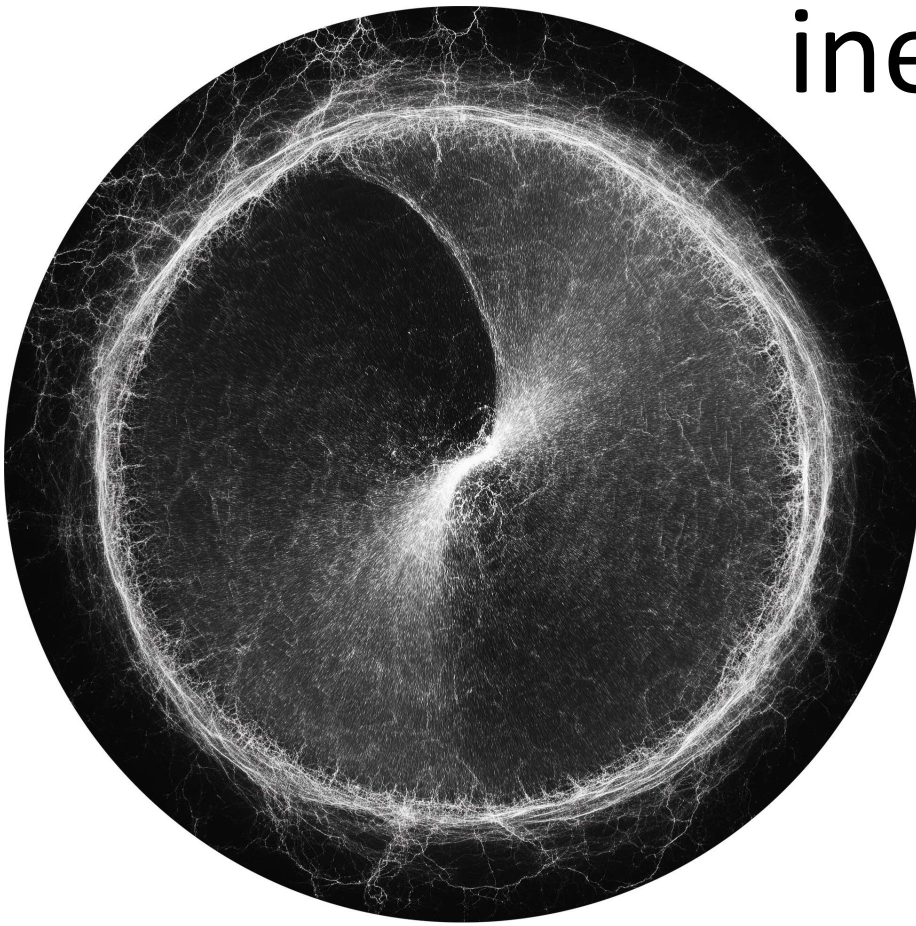
ESRF | The European Synchrotron

Uniaxial strain for diffuse and inelastic scattering: ID28 tandem at ESRF

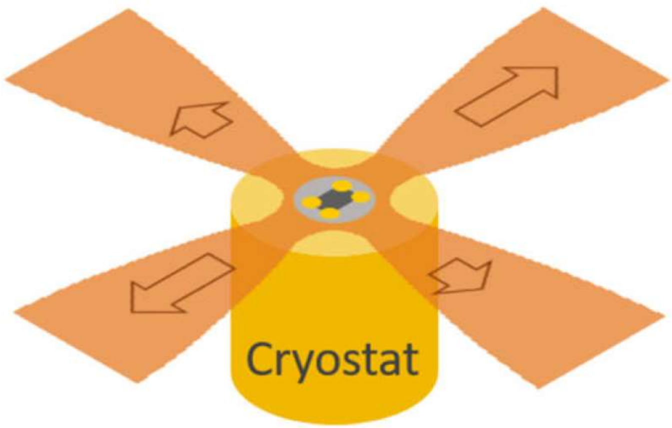


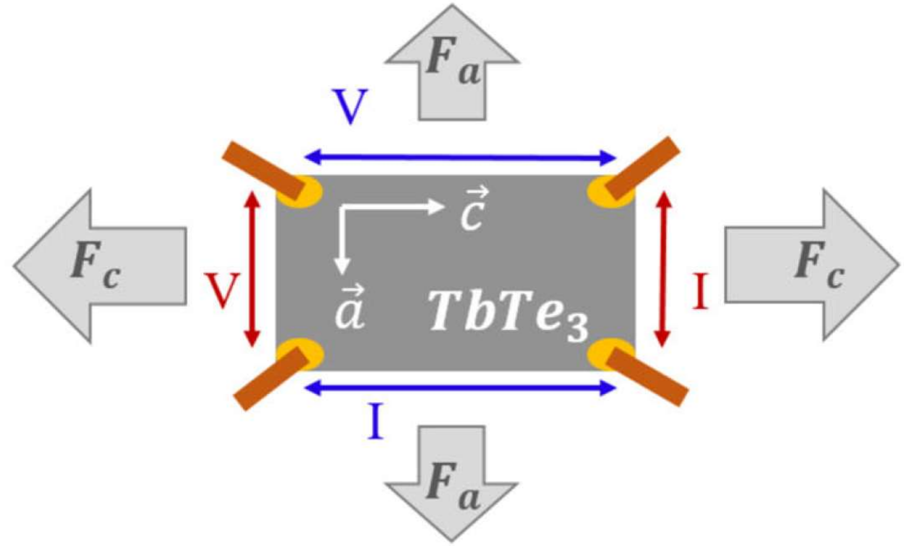
Alexeï Bosak

Outline

- ✓ Something on elasticity
- ✓ Inelastic and diffuse x-ray scattering
- ✓ Uniaxial strain hardware
- ✓ …with examples
- ✓ Plans

Biaxial strain / low temperature





sample glued to polyimide cross
arms are stretched by independent motors
cold finger behind the cross

[A. Gallo-Frantz et al., Nature Comm. 15, 3667 (2024)]
overcoming the symmetry lowering by strain

Something on elasticity

Elasticity

C_{ijkl} elastic tensor
S_{ijkl} compliance tensor

$C_{ijkl} = C_{jikl}$ $\quad C_{ijkl} = C_{klij}$
$S_{ijkl} = S_{jikl}$ $\quad S_{ijkl} = S_{klij}$

$11 \leftrightarrow 1$ $\quad 22 \leftrightarrow 2$ $\quad 33 \leftrightarrow 3$
$23 \leftrightarrow 4$ $\quad 13 \leftrightarrow 5$ $\quad 12 \leftrightarrow 6$

$C_{ijkl} \leftrightarrow c_{IJ}$ $\quad S_{ijkl} \leftrightarrow s_{IJ}$ $\quad \mathbf{s} = \mathbf{c}^{-1}$

but

$$\begin{pmatrix} s_{11} & s_{12} & s_{13} & s_{14} & s_{15} & s_{16} \\ s_{12} & s_{22} & s_{23} & s_{24} & s_{25} & s_{26} \\ s_{13} & s_{23} & s_{33} & s_{34} & s_{35} & s_{36} \\ s_{14} & s_{24} & s_{34} & s_{44} & s_{45} & s_{46} \\ s_{15} & s_{25} & s_{35} & s_{45} & s_{55} & s_{56} \\ s_{16} & s_{26} & s_{36} & s_{46} & s_{56} & s_{66} \end{pmatrix} = \begin{pmatrix} S_{1111} & S_{1122} & S_{1133} & 2S_{1123} & 2S_{1113} & 2S_{1112} \\ S_{1122} & S_{2222} & S_{2233} & 2S_{2223} & 2S_{2213} & 2S_{1222} \\ S_{1133} & S_{2233} & S_{3333} & 2S_{3332} & 2S_{1333} & 2S\,s_{3312} \\ 2S_{1123} & 2S_{2223} & 2S_{3332} & 4S_{2323} & 4S_{1323} & 4S_{2312} \\ 2S_{1113} & 2S_{2213} & 2S_{1333} & 4S_{1323} & 4S_{1313} & 4S_{1213} \\ 2S_{1112} & 2S_{1222} & 2S_{3312} & 4S_{2312} & 4S_{1213} & 4S_{1212} \end{pmatrix}$$

Stability condition and Poisson's ratio

Stability condition: c_{IJ} (s_{IJ}) positively defined (*i.e.* all eigenvalues are positive)

Poisson's ratio – transverse deformation to longitudinal deformation under the longitudinal strain

$$\nu_{\alpha\beta} = -\frac{S_{\alpha\alpha\beta\beta}}{S_{\alpha\alpha\alpha\alpha}} (\alpha \neq \beta)$$

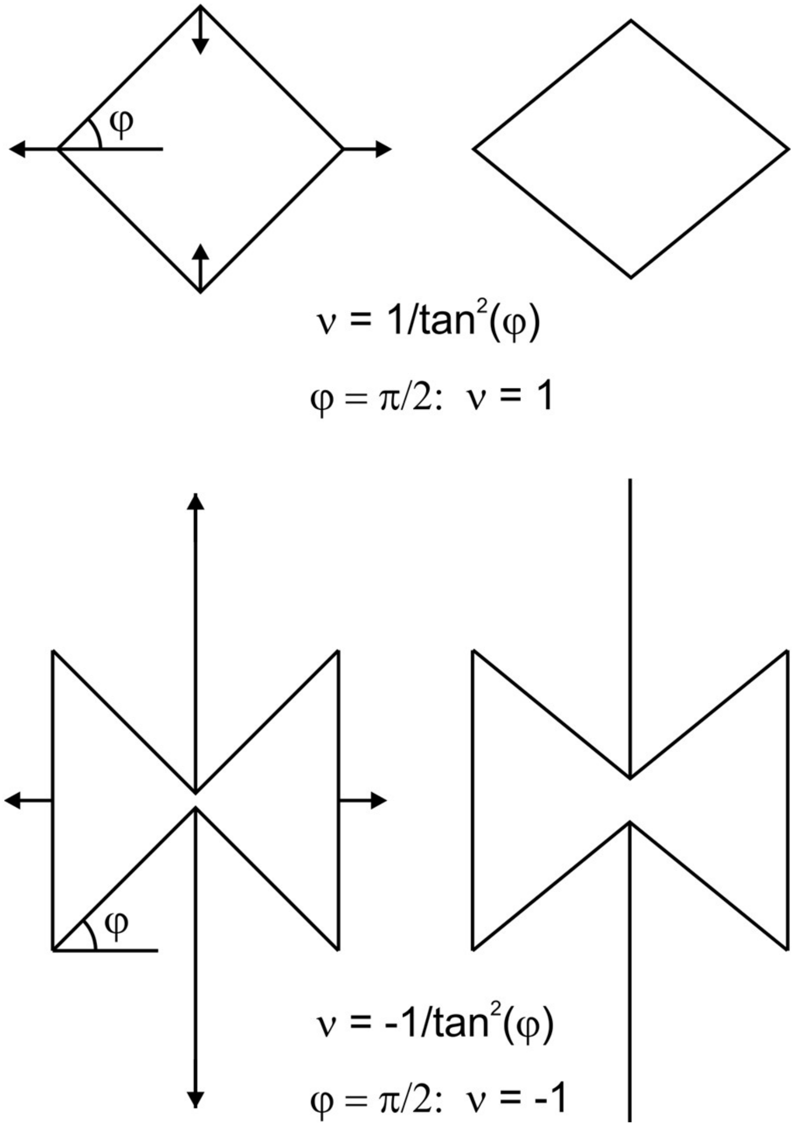
isotropy
$-1 < \nu < 1/2$

cubic symmetry
$-1 < \nu < 1/2$

lower symmetry
<u>no limits</u>

[L. Wheeler, C.Y. Guo, J. of Mechanics of Materials and Structures 2, 1471 (2007)]

Handwaving in 2D



2D assembly of rigid rods:
any values of ν

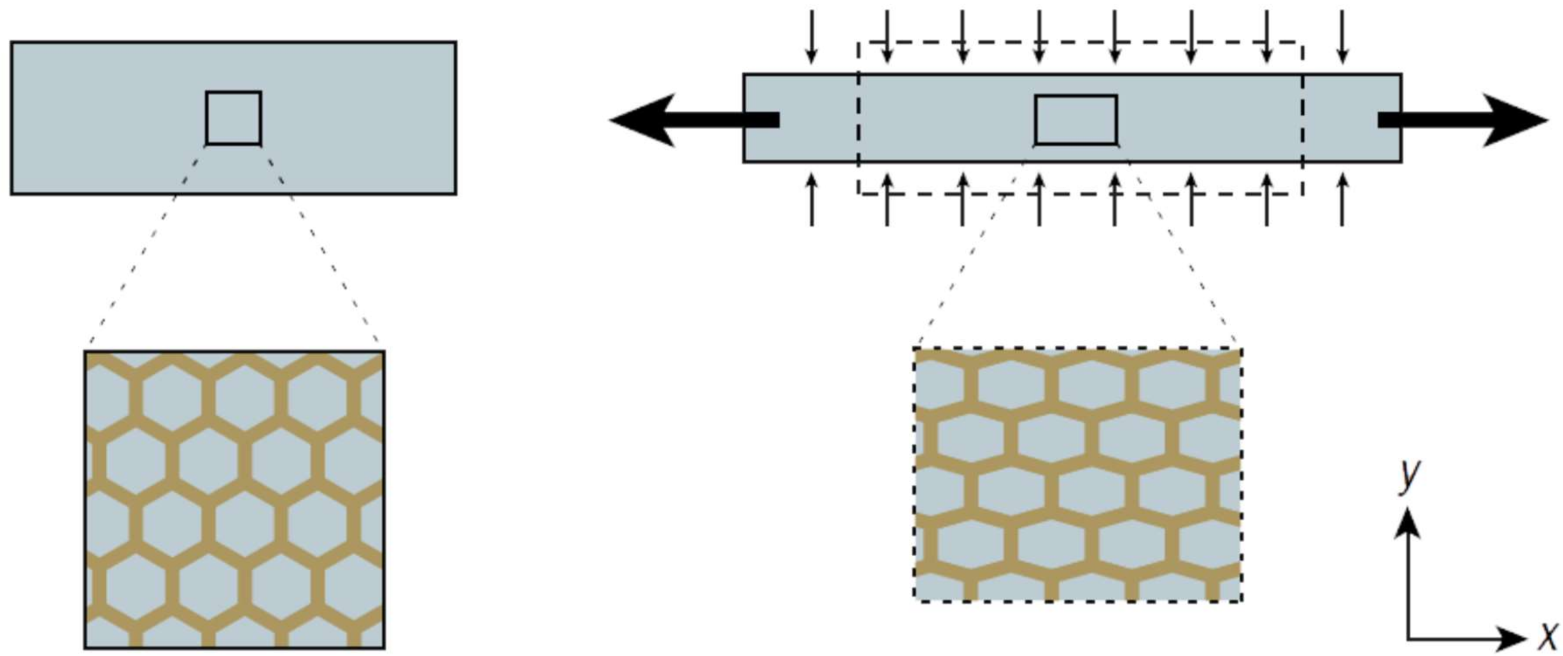
no forbidden values for
layered structures

layered structures
have symmetry lower than cubic

Normal materials

Non-auxetic material

As the material is stretched the component cells get longer in the x-direction but become compressed in the y-direction

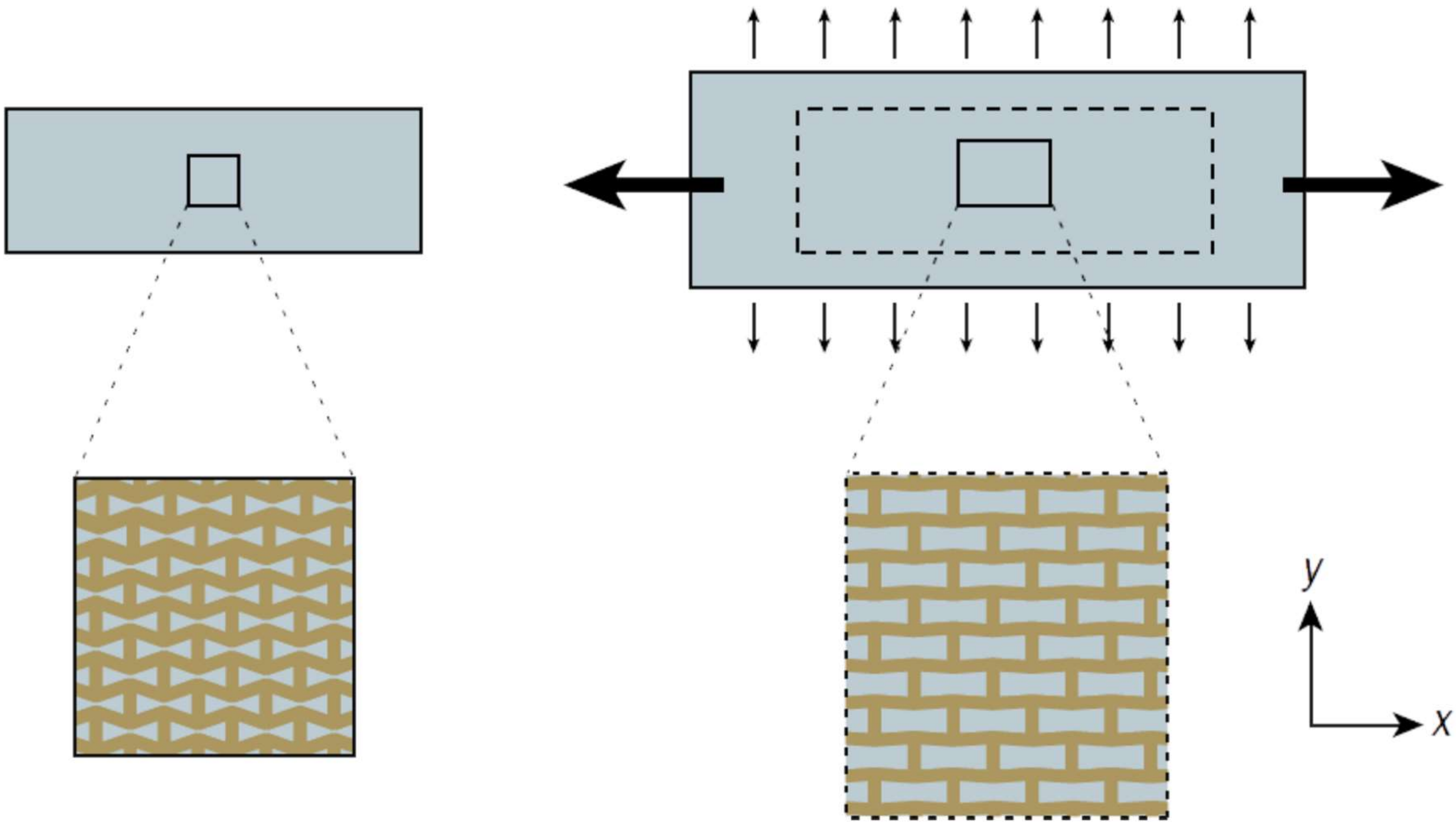


[A. Alderson, Chemistry & Industry 384-391, 1999]

Auxetics

Auxetic material

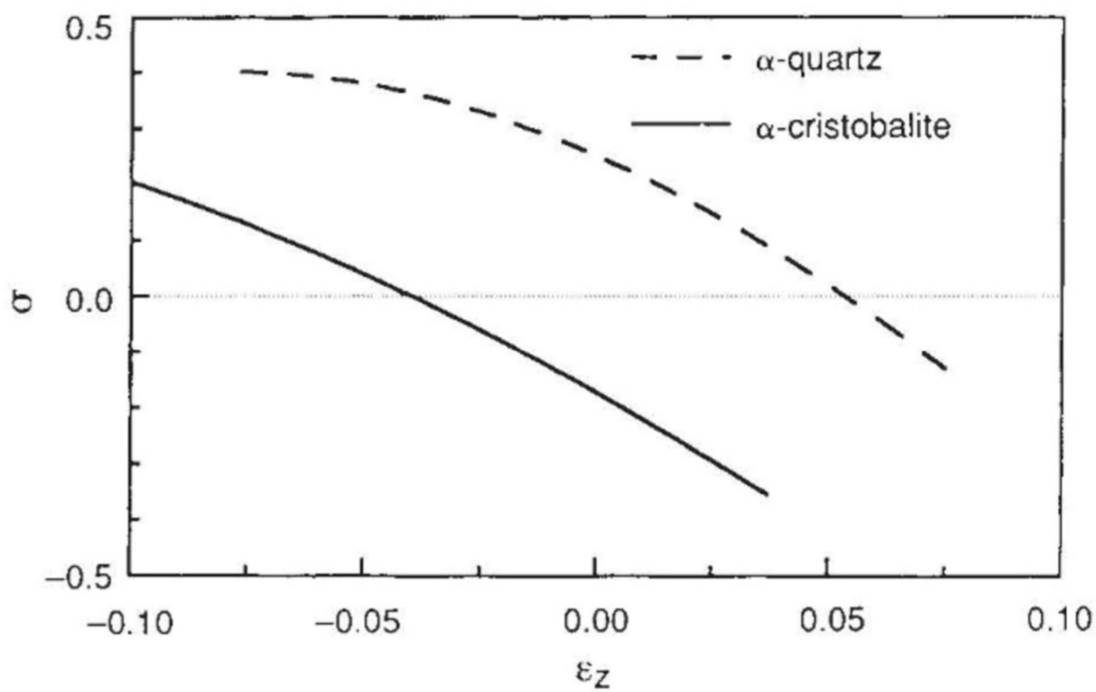
As the material is stretched, the cells get larger in both the x- and y-directions



[A. Alderson, Chemistry & Industry 384-391, 1999]

Auxetism in "usual" materials

nearly 70% of the cubic elemental metals have a negative Poisson's ratio when compressed along the <110> direction (contraction along <1-10>)



quartz should become auxetic when stretched by 5%
[N.R. Keskar, Nature 358, 222 (1992)]

Easy search of auxetism

$$S_{ijkl} = a_{mi}\ a_{nj}\ a_{pk}\ a_{ql} S^0{}_{mnpq}$$

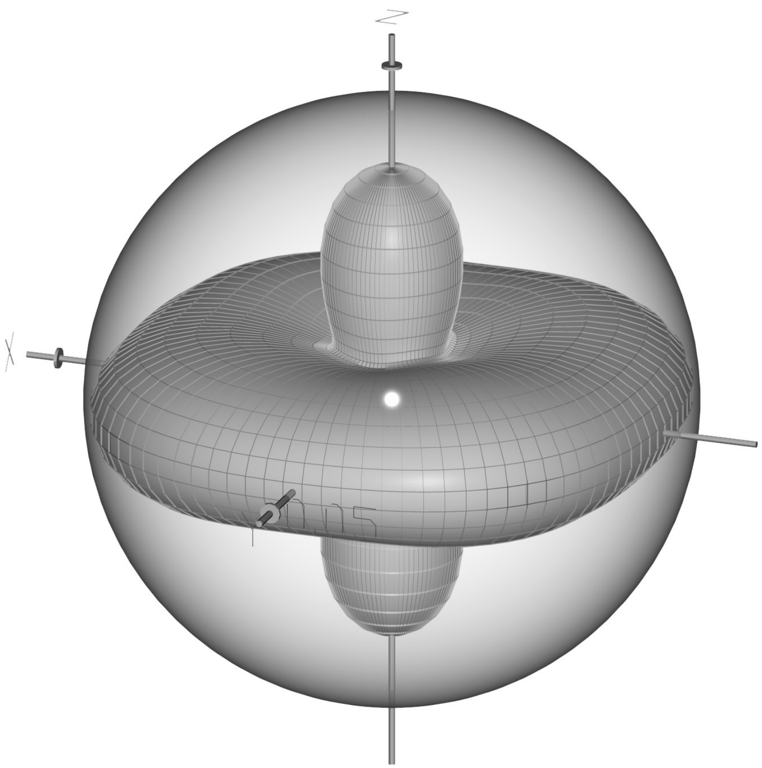
$$\nu_{\alpha\beta} = -\frac{S_{\alpha\alpha\beta\beta}}{S_{\alpha\alpha\alpha\alpha}} (\alpha \neq \beta)$$

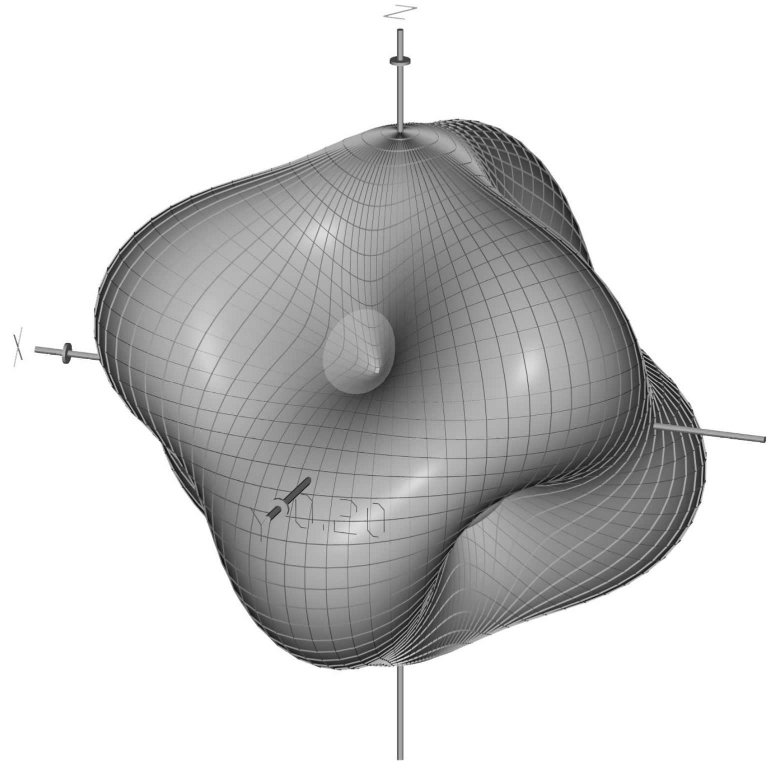
$$\tilde{\nu}_{1,2} = -\left(S_{\alpha\alpha\beta\beta} + S_{\alpha\alpha\gamma\gamma} \pm \sqrt{4 S_{\alpha\alpha\beta\gamma}{}^2 + (S_{\alpha\alpha\beta\beta} - S_{\alpha\alpha\gamma\gamma})^2} \right) \Big/ 2 S_{\alpha\alpha\alpha\alpha}$$

$$\bar{\nu} = -(S_{\alpha\alpha\beta\beta} + S_{\alpha\alpha\gamma\gamma}) / 2 S_{\alpha\alpha\alpha\alpha}$$

1. Normal materials: $\nu_{\alpha\beta} > 0$
2. The axial deformation is accompanied by transverse deformations of different signs in main directions ($\nu_1 \nu_2 < 0$)
3. The axial deformation is accompanied by the transverse deformations of the same negative sign

SiO_2

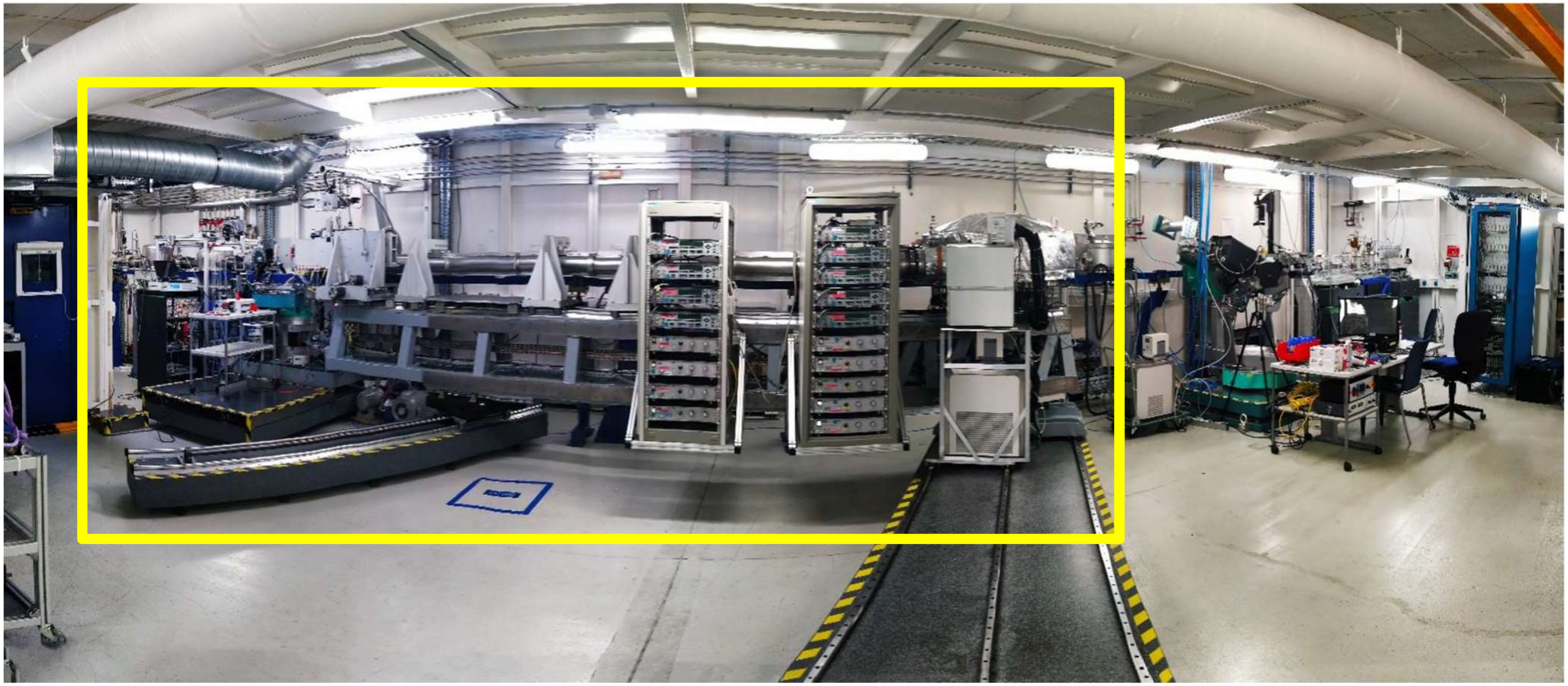




α-cristobalite α-quartz

quartz is *already* auxetic if deformed in right direction

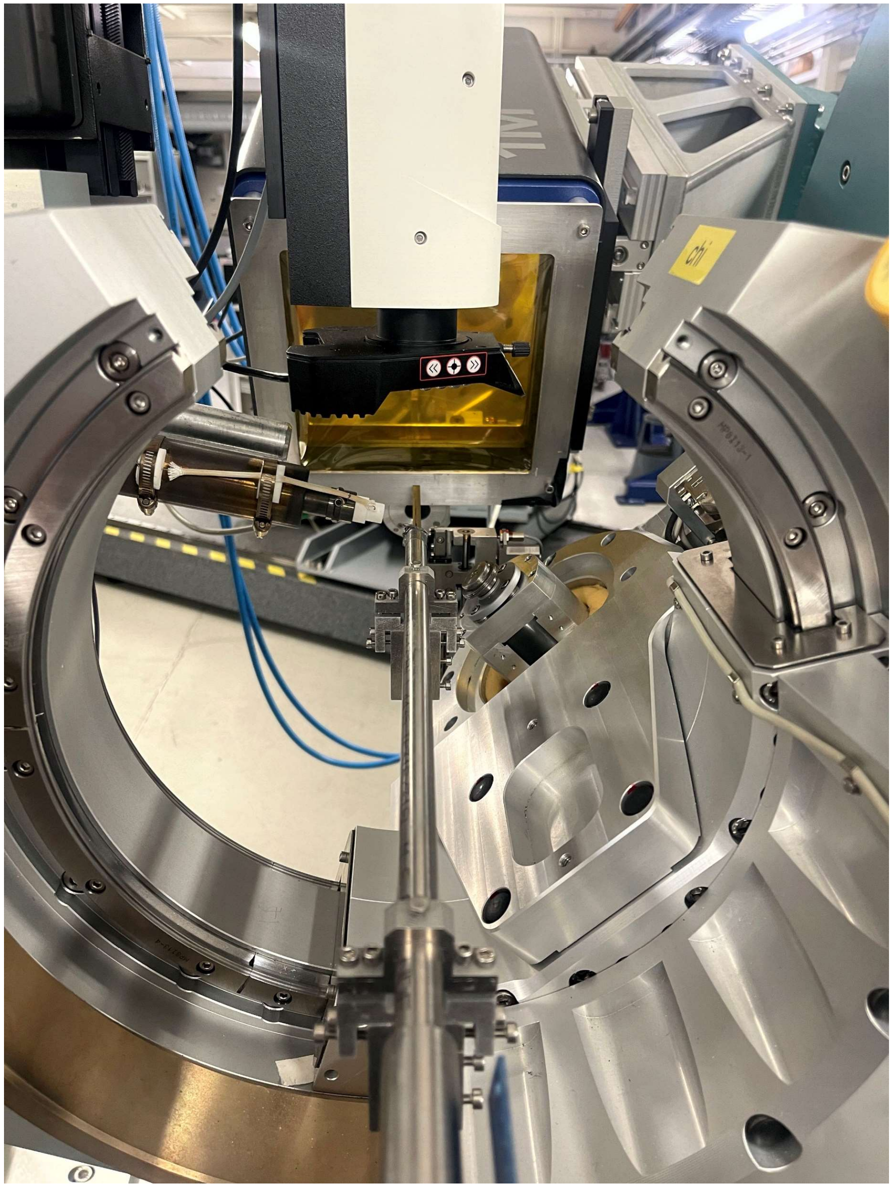
ID28: phonon spectroscopy



3.0 meV resolution at 17.8 keV
1.5 meV resolution at 23.7 keV

phonons and phonon-like excitations (slow)

ID28: diffuse scattering



fast mapping of reciprocal space
12-24 keV
10-1200 K
crystals + films

NB: here fast is ~10^{18} fs

¡Two techniques are complementary!

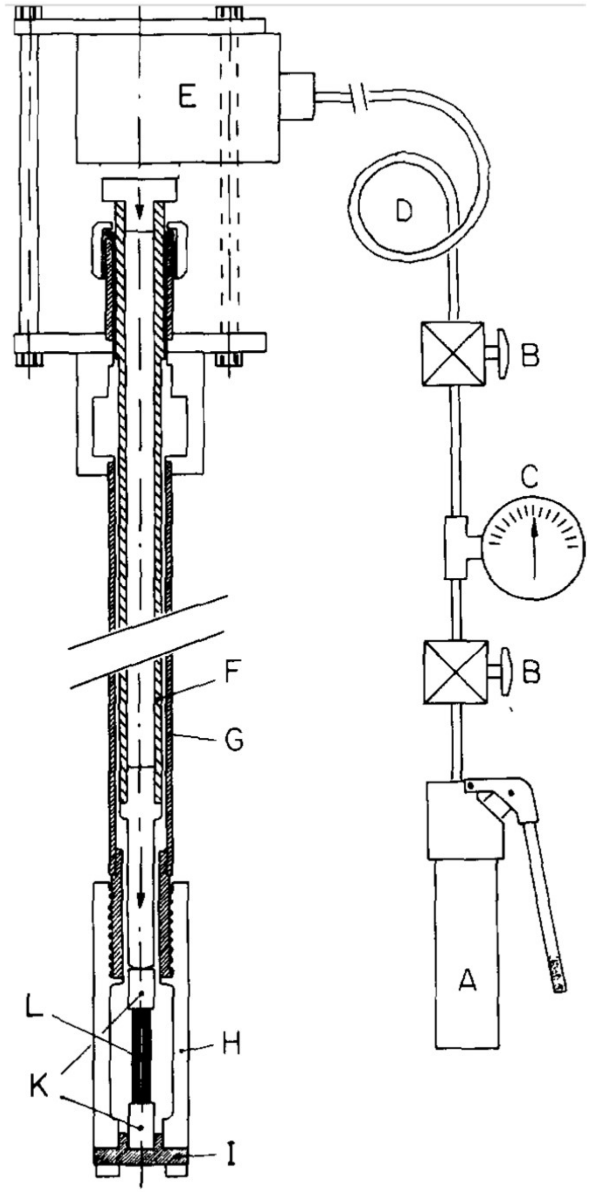
Strain for x-rays

- absorption length down to few μm
- focal spot down to few μm and below

✓ perfect fit for diamond anvil cells
✓ delicate fit for uniaxial strain

$p < 50$ GPa – we can help
$p > 50$ GPa – bring your own

Uniaxial strain for neutrons



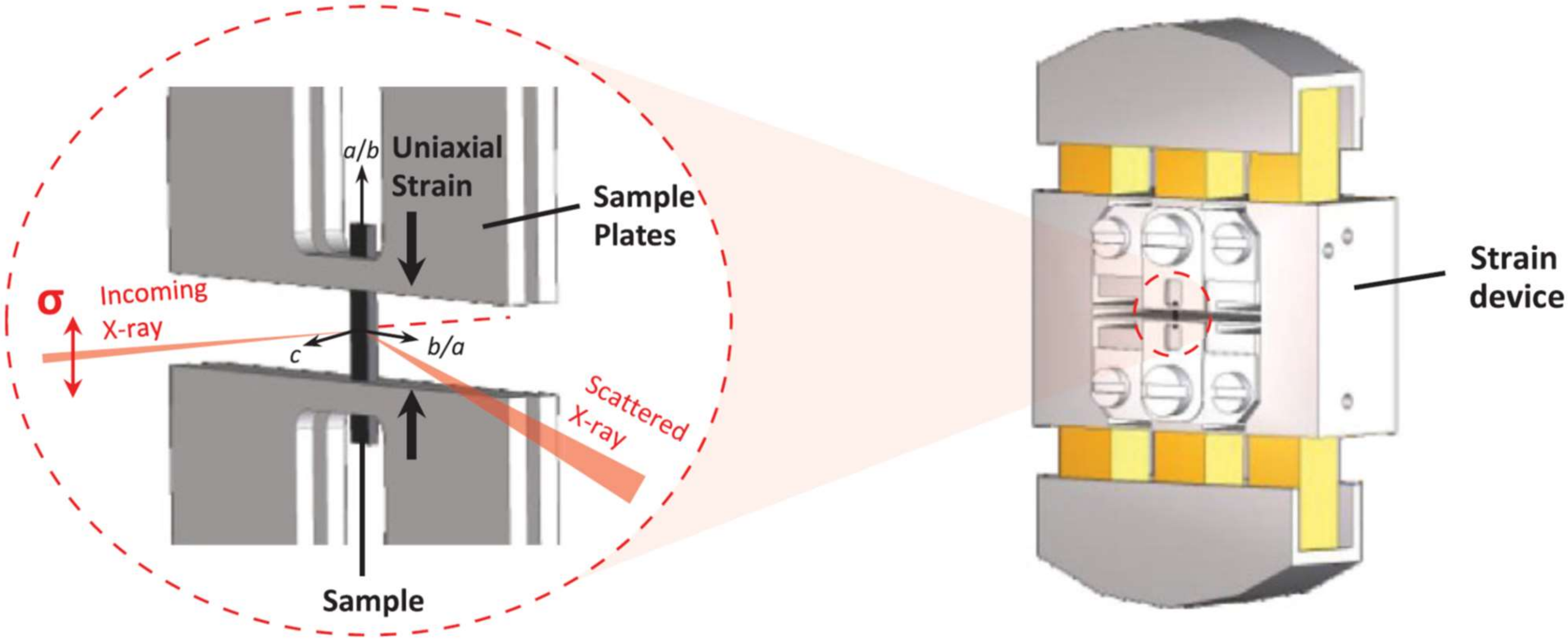
[B. Halg et al., Nuclear Instruments and Methods in Physics Research A253, 61-64 (1986)]

uniaxial pressure device for neutron scattering experiments

CeSb 3x3x3 mm^3

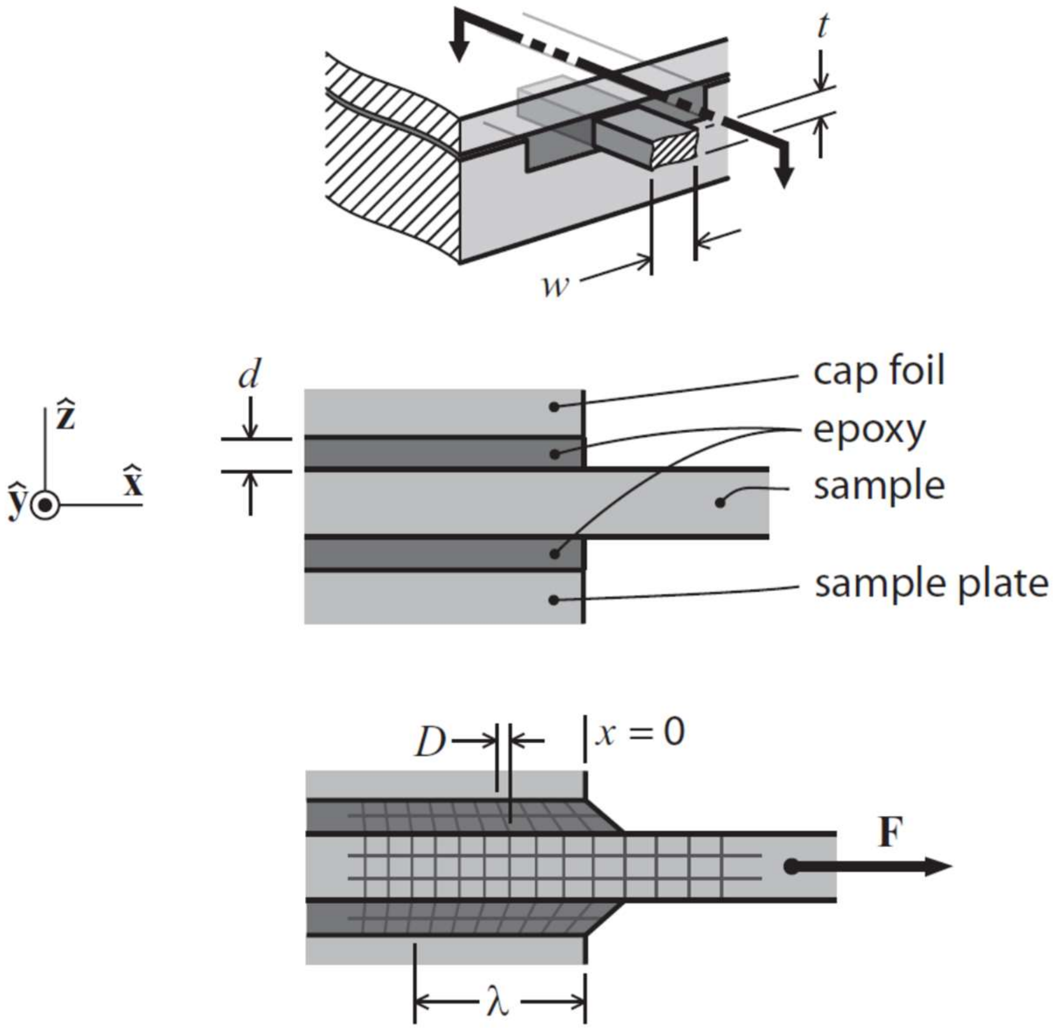
we cannot just borrow

Razorbill solution



piezo actuators + robust structure
force and displacement measured
special edition – OK for transmission
delicate points – sample preparation and gluing

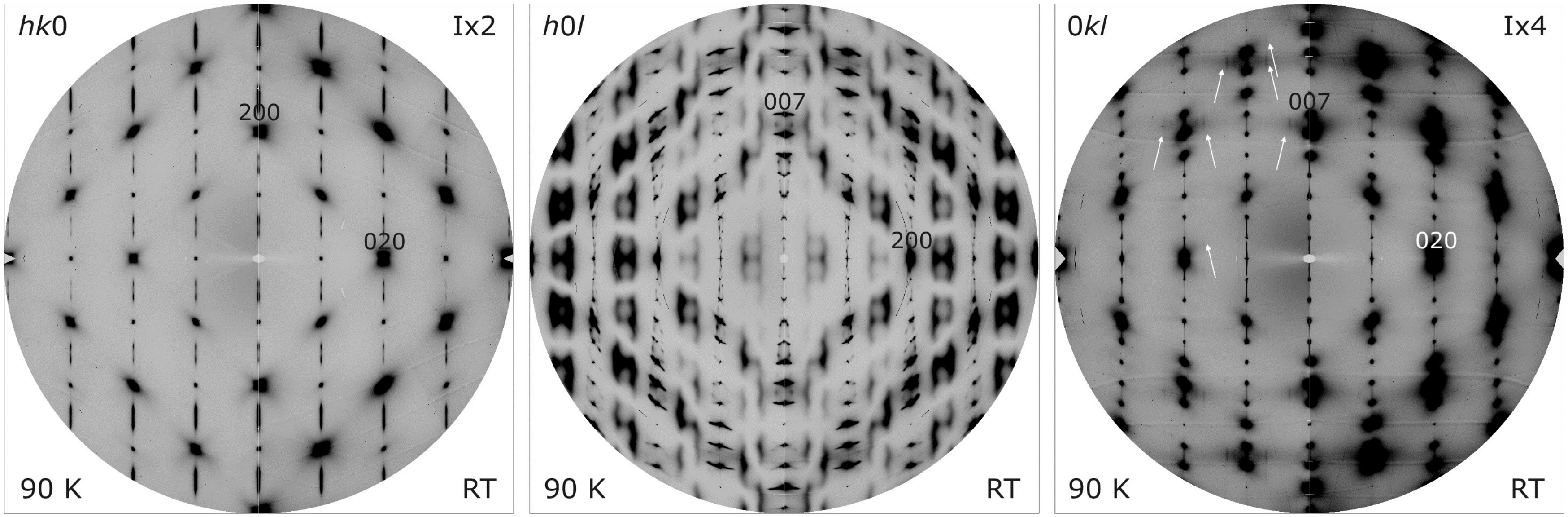
Trickiest part



[C.W. Hicks et al., Rev. Sci. Instr. 85, 065003 (2014)]

likely to remain a pain for many more years

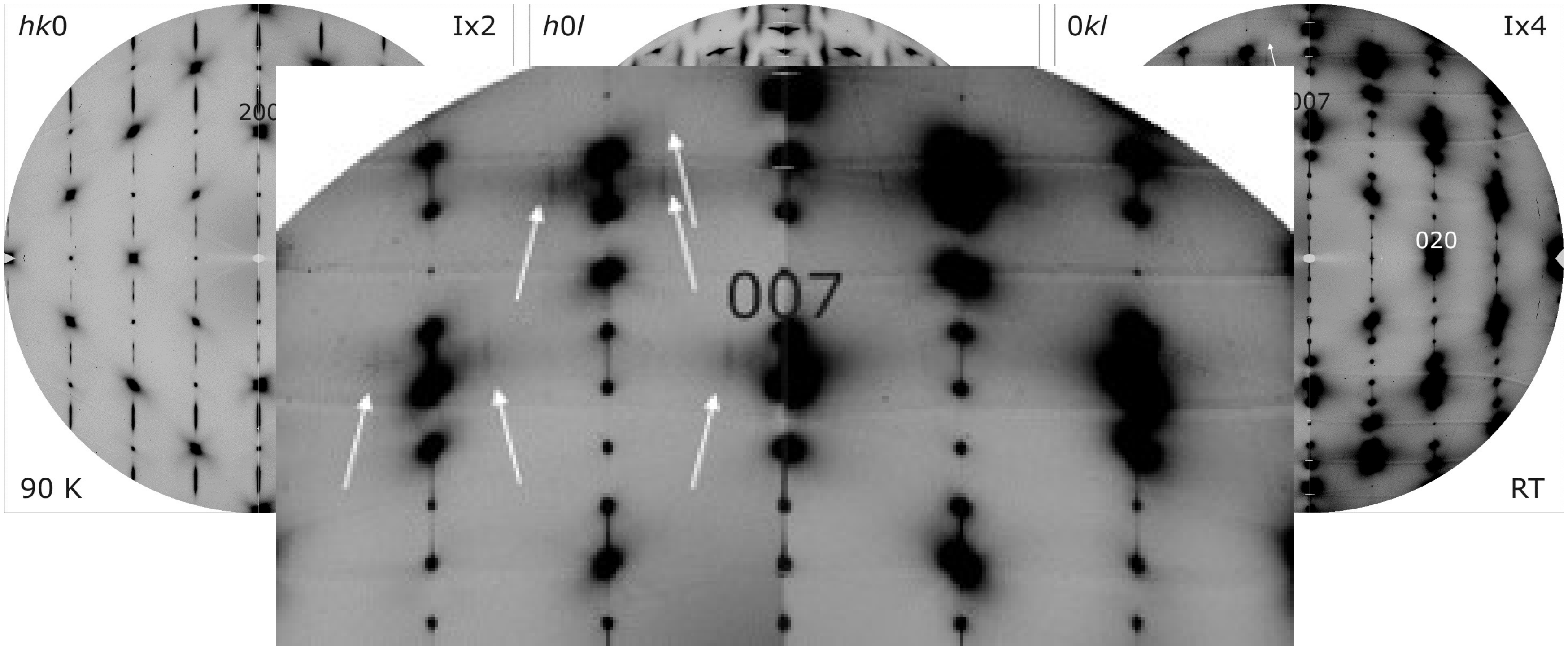
YBCO: diffuse maps



$YBa_2Cu_3O_{6.6}$: orthorhombic phase, less boring than tetragonal $YBa_2Cu_3O_7$

[M. Le Tacon et al., Nature Phys. 10, 52-58 (2014)]

YBCO: diffuse maps



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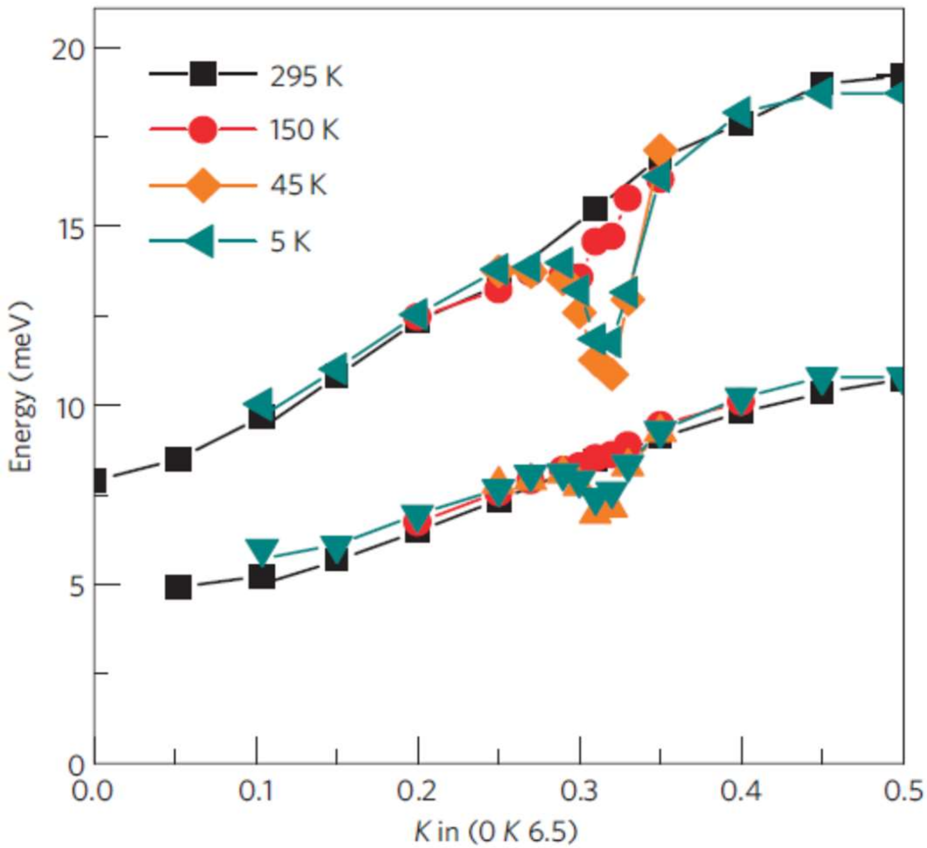
[M. Le Tacon et al., Nature Phys. 10, 52-58 (2014)]

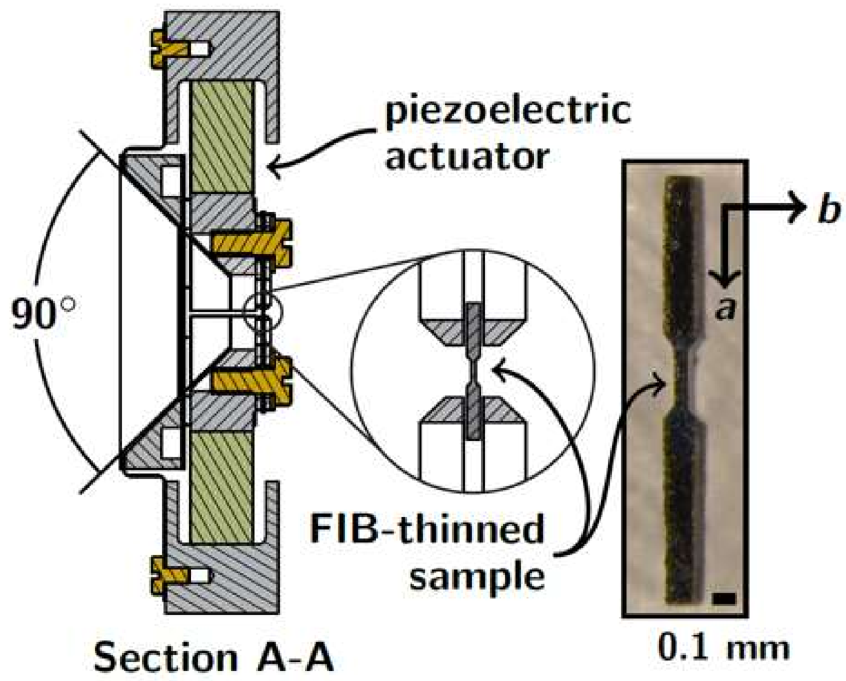
YBCO: inelastic scattering in narrow region of interest



- softening
- broadening
- central peak

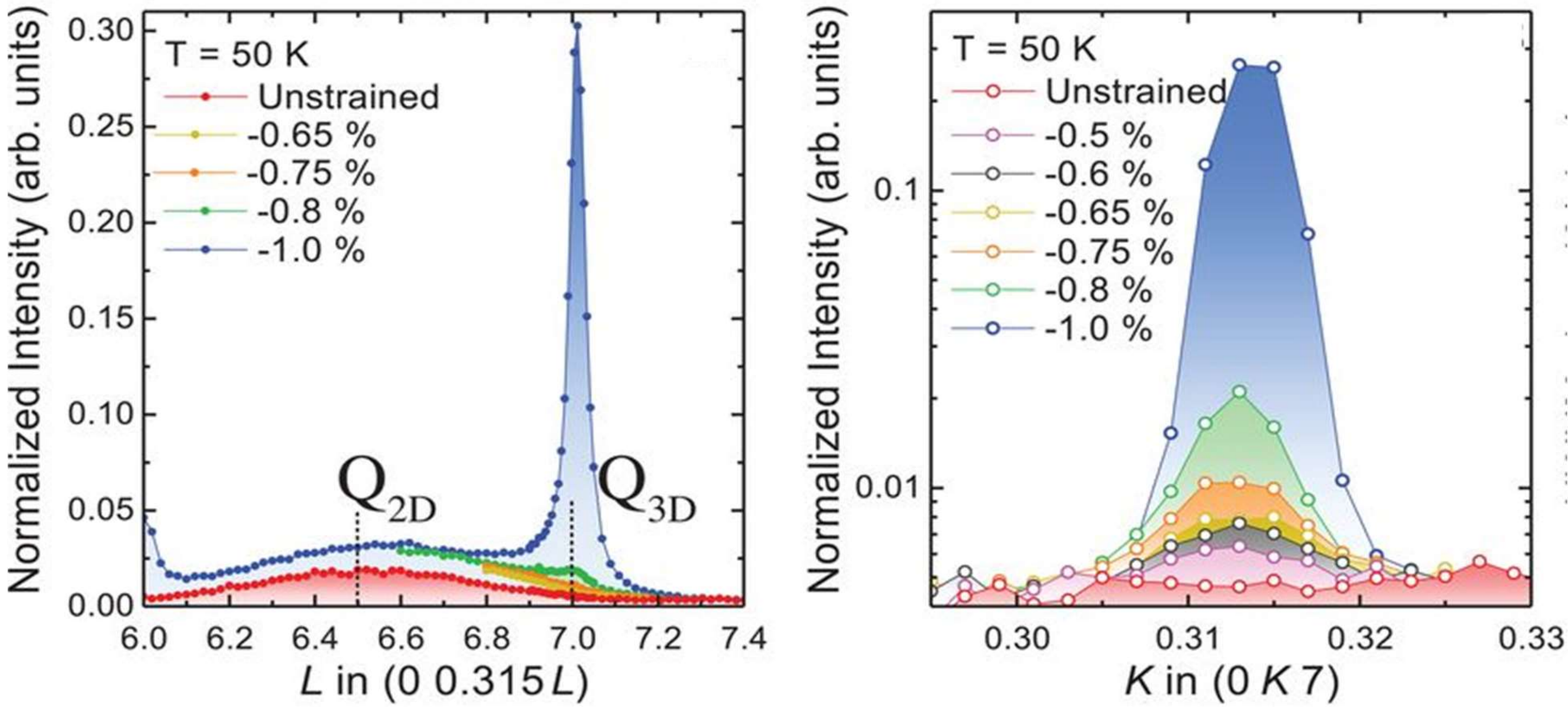
strong and sharp anomaly ☺
competitor of superconductivity ☹

Uniaxial strain + cooling



liquid helium cryostat (as for DAC)

Uniaxial strain + cooling



quasielastic intensity (energy window 3 meV FWHM)

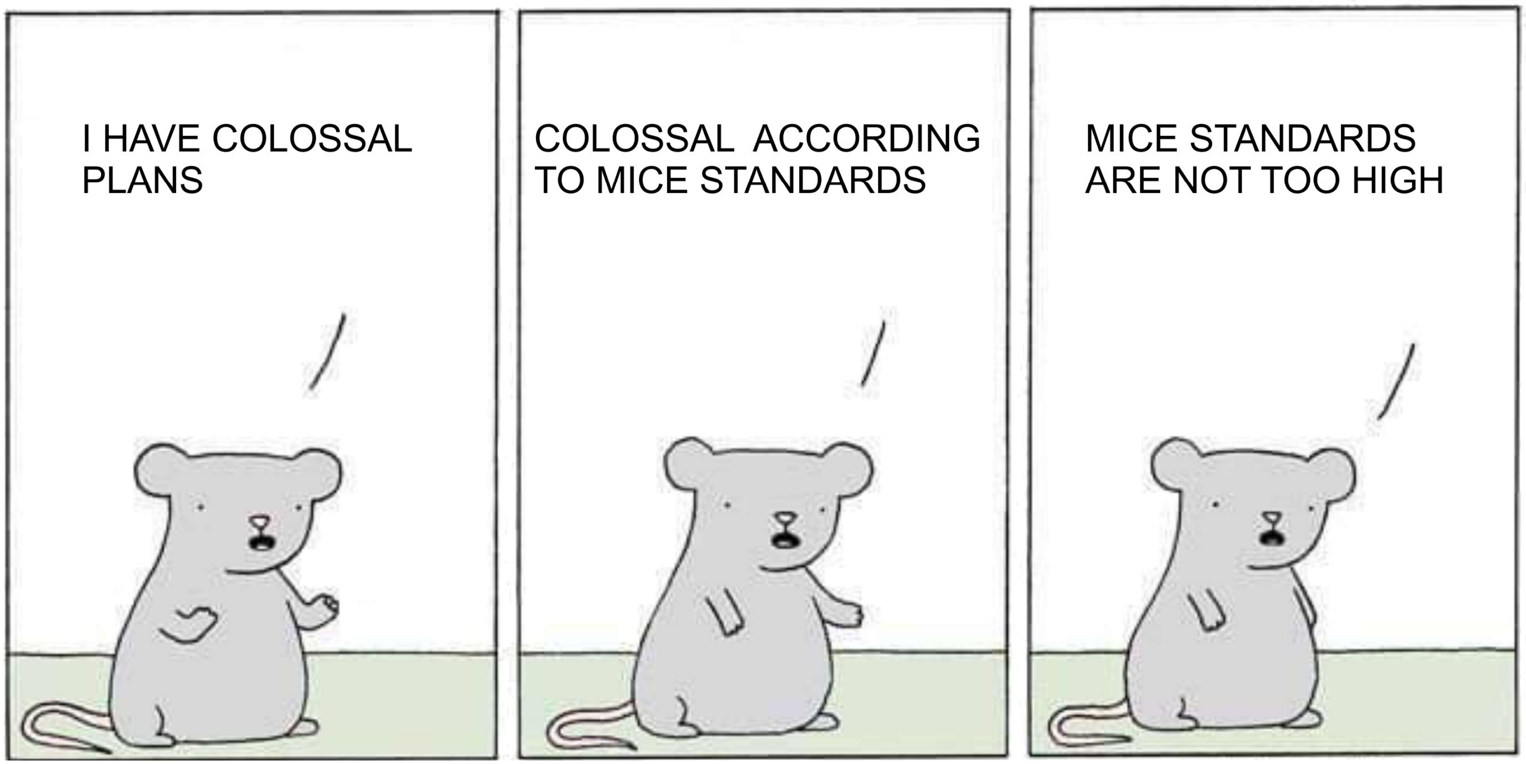
3D long-range-ordered CDW state induced by *a* axis compression

[H.H. Kim, S.-M. Souliou et al., Science 362, 1040–1044 (2018)]

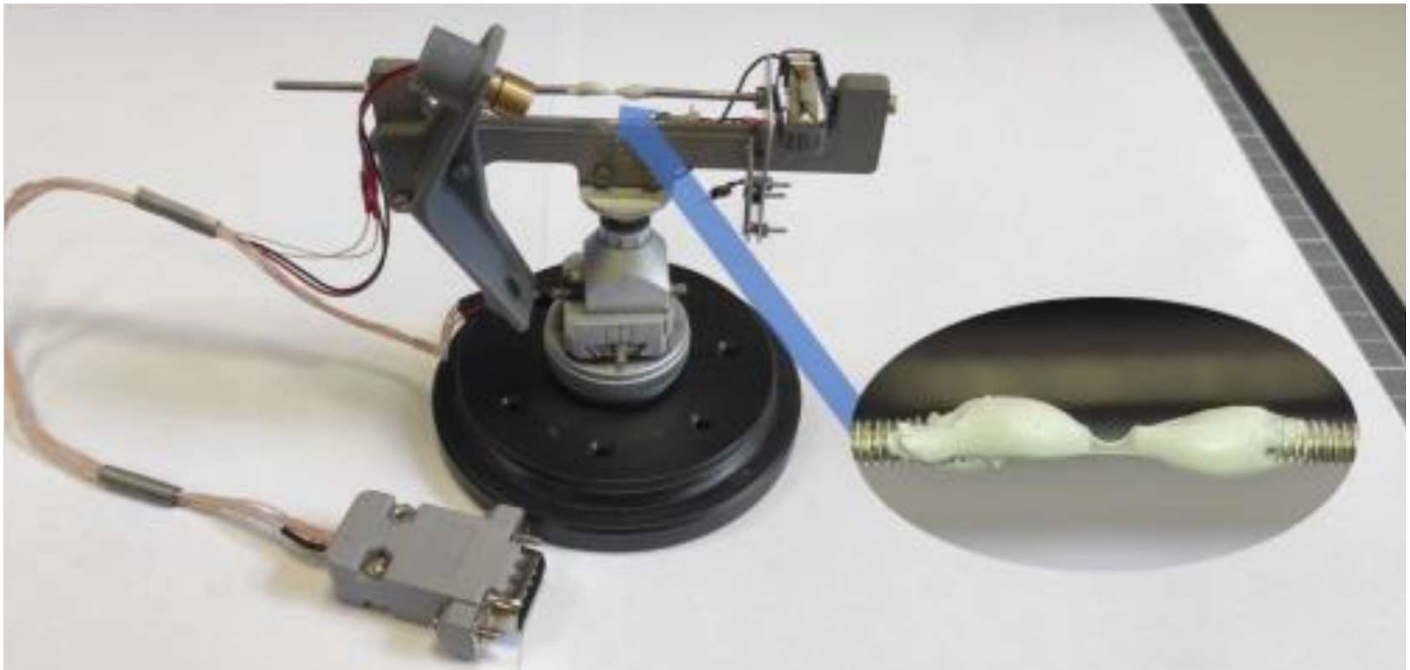
Failed attempt

Closed-cycle cryostat – not enough cooling power

Plans and dreams



High temperature setup



[S. Udovenko et al., proceedings of 2021 International Conference on Electrical Engineering and Photonics]

- bulky | limited rotation range
- no cooling possible | ice formation
- only stretching recommended

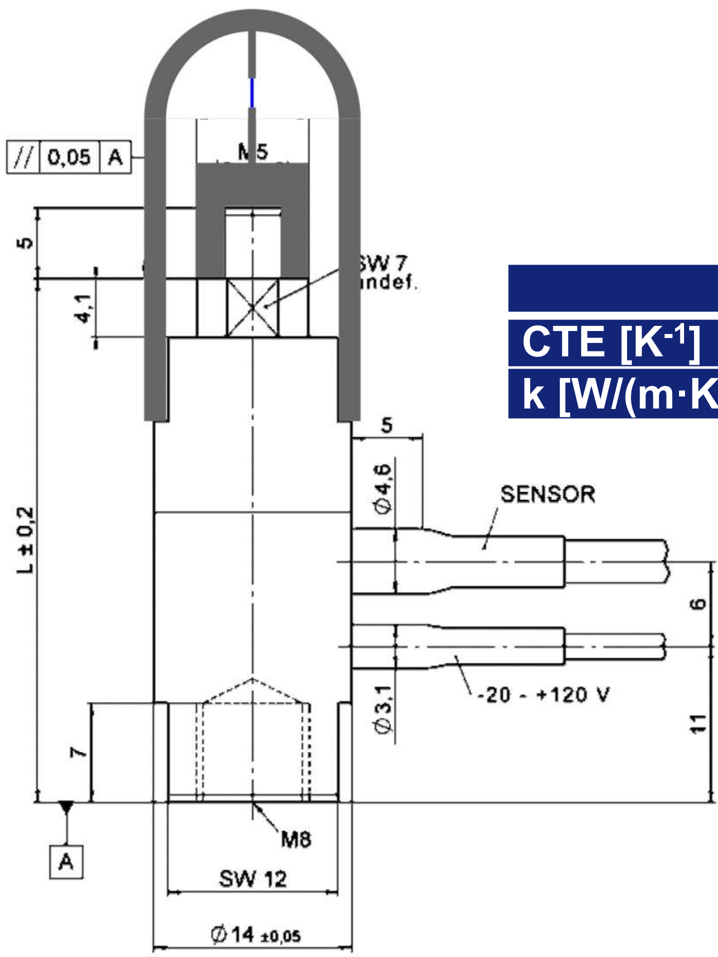
“I'll go build my own lunar lander”

Main destination - detwinning

Desired compatibility with gas blowers

- piezo element away from heating/cooling
- low thermal conductivity
- low thermal expansion
- easy sample mounting

"Detwinning" device

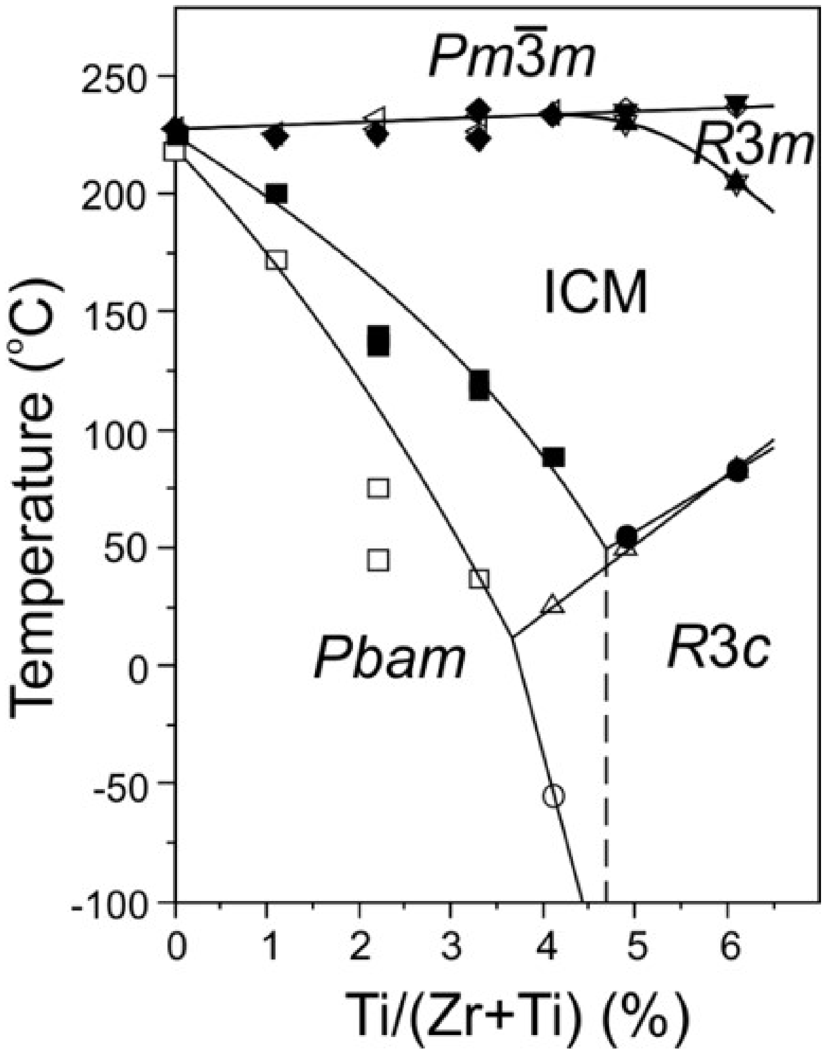


quartz glass or invar frames

	quartz glass	invar	copper
CTE [K^{-1}]	$0.5 \cdot 10^{-6}$	$1.2 \cdot 10^{-6}$	$16 \cdot 10^{-6}$
k [W/(m·K)]	1.4	12	398

…in preparation…

PZT with low Ti content



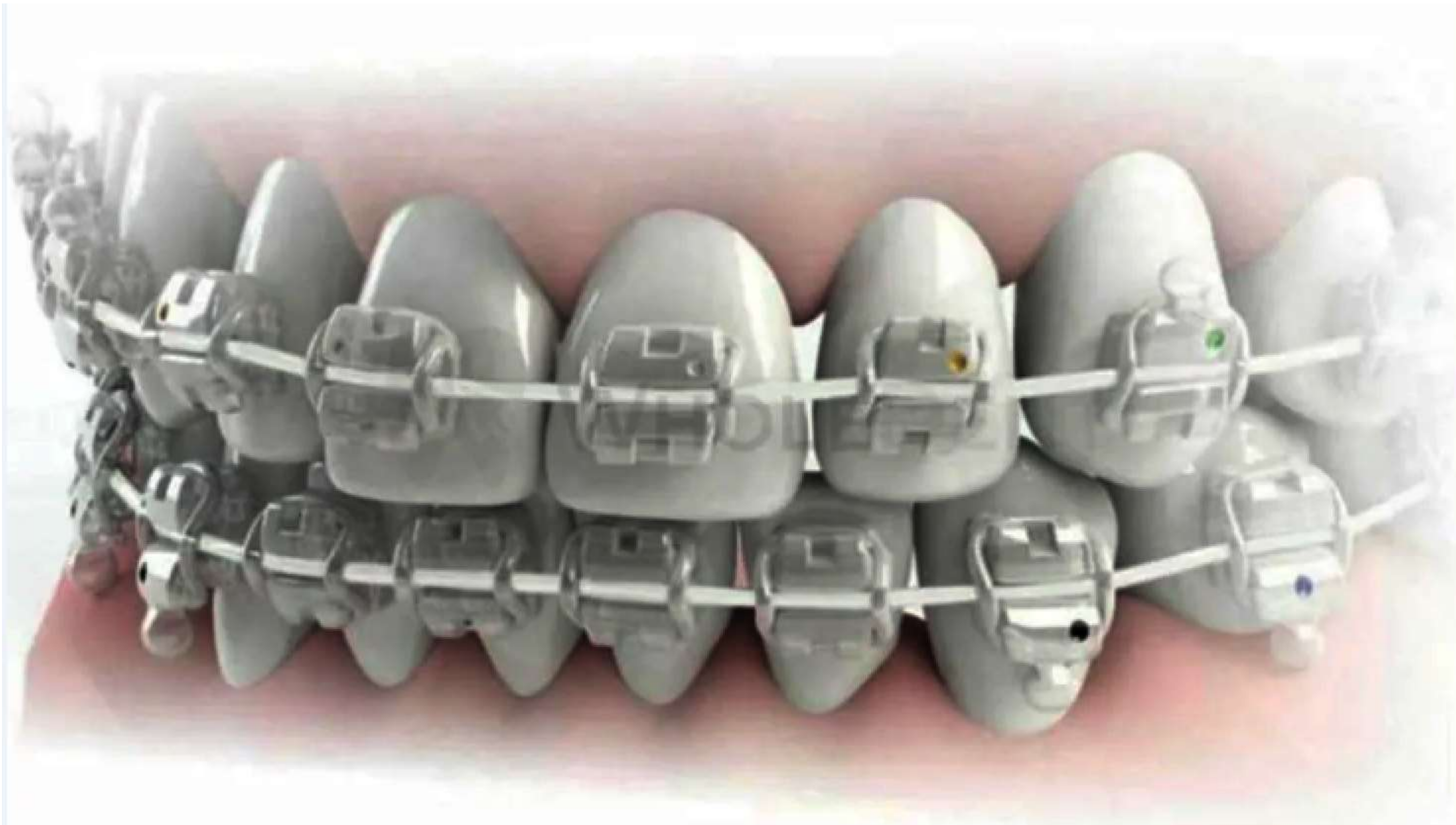
ICM – incommensurate monoclinic phase

…up to 12 centrosymmetric twins

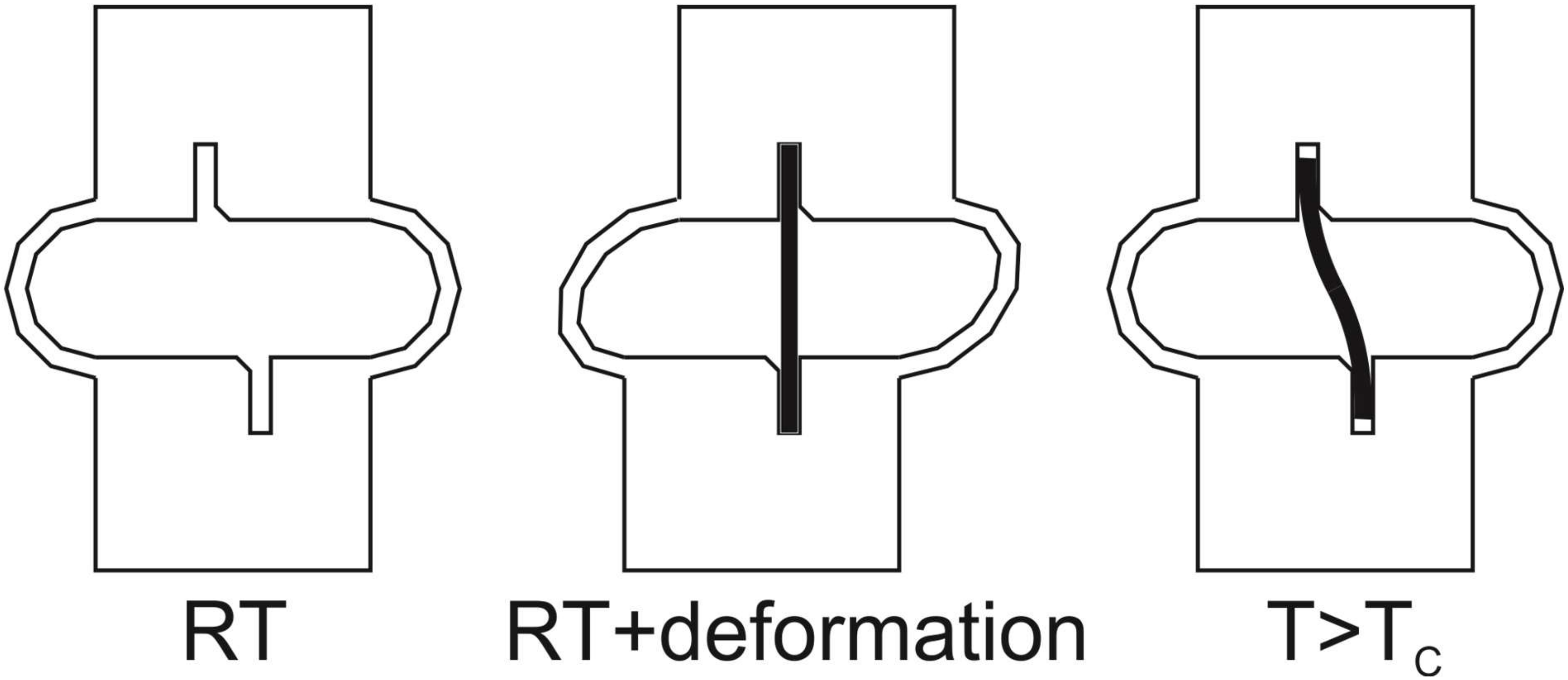
detwinning by cooling under strain?

HT strain device needed

Superelastic actuator

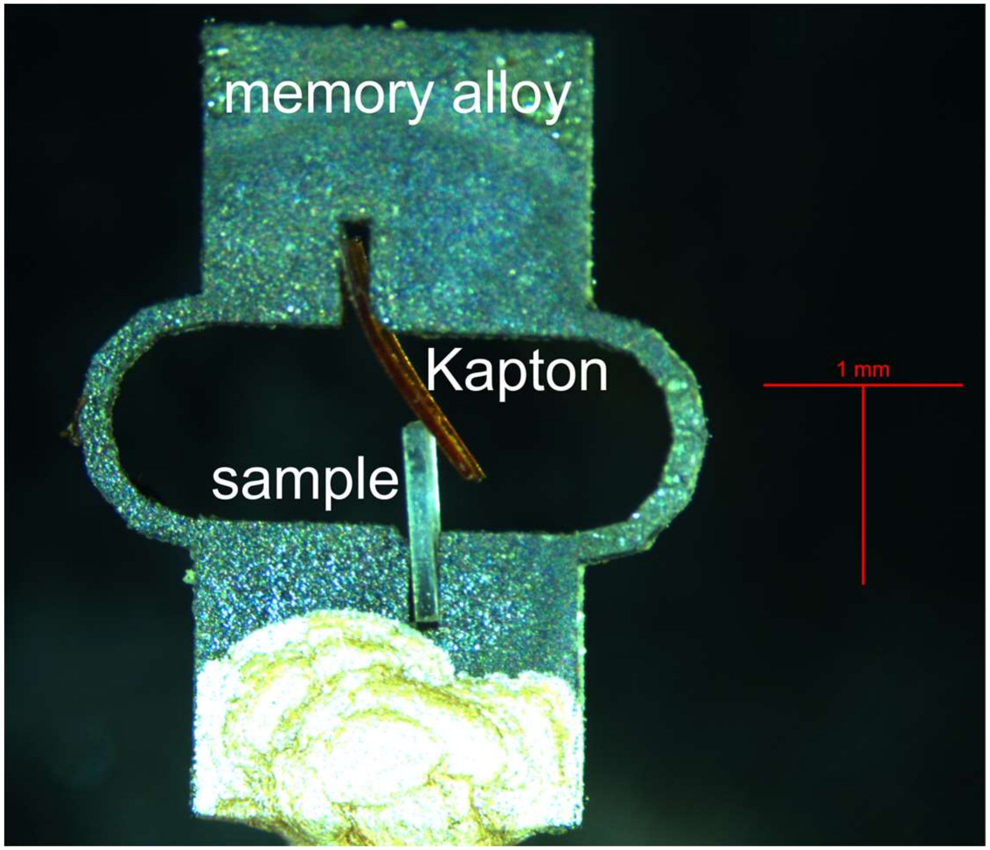


Lazy person strain device



if T_C of memory alloy is below the transition of interest, its superelastic state may kill some domains (or break the sample)

PZT detwinning

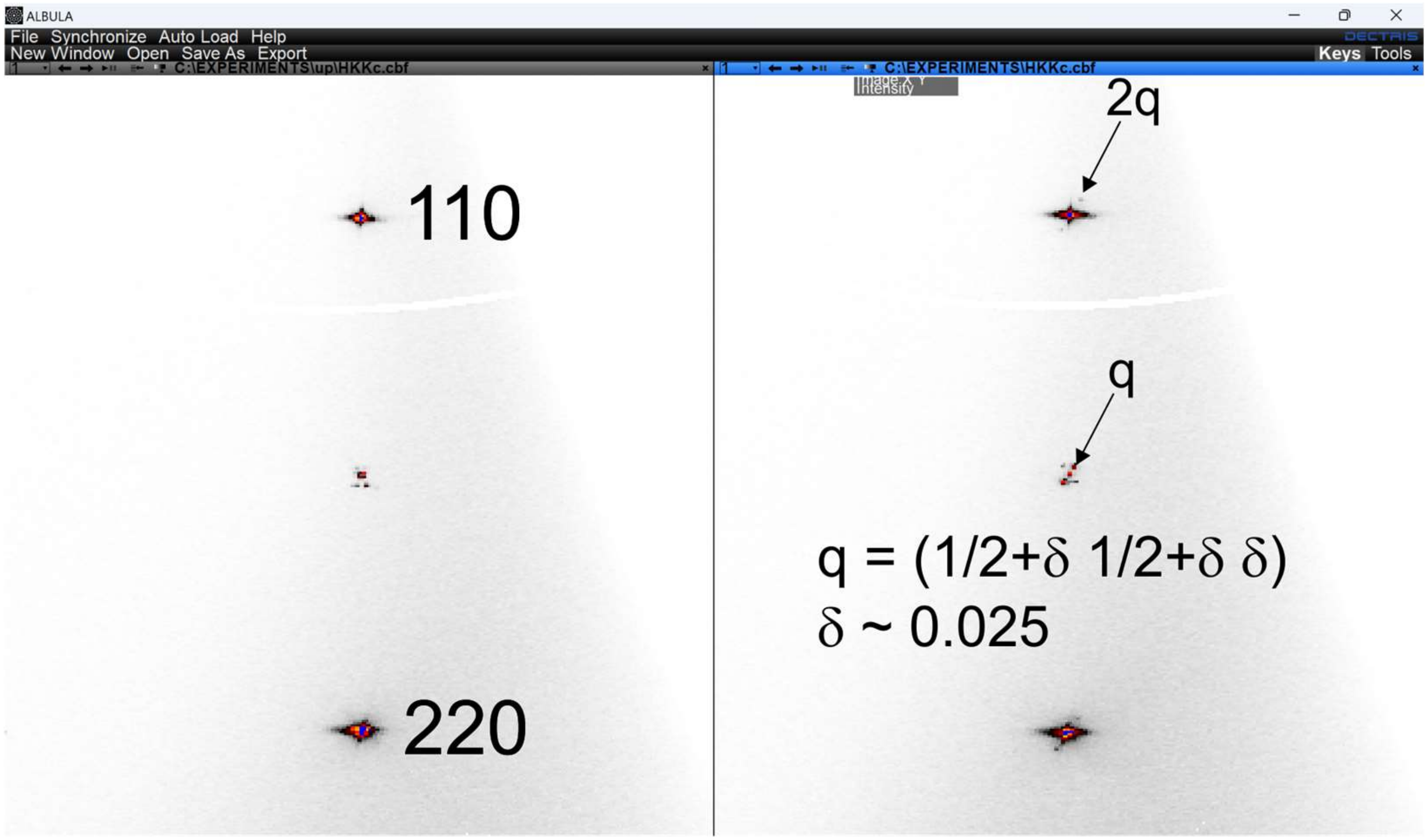


nitinol / 0.125 mm thick
laser cutting
T_C ~ 40°C

Kapton spring added
to reduce the force
(Kapton is OK to 400°C)

sample heated to 350°C
and then cooled

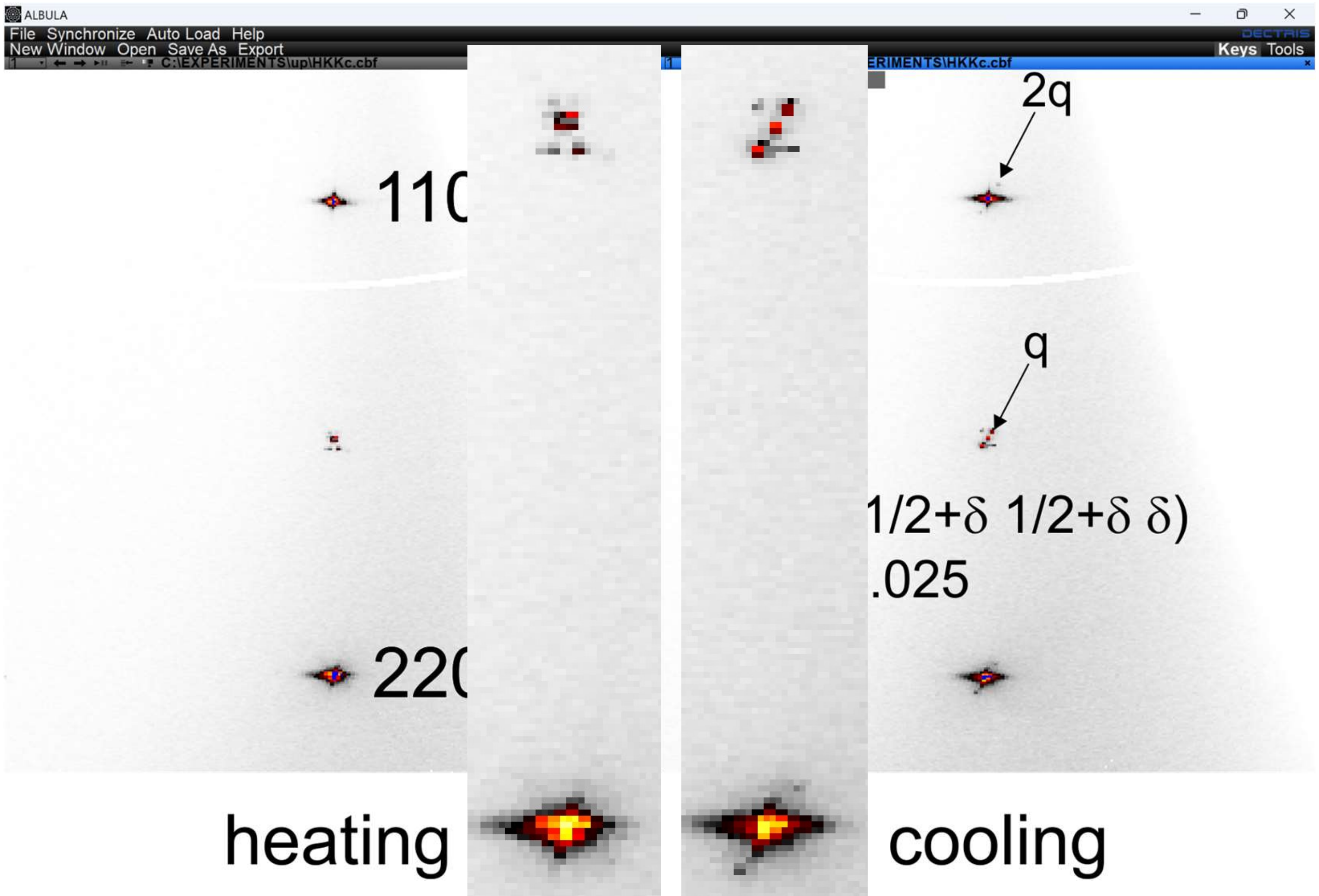
PZT detwinning



heating cooling

brut force attempt removed the most of twins
- to be optimized and cross-checked

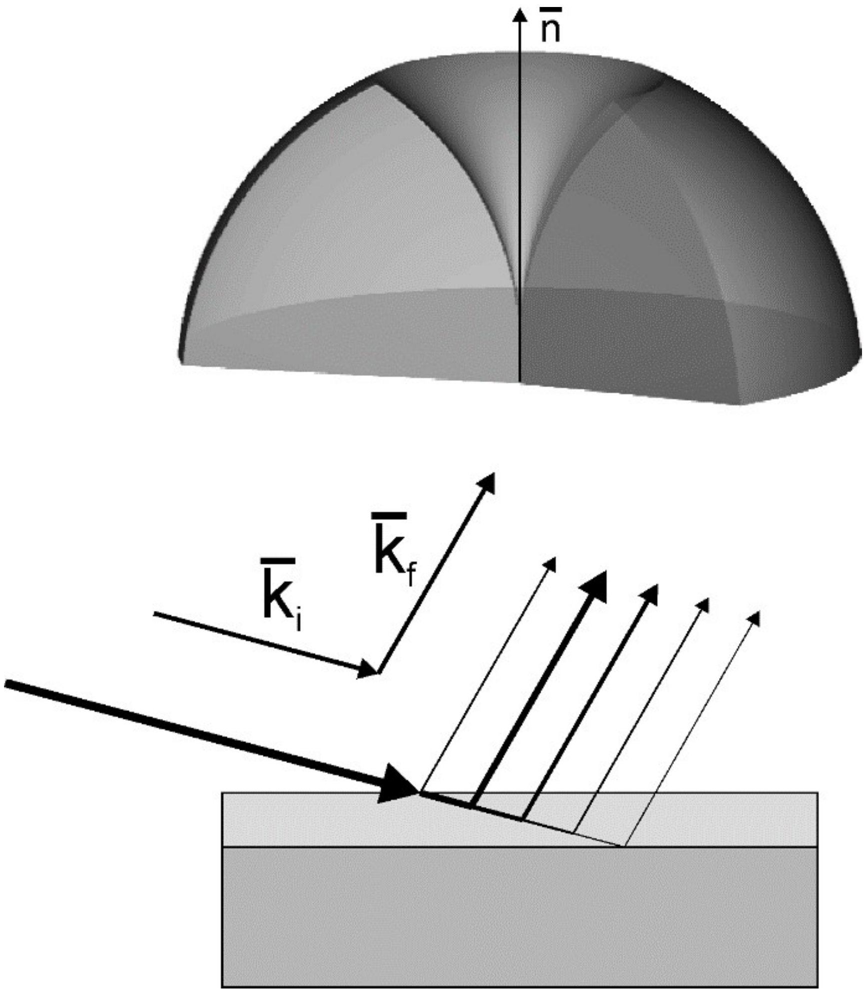
PZT detwinning



brut force attempt removed the most of twins
- to be optimized and cross-checked

Advertisement

Scattering from thin films

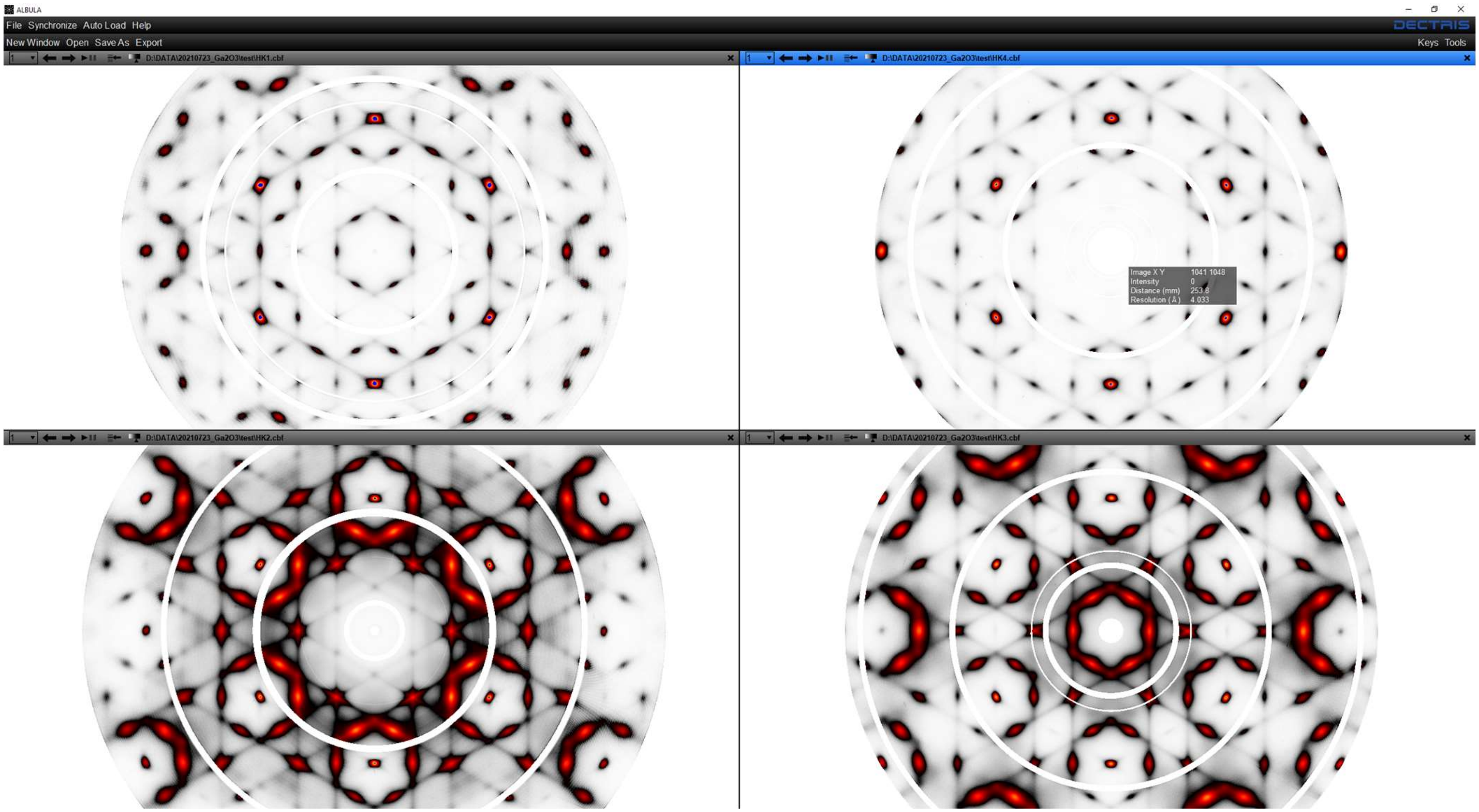


our favorite mode:
360° rotation with fixed incidence angle (0.1-0.5°)

angle optimized on film/substrate contrast

- nearly complete hemisphere in reciprocal space
- easy absorption correction

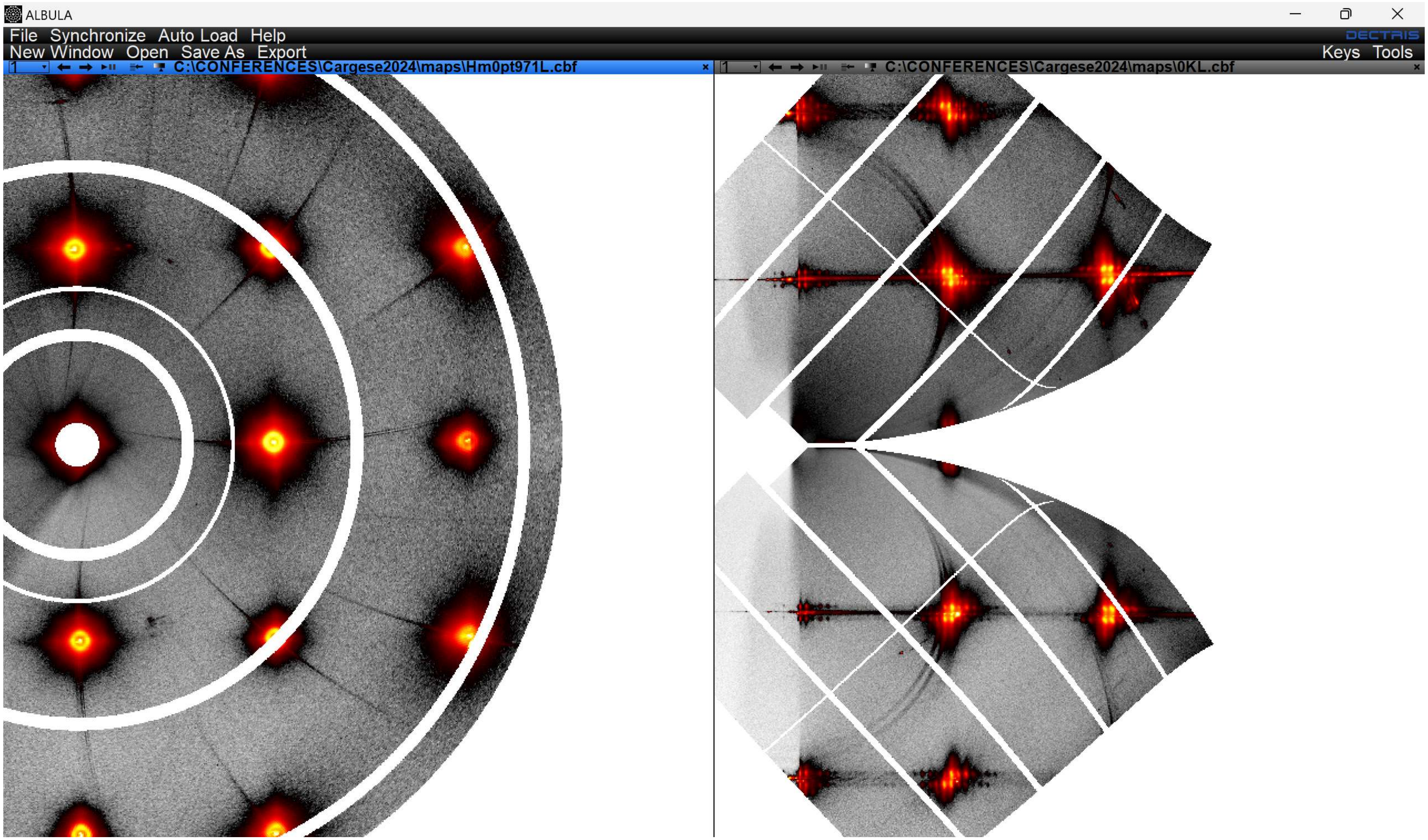
Orthorhombic κ-Ga_2O_3



data collection time ~10 min

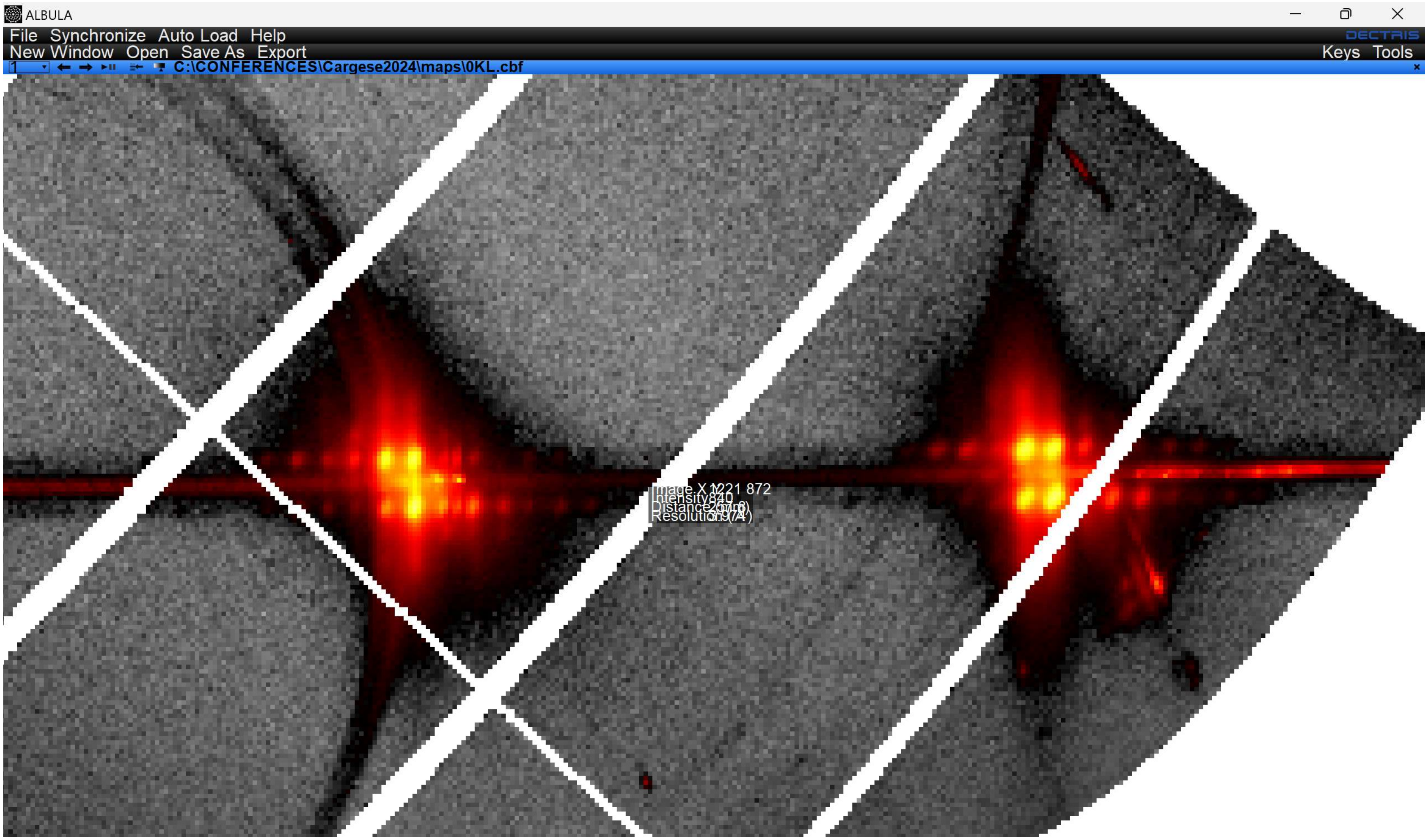
[Matteo Bosi, Jorge Serrano]

Ferroelectric superlattices



[M. Hadjimicael et al., under review]

Ferroelectric superlattices



[M. Hadjimicael et al., under review]

Near future (?)

ID28 ongoing optics upgrade =>
Ø2 μm focal spot + good stability

Integration of strain device in closed-cycle cryostat?
Uniaxial strain with hot+cold gas blowers?

Proposals

Deadline: 10th September 2024

IXS spectrometer: phonon spectroscopy | phonons for given momentum transfer

Diffractometer: diffraction and diffuse scattering | fast mapping of reciprocal space