

[o.m.4.p.1] Solving the structure of a new Ca-Sc-Sn disilicate twinned by metric merohedry G. Ferraris¹, A. Gula¹, G. Ivaldi¹, M. Nespolo², G. Raade³ - ¹DSMP Univ. Torino, Italy; ²NIRIM Tsukuba, Japan; ³Mineral-Geol. Museum Oslo, Norway; ferraris@dsmp.unito.it
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Structural crystallography is giving a revival to twins¹ and even new aspects have been recently explored^{2,3,4,5}.

Twinning by merohedry is the oriented association of two or more individuals of the same crystalline compound, in which pairs of individuals are related by a *twin operation* belonging to the point group of the lattice but not to the point group of the individual. Compounds with a *Lattice Metric Symmetry* (LMS) higher than that required by their syngony (syngony = crystal system) are well known, at least within the experimental error. In this case a twin operation may or may not belong to the syngony. These two cases correspond to twinning by *syngonic merohedry* (class IIA) and by *metric merohedry* (class IIB) respectively^{5,6} and both can produce a twin in which the twin operation overlaps reciprocal lattice nodes that are not equivalent under the Laue symmetry. The crystal structure cannot be (satisfactorily) solved without identification of the twin law⁶.

A new natural silicate, ideally Ca(Sn,Sc,Fe,Nb)Si₂O₇, from Tørdal (Telemark, Norway) is metrically *C*-centred monoclinic [*a* = 10.028(1), *b* = 8.408(1), *c* = 13.339(2) Å, α = 90.01(1), β = 109.10(1), γ = 90.00(1)°]. The statistical analysis of the X-ray diffraction intensities showed contrasting results; a structure solution obtained in *C2* could not be refined better than R = 0.092 and gave uncertain polarity. Switching to space group *C1* with allowance for {010} twinning¹ lead to a successful refinement of the structure (R = 0.025; almost same volume for the two components of the twin).

Conditions for twinning are, in order of priority, both metric and structural. The higher the degree of pseudosymmetry of the structure, the higher is the probability of getting an "acceptable" solution of the structure. However, (i) ambiguous intensity statistics, (ii) R values higher than those obtained for similar cases, (iii) disorder, (iv) not-positively-defined anisotropic temperature factors, (v) uncertain polarity and (vi) high residues of electron density (particularly on special positions) are some of the signals which can indicate a lower symmetry.

[1] Herbst-Imer R., Sheldrick G. M. "Refinement of twinned structures with *SHELXL97*". *Acta Crystallogr.*, 1998, B54: 443-449.

[2] Nespolo M., Kogure T., Ferraris G. "Allotwinning: oriented crystal association of polytypes – Some warnings on consequences". *Z. Kristallogr.*, 1999, 214: 5-8.

[3] Nespolo M., Ferraris G., Takeda H., Takéuchi Y. "Plesiotwinning: oriented associations based on a large coincidence-site lattice". *Z. Kristallogr.*, 1999, 214: 378-382.

[4] Nespolo M., Ferraris G., Durovic S. "OD character and twinning – Selective merohedry in class II merohedric twins of OD polytypes". *Z. Kristallogr.*, 1999, 214: 776-779.

[5] Nespolo M., Ferraris G. "Twinning by syngonic and metric merohedry. Analysis, classification and effects on the diffraction pattern". *Z. Kristallogr.*, 2000, 215: 77-81.

[6] Catti M., Ferraris G. "Twinning by Merohedry and X-ray Crystal Structure Determination". *Acta Crystallogr.* 1976, A32: 163-165.

[o.m.4.p.2] Opposite Polarity Twin Domains of KLiSO₄. Ch. Scherf¹, D. Rouède², Y. Le Grand², N.R. Ivanov³, Th. Hahn¹, H. Klapper⁴, G. Heger¹. ¹ *Institut für Kristallographie, RWTH Aachen, 52056 Aachen, Germany.* ² *Groupe Matière Condensée et Matériaux, U.M.R. 6626, Univ. Rennes I, 35042 Rennes Cedex, France.* ³ *Institute of Crystallography, Russ. Acad. of Sciences, 117333 Moscow, Russia.* ⁴ *Mineralogisch-Petrologisches Institut, Universität Bonn, 53115 Bonn, Germany*
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The tetrahedral framework structure compound KLiSO₄ is known for its rich polymorphism resulting in a large variety of transformation twinning. In this work we report some new results about the grown-in twin domains of the hexagonal *P6₃* modification at ambient temperature. The investigated large crystals with low dislocation densities were grown from aqueous solutions at 323 K.

Twin structures and twin boundaries related to the three possible merohedral twin laws (*m* || *c*, 2 ⊥ *c* and *m* ⊥ *c*) were observed within a spatial resolution of 30 μm by means of X-ray topography, polarized light and pyroelectric investigations (decoration with charged particles)¹. The two latter methods are able to detect the two twin laws 2 ⊥ *c* and *m* ⊥ *c* reversing the direction of the polar *c*-axis.

Our latest second-harmonic scattering experiments show that some chromium-doped KLiSO₄ crystals with 0.5 at% CrO₄ incorporation on SO₄ sites additionally contain a dense network of regularly shaped small (≈ 1.5 μm) growth twins of opposite polarity. They were invisible by means of optical activity observations with a polarization microscope. Thus, they obviously belong to the twin law 2 ⊥ *c* that does not reverse the sense of the optical activity. After transforming the crystals into the non-polar *Pcmm* high-temperature modification and their re-transformation into the *P6₃* modification no second harmonic scattering could be observed anymore, *i.e.* the small antipolar domains mentioned above vanish after the transformation cycle.

Also surface decoration experiments with nematic liquid crystal mixtures (static nematic liquid crystal method) showed fine stripe contrasts in as grown chromium-doped crystals which are obviously related to the small antipolar domains. As this experimental technique is not exclusively sensitive to twin domains of opposite polarity other experiments will be necessary to prove the twin law of the small domains.

[1] Klapper H., Hahn Th., Chung S.J. "Optical, Pyroelectric and X-ray Topographic Studies of Twin Domains and Twin Boundaries in KLiSO₄". *Acta Cryst.* B43, (1987), 147-159.