

## Application Note SCD 375

# Pseudo Merohedral Twinning: How to Treat a Six-fold Twin

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 in which to refine the cell 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 approach described here 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.

### Indexing and Domain Orientation

The initial indexing result is severely 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 identify in which planes spots can be separated.

### 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<sup>1</sup>.
- Solve the structure from the HKLF4 data.
- Refine the structure against the HKLF5 data.

It is also apparent 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<sup>2</sup>.

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 the 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.

## Data Collection

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

## Integration

All six domains were integrated concurrently using SAINT<sup>3</sup>. The cells were not refined to avoid refining on peaks affected by overlap.

## Results

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

### Refinement results (shelxl-97<sup>4</sup>):

R1=0.0673, 24906 Fo>4sig(Fo)

R1=0.0754 for all 29361 data

wR2 = 0.2402,

Restrained GooF= 1.032 all data

< $\sigma$ (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).

## References

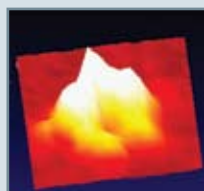
- 1 Sheldrick, G.M. (2002). TWINABS, Bruker AXS, Madison.
- 2 Sheldrick, G.M. (2003). CELL\_NOW, Bruker AXS, Madison.
- 3 Bruker (2001). SAINT, Bruker AXS, Madison.
- 4 Sheldrick, G.M. (1997). SHELXL-97, Uni. Göttingen.

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Figure 1: Visual representation and selection tools



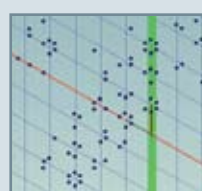
6-fold spot



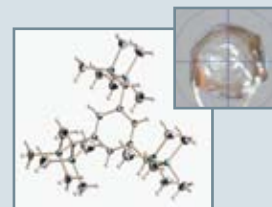
3D lattice view



The hk8  
"precession"  
plane



Lattice overlay  
selection tool



Refinement  
result using all  
domains

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