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Pseudo merohedral twinning - How to treat a six-fold twin.

Multiple domains, Six-fold twin

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Introduction

In dealing with pseudo-merohedral twins, cell refinement and data integration need special care, due to the presence of closely overlapping spots. Careful selection of trusted areas to refine the cell in, will improve the reliability of the cell parameters.

One of the pitfalls during integration of data from pseudo-merohedral twins is that the spot shape changes continuously due to the systematic overlap of areas of adjacent spots.
The described approach can be used for all multi-domain pseudo-merohedral cases. It is illustrated using a six-fold twin that shows a reversible transition from triclinic cells to single crystal state with a hexagonal cell.

Steps and tools

- *Harvest spots* from all frames (~10.000 spots.)
- *Index* on low resolution data.
- *Lattice Overlay* to isolate one domain.
- *Select area* to remove overlapping peaks.
- *Refine* cell of domain 1 against the selected peaks.
- *Transform unit cell* to create 5 remaining cells by rotating multiples of 60 degrees around the c-axis.
- *Integrate* the images with all 6 cells.
- *Scale* and correct for absorption using TWINABS [1].
- *Solve* the structure from the HKLF4 data.
- *Refine* the structure against the HKLF5 data.

Indexing and domain orientation

The initial indexing result is severally biased towards the overlap position, resembling hexagonal setting. By visualizing the peaks in 3D space, 'trusted' areas can be identified. The pseudo precession pictures will help identifying in which planes spots can be separated.
It also can be seen that the spot positions of the triclinic cell at low temperature, have inherited the hexagonal arrangement from the room temperature cell.
This relation was confirmed by CELL_NOW [2].
The hk0 plane only contains exactly overlaid peak positions and should be included in the cell refinement. Planes from hk1 to hk5 were excluded due to presence of systematically partial overlapping peaks. The higher order hk-planes have separated spots.
The reversible (triclinic/hexagonal) cell properties of this sample favors the use of generated cells derived from one well refined domain.

References
[1] Sheldrick, G.M. (2002). TWINABS, Bruker AXS, Madison.
[2] Sheldrick, G.M. (2003). CELL_NOW, Bruker AXS, Madison.
[3] Bruker (2001). SAINT, Bruker AXS, Madison.
[4] Sheldrick, G.M. (1997). SHELXL-97, Uni. Göttingen.

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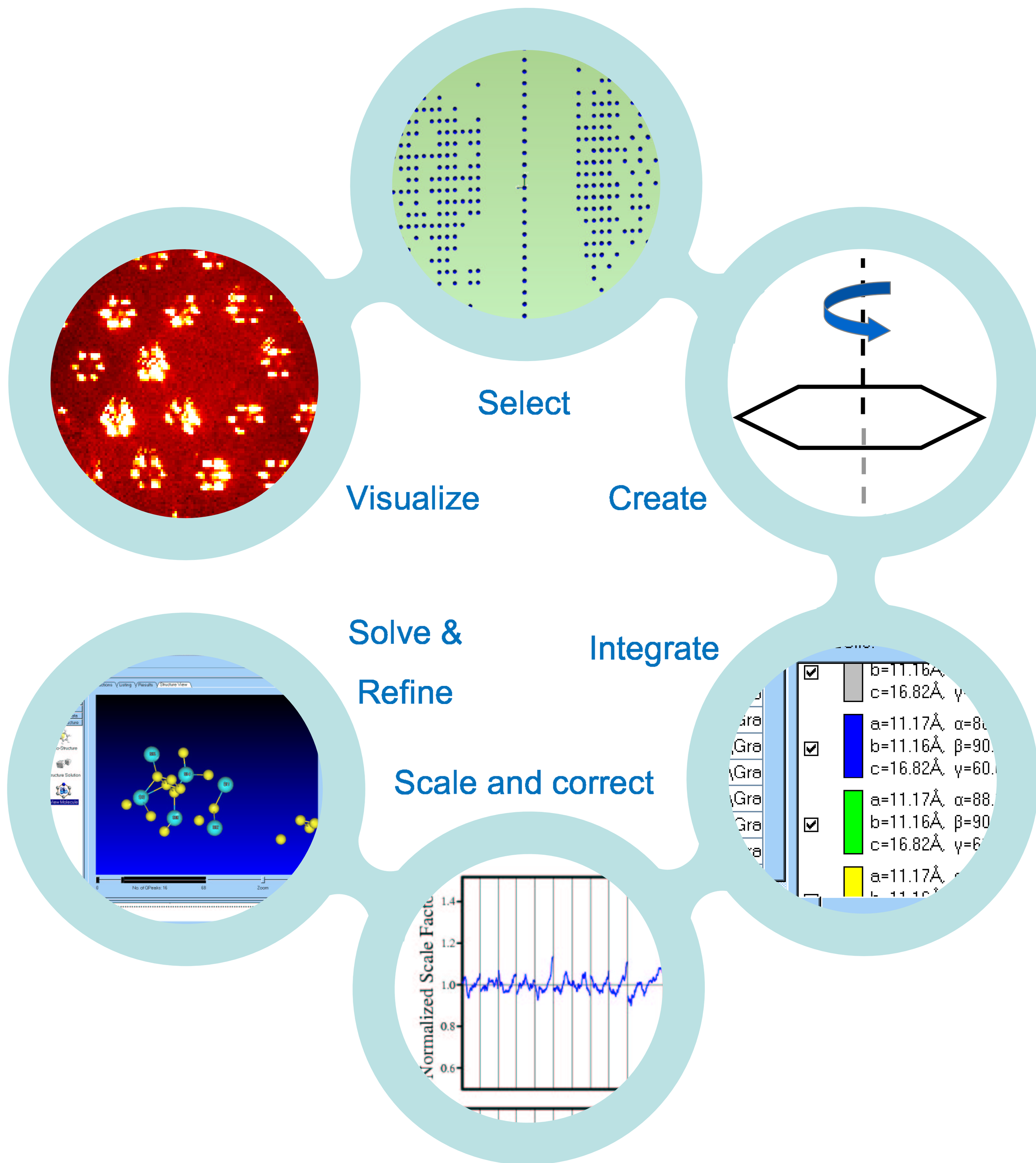

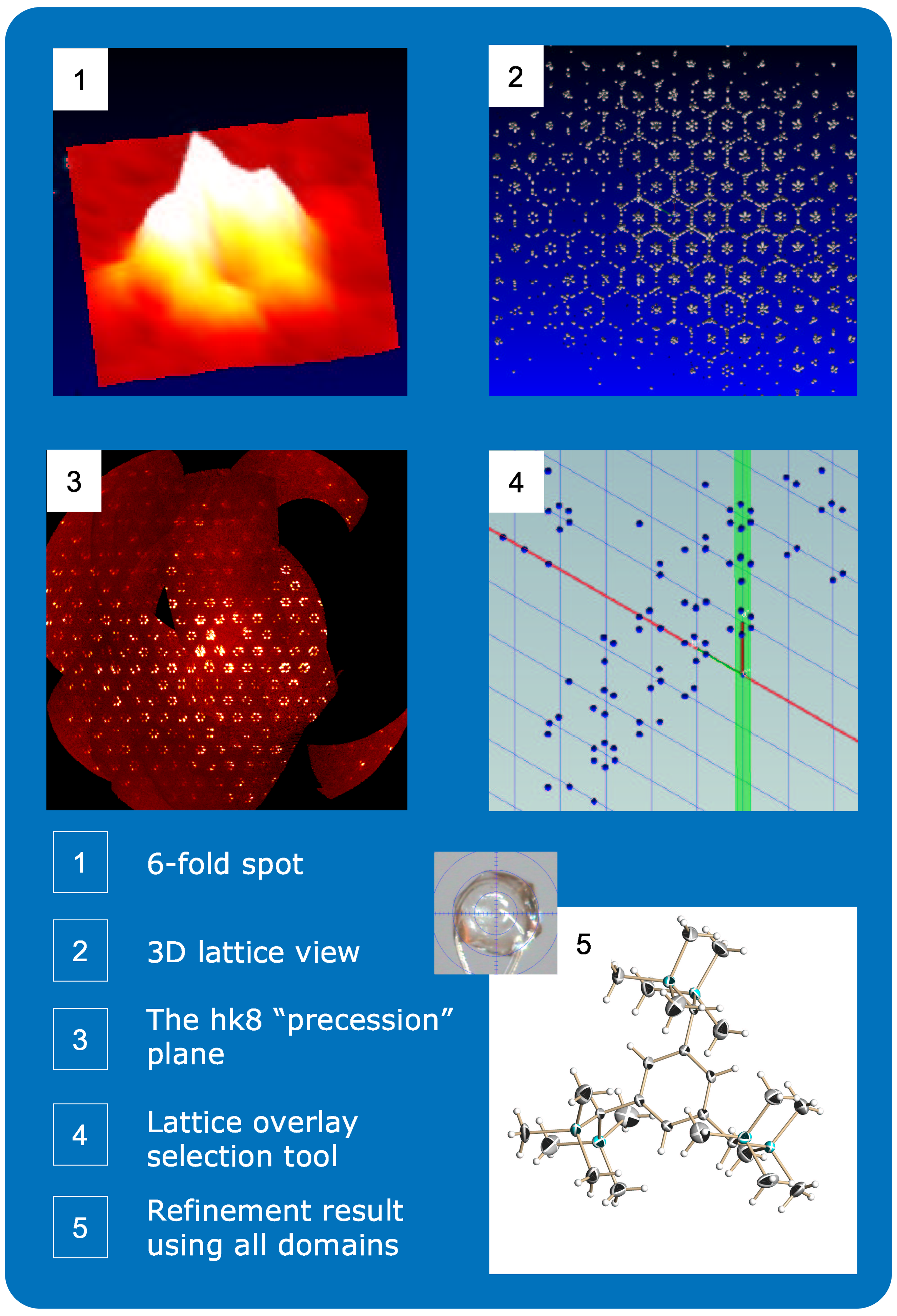

Fig. 1 Visual representation and selection tools

Data Collection

Measurements were done using Cu radiation. Data collection with 0.5 degrees images from 10 to 30 sec. per image. Detector distance 50 mm.

Integration

All 6 domains were integrated concurrently using SAINT [3].
The cells were not refined to avoid refining on peaks affected by overlap.

Results

The structure behaves well in unconstraint refinement using the HKLF5 data. The freely refined batch factors for each domain range from 0.154 to 0.191

Refinement results (shelxl-97 [4]):

R1=0.0673, 24906 Fo>4sig(Fo)
R1=0.0754 for all 29361 data
wR2 = 0.2402,
Restrained GooF= 1.032 all data
Highest peak 1.23
Deepest hole -1.13
<σ(bond)> = 0.0026

Conclusions

- Visual representation and interactive tools to select reliable peaks are essential.
- Absorption correction and scaling can be successfully applied through TWINABS.
- Generating idealized classical twin cells by pseudo symmetry operations, provides an excellent description of the spot positions
- All operations are possible through the Apex2 graphical user interface. (version2.2)

Technology