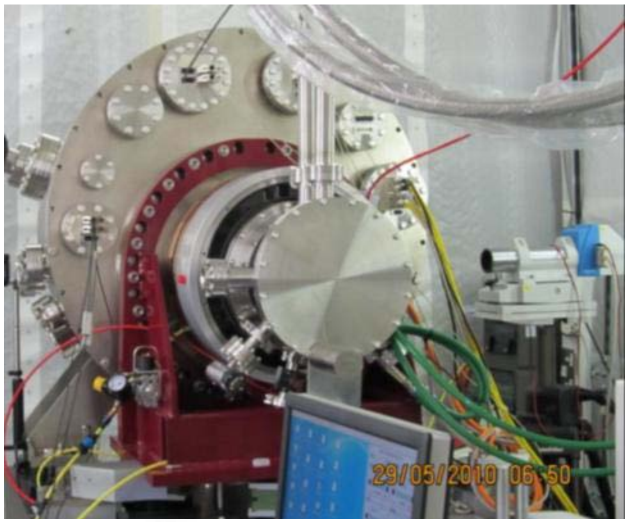
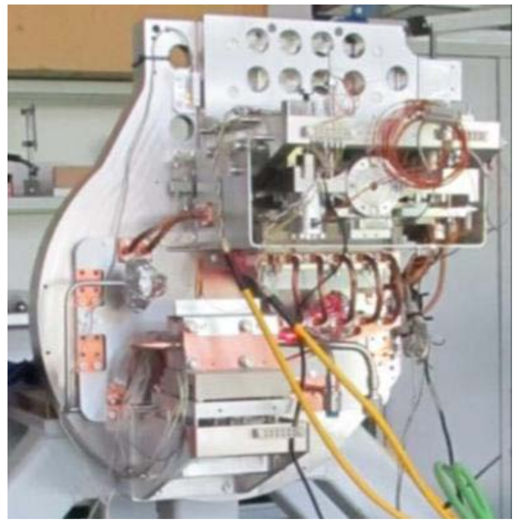
Off-line characterization & optimization of monochromators

Dr. Simon Alcock
Senior Metrology Scientist, DLS

Off-line characterization & optimization of monochromators





Simon Alcock, John Sutter, Alex Bugnar, Hiten Patel, Ioana Nistea, Kawal Sawhney, Jon Kelly, Andy Peach, Peter Docker, Graham Duller
(Diamond Light Source)

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The Problem

DCM performance is a critical issue for many synchrotron beamlines … particularly with high power densities

1) Thermo-mechanical deformation of mono crystal assemblies:

- mechanical clamping
- localised heating by high intensity X-rays
- contraction of dissimilar materials during cryo-cooling

→ Strains in crystal lattice impart aberrations to diffracted X-ray beam & degrade transmitted bandwidth

2) X-ray beam drift & vibration (parasitic motions & cooling)

Problems will worsen for upgraded & future light sources ☹

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Testing DCMs

- Ideal solution: use synchrotron X-rays for DCM commissioning

...BUT

- Limited access to beamtime ☹
- Difficult to decouple effect of individual components ☹
- Build & testing over an extended period(s) → scheduling! ☹
- During beamline construction, optics hutch unsuitable for sensitive work? ☹

Much of the work can be performed using off-line techniques prior to synchrotron X-ray tests ☺

Off-line pre-commissioning & optimisation

- Are crystals strained by clamping?
- Efficient thermal interface?
- Crystal deformation during cryo-cooling?
- Crystal deformation during X-ray beam heating?

- Do motorised stages meet specification? (range, resolution, repeatability, etc)
- Parallel / perpendicular / alignment of axes?
- Parasitic motions or long term drifts?
- Vibration induced by cooling?
- [+ Motion controls commissioning]

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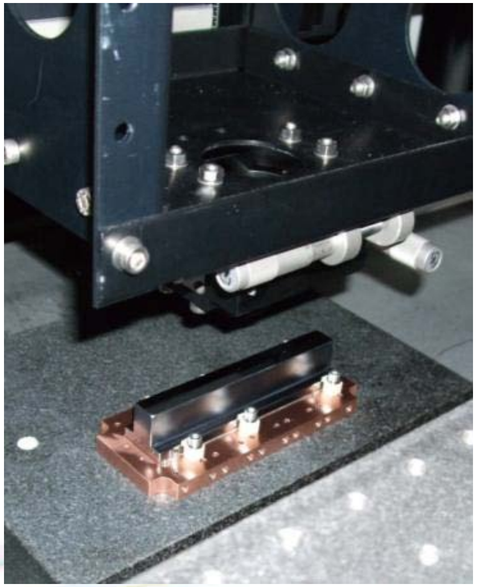
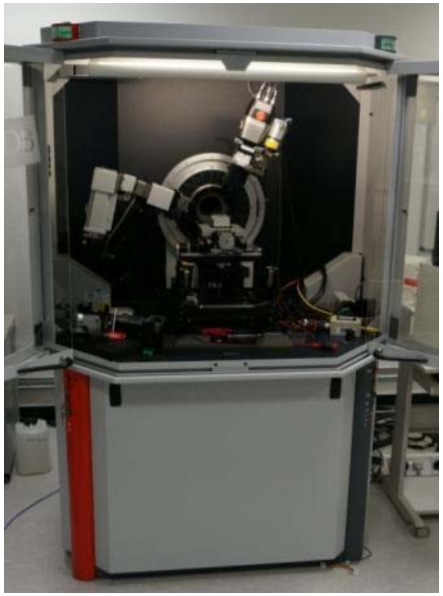
Section 1: Mono crystals

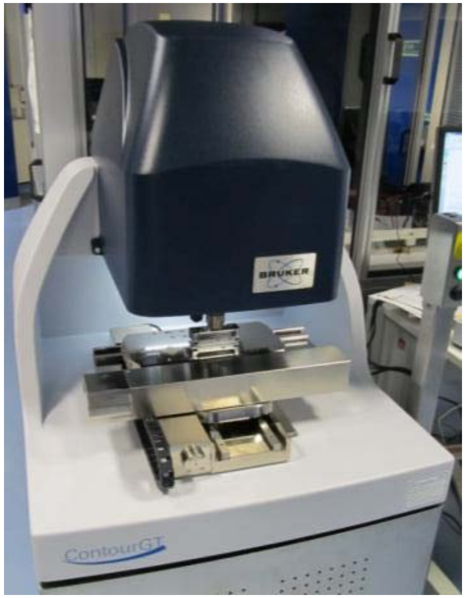
- Mono crystals procured from vendors & acceptance tested by DLS Optics & Metrology group



Diamond-NOM slope profiler



Bruker D8 Discover X-ray diffractometer

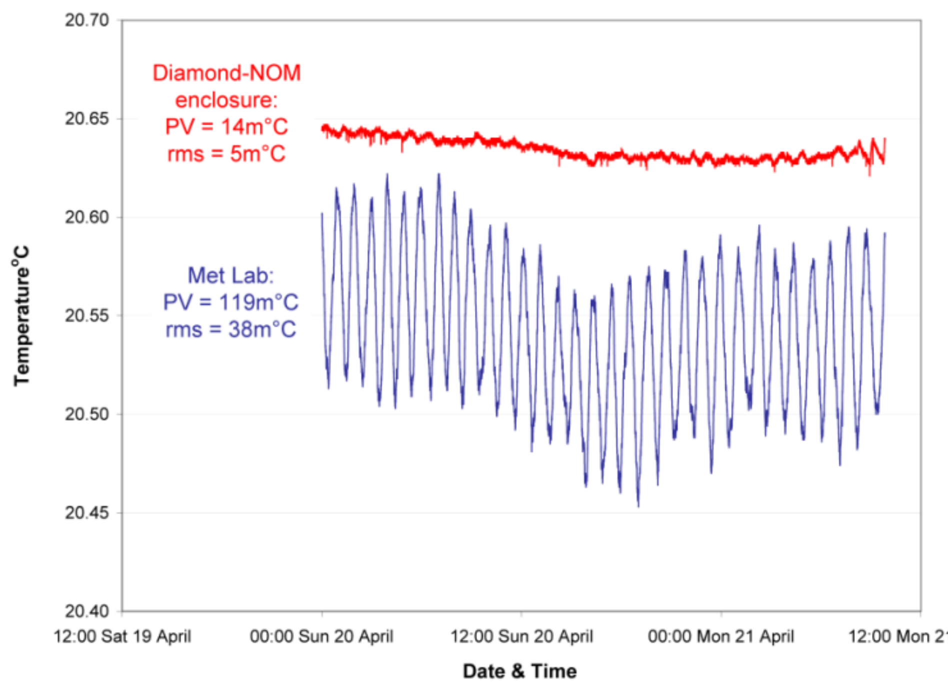


Bruker Contour GT-X micro-interferometer

→ miscut angle, rocking curve, micro-roughness, flatness, …

DLS Optical Metrology lab

- Class 10,000 (ISO7) cleanroom
- $100m^2$
- Active temperature control
- Passive environmental enclosures
- Excellent vibration stability



Temperature stability

<5m°C rms over ~36hrs

~1m°C rms over ~5hrs

Essential for nano-metrology!

Optical Metrology instruments

High spatial frequency errors

Mid spatial frequency errors

Low spatial frequency errors

Atomic Force Microscope (AFM)

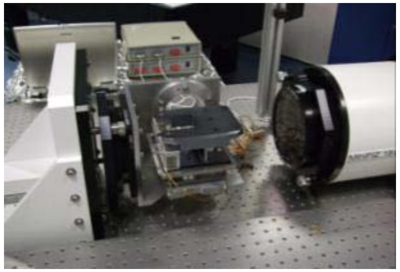
- Atomic defects & gratings
- Lateral scan size: up to 50µm x 50µm
- Lateral resolution: <1nm
- Vertical resolution: 0.01nm

Phase shifting micro-interferometer

- 3-D micro-topography
- Lateral scan size: 50µm → 5mm (+stitching)
- Lateral resolution: 0.1µm → 9µm
- Vertical resolution: 0.05nm (rms)

Fizeau interferometer

- 3-D height measurement
- Beam diameter: 150mm
- Lateral scan size: 150mm → 1800mm
- Planar & spheric accuracy: λ/100 PV



Diamond-NOM

- Slope profilometry
- Lateral scan size: 1500mm
- Lateral resolution: <1mm
- Repeatability <50nrad

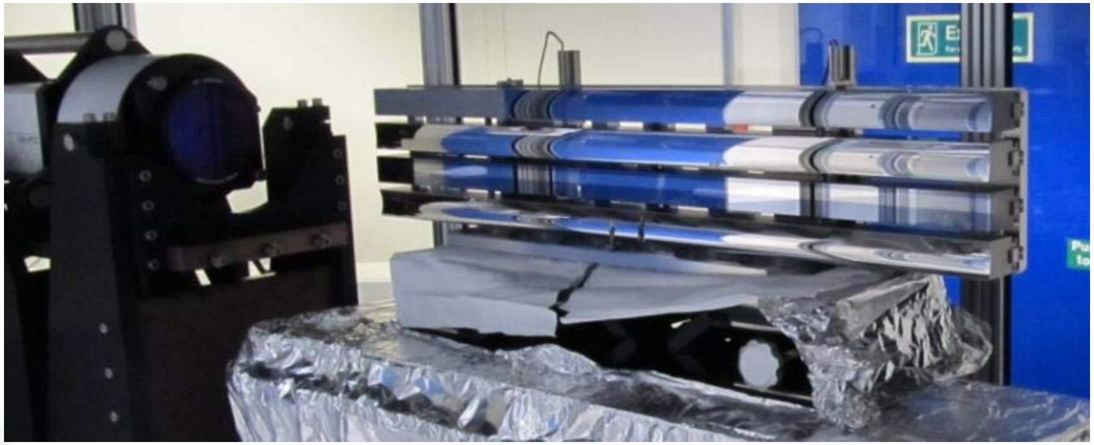
Optimise fully assembled beamline optic!

HR mirrors (side cooling)

PGM mirror (side cooling)

Internally cooled mirror

4 x 1.2m stacked mirrors!!!



How do crystals deform when clamped?

1) Indirect measurement of crystal lattice (*change* in optical surface)

   a) *Diamond-NOM slope profiler*
   b) *MiniFiz150 Fizeau interferometer*

2) Direct X-ray measurement of crystal lattice

   a) *Bruker D8 Discover X-ray diffractometer*
   b) *Synchrotron light at B16 Test beamline*

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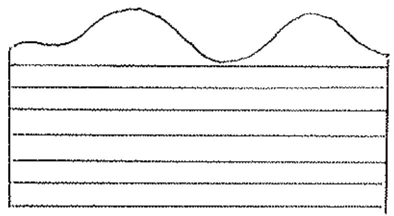
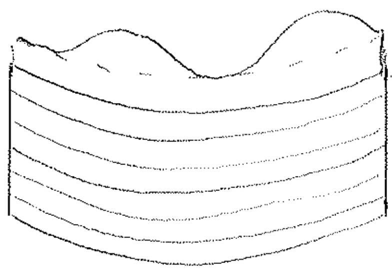
1) Indirect measurement of crystal strain

A



Measure external surface of unclamped, unstrained crystal

B



Remeasure external surface of clamped (& strained?) crystal

B – A = change in external surface

→ Infer that internal crystal planes have also deformed

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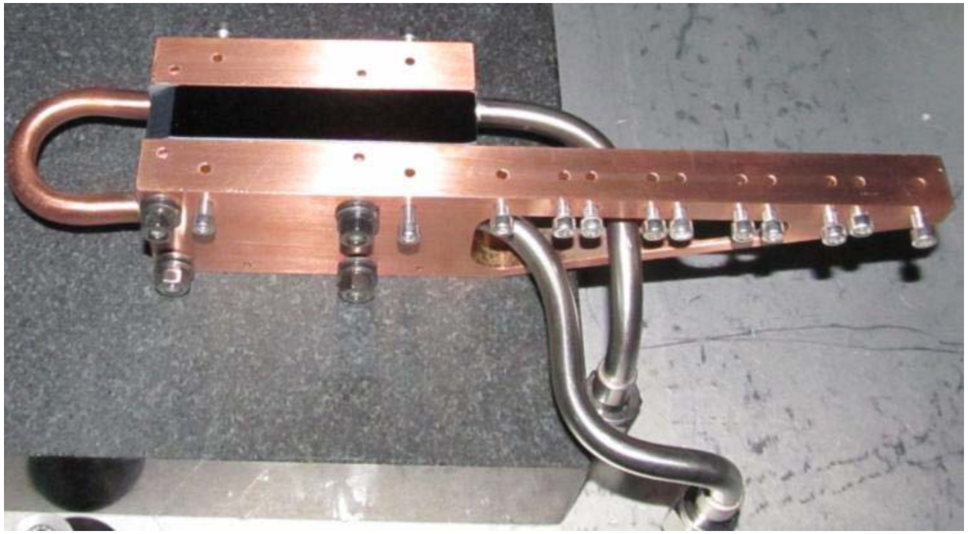
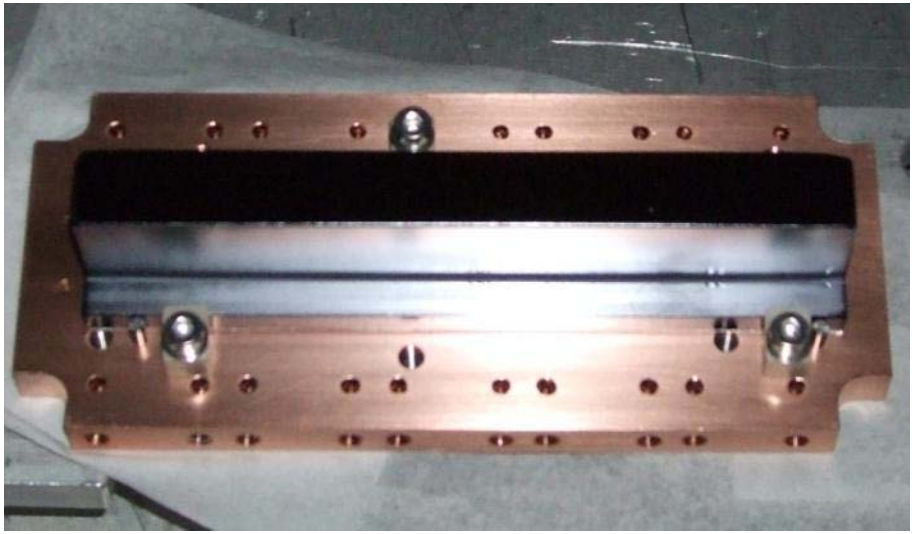
1a) I09 DCM crystals

Jon Kelly & Simon Alcock

I09 1st crystal | **I09 2nd crystal**





Side cooled

2 x clearance holes

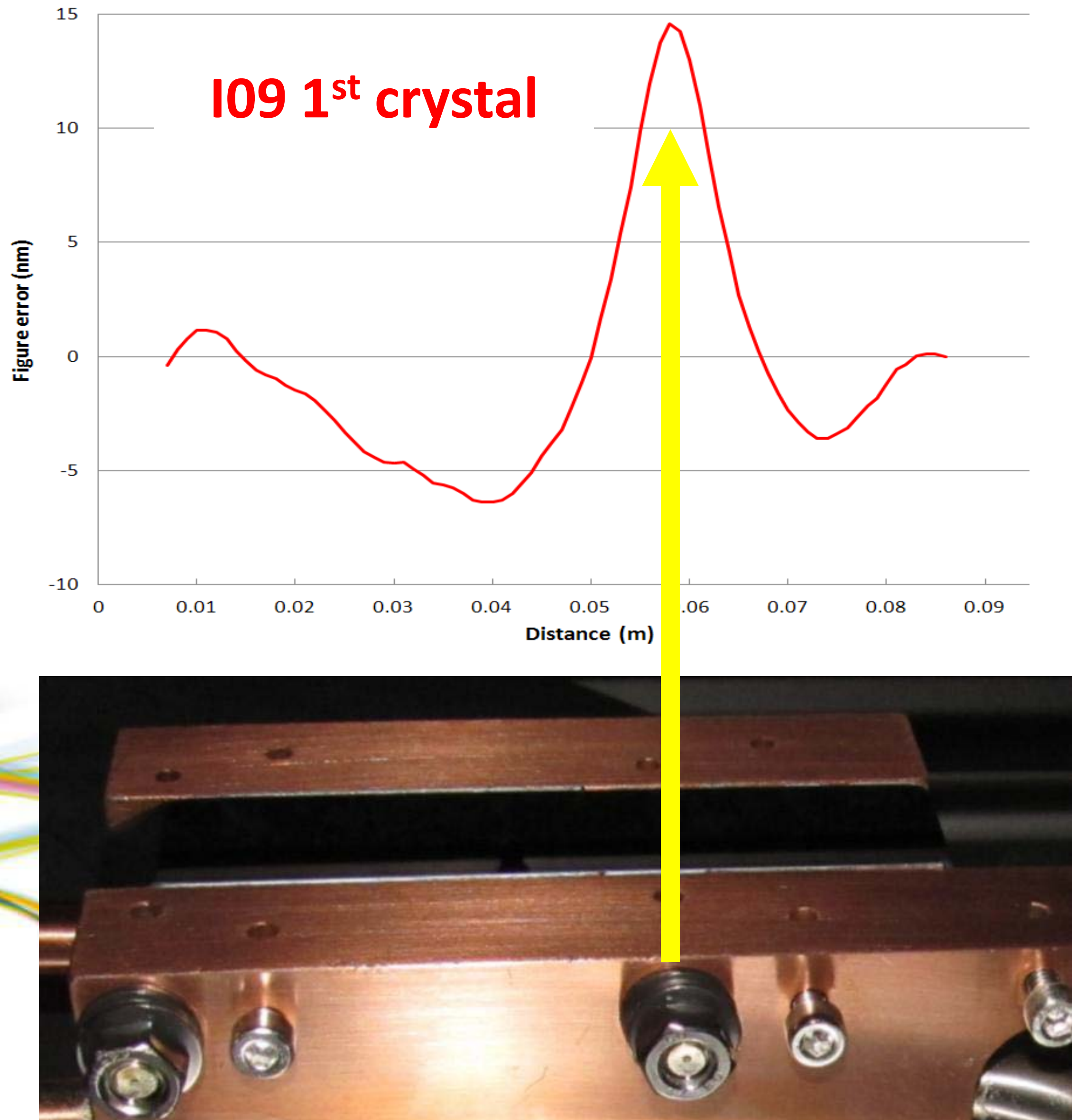
Bottom cooled

Indium pads or sheet?

Number & location of clamps?

Pressure?

Diamond-NOM metrology



"Bump" (~15nm high) directly above through hole

→ **bolt is touching silicon crystal?**

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1b) A novel apparatus to investigate deformation of cryo-cooled DCM crystals

Simon Alcock, Ioana Nistea, John Sutter, Graham Duller, Peter Docker

- Versatile chamber to accommodate a variety of DCM crystal assemblies
- Back or side cryo-cooling of crystals
- High vacuum ($<10^{-7}$mbar)
- Entrance & exit ports to view crystal surfaces
- Diagnostics (pressure, temperature)

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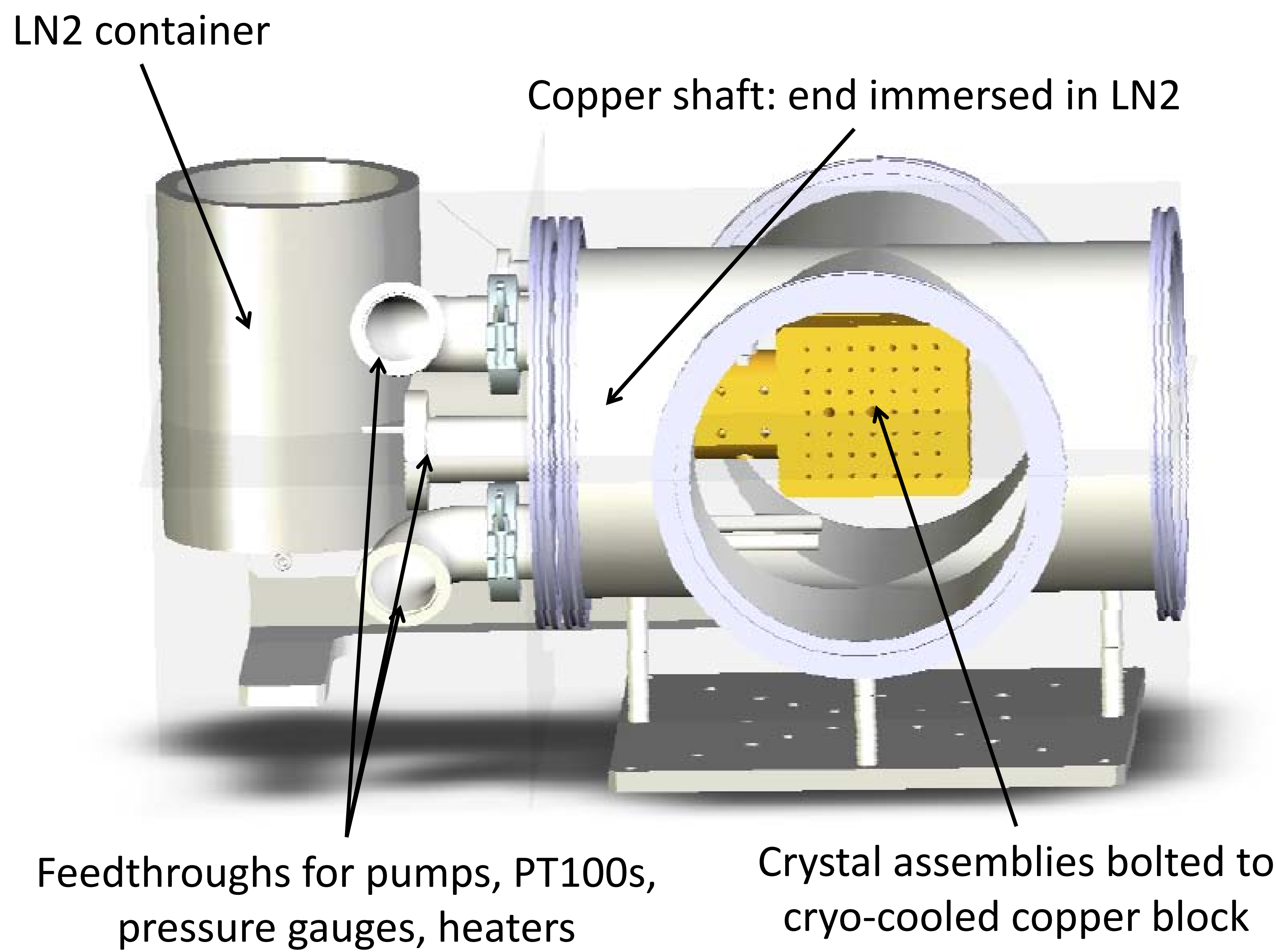
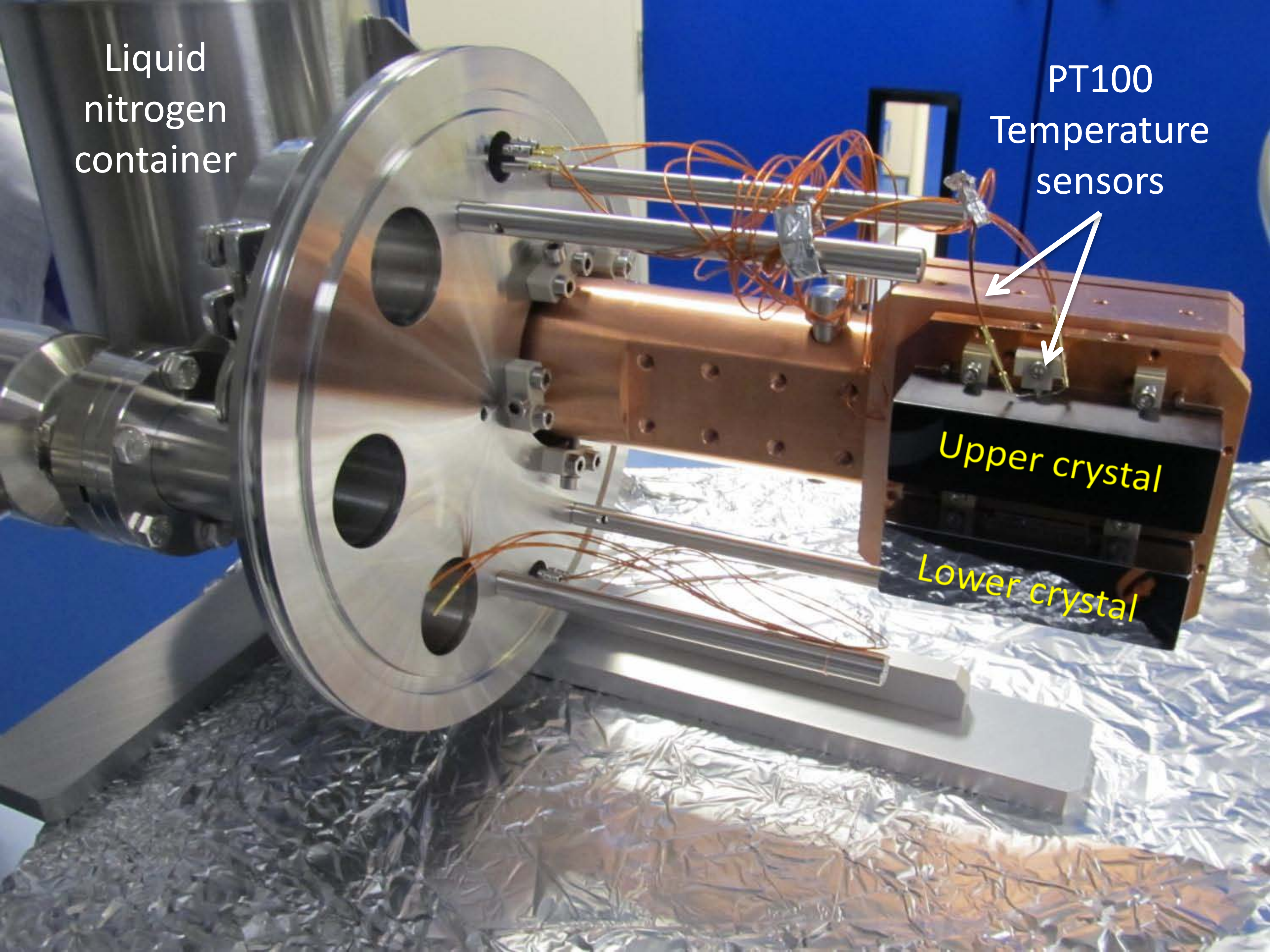
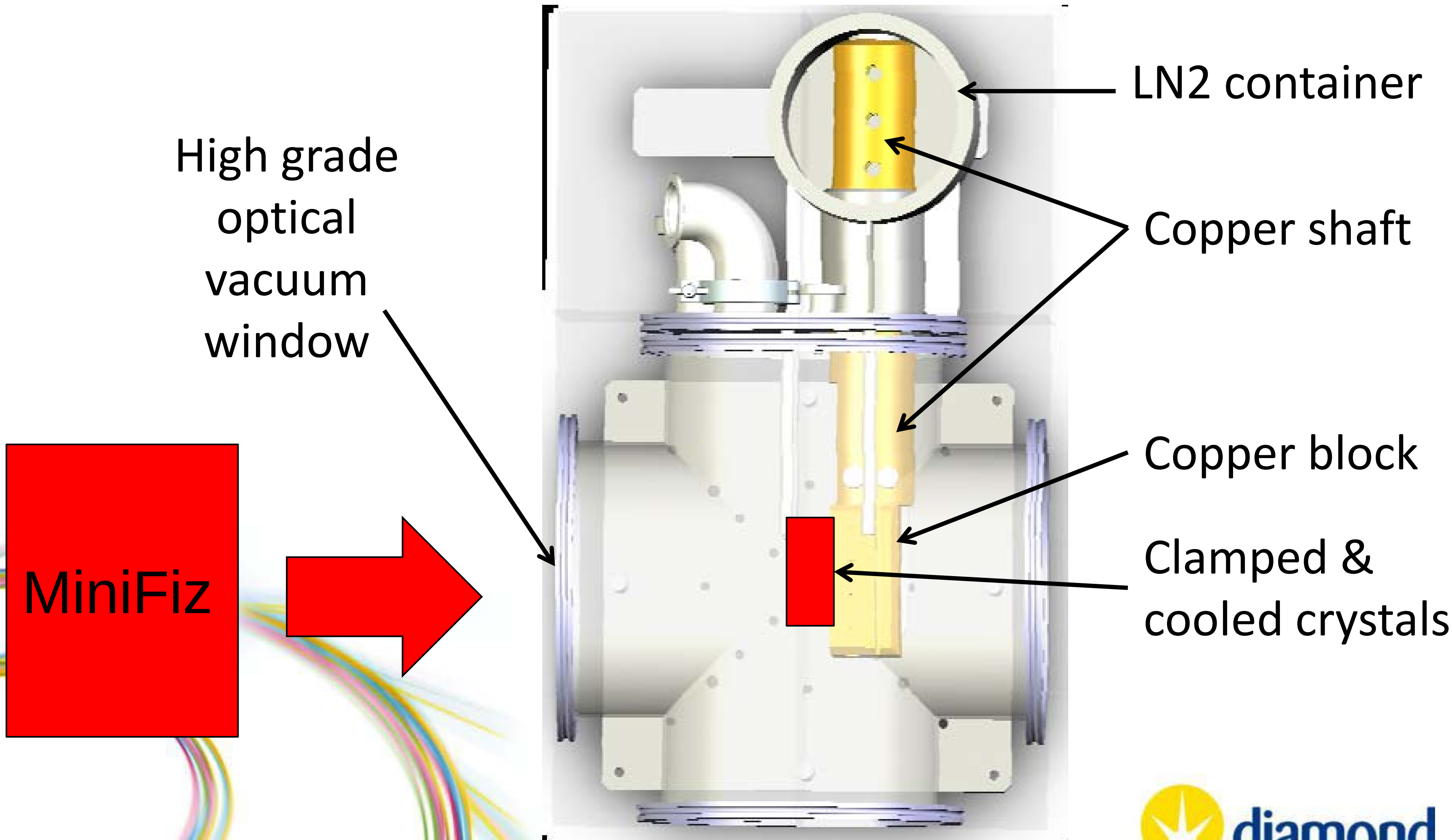

LN2 container
Copper shaft: end immersed in LN2
Feedthroughs for pumps, PT100s, pressure gauges, heaters
Crystal assemblies bolted to cryo-cooled copper block

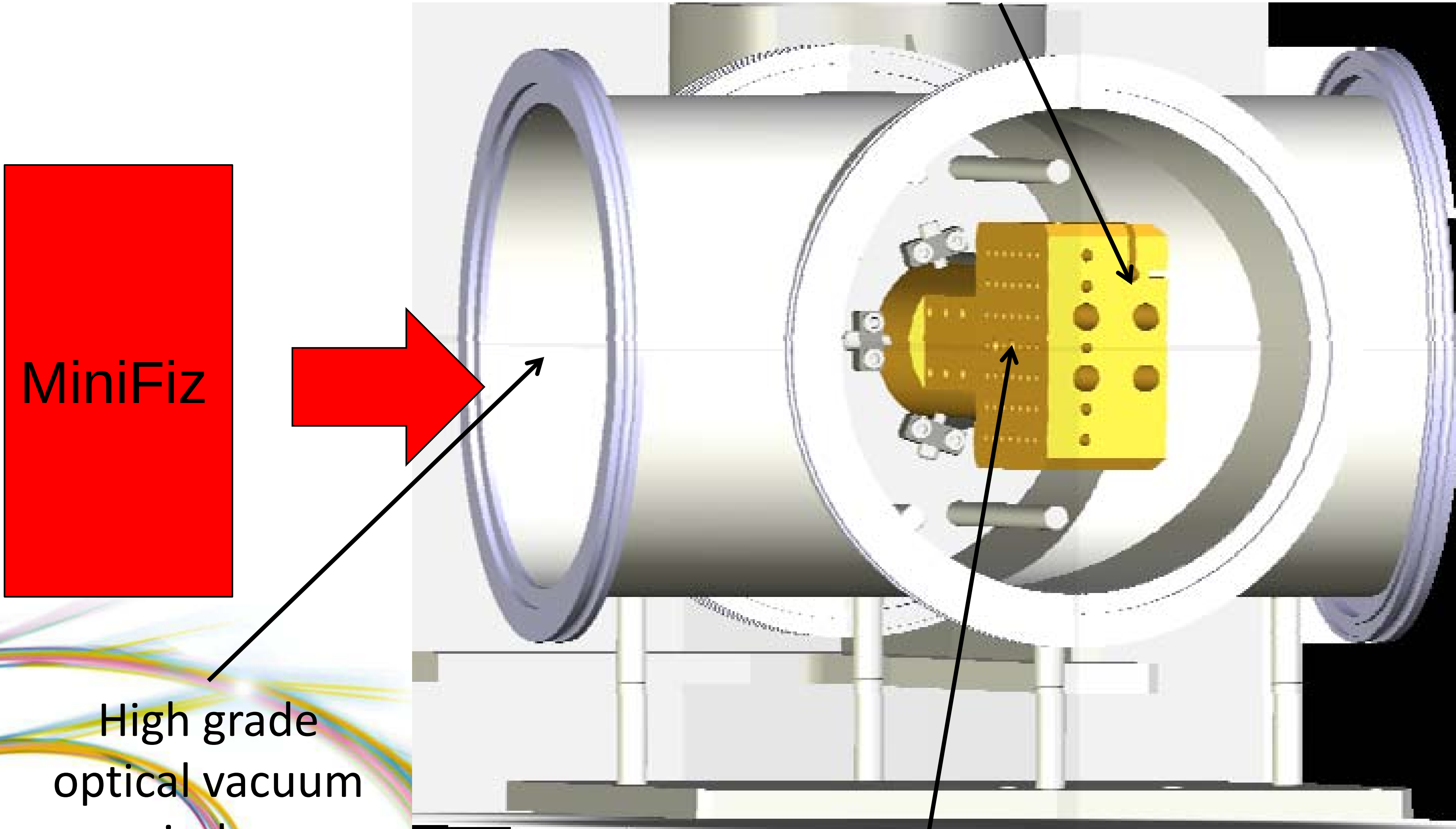

Liquid nitrogen container
PT100 Temperature sensors
Upper crystal
Lower crystal

Plan view (with transparent vacuum vessel)



Side view

Heater and thermocouples



High grade optical vacuum window

Clamped & cooled crystals

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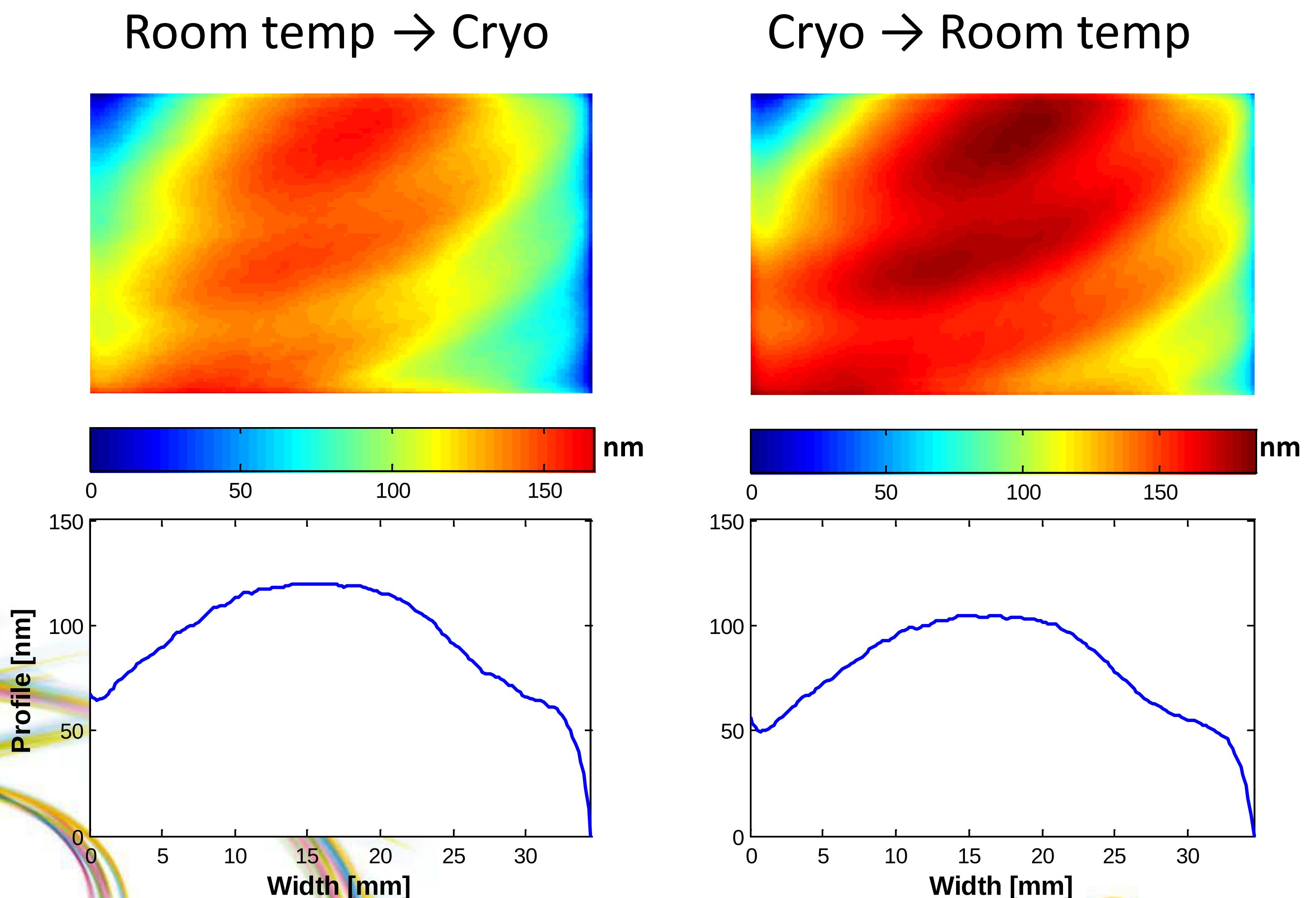
Nitrogen gas!!!
Bubbling LN2!!!
Vibrating pump cart
Fizeau beam
MiniFIZ 150

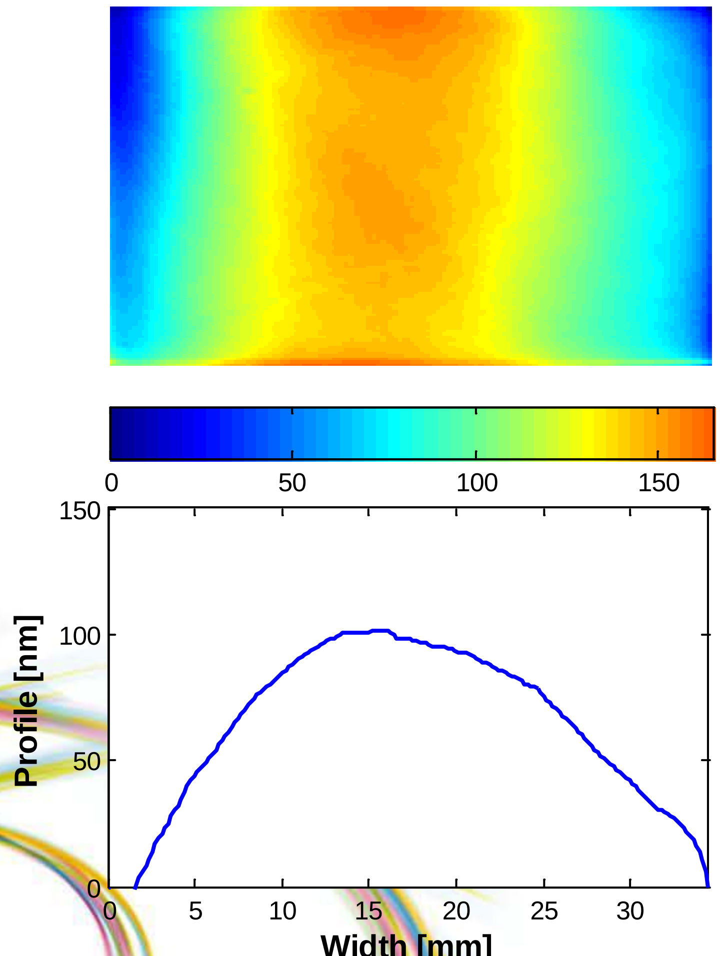
How do crystals deform on cryo-cooling?



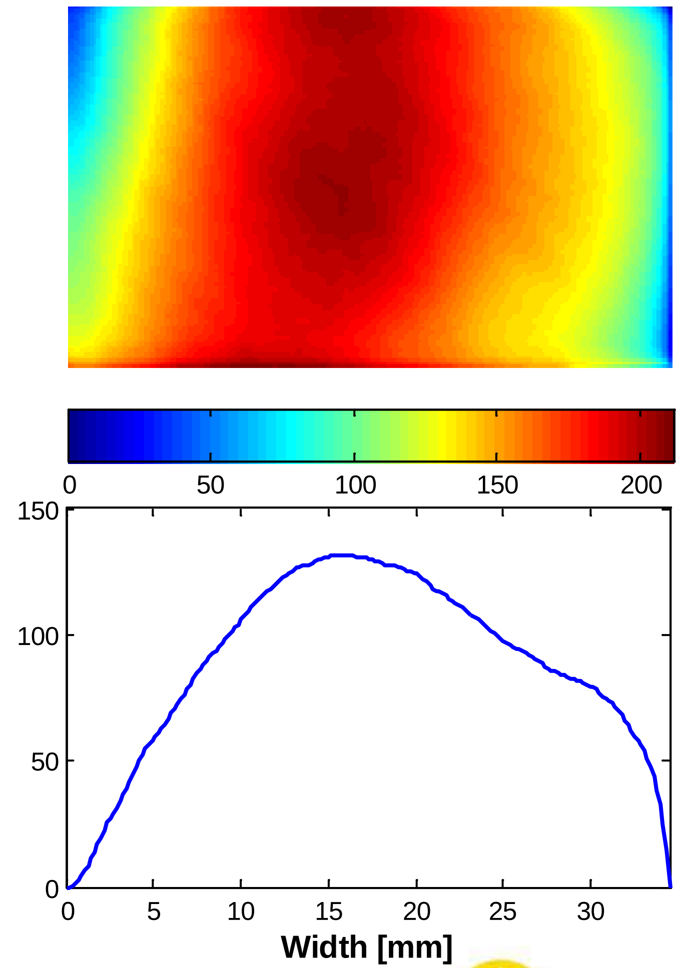
Si111 crystal

How do crystals deform on cryo-cooling?

Room temp → Cryo



Cryo → Room temp



Si311 crystal

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Fizeau interferometry

- Fizeau interferometer provides rapid (<1min) measurement of **3D topography** of entire crystal surface
- With care & skill, can acquire nm quality data in a remarkably noisy environment!!!
- Two crystals can be imaged simultaneously
- ~1 hour to cool to -196°C (& ~1 hour to warm up …using my wife's hairdryer!) → **several iterations per day** ☺
- Only provides a relative change in crystal surface (window introduces aberrations), NOT an absolute value ☹

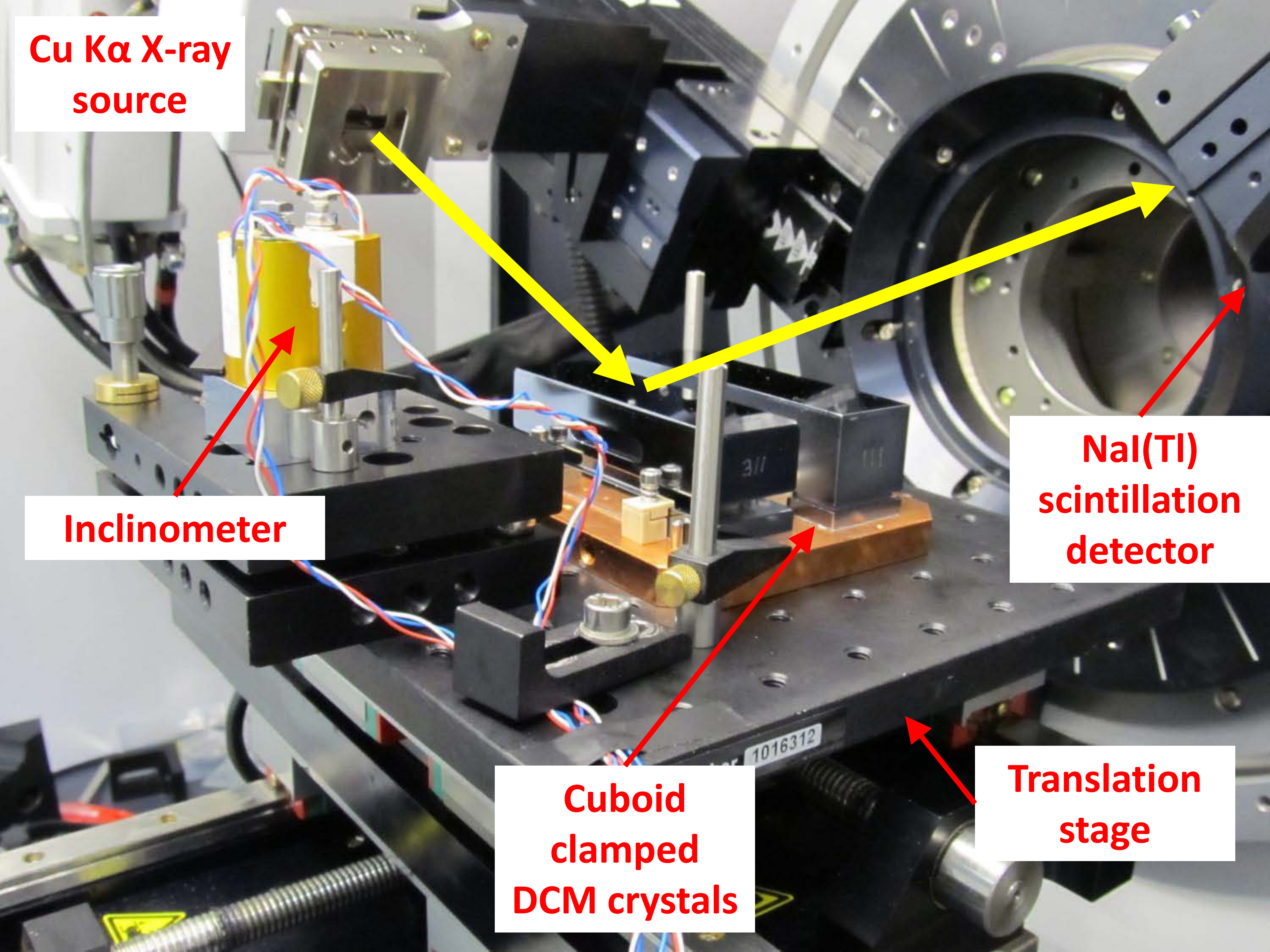
2a) Direct measurement of crystal strain

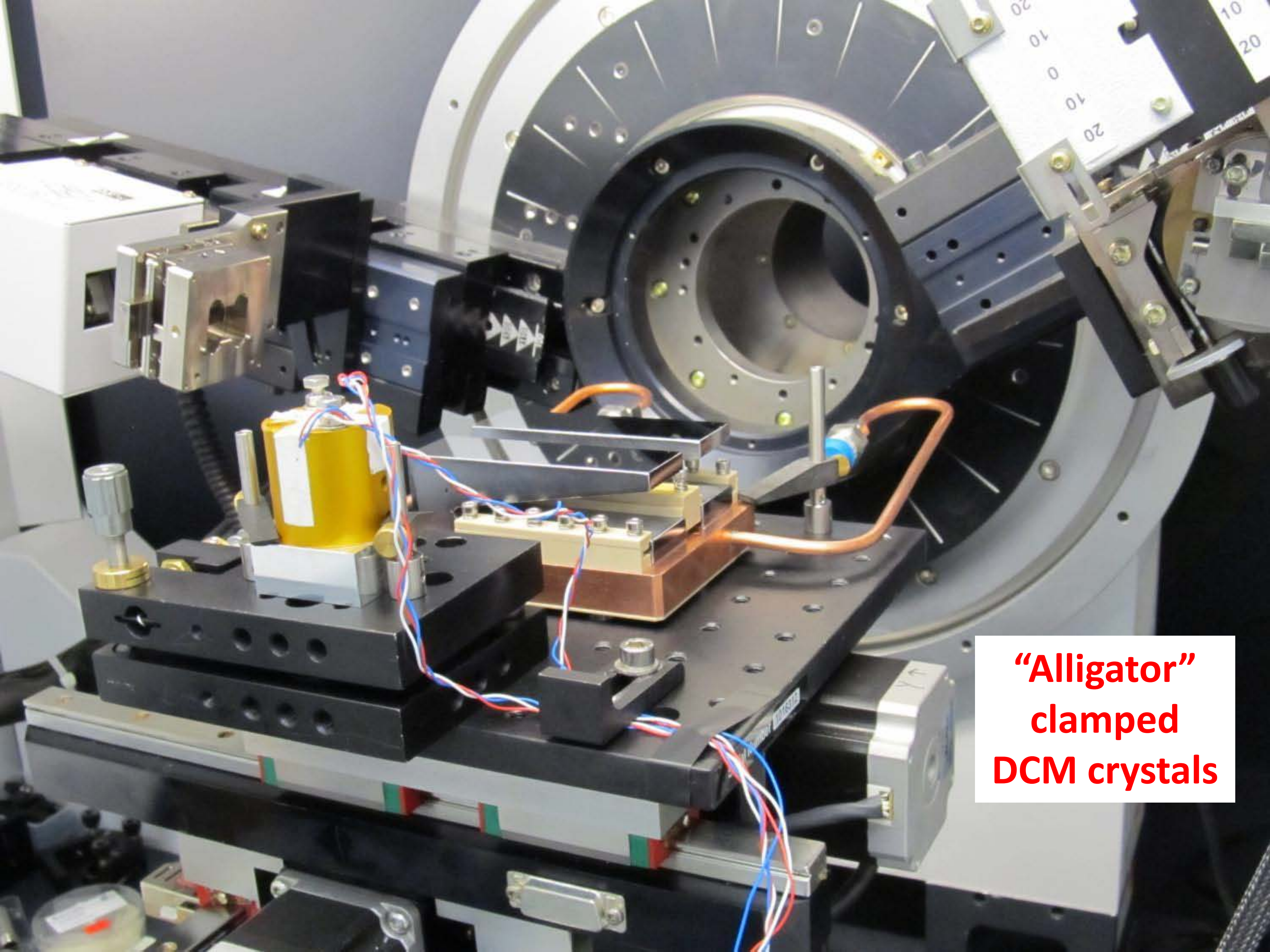
John Sutter, Hiten Patel
November 2013

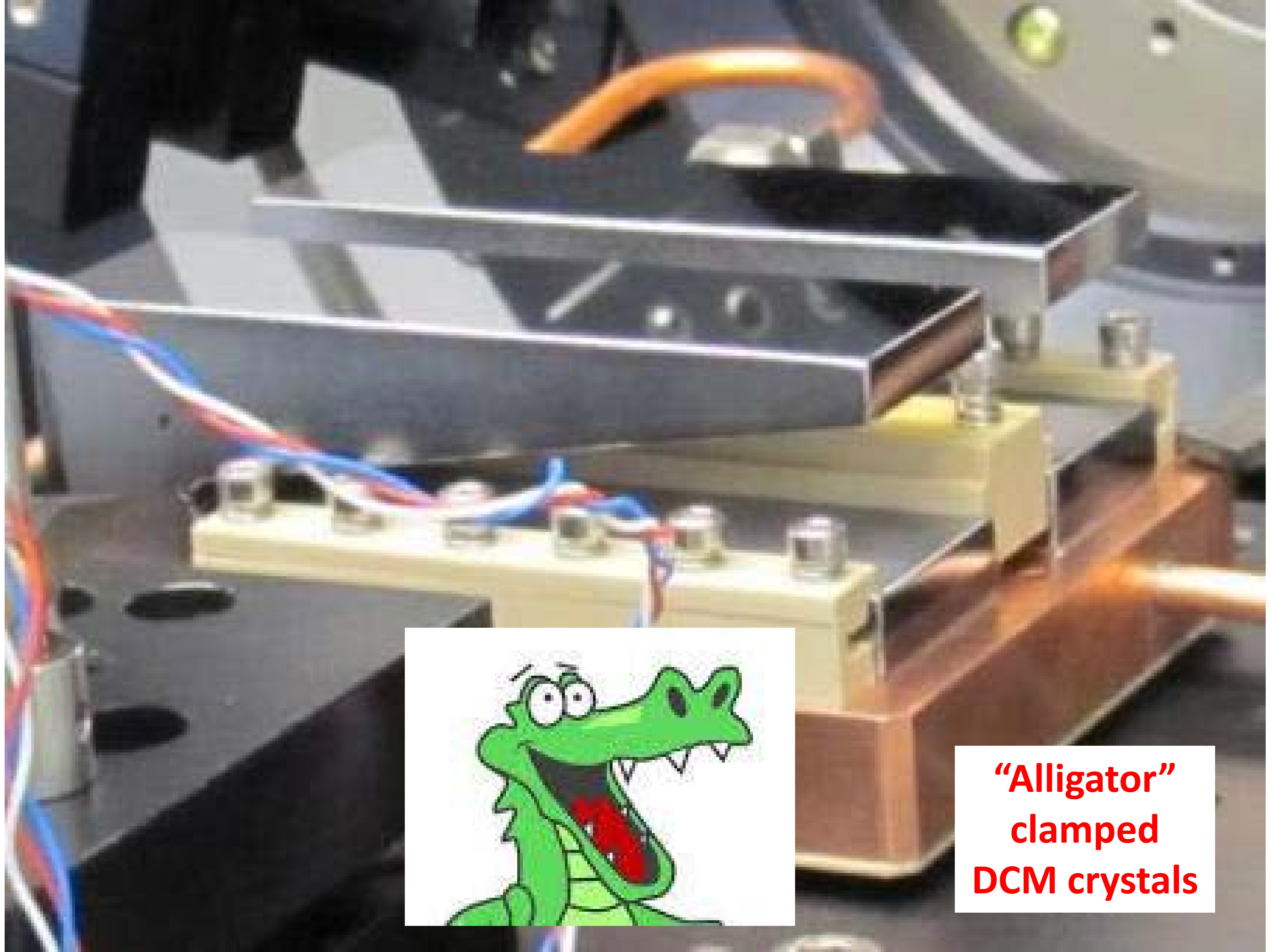
- Off-line, X-ray study of clamped DCM Si111 & 311 crystals
- Room temperature & atmospheric pressure
- Investigate : crystal shape (cuboid vs. “alligator”), clamping pressure, indium foil thickness, etc
- Measure local angle of diffraction planes at discrete positions

Bruker D8 Discover X-ray diffractometer

Cu tube (40 keV, 40 mA) + selection of optics & detectors, including:
Göbel parabolic multilayer mirror, Ge (220) symmetric channel cut,
NaI(Tl) scintillation detector +…

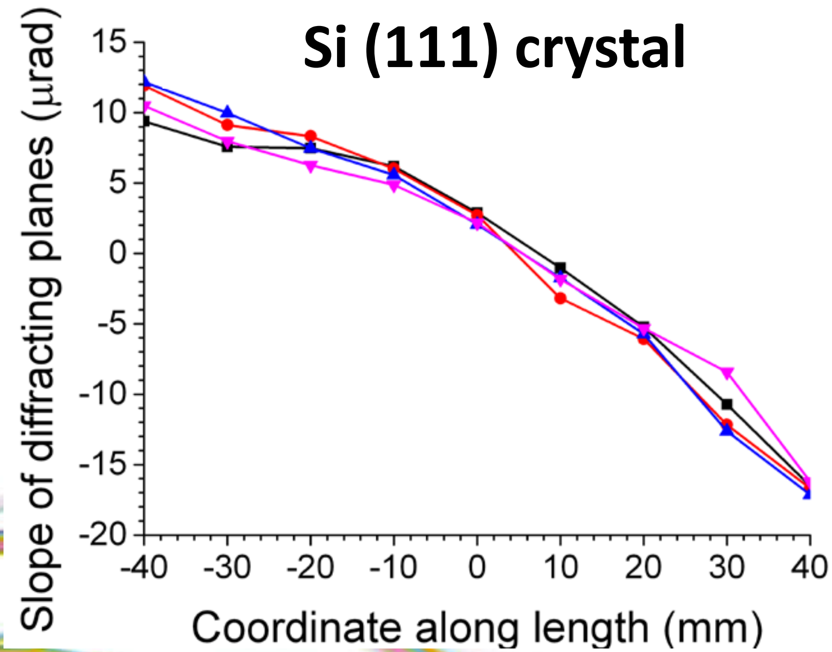

Cu Kα X-ray source
NaI(Tl) scintillation detector
Inclinometer
Cuboid clamped DCM crystals
Translation stage
1016312

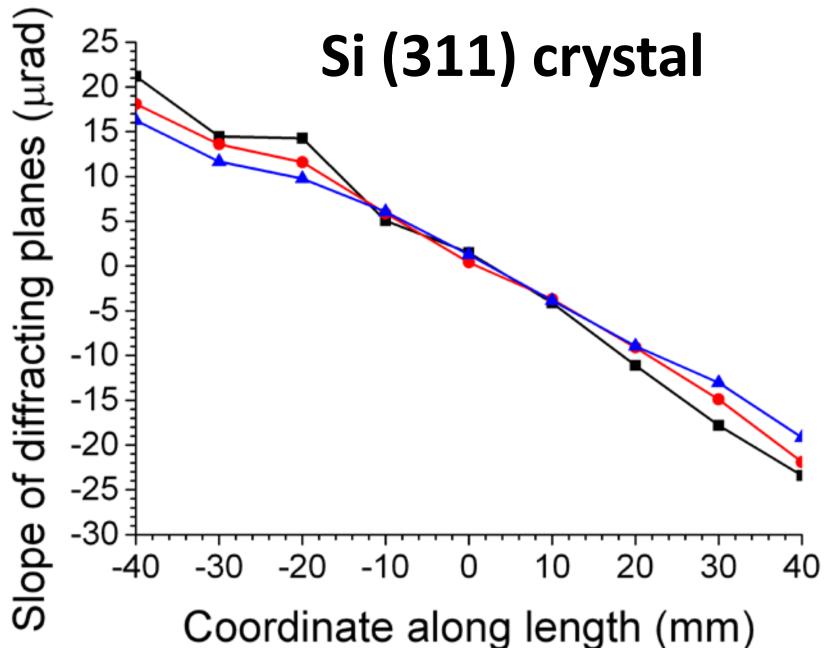

“Alligator” clamped DCM crystals


"Alligator"
clamped
DCM crystals

Cuboid crystals

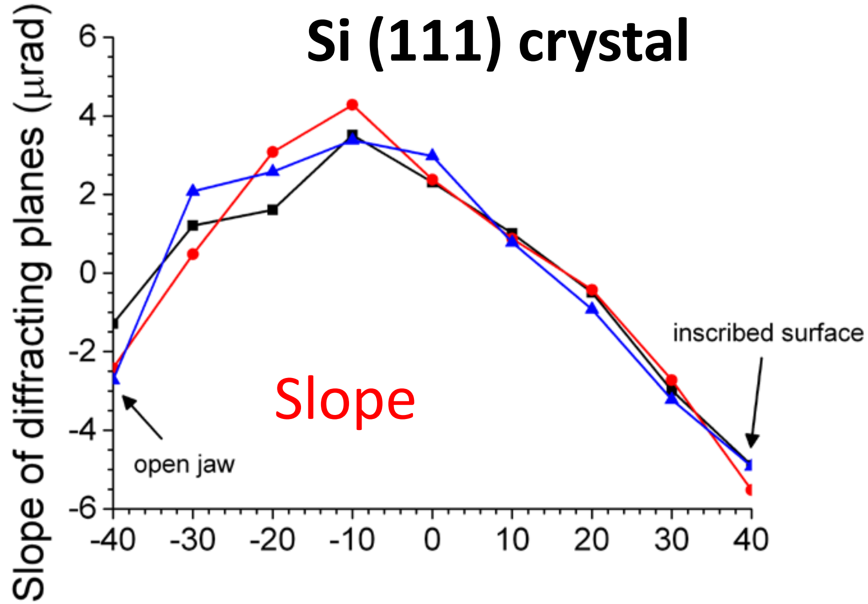
- Measure local slope of crystal planes along length of crystals
- Inclinometer used to monitor parasitic pitch / roll of translation

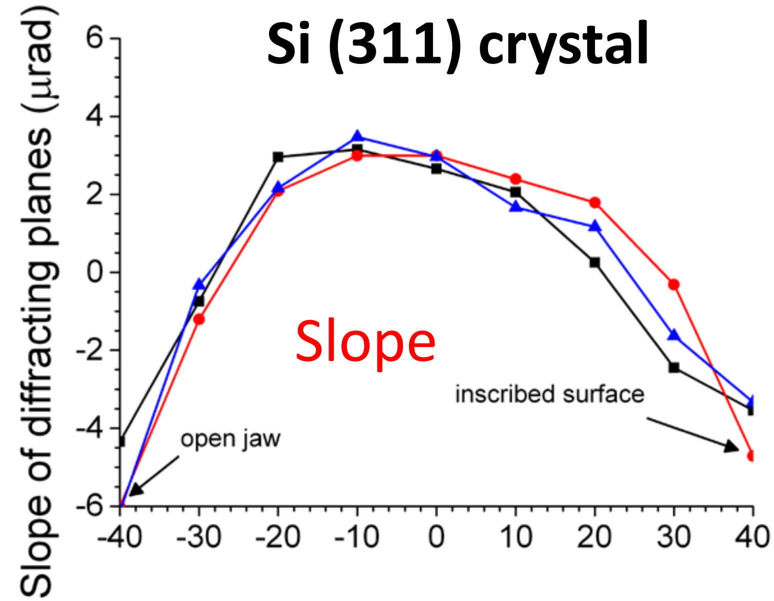


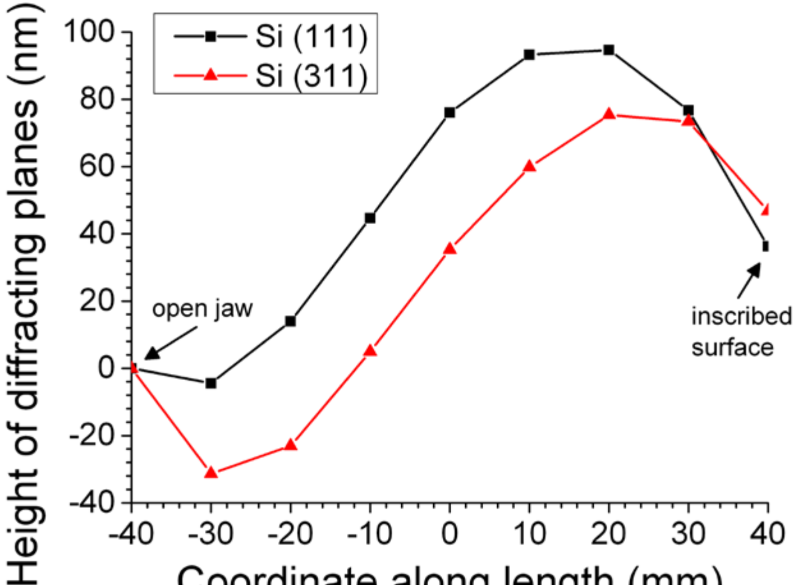


Curvature of diffracting planes is **convex (2 & 3km)**

"Alligator" crystals







- S-shape distortion
- Slope variation for "Alligator" crystals (~6urad PV) is much less than cuboid crystals (30 - 40urad PV)

2b) Direct measurement of crystal strain

John Sutter, Peter Docker, Steve Keylock,
Mónica Amboage, Sofía Díaz-Moreno

November 2013

- **B16 Test beamline** using attenuated white beam
- Crystal clamping chamber modified for X-rays (Kapton windows)
- I20 Si111 crystals with 250 µm thick indium foil
- Clamping pressure: compressed to 20 bar, then released to 1 bar
- Low vacuum, cryo-cooling
- 18.9keV selected by crystals

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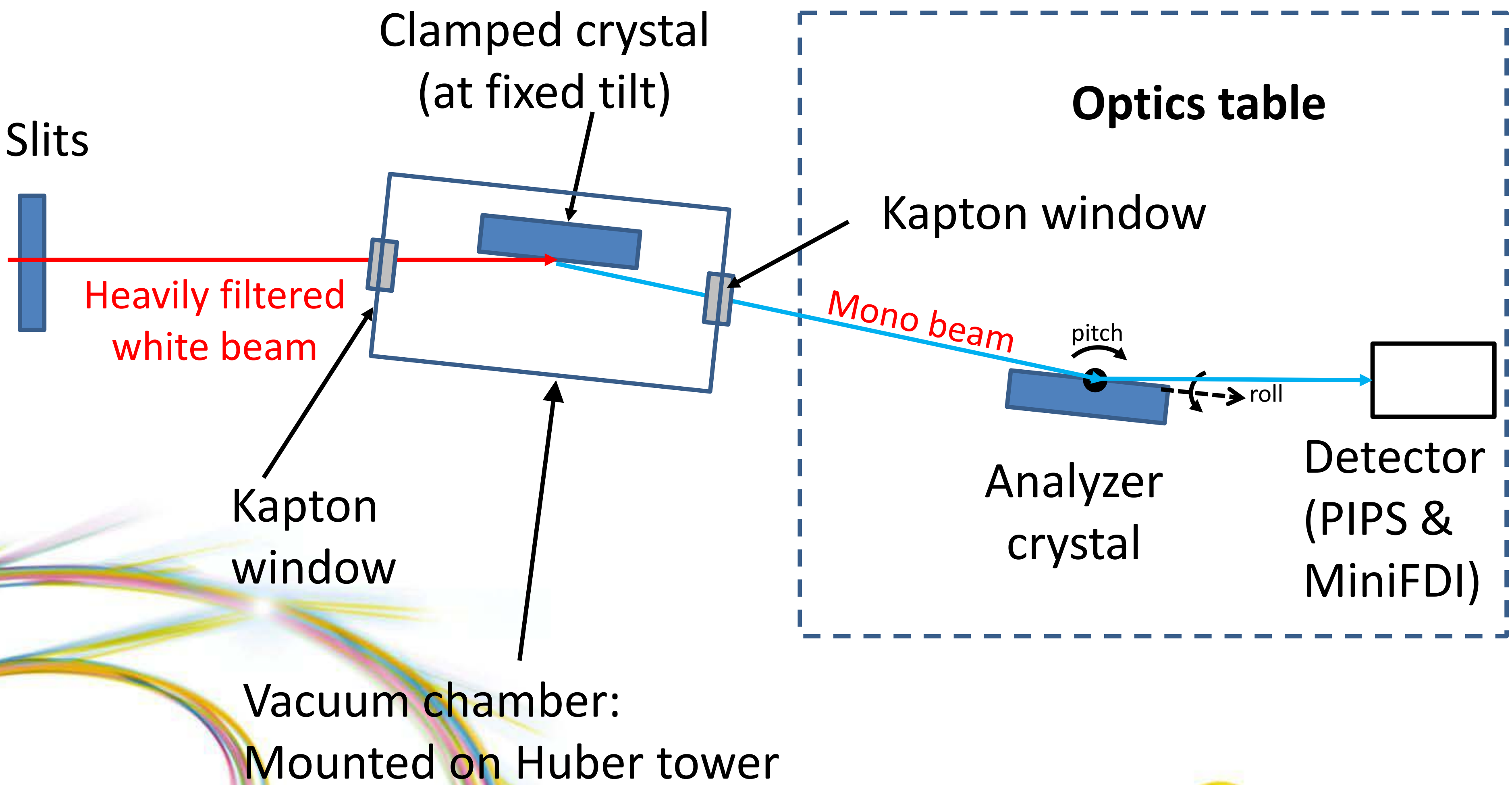
Plan view of B16 set-up



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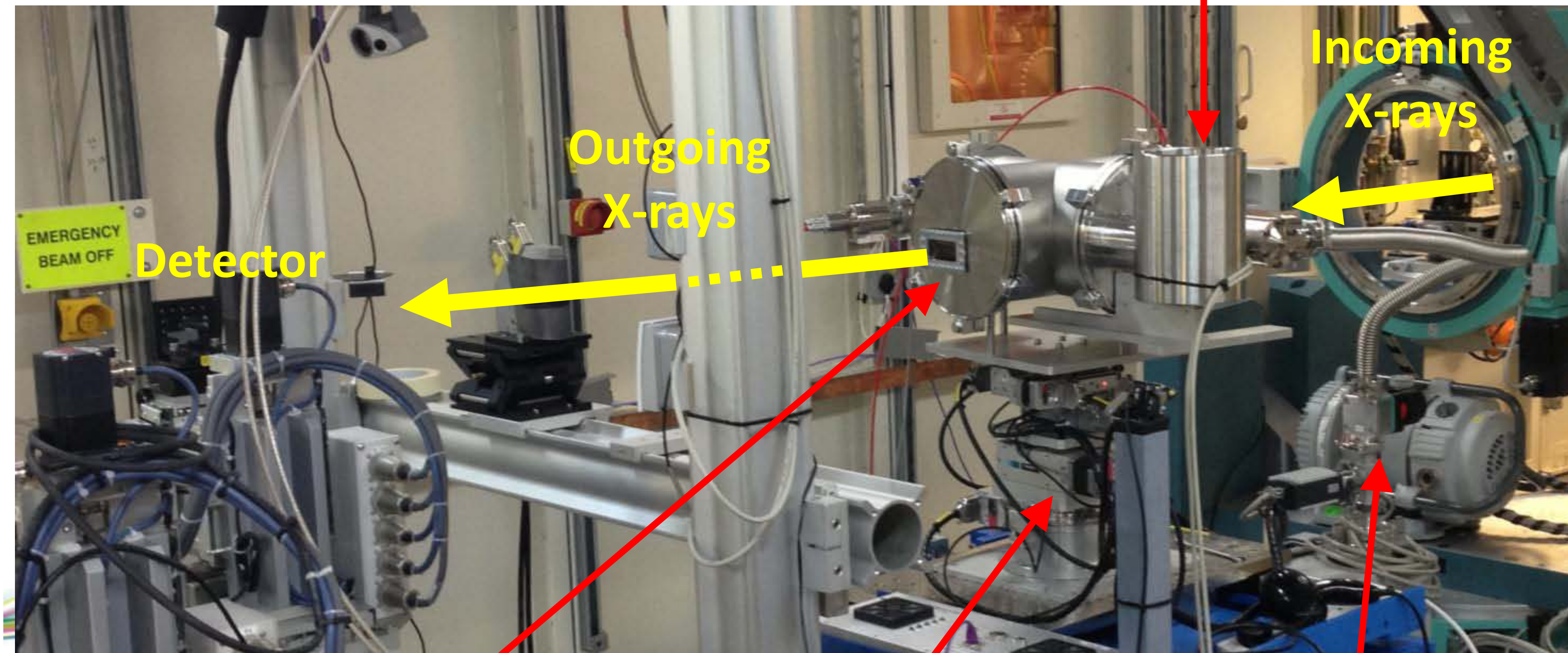
B16 set-up



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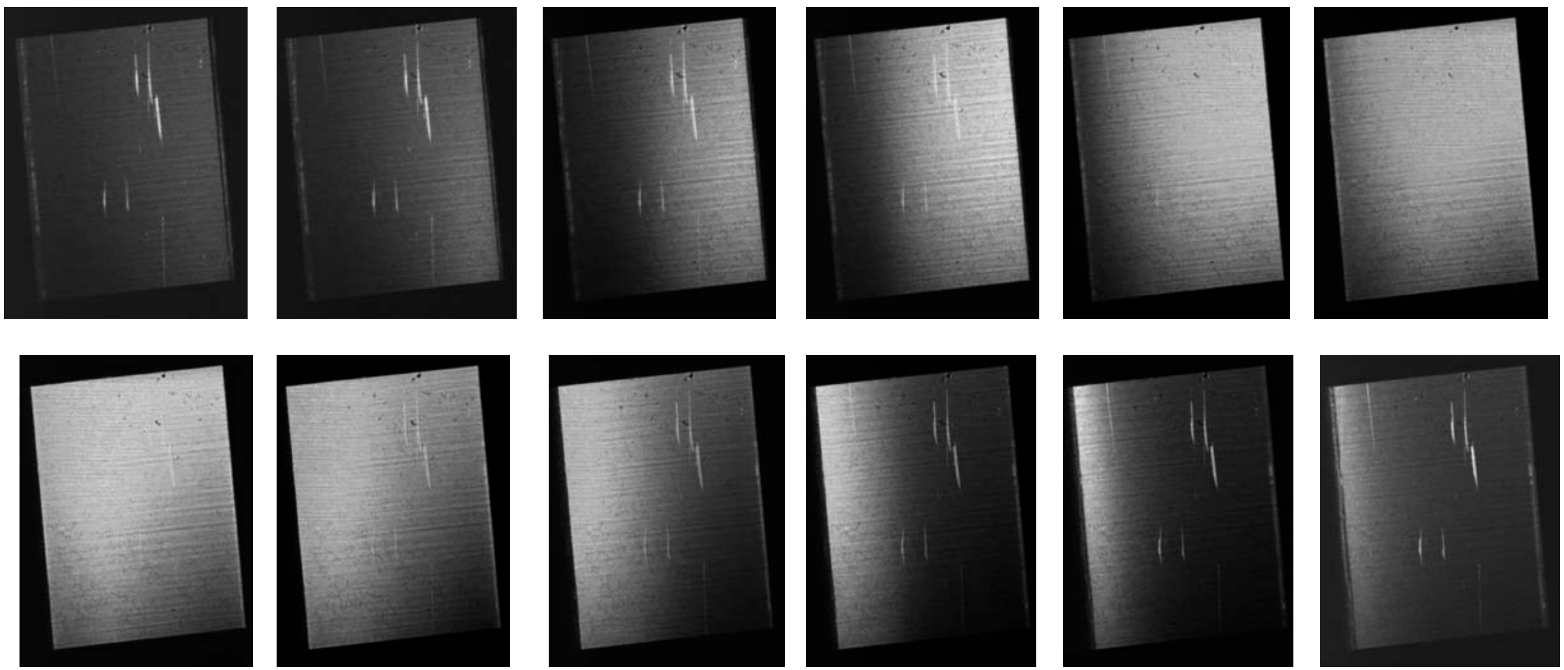
Double-crystal topographs: diffracted beam imaged as analyzer pitch is stepped through Bragg reflection

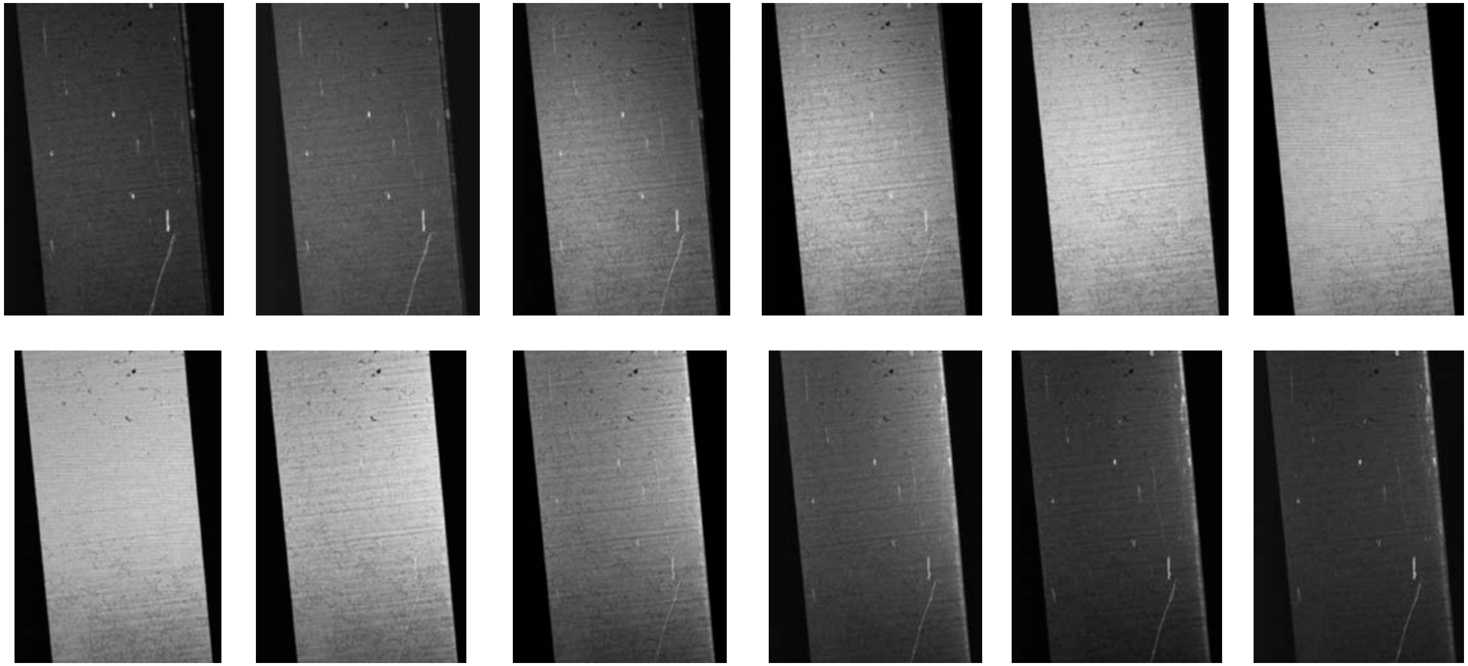
Perfect lattice matching → uniform diffracted beam
Imperfect lattice matching → diffracted beam has bright/dark regions that move with anaylzer pitch

Crystal cryo-cooled (−190°C)



1 mm Al filtering, analyzer pitch step 0.250 millidegrees

Crystal warmed to +32°C



Note that beam is much more uniform!

→ Crystal was significantly strained under cryo-cooling, but relaxed when warmed

After X-ray tests, crystal alignment checked by laser:
Δ roll ~1.0 mrad & Δpitch ~0.6 mrad

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Section 2: DCM mechanics

- Procured DCMs had problems, particularly with vibrations

  → liaise with suppliers to improve (I07, I13, B18, B21, etc)
  → upgrade programme (I18 & I22 Andy Peach *et al*)
  → **in-house development** of DCMs (I09 & I23 Jon Kelly *et al*)

- Metrology feedback essential to understand the nature of problems & quantify effectiveness of repairs / upgrade

Precision Metrology

- Precision Metrology performed in new Precision Metrology Lab or beamline

- Autcollimators, interferometers, vibration sensors, measure:
  - Parallelism between crystals
  - Angular stability
  - Parasitic pitch & roll errors during Bragg rotation
  - Linearity, repeatability & positional errors
  - Stiffness of mechanics (gravity sagging / unbalanced)
  - Vibration spectra of major DCM components
  - Dynamic (vibration & drifts) changes during cooling
  - Diagnose errors with Motion Controls systems
  - + …

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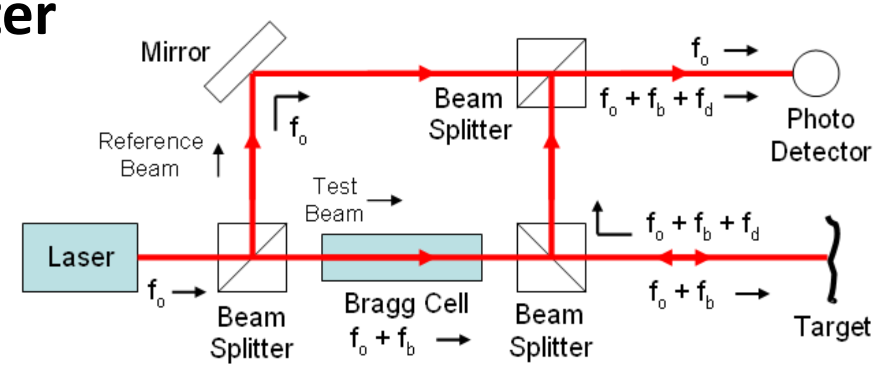
Precision Metrology instruments

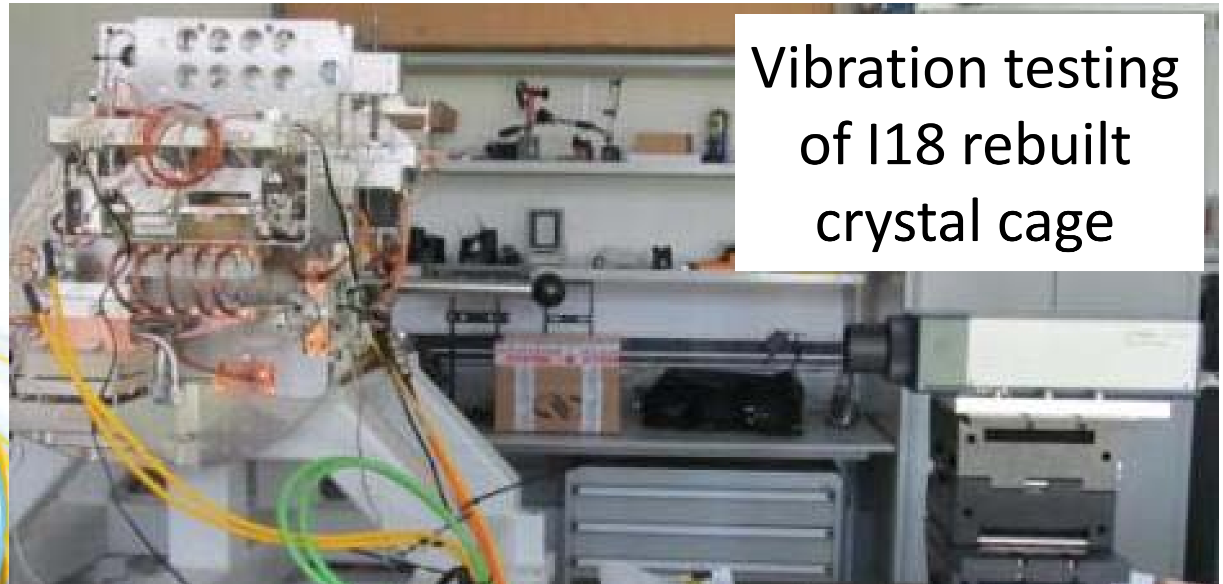
- **2 x "Elcomat 3000" autocollimators** — **Angular**
  - Angular range ±10,000µrad
  - Angular resolution <0.05µrad
- **Laser & position detector** — **Lateral displacement**
  - Lateral resolution ~1µm
- **"XL80" (DMI) & RLE interferometer** — **Linear + Angular**
  - Linear range >10m
  - Linear resolution ~ 10nm
- **Heidenhain gauges** — **Linear** (contact)
  - Linear range 25mm
  - Accuracy ±200nm
- **Capacitive sensors** — **Linear** (non-contact)
  - Linear range 100's um
  - Linear resolution <1nm
- **Polytec vibration sensor** — **Vibration**
  - Doppler shift (1Hz to kHz)

Non-contact, vibration sensing

Polytec (Doppler shifting) vibrometer

- linear vibrations
- **<1 nm** resolution
- **10 kHz** max acquisition rate
- Reflective surface not required
- Can measure **through** vacuum port!





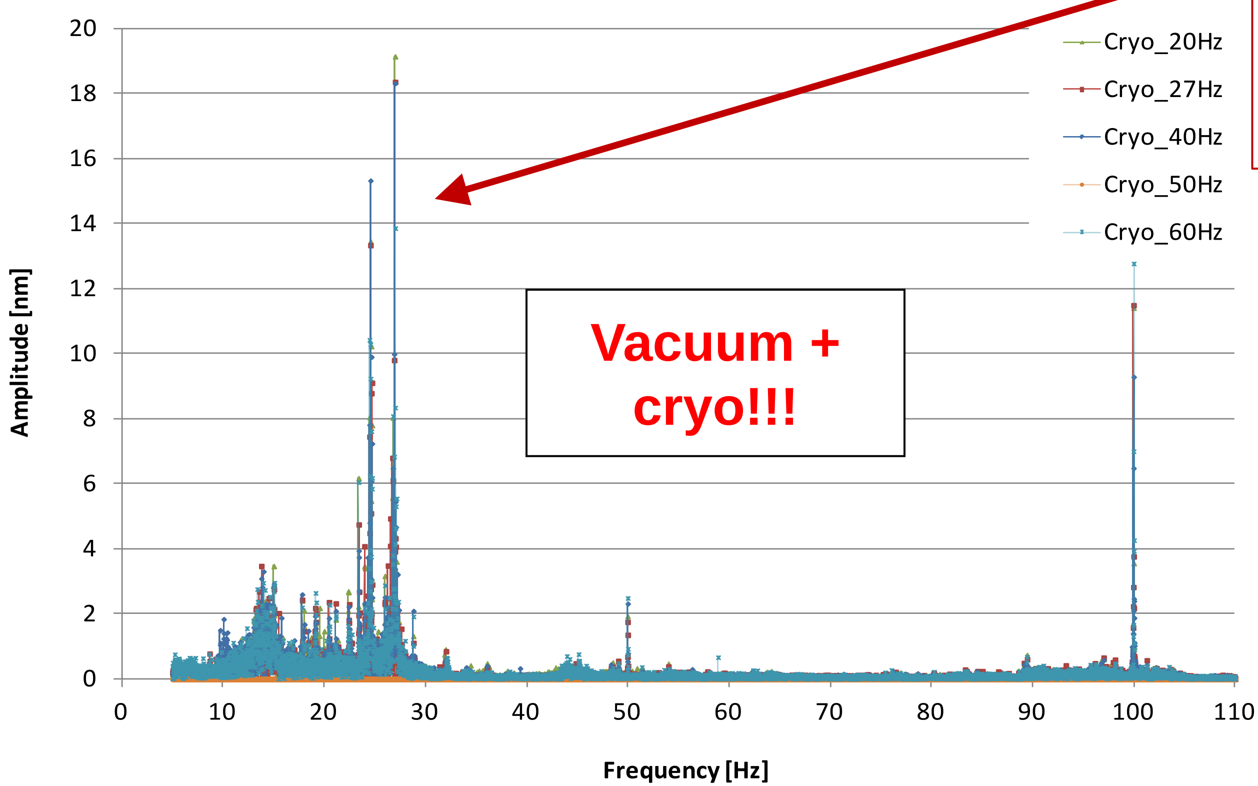
Vibration testing of I18 rebuilt crystal cage

I23 DCM vibrations

Jon Kelly, Hiten Patel, Alex Bugnar

Investigate vibrations at different coolant flow rates

Tapping vessel confirmed 27Hz peak (& higher harmonic at 53Hz) is natural frequency of DCM



- No significant effect on vibration spectra by adjusting cryo-cooler (20 – 60Hz)

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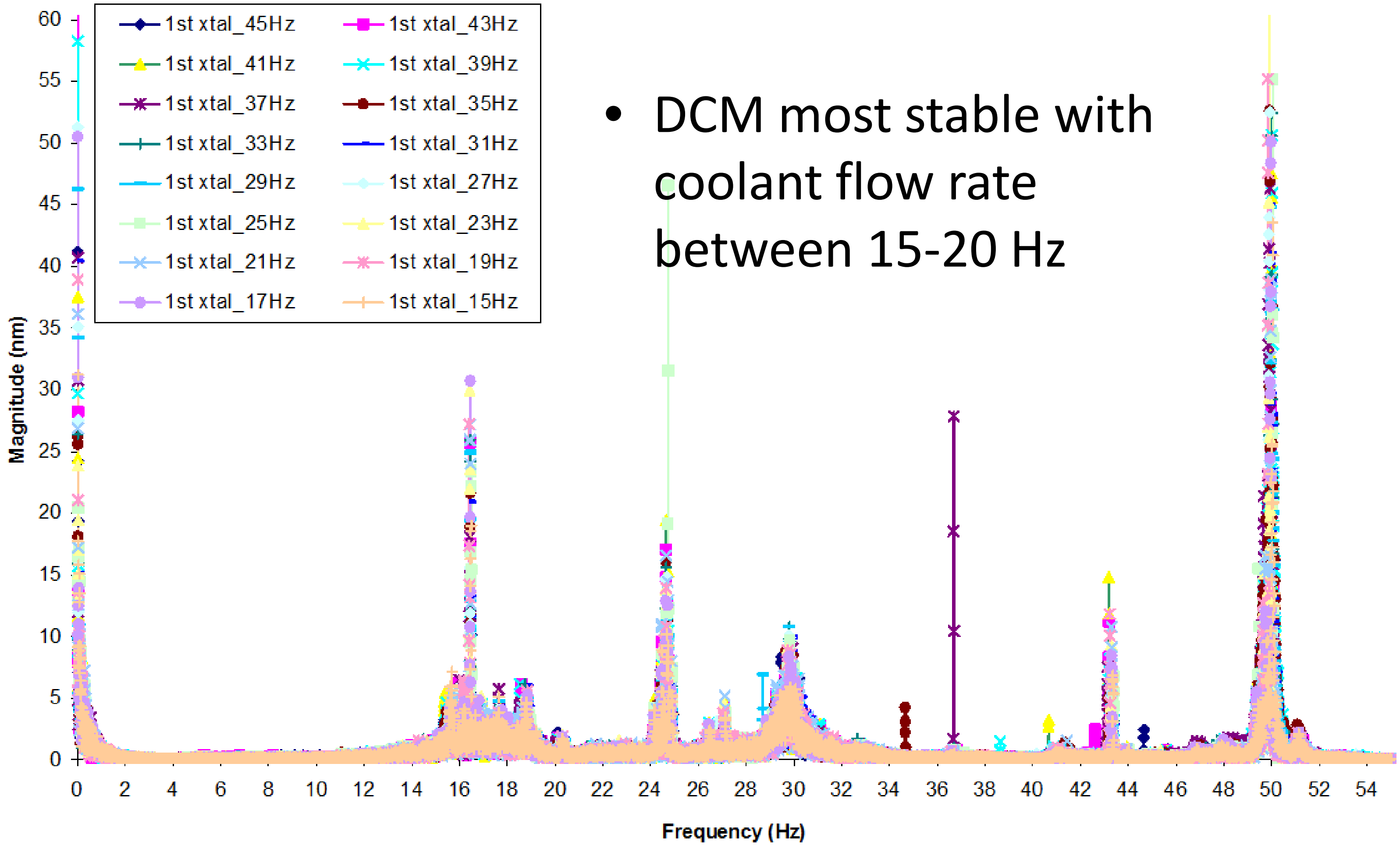
I07 DCM vibrations

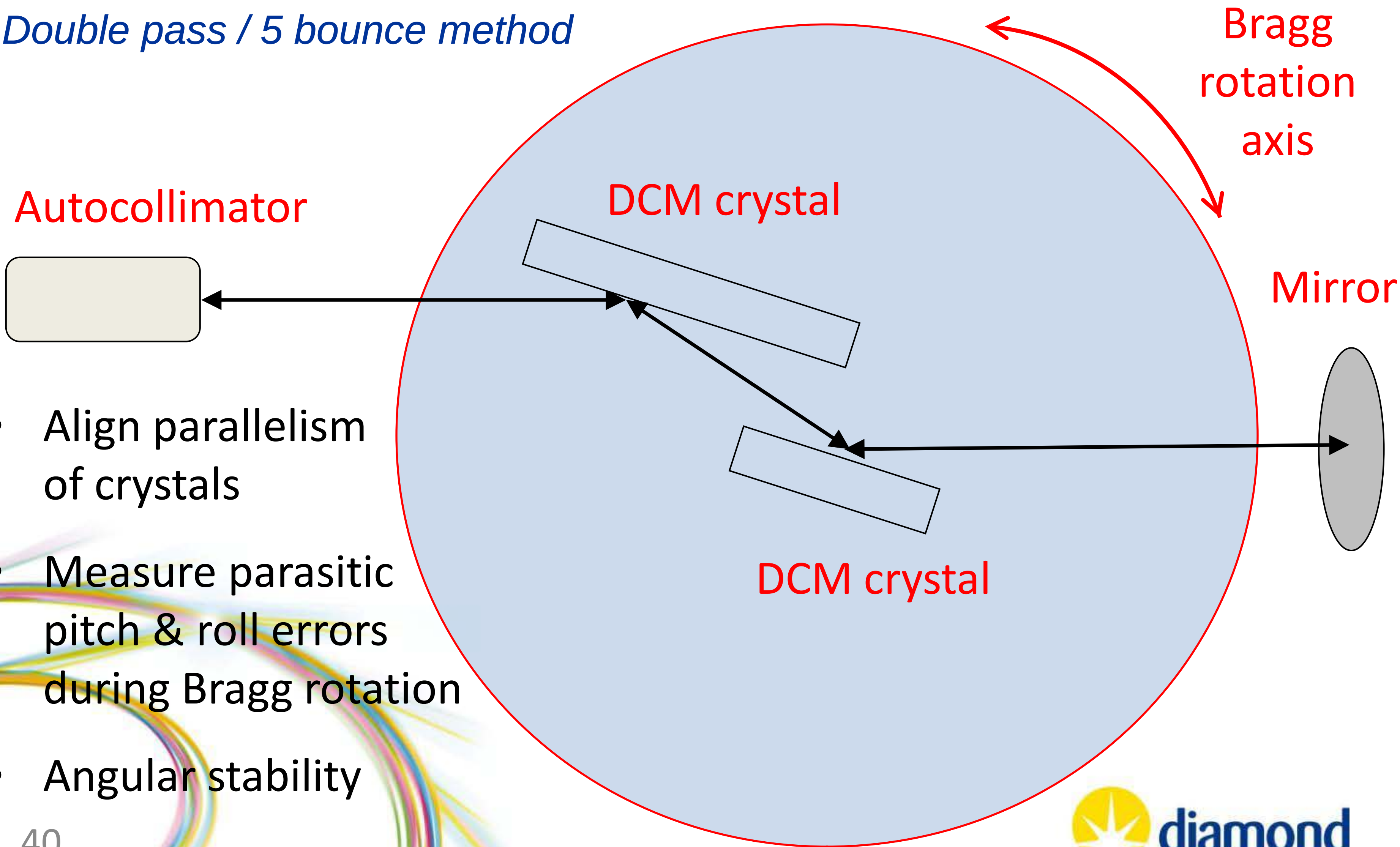
Hiten Patel, Alex Bugnar



- DCM most stable with coolant flow rate between 15-20 Hz

Crystal alignment vs. Bragg rotation

Double pass / 5 bounce method



- Align parallelism of crystals
- Measure parasitic pitch & roll errors during Bragg rotation
- Angular stability

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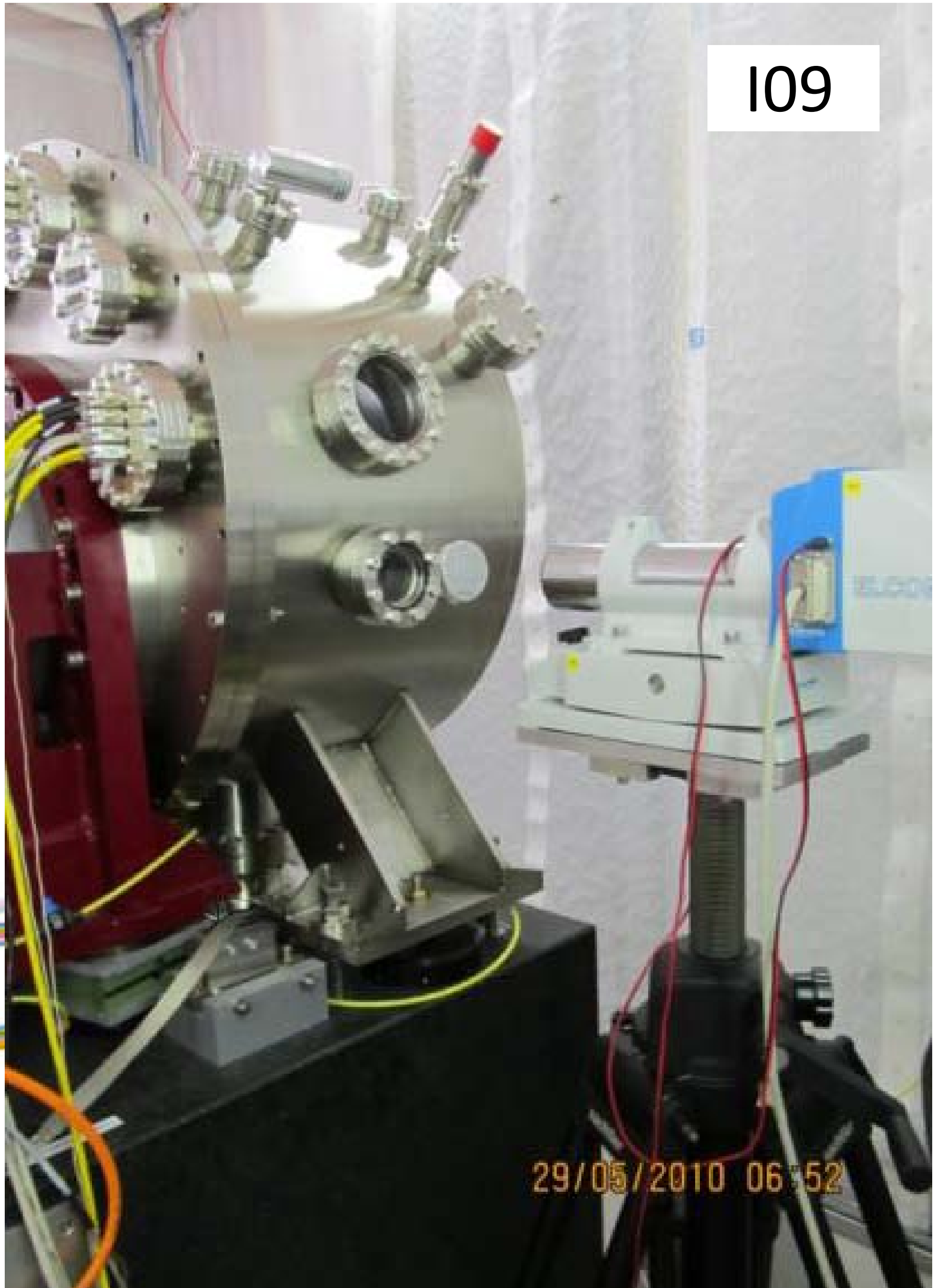
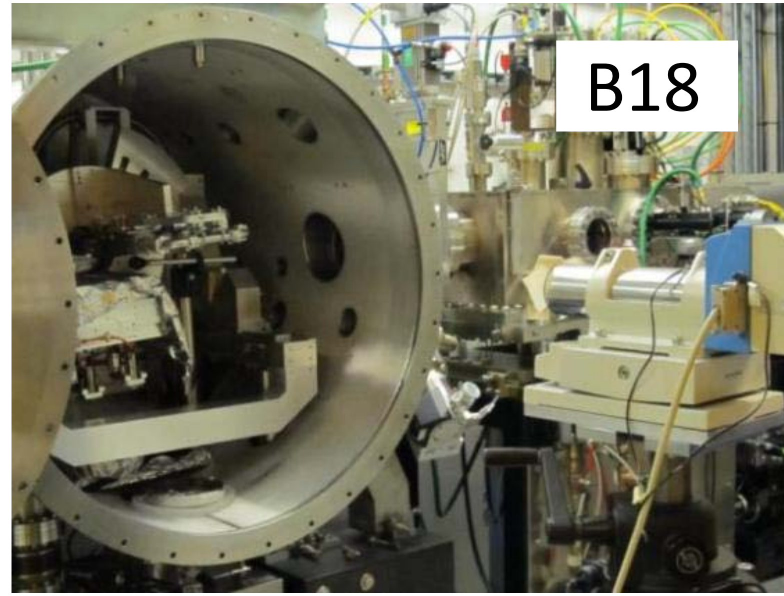
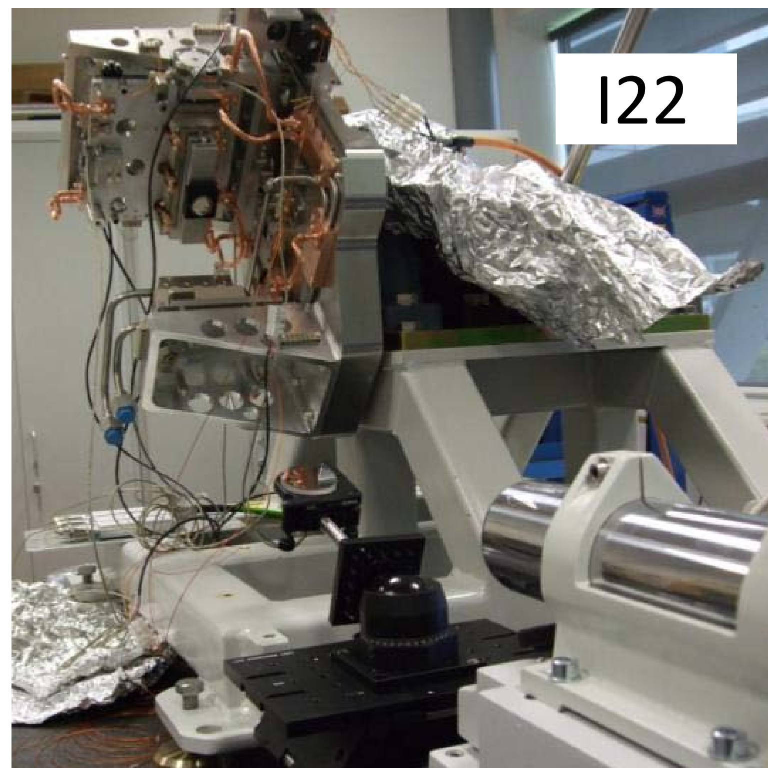
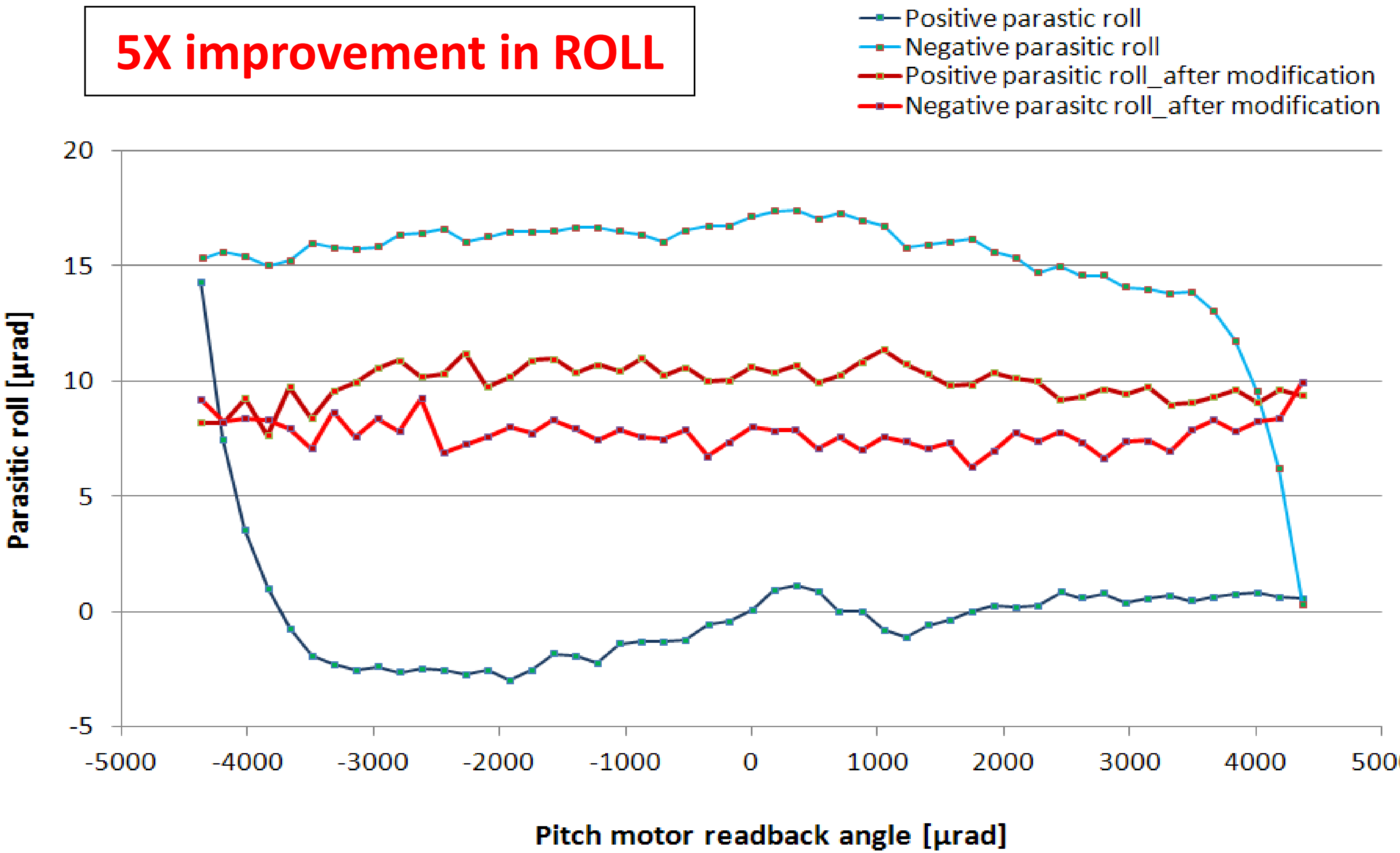

I09
29/05/2010 06:52


B18

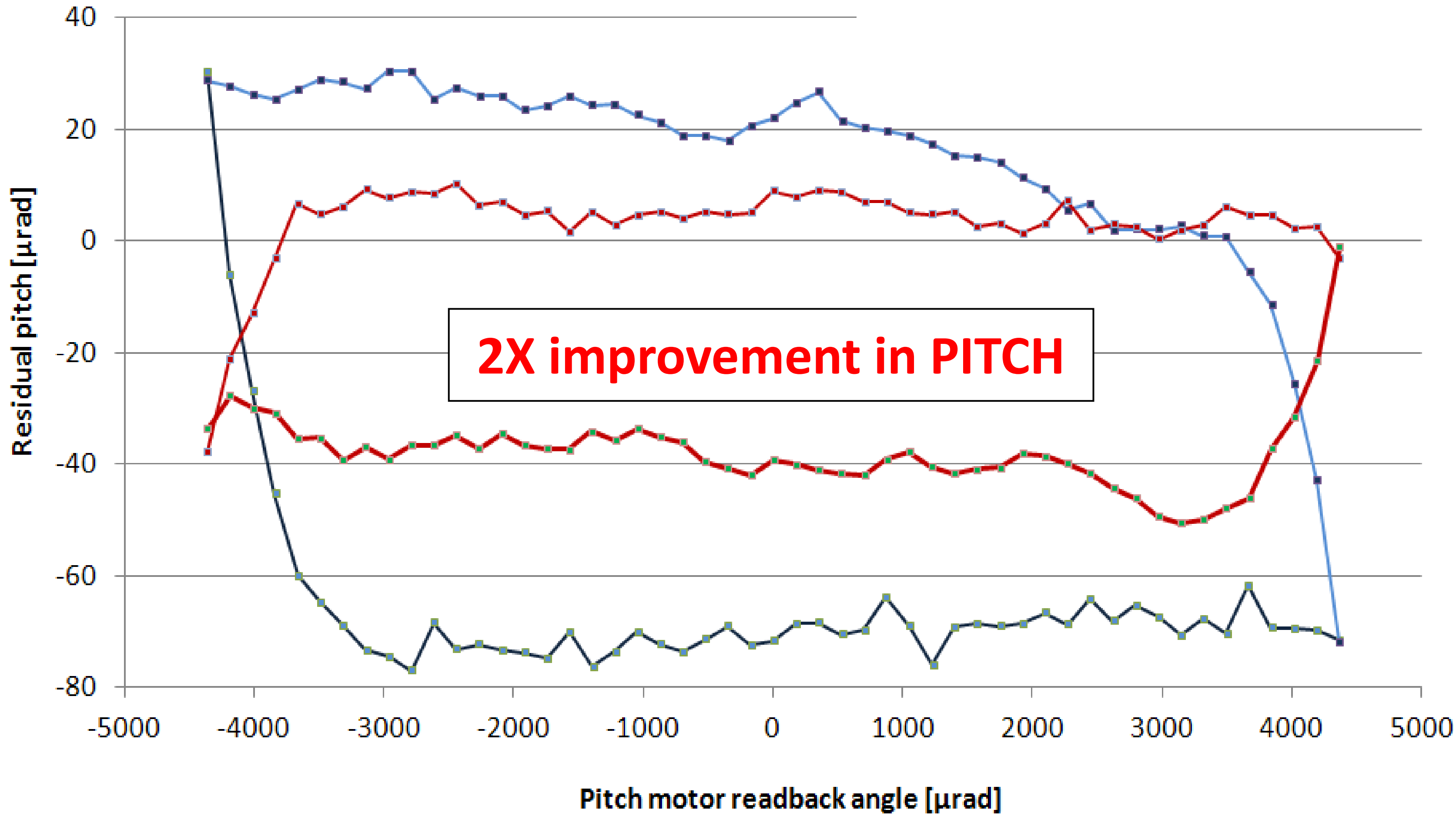

I22

Improvements during I09 DCM construction

- Flexure hinges replaced with thicker, stiffer version (**Jon Kelly**) → reduced parasitic hysteresis for roll & pitch angles



Improvements during I09 DCM construction



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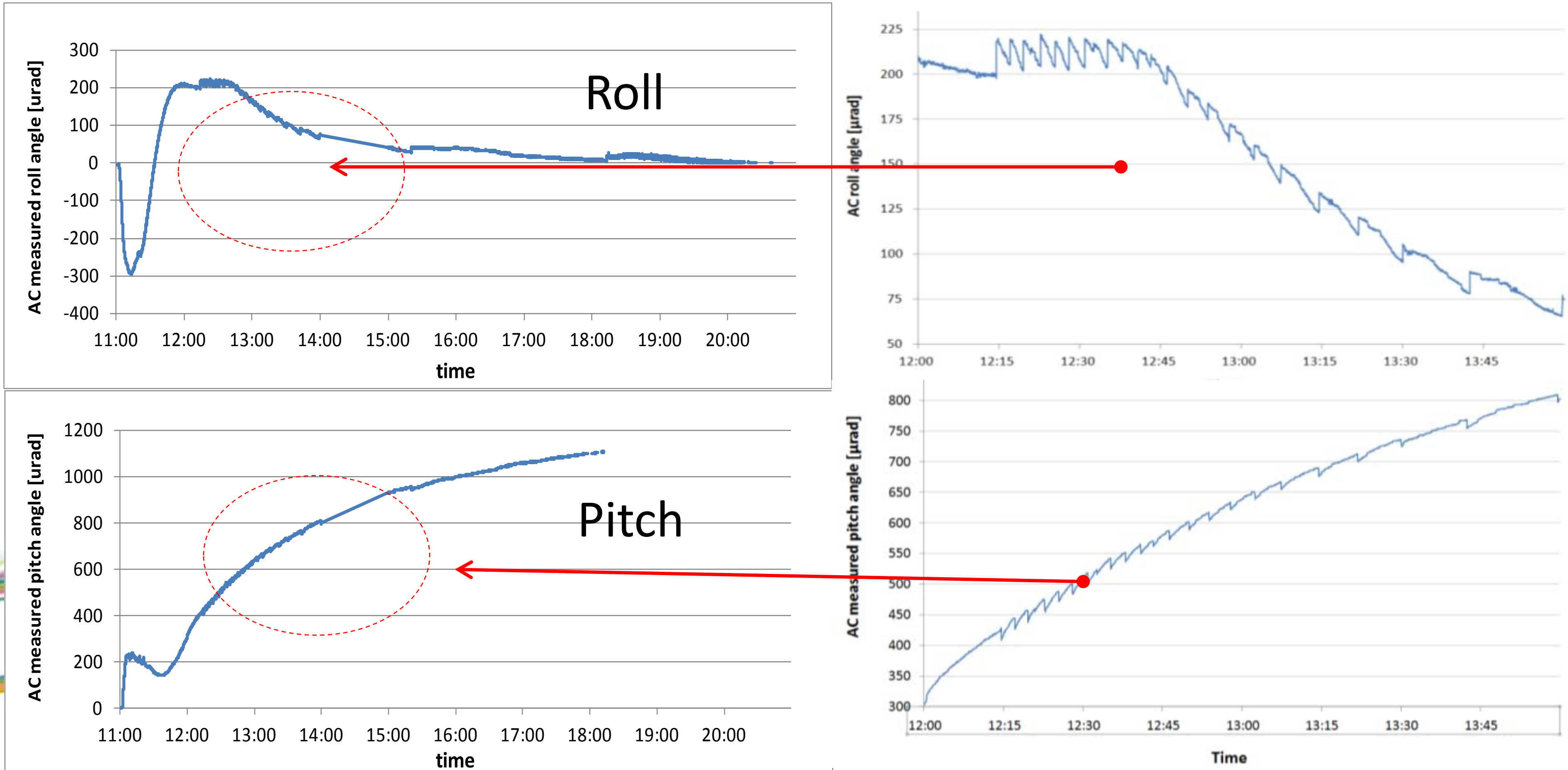
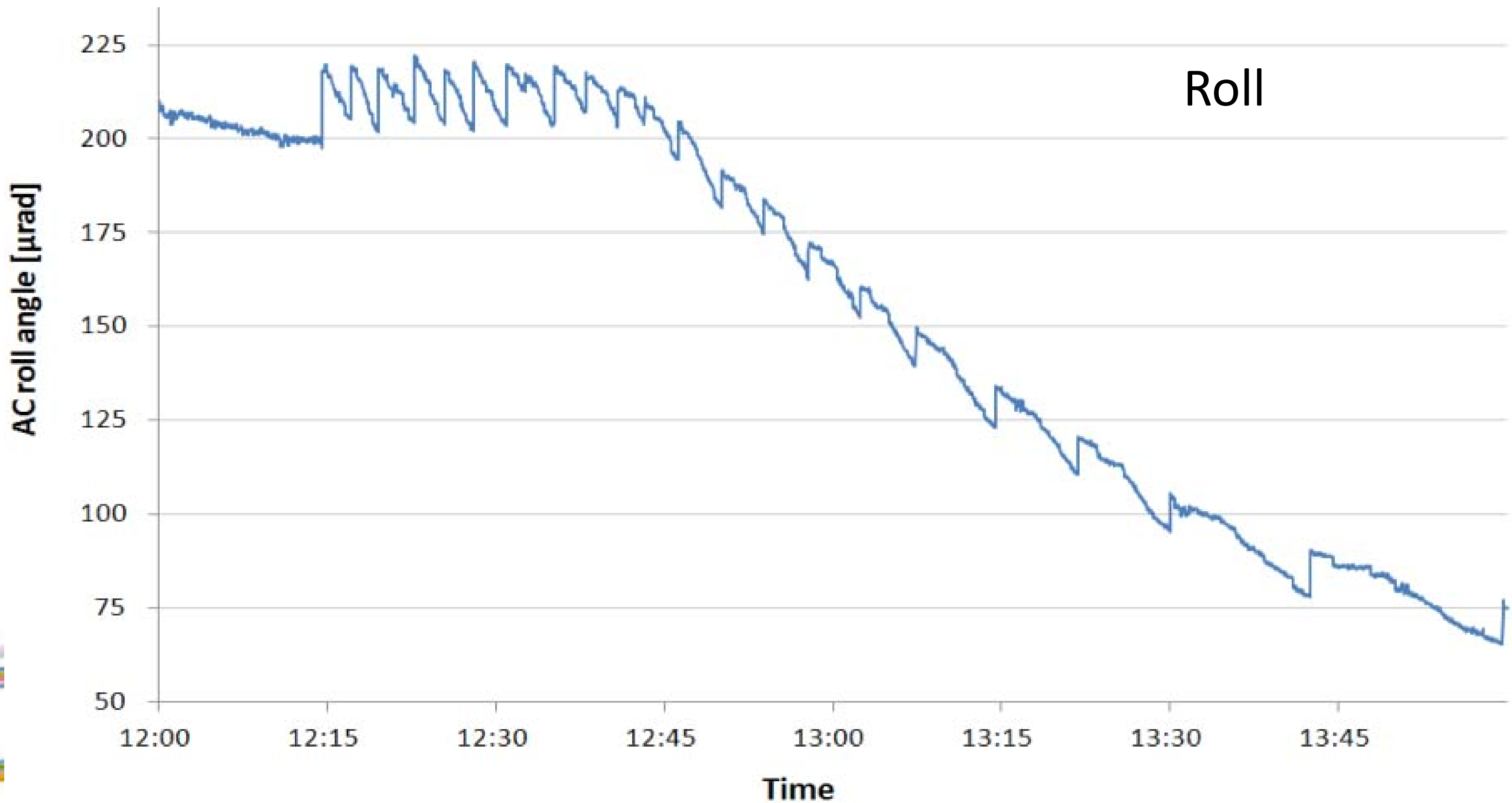
I23 DCM …during cryo-cooling!

Jon Kelly, Hiten Patel, Alex Bugnar



- During cryo-cooling, crystal parallelism changed by ~1mrad (pitch)

I23 DCM …during cryo-cooling!



- Stick / slip period extends as rate of cooling falls

Summary

1) Characterisation & improvement of crystal clamping:
   - *Indirect (interferometry / profilometry)*
   - *Direct (lab based X-ray source or beamline)*

2) Precision metrology (AC, vibrometer, interferometer)
   - *More reliable, accurate motions & alignment*

→ Significantly improved DCM performance!!! ☺

→ DLS in-house DCMs outperforming commercially available systems

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