

**A Light for Science**



**European Synchrotron Radiation Facility**

## **EDXAS: a Great Technique. Why is it so Difficult?**

Mairs T.R., Mathon O.

EDXAS: From a mechanical engineering view. Its main characteristics and their consequences.

- **No moving parts during data acquisition.**
- **Small and uniform focal spot using diffractive optics.**
- **$I_1/I_0$  normalisation not done simultaneously**
- **Fast acquisition times**
- **2d position sensitive detection**

**Firstly.....my apologies. The images you will see in this presentation have almost all been shown already in this workshop by my colleagues. However, I hope I will say something different!**

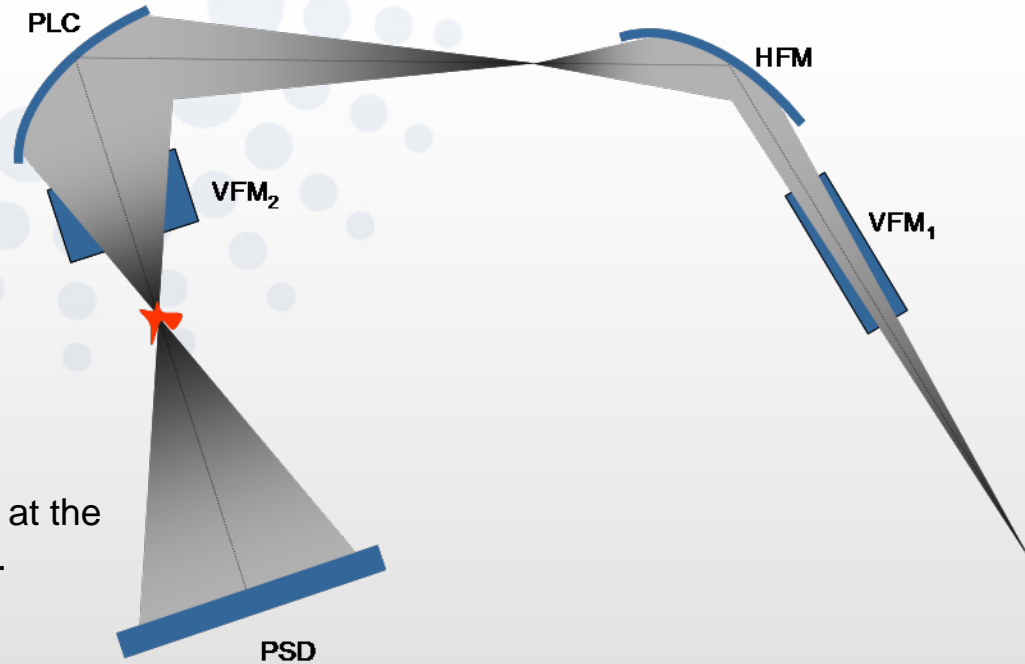
## No moving parts during data acquisition.

Small and uniform focal spot using diffractive optics.

$I_1/I_0$  normalisation not done simultaneously

Fast acquisition times

2d position sensitive detection



Great...easy.

Alignment of three mirrors and one polychromator at the beginning of the week then sit back and take data.

More rationally

A careful alignment of mirrors using a pre-defined procedure. Changing energy requires changing stripes on mirrors and refocusing the polychromator. All actions are well known and could be automated.

Automation requires reproducible mechanics and diagnostics interfaced to the software.

**No moving parts during data acquisition.**

**Means no parts deliberately driven during data acquisition.**

**BUT as we see later “moving” needs to be interpreted differently in order to get good data.**

No moving parts during data acquisition.

**Small and uniform focal spot using diffractive optics.**

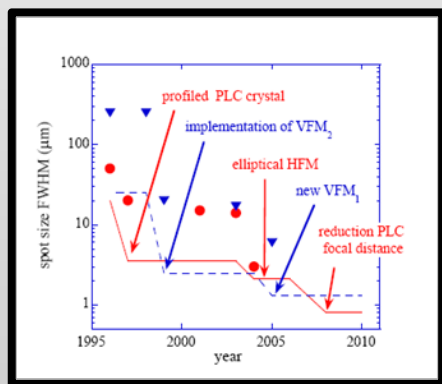
$I_1/I_0$  normalisation not done simultaneously

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## Polychromator

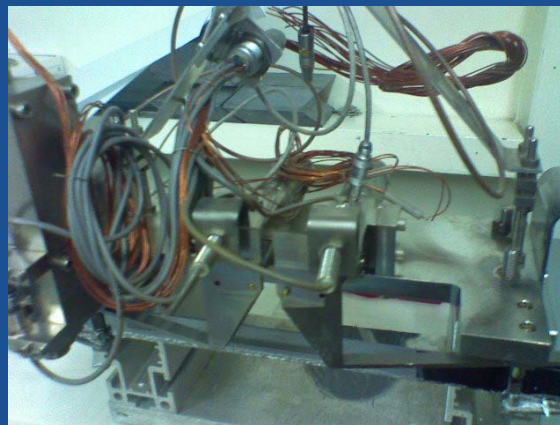
- A device specific to EDXAS.
- Limited development time.
- Large variation of energy, focal distance, resolution.
- Heatload



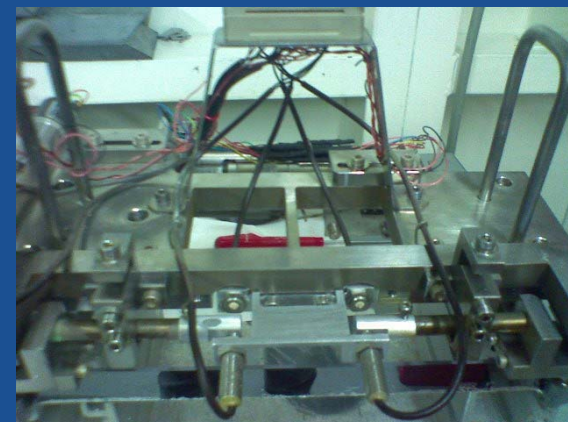
Reduction in focal spot size.  
What did we actually do?

## Evolution of Polychromator at ESRF

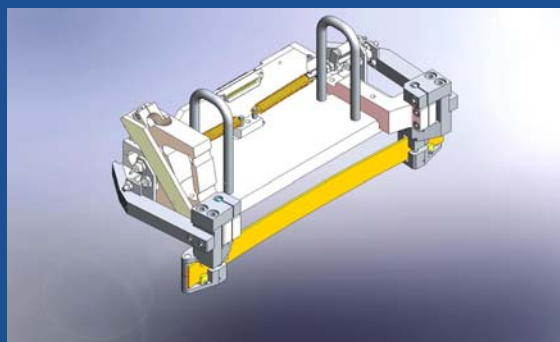
### BRAGG



1995 Beamline Commissioning



2001 new bender  
Crystal 1mm—2mm thick



2008 new bender 2 (see poster)

→ Improved reproducibility and precision. More stable and rigid. Possibility to automate focusing.

↓ BUT major improvements in focal spot size are due to crystal quality

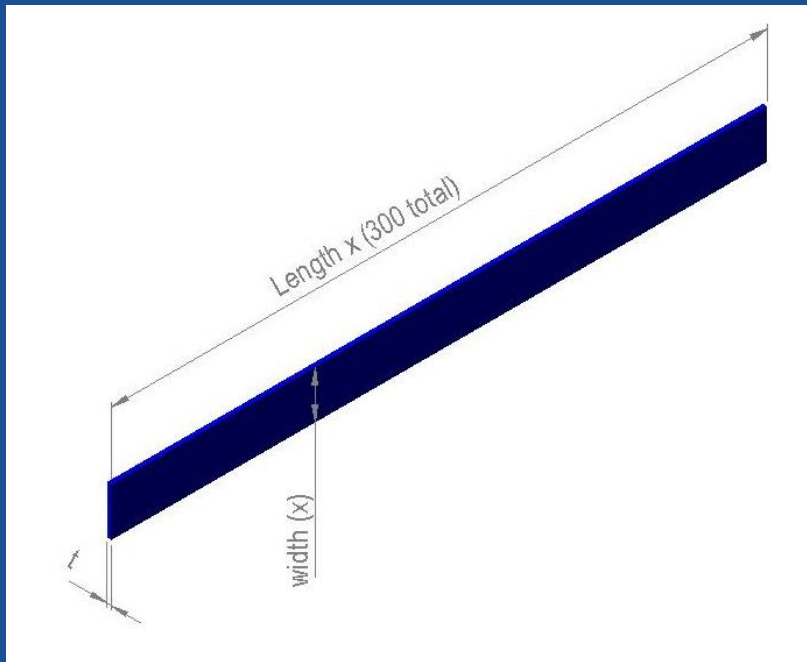
No moving parts during data acquisition.

**Small and uniform focal spot using diffractive optics.**

## Evolution of Polychromator at ESRF

### BRAGG– The crystal

1998 Problems focusing the beam. Investigation into quality of bender, but also quality of the crystals.



Curvature is proportional section inertia

$$\text{Inertia} = \text{width} \times t^3/12$$



Error in thickness  $t$  has a very significant effect on quality of focus

1999 typical errors in  $t$

$$t = 1\text{mm} \pm 0.05 \text{ gives error in } t^3 \text{ as } \pm 16\%$$

2008 typical errors in  $t$

$$t = 2\text{mm} \pm 0.005 \text{ gives an error in } t^3 \text{ as } \pm 0.075\%$$

Reduction in focal spot size.  
What did we actually do?

PLUS systematic quality control of crystals.



No moving parts during data acquisition.

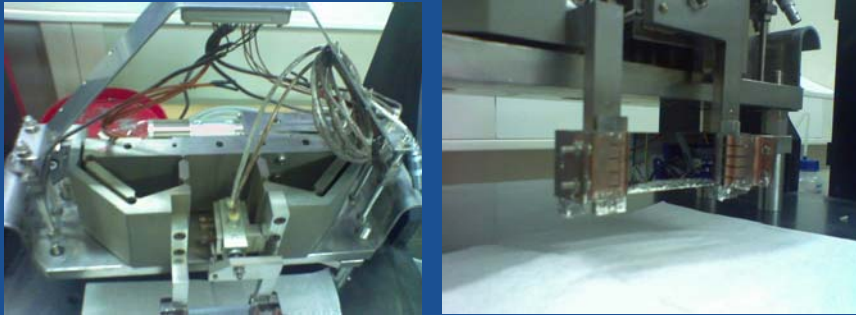
**Small and uniform focal spot using diffractive optics.**

$I_1/I_0$  normalisation not done simultaneously

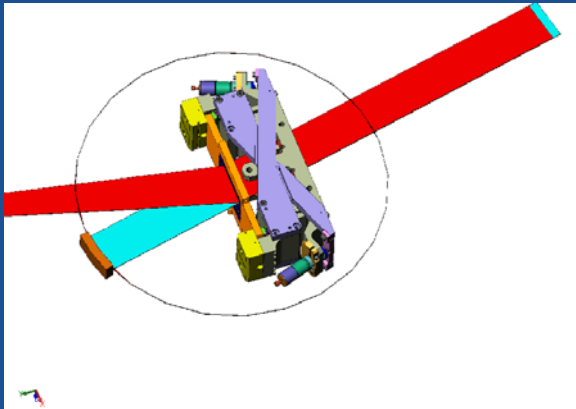
Fast acquisition times

2d position sensitive detection

## Future Improvements:Laue



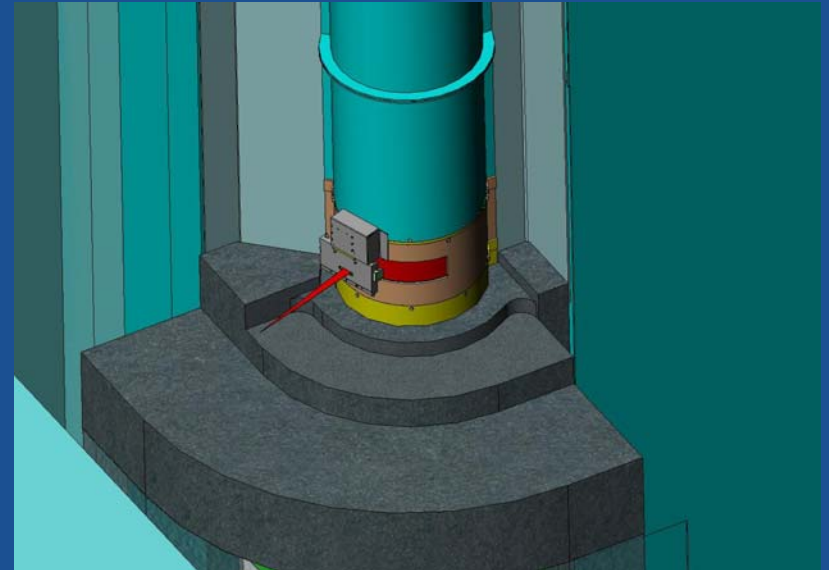
Theoretical calculations completed  
 Crystal fabrication problems as for Bragg  
 Crystal cooling...try to find alternative to In/Ga bath



Prototype to be built in 2009.

Based on pre-shaped water cooled copper block in KB mirror bender

## Future Improvements:Chamber/shutter



Main features;  
 "Small shutter" limited vibrations when opening.  
 Reduced focal distance to 650mm.  
 Stable Granite table for sample/mirror  
 Improved positioning precision.

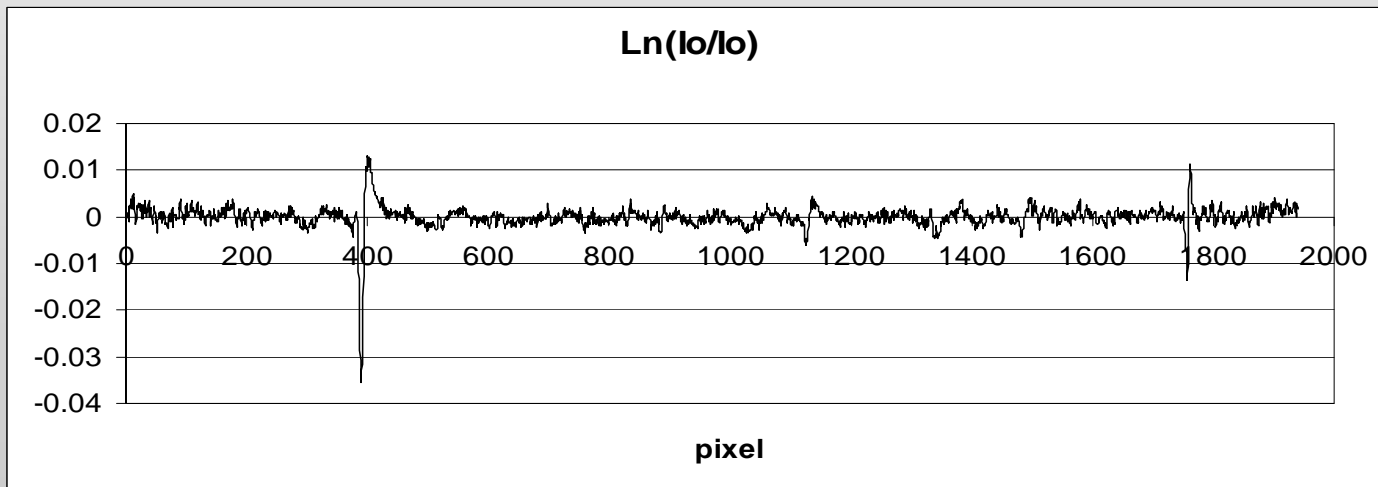
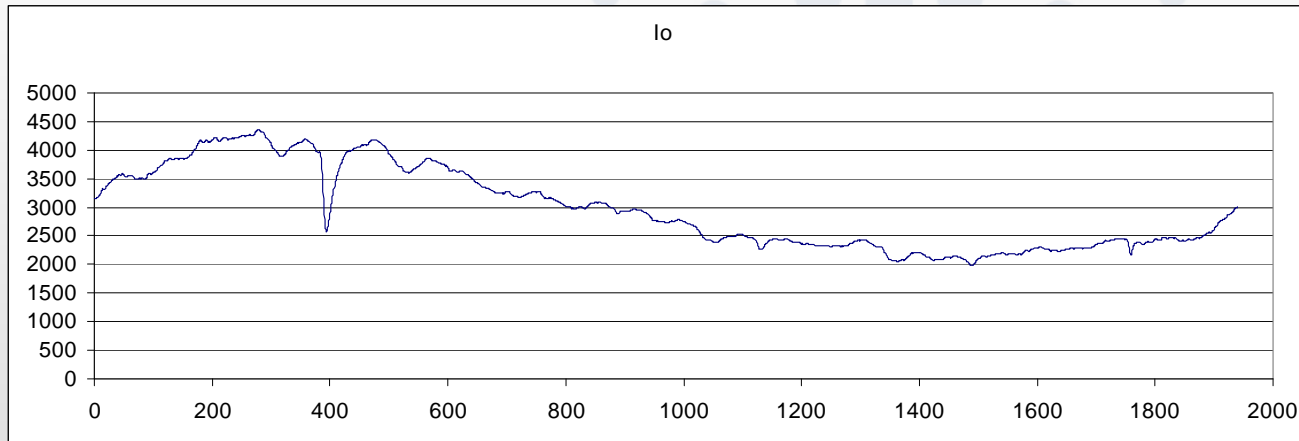
Not much work has been completed yet

No moving parts during data acquisition.  
 Small and uniform focal spot using diffractive optics.

**$I_1/I_0$  normalisation not done simultaneously**  
**Fast acquisition times**  
**2d position sensitive detection**



These 3 characteristics create the challenges.  
 (assuming transmission mode)



A typical (if not very nice)  $I_0$



$\ln(I_0/I_0)$  is a good measure of whether the beamline is working correctly



What happens if the beam moves horizontally by  $\frac{1}{2}$  pixel on the detector. (about 10 microns)



We must stabilise the beam on the detector AND on the sample

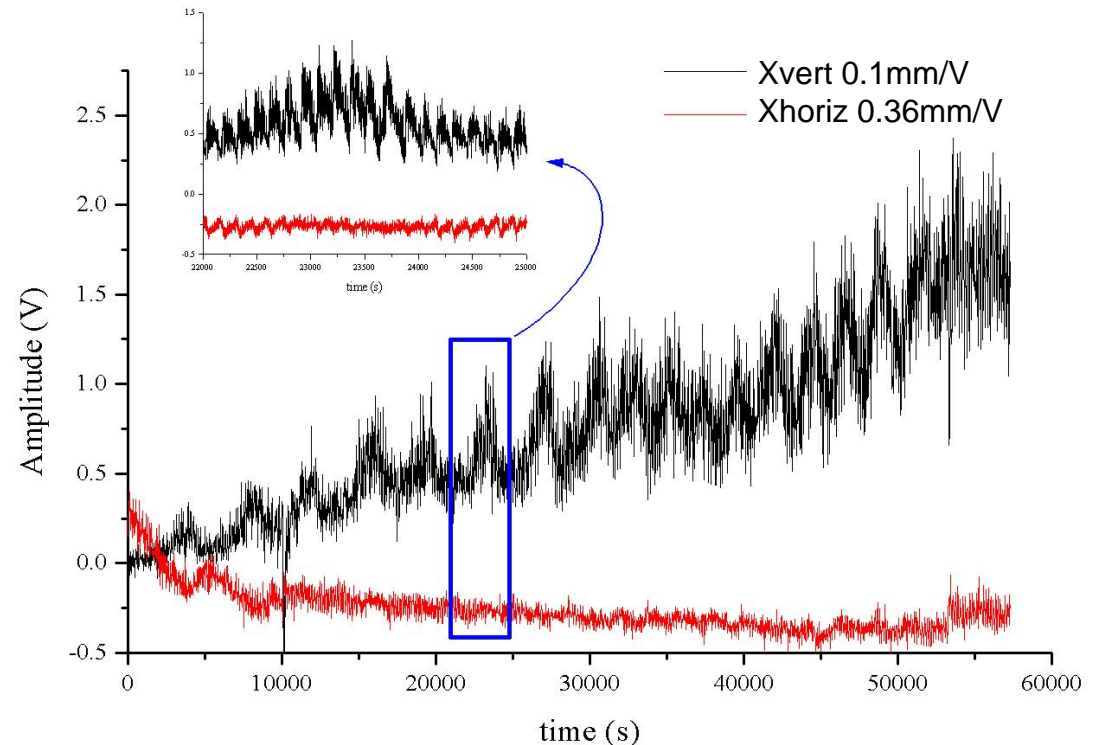
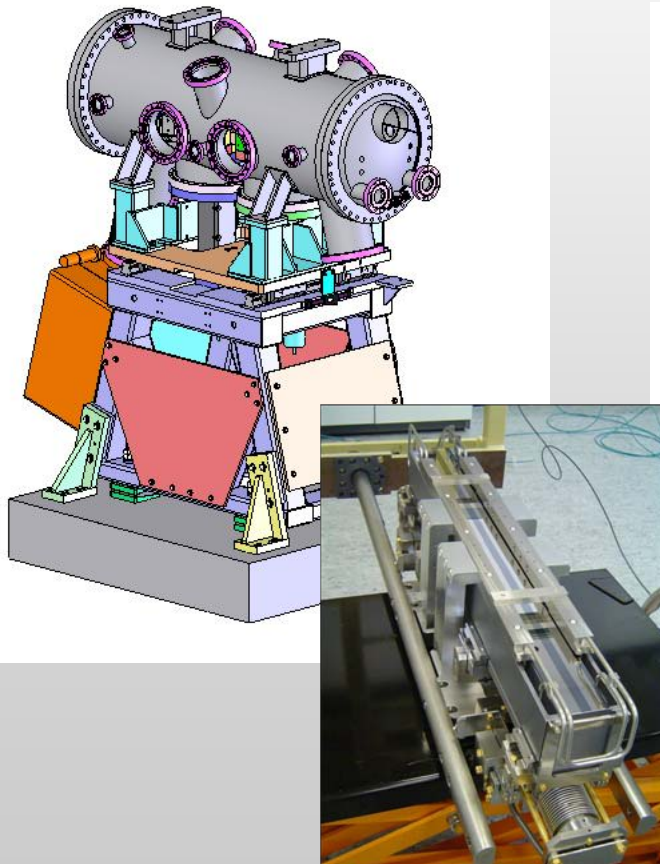
**No moving parts during data acquisition.**

This means no motion of the beam relative to the sample or to the detector

Stability of the optics

Thermal stability: slow drifts of the beam

**Diagnostic:** Is there a problem? **Action:** Are there solutions?



**Typical scan of x-ray position over 16 hours. Low heatload**



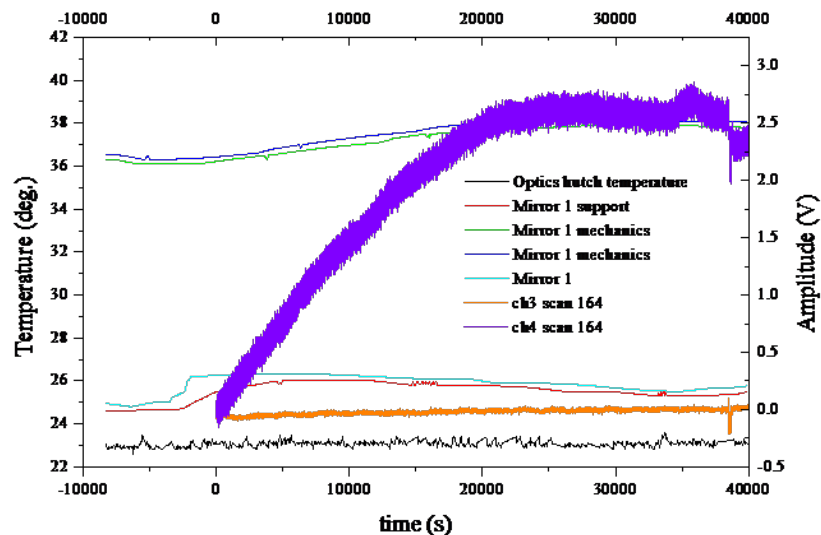
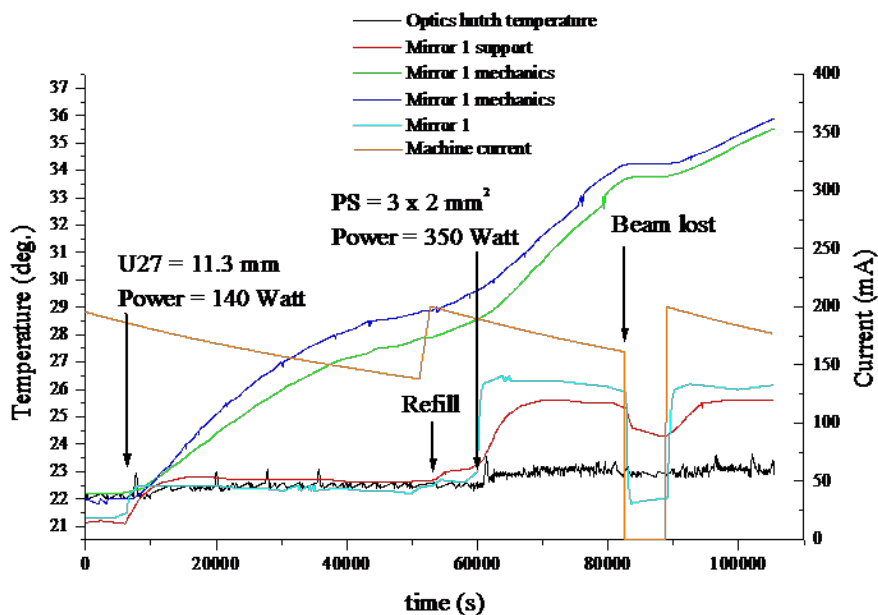
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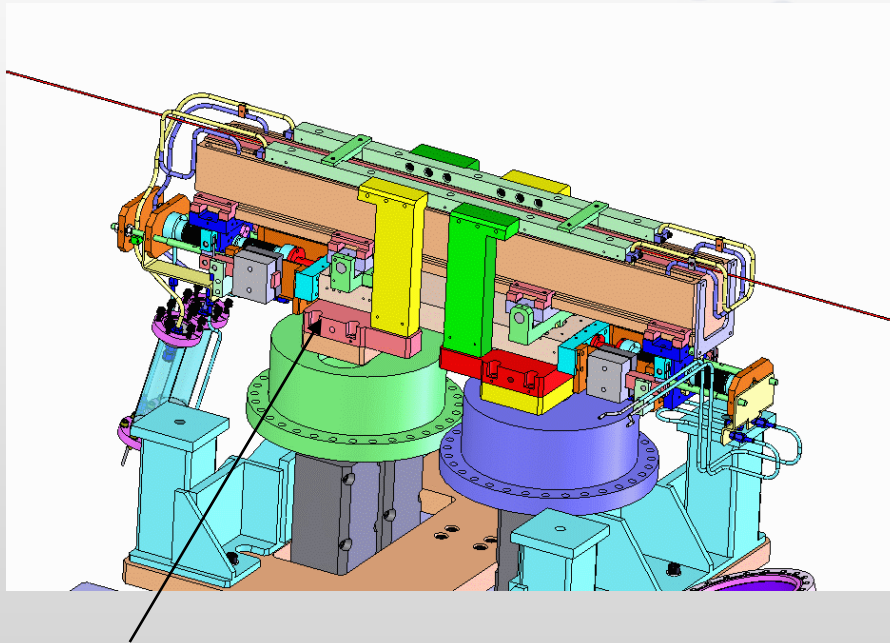
Temperature variations in mirror 1 with high heatload 350W

Correlation Mirror temperatures and beam position

## No moving parts during data acquisition.

### Stability of the optics

What can cause beam drifts?



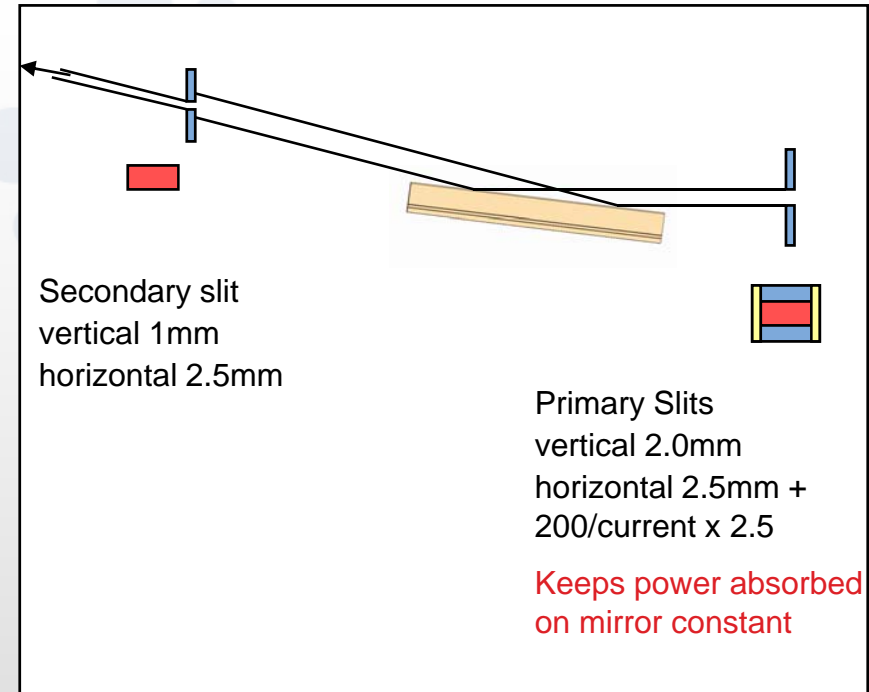
Main problem heating of the mechanics in particular the tilt.

Long reaction times

Solution: cool the mechanics

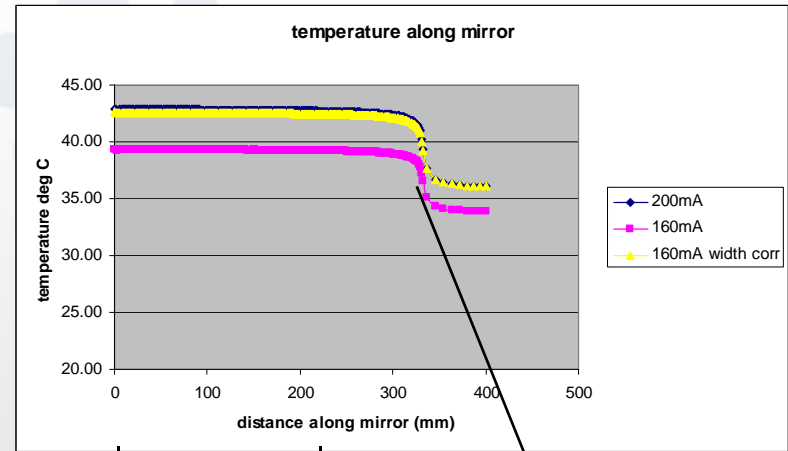
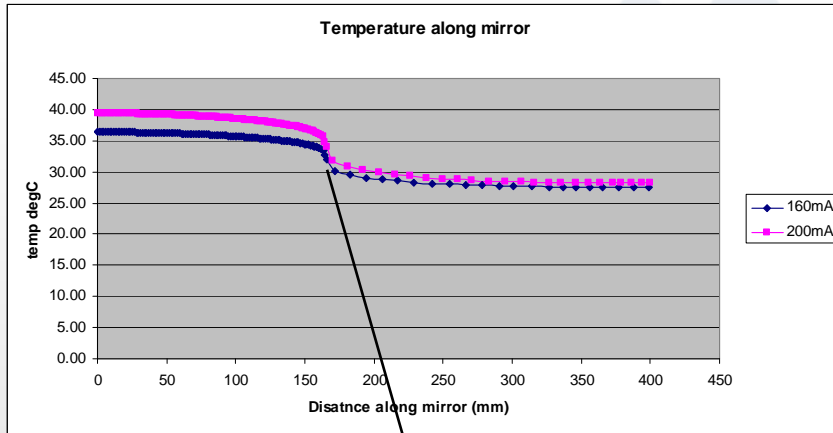
AND

Overfill the mirror



Principle of mirror overfilling

# Mirror Overfilling

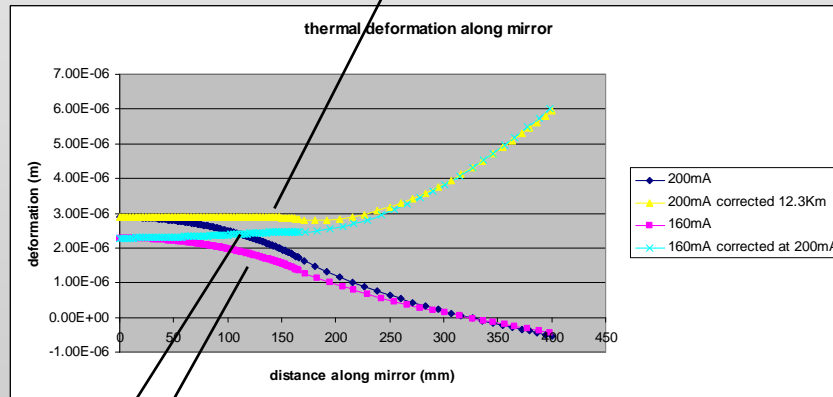


Useful beam

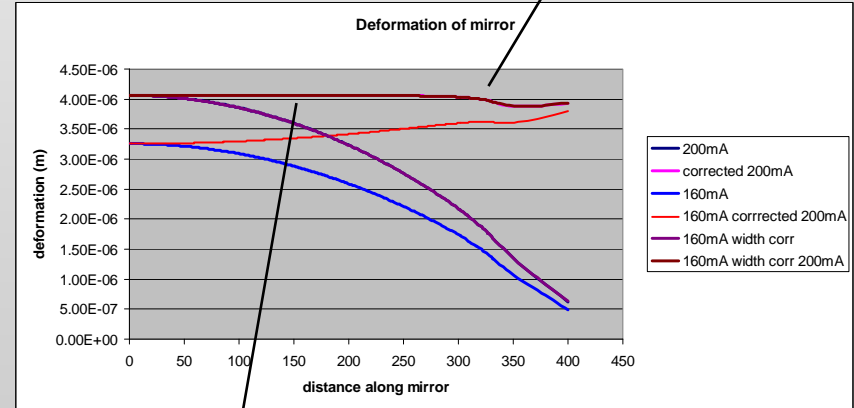
End effects, where beam is, cause local deformation

Useful beam

End effects, where beam is, are outside useful beam



Bender can correct for thermal slope errors but the correction is different at different currents



Bender can correct for thermal slope errors. With constant power on mirror same correction at 200mA and 160mA

## No moving parts during data acquisition.

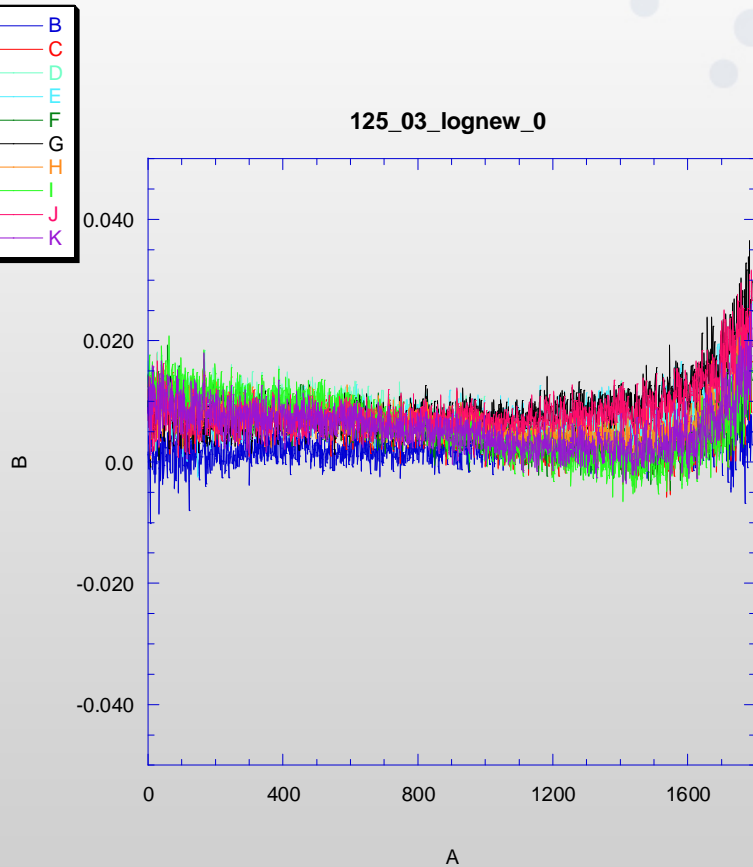
This means no motion of the beam relative to the sample or to the detector

### Stability of the source

If we measure  $\ln(i_0/i_0)$  on a good day what can we expect.

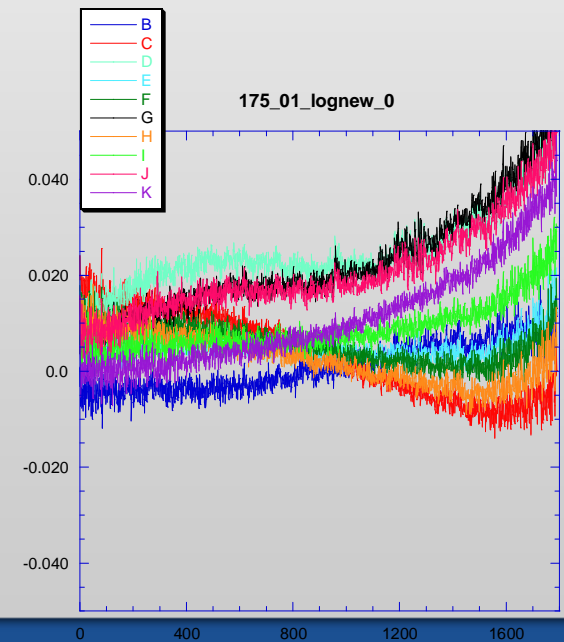
Spectrum every 26ms (13 accumulations@ 2ms)

Data taken in September 2006 during tests of the machine at different currents



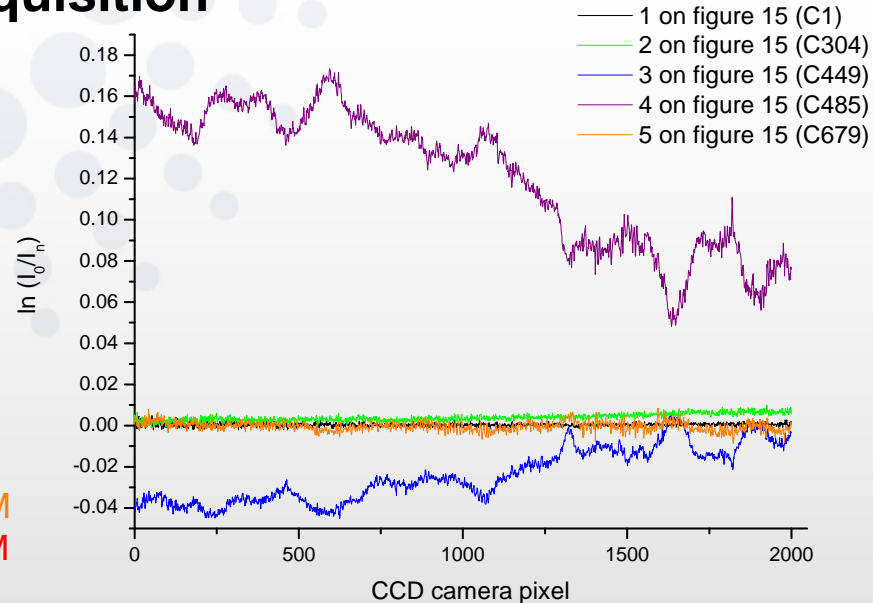
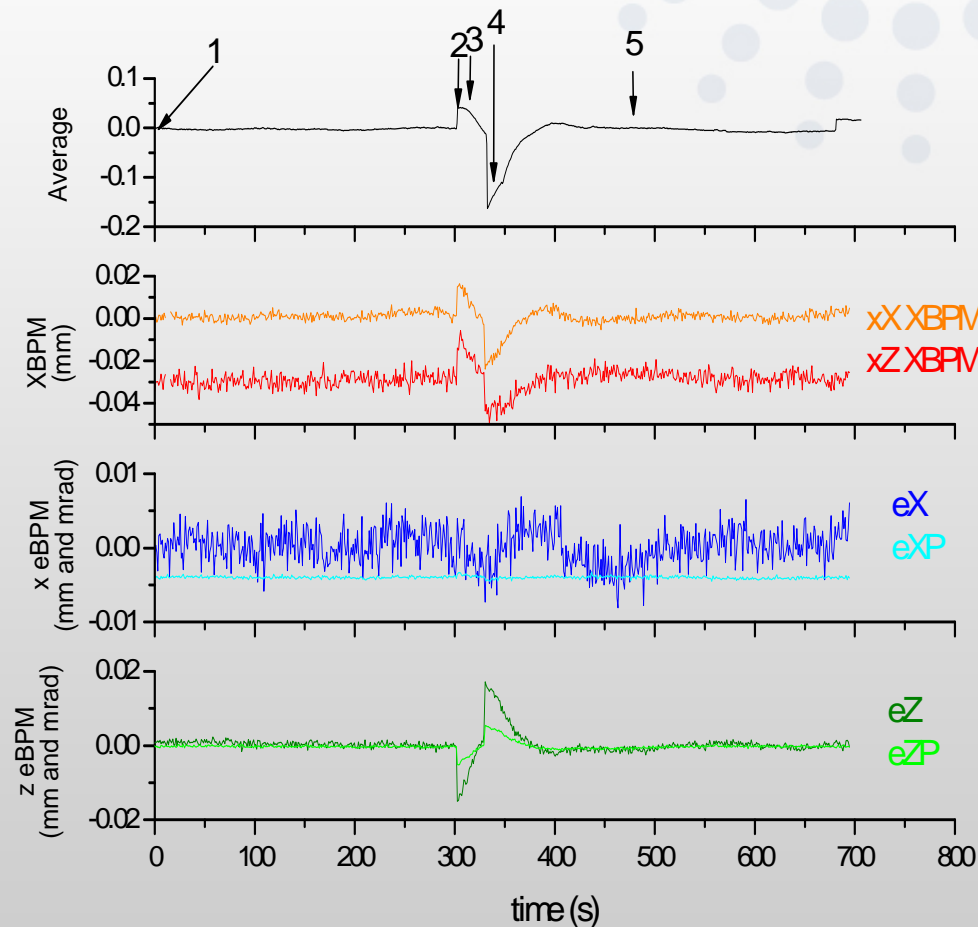
During injection.

Topping up mode could cause problems



And on a good and bad day....

# Effect on data acquisition



**“Accidents” of e-beam affects drastically the data acquisition quality**

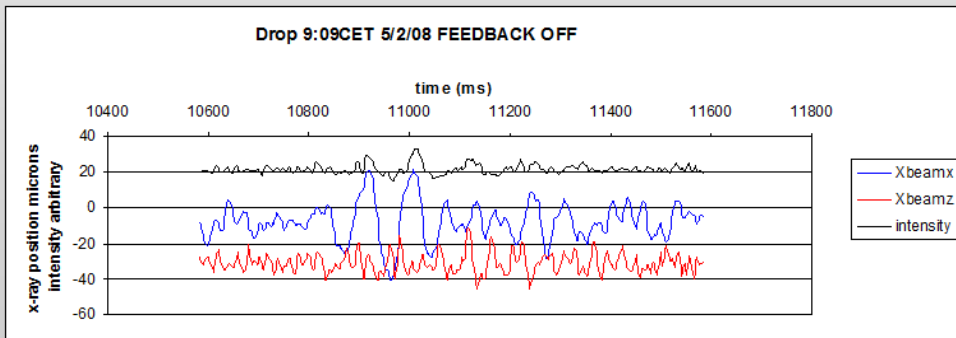
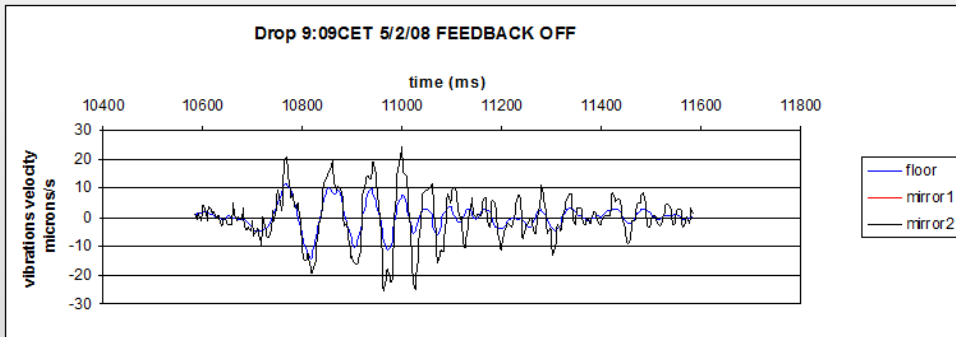
**Data taken Feb 2008 after improvements to mirror cooling.**



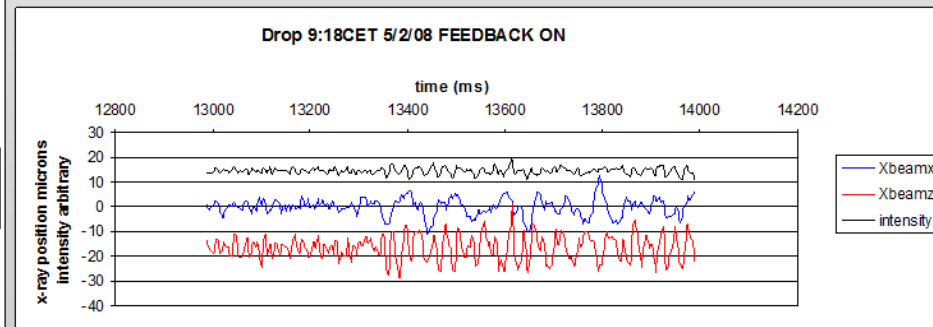
## External Influences on Beam stability

As preparatory work for the ESRF upgrade investigations on the quality of the site were made and to analyse the possible effects future infrastructure project: extension tramline etc.

Method: Drop a 1.2Tonne mass from 3m in centre of ring and see what happens to the beam.



Electron feedback OFF gives disturbance +/-20 microns in x-ray position for approx 0.5s



Electron feedback on gives disturbance +/-10 microns in x-ray position for approx 0.5s.

# No moving parts during data acquisition.

Stability of the optics:

fast beam movements > 1Hz

Measurements on ID24 trying to correlate variations of intensity with e-beam, floor vibrations and mirror vibrations.

Immediate actions: Remove sources of vibrations. chillers, pumps etc. Control water flow.

Future actions: Redesign of mirror supports and cooling systems. Possible feedback on beam position.

Coherence with mirror 1 (vertically deflecting)

MassDropVibrationStudy.doc

L. ZHANG et al., March 2008

Hz for mirror-1, and 21, 43, 49, 54.5, 68, 72, 78, 97.7 Hz for mirror-2. The peaks at 20~21, 49 and 97.7 Hz are also visible in the spectrum of the floor vibration. The much higher vibration level in a large frequency range (above 15 Hz) of the mirrors probably indicates the cooling flow induced vibrations.

### 6.4. Spectral analysis of X-ray beam Intensity fluctuation

One of the indicators of the X-ray beam instability is the X-ray beam intensity fluctuation. The X-ray beam intensity fluctuation could come from e-beam instability, the vibration of the floor, optics (mirrors and monochromator...) and some other beamline components. To track the sources of the X-ray beam instability, the PSD of the X-ray beam intensity fluctuation is shown in Figure 23 as well as the vertical and horizontal displacement PSD of the e-beam, the horizontal displacement PSD of the 2 mirrors and of the floor on the ID24 beamline. There are many more peaks in the X-ray beam intensity spectrum than in the other spectra. There is a peak at 49 Hz in the spectra of the floor, 2 mirrors and X-ray beam. In the e-beam spectra, the

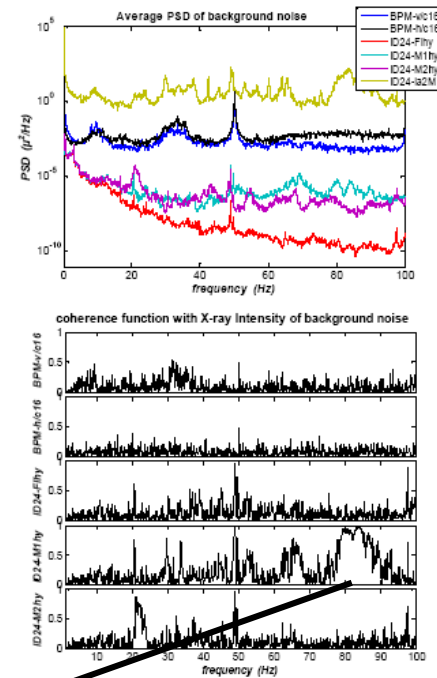
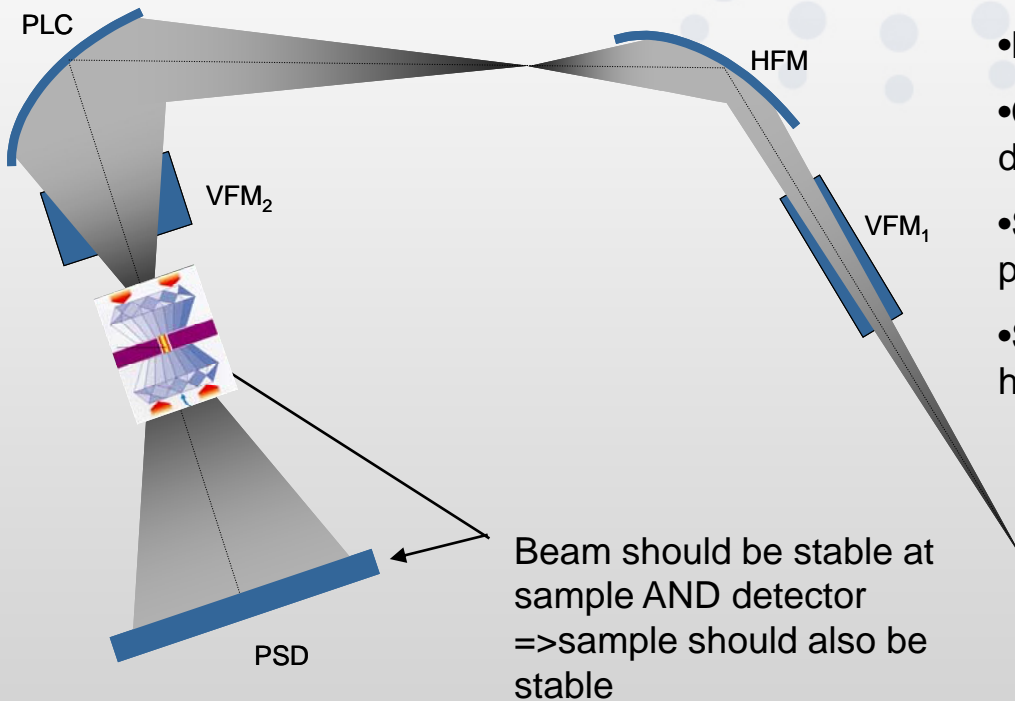


Figure 23: PSD (arbitrary unit) of the X-ray beam intensity, vertical and horizontal displacement PSD ( $\mu\text{m}^2/\text{Hz}$ ) of the e-beam, horizontal displacements PSD of the 2 mirrors and of the floor on the ID24 beamline (top), and coherence functions between the X-ray beam and vibration of the e-beam, 2 mirrors and floor in ID24

## No moving parts during data acquisition.

This means no motion of the beam relative to the sample or to the detector

### Stability of the sample



No real analysis done, but anecdotal evidence

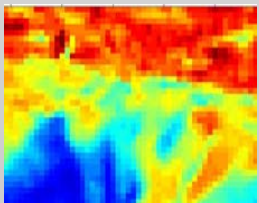
- Beryllium windows at VFM2 show structure
- Cannot cut beam between polychromator and detector without data degradation
- Surface quality of attenuators inserted in polychromatic beam is critical
- Small apertures (ie DAC) at sample position have to be accurately aligned

### The future sample environment:

High stability

Sub-micron resolution and repeatability

High quality temperature control of hutches,



In fluorescence and Turbo Exafs the resolution of these maps is dependent on precision of the alignment stages AND the stability of beam and sample.

## Acknowledgements

### ID24 team

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