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Non-merohedral Twinning in Small Molecule and Protein Crystallography

Regine Herbst-Irmer, *Department of Structural Chemistry, University of Göttingen, Germany*. E-mail: rherbst@shelx.uni-ac.gwdg.de

In contrast to twinning by merohedry the reciprocal lattices of the different domains of non-merohedral twins do not overlap exactly. If both twin domains are similar in size, there are often problems with the cell determinations and usual automatic indexing programs fail. But nowadays there are several programs that can deal with such kinds of problems [1], [2], [3].

In principle two different integration procedures are possible. The simpler one would be to integrate with all orientation matrices separately in different runs. This leads to three kinds of reflections: the reflections with no overlap, the reflections with an exact overlap, and the reflection with a partial overlap of a reflection of a second domain. A better way of integration is to use all orientation matrices in one step. Then the whole intensity of every reflection is integrated and we only have non- or exactly overlapped reflections [4], [5].

A special program for scaling and absorption correction is necessary to handle such data sets [6].

Refinements with data sets generated by different integration processes will be compared [7].

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Keywords: twinning, non-merohedral, data processing

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Additional Reflections and Polytypic Sequences in Polygonal Serpentine

Cecilia Viti[a], Mihovil Logar[b], Marcello Mellini[a], Enrico Mugnaioli[a], [a]*Earth Sciences Department, University of Siena, Italy*. [b]*Department for Mineralogy, University of Belgrade, Serbia*. E-mail: vitic@unisi.it

The existence of polygonal serpentine was first noticed trough additional reflections in XRPD patterns. Later, HRTEM revealed micrometric cross-sections, consisting of polygonally arranged lizardite sectors. They occur rotated by 12° or 24°, originating the "magic" numbers of 30 and 15 sectors per fiber (PS-30 and PS-15).

Adjacent sectors are based on different, regularly repeated polytypic stacking sequences. The stacking vector between adjacent layers changes by +/- *b*/3 (equivalent to -/+ *b*/6) from one sector to the next. Moving counterclockwise, couple of layers are stacked by (*o*) orthogonal, (*r*) right or (*l*) left pointing vectors in PS-30, and by (*o*), (*l*) and (*r*) pointing vectors in PS-15. The two sequences arise because adjacent sectors have (001) rotated by 24° and 12° in PS-15 and PS-30, respectively; therefore, the PS-15 *olr* sequence arises eliminating bracketed sectors in the *o(r)l(o)r(l)o(r)l.*. sequence of PS-30. Two-layer and multilayer polygonal polytipic arrangements are common, still matching the same rules as one-layer arrangements.

HRTEM observations indicate continuous 1:1 layer, with no tetrahedral inversion between adjacent sectors. The complex [100] SAED patterns with five-fold symmetry are reproduced by properly overlapped *hk0* reciprocal lattice planes.

The contemporaneous presence of different unit-cells removes degeneracy in d_{hkl} values of adjacent sectors, thus leading to clusters of additional reflections (*e.g.,* 020 and $020_{r,l}$; 021_r, 021_o and 021_l; 022_r, 022_o and 022_l in PS-30) in the XRPD patterns.

Keywords: serpentine, polytypism, microstructure

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Order-disorder, Polytypes and Twinning in the Crystal Structure of Vurroite

Daniela Pinto[a], Elena Bonaccorsi[b], Emil Makovicky[c], Tonci Balić-Žunić[c], [a]*Dipartimento Geomineralogico, Università degli Studi di Bari, Via E. Orabona, 4, I-70125 Bari, Italy.* [b]*Dipartimento di Scienze della Terra, Università di Pisa, Via Santa Maria 53, I-56126 Pisa, Italy.* [c]*Geological Institute, University of Copenhagen, Ostervoldgade 10, DK-1350 Copenhagen K, Denmark.* E-mail: d.pinto@geomin.uniba.it

Vurroite, ideally $Pb_{20}Sn_2(Bi,As)_{22}S_{54}Cl_6$, is a complex mineral type, where the two minor chemical components, Sn and Cl, act as essential constituents together with Pb, Bi, As and S [1, 2]. X-ray single crystal data on vurroite strongly indicate an orthorhombic F-centred symmetry [1, 3]. In this study the crystal structure of vurroite is interpreted as an OD structure belonging to the category III of OD structures composed of equivalent layers [4]. The application of the OD procedures allowed the derivation of the OD-groupoid family (λ and σ operations), as well as the MDO (Maximum Degree of Order) structures. The layer symmetry (λ) is *A(2)mm,* the interlayer symmetry (σ) consists of a glide plane $n_{1/2,1/2}$ and two-fold screw axes parallel to [010] and [001] with the translation components ¼ *b* and ¼ *c*, respectively. For this OD family two MDO polytypes exists. The former has monoclinic symmetry, *C*12/*c*1, whereas the latter is monoclinic, *P*12/*c*1. The OD treatment of the crystal structure of vurroite allowed to prove that the true symmetry of this mineral is monoclinic and that the apparent orthorhombic symmetry observed for the X-ray pattern of the measured crystal is due to a twinning phenomenon.

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Simulation of a Polytypic Family from an Incommensurately Modulated Member

Alla Arakcheeva, Gervais Chapuis, *Laboratoire de Cristallographie, École Polytechnique Fédérale de Lausanne. BSP, CH-1015 Lausanne, Switzerland.* E-mail: Alla.Arakcheeva@epfl.ch

The superspace formalism is applied in order to simulate a polytypic family from the structure of an incommensurately modulated member.

The complete set of structure characteristics (symmetry, unit cell parameters and atomic coordinates) of both periodic and aperiodic members can be generated from the incommensurate structure by changing the value of the modulation vector $\boldsymbol{q}$.

One of the interesting properties of the aperiodic structure is to present the full spectrum of the crystal chemical information (*e.g.* interatomic distances, tilts and displacements of rigid building units and rules defining the sequence of layers) for the most probable members of the polytypic family, respectively for the most probable values of the modulation vector $\boldsymbol{q}$.

These conclusions are illustrated on the basis of the incommensurately modulated phase β-$K_5Yb(MoO_4)_4$. The simulated structures of the highest-temperature phase α ($\boldsymbol{q}$ = 1) and the lowest-temperature phase γ ($\boldsymbol{q}$ = 1/2) show a good agreement with the corresponding experimental data [1].

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Keywords: polytypes, incommensurate modulated structures, crystal structure prediction