

High pressure, large volume press, and white beam	
Current designated sector:	Facility goes to:
ID09A, ID06LVP, ID15A	ID15

1.1 ID CARD

ID15 will become one of the most multifunctional beamlines at the ESRF, making full use of its high energy, white beam and focal qualities to suit investigations into materials science and extreme conditions research. The augmented high pressure diffraction beamline, previously at ID09A, will be located here, using the current ID15B branch of the specially revised canted straight-section. The ID15A branch will be used, independently of B-branch operation, and house two stations; one for the large-volume press and the other for materials science applications. Each of these will be open to users for use with high energy monochromatic and white beam data collections. Natural applications include mineral physics, geology, materials science, solid state chemistry and physics.

1.2 SCIENTIFIC CASE

High pressure (HP) research will become a major activity of the future ID15 beamline, where it will share facilities with the high energy (HE) programme of the engineering, materials science and inelastic scattering communities. The new HP DAC beamline, ID15B, will operate independently with its own insertion device and the LVP (~80 %) and HE programme (~20 %) will share the beamtime on ID15A (see Figure 1 for the overall layout of the beamline).

The proposed project will improve the ESRF infrastructure by providing, together with ID27, three independent beamlines for HP experiments. ID09A and ID27 are world-leading beamlines designed to determine structural properties of matter at extreme conditions with the greatest accuracy possible, using monochromatic diffraction and large area imaging detectors. The complementary specialisations of ID09A and ID27 make the ESRF the primary source for HP expertise and experimentation. The beamlines are highly productive and are competitive with the foremost equivalents in Japan (SPring-8) and the US (HPCAT and GSECars at APS). The research produced is of highest ranking, as judged by the ratio of publications to experiments and the number of papers appearing in top-ranked journals. All implicated beamlines are major resources for the European physics, structural chemistry, structural biology, materials science and earth science

communities. The installation of a LVP has increased the ESRF's repertoire in an area where it has not been competitive.

The HP-DAC beamline at ID15B will continue to use monochromatic diffraction with large area detectors on single crystals and powdered samples at high pressures in diamond anvil cells. It will provide variable beam sizes down to $<10 \times 10 \mu\text{m}$, to study samples from a few GPa to approximately 200 GPa, at fixed energies of 30 and 58 keV and very high photon fluxes of $\sim 10^{13}/\text{sec}$ with a dE/E resolution of $\sim 2 \times 10^{-3}$. The upgraded beamline will continue to offer state of the art optical systems for additional *in-situ* characterisation of the samples at high pressure (Raman, etc.) and, in addition, it will offer improved sample environment and both a better temporal and spatial resolution through the use of new and improved area detectors.

New detectors with faster readout coupled with recent developments, such as computer-controlled automated pressure controllers, allow new types of experiments such as real-time data collection while changing pressure and/or temperature. This permits well-controlled *in-situ* monitoring of intermediate states or phases, essential for understanding the formation of new metastable materials that are expected to be of technological interest. Mechanisms of pressure or temperature induced phase changes can be studied in detail. The combination of a higher dynamic range, larger detector surfaces and the increase in real space resolution offered by use of higher energy than presently available allows the total elastic scattering signal from the sample (Bragg reflections and diffuse scattering between the reflections) to be collected. From these, users can derive simultaneously structural and dynamical properties of matter under pressure to give insight into, for example, pressure-induced amorphisation, fast ion conduction, quasi-crystals. Further gains can be made through evolution of the sample environment; presently, a new cryogenic system is under development; including a tailored diamond anvil cell and a gas loading system, for diffraction measurements down to, and below, 4 K.

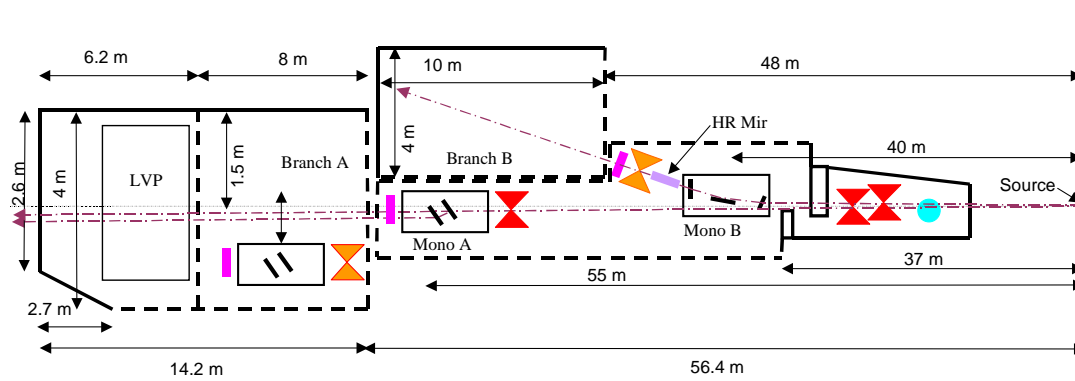


Figure 1. Schematic representation of the beamline optical layout for the future ID15. The beamline is canted; with Branch A, profiting from the use of the AMPW and U22 for high energy and white beam operation and Branch B using a U20 undulator and focusing optics for monochromatic diffraction experiments at extreme conditions.

Most LVP developments worldwide are currently operating at bending magnet sources and some make use of limited angle-dispersive facilities. Extending the opportunities presented with the AMPW/U22 source and white beam hutch at ID15

will provide both EDX and ADX capabilities up to the highest energies. With the effective source upgrade offered by this project, not only will all current experiments be improved from the outset – EDX from a wiggler or ADX from an undulator rather than both from a bending magnet – but this opens up a range of new experiments. Quite simply, the combination of the LVP with the source at ID15A is an extremely attractive proposition that would be in high demand from worldwide users. By design, we would also increase the stress/strain expertise currently operating at ID15A with those users who conduct similar experiments under HP conditions, through large-volume D-DIA, rotation strain or radial uniaxial DAC techniques.

The future HE station on ID15A will focus on engineering materials methods and instrumentation, as well as inelastic scattering. These experimental techniques benefit from the unique properties of the AMPW source that can provide circular polarised photons up to 750 keV. The major user activity will be in modern materials engineering research where non-destructive diffraction techniques to investigate materials properties on multiple length and time scales are state-of-the-art. Today this field has evolved much beyond routine 3D strain mapping and specifically makes use of the advantages evolving from high energy X-rays in the range 100 to 500 keV. These X-rays can even penetrate through 70 mm of steel, allowing ‘real’ components to be investigated. New research topics will be driven by the development of novel 1D and 2D segmented or pixellated detectors, optimised for the high energies that are proposed here. This will push studies of dynamics in thermo-mechanical loading processes to the sub-second time scale and to ‘single-shot’ experiments with higher spatial resolution when combined with special collimating slits, offering new insight into materials processing. Inelastic scattering research will focus on spin-polarised studies at high fields, high pressure and low temperatures to fully exploit the recently commissioned 9 T cryo-magnet.

1.3 PROJECT HISTORY

In line with the recommendations of the last beamline review (May 2006), SAC feedback from the Purple Book round (May 2007) and the Upgrade user meeting (October 2007), ID09A will be extended to a full beamline dedicated to diffraction experiments using diamond anvil cells. A further proposal of the beamline review and an integral part of the Purple Book HIPRE CDR was to install the large-volume press (LVP) on an integrated HP beamline. The second branch at ID15 will therefore become the final location of the LVP and will make use of existing infrastructure to provide a facility for monochromatic and EDX diffraction through use of the combination of facilities offered by the future ID15 source.

The main scientific activities of the HE beamline ID15 are focused on engineering materials research and studies on surfaces and buried interfaces. As a result of this successful research programme and due to insufficient beam time a new HE beamline, UPBL2, has been proposed following the recommendations of the last beamline review panel (May 2007). UPBL2 will host the major part of the present research programme. The research activities engineering materials science and inelastic scattering will continue on the ID15A beamline since they require the uniform spectrum of the high energy AMPW.

For a fuller discussion of other HP and HE programmes, the reader is also referred to the EMS, HIPRE and HIENE CDRs of the Purple Book May 2007 and the current CDRs for UPBL2 and ID27.

1.4 BASIC TECHNICAL CONSIDERATIONS

At present, ID09A shares a straight section and beamtime with ID09B, which is dedicated to ultra fast time-resolved diffraction. The installation on one branch of a canted undulator straight section will double the available beam time. However, two technical developments are crucial for the realisation of the new beamline: location of three IDs on the same 6 m or 7 m straight section and a large bandwidth non-dispersive monochromator (LBNDM).

The U20 in-vacuum undulator from ID09 will be used on one branch of a canted straight section. Following discussion with the Accelerator and Source Division, it has been proposed that the AMPW be rebuilt with approximately half the carriage length and double the field. This has considerable advantages. In addition to fitting the U20 and U22 2 m undulators and the AMPW in a canted (<1.7 mrad and ~2.0 mrad) 6 m section, it will also halve the wiggler divergence – effectively improving the source. Whilst the total power is reduced, this is at no loss to the power density, or flux on sample. This would allow the option of building the beamline on an existing 6 m straight section as either a temporary or final solution according to results of future 7 m testing.

ID09A uses an asymmetrically cut bent Laue monochromator (Schulze et al, 1998). The monochromator combines high flux with a small horizontal focus (<10 μm). Due to the heavy shielding needed for the white high energy beam, the deviation of the beam resulting from the canting (<<5.4 mrad due to the wiggler) will not provide the necessary distance between the experimental setup and the beam path for the combined LVP-HE station. The LBNDM, a combination of two Laue monochromators and a multilayer mirror, designed to preserve the small horizontal focus and high flux, will have to be inserted 5-10 m upstream from the experiment to realise a comfortable working distance. The proposed ID15B hutch (Figure 2) will extend to the current ring wall to allow for the combined 15° mono takeoff. Different energies will be achieved with different sets of crystals (111 for 30 keV, 311 for 57.9 keV). Vertical focusing and harmonic suppression can be achieved with the mirror from ID09 or if the harmonic rejection from the multilayer mirror is sufficient with compound refractive lenses. The experimental setup will be taken from ID09A and modified for the new location.

To fully develop the potential of the beamline, a new detector with the following characteristics is imperative: large area, high resolution (e.g. 50 x 50 cm with 5000 x 5000 pixels), as high dynamic range as possible (>18 bit), fast readout (10 ms), no crosstalk between pixels. Pixel detectors are under development to achieve this kind of performance. To work at high energies (> 30 keV) the detectors need GaAs, or similar, conversion layers.

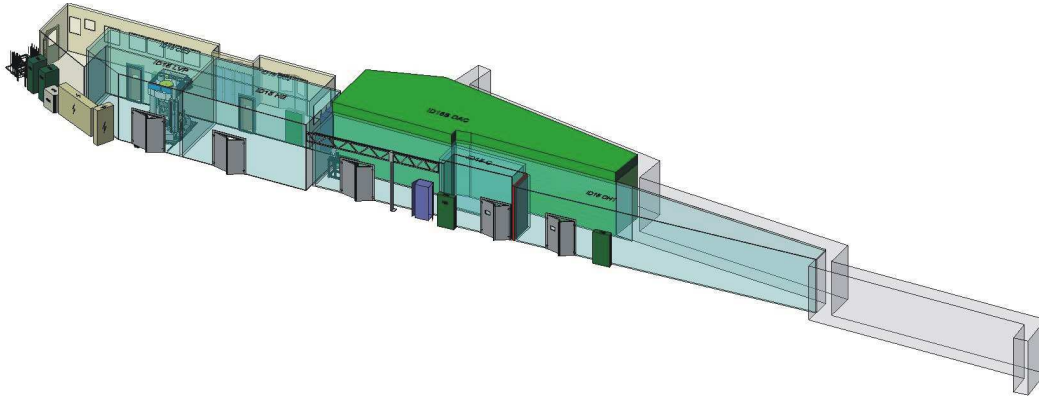


Figure 2. Upgrade of ID15B (green) will involve extension of the current monochromatic hutch to the ring wall.

Concerning the LVP, the use of the current white beam hutch at ID15A necessitates significant reduction of the transverse length of the parts of the press beneath the floor. The pit and supporting floor would be reduced in width (across the beam) by approximately 0.8 m. This involves a similar reduction of the base plate width and the long transverse-acting translation stage. A redesigned supporting floor (with steel framed insert) would have the capacity to bear a 25% increase in floor loading, in addition to any further constraints from seismic qualification and additional prerequisite radiation shielding. The reduction in base plate length would require relocation of motor and signal cabling, which is currently all beneath floor level. Similar treatment of the long axis stage will require replacement or re-engineering of its long-acting spindles, guide-rails and braking systems. It is not expected that any of the higher-acting motions will be affected. These modifications must be made both before relocation of the press and concomitant with reinstallation, which would require a total of four to six weeks. The design and manufacture of supporting floor, with any necessary seismic qualification or geotechnical testing, should be carried out at least three months in advance of any move.

The current LVP setup at ID06 has no optical component specifically assigned to the instrument and a full suite of optical elements will therefore be required. This request will, in the medium term, be limited to necessary primary and secondary slits, shutters, photon absorbers, etc and a liquid-nitrogen-cooled channel-cut Si111 monochromator. LVP-related beamline and motor control systems are up to date with the current ESRF standards, as are all counting chain elements and their control and gating systems. The LVP is currently dependent upon the availability of detectors from the Detector Pool. Once short-term testing of suitable detector components or other optical devices is completed, a separate budget request will be made for the selected items as part of the continual evolution of the instrument.

In order to locate the new ID15HE station and the LVP in ID15A (Figure 3) a shielded dividing wall must be inserted to allow the press to function for offline synthesis during the 20% operating time of the HE branch. Any changes to ID15A or the optic hutch will necessitate the removal of the control cabin facing BM14, for relocation of racks, placement of cryo-equipment and downstream OH access (Figure 4).

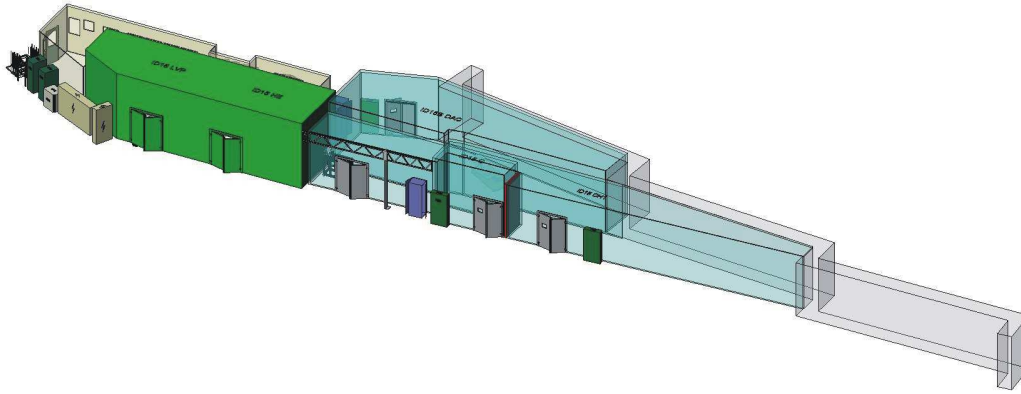


Figure 3. Redesigning the current ID15A white beam hutch (*green*) will involve removal of two existing hutches (at ID15-ILL) and extension of ID15A. Additional doors are required for access from the control cabins and additional roof access is required to make use of the overhead crane in both of the divided portions.

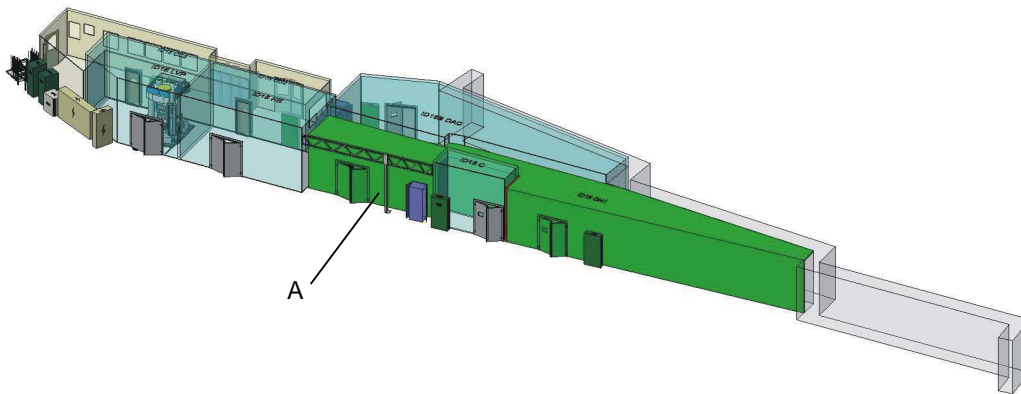


Figure 4. Upgrade of the current OH requires essential (~15 m) movement of the wall indicated (A) – and removal of the control cabin – with the addition of double door for access to the two monos, shutters and downstream photon absorbers. Further optional changes, *recommended by the Safety Group*, involve removal of the local shielding and rebuilding a 50 mm lead hutch (~45 m² extra).

1.5 REFERENCES

Schulze, C., Lienert, U., Hanfland, M., Lorenzen, M. and Zontone, F. (1998) Microfocusing of hard x-rays with cylindrically bent crystal monochromators. *Journal of Synchrotron Radiation* **5**, 77-81.