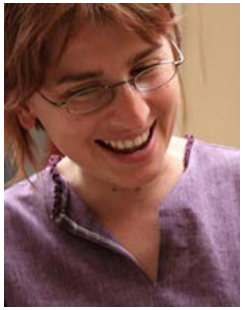
Bragg ptychography: Recent results and perspectives at 4th generation synchrotron sources

Virginie Chamard

The COMiX team at Institut Fresnel

Coherent Optical Microscopy and X-rays



V. Chamard
Coherent X-rays

M. Allain
Inversion

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Optical microscopy

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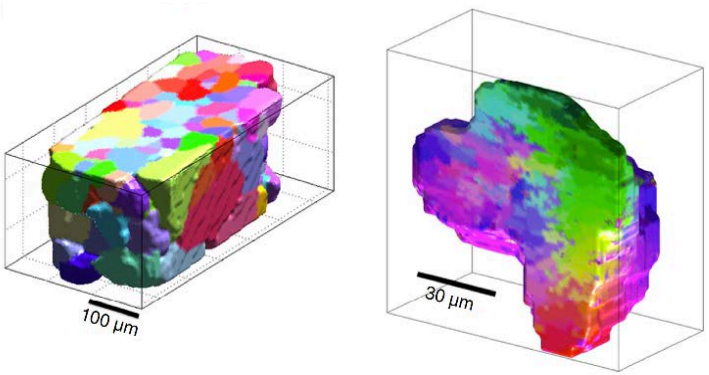
European Research Council

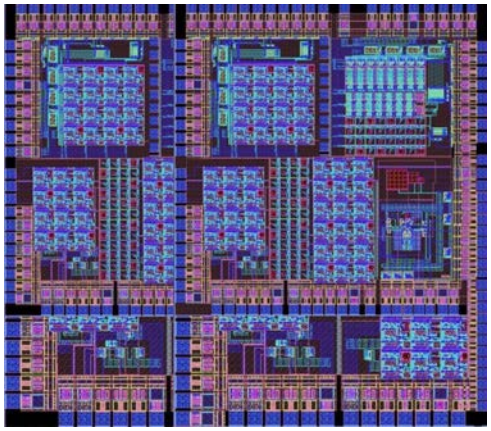
This project has received funding from the European Research Council (ERC) under the European Union's Horizon H2020 research and innovation program grant agreement No 724881

Imaging crystalline materials at the nanoscale

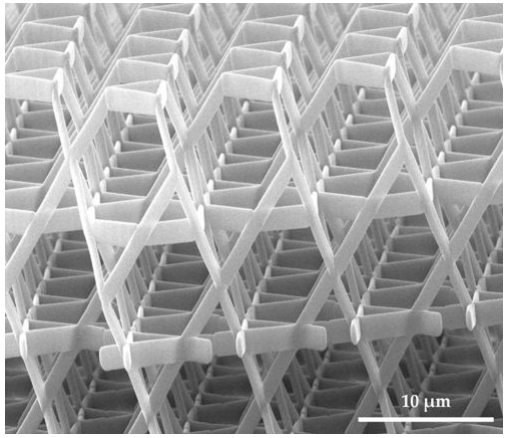
Understand



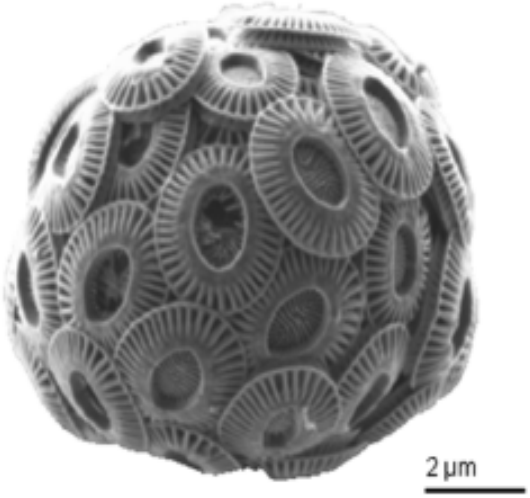
Assemble



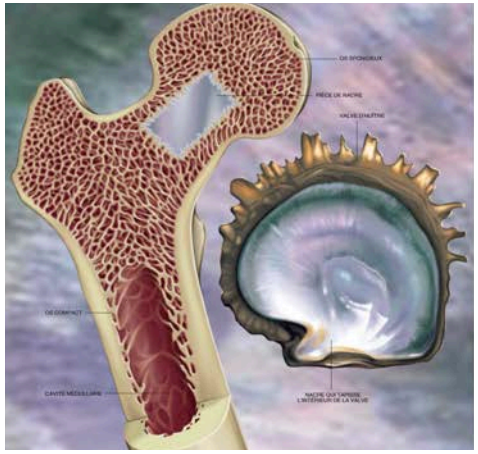
Design



Mimic

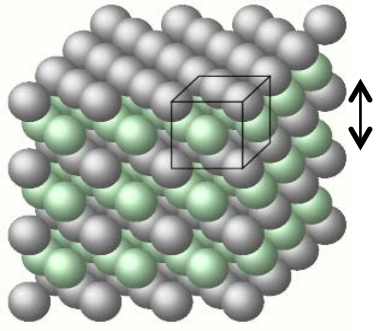


Use



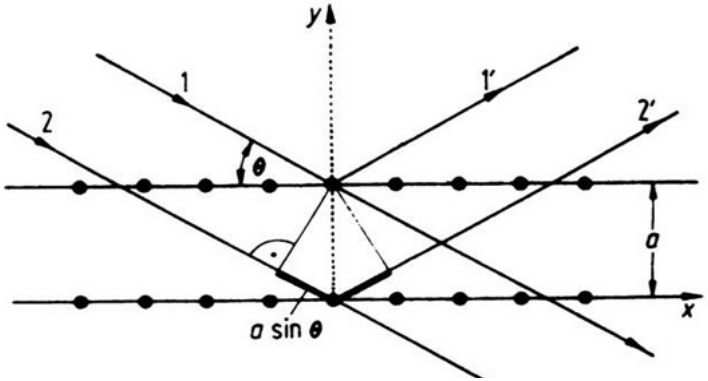
Predict

Imaging crystalline materials at the nanoscale



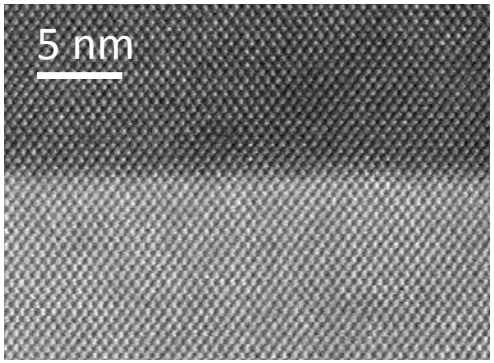
a ≈ 5 Å

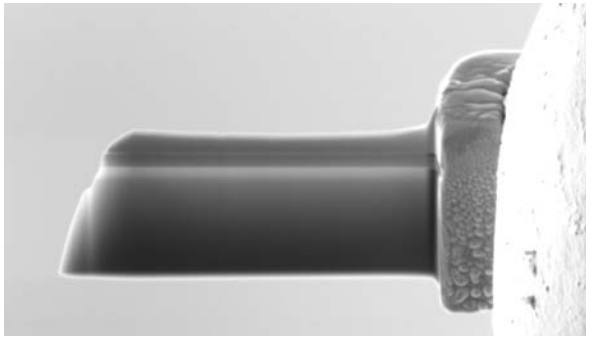
Bragg's law
$2a \sin\theta = n\lambda$
$\rightarrow \lambda < 2a$



- High Resolution electron transmission microscopy

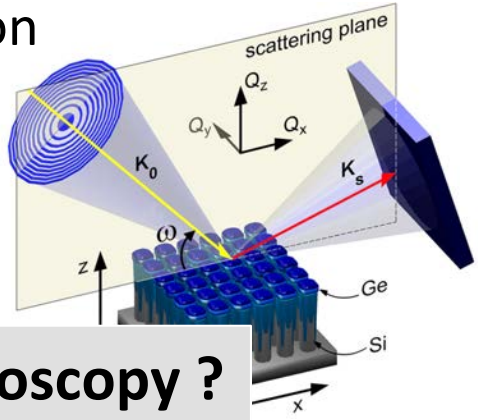
✔ Resolution
✗ Invasive

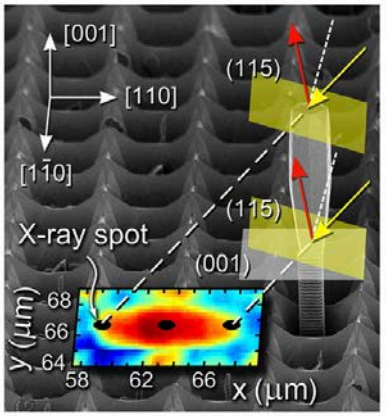




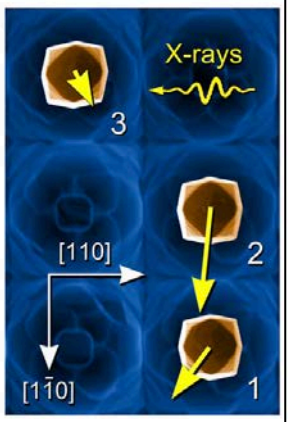
- X-ray nano-diffraction

✔ Non-destructive
✗ Resolution
✗ 2D



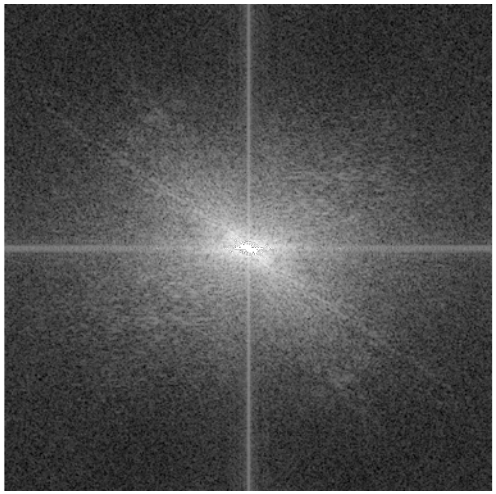


b



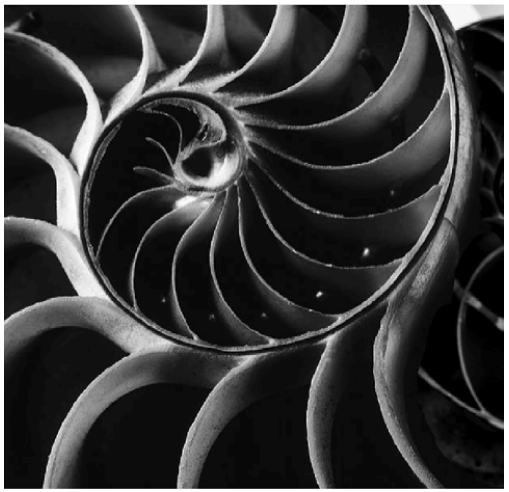
X-ray lens-less microscopy ?

Solving the phase problem: strategies



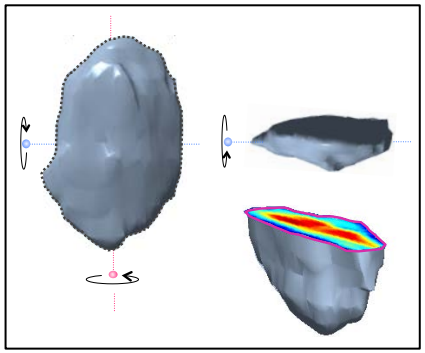
Inverse problem

with intensity data



Experimental set-up

- Encode phases into a ***known*** reference
  → Holography

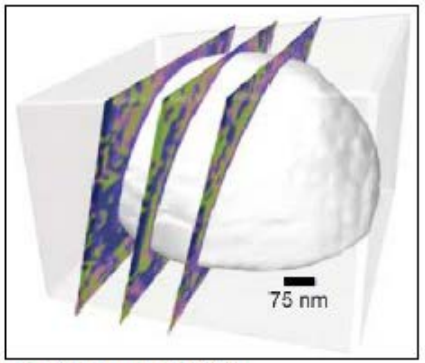


Chamard *et al.*,
Phys. Rev. Lett. (2010)

Sample information

- Add ***known*** constraint

  → Finite support CDI



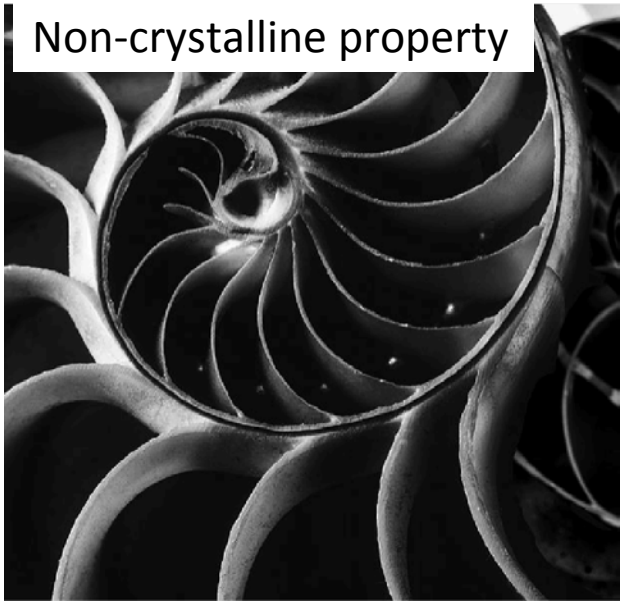
Pfeifer *et al.* (2006).
Nature, **442**, 63–66.

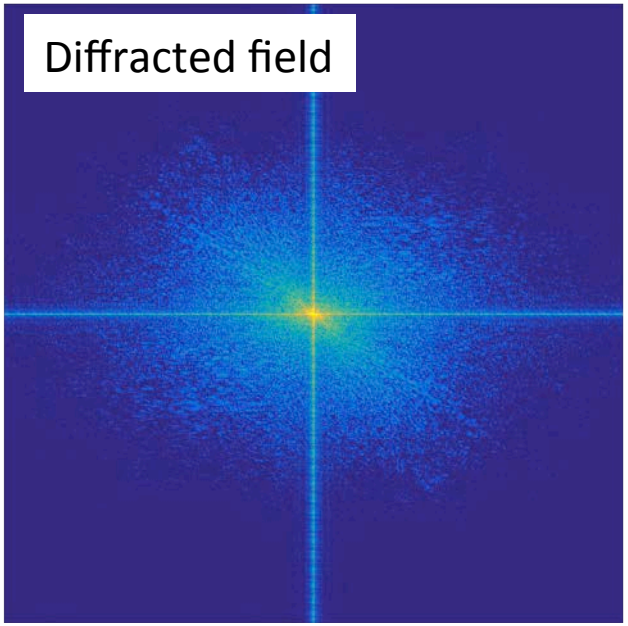
Exploit the probe

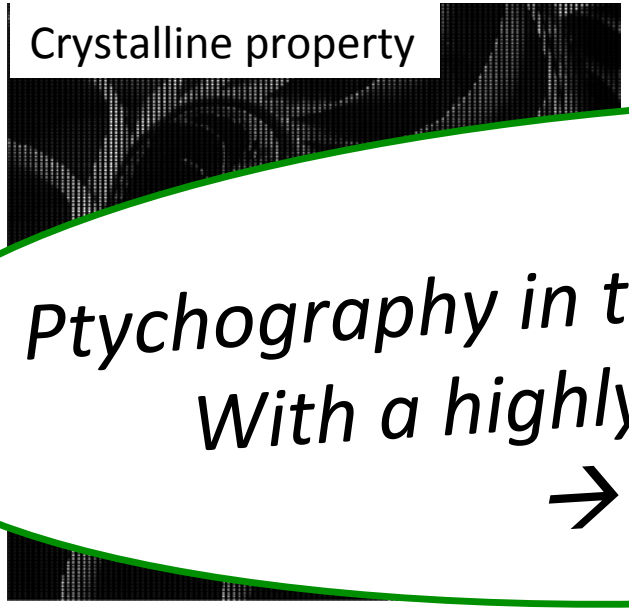
- **Divide the problem into simpler sub-problems with partial *redundancy***

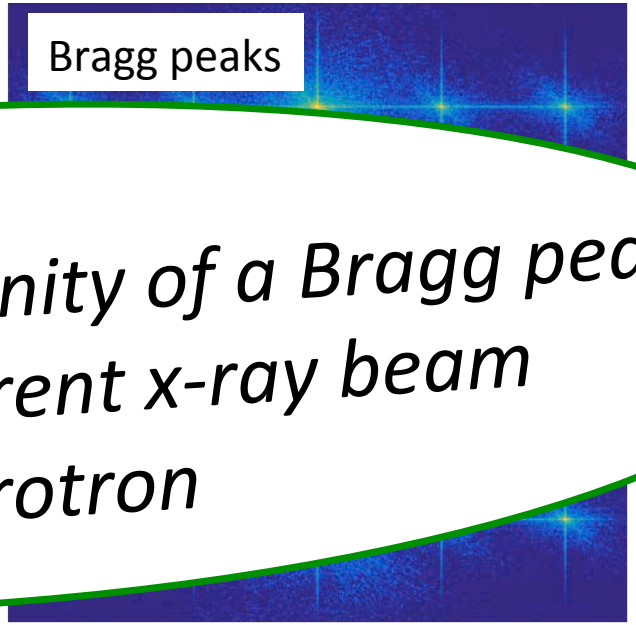
→ Ptychography

Ptychography for crystalline microscopy



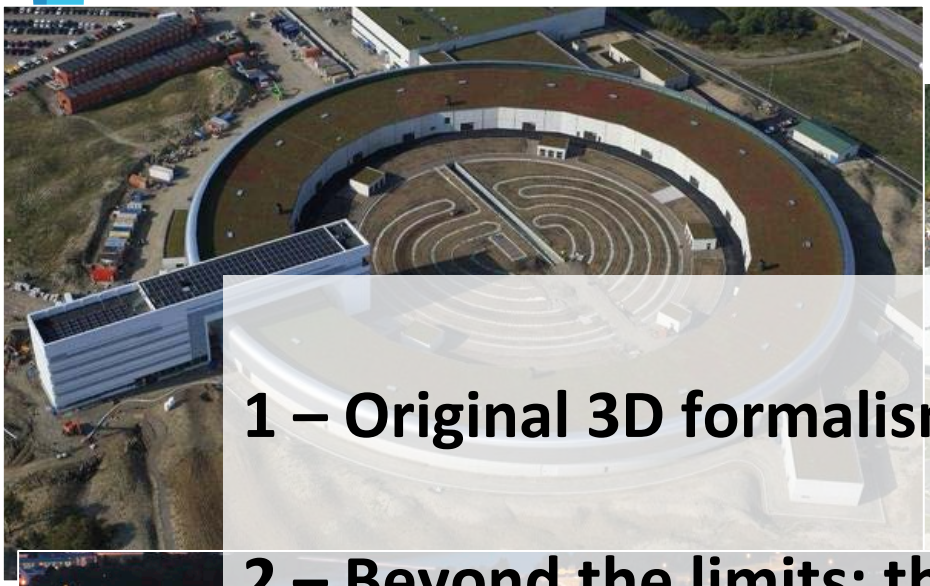


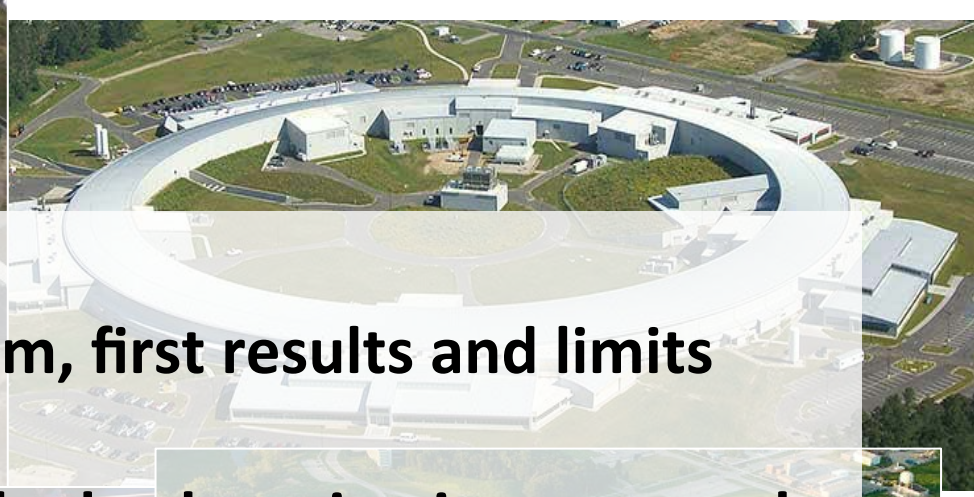




Ptychography in the vicinity of a Bragg peak
With a highly coherent x-ray beam
→ synchrotron

3D x-ray Bragg ptychography



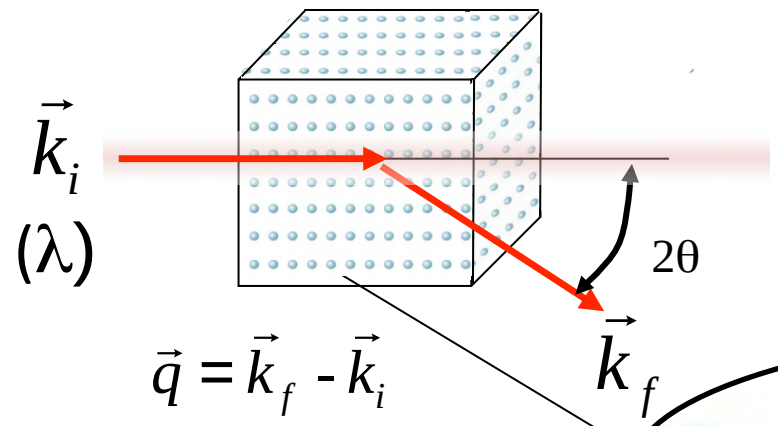


1 – Original 3D formalism, first results and limits

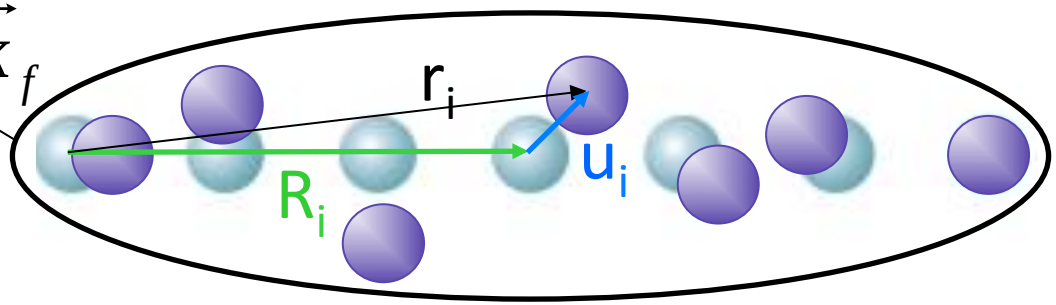
2 – Beyond the limits: the back projection approach

3 – Perspectives: 4th generation synchrotron sources

3D x-ray Bragg ptychography: lattice distortion sensitivity



Assumption on the lattice distortion
+
Fourier synthesis operation



$$\vec{r} = \vec{R} + \vec{u}(\vec{R})$$

$\vec{r}$: strained solid

$\vec{R}$: unstrained solid

$\vec{u}(\vec{R})$: displacement field

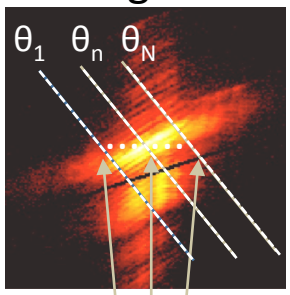
$$I_i(\vec{q}) \propto \left| 3DFT\left\{ P(\vec{R} - \vec{R}_i)\rho(\vec{R})e^{i\vec{G}\cdot\vec{u}(\vec{R})} \right\} \right|^2$$

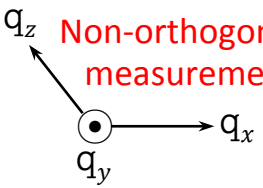
- **3D approach (probe, object)**
- **Complex-valued electron density**
- **Crystal lattice distortion → phase**

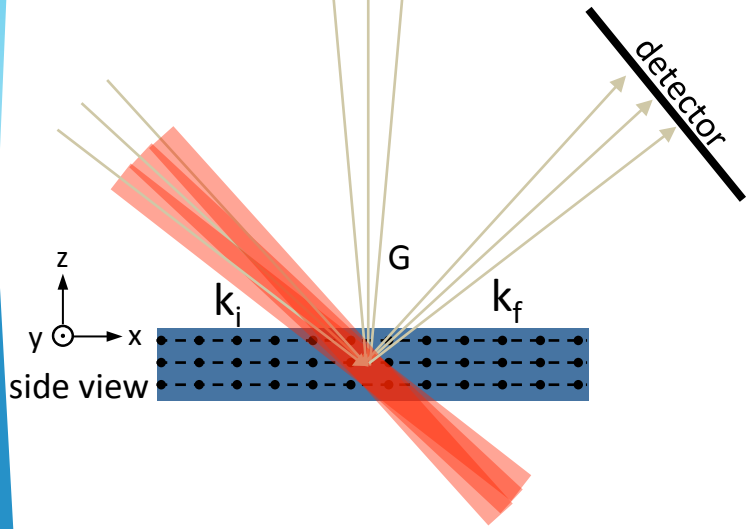
Data acquisition scheme: 3 main steps

3D Fourier components
→ rocking curve scan

(I)

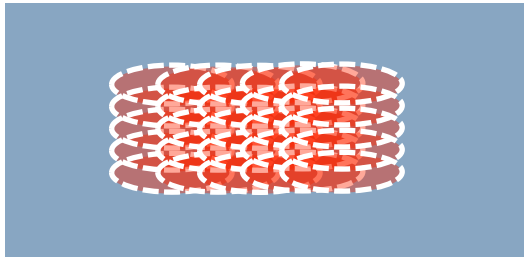


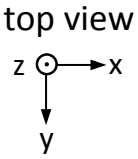




Ptychography: redundancy
Sample translation

(II)

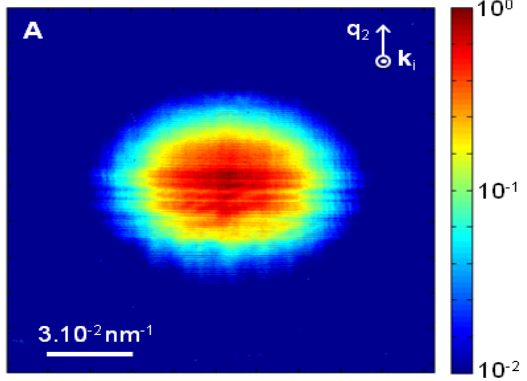


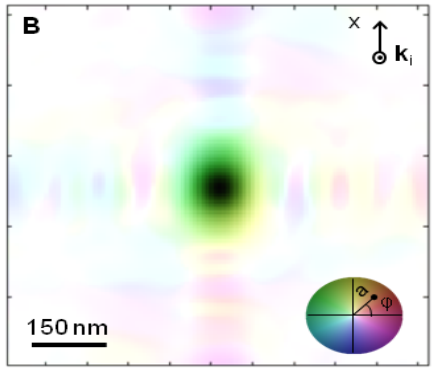


For each probe position,
a full rocking curve scan is
performed

(III) Probe pre-characterization

ID13 (ESRF), Si Refractive lenses (f = 0.01 m), 14.9 keV

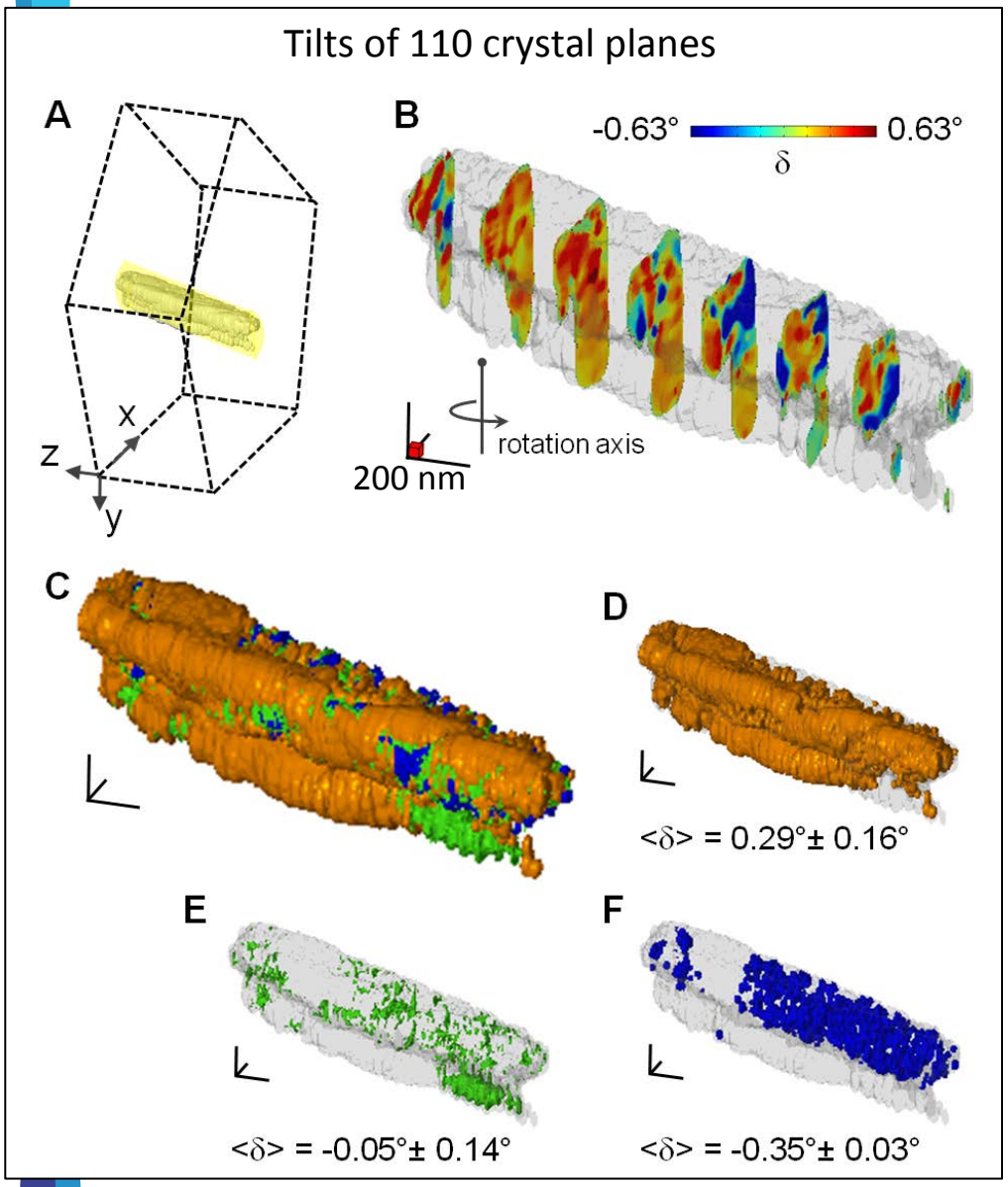


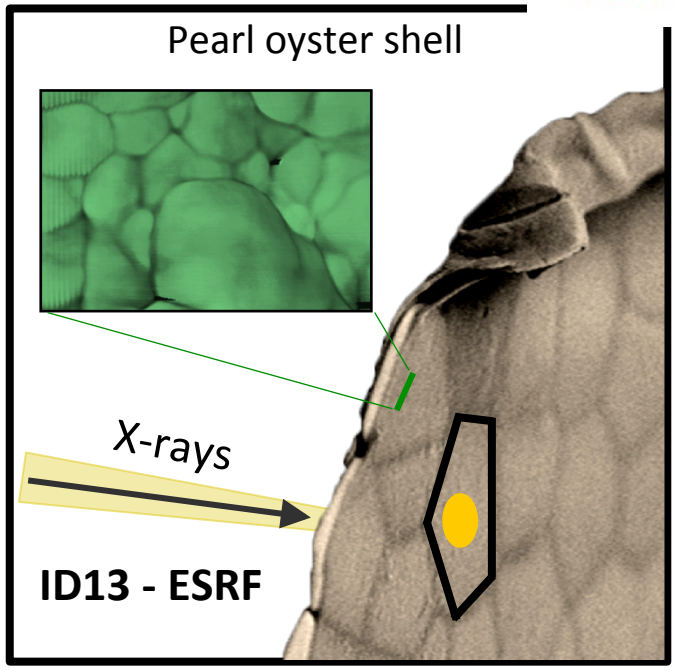


Pateras et al., Phys. Rev. B (2015)

3D x-ray Bragg ptychography: ***Biomineral mesoscale structure***

Organo-mineral granular nano-structure → single crystalline material?



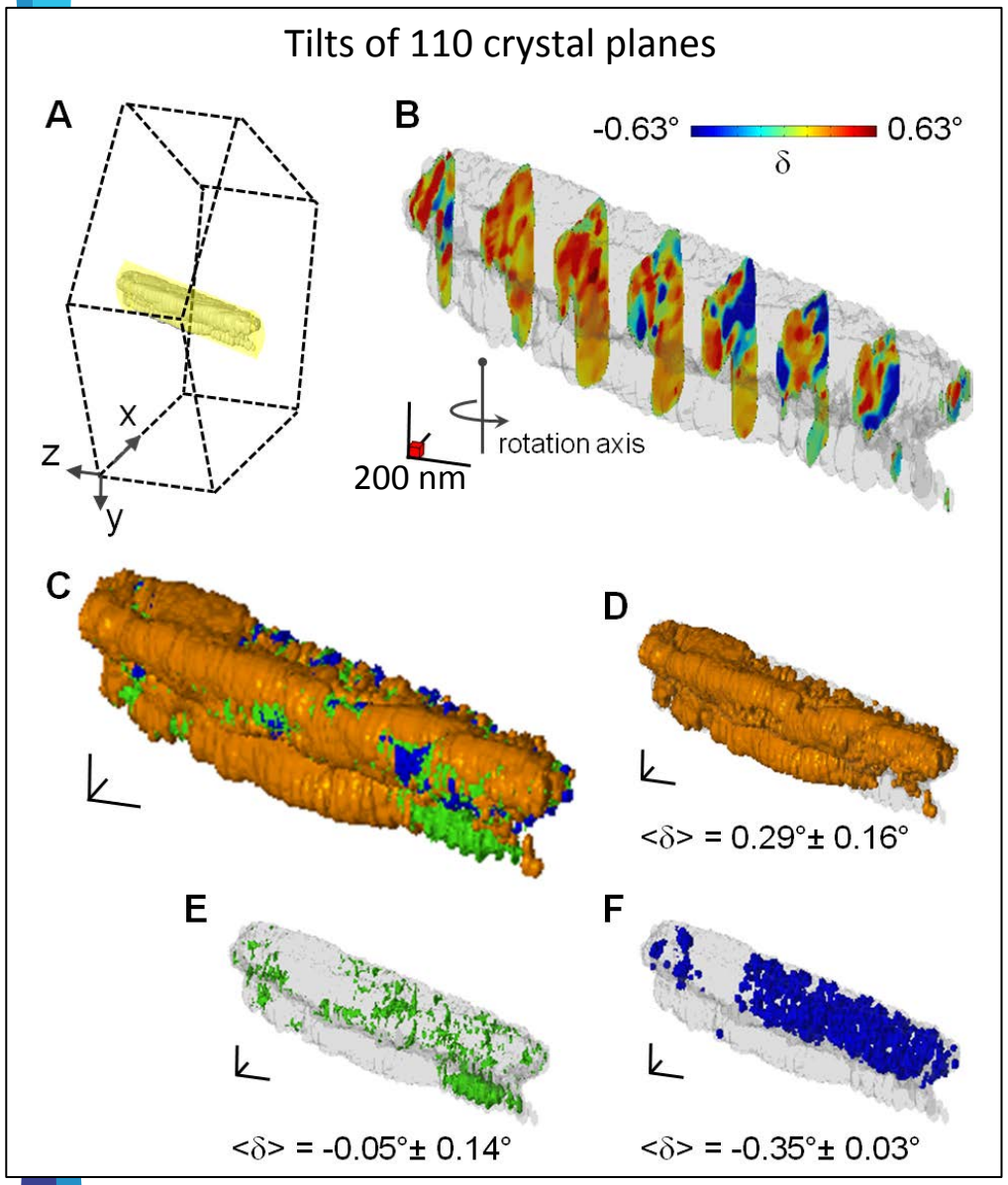


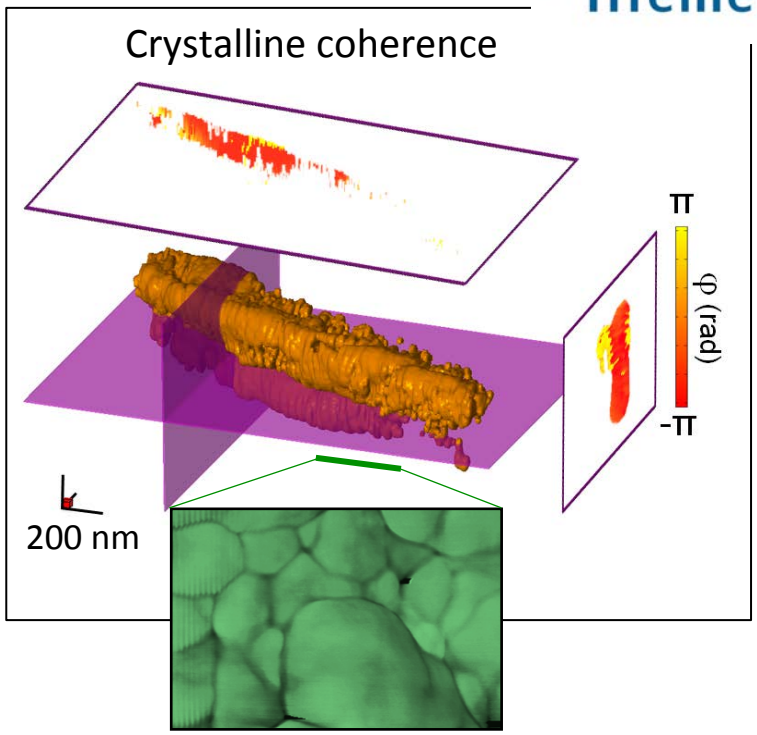
→ Slightly mis-orientated crystalline domains

F. Mastropietro et al., Nature Materials (2017)

3D x-ray Bragg ptychography: ***Biomineral mesoscale structure***

Organo-mineral granular nano-structure → single crystalline material?





→ Slightly mis-orientated crystalline domains

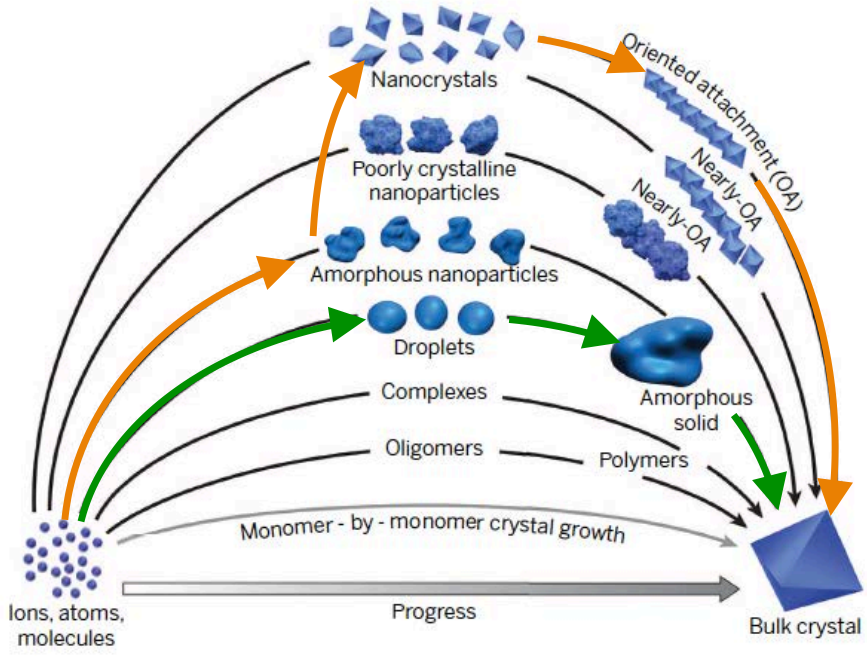
→ Each iso-oriented domain contains several coherent crystals larger than a granule

F. Mastropietro et al., Nature Materials (2017)

3D x-ray Bragg ptychography: ***Biomineral mesoscale structure***

Organo-mineral granular nano-structure → single crystalline material?

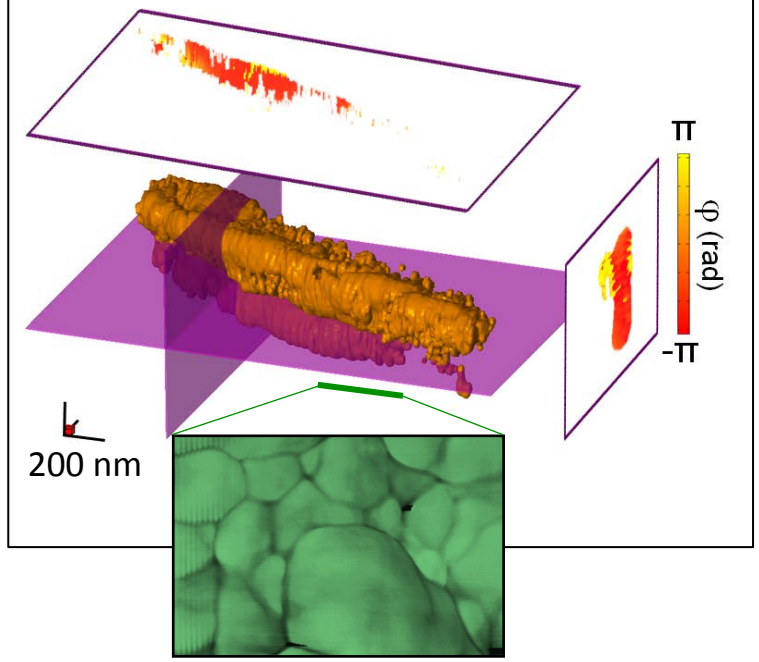
In vitro synthesis



De Yoreo et al., Science (2015)

→ **mesocrystal formation + partial fusion**
or/and
→ **liquid/amorphous droplet precursor**
or/and ?

Crystalline coherence



→ Slightly mis-orientated crystalline domains

→ Each iso-oriented domain contains several coherent crystals larger than a granule

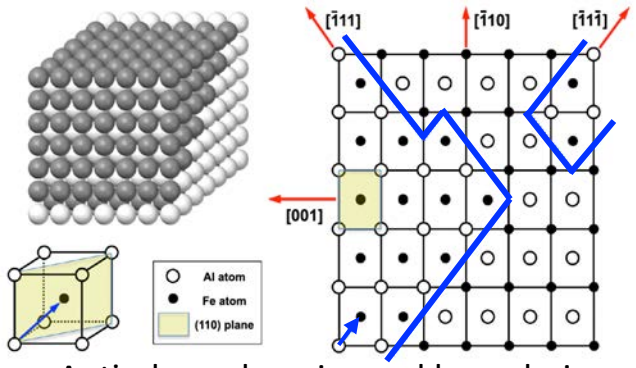
F. Mastropietro et al., Nature Materials (2017)

3D x-ray Bragg ptychography: ***Anti-phase domain Boundaries***

3D ADB structure and strain relationship?

Binary intermetallic alloy (Fe-Al) → Rich phase diagram (several ordered phases, order-disorder transition)

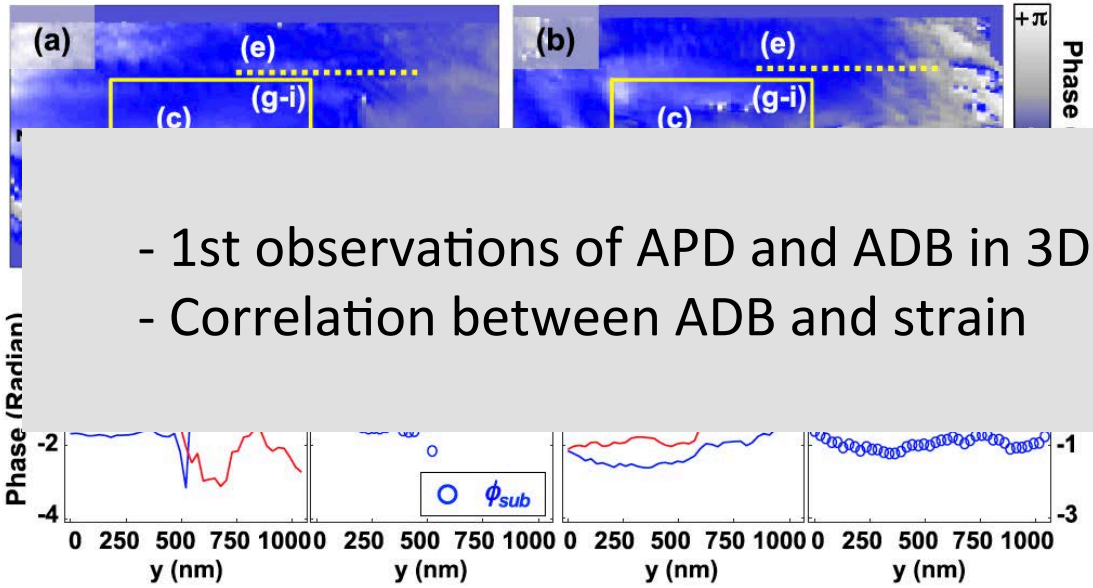
- Several attempts to image with TEM, BCDI…
- Difficulties: **3D sub-µm *phase* domains**.



Anti-phase domains and boundaries

ID01 - ESRF

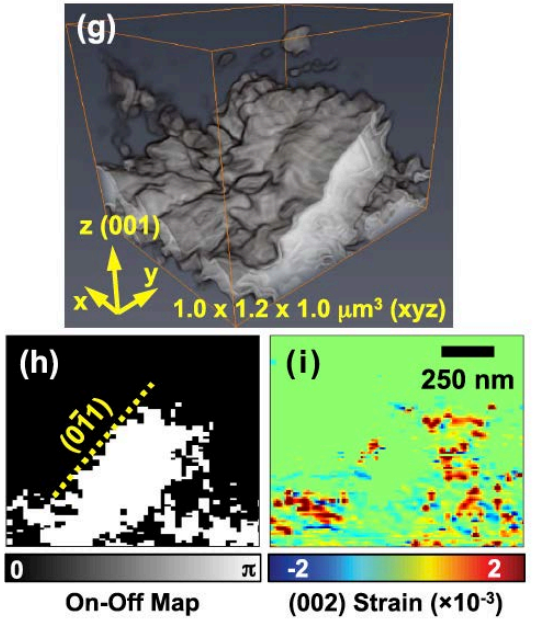
Comparing 001 and 002 3D lattice displacements



- 1st observations of APD and ADB in 3D
- Correlation between ADB and strain

001→ boundaries and strain, 002 → strain

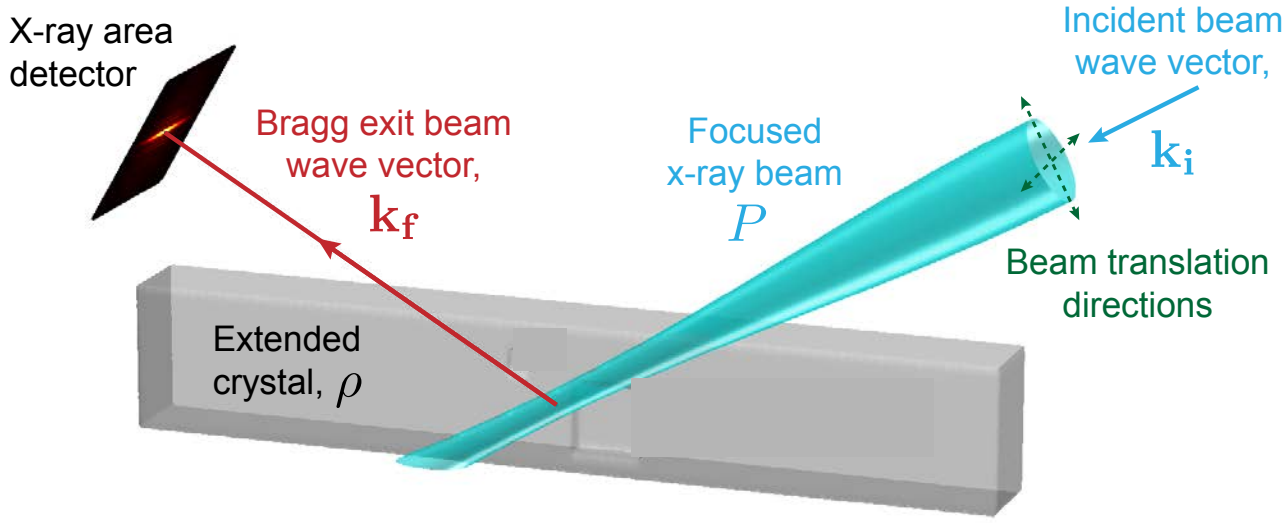
Boundaries vs strain



C. Kim et al., Phys. Rev. Lett. (2018)

2D X-ray Bragg ptychography approaches

2D x-ray *Bragg* ptychography: a straightforward development of 2D ptychography



In the 2D detector :
(Fourier slice theorem)

$$\int P(\vec{r}-\vec{R}_n)\rho(\vec{r})d\vec{k}_f$$

is approximated to $\int P(\vec{r}-\vec{R}_n)d\vec{k}_f \int \rho(\vec{r})d\vec{k}_f$ for 2D Bragg ptychography analyses

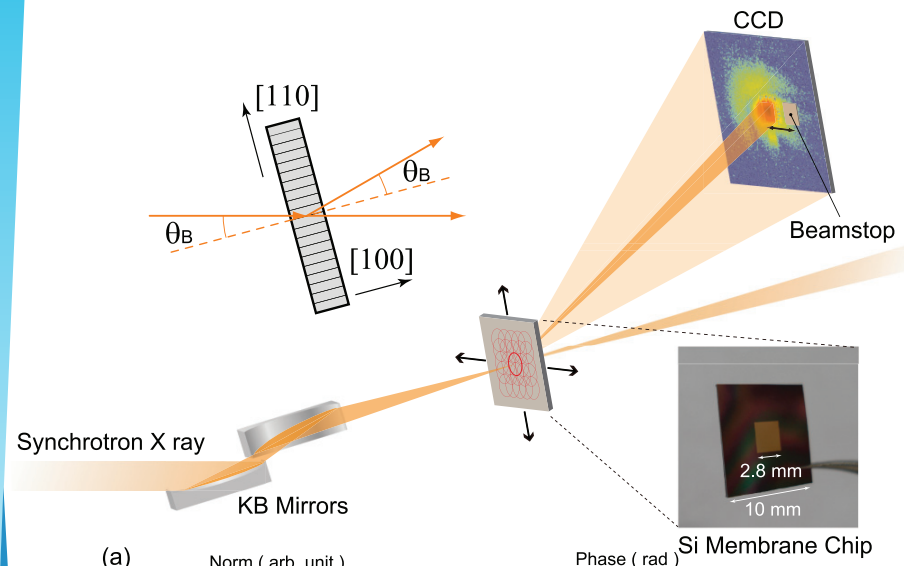
It works, if

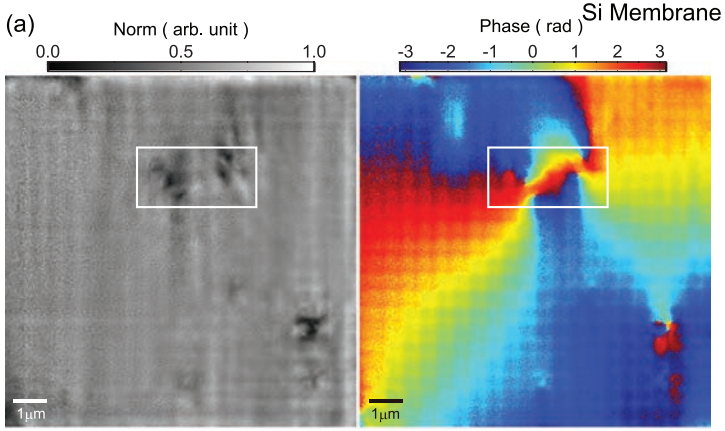
- ✓ $\vec{k}_f \approx \vec{k}_i$
- ✓ Or film thickness < beam size

2D X-ray Bragg ptychography approaches

Powerful approximation of the 3D Bragg ptychography formalism

Dislocation strain field in a Silicon thick film

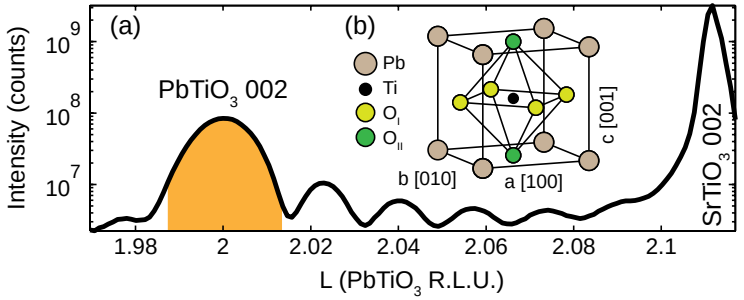


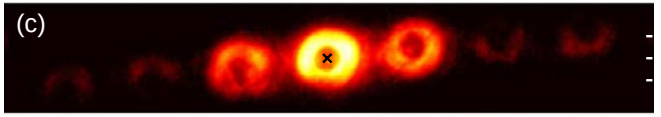


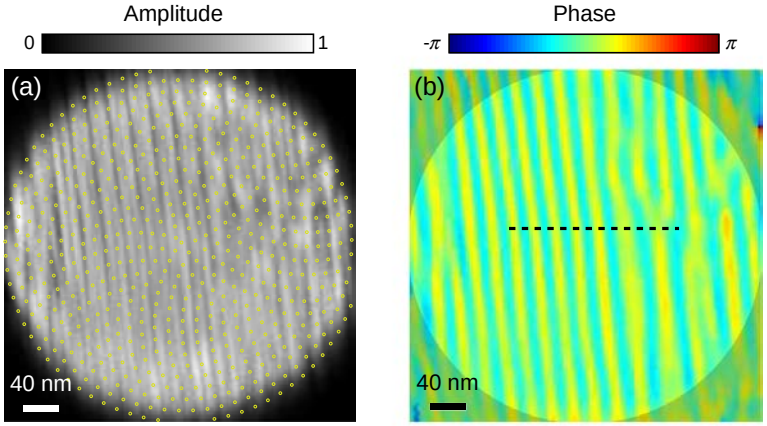
Laue geometry ($2\theta_B \approx 32°$)

Y. Takahashi et al., Phys. Rev. B (2013)

Polarization domains in ferroelectric film







25 nm $PbTiO_3$ thin film

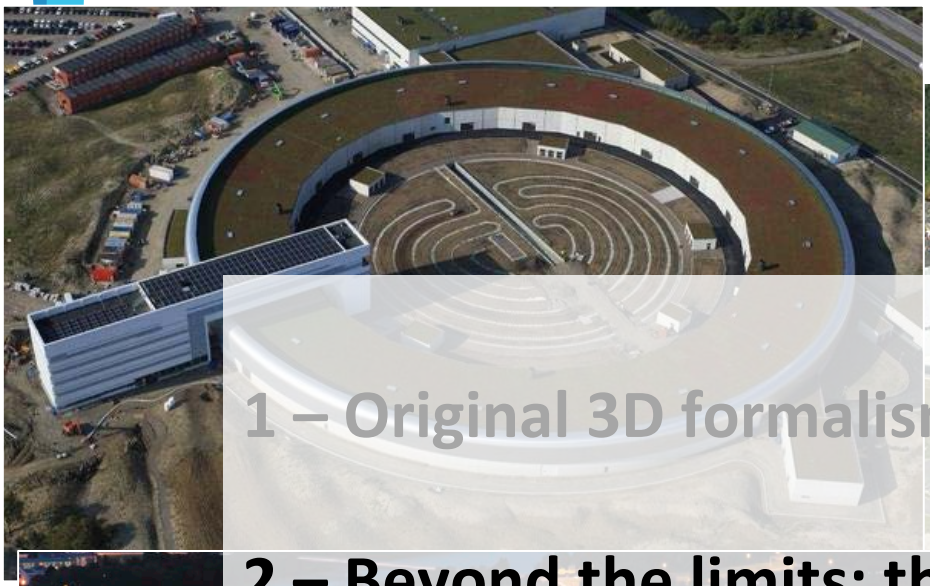
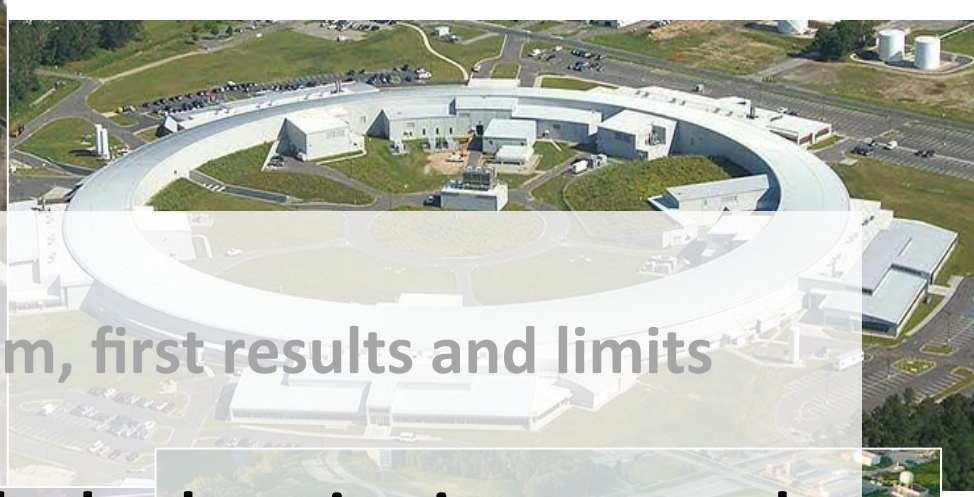
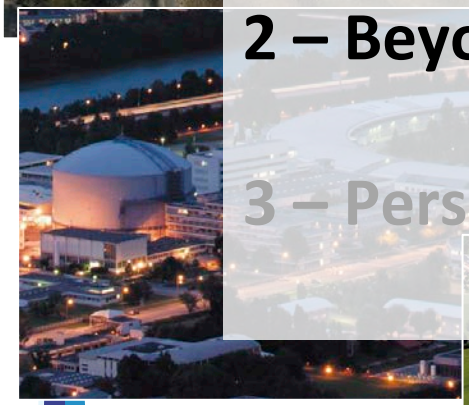
S. Hruszkewycz et al., Phys. Rev. Lett. (2013)

3D x-ray Bragg ptychography: ***some numbers***

- **Spatial resolution**
  → 3D, 10 – 30 nm, anisotropic
- **Strain sensitivity**
  → 10^{-4} – 10^{-2}
- **Lattice rotation sensitivity**
  → 5.10^{-3} – 1°

- Field of view
  → < 1µm^2
- Thickness
  → 1-2 µm
- Total acquisition time
  → From 10 hours to 3-4 hours
- Probe size
  → 80 nm to 1 µm
- Translation step size
  → (probe size) 25 nm
- Angular step size
  → 0.003° to 0.01°
- Data size
  → 20 x 20 x 300 x 256 x 256
- Inversion time
  → A few hours

3D x-ray Bragg ptychography

1 – Original 3D formalism, first results and limits

2 – Beyond the limits: the back projection approach

3 – Perspectives: 4th generation synchrotron sources

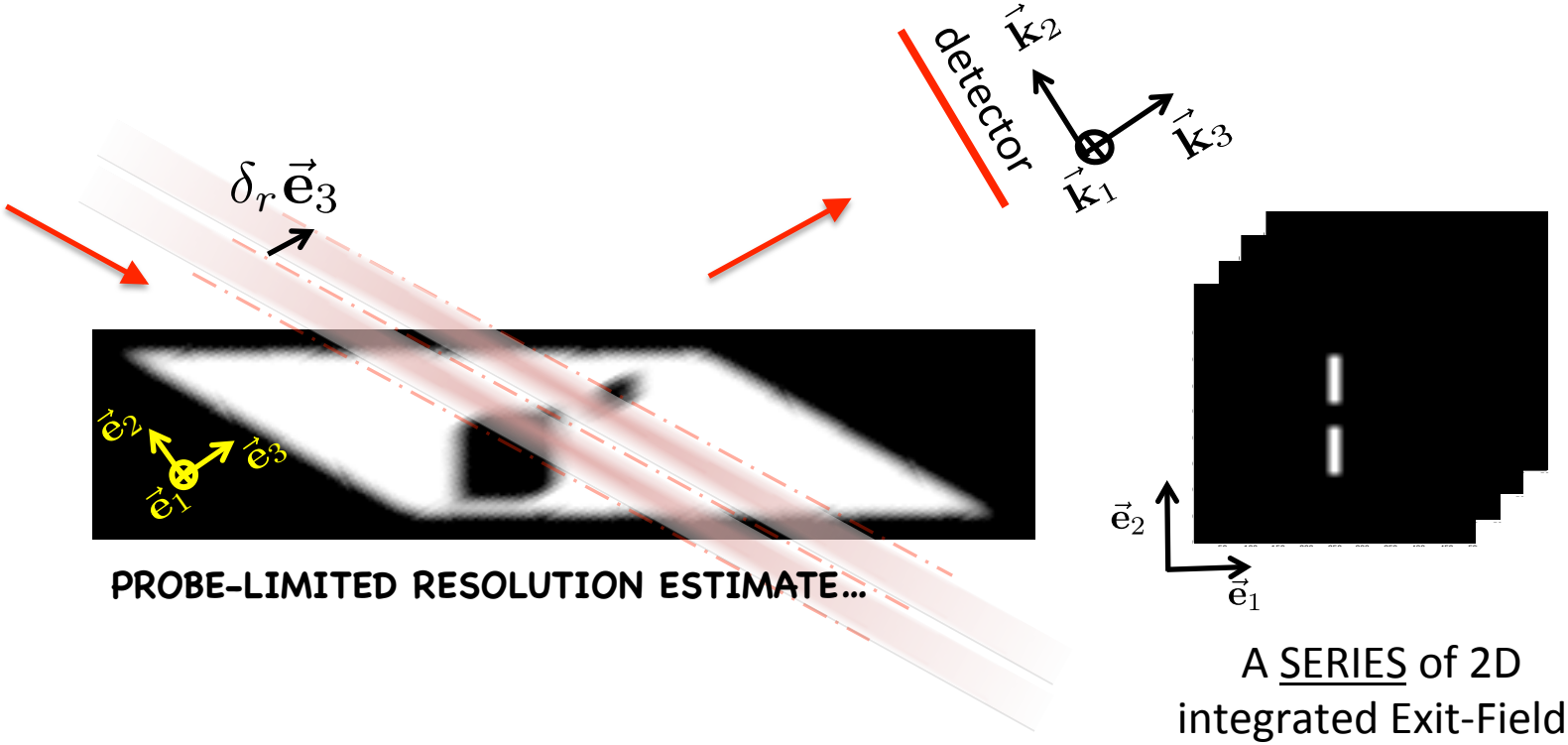
Fast 3D x-ray Bragg ptychography: single angle

3D Bragg projection ptychography: 3D imaging at a **single** tomographic angle

Coll. S.O. Hruszkewycz, M.V. Holt, P.H. Fuoss
(APS/Nanoprobe beamline, CNM)

Argonne NATIONAL LABORATORY

The scanning probe encodes some spatial information along e3



A SERIES of 2D integrated Exit-Fields

S. O. Hruszkewycz et al., Nature Materials (2017).

Fast 3D x-ray Bragg ptychography: single angle

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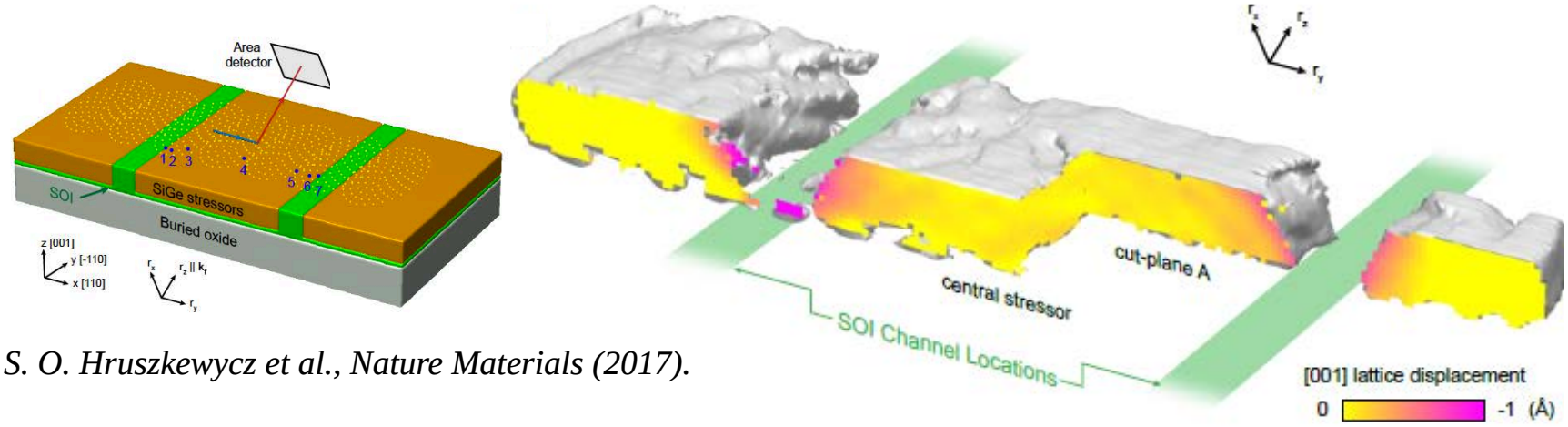
- Modified inversion algorithm based on back-projection operator
- No rocking curve needed
- Reduced acquisition time: x **50-100** gain



S. O. Hruszkewycz et al., Nature Materials (2017).

- No angular/translation registration
- No fine angular steps
- Efficient for energy scan

→ **New standard for Bragg ptychograpy**

Fast 3D x-ray Bragg ptychography: a few angles

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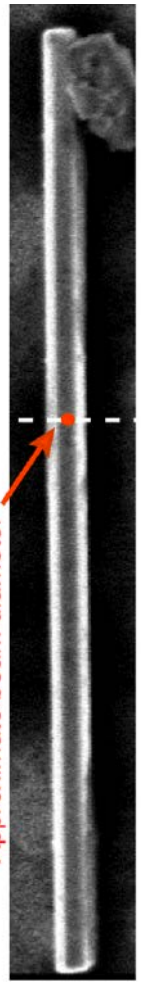
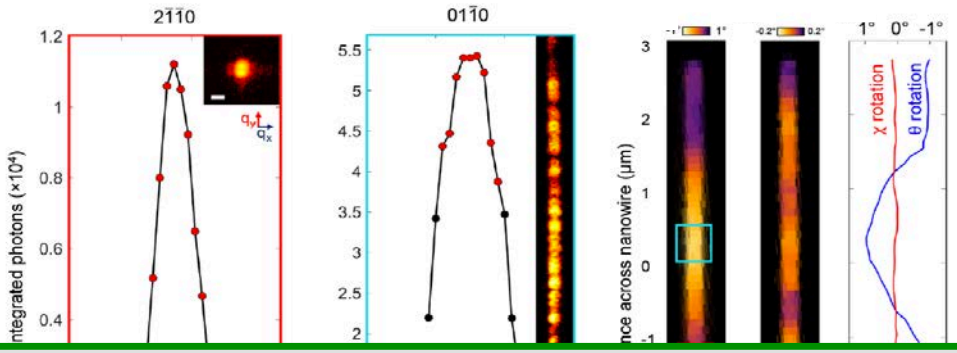
Detailed structure of III-V nanowire → defaults ? Strain ?



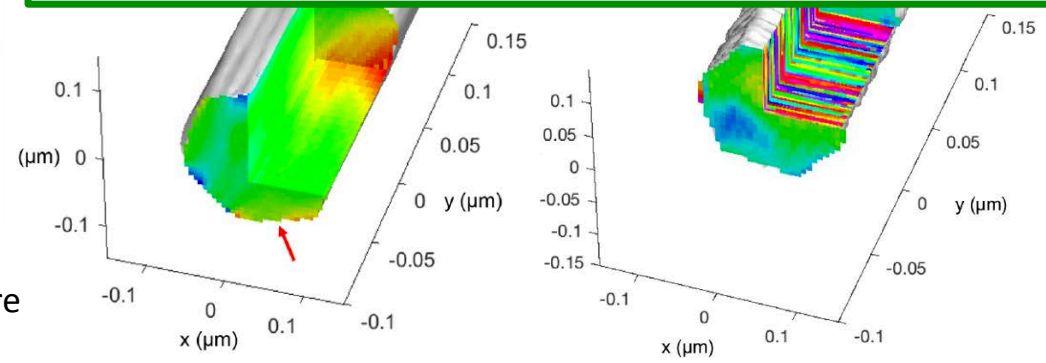
200 nm
InGaAs wire

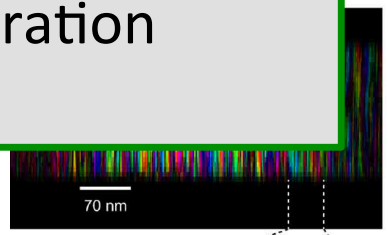


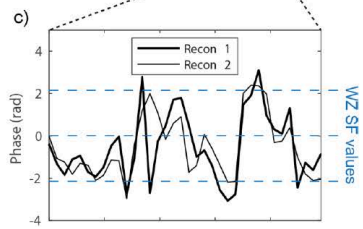
$2\bar{1}\bar{1}0$ Strain sensitive

$01\bar{1}0$ Strain/stacking sensitive

Reduced number of angles along rocking curve
(→ reduced acquisition time)
Decoupling angle and spatial registration







M. Hill et al., Nano Letters (2018)

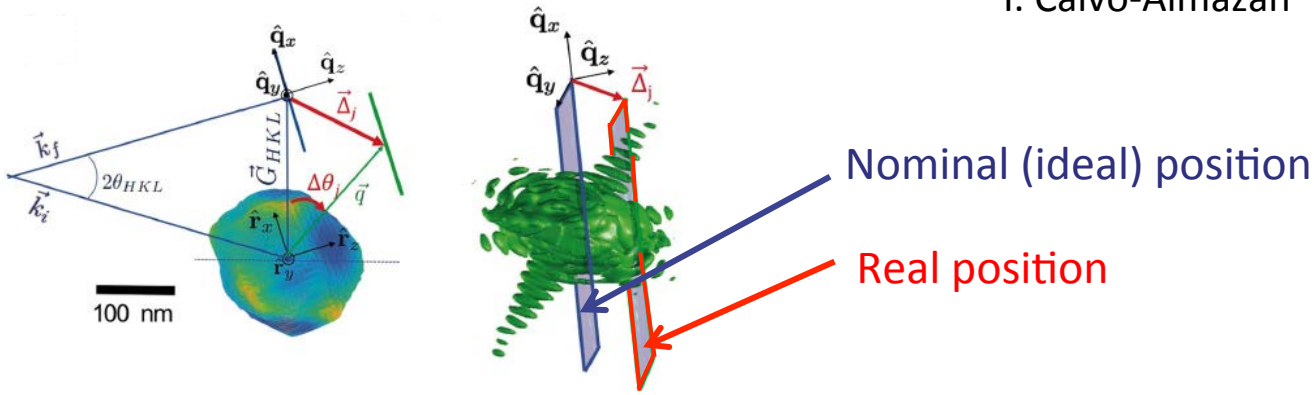
Mitigation of angular uncertainties in Bragg CDI

3D Bragg CDI and ptychography → small angular steps

- Mechanical limits of the instrument
- Environment conditions (heat, pressure, *etc*…)

Argonne NATIONAL LABORATORY

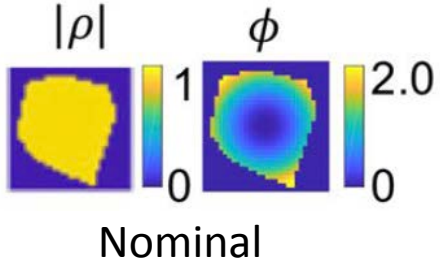
I. Calvo-Almazan



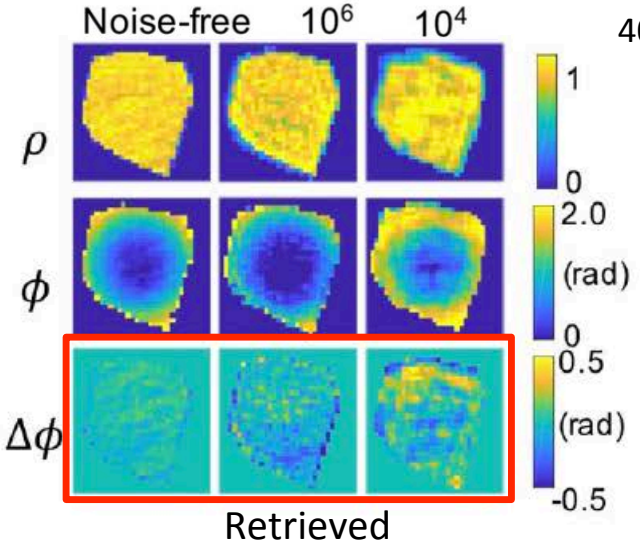
I. Calvo-Almazan et al., Scientific Reports (2019)

Impact of uncertainties

No angular error



40% angular error



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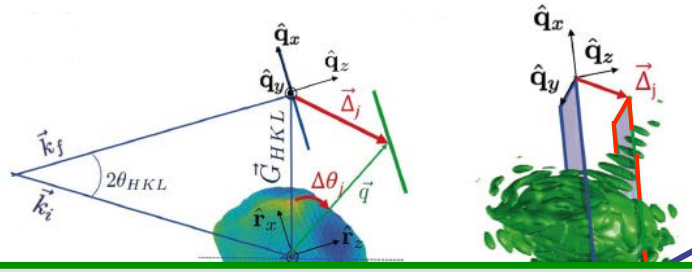
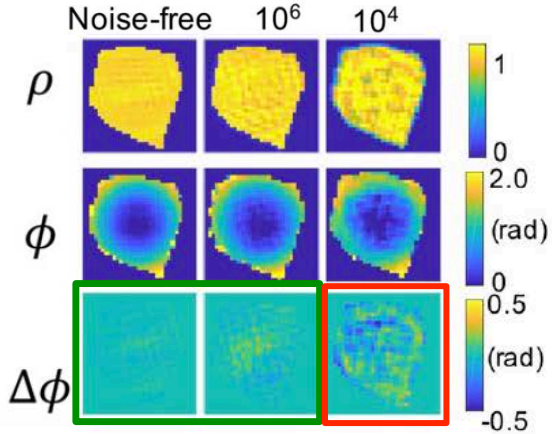


Nominal (ideal) position

Real position

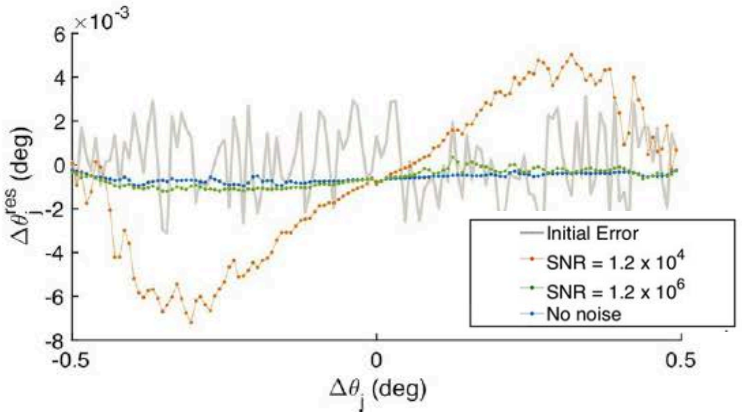
Allowing for some angular uncertainties

Mitigation of uncertainties during inversion process

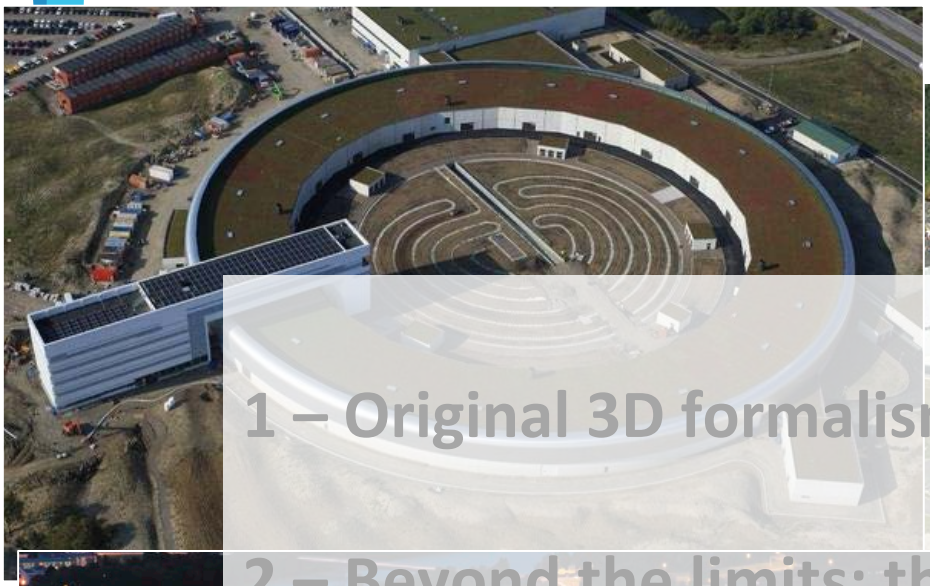


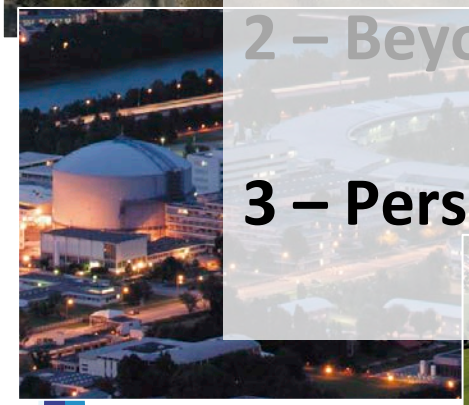
40% angular error

Angular residuals



3D x-ray Bragg ptychography





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What's next ?

Future should be bright: New or upgraded synchrotron sources

→ 100 times more coherent flux !

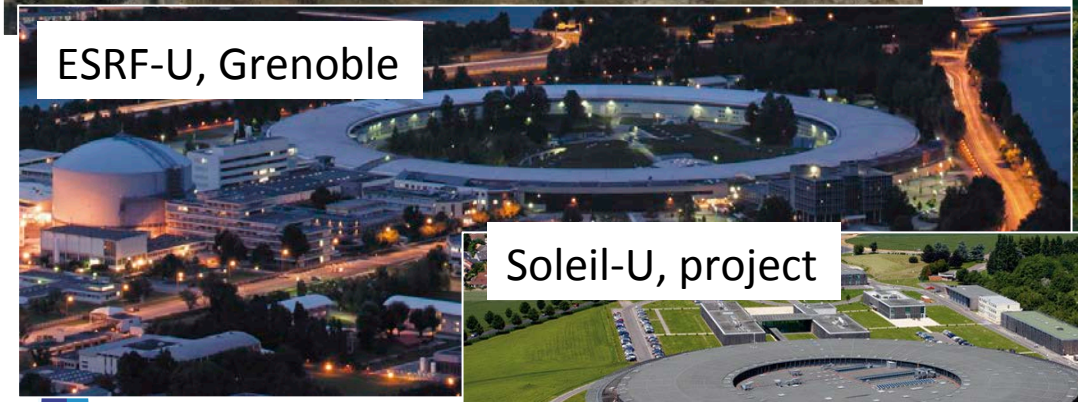
→ Several Bragg ptychography compatible beamlines

MAXIV, Sweden

NSLS2, Brookhaven

APS-U, Argonne

ESRF-U, Grenoble



Soleil-U, project

Conclusions

More difficult than Bragg CDI and forward ptychography, but:

- Weak and strong defects in energy related material
- Iso-oriented domains in biominerals
- Stacking faults in quantum wire
- Anti-phase domains and boundaries in metallic alloy

First tests at 4th generation synchrotron source MAX IV

(Coll. D. Carbone)

→ a full 3D Bragg ptychography in **20 min**

We appreciate supports from synchrotrons, to bring Bragg ptychography to users

We look for strongly motivated users with challenging problems and ready to suffer

We love stable set-ups

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