X-Area

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

from STOE image plates and PILATUS detectors

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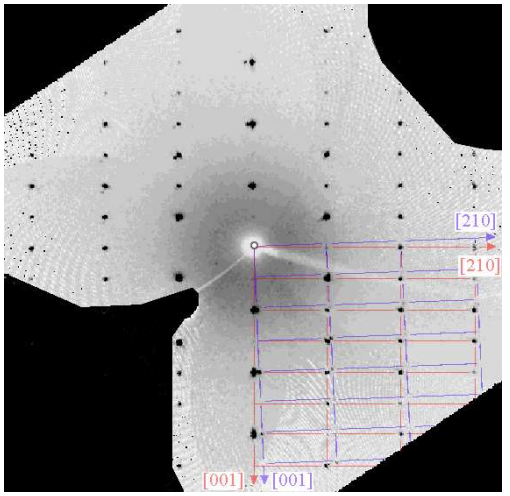
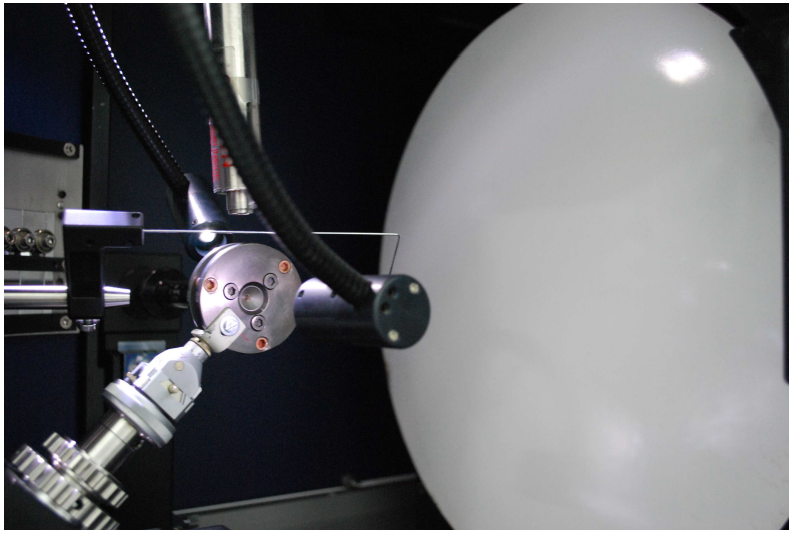
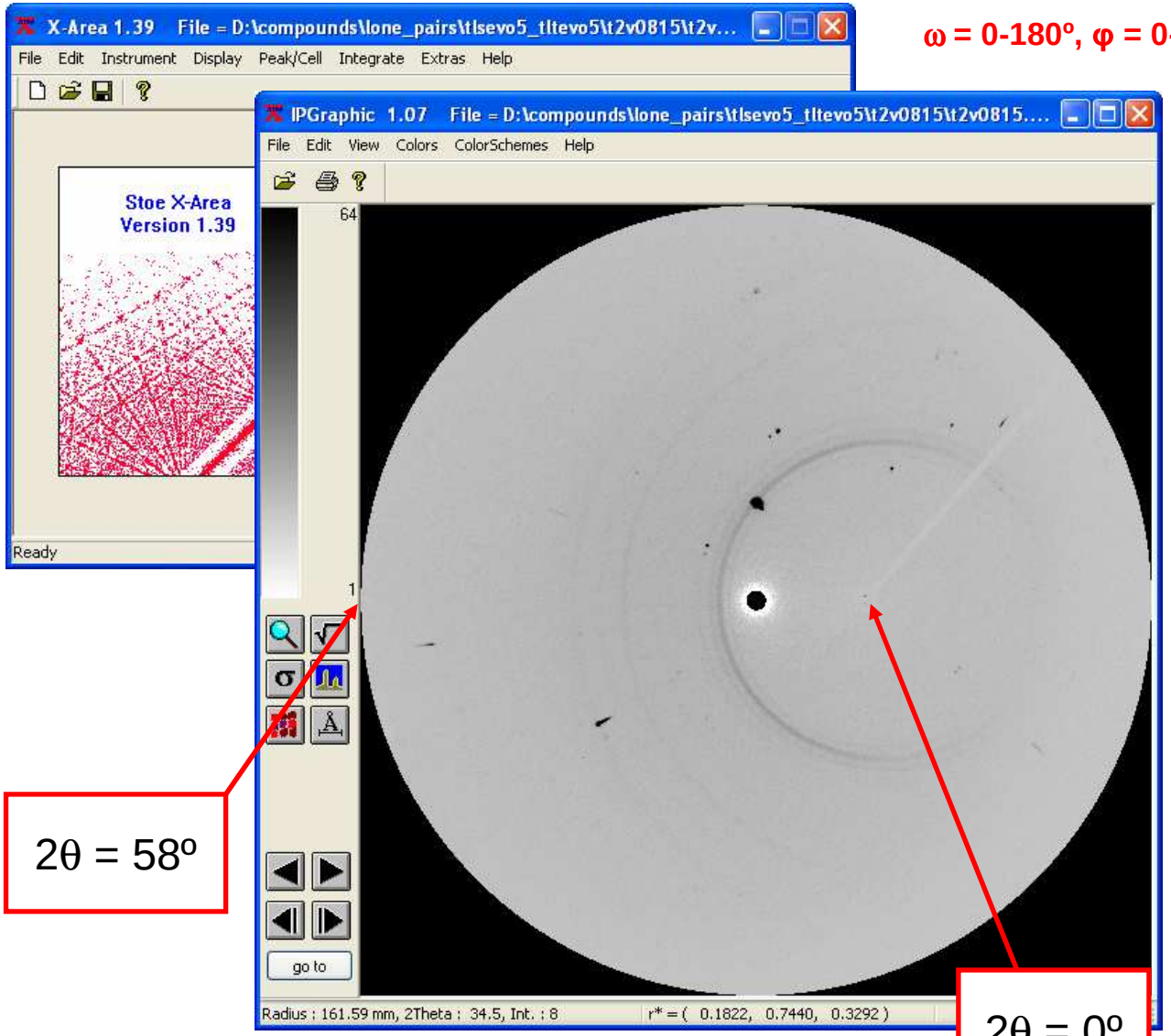
Crystallographic problems that X-Area handles without any problems

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



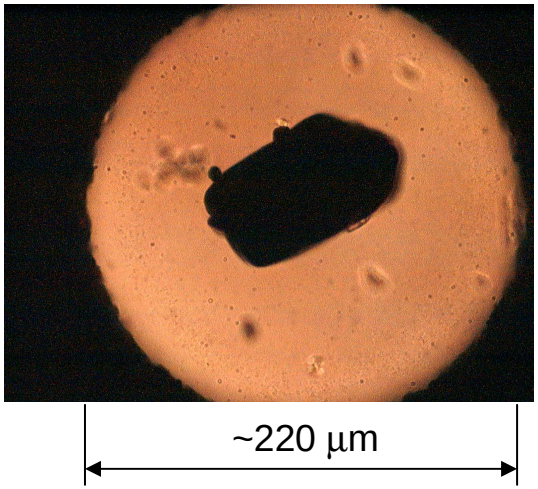


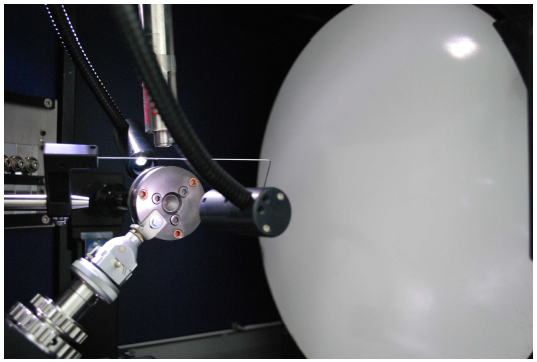
X-Area routines to treat single-crystal data at high pressures



The diameter of the image plate of 340 mm

ω = 0-180º, φ = 0-360º, χ = 45º, 2θ = 0, 15, 30, 45, 60º





IPDS-2T (STOE)
Mo-Kα

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

Exposures from both sides of the diamond anvil cell at $2\theta = 0^{o}$ and $2\theta = 15^{o}$

$\Delta\omega = 2^{o}$

Exposure time per frame between 2 and 10 minutes

Nearly 400 frames for one measurement (depending on the opening angles of the diamond anvil cell)

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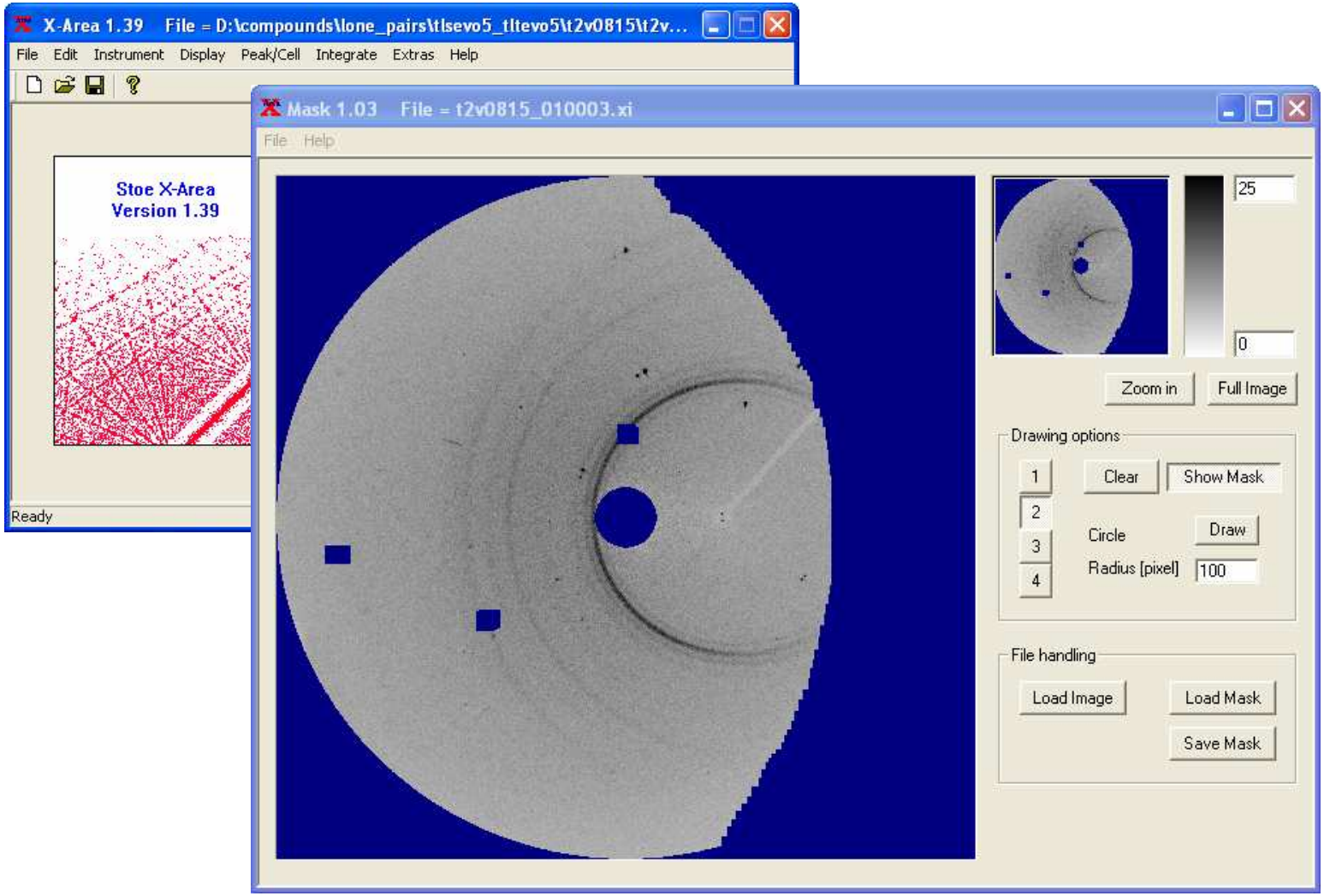


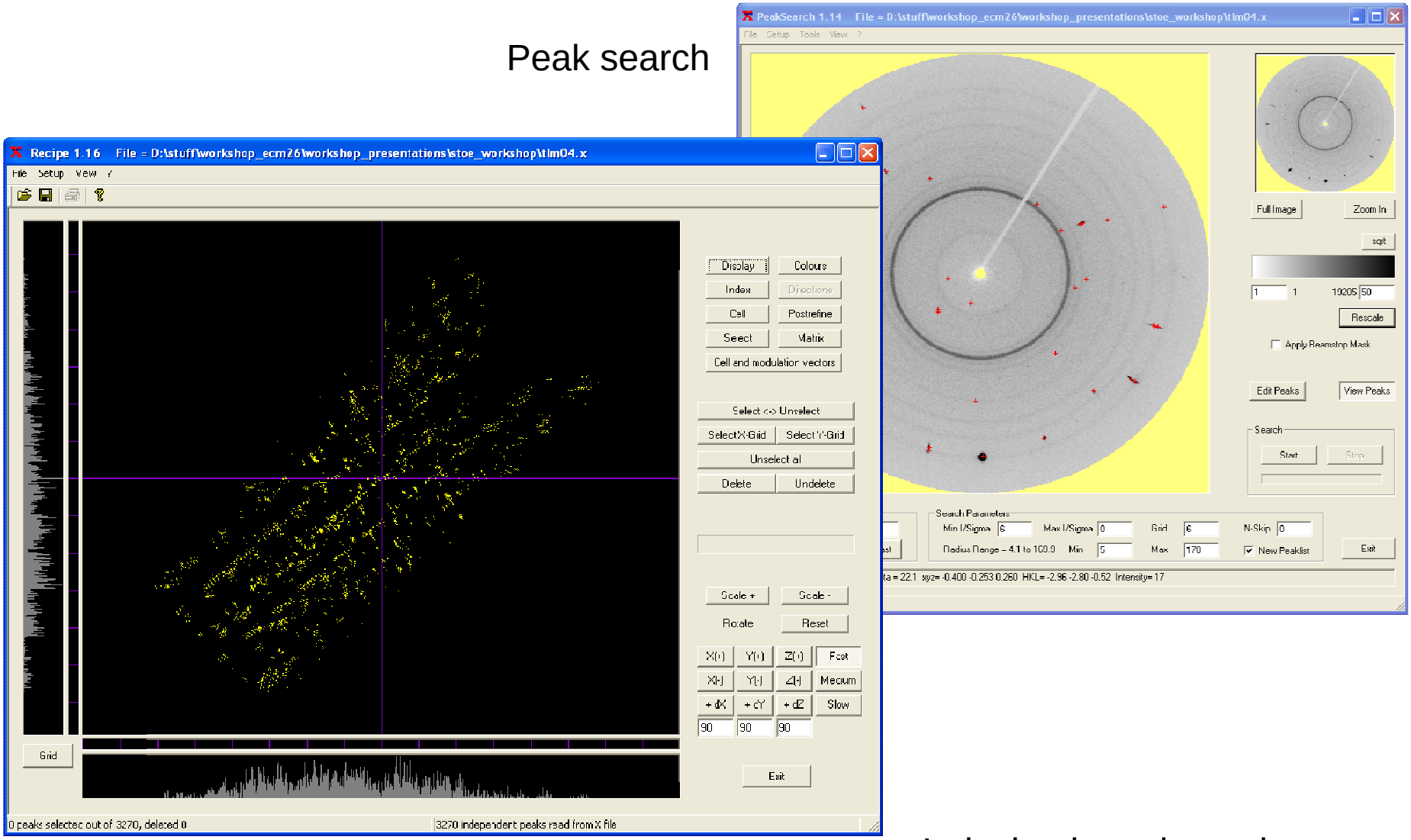
Masking the shaded areas of the detector (each frame has its own mask)

The masks could be re-used for any other data collected in the same DAC.

Instead of masks, the program ABSORB could be used after integration to eliminate the shaded reflections.

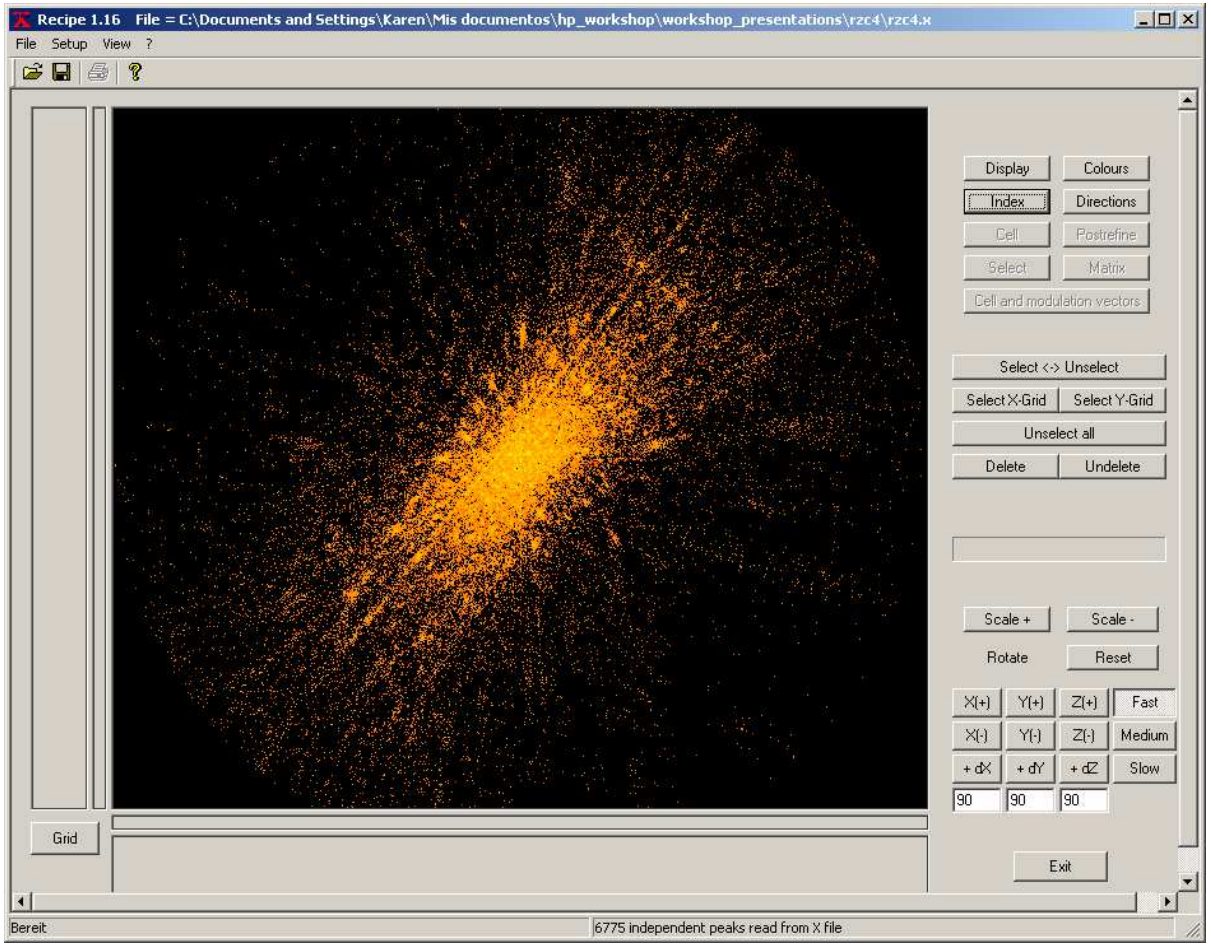
X-Area routines to treat single-crystal data at high pressures

Peak search



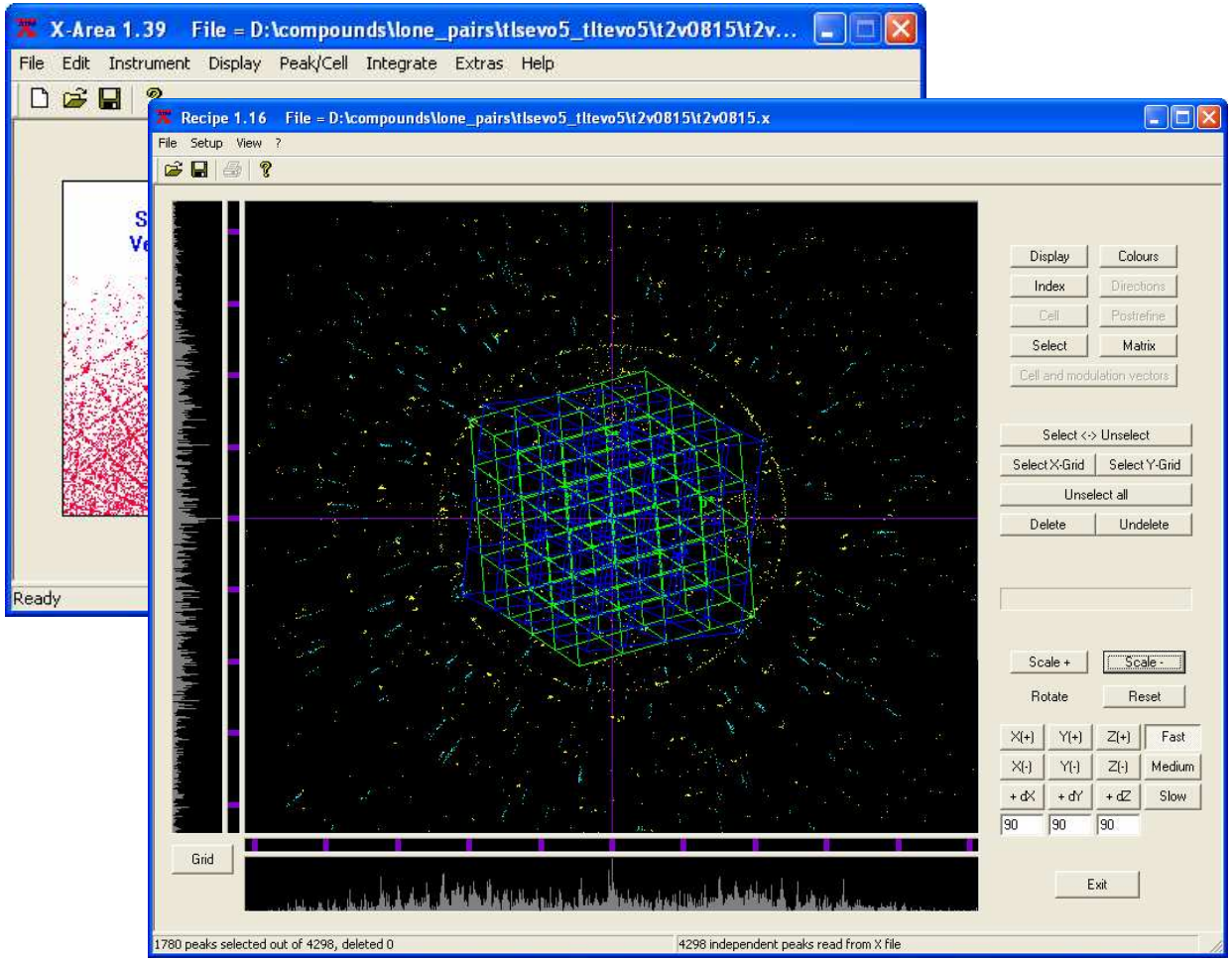
Indexing in reciprocal space

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



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



The diamond orientation matrices are useful to check the alignment. They could be used for any other measurements in the same DAC.

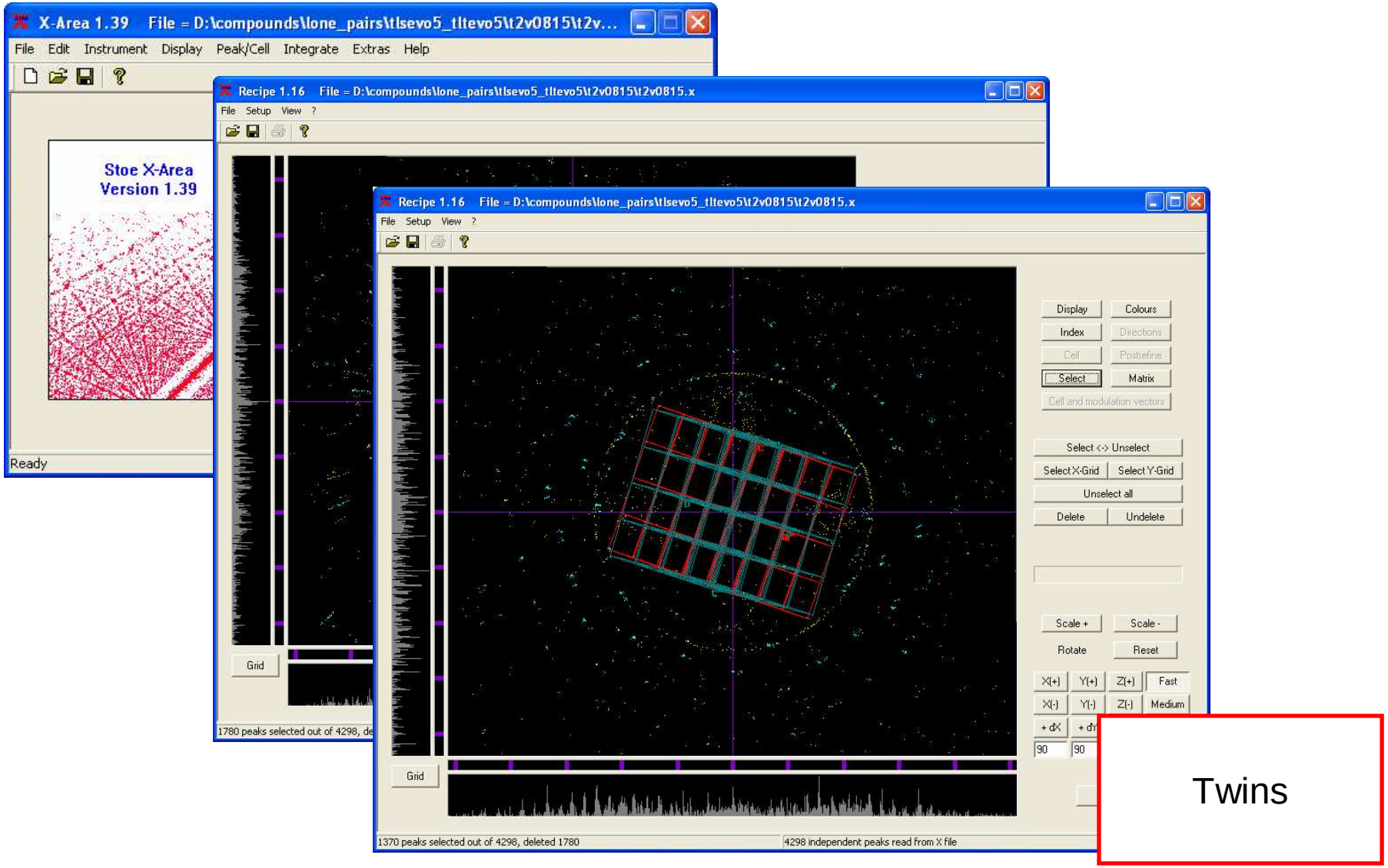
Two diamond matrices

cF, a ≈ 7.1 Å

Diamond λ/2 reflections

A *contamination* by the "half-wavelength" component of the Mo spectrum both in a crystal-monochromated beam and in a filtered beam.

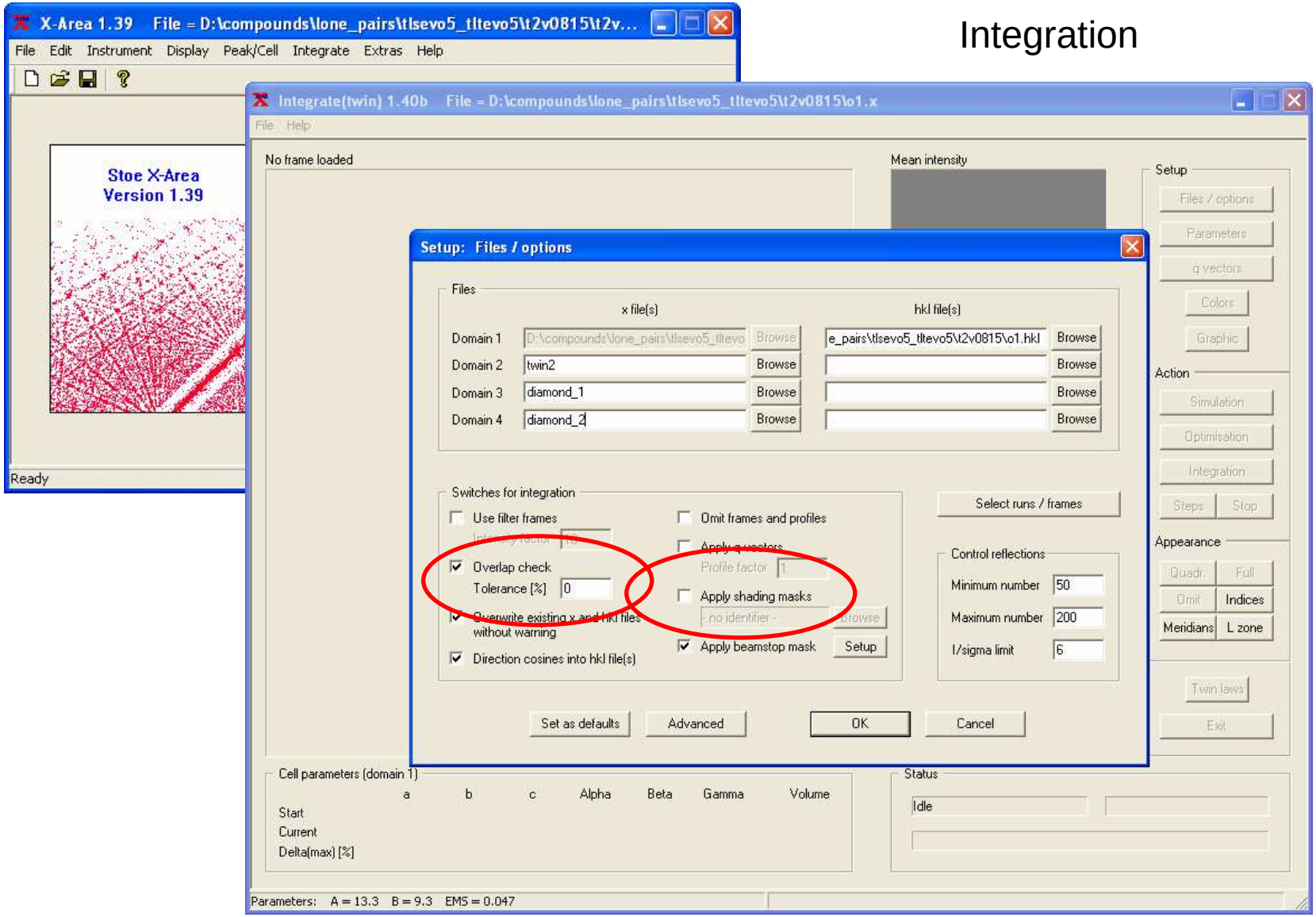
X-Area routines to treat single-crystal data at high pressures



Twins

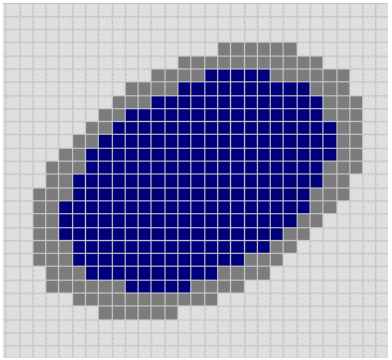
X-Area routines to treat single-crystal data at high pressures

Integration



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

Integration masks

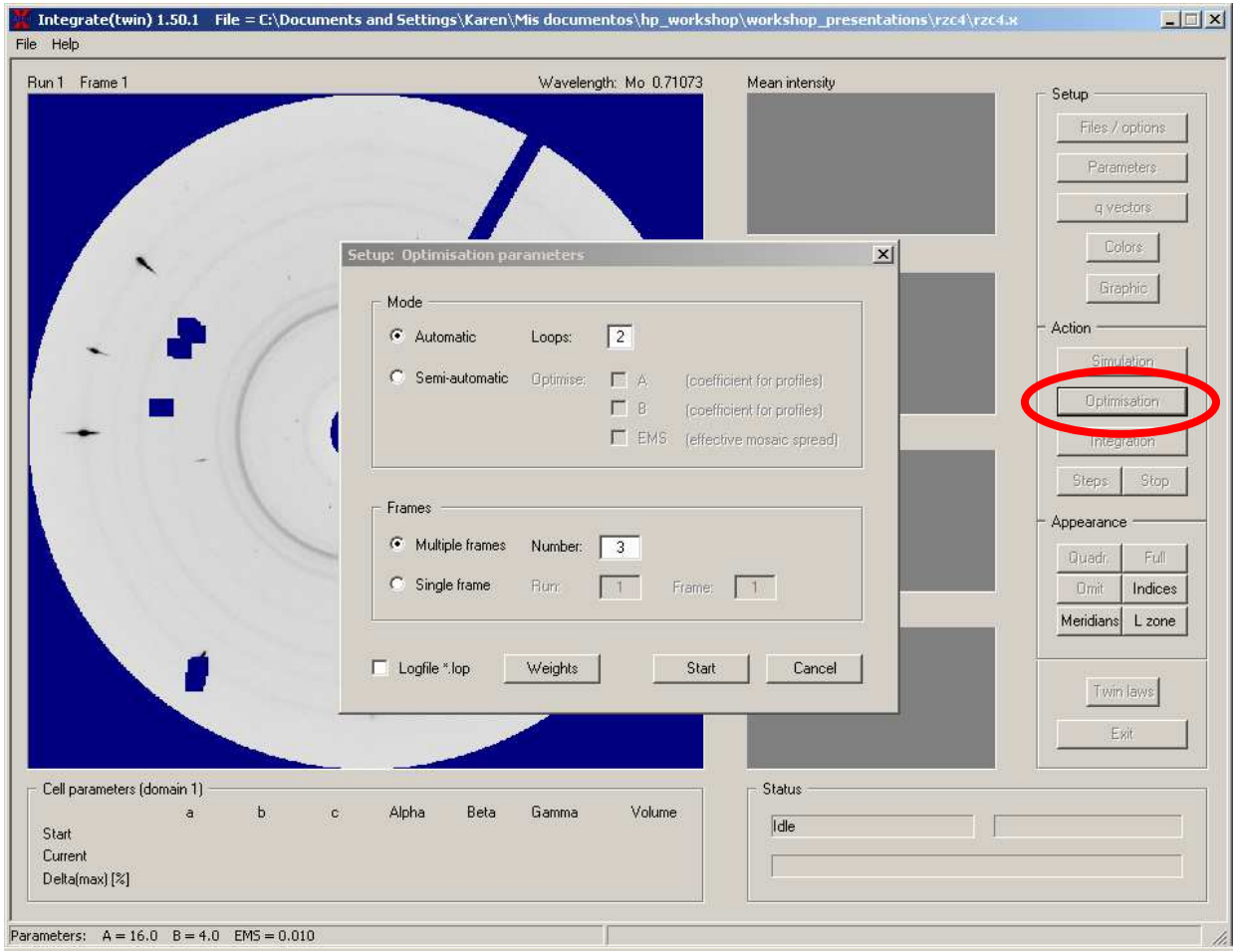


Elliptical masks are used to integrate peak and background intensities. For each mask the smallest diameter is given by W = **A** + **B** tanθ, the largest diameter is defined by W / cos(2θ) + Δλ/λ tanθ (oblique incidence at higher 2θ angles and α1-α2 splitting). For obtaining the peak intensity the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

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

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

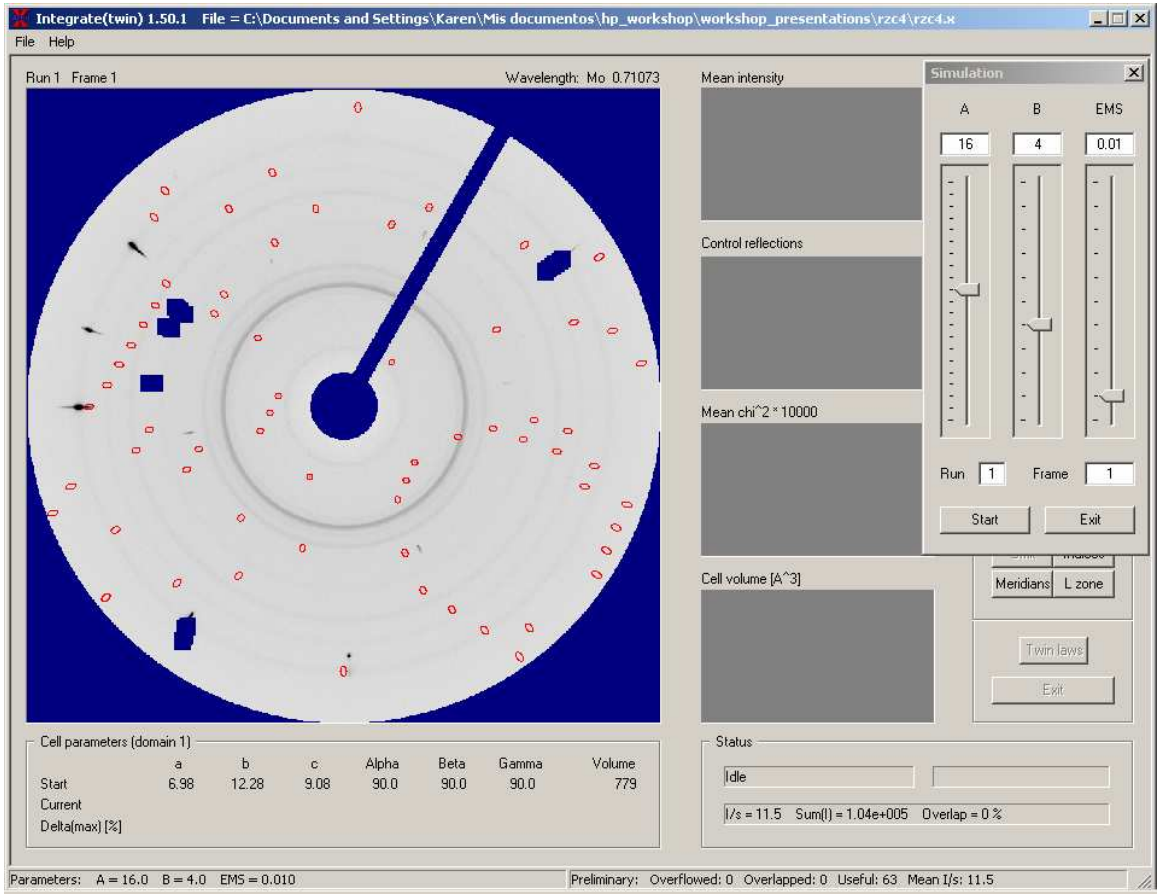


Automatic optimization of A, B, and EMS: multiple frames 3-7

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 proper 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

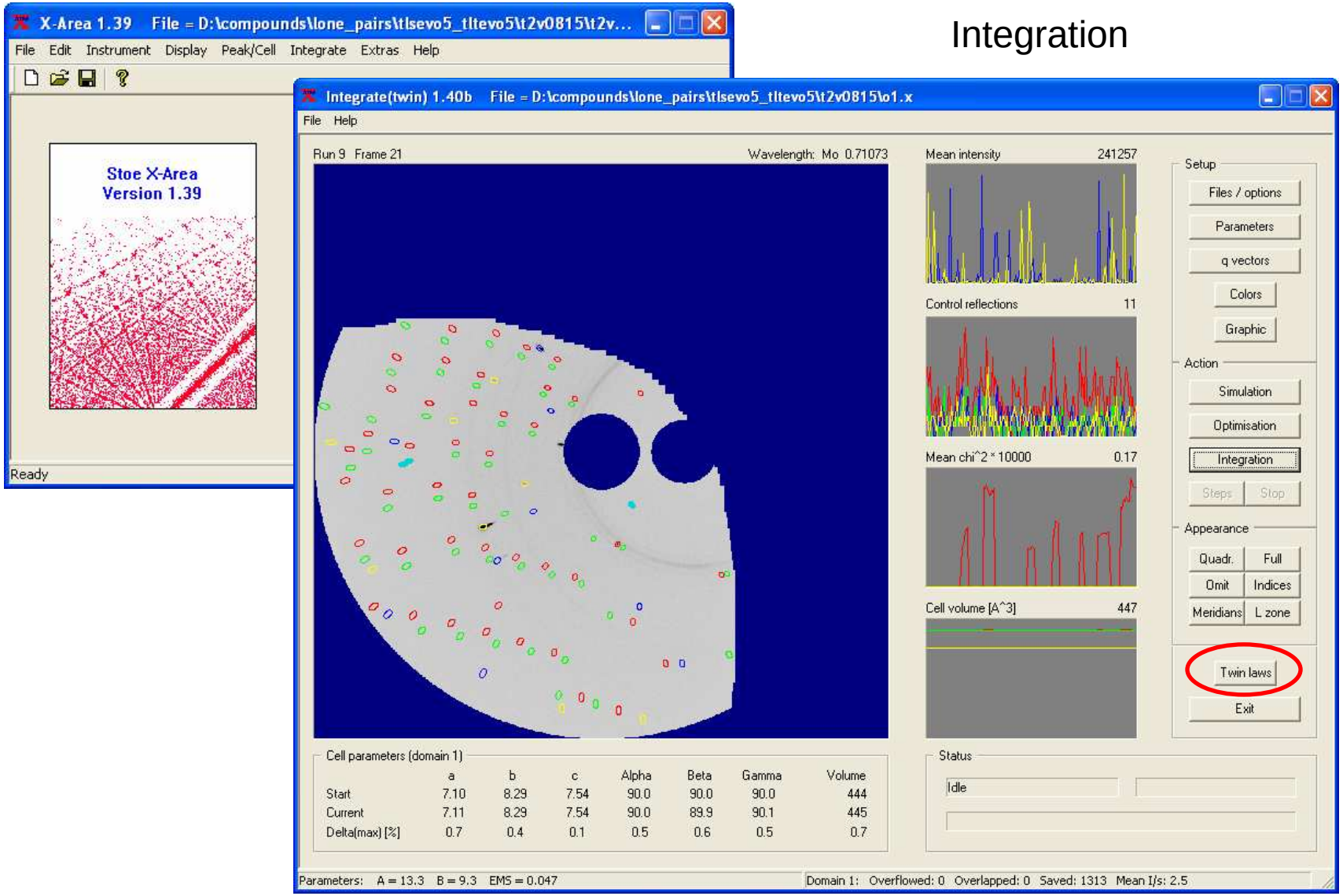


Simulation of the integration masks.

Usually the A, B, and EMS parameters for a good crystal of an inorganic solid in a DAC hardly ever are larger than 20, 6, and 0.04, respectively. If any of these parameters is bigger, there must be something wrong with your measurement and/or crystal.

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

Integration



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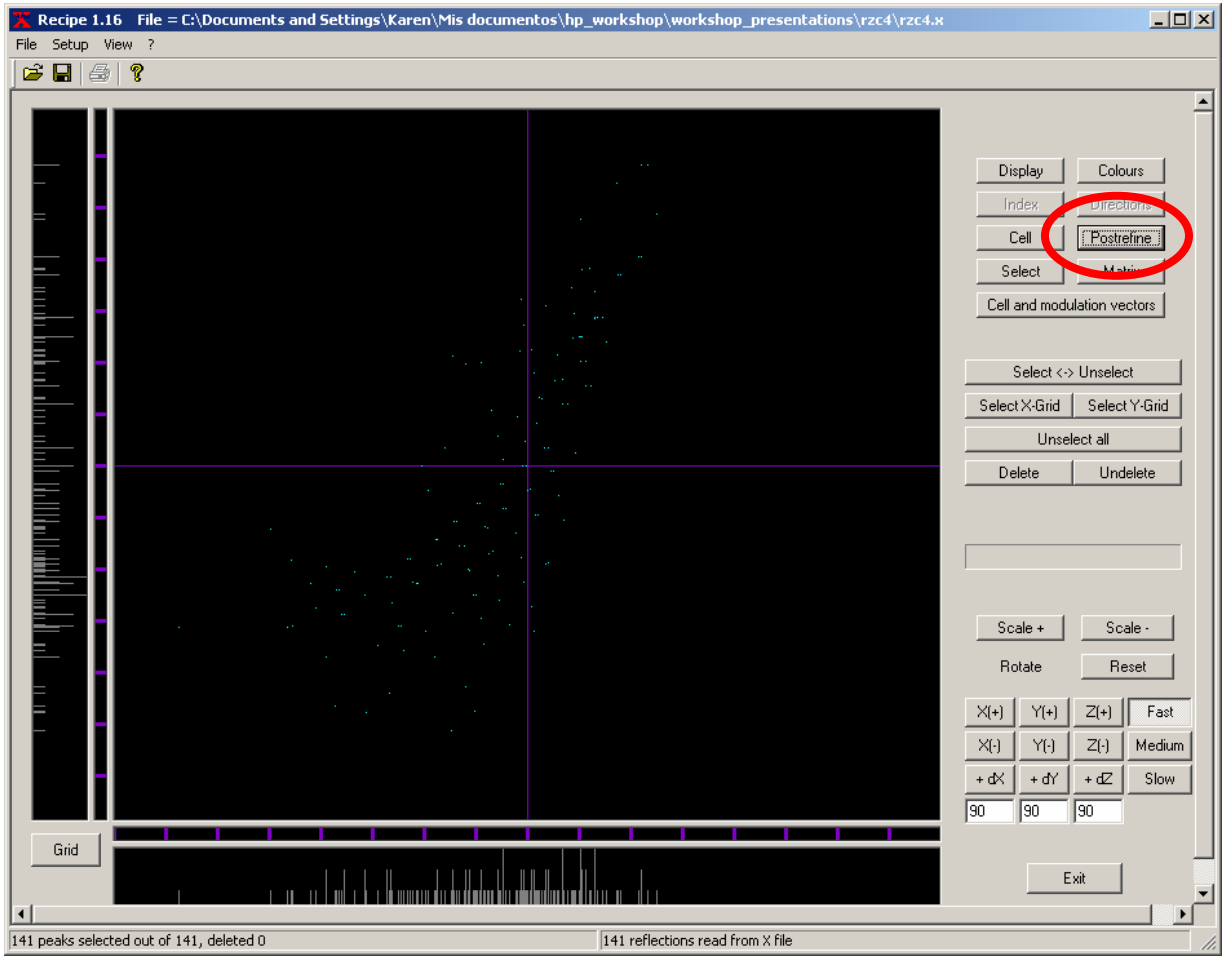
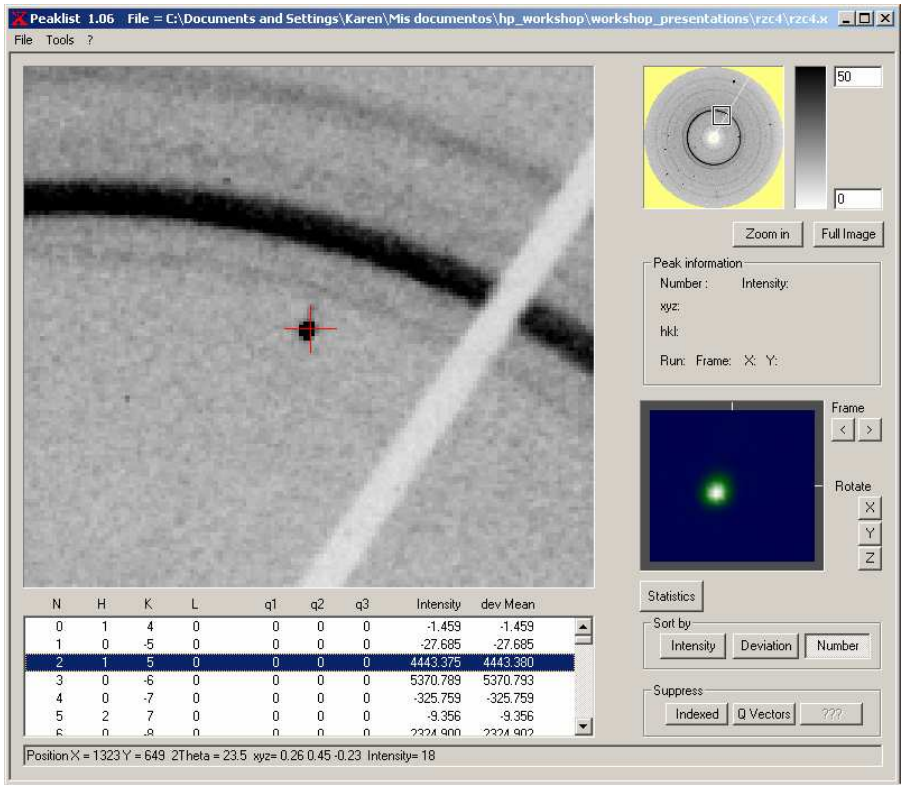
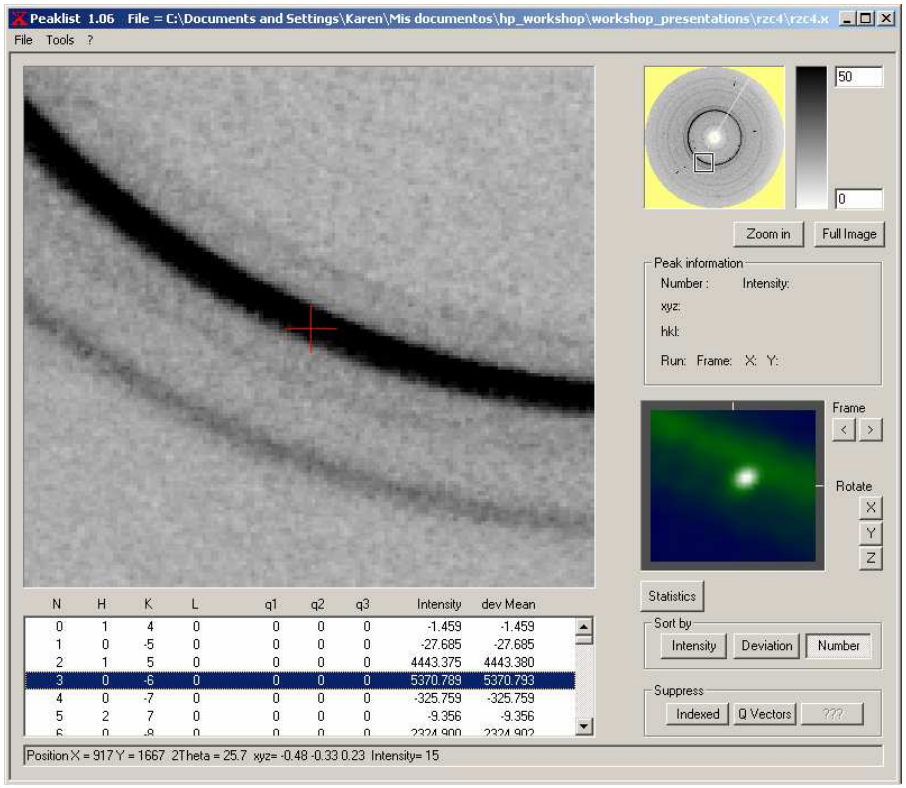


Postrefine – refinements of the orientation matrices of the crystal and two diamonds after integration

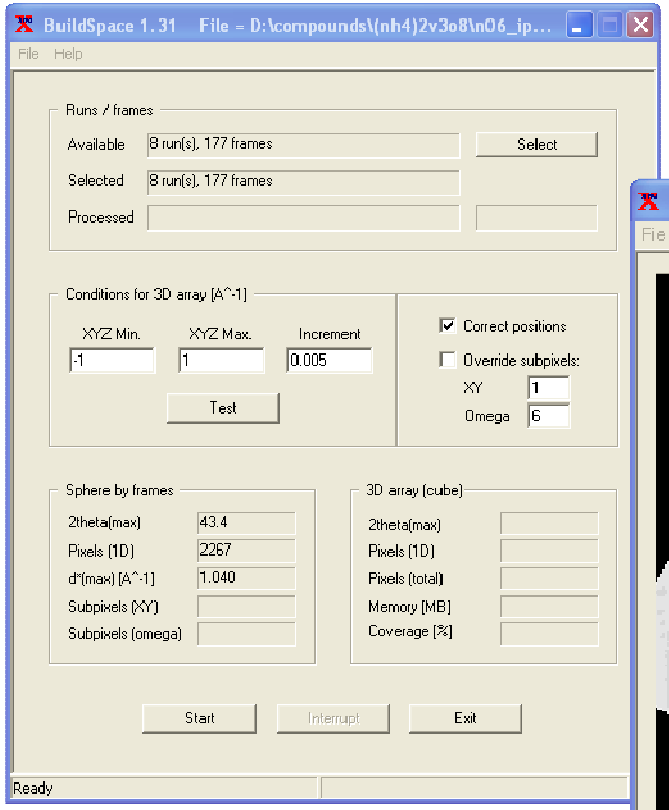
X-Area routines to treat single-crystal data at high pressures



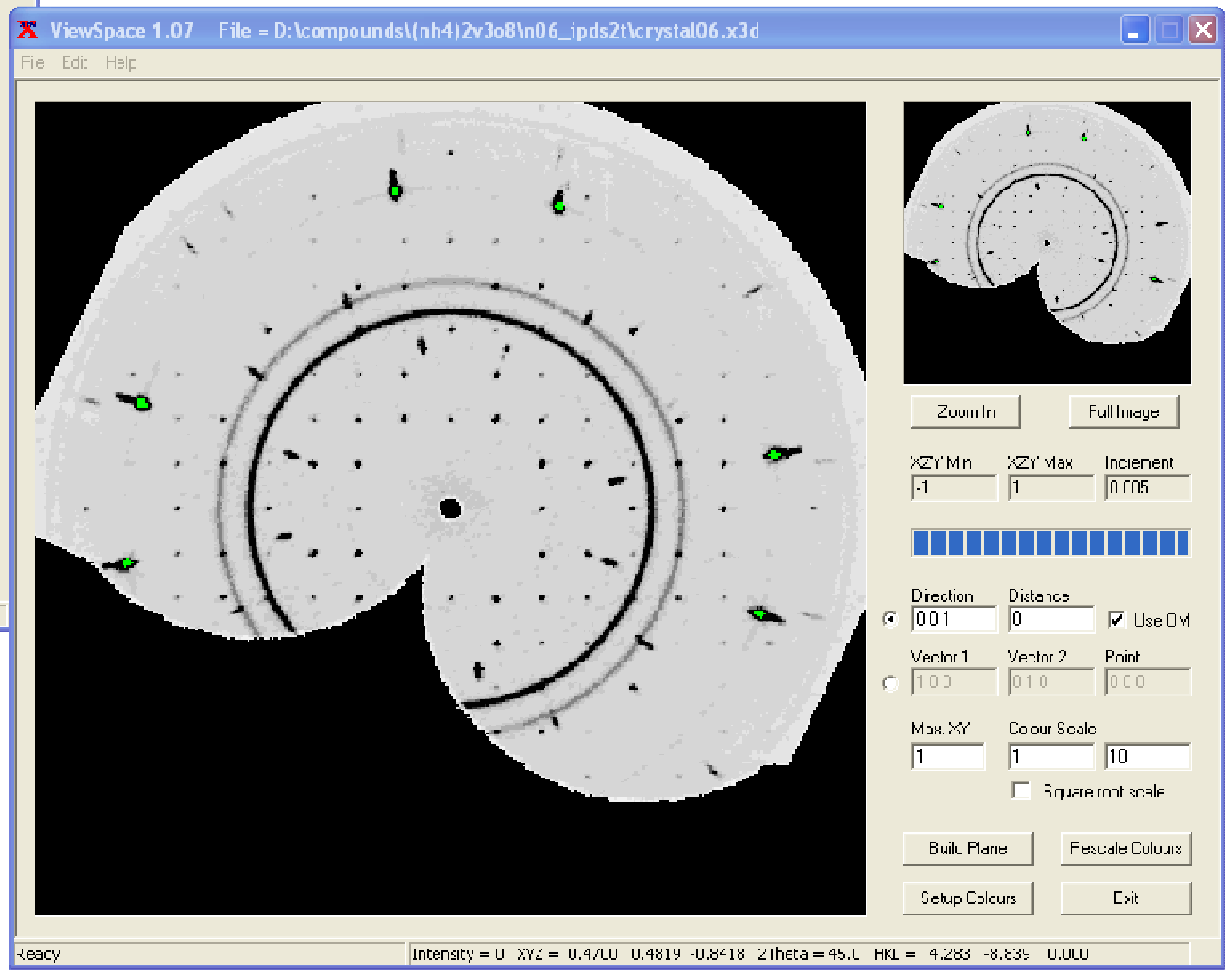


Each reflection could be inspected on the frames in the case you have doubts about its intensity during data reduction and analysis.

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



Building and viewing reciprocal space



Reconstruction of reciprocal space on the basis of the measured frames

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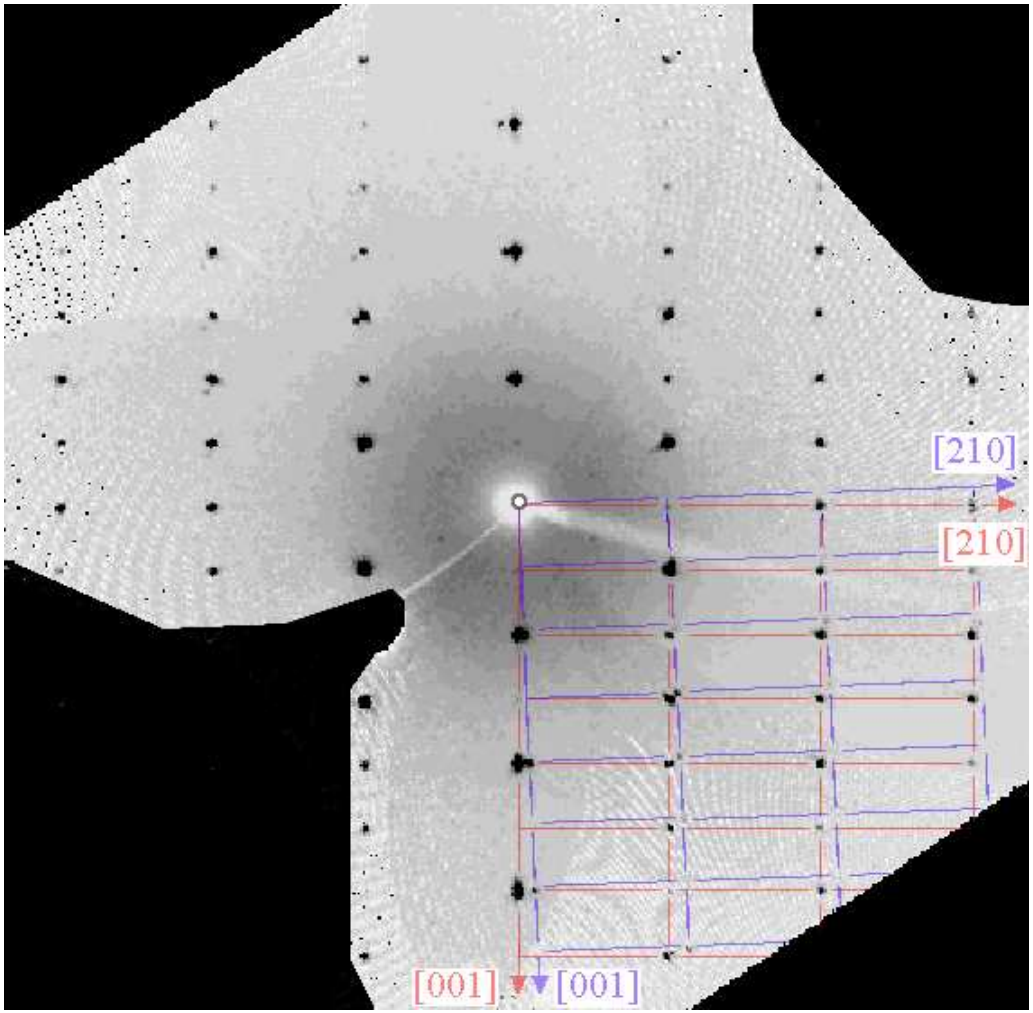


Twinning at high pressures

in the reciprocal space

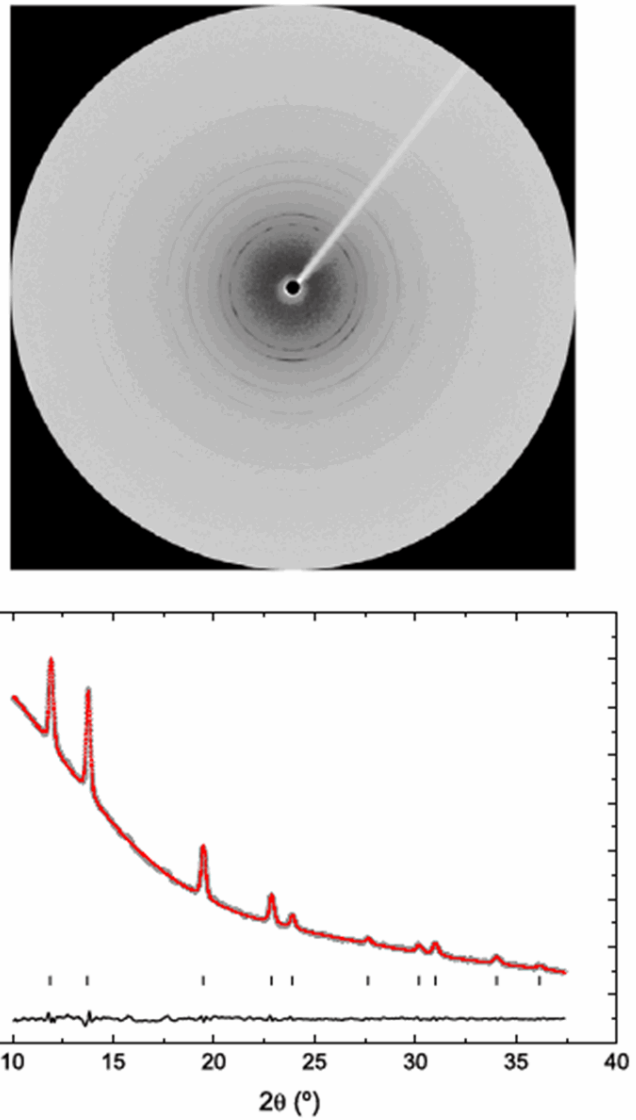
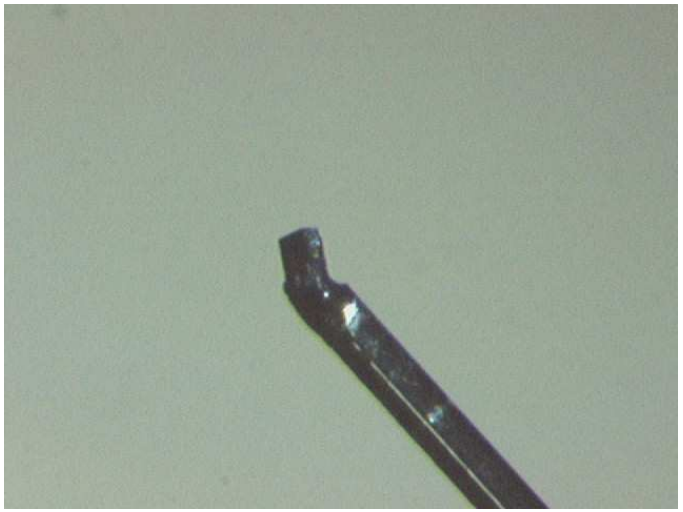


Figure 4. A Gandolfi-like diffraction diagram of the PbS single crystal decompressed from 7.73 GPa (top) and the corresponding integrated powder diagram refined with the Le Bail method assuming the $Fm\bar{3}m$ lattice (bottom). Vertical markers indicate Bragg reflections.

Gandolfi-like diffraction diagram of a single crystal processed with X-Area



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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.