The high pressure crystallography beamline
ID15B
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Two beamlines on a canted straight section.

ID15A: Materials chemistry/engineering, 20 \( \text{–} 100 \text{(500)} \) keV (M. DiMichiel, T. Buslaps, G. Vaughan)
ID15B: High pressure diffraction, 30 keV (M. Hanfland, G. Garbarino)
Engineers: K. Martel, J. Leonardon, J. Bonnefoy, …
Technician: D. Duran
Replaces ID09A
ID: U20
Energy: 30 keV
Optics: Designed with the machine upgrade in mind
Beamsize: Variable, minimum 7 x 7 μm², with low divergence
Flux: $10^{12}$ ph/sec
Other: Camera for HRXTM on a table at installed the end of the experimental hutch
Beam through 30 µm pinhole

vertical

horizontal
Large opening angle, ±32° or ±38° rotation range,
Pressures (with He): 30 GPa with 600 μm, 85 GPa with 300 μm and 150 GPa with 120/350 μm culet (with Re gasket).
High resolution phase contrast imaging

- Localization of new phases within the DAC.
- Creation of precise maps of their position.
- Limited 3D information.

Details see: P1, A. Barannikov et al.
Low (horizontal) emittance machine.

Changes for ID15B:
~ 20 x increase in flux, due to low horizontal divergence.
Smaller (horizontal) beam, due to smaller source size.
Coherence.

Also: New detector, Eiger 2, 9M, CdTe.
He intercalation (As$_4$O$_6$ + 2 He) at 2.5 GPa. H$_2$ intercalation (As$_4$O$_6$ + 2 H$_2$) at 1.8 GPa. No intercalation with Ne. Can be suppressed by rapidly increasing P to > 10 GPa. No evidence of a structural phase transition or amorphisation up to 40 GPa*.

Study related systems looking for comparable phenomena. System selected: Realgar (As$_4$S$_4$).

Method: Single diffraction in a diamond anvil cell with He as quasi hydrostatic pressure transmitting medium. Rotation images, 0.5° image, 1 sec exposure, 156 images. Peak search, orientation matrix determination and integration with CrysAlisPro (Oxford diffraction). Refinement with Jana 2006.

Sulfur, 17.5 GPa
REALGAR ($\text{As}_4\text{S}_4$)

$\text{As}_4\text{S}_4$-molecules, monoclinic, $V_0 = 800 \, \text{Å}^3$, S.G.: $\text{P}2_1/n$ with 4As and 4S in 4e positions. Transforms under light into Pararealgar, high temperature polymorph (C2/c). SC studies to 5 GPa, Powder to 45 GPa*. *C. Hejny et al., Physics and Chemistry of Minerals 39, 399 (2012)

Realgar, $P = 0.5 \, \text{GPa}$, $wR_{\text{all}} = 5.6 \%$

Realgar, $P = 41 \, \text{GPa}$, $wR_{\text{all}} = 3.97 \%$

Stabilization of structure under pressure.
REALGAR \((\text{As}_4\text{S}_4)\)
REALGAR (As$_4$S$_4$)

One As – S distance becomes significantly longer and a second one significantly shorter at high pressure.
Above 42 GPa Realgar becomes amorphous. Similar to a 1\textsuperscript{st} order phase transition. Amorphous phase can be recovered at ambient P. As\textsubscript{4}S\textsubscript{4} molecules remain intact.
ID15B is designed for crystallographic studies at extreme conditions. It is extremely stably and performs better than planned. A substantial increase in flux is expected after the machine upgrade.

Realgar: no intercalation, stable to 41 GPa, amorphisation at 42 GPa.

Arsenolite: no amorphisation, crystal just gets bad (increase in mosaicity) above 15 and 40 GPa with Ne and He as pressure transmitting medium, respectively.

As$_4$O$_6$ single crystals by P. Guńka.