Conceptual Design Report of the Swedish Materials Science Beamline at PETRA III

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1 Opportunities enabled by High-Energy x-rays

The use of high-energy x-rays (HEX) for materials science applications at 3rd generation high-energy synchrotron facilities has significantly increased during recent years. At present, high-energy beamlines are severely oversubscribed, restricting access for novel users. Most HEX experiments exploit the unique properties of high penetration power (and contrary to neutron diffraction high spatial and temporal resolution) and/or compression of reciprocal space onto area detectors due to the small scattering angles. Also, optimized instrumentation has overcome many limitations that arise when instruments designed for the conventional energy range are used by HEX.

Due to instrumental advances such as broad band monochromators, efficient focusing devices and area detectors, and sample positioning with high angular precision, a wide variety of techniques are now available covering atomistic to macroscopic length scales: wide angle diffraction (WAXS), pair distribution function (PDF) measurements, small angle diffraction (SAXS), and imaging. In particular, novel opportunities arise for *in-situ* experiments during sample processing since bulk samples representative of actual devices can be used, requirements on sample environments are relaxed, and fast data acquisition is enabled by the large reciprocal space coverage. Many modern materials have a complex and hierarchical structure suggesting the simultaneous combination of complementary techniques. HEX not only provide penetration power but also full three-dimensional mappings of heterogeneous samples, either by computerized tomography methods (imaging, or diffraction signals integrated along pencil beams (XRD-CT)), multi-grain diffraction (3DXRD), and/or depth selective apertures. WAXS will be the primary technique and combined with SAXS and imaging as required. The latter also provides efficient mappings to identify volumes of interest for further “zoom in” with a focused beam.

2 Beamline design objectives

The primary mission of the Swedish Materials Science Beamline (P21 in PETRA III nomenclature) is to provide a world class high-energy diffraction and imaging facility to the Swedish materials research community, complementary to the scope of MAX IV. The core energy range will be around 50-150 keV which cannot be reached by undulators at MAX IV (a possible extension of the energy range down to about 30 keV is proposed based on the particular undulator spectrum but performance in the core range should not be sacrificed). To increase the portfolio of techniques available at PETRA, some capabilities that were provided at the DORIS facility should be accommodated, including wide beam tomography, pair-distribution function measurement, and a heavy load diffractometer. Since the user agreement with the Swedish community will include access to other instruments at PETRA, existing capabilities should not be duplicated except required by user demand. Two existing beamlines at PETRA III operate within the targeted energy range. Beamline P02 is dedicated to wide angle diffraction under extreme conditions. This is seen as complementary to the sample environments of technological importance targeted at P21. P02 also has a side station operated at 60 keV and dedicated to high resolution powder diffraction and PDF. The high resolution powder diffractometer will feature multiple crystal analyzers and is clearly complementary to instrumentation proposed at P21. Likewise, the proposed PDF instrument at P21 will operate mainly with lower q-resolution but improved temporal resolution. The energy range of the P07 beamline is essentially identical with the range proposed for P21. However, the layout of P07 with four experimental in-line stations each housing a dedicated instrument is promoting peak performance of individual techniques while the P21 layout should facilitate the combination of techniques and exploitation of advanced detectors and focusing optics.

The beamline design should exploit unique features of the PETRA storage ring (small emittance) and the large distance between the source point and the monochromator and sample locations. The combination of these properties is particular beneficial for focusing and high resolution optics. However, development beyond off-the-shelf optics will be required. The common interest of user groups in *in-situ* experiments would benefit from fast data acquisition and simultaneous access to complementary techniques. Novel opportunities have developed recently by the tremendous progress of area detectors. Further development is anticipated and efficient exploitation of state of the art area detectors is a high priority. Beyond instrumentation, significant development of data acquisition software for beam position feedback, fast synchronization of detectors, and online data visualization is required. For efficient implementation a large experimental station is proposed enabling the use of all optics and detectors with a set of complementary sample positioning units. This approach is also rather complementary to other high-energy instruments at PETRA. On the other hand, the efficiency of the proposed approach requires pre-aligned components and would not be amenable to accommodating all of the proposed techniques for the beamline. Therefore, two other stations are proposed. An upstream inline station should accommodate large equipment including a setup for wide beam tomography. The hutch design should enable “roll-in” of large scale sample environments but the choice of optics and detectors would be reduced. Longer turn over periods should enable efficient operation. Second, a side station is proposed that should be served by a canted undulator and therefore be operated independently. This station would be well suited for a PDF instrument and a heavy load diffractometer.

3 Beamline layout

For reference by further discussion, a tentative floor plan of the P21 beamline is shown in Fig. 1 together with distances from the source points.

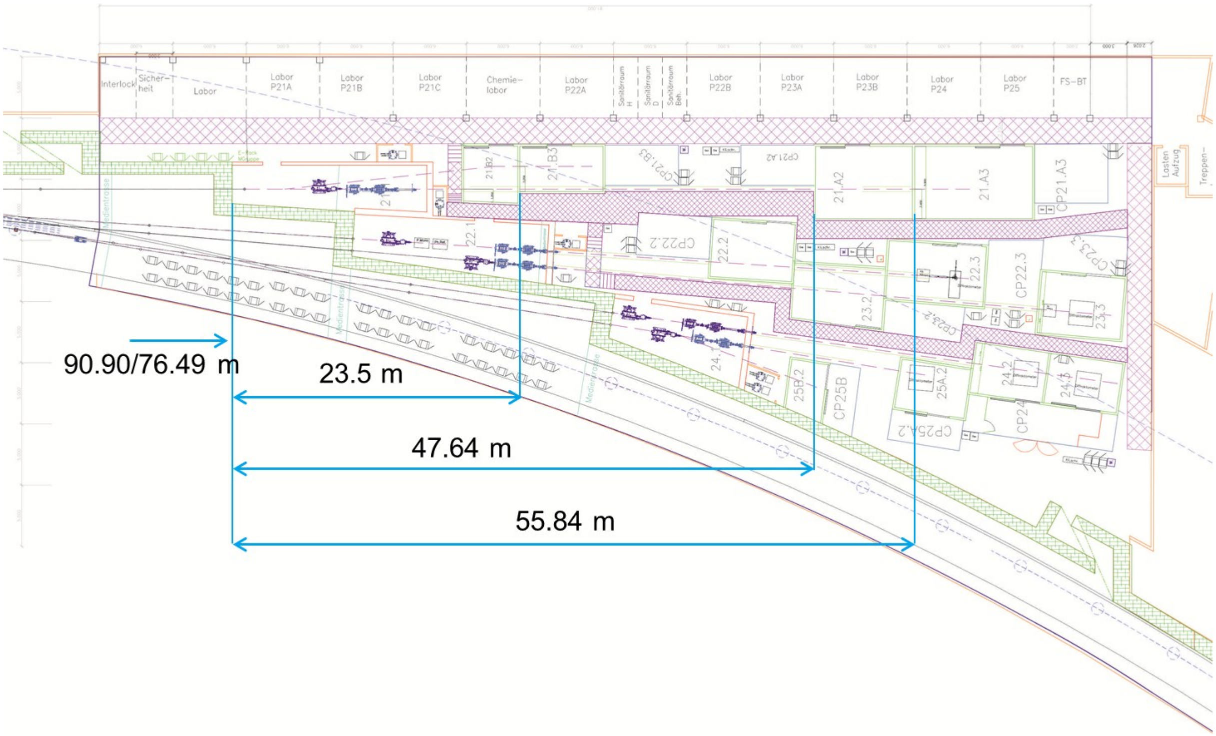

Fig. 1: Tentative floor plan. The P21 hutches are: 21.1-white beam optics; 21.B2-monochromatic beam optics; 21.B3-side station; 21.A2-tomography and “roll-in” station; 21.A3-diffraction station; CP-control hutches.

3.1 Insertion devices

A 4 m short-period in-vacuum undulator is proposed for the upstream straight section and would serve the inline stations. It is proposed to split the downstream 5 m straight section into two 2 m devices. Upstream, a canted undulator would serve the side station and an inline wiggler would provide wide beams to the inline stations, alternatively to the in-vacuum undulator.

An in-vacuum undulator with 19 mm period is under procurement for the P07 beamline. The brilliance is shown in Fig. 2. Parameters may be fine-tuned but the predicted performance matches well to the scope of the P21 beamline.

As a starting point, a wiggler with a Kmax of 5.6 and critical energy of 32 keV is considered to serve the wide beam tomography experiment. The horizontal intensity distribution is reasonably flat over 0.5 mrad which would expand to about 60 mm at the sample position. The total power is calculated as 8.2 kW which is significant.

A suitable 2 m short period undulator (not in-vacuum) would serve the side station.

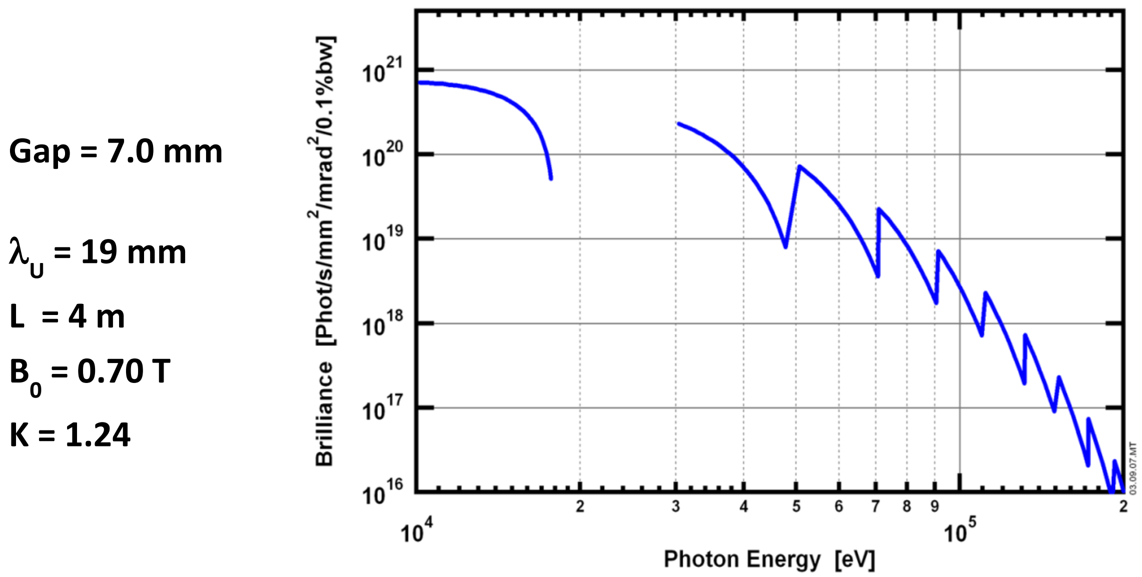

Fig. 2: Brilliance of the 4 m in-vacuum undulator proposed for P07.

Fig. 3 shows the proposed arrangement of the insertion devices. Feasibility of the canting is currently investigated by the machine group, a conclusion is expected by end of July. Also, no frontend design exists for the canted undulator and wiggler beams. Initially, a standard frontend will be used providing an undulator beam from the in-vacuum device for the inline stations.

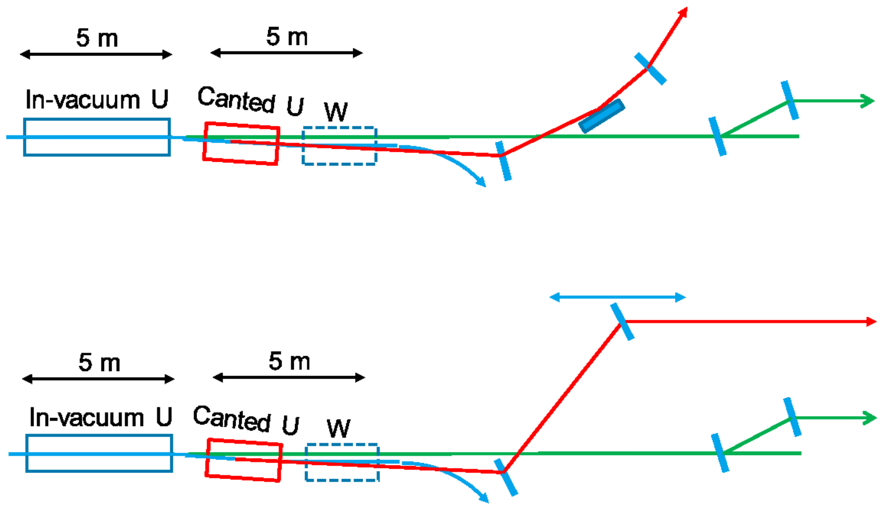

Fig. 3: Proposed arrangement of P21 insertion devices. Two different monochromator schemes for the side station are shown.

3.2 Optics

3.2.1 In-line Laue-Laue monochromator

Due to the broadened bandwidth and several technical advantages, Laue-Laue mononchromators are the default choice at high-energy beamlines on 3rd generation synchrotron sources. For P21 a horizontally diffracting Laue-Laue monochromator is proposed. Due to the large source-to-monochromator distance, the geometric bandwidth due to the finite horizontal source size is small and the vertical source size is preserved for possible sub-micron focusing. On the other hand, the horizontal beam divergence would cause a significant geometrical bandwidth on a flat crystal and bending to Rowland circle geometry is therefore suggested.

Important parameters of the electron beam are compiled in Tab. 1.

FWHM	σ_X [μm]	σ_Y [μm]	$\sigma_{X'}$ [μrad]	$\sigma_{Y'}$ [μrad]	S_X (100m) [mm]	ΔE/E σ_X 100 keV R=100m	ΔE/E $\sigma_{X'}$ 100 keV
high-β	333	13.9	16.7	3.98	1.70	1.67e-4	8.4e-4

Tab. 1: Electron beam parameters (note FWHM is given, not RMS values). Sx is the horizontal size of an undulator beam at the monochromator location. ΔE/E σ_X is the geometric bandwidth due to the finite source size on a Si(111) crystal bent on Rowland geometry, ΔE/E $\sigma_{X'}$ is the geometric bandwidth due to the source divergence on a flat crystal.

For many applications the bandwidth can be broadened to match the resolution of typical area detectors of about 10^{-3}. However, the large source-to-monochromator distance entails a large bending radius and therefore a 10^{-3} bandwidth cannot be reached. It is proposed to also bend the monochromator crystal in the vertical plane by which the desired bandwidth could be achieved due to the elastic anisotropy of Silicon. A proposal will be submitted to test a prototype before the PETRA shutdown in 2013. Fall back solutions are temperature gradient or cylindrically bent crystals. The choice of water or cryogenic cooling will be made based on detailed heat load calculations.

Motivated by the strong 3^{rd} harmonic of the in-vacuum undulator it is suggested to investigate extension of the energy range down to 30 keV. A lower β mode would be desirable to reduce the source size asymmetry for point focusing and increase the beam width for larger samples up to a diameter of a few millimeters.

3.2.2 Tomography monochromator

Because of the wide horizontal beam of the wiggler at the monochtromator position (about 50 mm), diffraction in the horizontal plane would lead to excessively long footprints on the crystals. A bent Laue-Laue Si(111) monochromator with vertical diffraction plane will therefore be chosen. Suitability of a monochromator presently used at DORIS needs to be investigated. Maintaining the narrow vertical source size is not critical for tomography.

3.2.3 Site station monochromator

Two options are considered (see Fig. 3). The first consists of two Si (111) Laue crystals in dispersive +/+ arrangement and a multilayer mirror in between. The multilayer reflection creates a virtual non-dispersive setting such that a large bandwidth (up to about 1%) could be delivered to the side station without significant source broadening by the polychromatic divergence. The energy would be fixed to about 100 keV.

The second option consists of two widely spaced Laue crystals in non-dispersive setting. Increased bandwidth leads to polychromatic broadening. However, a few different energies could be selected by different reflections, and by suitable translation of the crystals along the beam a fixed exit arrangement can be realized with some energy tunability.

3.2.4 Inline multilayer monochromator

An inline double bounce multilayer monochromator has been considered to provide (i) an about 1% bandwidth at the lower end of the proposed energy range, and (ii) a wide bandwidth of about 20-30% for single grain Laue type diffraction. The latter could enable very high temporal resolution if no sample rotation is required. However, broadening of the harmonics of the in-vacuum undulator by tapering is challenging. Also, the intrinsic width of the harmonics is likely below 1%. Quantitative simulations should be made. Given the already complex beamline optics, exceptionally performance would be required to pursue this option. In particular, comparison to the damping wiggler should be made for the wide bandwidth Laue diffraction option.

3.2.5 High-resolution monochromator

A high-resolution monochromator is proposed for the inline stations. It would be accommodated in the monochromatic optics hutch and consist of collimating refractive lenses, a four bounce flat crystal monochromator and focusing refractive lenses. Efficient monochromatization is enabled by the small vertical source size. Feasibility of such a setup has been demonstrated at the APS 1-ID beamline, the setup is sketched in Fig. 4. The high-resolution monochromator would also enable feasibility studies of RIXS experiments.

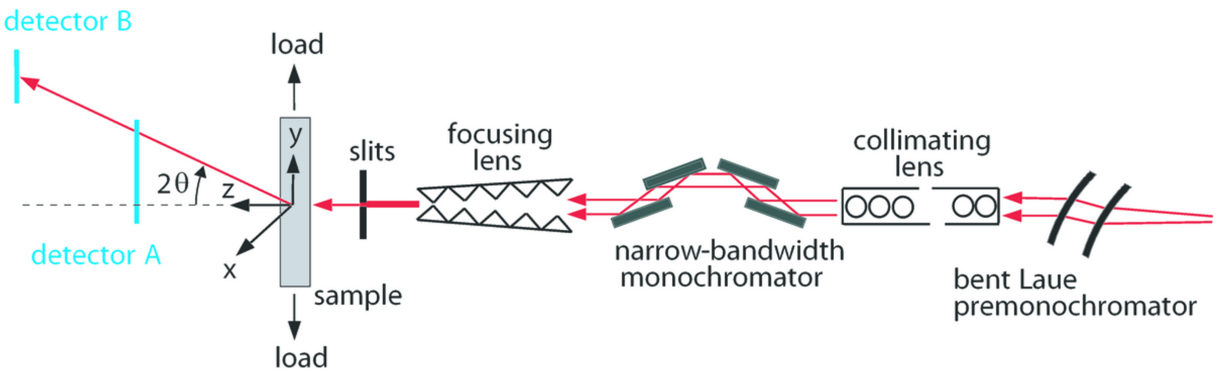

Fig. 4: High-resolution monochromator as implemented at the APS 1-ID beamline (Shastri, JSR 2004).

3.2.6 Refractive lenses

Refractive lenses will play an important role for long and short focal length focusing. They will be installed in the monochromatic optics hutch, on a removable mount in the tomography station, and on the optics table in the diffraction hutch. Also, a small enclosure between the control hutches of the side and tomography stations will be considered. The development of refractive lenses by lithographic procedures is still ongoing and promising due to the favorable refractive properties of Si at high energies.

3.3 Instrumentation

3.3.1 Side station (21.B3)

The width of the side station is limited to about 3 m by the in-line beam and freeway and the proposed length is about 6 m. The length could be somewhat increased if required by specific instrumental requirements. The side station could accommodate one or two instruments. Focusing optics can be placed in the monochromatic optics hutch (possibly even in the white beam optics hutch).

At present, a PDF instrument and a heavy load diffractometer are considered. Main components are a multi-purpose, heavy load goniometer, a large area detector(s) and a crystal analyzer. The sample-to-detector distance should be adjustable to adjust reciprocal space coverage but will be below 1 m. Rotation of the detector around the sample should be possible to increase reciprocal space coverage. The desired beam size is about 0.05 to 1.5 mm. Depending on the sample, a bandwidth of up to a few times 10^{-3} could be tolerated and time resolution down to milliseconds is desirable. The broader bandwidth and hence improved temporal resolution would make the instrument complementary to P02.1.

3.3.2 Tomography and “roll-in” station (21.A2)

The station can be served by either the in-vacuum undulator or by the wiggler. It is proposed that the hutch accommodates large scale user equipment that is not compatible with the sample stages proposed for the diffraction hutch, one of the instruments being a wide beam, high-energy tomography setup. Reproducible repositioning should be provided such as by rails. Refractive lenses in the monochromatic optics hutch and possibly in an enclosure about 10 m upstream are available for focusing. General purpose diffraction ancillaries, heavy load positioning, and an area detector will be provided. The hutch will be operated in slow turn over mode such that several experiments can be performed before instruments are changed. It should be noted, that the hutch is not covered by the hall crane and equipment needs to enter through the door and hallway.

The station could also accommodate a RIXS spectrometer. However, a detailed design would need to be developed and checked for feasibility. Some manpower would need to be provided by the community.

3.3.3 Diffraction station (21.A3)

The diffraction station will be served by a 4 m in-vacuum undulator (and could also take the wiggler beam). An outline of the instrumentation is sketched in Fig. 5. First, there is a beam conditioning unit that will be equipped with refractive lenses and possibly bent multilayers as focusing. Furthermore it will hold slits, beam monitors, a fast shutter and an optional larger scatter shield. Possibilities will be investigated to evacuate most of the flight path. A monochromator for scattering on liquid interfaces is not considered initially.

Next are three sample stages mounted on horizontal rails transverse to the beam. Proposed are (i) an air-bearing rotation stage with vertical rotation axis and about 100 Kg on axis load capacity. Electrical, fluid, and gas rotational unions are considered that could enable continuous rotation of sample environments.

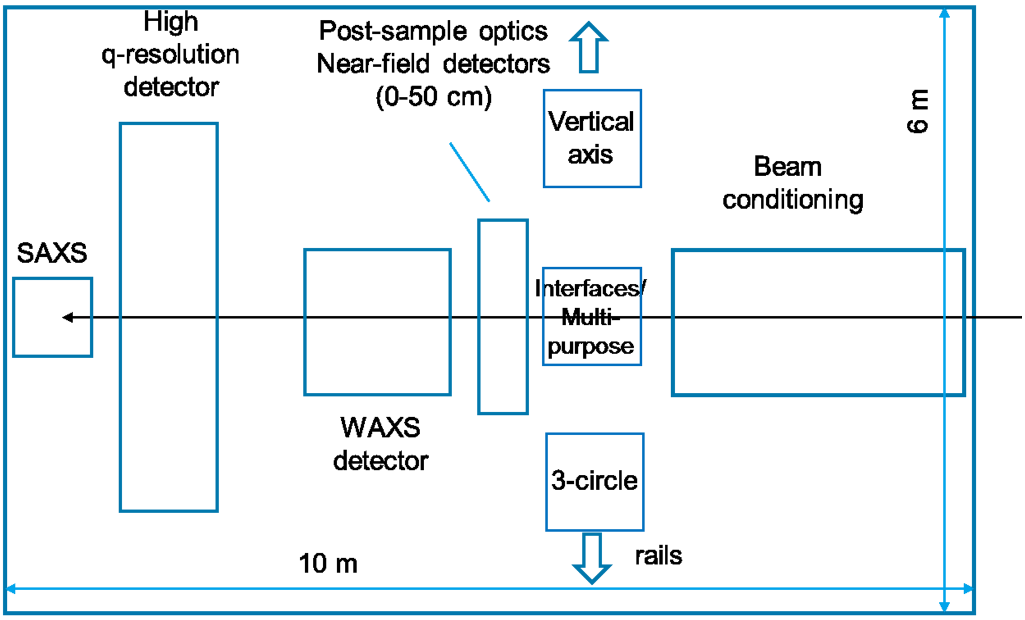

Fig. 5: Sketch of principle components of the diffraction hutch instrumentation.

(ii) A stage optimized for interface diffraction. The main feature is a highly precise horizontal rotation axis transverse to the beam. The design will follow the principle of the interface diffractometer in the EH2 hutch at P07. As in EH2/P07 the diffractometer could also serve as multi-purpose mount. The central mounting position would provide ample space laterally for sample environments.

(iii) A three circle goniometer preferably with air-bearing phi-circle.

Next downstream will be a mounting frame for detectors and apertures that need to be positioned closely behind the sample. The frame itself should not interfere with sample environments. Foreseen are a semi-transparent imaging detector and holder for depth resolving apertures. The imaging detector would enable tomographic imaging of samples of a diameter up to few millimeters. However, imaging per se is not inteded but rather as secondary technique to facilitate the identification of areas of interest for diffraction measurements.

Next will be an xyz stage for the positionig of large area detectors, followed by an xy frame to mount an area detector that provides high reciprocal space resolution. Finally, a SAXS detector is proposed that could be operated simultaneously with WAXS detectors.

3.3.4 Sample environments and supplementary characterization techniques

The capability of performing in-situ experiments is crucial to both the interfaces and bulk materials diffraction communities. Of particular interest are elevated temperatures (and to lesser extend low temperatures), chemical environments (gases), and compact devices for mechanical loading. Beamline P07 should be considered for large engineering equipment. The supply and exhaust of gases, electric power, and cooling water must be supported by the infrastructure. Suitability and availability of sample environment from BW5 and P02/EH2 should be investigated. However, user groups will also custom design and/or modify commercial sample environments. The user driven development of sample environments will be supported, in particular if the devices can be made available for other users.

It is also expected that the availability of supplementary sample characterization techniques such as optical methods will be important. The successful implementation of supplementary sample characterization techniques will require collaboration between user groups who have the technical expertize of the method and beamline staff to accommodate constrains by the diffraction techniques. Again, projects that aim at providing a capability for general users are particularly encouraged.

3.4 Data acquisition, reduction, and visualization software

At present, limitations with the data acquisition, reduction, and visualization software are often limiting factors during in situ experiments. The situation is expected to aggravate due to the increasing data acquisition rates and volumes and the variety of sample environments and control parameters. General approaches need to be developed by PETRA but the implementation at the beamline and interfacing with user equipment will require the support by a software engineer.