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Structural interpretation of a new twin in staurolite from Coray, Brittany, France

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Abstract: A new twin in staurolite has been recently reported from Coray (Brittany, France). From morphological measurements the twin plane has been identified as (202), which corresponds to twinning by reticular pseudomerohedry with index 3 and obliquity 0.97°. On the basis of the high degree of lattice restoration, significantly higher than that in the other well-known twins in staurolite (Greek cross and Saint Andrews cross), one would expect a high probability of occurrence for this twin. Its rarity can be explained on structural bases. The interface corresponding to the twin plane corresponds to a highly disrupted coordination, where only half of the tetrahedra are restored. This makes a contact twin highly unlikely. The reported twin is actually a penetration twin, in which the irregular composition surface may have compensated, to some extent, the unfavourable structural match.

Key-words: staurolite; crystallographic orbits; twinning.

1 Introduction

Staurolite is a well-known index mineral used to estimate the degree of metamorphism of the host regional metamorphic rocks. The idealised formula of staurolite can be written as X_9 ₁₂Y₂Z₄O₂₀(O,H)₄, with ^[6]X = (Al³⁺, Fe²⁺, □), ^[4]Y = (Fe²⁺, Mg²⁺) and ^[4]Z = Si⁴⁺ (□ means vacancy). The structure is based on a slightly distorted *fcc* packing of oxygen atoms, with the cations partly occupying the octahedral (^[6]) and tetrahedral (^[4]) cavities. The distribution of the cations results in a

26 space group of type $C2/m$, with however a lattice that is metrically orthorhombic or very close to
27 orthorhombic. The cell parameters reported by Hawthorne *et al.* (1993), and used hereafter, are $a =$
28 $7.8695(3) \text{ \AA}$, $b = 16.6076(6) \text{ \AA}$, $c = 5.6658(2) \text{ \AA}$, $\beta = 90^\circ$.

29 Smith (1968) called staurolite “an enigma” because of the high frequency of occurrence of its two
30 well-known twins, the 90° twin or Greek cross twin, and the 60° twin or Saint Andrews cross twin¹.
31 Hurst *et al.* (1956) showed that these are rotation twins, and not reflection twins as often reported in
32 the literature. Nespolo & Ferraris (2007), by applying the theory of hybrid twinning (Nespolo &
33 Ferraris, 2006), were able to remove the apparent contradiction related to a higher twin index in the
34 more frequent Saint Andrews cross twin, showing that the two twins actually have the same twin
35 index, once the Saint Andrews cross twin is treated as a hybrid twin. This did not however explain
36 why the two twins have a different frequency of occurrence. The explanation was obtained by
37 Marzouki *et al.* (2014b), by applying the structural analysis based on the study of the
38 eigensymmetry of crystallographic orbits² (Marzouki *et al.*, 2014a). The “staurolite enigma” was
39 therefore considered solved, until Moëlo (2018) reopened it by reporting the first occurrence of a
40 new twin from Coray (Finistère department, Brittany, France), which, as we are going to show, has
41 a lower twin index than the two other well-known twins.

42 The studied sample comes from the well-known staurolite-rich micaschist area close to Coray, a
43 little town in the South of Finistère department, Brittany, France (Chauris, 2014). The twin (Figs. 1
44 and 2) is a penetration twin in which the two individuals make an angle of about 108° between the
45 respective c axes (Moëlo, 2018). The possible twin plane belongs therefore to the form $\{202\}$,
46 which corresponds to an angle of 108.5° between the c axes of the two individuals (computed with

3 1 Dana (1876) reported another twin, with two individuals forming an angle of $70\text{-}71^\circ$ (or the complementary
4 $110\text{-}109^\circ$) between the respective b axes. To the best of our knowledge, this twin has never been found later.

5 2 A crystallographic orbit is the infinite set of atoms obtained from one atom in the asymmetric unit by
6 applying all the operations of the space group.

47 the software Twiny: Nespolo & Iordache, 2013)³. The direction quasi-perpendicular to the plane
48 (202) is [102], resulting in twin index 3 and obliquity 0.97° (computed with the software
49 Geminography: Nespolo & Ferraris, 2006); from the reticular viewpoint, this twin is therefore by
50 reticular pseudomerohedry (Friedel, 1904). For comparison, the Greek cross twin has index 6 and
51 obliquity 1.33°, whereas the Saint Andrews cross twin has effective twin index⁴ 6.0 and obliquities
52 1.01° and 6.12° respectively for the two concurrent sublattices. The rarity of the Coray twin is
53 therefore in striking contrast with the predictions of the reticular theory of twinning, according to
54 which the occurrence frequency should be inversely proportional to the twin index and obliquity.
55 The reticular theory of twinning provides, however, only the necessary conditions: whereas a twin
56 with very high twin index should not occur (when it does, it normally is a hybrid twin), a twin with
57 low index does not necessarily occur. This is precisely the situation in staurolite. To explain the
58 rarity of the Coray twin we need to analyse the structure at the interface.

59 **2 Structural analysis of the Coray twin in staurolite**

60 Twinning can occur as the result of a phase transition (transformation twins), of a physical action
61 (mechanical twins) or as a perturbation of the process of nucleation and crystal growth (growth
62 twins). Transformation twins typically result in domain structures, in which several physically
63 distinct domains are distributed over a few (often two) orientations (domain states) (Nespolo,
64 2015b). In mechanical twins, the interface between individuals are characteristically plane (Buerger,
65 1945), so that this type of twinning should result in contact twins. The Coray twin in staurolite
66 being a penetration twin with two macroscopic individuals clearly visible, it must be a growth twin.
67 A growth twin is the result of a change in orientation during crystal growth, or of the oriented

9 3 The (101) indexing in Moëlo (2018) has to be corrected to (202) because the conventional unit cell of
10 staurolite is *C*-centred (Nespolo, 2015a).

11 4 The effective twin index is the generalization of the twin index to hybrid twins and is defined as the inverse
12 of the fraction of the number of lattice nodes belonging to any of the concurrent sublattices based on the twin
13 plane (or axis, for rotation twins) (Nespolo and Ferraris, 2005).

68 attachment of crystals (Nespolo and Ferraris, 2004). Both origins can be seen as the result of a
69 perturbation with respect to normal crystal growth process, which results in a heterogeneous
70 crystalline edifice that should be less favourable, from a thermodynamic viewpoint (especially in its
71 enthalpic term), with respect to an untwinned crystal. When a twin occurs with a certain frequency,
72 there must be structural reasons that make the change in orientation a tolerable situation; otherwise,
73 the twin either does not form or occurs very occasionally. The two well-known cross twins of
74 staurolite correspond to the first situation, while the rare (so far unique) Coray twin must instead
75 belong to the second category. To ascertain whether this is really the case, we need to analyse the
76 pseudo-eigensymmetry of the crystallographic orbits to find whether it contains an operation whose
77 linear part coincides with the twin operation. The approach has been described in details in our
78 previous articles; in particular, we have shown that the cross twins of staurolite indeed present a
79 good structural match across the interface, which justifies their occurrences (Marzouki *et al.*
80 2014b). The same approach allowed us to explain, from on a structural viewpoint, twinning in
81 melilite (Marzouki *et al.* 2014a), aragonite (Marzouki *et al.*, 2015), marcasite (Nespolo and
82 Souvignier, 2015a), cassiterite/rutile (Nespolo & Souvignier, 2015b), forsterite (Azevedo &
83 Nespolo, 2017), chalcostibite (Nespolo, 2018) as well as the Carlsbad twin in orthoclase (Nespolo
84 & Souvignier, 2017). As we are going to show, the same approach explains the rarity of the Coray
85 twin.

86 The unit cell of the twin lattice of both the Greek cross twin and the Saint Andrews cross twin is
87 based on the so-called Mallard's pseudo-cube⁵, *i.e.* a metrically cubic supercell whose basis vectors
88 are obtained from those of staurolite by the transformation $\mathbf{b}+3\mathbf{c}$, $-\mathbf{b}+3\mathbf{c}$, $3\mathbf{a}$. The structure of
89 staurolite contains seven oxygen orbits; the union of all these orbits has pseudo-cubic (*fcc*)
90 symmetry and is therefore approximately restored by the twin operations. The individuals in both

16 ⁵ This supercell is called “*pseudo-cube*” because the metric of the lattice based on the unit cell is cubic but the
17 unit cell is *C*-centred and therefore not compatible with the cubic symmetry (Friedel, 1904).

91 twins have therefore a substructure based on the oxygen atoms that is approximately common. The
 92 higher occurrence frequency of the Saint Andrews twin was explained on the basis of a higher
 93 degree of restoration for the cations (Marzouki *et al.*, 2014b).

94 The unit cell of the twin lattice of the Coray twin is based on the [102]/(202) pair of lattice
 95 elements. In the cubic basis of the Mallard's pseudo-cube these become [111]/(111). The [111]
 96 direction (*i.e.* [102] in the setting of staurolite) corresponding to a threefold axis in a cubic
 97 (sub)lattice: indeed, this direction acts as a three-fold rotation axis in the Greek cross twin. A
 98 twofold rotation about the same axis is not a symmetry operation for a cubic (sub)lattice; neither is
 99 a reflection through the (111) cubic plane. Therefore, the union of the oxygen orbits is no longer
 100 restored in the Coray twin, as is instead the case in the cross twins. We have therefore to explicitly
 101 obtain the degree of restoration for both the anions and the cations in this new twin.

102 The basis vectors of the individual ("I") and of the twin ("T") are related by the following
 103 transformation:

$$(\mathbf{a} \ \mathbf{b} \ \mathbf{c})_I \begin{pmatrix} 1 & 0 & \bar{1} \\ 0 & 1 & 0 \\ 2 & 0 & 1 \end{pmatrix} = (\mathbf{a} \ \mathbf{b} \ \mathbf{c})_T$$

104 where [010] and $[\bar{1}01]$ are the two shortest direction in the (202) plane at right angles from each
 105 other. The cell parameters of the twin lattice are $a = 13.796 \text{ \AA}$, $b = 16.608 \text{ \AA}$, $c = 9.697 \text{ \AA}$, $\beta =$
 106 89.03° and the unit cell is *C*-centred (Fig. 3). The highest subgroup of *C2/m* compatible with this
 107 lattice is obtained as $\mathcal{H} = \mathcal{G}_1 \cap \mathcal{G}_2$ where \mathcal{G}_1 and \mathcal{G}_2 are the space groups of the two individuals, both
 108 of type *C2/m*, in the respective orientations; in particular \mathcal{G}_2 is conjugate of \mathcal{G}_1 with respect to the
 109 twin operation. In our case, $\mathcal{G}_2 = m_{(202)}\mathcal{G}_1m_{(202)}$ and $\mathcal{H} = C2/m$, *i.e.* an isomorphic subgroup of the
 110 space group of the individual: same type of space group but whose translation subgroup is a
 111 subgroup of the translation subgroup of the space group of the individual. In symbols, $\mathcal{T}(\mathcal{H}) \subset \mathcal{T}(\mathcal{G})$.

112 The Miller indices of the twin plane in the axial setting of the twin are (200).
113 In order for a substructure of staurolite to cross the twin plane more or less unperturbed, the
114 crystallographic orbits building this substructure must contain, in their eigensymmetry, an
115 operation, known as the *restoration operation*, whose linear part coincides with the twin operation.
116 In the case of the Coray twin in staurolite, the twin operation is a mirror reflection through the (200)
117 plane (in the twin setting); the restoration operation can therefore be a mirror or glide reflection
118 through a plane parallel to (200). Fig. 4 shows a projection along the *b* axis of the structure of
119 staurolite in the axial setting of the Coray twin, with planes of the (200) family on or close to
120 oxygen atoms - black planes at $(2n+1)a/12$, *n* integer - on or close to the X atoms – grey planes at
121 $na/6$, *n* integer. The two nearest planes of either set are $a/6$ apart, while two nearest planes of
122 different sets are $a/12$ apart. Twinning would be explained on a structural basis if any of these
123 planes is a pseudo mirror or glide plane for a significant subset of atoms close to that plane, because
124 in this case a significant substructure would cross the plane without a large deformation. Because of
125 the $C2/m$ symmetry, only two positions for the each of the two sets have to be considered, the others
126 being related by symmetry operations of the space group; in the following we analyse positions at x
127 $= 1/4$ and $x = 5/12$ for the black planes, and positions $x = 1/3$ and $x = 1/2$ for the grey planes.
128 A slab of the structure corresponding to the coordination polyhedra of staurolite possesses only the
129 translational symmetry of staurolite; it has therefore to be described in the *translationengleiche* $P1$
130 subgroup of $C2/m$ (basis vectors of the twin lattice), in which every atom in the unit cell is
131 symmetrically independent and defines a crystallographic orbit. Restoration, if it occurs, concerns
132 atoms with the same structural role: we have therefore to consider the pseudo-eigensymmetry of
133 oxygen atoms, octahedral (X) cations, and tetrahedral (Y and Z) cations separately (we have
134 omitted the Fe1 and Fe2 sites because their very low occupancy makes their structural role
135 negligible). These are (quasi)-restored if the union of the corresponding crystallographic orbits

136 contains a mirror or glide reflection about the (200) plane; in other words, if the pseudo-
137 eigensymmetry of the union of these orbits is of type $Pm11$, $Pb11$, $Pc11$ or $Pn11$. The analysis is
138 presented in Tables S1-S48 (deposited material). Each table presents, for each of the three union of
139 orbits (oxygen atoms, X cations, Y&Z cations) and for each position of the plane:

- 140 • the fractional coordinates of the atoms on the two sides of the plane nearest to that plane:
- 141 • the fractional coordinates of the same atoms on which the plane has acted as a mirror plane,
142 b -glide plane, c -glide plane or n -glide plane, ordered in the sequence that minimizes the
143 distance between corresponding atoms;
- 144 • the difference of fractional coordinates;
- 145 • the distance in Å between corresponding atoms:
- 146 • when the position of an atom obtained by mirror or glide-reflection is relatively close to
147 more than one atom in their original position, the data of the former are shown in red: this
148 means that the 1-1 correspondence is broken and the atomic position obtained by mirror or
149 glide reflection is midway between the atomic positions of two atoms in the slab of
150 staurolite; this also implies that some of the atoms obtained by mirror or glide reflection do
151 not have any of the atoms in the slab of staurolite as first neighbours; in this case, the atomic
152 coordination on the two sides of the plane is severely affected by the mirror or glide
153 reflection.

154 The results, summarized Table 1, show that:

- 155 • the oxygen orbits have a very high degree of pseudo-symmetry (very low distance d) for all
156 the four cases $Pm11$, $Pb11$, $Pc11$ or $Pn11$ for black planes (which is not surprising because
157 the corresponding atoms are almost on those planes); this is instead not the case for the grey
158 planes;
- 159 • the X orbits have a very high degree of $Pm11$ pseudo-symmetry for the grey planes, but not

160 for the black planes; this is also true for a subset of the orbits for $Pn11$ (75%) and for an
161 even smaller subset for $Pc11$ (33%), whereas no $Pb11$ pseudo-eigensymmetry exists for the
162 black planes;

163 • a high degree of $Pb11$ pseudo-eigensymmetry exists for half of the Y and Z cations with
164 respect to the black planes; no other pseudo-eigensymmetry for any of the other cases.

165 Restoration is considered to be achieved if the distance between corresponding atoms is shorter than
166 a certain threshold, which depends on the atomic size. In our case, a clear gap is evident, between
167 atomic positions very close to each other (below 0.5Å) and the other positions (above 0.9Å). The
168 best atomic restoration obtained in the Coray twin corresponds to the pseudo-eigensymmetry $Pb11$
169 on the black planes, *i.e.* planes at $(2n+1)a/12$, for which the whole set of oxygen atoms and half of
170 the tetrahedral cations (of these, $\frac{1}{3}$ of Y and $\frac{2}{3}$ of Z) are almost perfectly restored. This is however
171 unlikely to be sufficient to **give** the twin a reasonable mechanical stability, considering that 1) in a
172 silicate, the bulk of the structure is represented by the tetrahedra centred on silicon, whereas in this
173 twin only half of the tetrahedra are restored across the twin plane (Fig. 5); 2) none of the octahedral
174 cations are restored. For the other eigensymmetries the situation is even worse, with either the
175 cations or the anions, but not both, restored. The structural continuity is therefore severely affected
176 and the coordination disrupted.

177 **3 Discussion**

178 The newly reported (200) twin in staurolite from Coray represents an exception with respect to the
179 inverse trend usually observed between occurrence frequency on one hand and the degree of lattice
180 restoration (as measured by the twin index and obliquity) on the other hand. Despite a degree of
181 lattice restoration much higher (double) than that in the cross twins (Greek and Saint Andrews), it
182 was never reported before.

183 The analysis of the structure at the twin plane, in terms of pseudo-eigensymmetry of the

184 crystallographic orbits close to it, shows significant disruption of the coordination, which is instead
185 largely preserved in the cross twins. This result naturally prompts the question as to the possible
186 origin of this newly reported twin. It is well known that in a contact twin the composition surface is
187 planar and, if twinning is by reflection, it is parallel to the twin plane (Sunagawa, 2005). On the
188 contrary, in penetration twins the composition surface is irregular and the original composition
189 plane, *i.e.* the one on which the change of orientation has originated (by the occurrence of a stacking
190 fault, the deposition of an impurity or other perturbations) does not coincide with the twin plane; it
191 plays an ephemeral role and disappears during the crystal growth (Kern, 1961). The Coray twin in
192 staurolite is a penetration twin, which means that the composition surface most likely deviates from
193 a plane; this may, to a certain degree, reduce the coordination disruption at the interface that would
194 otherwise most likely prevent the occurrence of the twin. The (200) twin was never reported before.
195 This may be an oversight, especially considering that morphological studies are less frequently
196 performed today and morphological information is often unfortunately missing from structure
197 refinements of twinned crystals. Otherwise, the extreme rarity of this twin, if confirmed, may
198 suggest that the supposed compensatory effect of the irregular composition surface on the
199 coordination disruption is probably limited.

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252 **FIGURE CAPTIONS**

253 **Figure 1.** (a) Photo of the Coray twin sub-parallel to b (reproduced from L.-D. Bayle, with his
 254 permission). (b) Schematic drawing, with twin angle between A and B crystals. Arrow: view
 255 direction of Fig. 2.

256 **Figure 2.** (a) Photo of the Coray twin sub-parallel to a (reproduced from L.-D. Bayle, with his
 257 permission). (b) Right. Schematic drawing.

258 **Figure 3.** (010) projection of the lattice of staurolite (black and yellow circles represent lattice
 259 nodes with integer and semi-integer values of the y coordinate). In black the unit cell of staurolite.
 260 In red, the unit cell of the twin lattice. The indices “I” and “T” stand for *individual* and *twin*
 261 respectively.

262 **Figure 4.** (010) projection of the structure of staurolite in the axial setting of the Coray twin, with
 263 planes of the family (200) passing on or close to the oxygen atoms (black) or X cations (grey). This
 264 and the following figure drawn with VESTA (Momma and Izumi, 2011).

265 **Figure 5.** Overlap of the structure of staurolite about the (200) plane at $y = 5/12$ (white atoms) and
 266 its mirror image obtained by b -glide reflection through this plane (black atoms). Only the quasi-
 267 restored atoms are shown in the second case, *i.e.* oxygen atoms and half of the Y and Z sites.

268 **Table 1.** Percentage of restoration of crystallographic orbits around the (200) plane (setting of the
 269 twin) of Coray twin in staurolite for the four possible eigensymmetries. Details of the calculation
 270 are shown in Tables S1-S48 (deposited material). An atom is considered restored if the distance
 271 with respect to the corresponding position obtained by acting on it with the mirror or glide plane is
 272 lower than 0.5 Å, as explained in the text.

	Planes at $(2n+1)a/12$ (black planes in Fig. 4)				Planes at $na/6$ (grey planes in Fig. 4)			
	$Pm11$	$Pb11$	$Pc11$	$Pn11$	$Pm11$	$Pb11$	$Pc11$	$Pn11$
O	100	100	100	100	0	0	0	0
X	0	0	0	0	100	0	33	75
Y&Z	0	50	0	0	0	0	0	0













