Online correlation of data quality and beamline/beam instabilities

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Abstract

The appearance and causes of poor quality data on ESRF beamlines is still unclear. Sources of poor data quality have been attributed to vibrations in the monochromator and mirror vessels, as well as due to instabilities of sample itself either due to its mounting or from sample environment considerations. There are also suspicions that the machine itself occasionally performs erratically in a way that is invisible to the machine group sensors.

When problems occur it is often difficult to trace back the origin of instabilities and solutions cannot be immediately found without stopping the experiment and requesting vibration analysis equipment to be installed. By the time this has been done the instabilities have often disappeared.

Vibration analyses have been completed on the monochromators and mirrors of various beamlines, and in certain cases the vibrations on these elements could be directly correlated with instabilities in the x-ray beam intensity. However, it has not been possible to perform these measurements whilst taking data, so no direct correlation between types of beam instabilities and poor data quality has been established. A measurement system is described which allows simultaneous logging of vibration data and intensity data whilst performing experiments. If data quality is suspect this system can be used for on-line diagnostic of instabilities.

1 History and motivation

The initial performance and reliability of a new beamline is critical in gaining its acceptance and popularity in the user community. In 1999/2000 the ID29 MAD beamline was constructed at the ESRF and its initial performance during the commissioning period showed data quality, as measured in the Macromolecular Crystallography community in terms of Rmerge [1], comparable with other beamlines. This method, which arises from the averaging of multiple measurements of symmetry related diffraction spots, reported values of 1.5 – 3% and were considered good enough to open the beamline to the user community. In the following twelve months the "quality" of the data taken at ID29 varied unpredictably and values as high as 10-15% were reported. As a consequence, an investigation into the causes of the poor data quality was launched between October 2000 and July 2001.

1.1 Symptoms

Taking data at a modern MX beamline has become extremely standardised and one user will be performing the same tasks as the preceding user. Between one user group and the next the two variables that change are the crystal under study and the human aspects of care in data collection. However, it was noted that the same user could take data on the same crystal on two different days and get values for Rmerge varying from 3% to 10%. Even expert users were unable to get consistently good data. The beamline hardware in this type of case was understandably suspected of being the cause.

Many things can affect a diffraction experiment of this type. The most likely were considered to be:
- Non-uniform rotation speed of spindle axis
- Poor shutter and spindle synchronisation
- Unstable sample position with respect to the beam
- Poor sample
- Unstable beam intensity and/or position

1.2 Action Taken

During this period each of the above causes was investigated individually by trying to deliberately simulate a misfunction of a particular piece of equipment and see its effect on data quality. Not surprisingly each of the above possible causes did, in reality, cause poor data quality when the equipment was not working correctly.

An interesting aspect of these tests concerned the beam stability and its correlation with optical elements on the beamline. One day of beam time was devoted to measuring the intensity of the beam at the sample point for varying sample slit openings and also the vibration levels on various components along the beamline. Figure 1 shows a typical frequency spectrum of the intensity of the beam at the sample point.

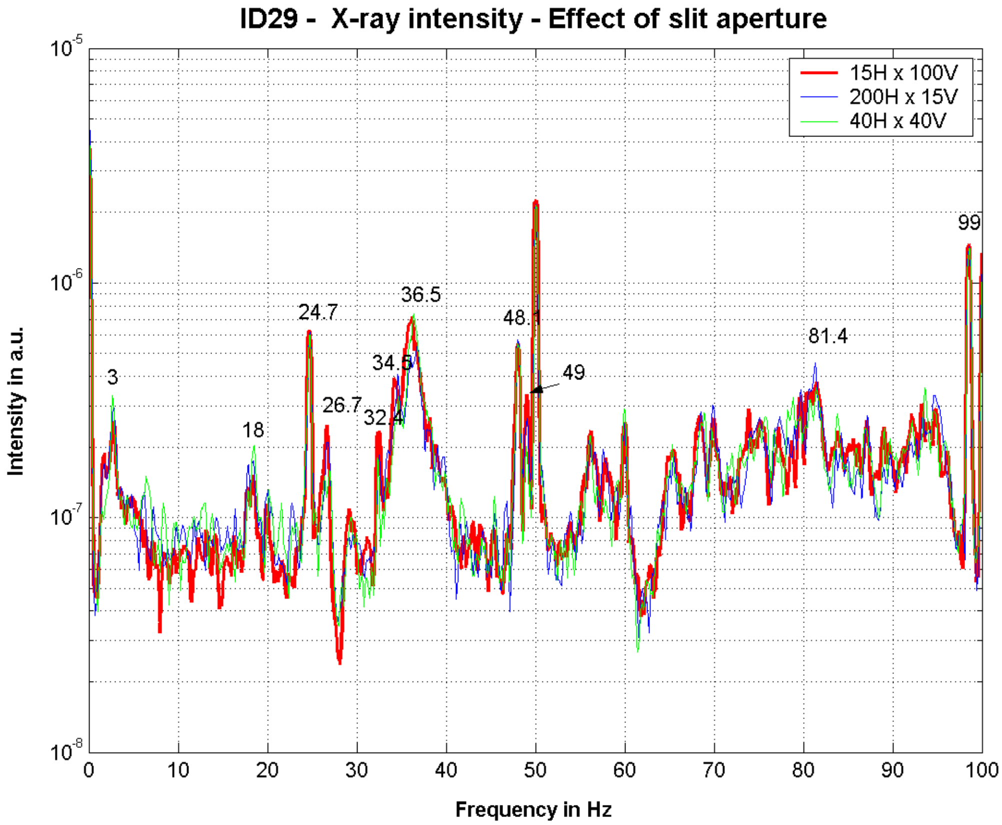

figure1 Frequency spectrum of x-ray intensity at the sample with different sample slit apertures

The intensity was then compared with the velocity of vibrations on the various optical elements. Figure 2 shows the intensity of the x-ray beam and mirror vibrations on the same graph. The 18 Hz vibration on the mirror can be seen also on the x-ray beam intensity.

This is done with the other elements along the beamline and expressed as a correlation factor. In figure 3 it can be seen that above 30 Hz any vibration that is detected on the monochromator is directly seen on the x-ray intensity. It can also be noted that at 18Hz both the mirror and monochromator have an effect and also to a small extent the table.

It was suspected that a possible cause for poor quality data was the possibility of the three elements mono, mirror and experimental table creating resonant pulses around 18Hz under certain variable conditions. Action was taken to shift the resonant frequency of the mirror to a higher value of 30Hz by linking it via damping links to the storage ring tunnel wall. The experimental table was also stiffened and its resonant frequency shifted to 21Hz.

In addition all sources of vibrations (rotary pumps, chillers etc) were removed from the experimental hutch. Users were also advised on how to take data on the ESRF beamlines. [2].

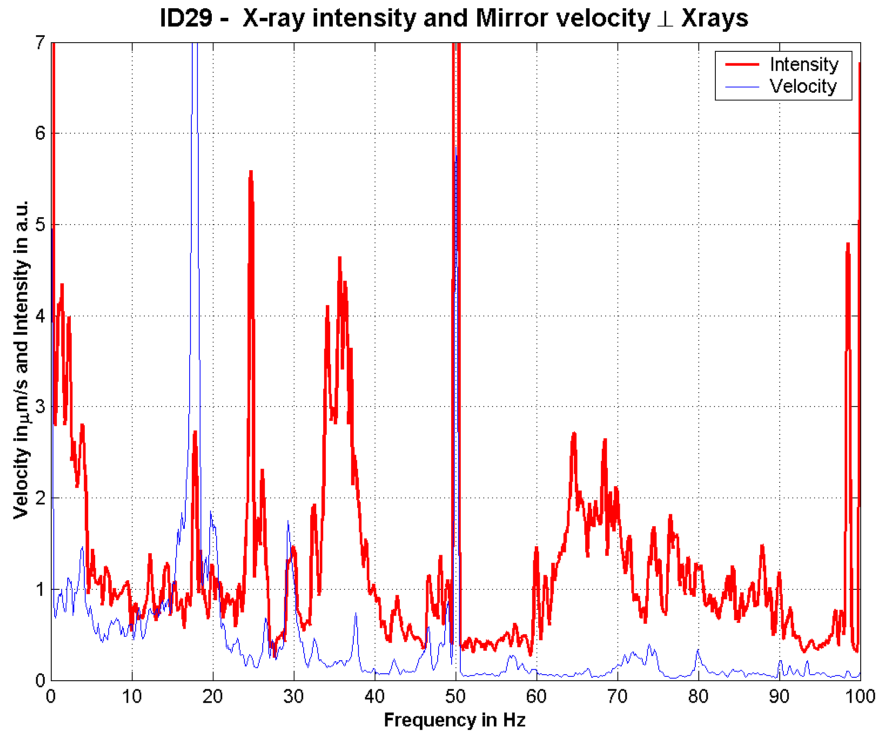

figure 2 x-ray intensity spectrum and mirror velocity spectrum

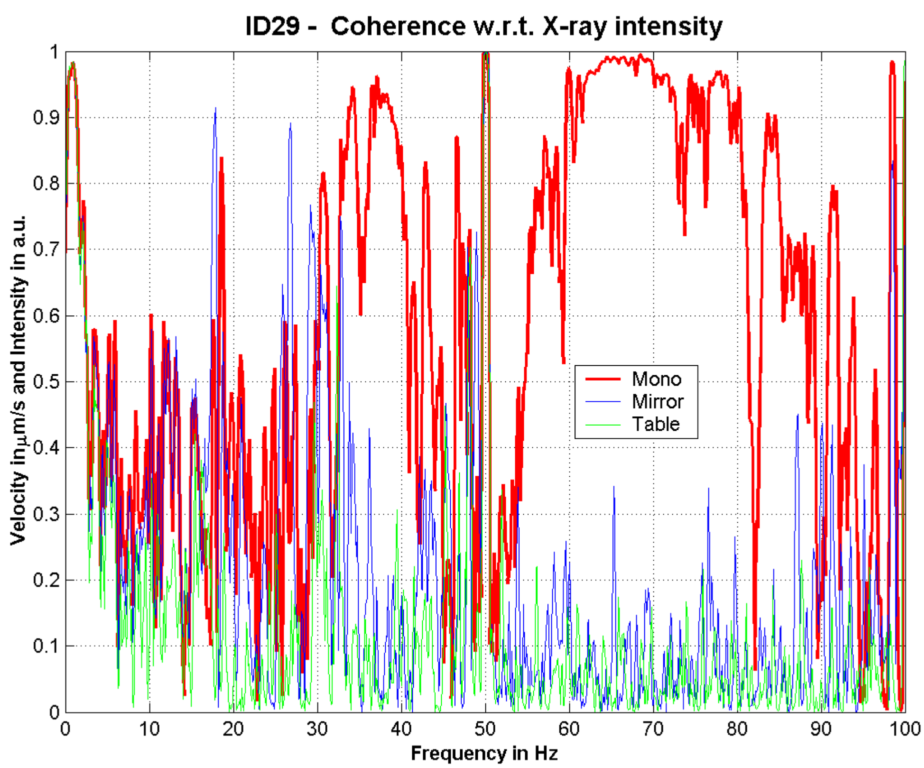

figure 3 Correlation between x-ray intensity and velocity spectrum of optical elements. A value of 1 indicates exact correlation

1.3 Conclusions drawn from this experience

After twelve months of investigations and adjustments on the beamline the data quality stabilised at an acceptable level. However, it is not known which improvements that were made were the ones that really counted. This is due to the fact that all measurements of equipment were taken off-line. This work would have been a lot easier if it had been possible to measure all of the conditions of the beamline during data collection. It was therefore decided to try and integrate as much diagnostics into the beamline acquisition software as possible.

2 Proposed Integrated System

Of the possible causes indicated for poor quality data the three below can be monitored without disturbing the data acquisition. This information is retained such that if a user has poor quality data it is possible to look back in the beamline log to verify that the beamline itself was functioning correctly.

- Non-uniform rotation speed of spindle axis
- Poor shutter and spindle synchronisation
- Unstable beam intensity and/or position

2.1 The proposed Integrated System

For the beamline it is important to know what is happening during each image that is taken, typically less than one second, and over the whole data set, typically 4-10 minutes. In addition it is useful to know what has happened over the last few days. This is dealt with by two distinct sets of instrumentation. The diagnostic of what is happening during each image is dealt with using an ESRF proprietary electronics MUSST [3]

A separate system is used to continually monitor vibrations and intensities. The data can be read in real time or archived for approximately 2 weeks

2.2 Acquisition Electronics

The requirement to be able to monitor an analogue signal for long periods of time and with a sampling time fast enough to allow us to analyse the signal in a frequency range of 0 -100 Hz is identical to seismic recording instruments.

ESRF has recently upgraded its seismic recorders for the machine and therefore, an identical instrument was purchased for these tests. A six channel broadband seismic recorder from Reftek [4] was selected. Its main interesting characteristic are:

- Analogue input 20 Volts pk to pk
- 24bit
- gain x1 or x100
- sampling rate 0.1/s to 1000/s
- communication ethernet

2.3 Vibration Sensors

ESRF has seen that the beam intensity is more sensitive to horizontal vibrations than in the vertical direction. Therefore, it was decided to monitor only in the horizontal direction. There is always a certain amount of crosstalk between vertical and horizontal modes so even vertical mode frequencies would be seen on the horizontal sensors. A Sercel L4C seismometer was chosen [5]

2.4 Intensity Monitors

The intensity monitor has to be transparent as it must register the intensity whilst the beamline is in operation. It should be sensitive to position as well as intensity although there is no need to calibrate the signal to a specific position change as it is the frequency of the perturbation that are analysed. Therefore, a scattering foil of carbon based foil where used. A 250 micron foil of pyrocarbonne was used for the white beam as in needs to cooled. Kapton foils of 100microns were used for the monochromatic sensors.

A silicon diode (Hamamatsu S1223 with window removed) is positioned at 45 degrees to the scattering foil. Figures 4 and 5 show the two configurations.

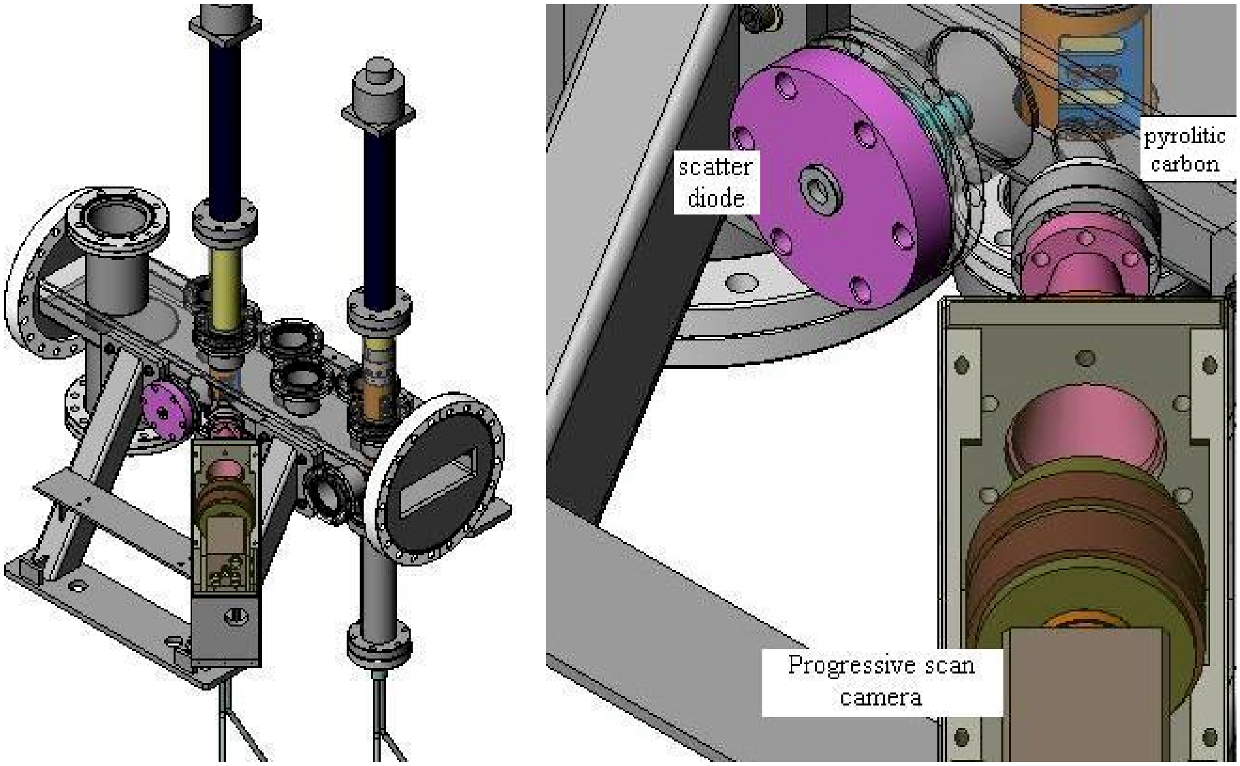

Figure 4 White beam intensity monitor and viewer

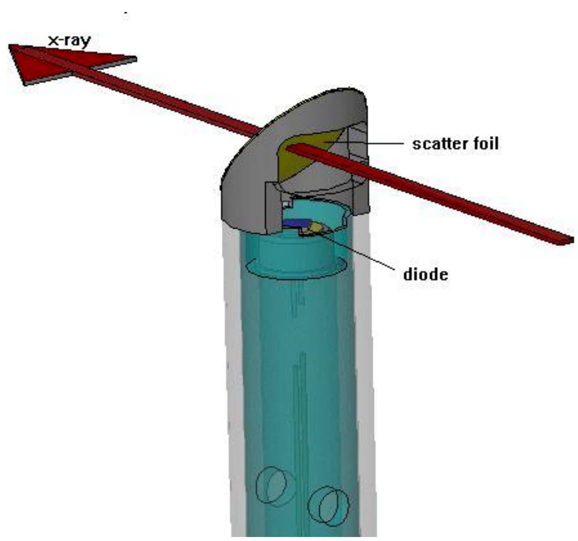

Figure 5 Monochromatic beam intensity monitor

2.5 *The Software*

The acquisition electronics is delivered with software that can run on Windows or Linux which allows the real time monitoring of data. It also allows the recovery of data archived by means of FTP. At ESRF the data is stored for 2 weeks. This allows for the investigation and analysis of problems reported by users after processing their data. The software can display the raw data and also has the possibility of displaying frequency spectra for selected signals in selected time windows. The figures shown in this paper are images of displays from this software. The data can also be analysed using Matlab to produce correlation data as in figure 3.

3 Integration into the Beamline

The system has been installed on the new MX beamline ID23. ID23 is a two end station beamline installed on a straight section equipped with canted undulators[6]. Initially the system was installed on the tuneable Mad station, it was then transferred to the fixed energy micro-focus side station(ID23-2). The data presented here comes from ID23-2. The beamline is simple and consists essentially of (in the direction of x-ray beam)

- Front end including diamond window
- Primary slits
- White beam intensity monitor
- Single bounce side reflecting LN2 cooled silicon 111 monochromator
- Secondary slits
- Monochromatic intensity monitor
- Optics hutch beamshutter
- KB mirror pair 300mm-300mm
- Sample slits
- Monochromatic intensity monitor I0
- Millisecond shutter
- Sample
- Detector

The seismometers were mounted on what was considered the three most sensitive items, the monochromator, the KB mirror support and the experimental table. Particular attention was paid during the design process to the vibration characteristics of the supports for these instruments. The monochromator and KB are mounted on concrete blocks, whereas the experimental table is mounted on a steel framework. The intensity monitor just before the sample is mounted on the experimental table and hence moves with the table when it is aligned during the experimental procedure. It is also after the optics hutch beamshutter and therefore, is subject to interuption of the beam when it is necessary to take a dark current for the detector.

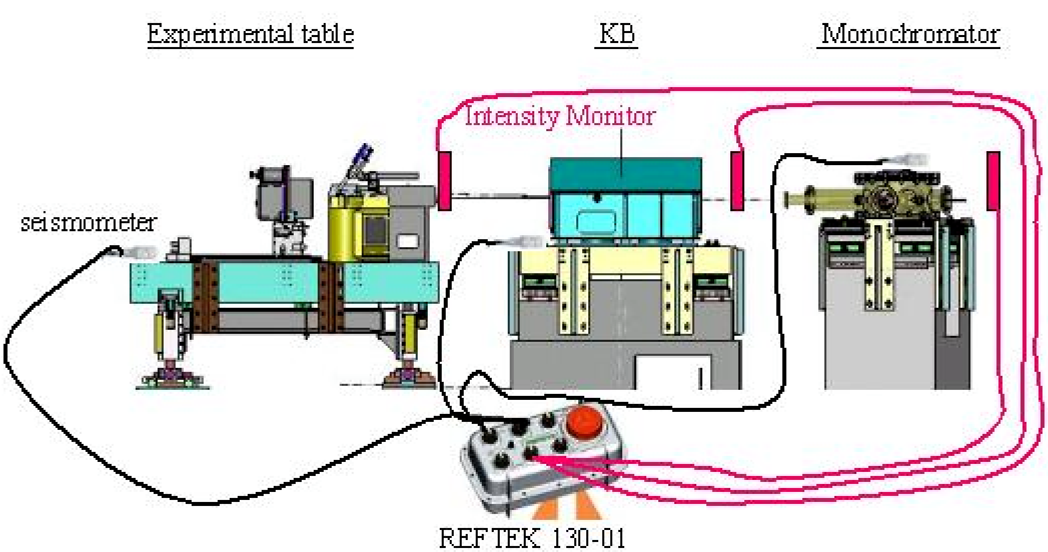

Figure 6 Implantation of the system on ID23-2

4 Results during normal operation

The system was conceived to help in diagnosing problems which may occur on the beamline. In order to do this it is necessary to have a standard performance for the beamline to which abnormal condition can be compared.

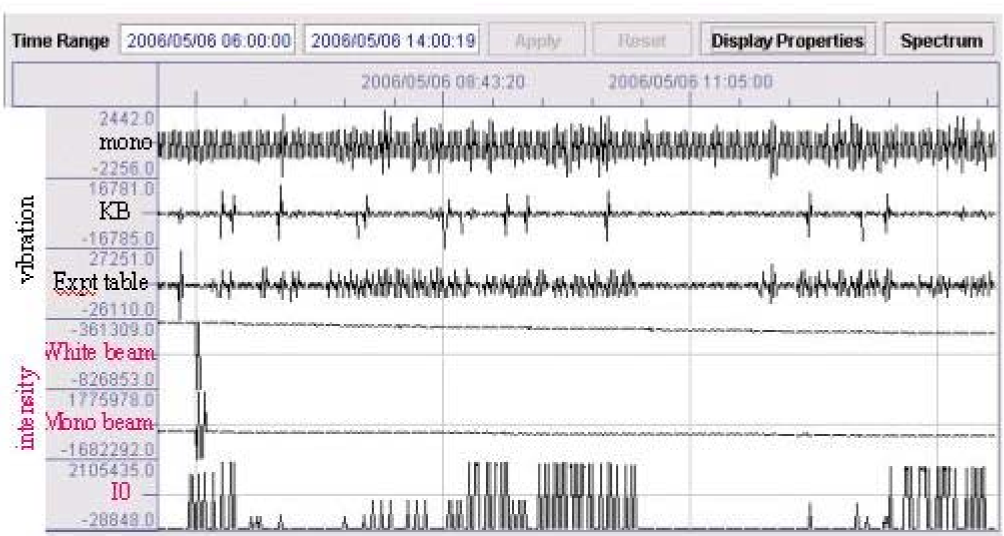

Figure 7 Typical shift (8 hours) at ID23-2

Figure 7 shows a typical shift at ID23-2. The seismometer on the monochromator picks up some occasional disturbance, but all of limited amplitude. The seismometer mounted on the KB show some occasional strong disturbances whereas the one on the experimental table picks up many smaller disturbances. These latter two are related to users entering into the experimental hutch and possibly slamming the door when leaving. The white beam and mono beam intensity monitors show the current decay in the machine with one event that was the realignment of the beamline. The intensity before the sample I0 has many steps each one corresponding to the acquisition of a data set. A zoom can be made of this area and is shown in Figure 8 and actually during data acquisition in Figure 9..

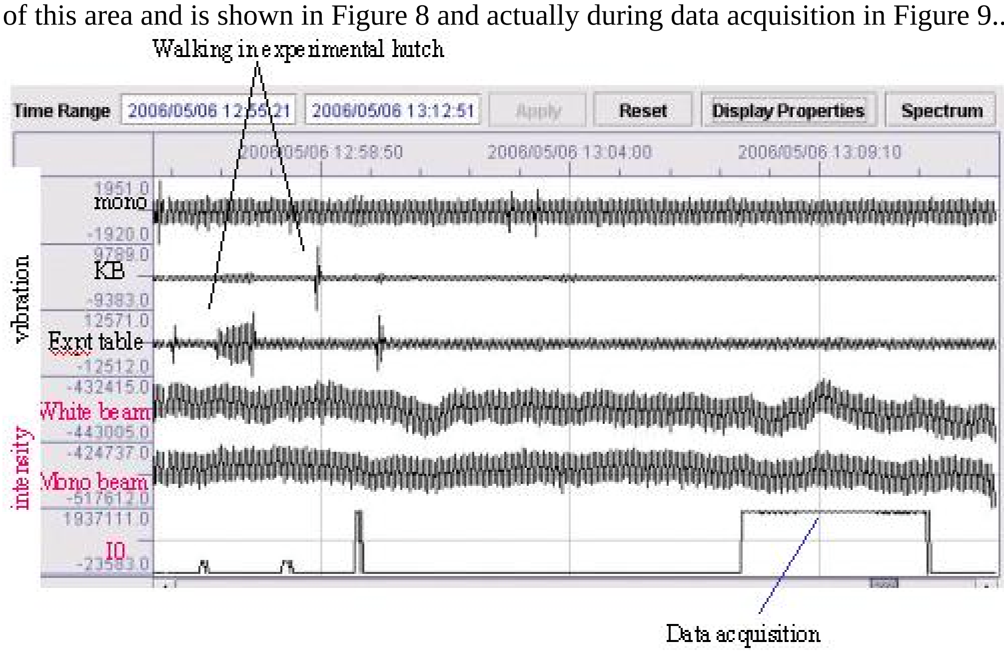

Figure 8 Zoom around a data acquisition period.

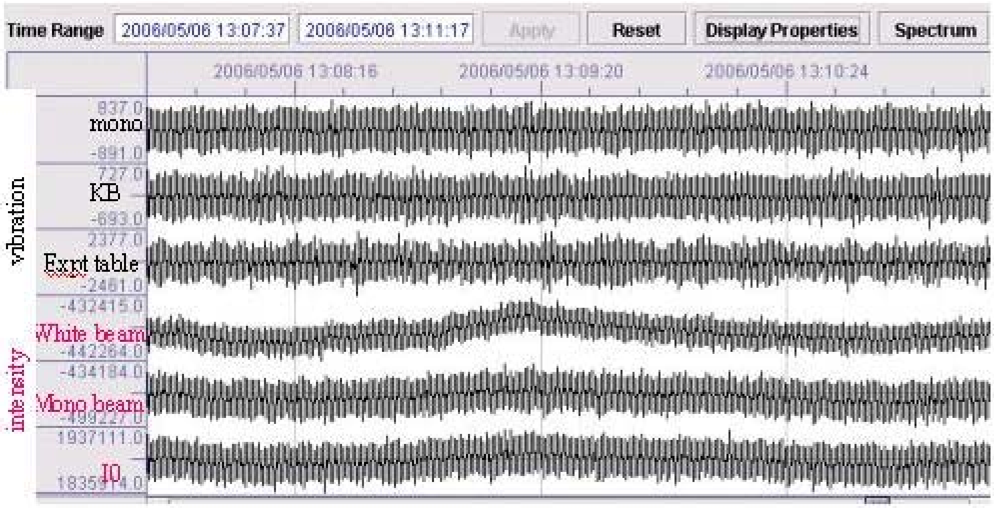

Figure 9 Intensity variations and vibrations during data acquisition

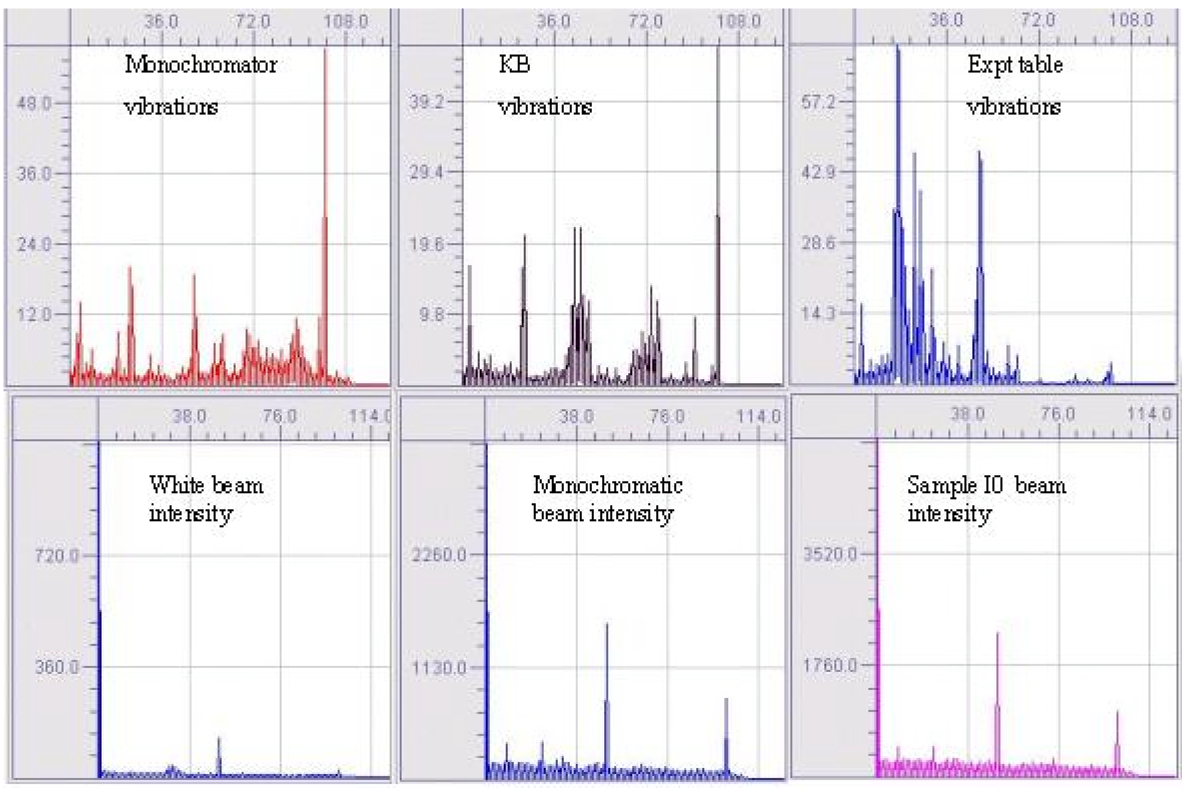

Figure 10 Power density spectrum of the six monitors during data acquisition.

If the intensities and vibrations are analysed during the data acquisition as shown in figure 10 it can be noted that the vibration levels on the experimental table at low frequencies of less than 30 Hz are relatively important compared with the monochromator and KB. There were vibrations on this particular day with a frequency of 23Hz on all three supports and although at a very low level this 23Hz can be seen on the monochromatic and sample I0 intensities but not at all on the white beam intensity. In this particular case where the beamline was working well the current decay (frequency less than 1 Hz) was far more important than any other effect. The 50Hz and 100Hz signals are assumed to be parasitical.

5 Results during non-optimal conditions

The system has been used to investigate the performance of ID23-2 under exceptional conditions when the performance of the beamline was not optimal in one case and also during topping-up tests of the ESRF storage ring.

5.1 Beam drifts

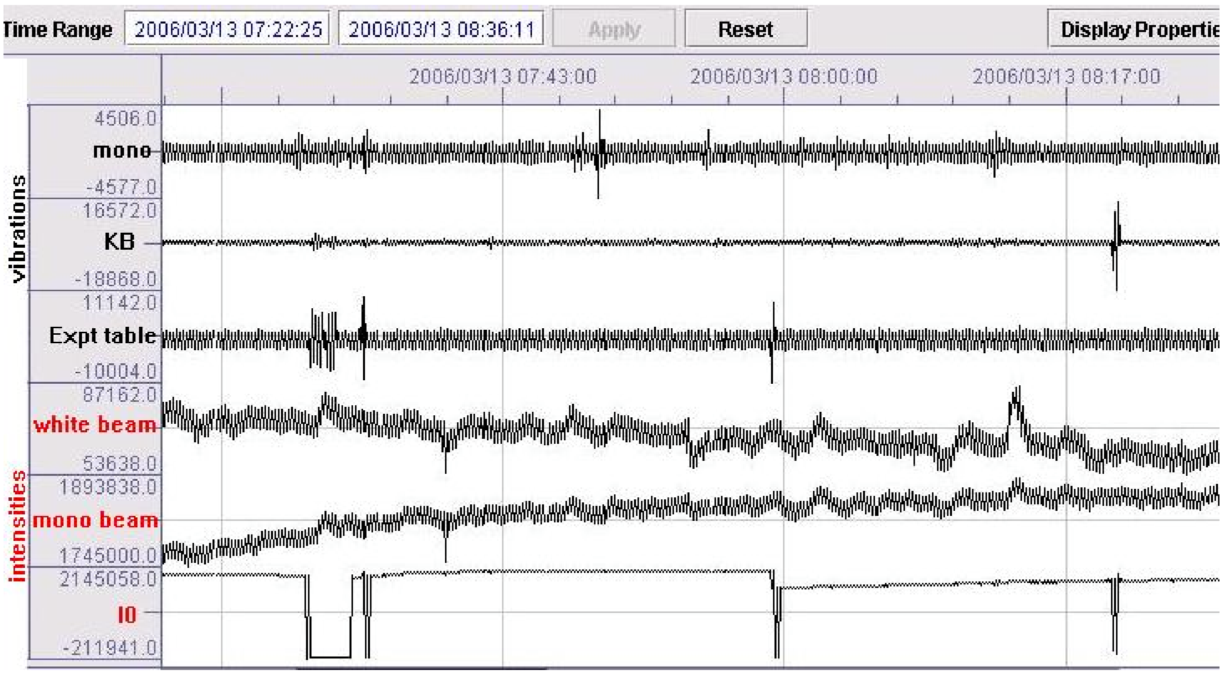

Figure 11 Drifts of beam intensity

Figure 11 shows one morning when the users complained that the beam was drifting at the sample in the order of magnitude of 50 microns during a period of 1 hour. As this data is archived it was possible to observe that there were indeed drifts of the beam. In the short term (1-2 minutes) the intensity of the beam after the monochromator follows that of the white beam, but over one hour the intensity of the monochromatic beam did not decay in the same way as the white beam. This indicated a problem with the monochromator.

5.2 Topping-up

A number of 3rd generation synchrotron sources operate a "topping-up" mode where electrons are injected into the storage ring periodically in order to keep the storage ring current at a constant level. The advantage is that the beamline optics do not change temperature and as a consequence beam drifts should be smaller. ESRF has never operated this mode, but is considering doing so. In order to evaluate this mode some initial tests have been made to look at the effect on data quality. Already the injection process is clearly seen on the intensity/position monitors. The huge variation in signal is certainly due to beam movements as the injection bump is activated. Figure 12 shows a typical injection cycle which occurs in the middle of a data acquisition period of 4 minutes. A zoom of the perturbations show that they last for about 40 ms and a frequency analysis shows that they occur at 10Hz which is the frequency of the injection bump.

During this period data sets were taken on a trypsin crystal with 1degree/1 second exposures/90 degrees of data. Comparisons were made between a period of storage ring operation with normal decay and a period of storage ring operation in this topping-up mode. Unfortunately for these tests the quality of data

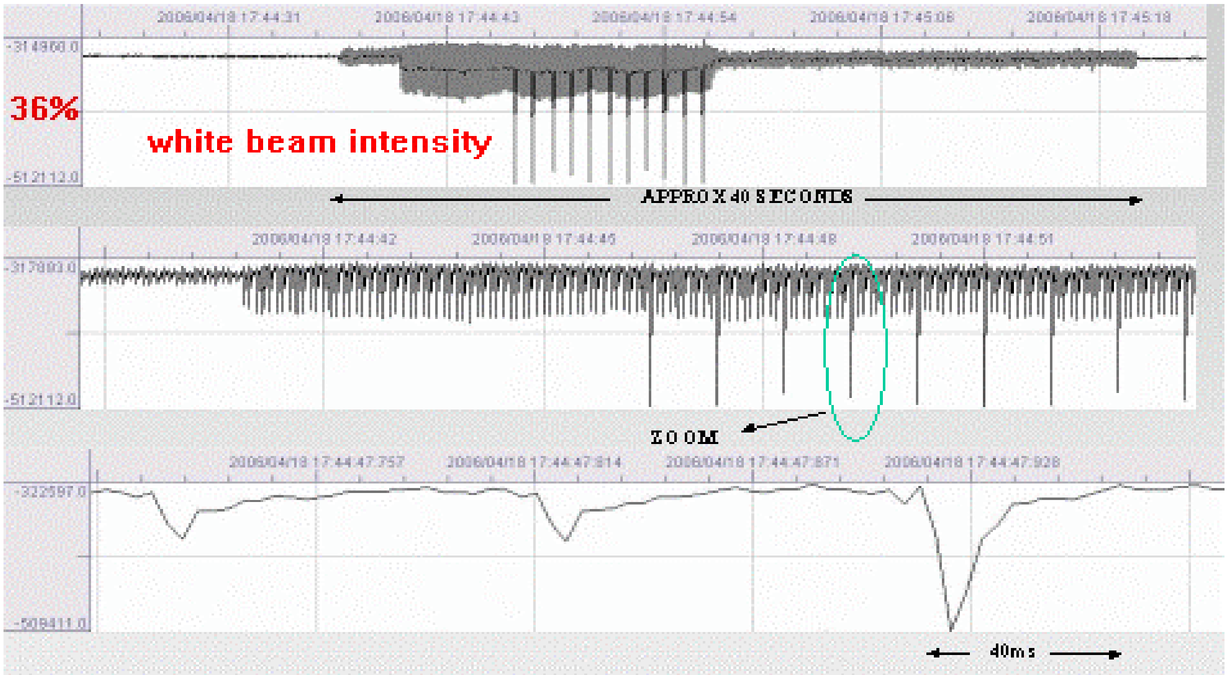

Figure 12 Injection of electrons in topping-up mode

in all periods was not high enough to draw any conclusions. Rmerge of 7-8% was achieved in both modes. This may have been due to poor experimental procedure or poor performance of the machine as there appeared to be some abnormal instabilities due to the global feedback not functioning correctly.
The system is clearly able to detect the rapid perturbation on the beam during injection of electrons into the storage ring. The tests need to be redone with careful control of the experiments in order to establish whether these perturbations have an effect on the data quality.

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References

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