AUTOMATION; DESIGNING THE BEAMLINE TO MAKE THE SOFTWARE POSSIBLE

T. Mairs, P Fajardo, J Meyer, E. Mitchell, D. Nurizzo, V. Rey-Bakaikoa, W Shepard, O Svensson

European Synchrotron Radiation Facility, 6 rue Jules Horowitz, BP220, 38043 Grenoble Cedex, FRANCE

Abstract

"Automation" and "High Throughput" are words that are becoming very common in synchrotron sources, and particularly, on Macromolecular Crystallography (MX) beamlines throughout the world. "Automation" is being implemented on all aspects of beamline operation including data acquisition strategy, sample changing, sample alignment, beam alignment and beamline quality control.

ESRF has recently had the opportunity with the construction of ID23, whose remit was to be a fully automated MX beamline, to design the beamline from the outset with automation in mind. This paper describes the specification and design of the hardware necessary to achieve a fully automated beamline alignment and calibration procedure. The beamline's performance will be logged and attached to the Beamline experimental data so that poor (or very good) data quality can be correlated to particular beamline conditions.

1. Automation: the Macromolecular Crystallography requirements

Macromolecular Crystallography at synchrotrons is no longer considered as an experiment where there are unknowns in the acquisition methodology. In effect, at the ESRF there can be several user groups using the same beamline in any 24 hour period all basically performing the same task. The restricted time available for data acquisition means that the beamline must be set up in a short time, be extremely reliable and very reproducible in performance.
Traditionally the tuning of beamline optics is carried out at the beginning an experimental run (4-6 week period) by the beamline scientist after which only small adjustments are made on a day to day basis. These methods, that have served the community well, have some drawbacks.

- The tuning of beamline optics is an expert process, which requires a good background in how the beamline was constructed.
- Different beamline scientists produce different results
- The process is time consuming.
- The beamline can become de-tuned during the experimental run.

All the above conditions lead to an overwhelming case for automating the whole of the data acquisition process for MX beamlines. This paper outlines the mechanical hardware necessary to automatically tune the beamline optics and to survey their performance during data acquisition.

2. General Philosophy

2.1. Beamline layout

MX beamlines are simple in conception and consist principally of a monochromator and a focusing device with the associated defining apertures along the beamline. When a problem is encountered, even with such a simple layout, it is often difficult to ascertain which of the

optical devices is at the origin of the problem. For example, a beam instability at the sample position could be caused by numerous devices including the machine, primary slits, monochromator, mirror etc.
Consequently the beamline has been designed so that each optical element has appropriate diagnostics before and after the element. These diagnostics have, as far as possible, been conceived to be capable of measuring the performance to a degree of accuracy sufficient to indicate whether the element is working to specification.

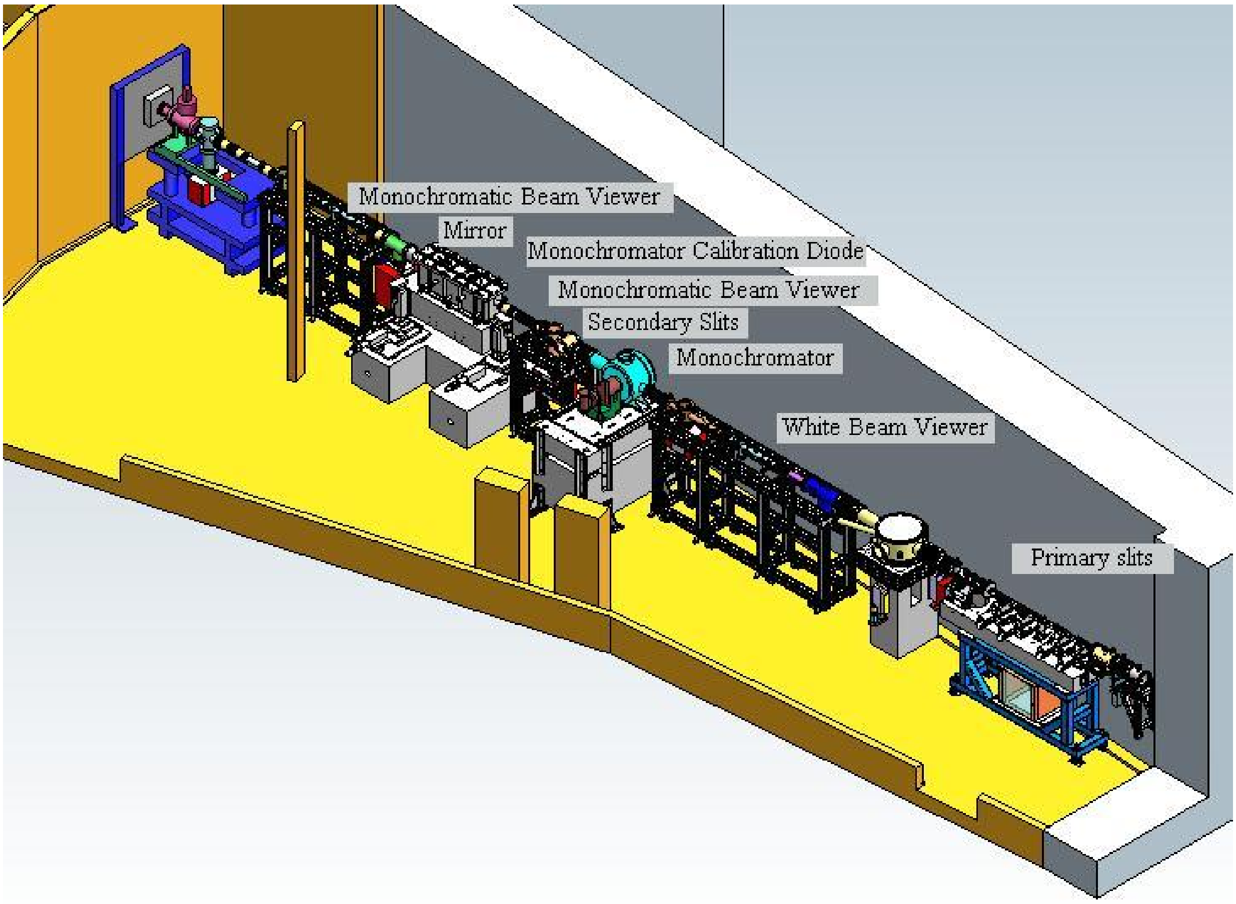


Figure 1 General Layout of ID23 optic hutch at ESRF

2.2 Optical element split

In order to achieve the automatic alignment of the optical elements it is necessary to split the beamline into sections that can function autonomously. Concentrating on the straight through MAD station of the ID23 beamline complex, the beamline was split into the following discreet parts.

- Primary Slits
- Monochromator and secondary slits
- Mirror
- Sample environment including sample slits, sample goniometer, detector and shutter

3. Primary Slits

3.1 Functional requirements

The primary slits mounted on ID23 are two apertures of 7mm horizontal x 3 mm vertical mounted on a YZ motorised table, which have to be aligned to x-ray beam. The primary slits are able to completely close in both directions and also to scan completely across the beam. An automated process must be able to logically accomplish a series of tasks in order to find the centre of the beam and then position the two apertures around this beam to give the desired slit opening. These tasks are complicated by the fact that the primary slits are dealing with white beam with a power density of 75 W/mm2.

An automated procedure needs to perform tasks to answer the following:

- Is there beam coming from the Front End?
- At what position of the motors are the slits fully close?
- Where is the centre of the beam?

3.2 White Beam Viewer

Situated immediately downstream of the Primary Slits, this device allows the user to visualise the white beam exiting from the slits. It consists of a CVD diamond foil with dimensions 20mm x 12mm x 0.250mm clamped mechanically to a water-cooled copper holder. See Figure 2.

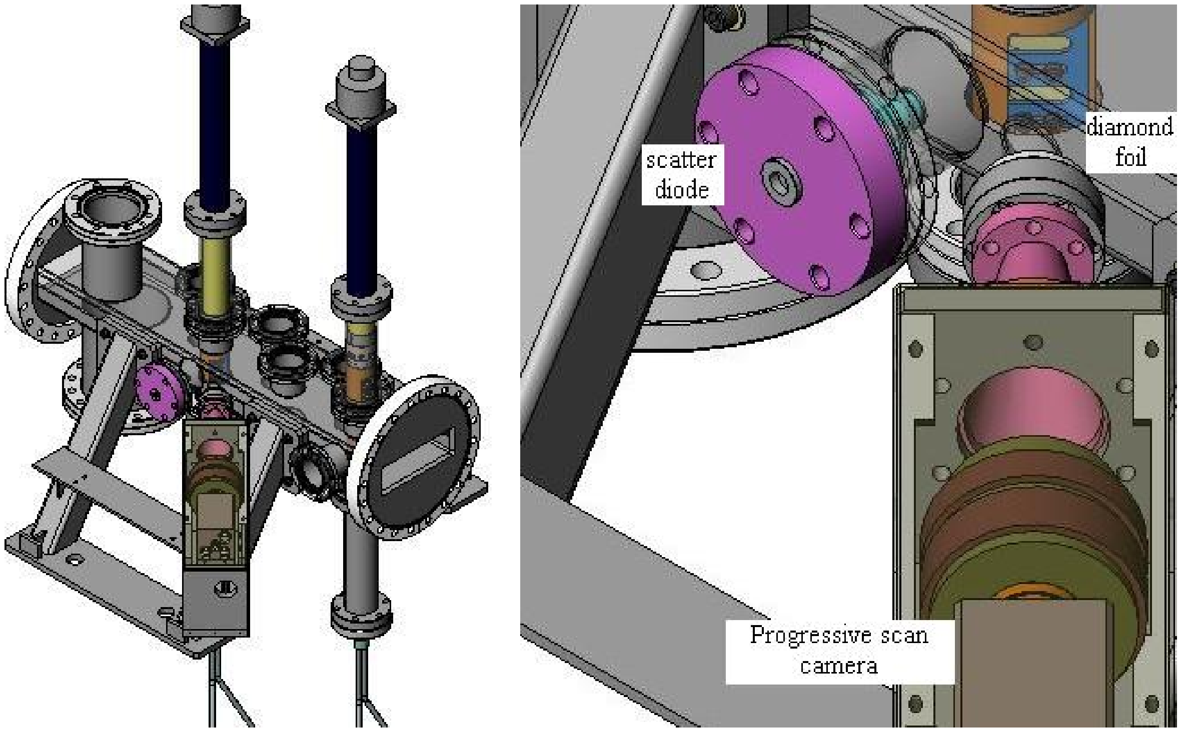


Figure 2 White Beam Viewer

The diamond foil, which is at 45degrees to the beam, emits visible light. A progressive scan black and white camera with appropriate lens is attached to a CF16 window flange. The progressive scan camera allows the adjustment of the acquisition time of the camera between 0.00002s and 2s, thus it is possible to have an unsaturated picture of the beam in all conditions from when the undulator is completely open to when the undulator is set at 11mm gap. Figure 3 shows the type of image that is produced with line profiles of the beam. The rounded corners of the Primary Slits can be seen.

It is possible to use this image to centre the Primary slits on the beam and the image is extremely reassuring for users to indicate the beam is there. It is indispensable for diagnosing misalignment of Front End elements. Unfortunately the diamond foil ages with time giving results that are incorrect. In figure 3 the dip in the centre of the horizontal line profile is an artefact of the diamond fluorescence.

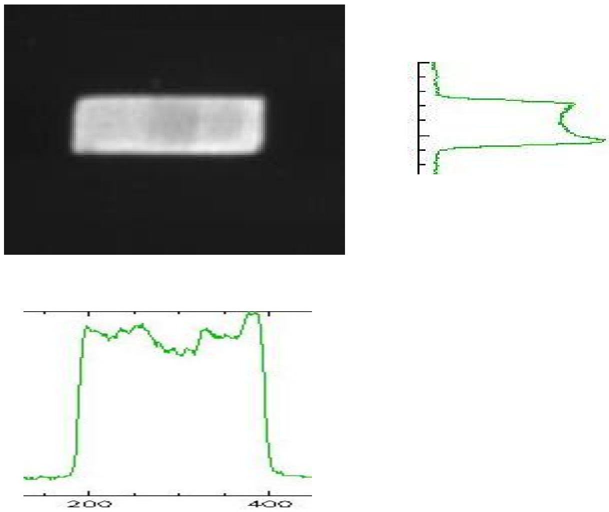
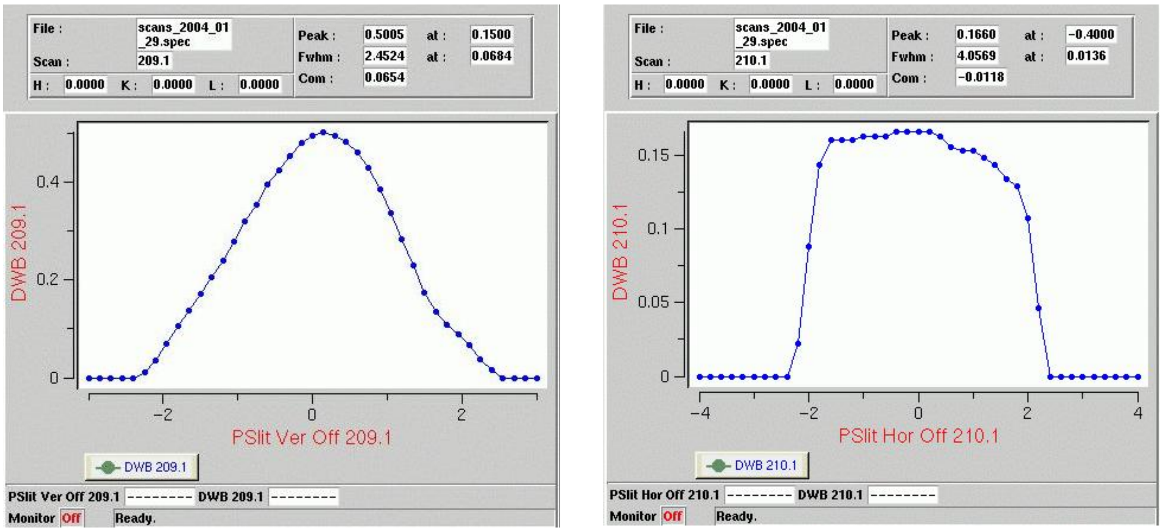


Figure 3 Images taken from the White Beam Viewer. Line profiles of intensity.

3.3 White Beam Scatter Diode.

In order to solve the problem of the non-homogeneity of the diamond foil, a pyrocarbon foil was also mounted on the same water-cooled copper block. As this is motorised using a commercially available vacuum actuator it is possible to insert either the diamond or pyrocarbon or nothing. A diode is attached to the CF35 flange and looks at the foil at 45degrees. Scanning the primary slits gives profiles shown in figure 4. It can be seen that the centre of the beam is more clearly defined, and thus, a reliable automatic centring of the Primary Slits can be implemented. This diode can also detect variations in beam intensity. See section 6.



a) b)

Figure 4a) scan of the primary slits vertically with a slit size 3mm horizontal x 0.1mm vertical

Figure 4b) scan of the primary slits horizontally with a slit size 0.1mm horizontally x 1mm vertical.

4. Monochromator and Secondary Slits

4.1 The Monochromator.

ID23 uses a channel cut silicon 111 monochromator. This design was chosen in part because of its simplicity and its easy alignment procedures. It has basically two movements: bragg angle and the tuning of the parallelism of the second reflecting face to the first. Figure 4 shows the crystal and the extremities of its rotational movement. The disadvantage of a channel cut monochromator is of course the intrinsic vertical movement of the beam when changing energy, however this can be easily compensated for automatically when changing energy.

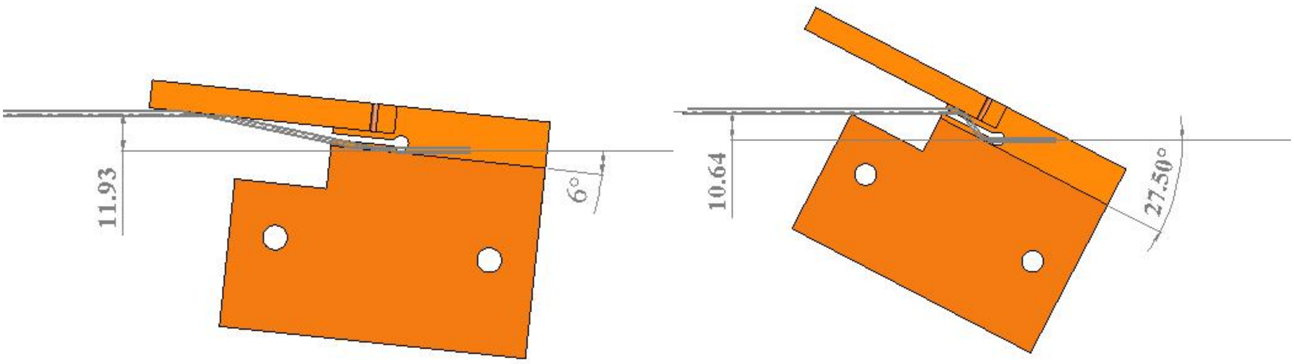


Figure 5 Channel cut monochromator crystal with vertical beam movement when changing energy

4.2 Functional Requirements

The monochromator has the basic function of selecting the energy of photons required for the experiment. The ID23 bragg angle is controlled with a stepper motor and active feedback on a high resolution incremental encoder. Tests in the ESRF Precision Engineering Laboratory showed that the rotation axis had an accuracy of 0.1mdeg. However, when installed on a beamline a monochromator performance needs to be verified and the energy calibrated. The second crystal needs to be tuned to eliminate harmonics, the undulator gap needs to be optimised for the chosen energy and finally the secondary slits need to be centred around the beam that exits the monochromator.

4.3 The diagnostics for monochromator and secondary slits

The beam is monochromatic so all problems of heatload no longer apply. Two diagnostic elements are installed after the secondary slits which when used together can achieve the required automatic alignment and calibration functionality.

4.3.1 Monochromatic beamviewer and foil holder

As is seen in figure 6 this unit is a derivative of the White beam viewer. The diamond foil is replaced by a YAG:Ce scintillator of the same dimensions, which is polished on both sides. See section 5 for details on the performance. The scintillator is mounted on a linear actuator, which allows the scintillator to be moved. There are 5 foils (platinum, copper, iron, zirconium, and molybdenum) mounted on the same actuator and, in addition, a Kapton foil mounted in a scattering configuration with an associated diode.

4.3.2 Monochromator calibration diode

Situated just downstream of the foil holder is a diode (10mm x 10mm) that can be inserted into the beam. This diode absorbs the entire beam and is consequently only used during calibration purposes. It

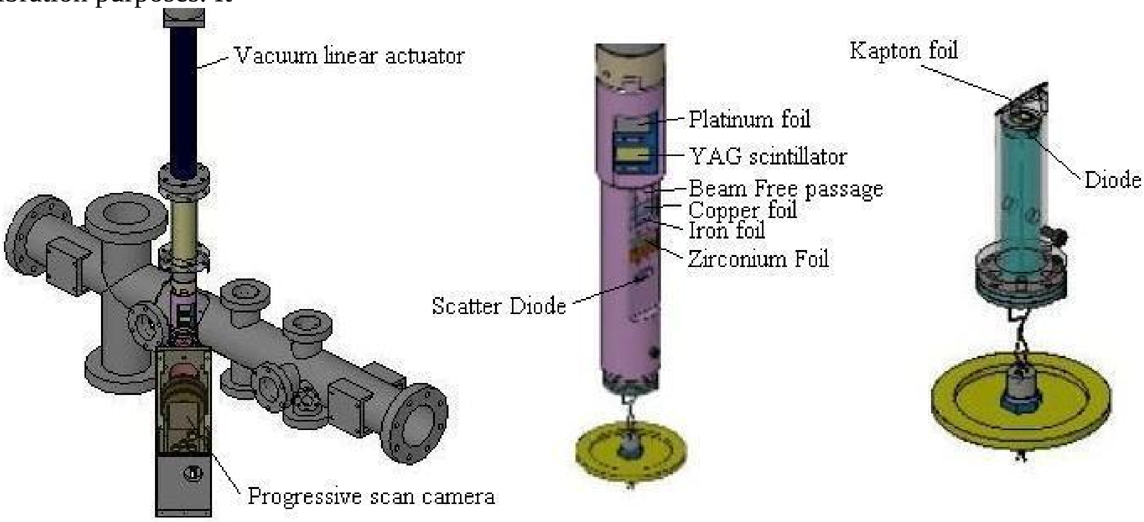


Figure 6 Monochromatic beam viewer and foil holder

is important that the signal given on this diode is independent of beam position as the beam moves in the calibration routines. Experience showed that a diode in a scattering configuration did not work.

4.4 Calibration Routines

Four routines have been developed for regular use to calibrate and check the functioning of the monochromator.

- Move Energy
- Energy calibration
- Undulator calibration
- Crystal tuning

The last two routines have been used for commissioning, but it has been shown that the repeatability of these measurements is such that they are now only used for special beamline requirements and are not described further here.

4.4.1 Move Energy

As can be seen from figure 5 the vertical position of the beam changes when the energy of the monochromator is changed. In consequence the optical elements downstream of the monochromator need to be adjusted. The first thing is to realign the secondary slits as misaligned secondary slits make any intensity variations due to beam instabilities worse. The beam height value derived from this procedure is used afterwards to adjust the height of the toroidal mirror. The Move Energy routine is performed every time the user requests a particular energy.

The macro Move Energy does the following actions

- Moves the monochromator Bragg angle to the appropriate value
- Moves the undulator gap to the correct value from a look-up table
- Inserts Monochromator Calibration Diode (MCD)
- Whilst reading the current of the MCD scans one vertical blade of Secondary slits until the value of MCD is reduced by 50%.
- Scans the other vertical blade until the value of the MCD goes to zero. Sets this value as zero for the gap of the secondary slits.
- Opens the vertical gap of the secondary slits to 0.1mm. Scans across the beam and centres the secondary slits on the centre of the beam exiting the monochromator.
- Opens the secondary slits to the value desired by the user.
- Calculates how much the beam has moved in height.
- Changes the height of the toroidal mirror by the same value (see section 5)

4.4.2 Energy calibration

The energy calibration of the monochromator is carried out at the beginning of each run and whenever there has been any possible loss of position of the monochromator angle. A loss of monochromator Bragg angle can occur if there is a power loss, during maintenance work and sometimes due to software problems.

The macro that does the energy calibration does the following actions:

- Moves monochromator to 11.56KeV which is the Platinum L3 edge and moves the undulator gap to the appropriate value from a look up table.
- Inserts Monochromator Calibration Diode (MCD) and the Platinum foil.
- Whilst reading the current of the MCD scans the energy from 11.52KeV to 11.62KeV in intervals of 0.5eV (scan I0).
- Removes the Platinum foil from the beam.
- Whilst reading the current of the MCD scans the energy from 11.52KeV to 11.62KeV in intervals of 0.5eV (scan I1).
- Performs data processing to produce the data logI1/logI0 see figure 7
- The energy at the inflection point on the edge jump is defined as 11.56276 KeV [1]. The value of monochromator energy is set to this exact value.
- To confirm the monochromator is working correctly the same routine is carried out at the copper K edge (8.98048KeV) and equivalent edges of iron, zirconium and molybdenum. The energy is not reset, but the calculated values should be within 1 eV if the monochromator is working correctly.

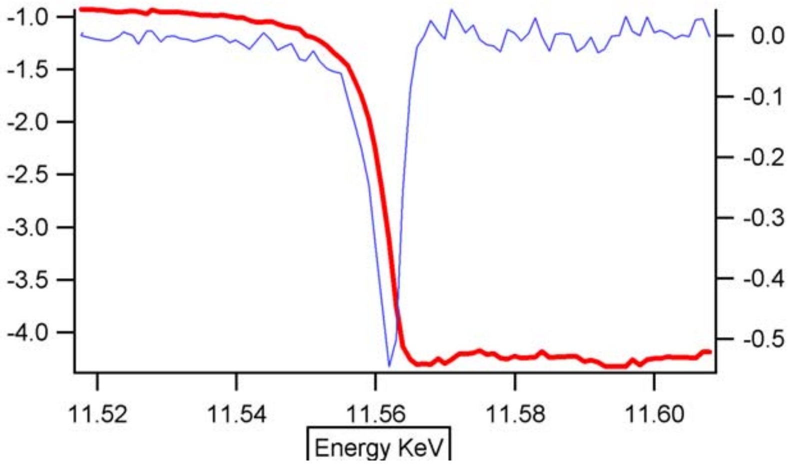


Figure 7 Edge scan of Platinum L3 for calibrating the monochromator with the derivative.

5. Toroidal Mirror

5.1 The Mirror Mechanics

The mirror itself is a silicon toroidal mirror of length 780mm with sagital radius 39mm and meridinal radius of with bender of 4.3Km. The distance between the source and mirror is 36.96metres and between the mirror and sample point is 7.94metres. The nominal angle of incidence of the mirror is 3 mrad. The mirror and bender are mounted in a vacuum chamber as shown in figure 8. The vacuum chamber and marble on which it is mounted are moved as a unit in order to align the mirror to the beam. The principal reason for mounting the mirror in this method was to try and reduce vibrations on the mirror.

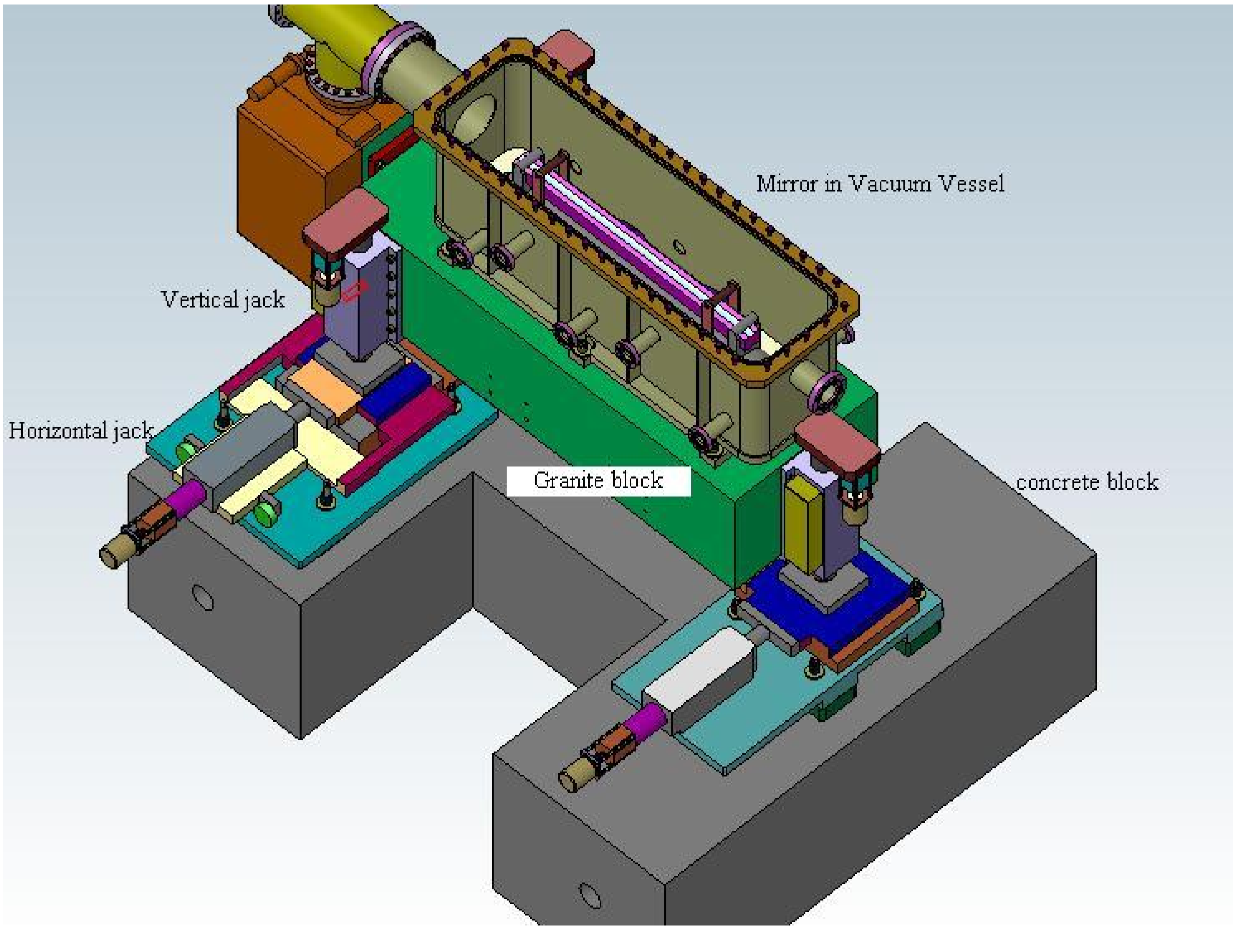


Figure 8 Toroidal mirror mounted in vacuum chamber

5.2 The diagnostics for mirror alignment.

In order to correctly align a toroidal mirror a simple diode is not sufficient as it does give any information on the shape of the beam. There are a number of adjustment that are necessary: a)Mirror height b)Lateral position c)Incidence d)Yaw e)Bend

All of these have an effect on the beam that can be distinguished by visualising the beam. The ESRF has developed a “wavefront analysis” method for aligning toroidal mirrors, which will be the subject of a future paper, which requires the following equipment.

- Secondary slits upstream of mirror

- Beam visualisation system just downstream of mirror that can detect the reflected beam and the non-reflected beam if the mirror is withdrawn. (Monochromatic Beamviewer)
- Beam visualisation system at the sample point with the resolution necessary to judge focus quality. (xray camera)

5.2.1 Monochromatic Beamviewer

The design for the monochromatic beamviewer just downstream of the mirror is based on the requirement to be able to see both the reflected beam and the non-reflected beam. The angle of the mirror is 3 mrad and the distance between the centre of the mirror and the beamviewer is 1.64metres. Hence the distance separating the two beams at this point is 9.84mm The screen is on a motorised vacuum actuator, so it can be adjusted in the beam and has a surface area available for the beam of 10mm vertically and 18mm horizontally. However, the camera is fixed so its field of view has to be larger. The lenses chosen give a field of view of approximately 25mm x 25mm and the number of pixels on the camera is 860 x 800 Hence, .the resolution of the beamviewer is 30microns per pixel. This is sufficient for the wavefront analysis method, using a 100micron slit, to prealign the mirror before optimising the alignment using an xray camera at the focal point.

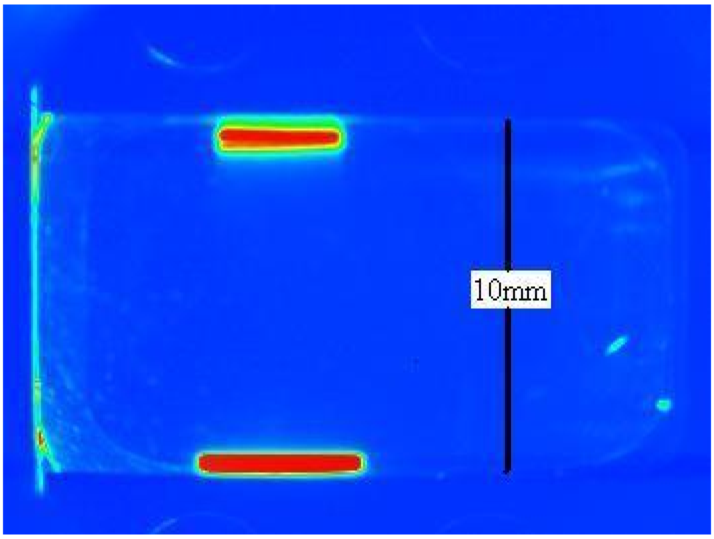


Figure 9 xray beam on monochromatic beam viewer

Figure 9 shows the two beams coming from the mirror when the mirror is half withdrawn, the angle of incidence of the mirror can be deduced from the image. The horizontal focusing effect can be clearly seen

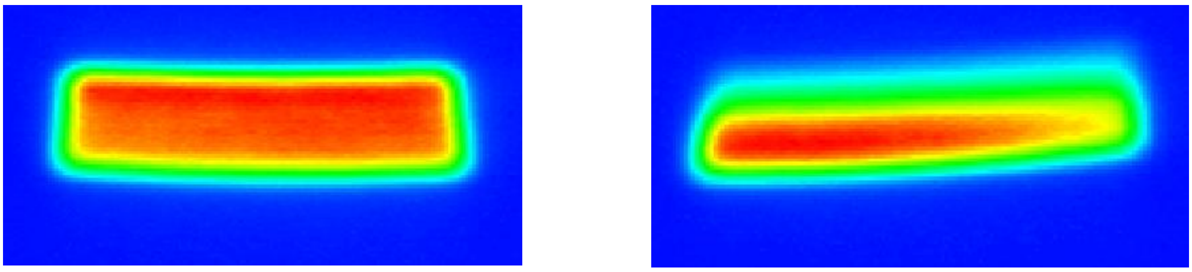
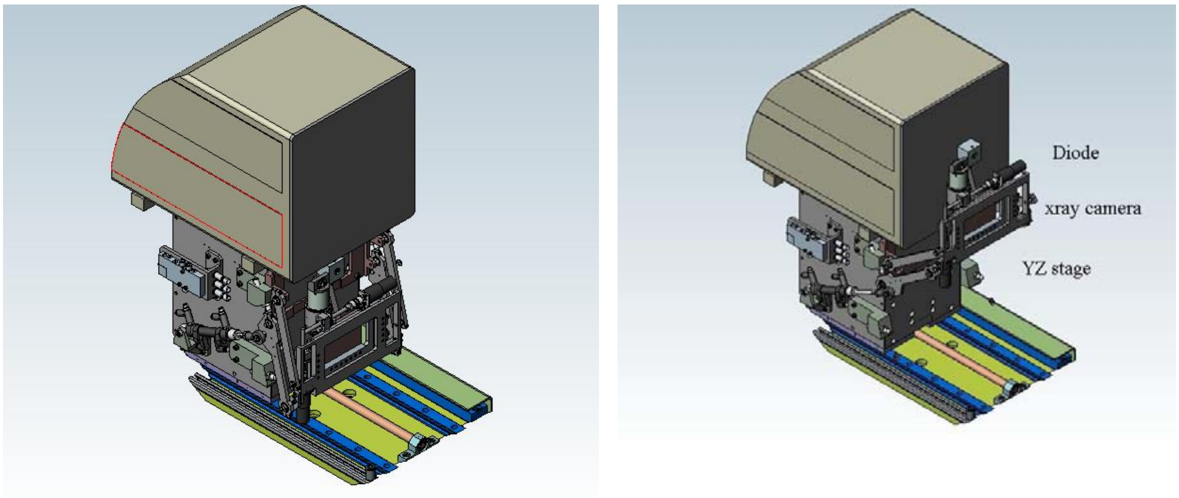


Figure 10 reflected beam mirror well aligned and when the yaw is deliberately misaligned by 0.5 mrad

5.2.2 Xray Camera at sample position

Xray cameras [2] have been used for some time at ESRF and elsewhere for monitoring and optimising the focus on various beamlines. However, its field of vision is relatively small, 2mm x 2mm, so it is always necessary to mount the camera on a motorised YZ table in order to align the camera onto the beam. The measurement of the focus at the sample position means that this instrument needs to be inserted into the beam for verification of the focus, but it needs to be withdrawn for taking data. Experience has shown that if this task is not easily and quickly achieved then the focus is rarely checked and optimised. On ID23 and ID29 at ESRF this xray camera has been mounted on a folding support mounted below the detector.



The support itself contains the YZ motorisation to align the camera to the beam.

Figure 11 Mounting of xray camera in front of the detector

6. The sample environment and checking the beamline is working

ID23 has been designed to easily check the quality of the xray beam as it is delivered to the sample. All diagnostics are permanently installed so that there is no time wasted in setting up to check the focus or the quality of the monochromator calibration. However, all the above checks are either working in the time domain of several seconds or are intrusive and therefore, cannot be used when actually taking data. The time of data acquisition on a MX beamline with the characteristics of ID23 ranges from 100ms to 5s. Figure 12 shows the sample environment.

In simplified terms the data acquisition consists of rotating the sample, which can be from 20-500microns in size, in the xray beam whilst taking a diffraction image on a 2D detector.
A typical simplified data acquisition procedure would be

- Take dark current of detector
- Accelerate sample to constant speed of rotation
- Open xray shutter at 0degree (for example) and close at 1degree whilst taking image on 2D detector
- Decelerate sample
- Repeat several times
- Repeat but between 1 and 2 degrees
- Etc

During this short data acquisition process a number of factors can affect the quality of the data. A non-exhaustive list could be: quality of sample, stability of the xray beam, accuracy and repeatability of shutter opening, quality of rotation axis, stability of the sample in the beam. The aim of the beamline design was to diagnose any problems with the beamline equipment before a data acquisition is made, and also, to monitor the quality of the beamline during data acquisition. Any problems with data quality can then be more easily analysed. Two diagnostic systems are or will be implemented.

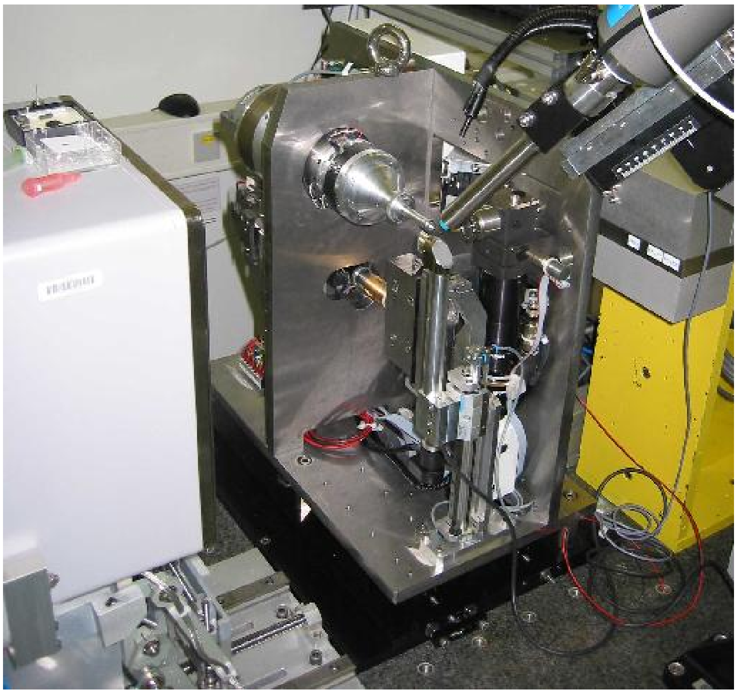


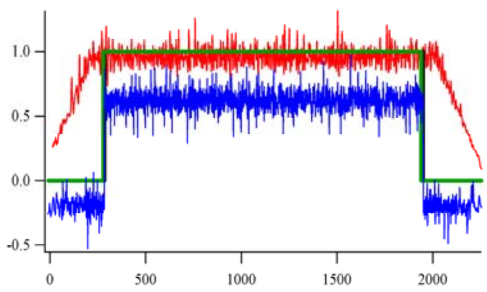
Figure 12 the sample environment of ID23

6.1 Rotation speed and shutter synchronisation analysis

The quality of the rotation axis and the synchronisation of the fast shutter with the rotation axis are critical. The fast shutter should open and close at the desired position and be repeatable in the millisecond range. These components have often been blamed for poor quality of the data in the past. It is, therefore, important that the quality of this synchronisation is monitored. At ID23 four signals are systematically recorded every time an image is taken.

- Encoder position of the rotation axis
- Status of the command to open the shutter
- Actual status of shutter (open or closed read by an infrared probe)
- Measurement of beam intensity after the shutter from a scatter diode.

These signals are entered into a synchronisation unit, which controls the fast shutter. The time between measurement points, which this unit takes, is a function of the acquisition time of the image.



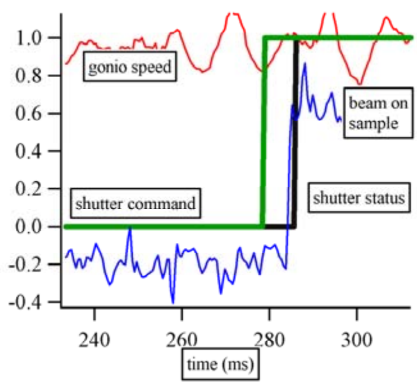


Figure 13 data attached to each image

The smaller the acquisition time is the smaller the time between the points. Figure 13 shows an example where there were 3955 points and the acquisition time was 2.2 seconds. It can be seen from this data that the fast shutter opened 7.5msec after the command was given, the beam arrived as the shutter opened and that the command for opening the shutter was given after the rotation axis had arrived at the correct speed. This particular acquisition was made at low rotation speed and low intensity and, hence the poor signal to noise ratio of both the speed and the beam intensity. This information is attached to the data that the user analysers. If data quality is poor then the quality of the synchronisation can be verified for each image.

6.2 Vibration analysis

Another possible cause of poor data quality is beam instabilities. An instability could be either in position or in intensity. If data is taken in 100msec, it could be sensitive to beam instabilities with a frequency of up to 100Hz. On ID23 the vibration levels have been measured on the 3 major optical components (Monochromator, mirror, sample table). However, this was a one off measurement and the day to day evolution of beamline equipment and beam instabilities is not routinely monitored.
It is proposed to install a system that will allow an on-line monitoring of both beam instabilities and vibrations on equipment. See figure 14.

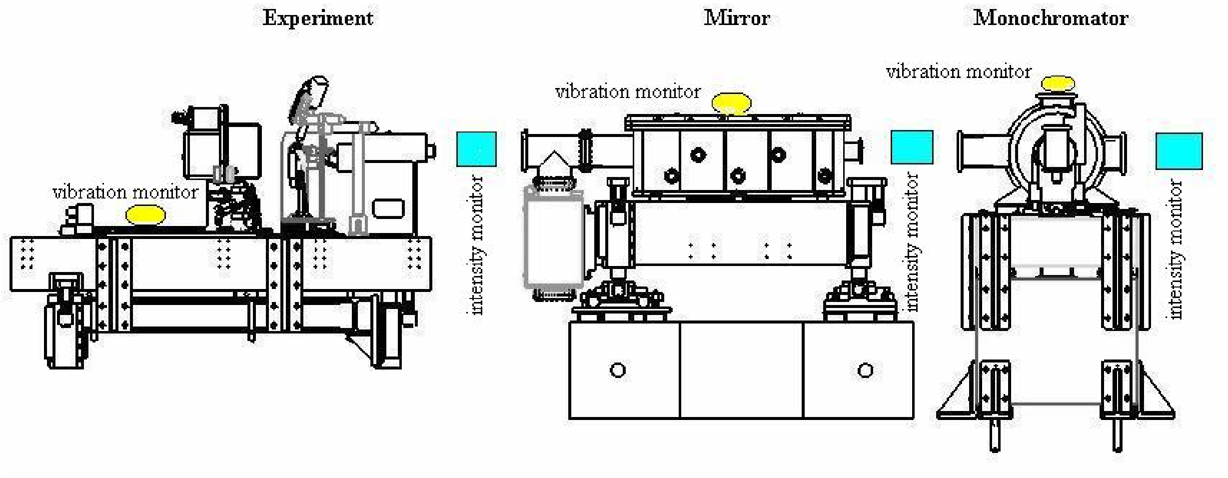


Figure 14 layout of intensity monitors and vibration monitors

The intensity monitors are the scatter diodes that are mounted in the white beam viewer and the monochromatic beam viewer. Their signal is sensitive to both positional changes of the beam and intensity changes. The scatter foil used is Kapton, so they are almost transparent to the beam. Vibration monitors will be attached to the critical optical element so that a correlation can be made between intensity variation and the optical component. This should mean that problems can be pinpointed and remedied more quickly than at the moment.

7. References

[1] High resolution x-ray absorption spectroscopy with absolute energy calibration for the determination of absorption edge energies. Kraft, J. Stümpel, P. Becker and U. Kuetgens. Rev. Sci. Instrum 67(3) March 1996
[2] http://www.seso.com/pub/X%20Ray%20Beam%20Monitor.pdf