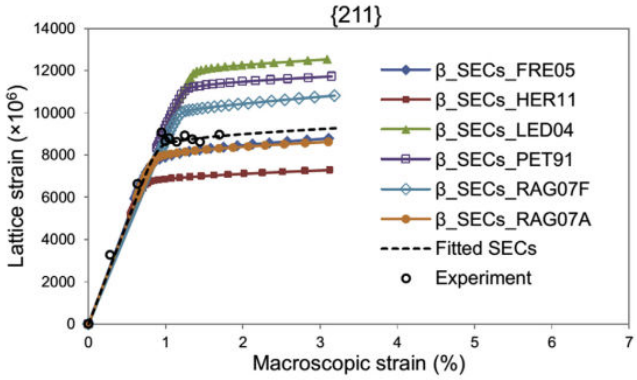
with neutrons

in-situ experiments

neutron setup → large sample environments

- tensile setup
- furnace (RT - 2000K)
- cryostat (4K - TR)
- magnets
- ...





Hounkpati et al. Acta Materialia 109 (2016) 341

Stress determination

Laboratory, synchrotrons, neutrons

	Laboratory	Synchrotron	Neutrons
residual stress	OK	OK	OK
internal stress	variable	OK	OK
multiple techniques	OK	OK	OK
flexibility	OK	OK	OK
time consuming	measurement	preparation data analysis	preparation data analysis
diffraction mode	reflection	reflection / transmission / small angles / ...	transmission / small angles
energy	limited by tubes	no limit	limited
analyzed volume	surface / μm^3	μm^3 to mm^3	mm^3 to cm^3
stress determination	biaxial	full tensor	full tensor

Laboratory, synchrotron and neutrons are complementary.

Large scale facilities are open for users !

Stress determination

Laboratory, synchrotrons, neutrons

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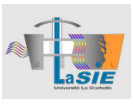
P.O. Renault, E. Le Bourhis, P. Godeau, R. Guillou, S. Diaziri. B. Girault

B. Panicaud



J.L. Grosseau-Poussard, F. Rakotovao, M. Guérain

B. Malard

F. Lefebvre, E. Wasniewski

P. Boulet, C. Rampelberg, B. Denand, L. Vautrot, D. Maréchal, J. Teixeira, S. Denis