Basics of X-Area for PILATUS

Commercial software to process single-crystal and powder x-ray data

from STOE image plates and PILATUS detectors

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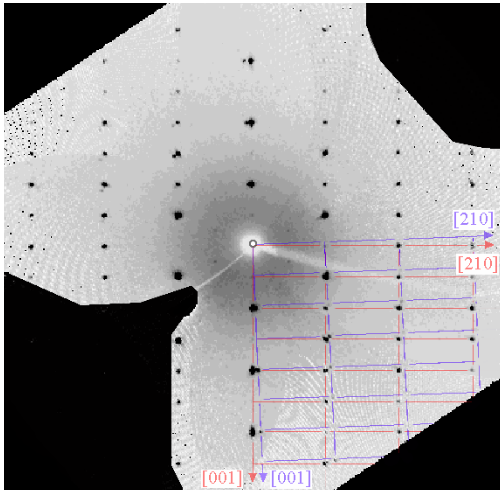
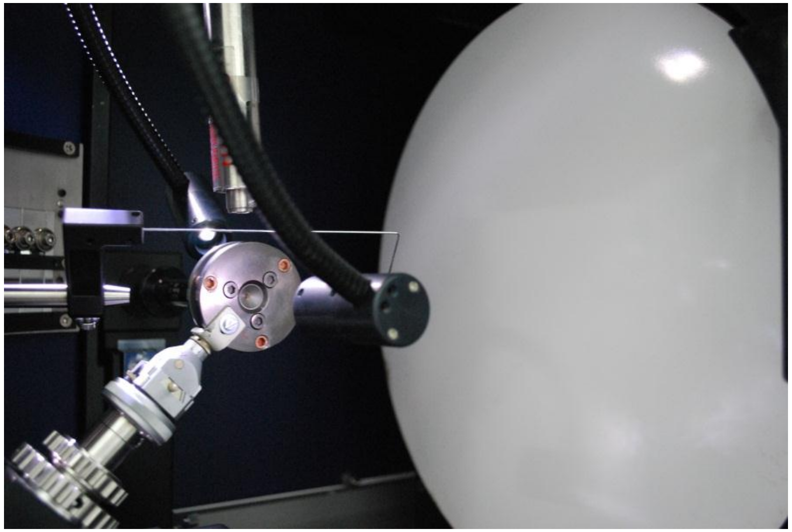
Crystallographic problems that X-Area handles without any problems

- twinning
- modulated structures
- composites
- polytypism
- **high-pressure data**





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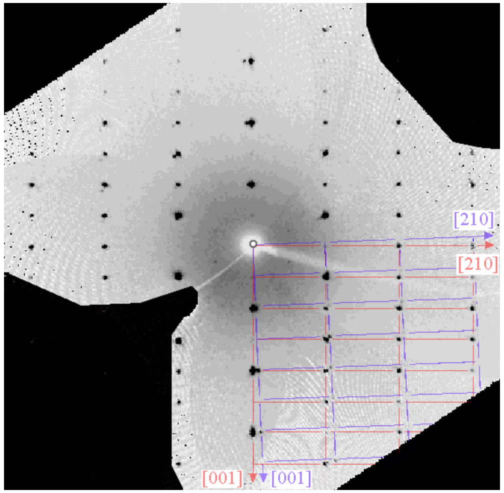
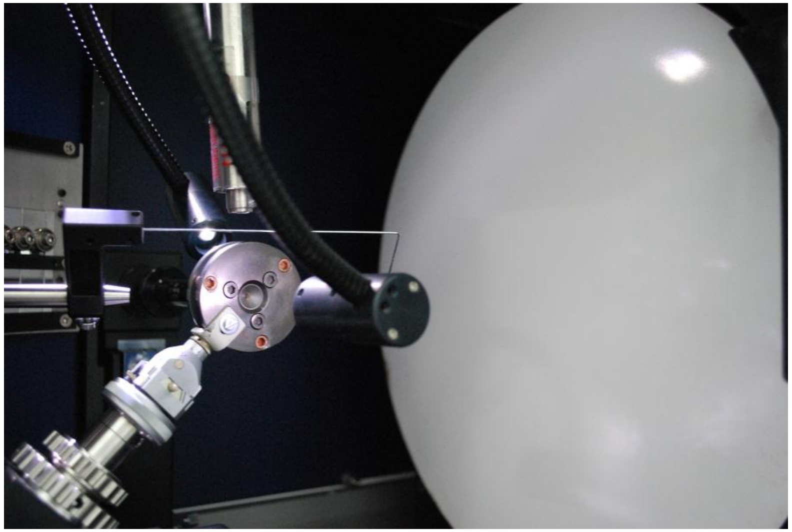
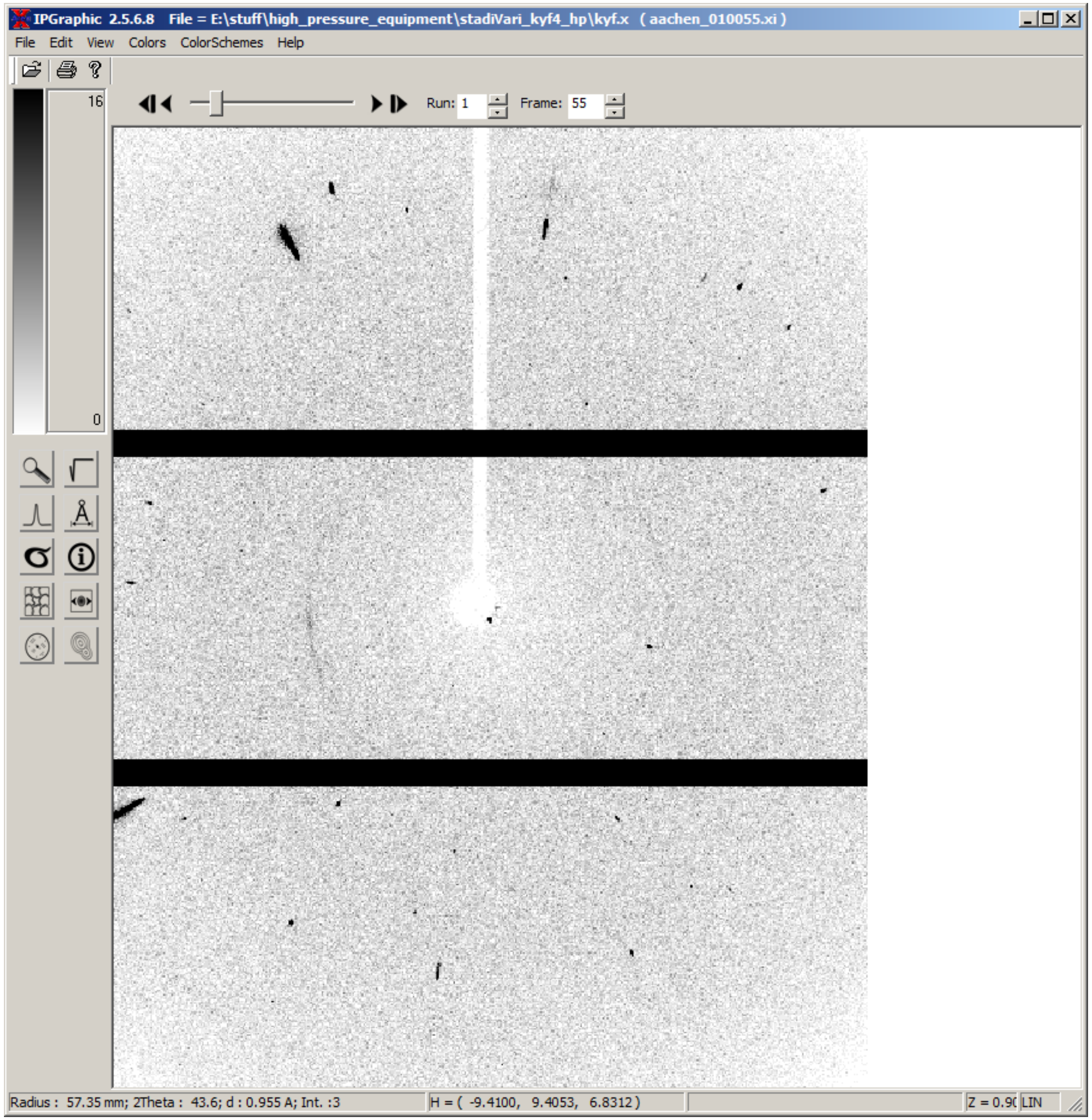




X-Area routines to treat single-crystal data at high pressures from PILATUS



Ahsbahs diamond anvil cell
(opening angle 90°)

PILATUS 300 K

GeniX 3D Mo high-flux delivery system
(beam size at focus: <130 μm)

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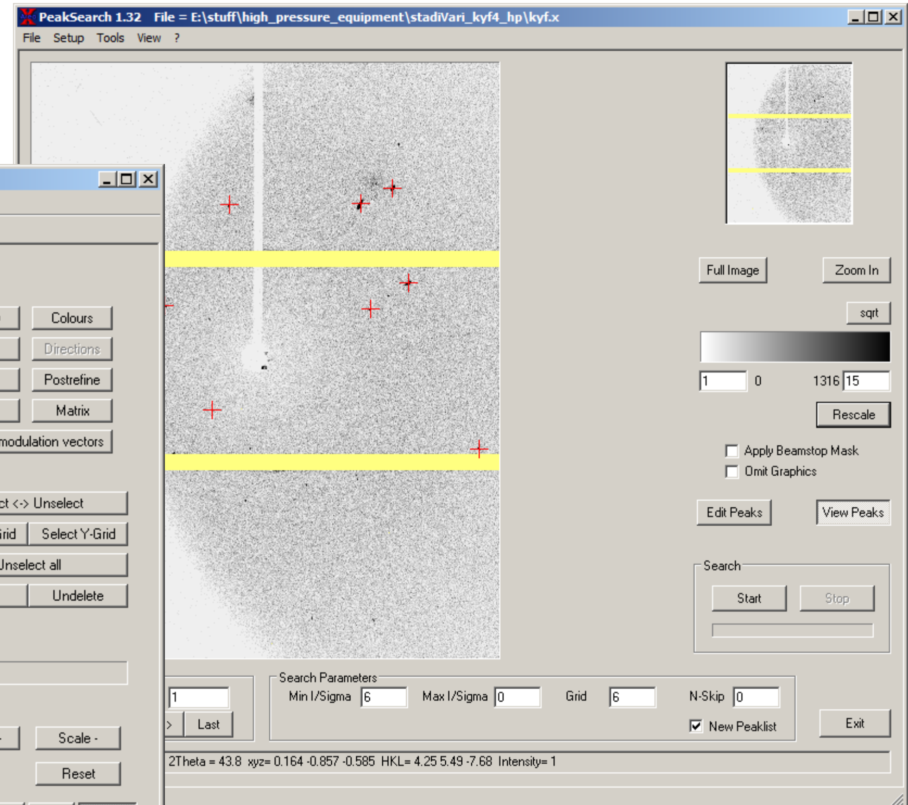
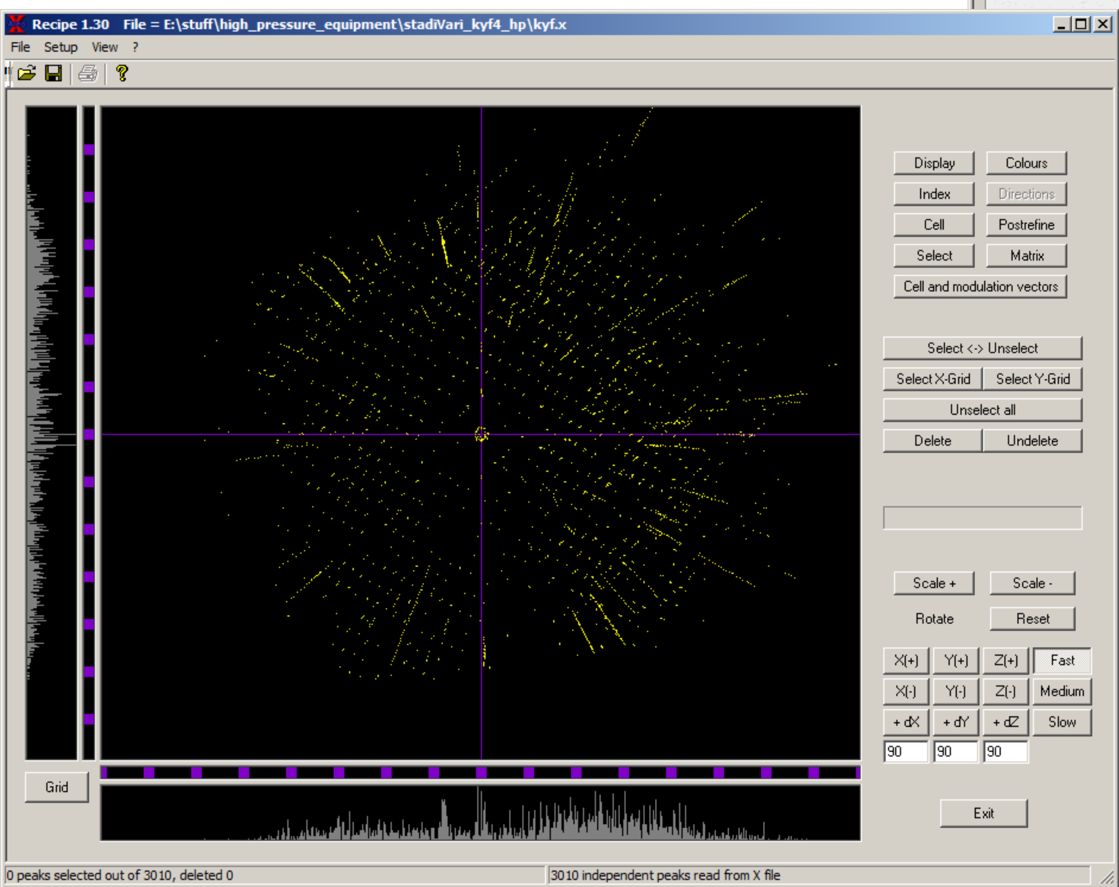
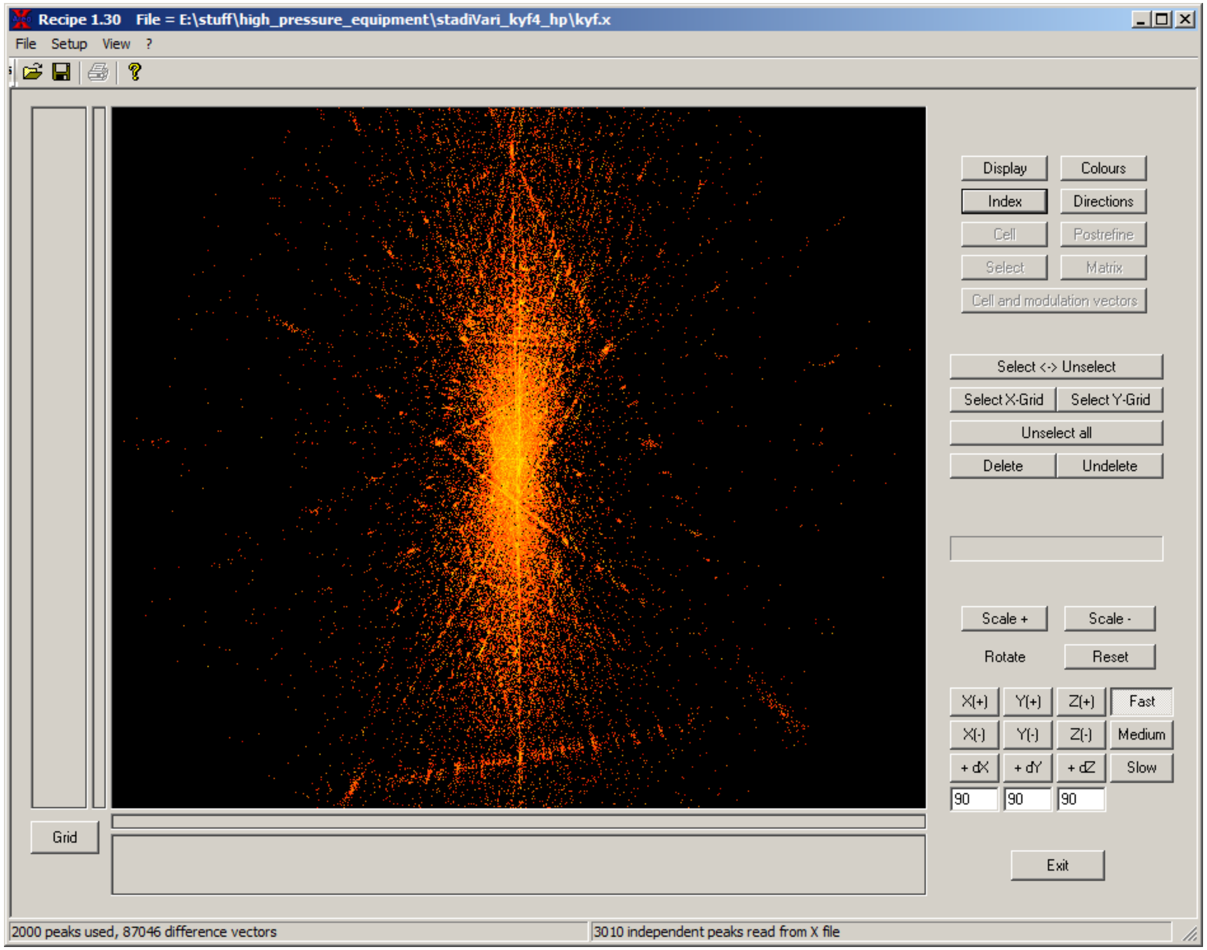
Peak search





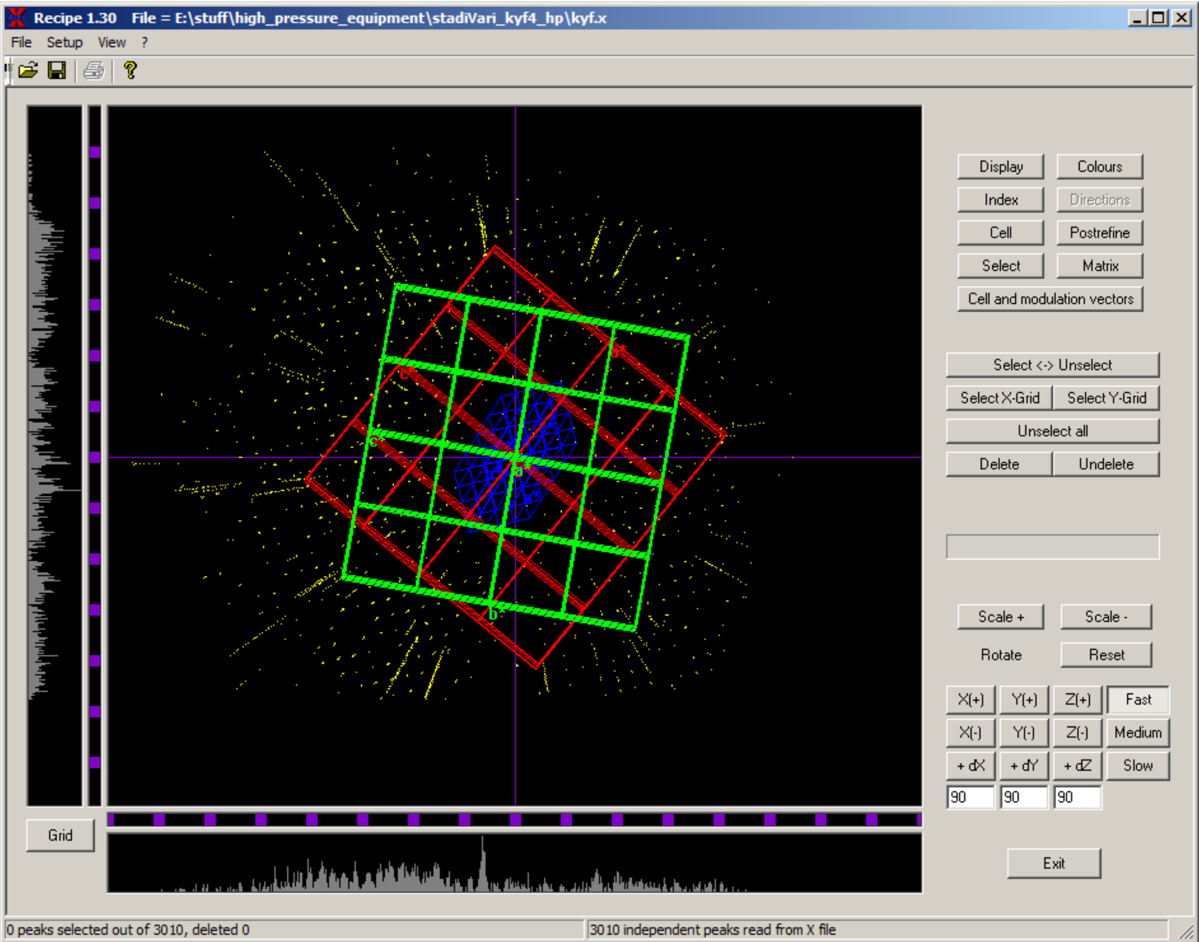
Indexing in reciprocal space

X-Area routines to treat single-crystal data at high pressures from PILATUS



Using the currently selected peaks all difference vectors between all peak positions are calculated, normalised, and projected onto the horizontal plane of the Ewald sphere. When the crystal is a *true* single crystal, a series of sharp "lines" can be seen. Each pixel represents a direction in the reciprocal space. The picture is colour coded, the brighter the pixel the larger the frequency of difference vectors in that direction. Each "line" corresponds to a set of parallel, equally spaced layers in the reciprocal space. **In the case of the high-pressure data, the "lines" from the crystal, two diamonds, and grainy spots of the gasket rings are superimposed.**

X-Area routines to treat single-crystal data at high pressures from PILATUS



The diamond orientation matrices are useful to check the alignment. They could be used for any other measurements in the same DAC. Hence, it is enough to find them once. The easiest way to determine them is to perform a full measurement with a very short exposure time per frame, e.g., 1 second or less.

Two diamond matrices

cF, a ≈ 3.55 Å

λ/2 reflections from the diamonds are not measured on Stadivari

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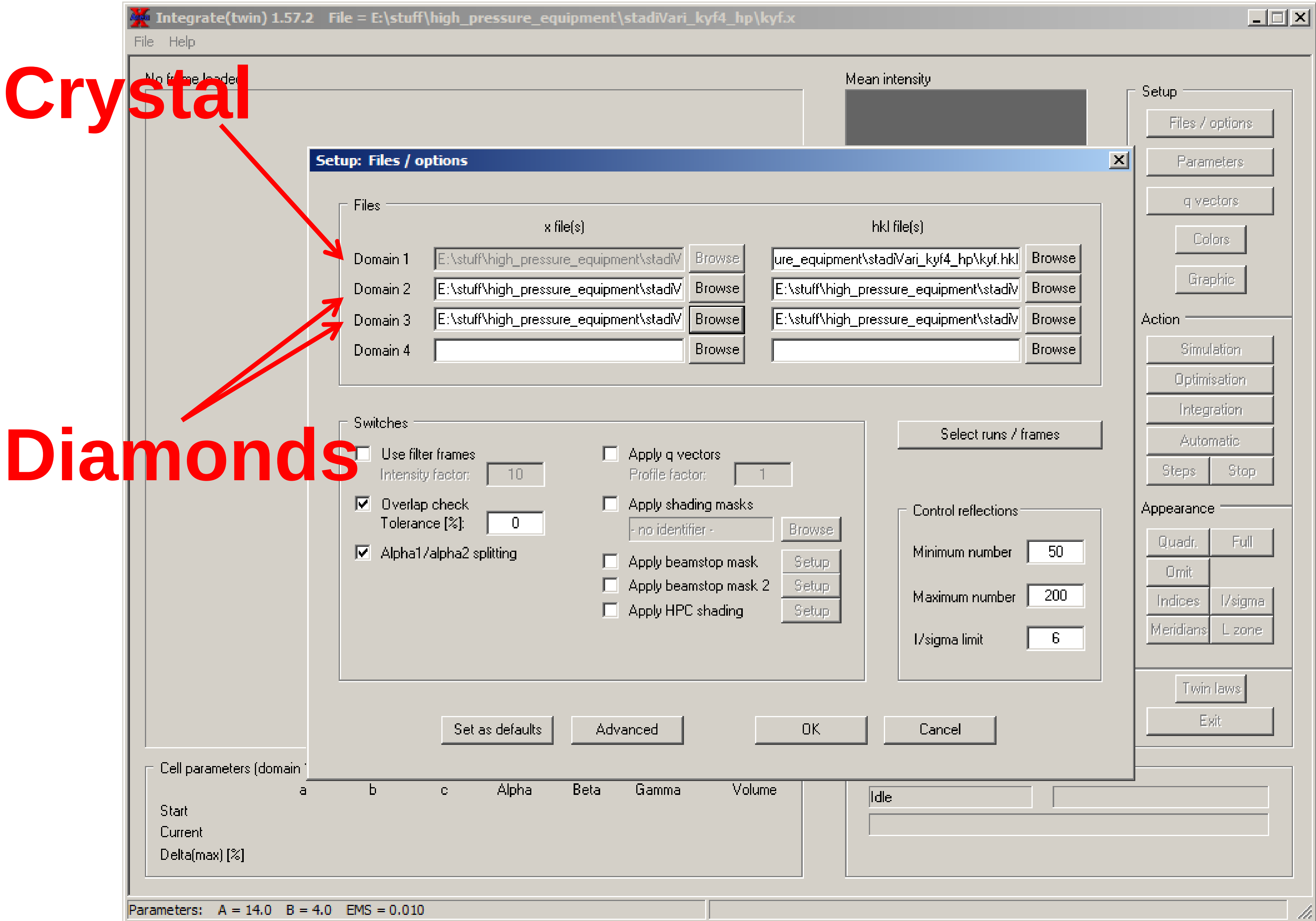

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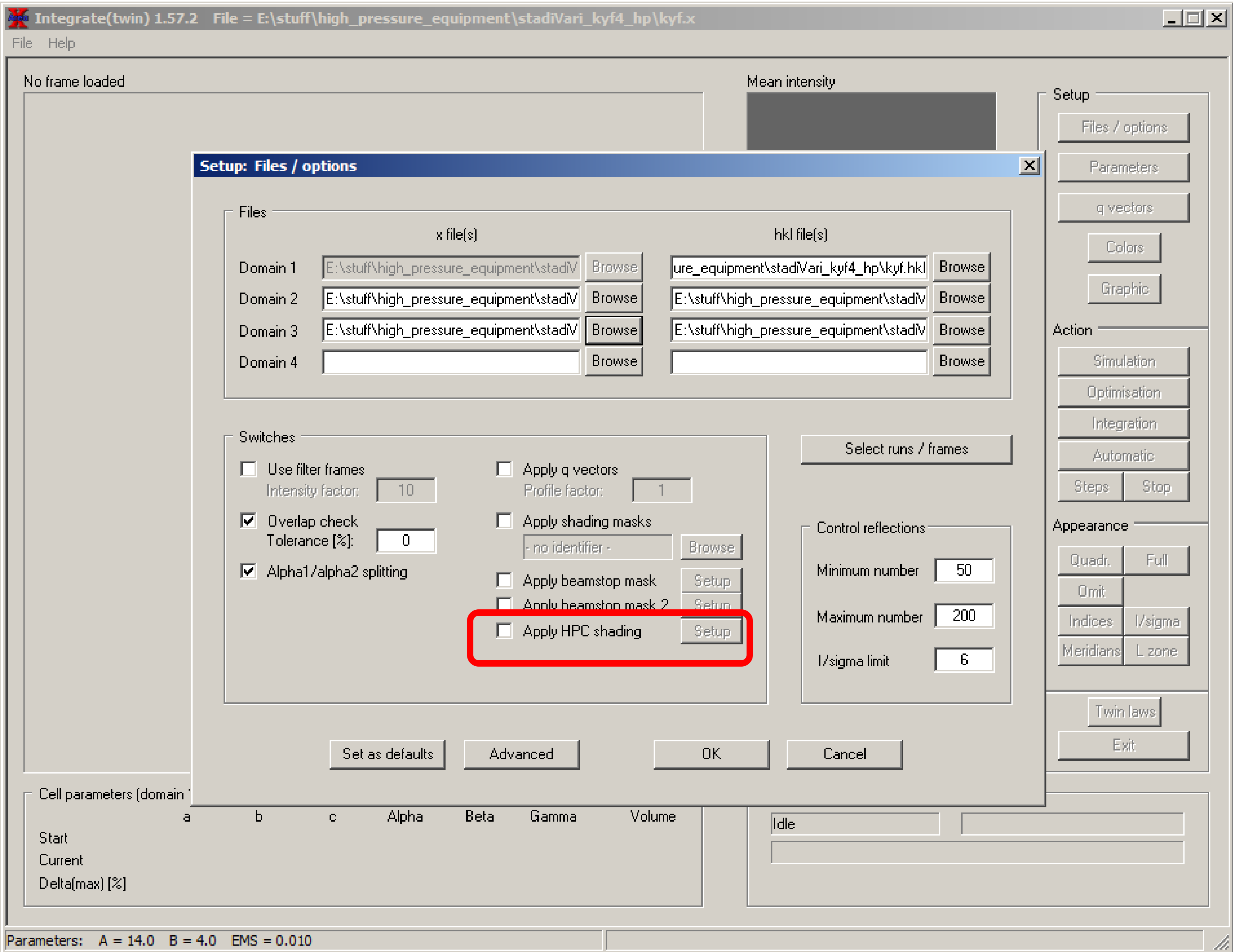

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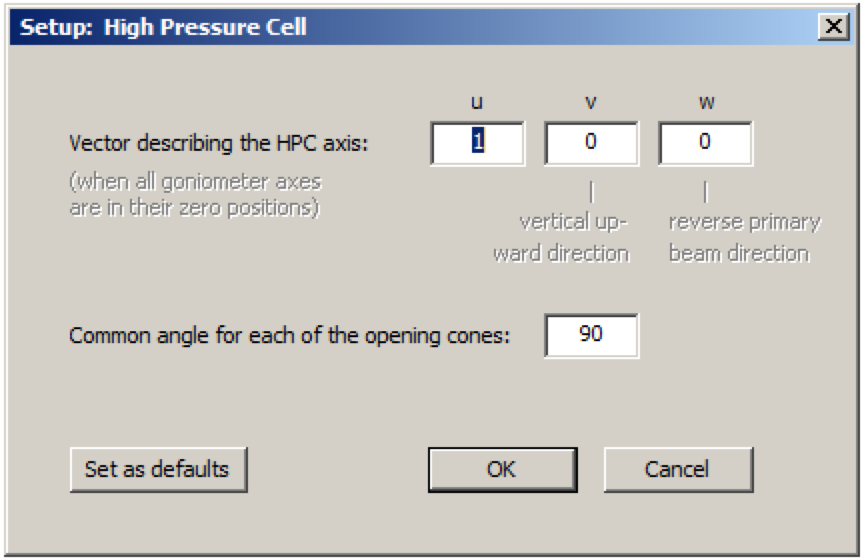


The coordinate system *uvw* is centered on the goniomter:

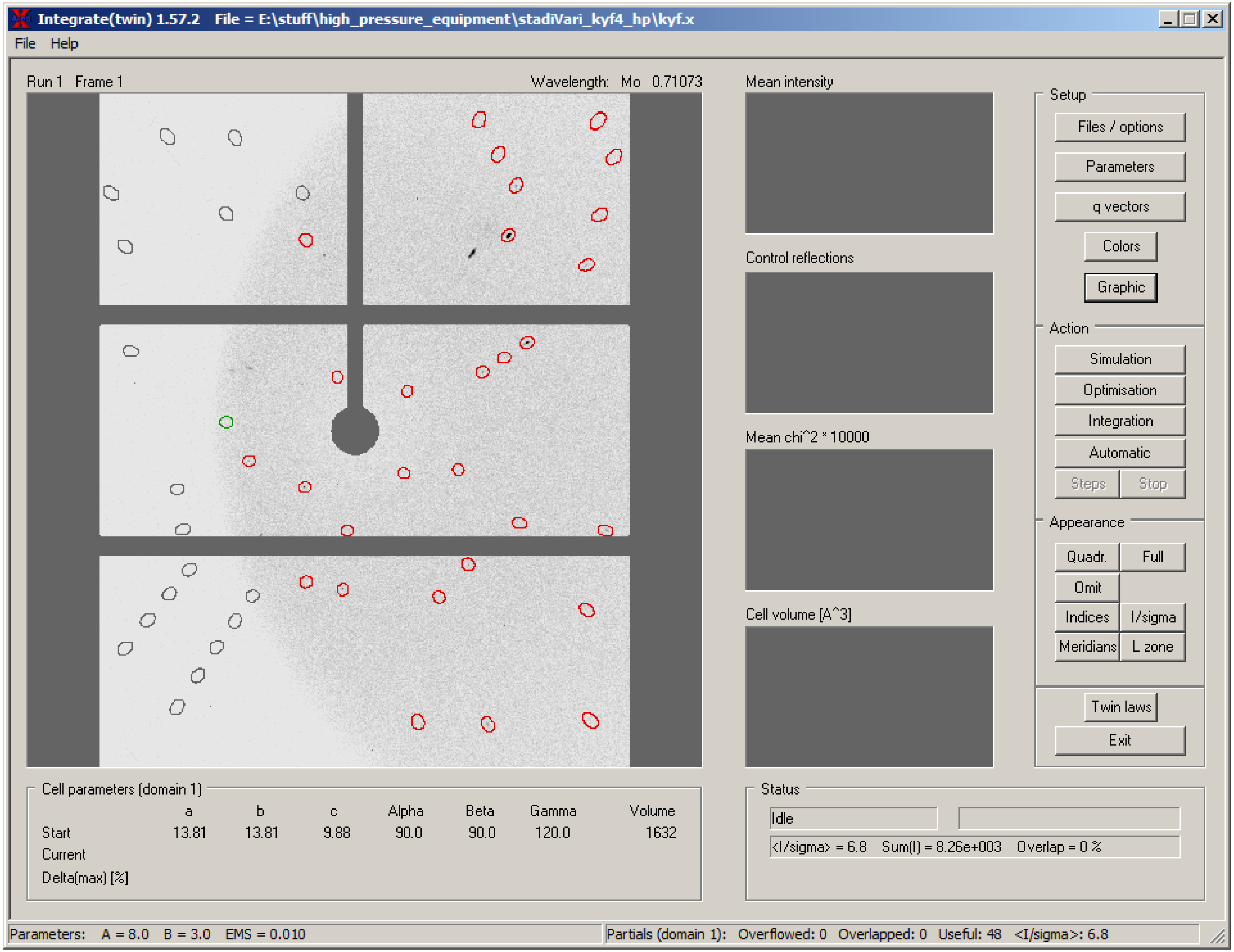
the vector *v* is directing upwards vertically, *w* is directing to the centre of the collimator, and *u* is oriented in a way that a right-handed Cartesian coordinate system is built.

The cone is defined by the vector relating the position of the DAC with respect to the direct beam and the opening angle. When the DAC is perpendicular to the beam at $\omega = 0°$, $\varphi = 0°$, and $\theta = 0°$, the vector is (1 0 0). For $\varphi = 90°$, it is (-1 1 0).

The reflections marked in grey on the following picture are the ones that are outside of the cone.

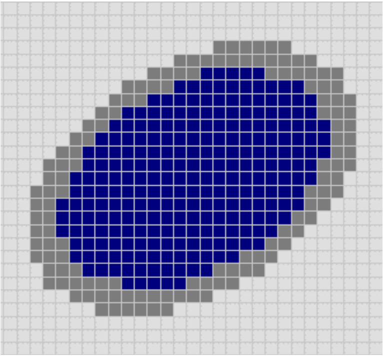
The diamond reflections could be masked by hand in “*Instrument → Define Shading Masks*” (see the presentation on the image plates).

X-Area routines to treat single-crystal data at high pressures from PILATUS



X-Area routines to treat single-crystal data at high pressures from PILATUS

Integration masks

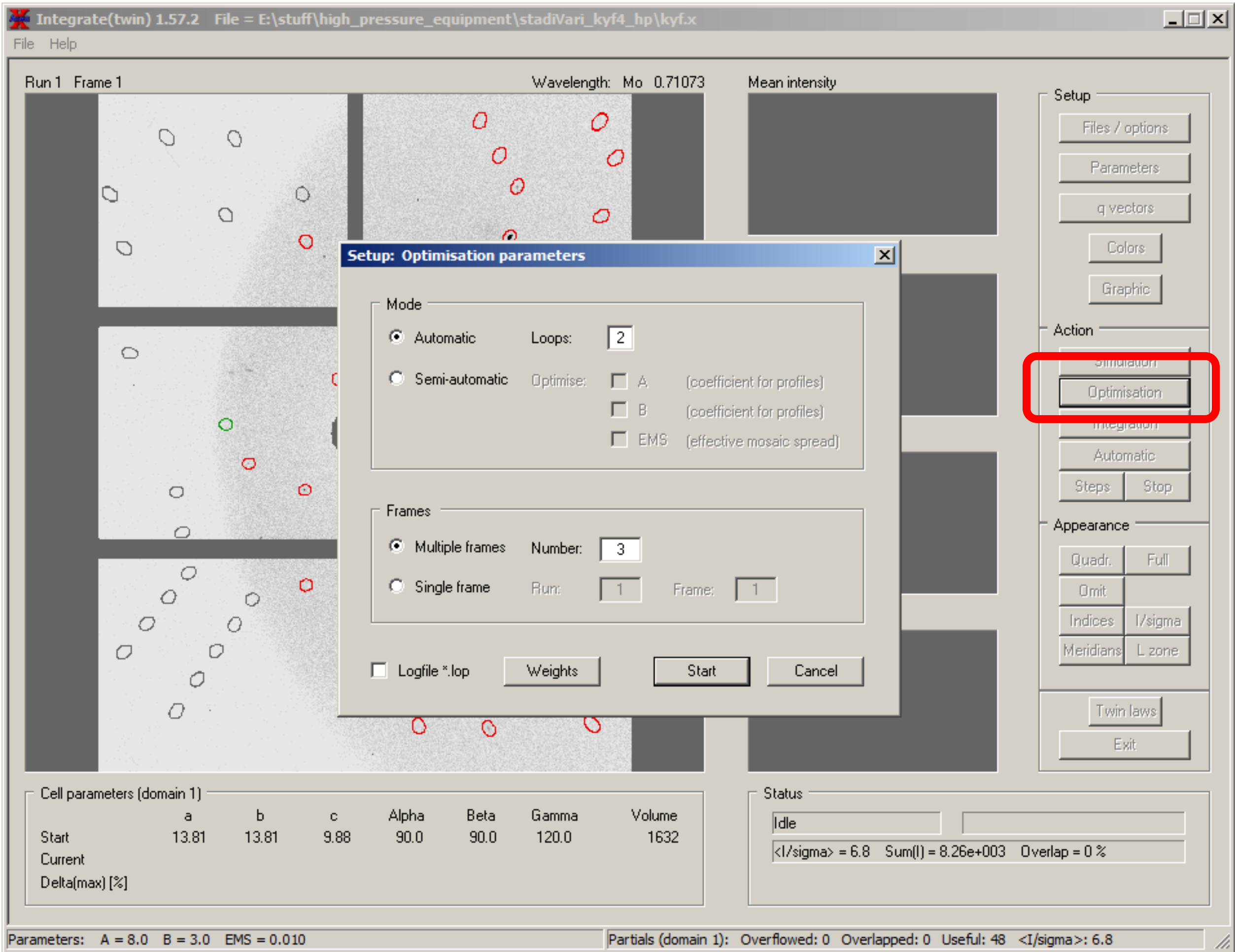


Elliptical masks are used to integrate peak and background intensities. For each mask the smallest diameter is given by W = **A** + **B** tanθ, the largest diameter is defined by W / cos(2θ) + Δλ/λ tanθ (oblique incidence at higher 2θ angles and α1-α2 splitting). For obtaining the peak intensity the inner area of the mask is used. For determining the background, the pixels at the border of the ellipse are taken.

EMS means *effective mosaic spread* and combines the divergence of the primary beam with the mosaic spread of a crystal. For a given instrument setup (the X-ray source, monochromator, and collimator) the beam divergence is constant. However the mosaic spread varies from crystal to crystal, so that **EMS** should be determined for each measured crystal. **EMS** determines how long a reflection is in the reflecting position when it passes through the surface of the Ewald sphere.

The default parameters are **A** = 14, **B** = 4, and **EMS** = 0.01

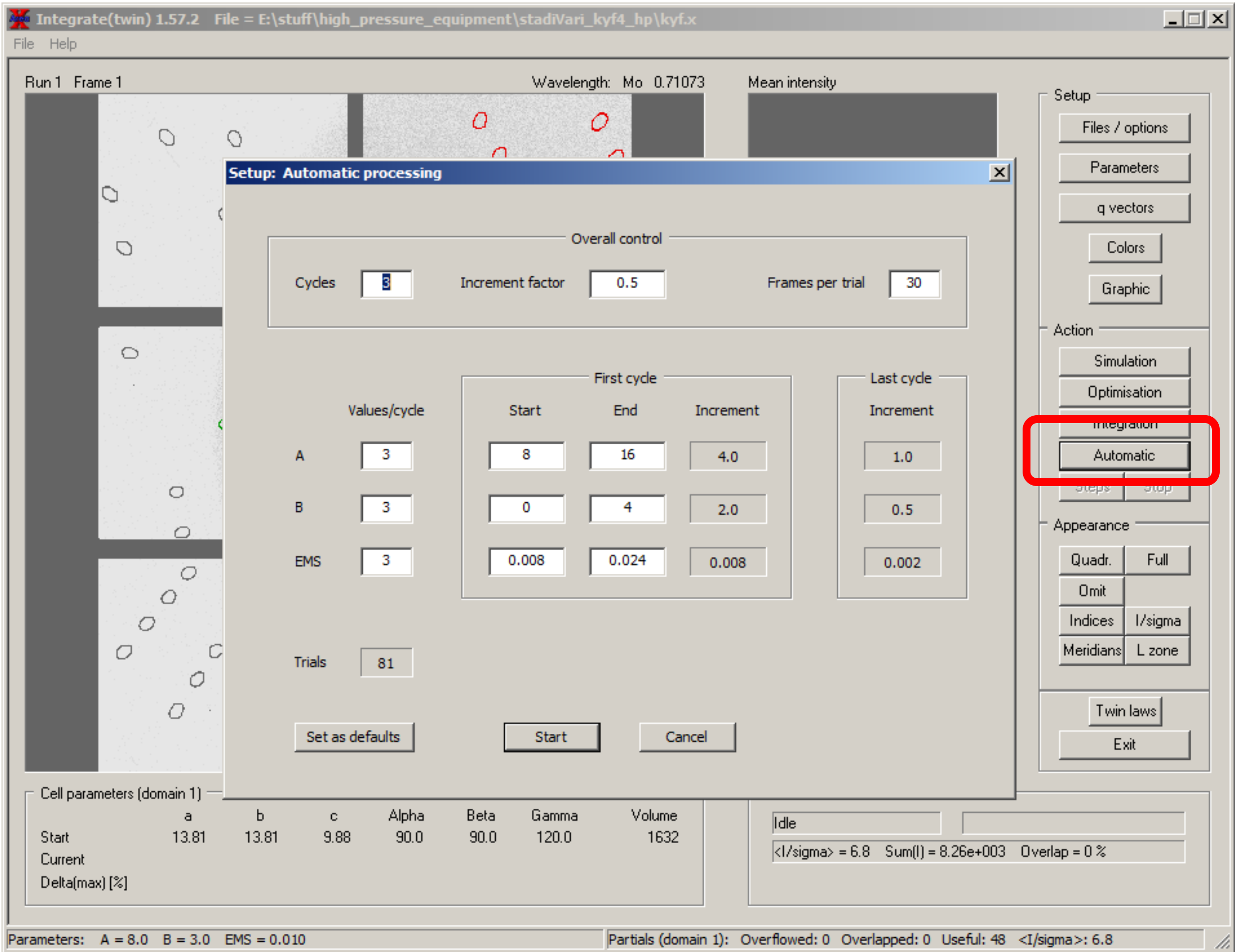
X-Area routines to treat single-crystal data at high pressures from PILATUS



Automatic optimization of A, B, and EMS: multiple frames 3-7

It is a very tricky procedure in the case of large overlap of crystal reflections with diamond reflections and gasket rings. You can't use it without having a proper cone or masks for shaded areas of the detector.
You should never fully trust what this procedure gives you. Visual inspection of the results is imperative.

X-Area routines to treat single-crystal data at high pressures from PILATUS



Automatic optimization of A, B, and EMS: searching for the lowest R_{int}

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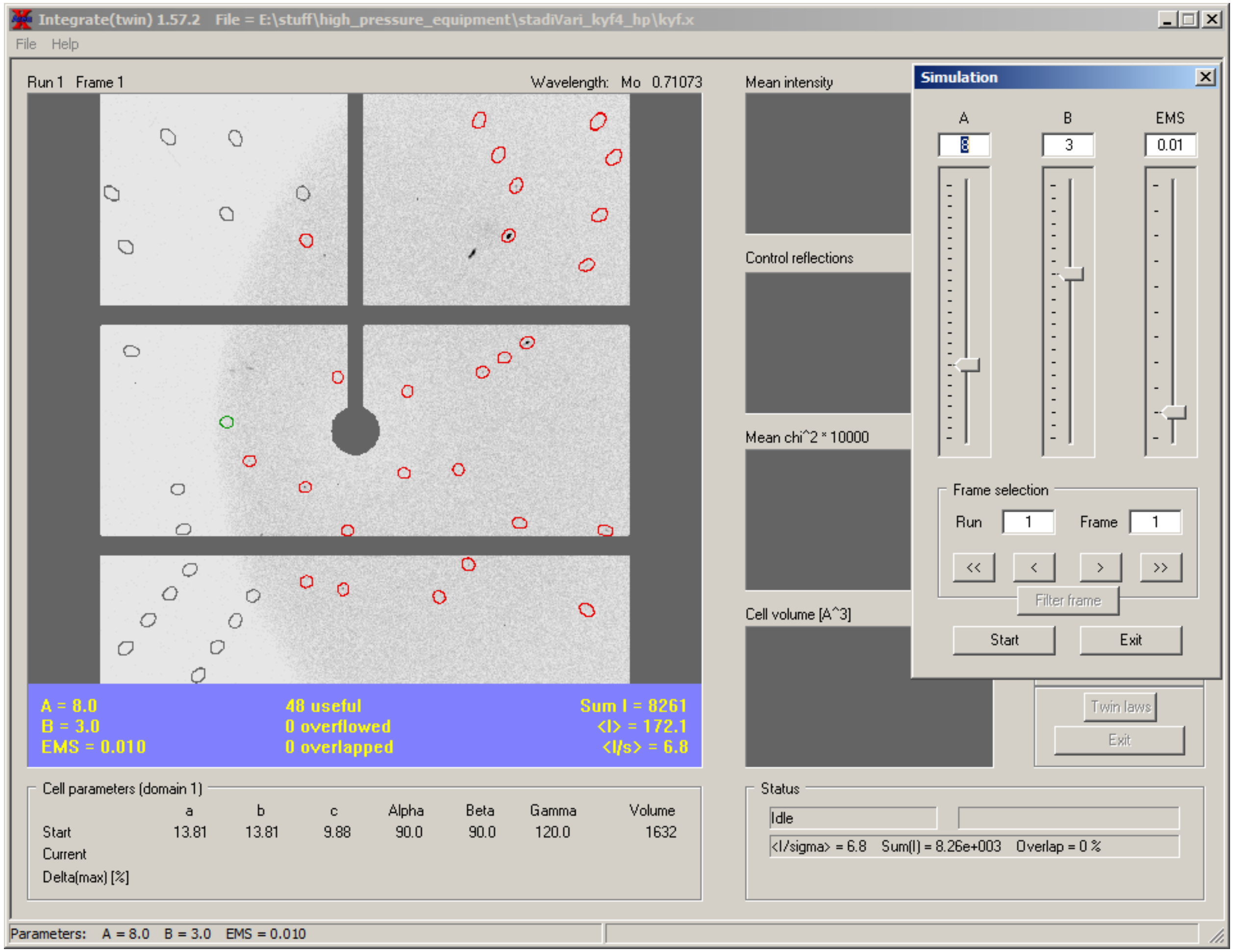

Simulation of the integration masks

Usually the A, B, and EMS parameters for a good crystal of an inorganic solid in a DAC hardly ever are larger than 12, 6, and 0.04, respectively. If any of these parameters is bigger, there must be something wrong with your measurement and/or crystal.

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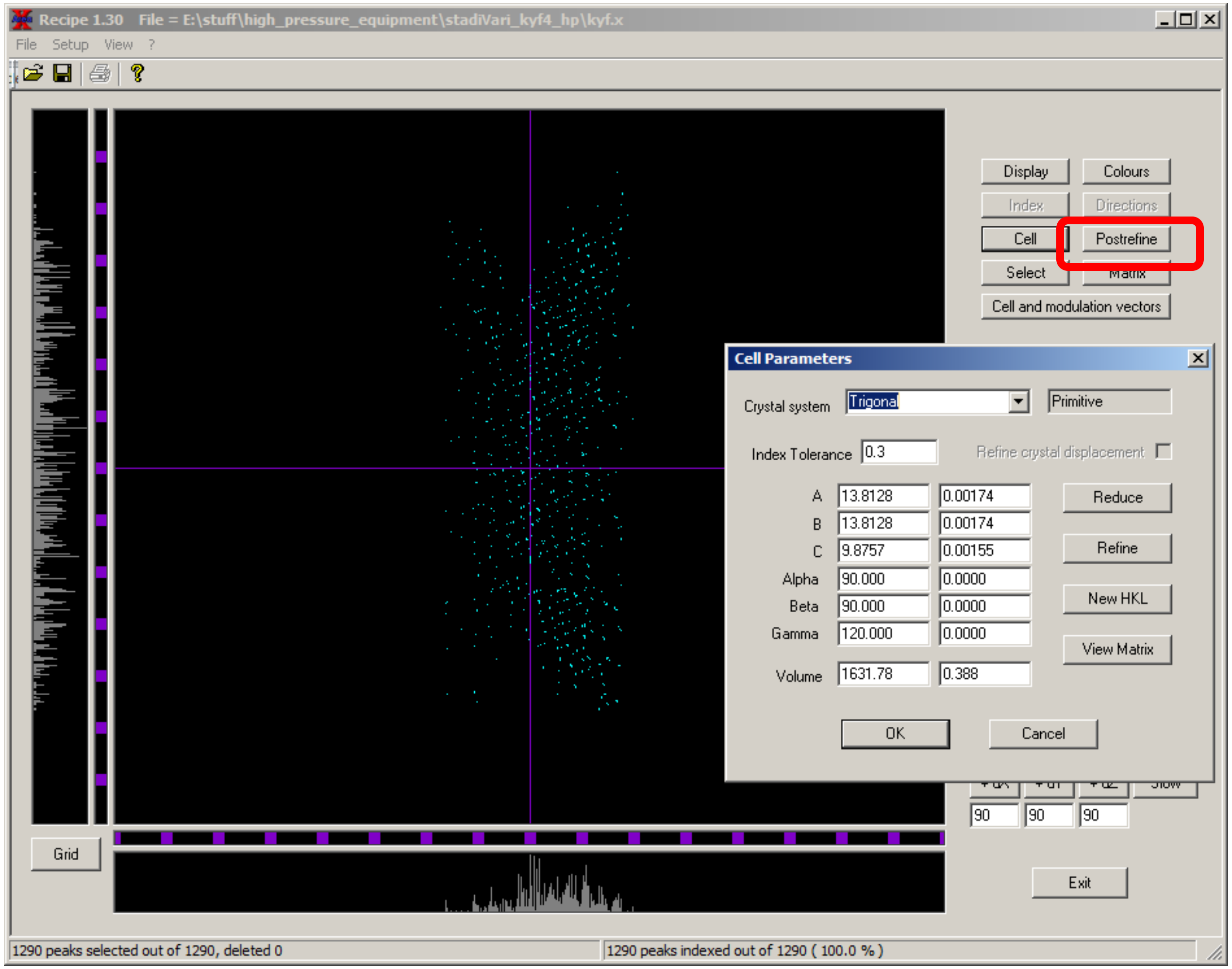

Postrefine – refinements of the orientation matrices of the crystal and two diamonds after integration

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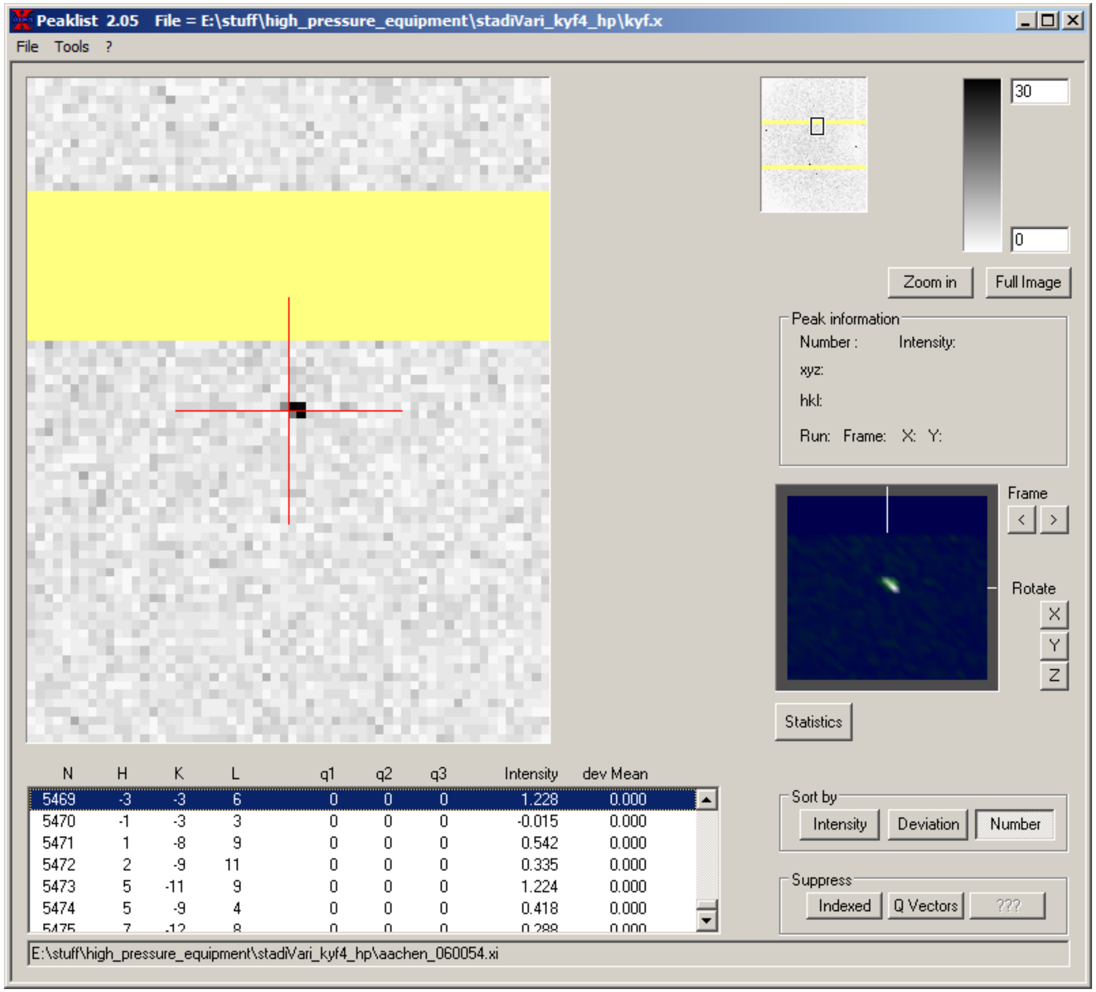

Each reflection could be inspected on the frames in case of doubts about its intensity during data reduction and analysis.

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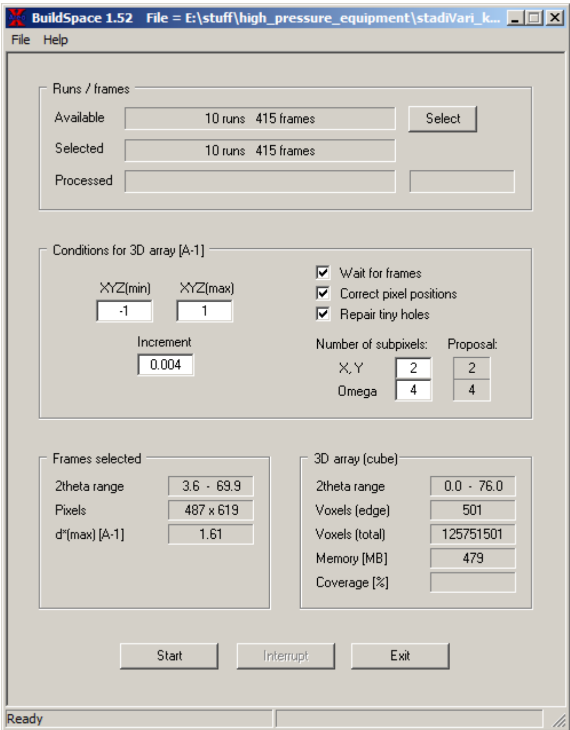
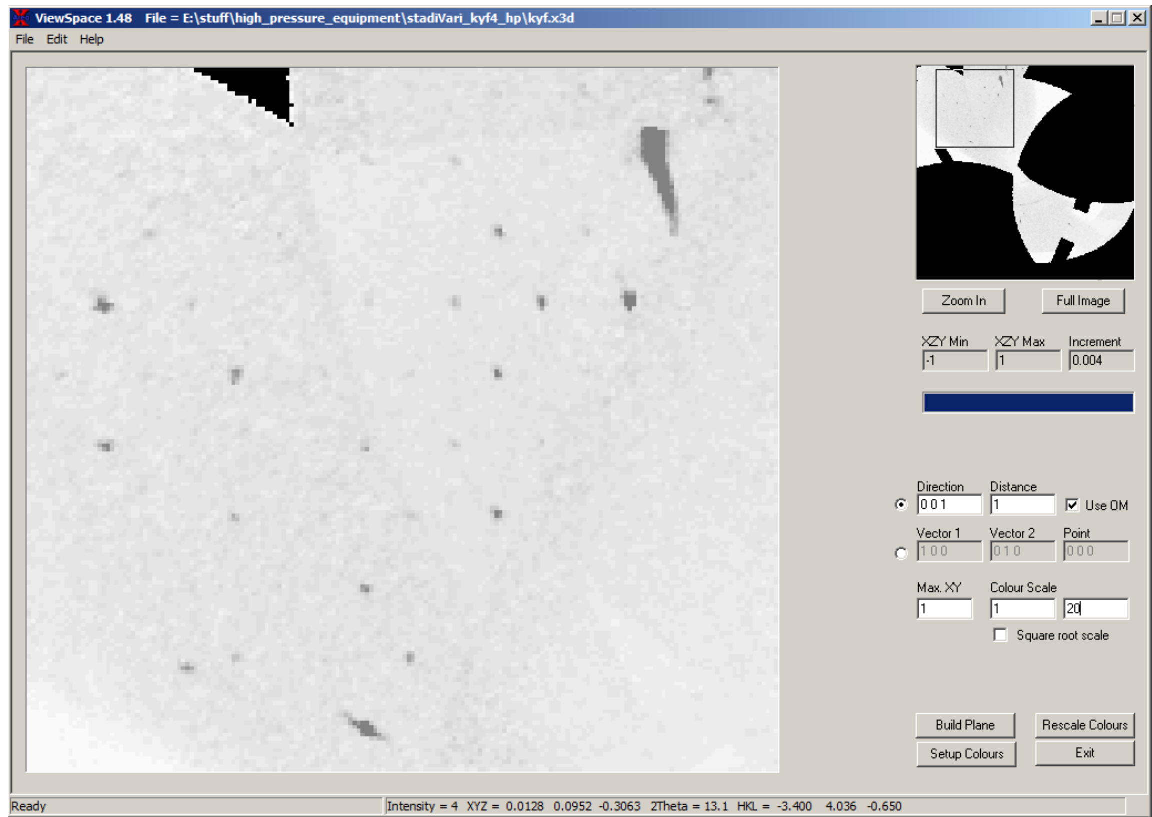
Building and viewing reciprocal space



Reconstruction of reciprocal space

on the basis of the measured frames



Some parts of the text and one figure were taken from the X-Area manual written by STOE. It is worth spending some time on reading it carefully. X-area is not a black-box software and requires conscious actions from the user.