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The non-crystalline Diffraction beamline for Diamond – An Update

N.J. Terrill[1], A.F. Grant[2], A.R. Marshall[1], A.D. Smith[2], K.J. Sawhney[1],

[1] Diamond Light Source, Rutherford Appleton Laboratory, Chilton, Didcot, Oxfordshire, OX11 0QX
[2] CCLRC Daresbury Laboratory, Daresbury, Warrington, Cheshire, WA4 4AD, UK

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ABSTRACT

Non-Crystalline Diffraction is an important method for studying the structural properties of non- or semi-crystalline states of matter. These include biological macro-molecules e.g. fibres or proteins and their complexes in solution, synthetic polymers, gels, liquid crystals, oils, paints, ceramics and environmental aggregates. The technique yields information on the shape and size of these molecular assemblies and is particularly sensitive to phase changes or conformational rearrangements on a length scale ranging from 10 to 10000 Å. Diamond is the new third generation synchrotron source that is currently being designed and constructed for the UK Science community. The 3GeV machine will begin operations in 2006 with commissioning of the first seven beamlines to follow. I22 is now in the detailed design phase after successfully passing through its concept design review. The science case has specified the beamline to operate at energies between 4-20keV utilising the low divergence beams from an undulator. The beamline is to include features for standard Small Angle Scattering and Fibre Diffraction together with a combined SAXS/WAXS capability. A modular Microfocus facility will also be incorporated.

Diamond is the largest scientific facility to be built in the UK for nearly thirty years. The facility when operational in 2007 will produce highly focused high brightness beams that will enable scientists and engineers to probe deep into the basic structure of matter and materials, answering fundamental questions about everything from the most sophisticated structures in a cell to the origins of our planet. State-of-the-art beamlines will maximise the effectiveness of the source, ensuring that researchers from the UK and abroad have access to cutting-edge analytical techniques and services for the next twenty years or more. Such fundamental research is at the heart of the competitiveness of the UK economy.

There has been strong pressure to provide the non-crystalline Diffraction community with a world class facility at the earliest possible point in the project. I22, the identifier for the non-crystalline Diffraction beamline, is being designed to meet those broad ranging needs.

The UK has an active community that has been at the forefront of developments in this field for many years. Community interests, as identified in the responses to the announcement for this beamline and in earlier consultations, encompass the fields of medicine, biology and materials processing. Interests include; studies of supramolecular organisation in biological systems, the structure and function of muscle filaments, elastic proteins, bone nanostructures, corneal transparency, biological membranes, polymer processing, self-assembly of mesoscopic metal particles, colloids, ceramics, environmental nanoparticles, barrier materials, mineral colloids, liquid crystals and nanoscale devices. The work is supported by the BBSRC, EPSRC, MRC, NATO, NERC and the Wellcome trust. Non-Crystalline Diffraction is one of the truly synergistic interdisciplinary sectors within UK science and the wider international arena.

At present, the programme is supported by three beamlines at the SRS, as well as others at the ESRF including ID02, ID13 and DUBBLE. The ESRF does not have the capacity to take up the expected demand after the closure of the SRS or to provide the variety of environments that will be needed to advance our knowledge of behaviour of the wide range of biological and materials systems that will be studied.

The non-crystalline Diffraction beamline will bridge the gap between optical and more conventional X-ray measurements. It will play an important role in developing our knowledge of macromolecular structure into function and into advancing the industrial development of biomaterials, biosensors, and lead to the manufacture of better designed polymers and a more efficient production technology. The beamline will represent a facility at the cutting edge of synchrotron technology that will provide a robust SAXS service for an international community. The design of the beamline also looks to the future challenges of SAXS where the objectives will be characterisation of larger and more transient structures.

Insertion Device - A consultation with the user community to gauge expected energy requirements is reproduced in Figure 1. The consultation clearly indicated that while there was excellent science to be performed at the extremes of the beamlines range, including Calcium edge, biomineralisation, bioceramics and food science at 4keV and high pressure studies above 16keV, the main focus of the work to be carried out on the non-crystalline Diffraction beamline will be between 10-12keV. This is also the experience of Beamline Principals at other third generation sources, including those from ID02 and DUBBLE at the ESRF and BIO-CAT on the APS.

A request was therefore made to ASTEC to design an insertion device with low divergence and high flux which delivered photons continuously over the energy range 4-20keV and gave good performance between 10-12keV. The requirements resulted in ASTEC supplying three options to consider (Figure 2).

Further analysis of the undulator performance suggests that a U25 in vacuum device gives the best overall performance. The 7th harmonic of the U25 device covers the primary range requested whereas to cover the same range with an ex-vacuum

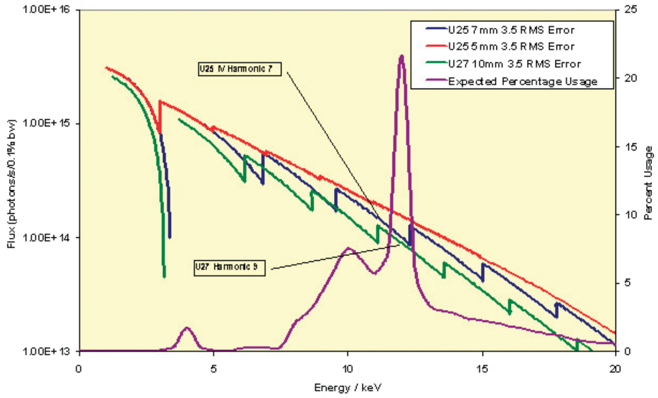

Figure 1. Undulator Options against Expected Energy Demand for I22

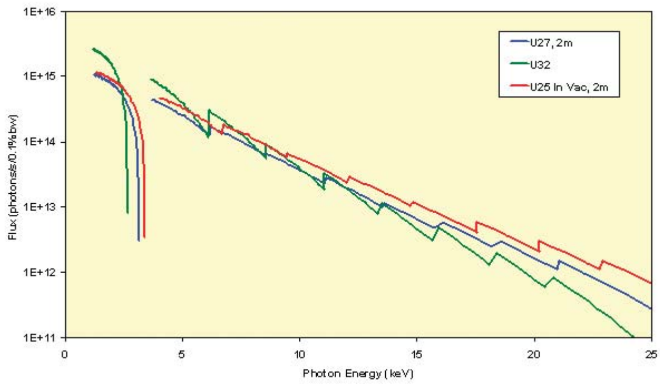

Figure 2. Undulator Options for I22

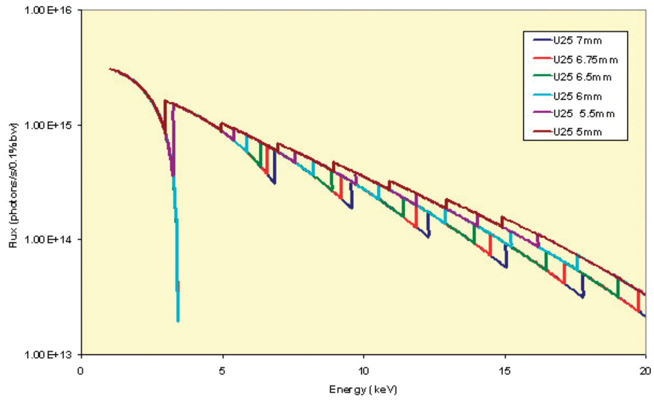

Figure 3. Effect of gap reduction on U25 IV Undulator.

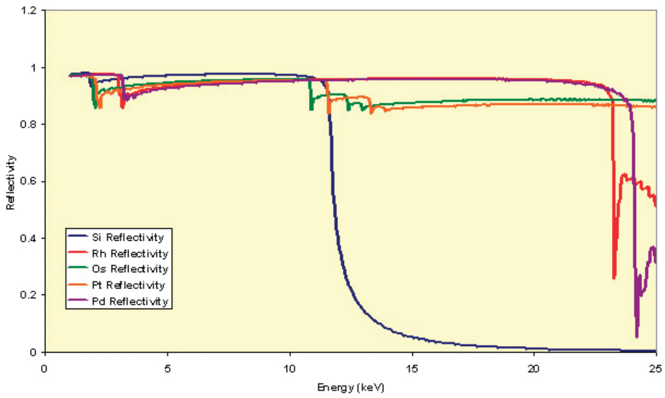

Figure 4. Reflectivity of different mirror coatings for a mirror inclined at 2.6mrad

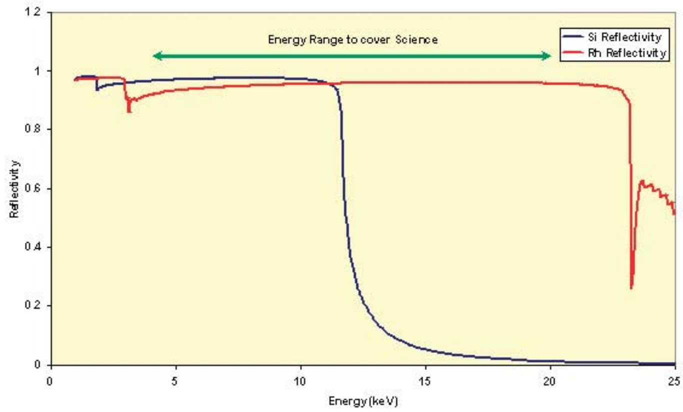

Figure 5. Reflectivity for a bare and Rh coated mirror (Rh layer 30nm thick) at grazing incidence angle 2.6mrad.

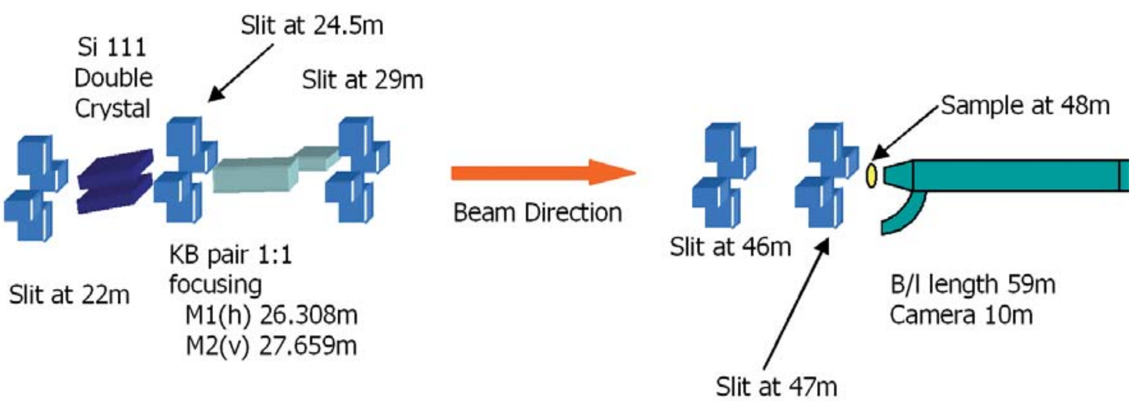

Figure 6. Outline layout of I22

U27 device use of the 9th harmonic is required. Real undulators have magnet phase errors associated with them (Figure 2). These degrade the performance output of the undulator and affect the higher harmonics more than the lower ones therefore working with lower harmonics is always preferable.

As the ring vacuum conditions it should be possible to reduce the gap of an in-vacuum device further, from 7mm to 5mm and possibly even narrower, thus giving additional performance gains (Figure 3).

Outline Optics Scheme - Flexibility is the key to the design of the optics as this beamline has to cater for a wide range of scientific challenges. For this reason interchangeable and adaptable components will be necessary.

Monochromators - The scientific case has identified anomalous small angle X-ray scattering (ASAXS) to be an important option. Such measurements call for good energy resolution, therefore a double crystal Si(111) monochromator (DCM) ($\Delta E/E$ 2 x 10^{-4}) will be included in the beamline design. There are other parts of the programme that call for less energy resolution but do call for a high flux on the sample. Macromolecular solution scattering measurements and many parts of the polymer case fall into this category. The science therefore calls for a multilayer-based monochromator to be considered where the bandpass is 1-2 orders of magnitude higher than a Si monochromator.

The monochromator will be the first optical element in the beamline located at approximately 23.5m. Having the DCM as the first optical element will allow simplification in design of all downstream elements. Mirrors and any microfocusing elements will not require cooling as most of the power will be removed from the beam by the 1st crystal of the monochromator. It is planned that the monochromator will be of a fixed exit design. This is to ensure that minimal movements of the beam occur at the sample when the wavelength is changed for anomalous (high angle non-crystalline /fibre) and microfocus work.

Mirrors - Primary focusing will be achieved with a Kirkpatrick-Baez (K-B) mirror pair; these are grazing incidence elliptical reflecting mirror pairs that have extreme surface precision. This combination has been chosen for two reasons; 1) the choice allows flexibility in the location of focus anywhere along the 10m SAXS camera and possibly in a pre-hutch for microfocus work, and 2) it allows decoupling of the horizontal and vertical focusing. This will enable experimentalists, if necessary, to split the focus between the sample (maximum photons on sample e.g. horizontally) and the detector (maximum resolution e.g. vertically).

The K-B mirror pair, located at approximately 26.3m (h) and 27.7m (v) will provide approximately 1:1 focusing into the experimental hutch at 54m. However, these positions will need validating in the detailed design in light of any further design work that may be required for a technical design review that is to take place in May 2004.

At these distances mirrors of length 1.2m (h) and 0.4m (v) respectively would comfortably collect the entire beam profile. Both mirrors will be set at 2.6mrad. The selection of coating is between palladium (Pd) and rhodium (Rh) which could both be utilised over the whole energy range of the beamline. Other common coatings have edges in the range so can be discarded (Figure 4).

The mirrors could have one coating. If, however, the mirrors are striped, i.e. only part of their surface is coated longitudinally with, for example Rh, the variation in cut-off energy between the different mirror coatings could be used to prevent contamination of the beam from energy harmonics transmitted by the monochromator. For example, with a Si(111) monochromator, if a beam of 6keV is used, then there is likely to be contamination from the Si 333 reflection coming in at (6x3) keV i.e. 18keV. However if the bare Si was used in this situation then there would be no contamination from the harmonic as Si does not reflect at 2.6mrad above ~12keV. So for routine operation between 10-20keV one could use a rhodium coated Si mirror, but for operation below 10keV one could use an uncoated Si mirror (Figure 5).

The decision on which stripe to chose in addition to the uncoated stripe will be made after discussions with manufacturers - it may be that one material is easier to handle than the other (rhodium doesn't require a binding layer of chromium) or the use of one would result in a significant cost saving (palladium is expensive).

This information leads to the following outline layout for I22 (Figure 6).

Preliminary ray tracing with 1:1 focusing at 54m, halfway along the maximum SAXS camera length, shows that a beam approximately 310 x 75mm can be expected (Figure 7). An estimate of the effect of slope errors using a simple wave oscillation has been included in the calculation of the focus spot. A more realistic model would include a randomisation effect and not produce the "dumbbell" shape illustrated below but a smoother beam profile.

Microfocusing Options - X-ray microfocus is the provision of small intense X-ray beams, using a variety of optical elements that focus rather than merely collimate the beam. The main advantages are two-fold; firstly the technique can be used to illuminate very small samples whilst minimising the scattering from the surrounding substratum. Secondly, X-ray microfocus can allow textures to be distinguished within a material on the micron and sub micron length scale. There are a number of materials science and biological investigations where the use of microfocus technology is becoming crucial for maximising the information that can be obtained on the structure and behaviour of materials. For example examining the microtextural variation of bone nanocrystallites is important in understanding bone-ligament and bone cartilage interfaces. The textural variation measured in skin-core experiments imparts information about polymer alignment and crystallinity within a single fibre.

Two options for the microfocusing capability of this beamline are currently being pursued.

Small K-B pair - This is the option chosen by the microfocus XAFS beamline on Diamond and is one of the more widespread

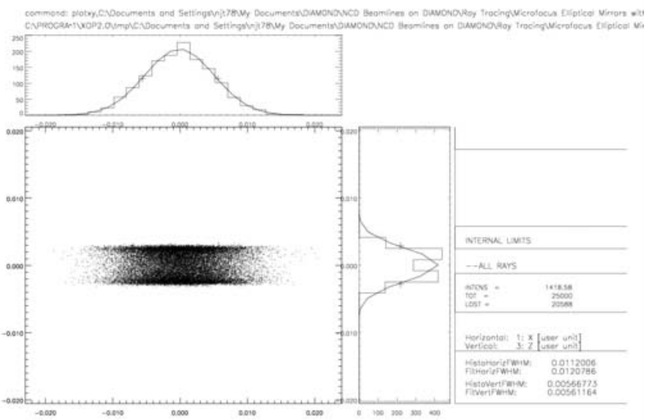

Figure 7. Shadow Output for Outline layout of I22

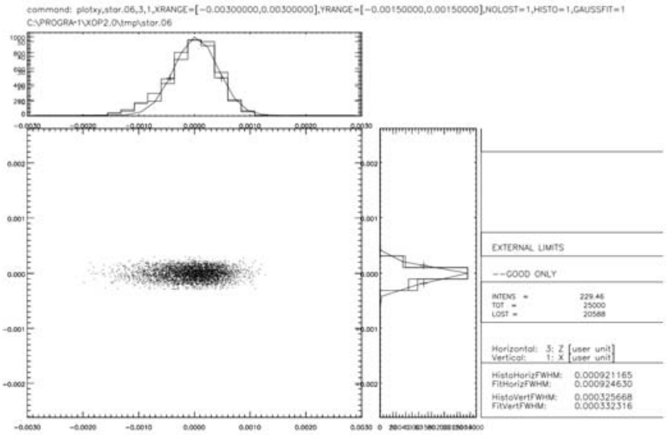

Figure 9. Shadow Output for microfocusing K-B pair for I22

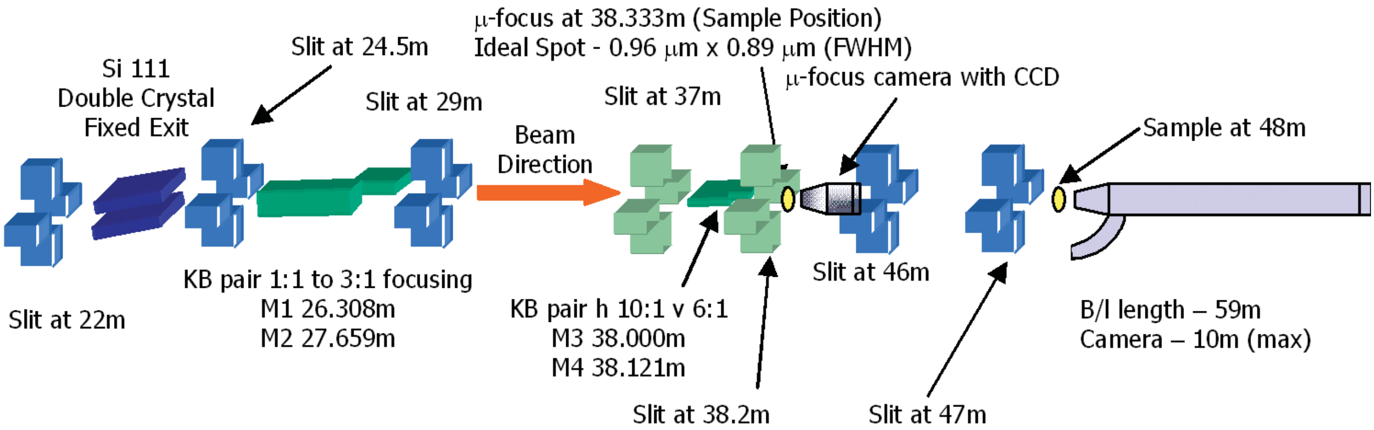

Figure 8. Outline layout for microfocusing K-B pair for I22

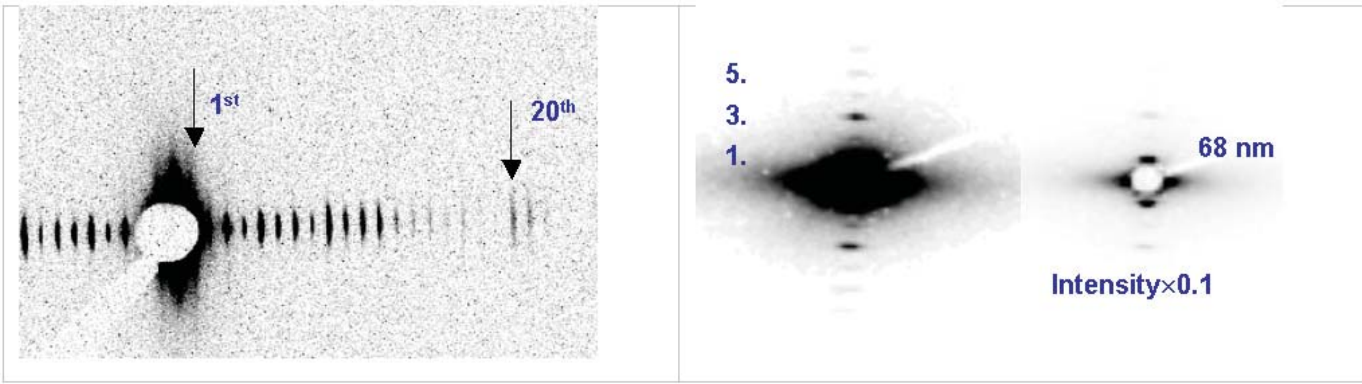

Figure 10. CRL output for collagen on ID18

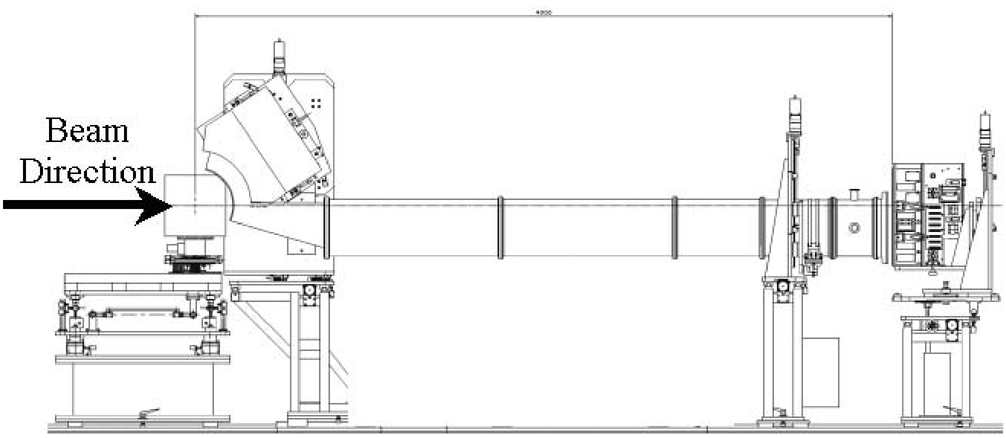

Figure 11. Possible End station layout for I22

choices now available. A number of the beamlines on the ESRF are adding this capability through their own design of small K-B mirror pair.

This option will require either a pre hutch or an extension upstream of the main experimental hutch. An outline of a possible configuration is given in Figure 8.

A preliminary ray tracing study (Figure 9) of this arrangement has been carried out using similar slope errors to those for the large K-B pair. This should give a worst case scenario for the focus. The spot size deliverable from this arrangement is of order 9 x 3 μm. Additional collimation would be required to deliver sub-micron beam sizes.

Further design work is ongoing to define the best arrangement for this optical option.

Compound Refractive Lenses - These are an attractive option if microfocusing is to be a complementary technique to the main function of the beamline. They are small, easy to align and relatively simple to maintain. There are encouraging results from the ESRF with a set of Aluminium lenses for SAXS experiments. The scattering patterns in Figure 10 show data obtained on ID18 for Collagen[i], a common SAXS standard. They clearly show the first order axial peak at 68nm.

This option would require a platform to be built to accommodate lenses, sample stage and detector. For a set-up similar to ID18 this experimental table would be located at approximately 56-58m from the source at the end of the experimental hutch.

End Station - A modular design that closely resembles MPW 6.2 at the SRS and DUBBLE at the ESRF will be considered for the end station. These are both productive stations which cater for the wide range of SAXS disciplines. The outline drawing below is taken from MPW 6.2 (Figure 11). Although not identical, I22 will share a number of features:

- All sections to be mounted from alignment rail in floor of experimental hutch.
- SAXS camera made up from 0.25, 0.5 and 1m sections to accommodate multiple camera lengths from 1-10m. This type of SAXS camera is chosen in preference to the large evacuated vessel found on ID02 at the ESRF as it enables the use of RAPID without major modification, an essential component of the detector portfolio.
- SAXS camera to be evacuated to roughing vacuum and to utilise interchangeable window materials for the variety of experiments and energies contemplated.
- Independent Y, Z mounting for front and back of SAXS camera.
- Separate mount for SAXS detector including Y, Z, pitch, yaw and roll motions.
- WAXS detector to be mounted directly to the nose cone section of the SAXS camera and given some 2θ movement.
- Nose cone to pivot about notional sample position.
- Flexible Sample platform to accommodate Y, Z, limited pitch and roll, and a rotary table mounted about the Z axis. Demountable precision X, Y, Z platform to be included for small sample environments.

Detector Options - The scientific case calls for both 1-d and 2-d capability from the detection systems on I22. Although the options will be evaluated in detail later an indication of the requirements is given below.

RAPID (for fast time resolved 2-d work) - This detector has been highlighted as the primary option for fast time resolved 2-d SAXS studies. RAPID has been tested at the ESRF on the high brilliance beamline ID02 with positive feedback from the user community. As an example of the sort of experiments which this detector is capable, included below is a muscle stretching experiment carried out over 40ms (Figure 12)[ii]. Time courses of temperature, force and intensities of M3 and A1 reflections are displayed opposite. M3 and A1 are the results of 162 T-jumps. Force and temperature are representative traces. All traces were normalised to facilitate superposition.

It must be noted that RAPID is the only detector technology currently available to this community which allows on-line data collection with sub-millisecond time resolution.

CCD (for Microfocus and Fibre Diffraction) - The science case calls for the beamline to perform fibre diffraction experiments. These experiments are not possible with the detector outlined for the fast dynamic experiments as RAPID does not have the spatial resolution capabilities for high angle diffraction. The Microfocus function of the beamline will also require a high spatial resolution detector. Here the obvious choice is a CCD where the majority of the work will be concerned with mapping samples rather than in dynamics. If fast dynamics are required then, due to the modular design of the end station and the data acquisition electronics the beamline's RAPID system could be utilised with the accepted loss in spatial resolution.

HOTSAXS/WAXS (for fast time resolved 1-d work) - To perform fast, combined, 1-D SAXS/WAXS well requires an excellently matched set of detectors. Until recently no such options were available. Now both wire and glass microstrip heads are feasible and offer high count rate capabilities >5MHz global, excellent dynamic range (107 - 108) and very fast time framing <10μs. This is again far in excess of anything possible with the latest CCD technology. It is also possible with the photon counting option to utilise the same backup electronics as will service the RAPID 2-D system thus saving on hardware costs.

The HOTWAXS detector is the subject of an ongoing project within CCLRC, the prototyping having been very successful on beamline 8.2 on the SRS and a build to cost option should be available for I22. HOTSAXS is the subject of a development project sponsored by Diamond and the Non-Crystalline Diffraction beamline. Early indications are very promising that the station can be supplied with a world leading fast 1-d detector pair.

Beamline layout - Reproduced in Figure 13 is a layout option for the Non-Crystalline Diffraction beamline. It includes provision for a microfocus hutch before the primary experimental hutch. This option is dependent on choosing a K-B pair for microfocusing. The decision will be the subject of a detailed study prior to the Technical Design Review for the beamline in May 2004.

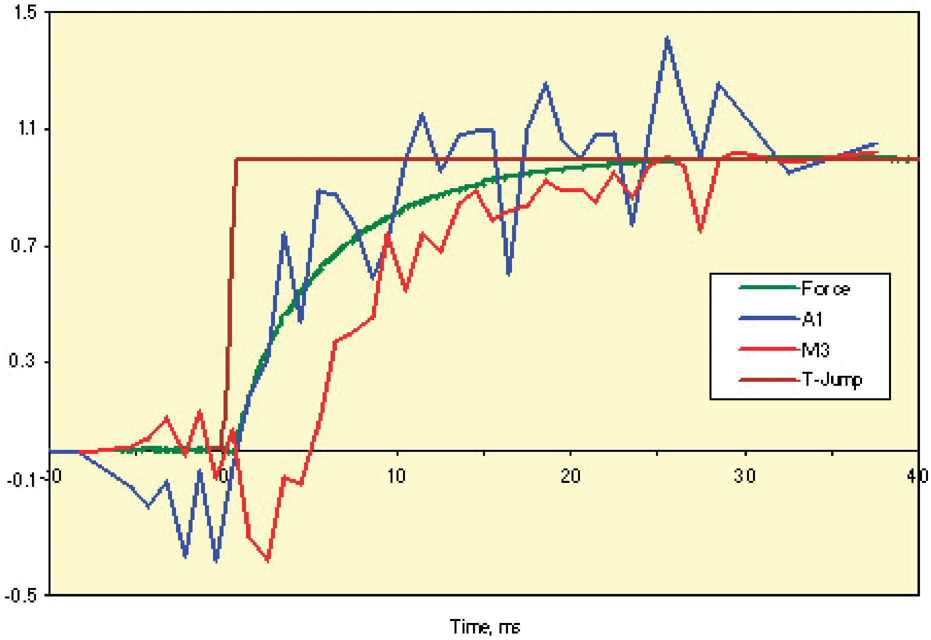

Figure 12. RAPID Results from ID02 experiments

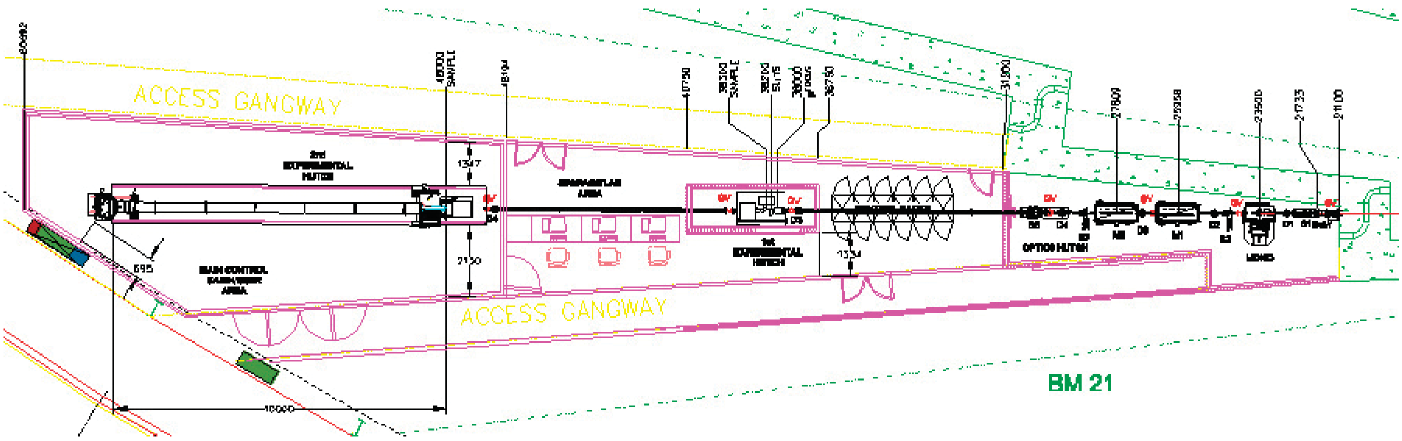

Figure 13. Overall Schematic Layout for I22

Sample environment - The beamline will be equipped with a range of integrated sample environments for simultaneous experiments including DSC, other furnaces/humidity chambers, stop flow cells, etc.

A purpose built platform will be provided for the manipulation of samples for microfocus work. Consideration will also be given to the demands of high throughput and facilities for automatic sample changing will be provided. An offline preparation facility will complete the beamline facilities.

Other Facilities - There are plans for a research complex at RAL to support Diamond; this is seen as an important opportunity to create a world-leading facility in the UK. It will be a research centre and not just a facility provider. Examples of the types of science that could take place in such a facility are in biology work on membrane proteins and large complexes. Examples in physical science could include be research on extreme conditions, environmental research and dynamic structural chemistry.

Timescale - It is hoped that the technical design for the beamline will be completed by the summer of 2004 with installation due to commence towards the end of 2005. Commissioning should commence during the autumn of 2006 for operation later in 2007.

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[i]Tim Wess, Michael Drakopolous, ID18, 2003, Collagen sample collected with CRL system.

[ii]Experiment SC889, Michael Ferenczi[*], Sergey Bershitsky[#], Andrey Tsaturyan[@] and Verl Siththanandan*, ID02 at the ESRF in Grenoble from 9th to 12th April 2003. The work benefited from the help of Daresbury Staff who moved, installed, tested and maintained the RAPID detector (William I. Helsby, Anthony Gleeson, Mark Hillon and Nick Clague) and ESRF Staff who modified ID02 to accommodate RAPID and aligned and tested the station (Theyencheri Narayanan, Pierre Panine, Manfred Roessle, Jacques Gorini).

[*]Biomedical Sciences Division, Imperial College, Sir Alexander Fleming Building, South Kensington, London SW7 2AZ; [#]Institute of Immunology and Physiology, Ural Branch, Russian Academy of Sciences, 91 Pervomajskaya, Yekaterinburg 620219 Russia; [@]Institute of Mechanics, Moscow University, 1 Mitchurinsky prosp., Moscow 119992 Russia.