International Tables for Crystallography (2006). Vol. D, Section 3.3.6.9, pp. 408–409.

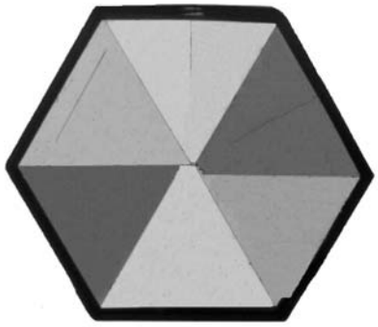

Fig. 3.3.6.7. Pseudo-hexagonal growth twin of K_2SO_4 showing six sector domains in three orientation states. (001) plate, about 1 mm thick and 5 mm in diameter, between polarizers deviating by 45° from crossed position for optimal contrast of all domains. The crystal was precipitated from aqueous K_2SO_4 solution containing 5% S_2O_3 ions. Courtesy of M. Moret, Milano.

(4) Twin law representative: 'reflection across $(11\bar{2})$';
Alternative twin operations: line (3) of case (*a*) above;
Reduced composite symmetry: $\mathcal{K}^* = 31m'$ (order 6).

The restriction to only one of the four spinel twin axes $\langle 111 \rangle$ combined with the application of the coset expansion to the *reduced eigensymmetry* $\mathcal{H}^*$ always leads to a crystallographic composite symmetry $\mathcal{K}^*$. The supergroup generated from the *full eigensymmetry*, however, would automatically include the other three spinel twin axes and thus would lead to the infinite sphere group $m\overline{\infty}$, *i.e.* would imply infinitely many cosets and (equivalent) twin laws. Higher-order spinel twins are discussed in Section 3.3.8.3.

3.3.6.7. *Growth and transformation twins of K_2SO_4*

K_2SO_4 has an orthorhombic pseudo-hexagonal room-temperature phase with point group $\mathcal{H} = mmm$ and axial ratio $b/a = \tan 60.18°$, and a hexagonal high-temperature phase (> 853 K) with supergroup $\mathcal{G} = 6/m\,2/m\,2/m$. It develops pseudo-hexagonal growth-sector twins with equivalent twin reflection planes (110) and $(1\bar{1}0)$ which are also composition planes, as shown in Fig. 3.3.6.7. As discussed in Sections 3.3.2.3.2 and 3.3.4.4 under (iii), this corresponds to a pseudo-threefold twin axis which, in combination with the twofold *eigensymmetry* axis, is also a pseudo-hexagonal twin axis. The extended composite symmetry is

$$\mathcal{K}(6) = \mathcal{K}(3) = 6(2)/m\,2/m\,2/m.$$

Upon heating above 853 K, the growth-sector twinning disappears. On cooling back into the low-temperature phase, transformation twinning ('domain structure') with three systems of lamellar domains appears. The three orientation states are identical for growth and transformation twins, but the morphology of the twins is not: sectors *versus* lamellae. The composite symmetry $\mathcal{K}$ of the twins at room temperature is the true structural symmetry $\mathcal{G}$ of the 'parent' phase at high temperatures.

3.3.6.8. *Pentagonal–decagonal twins*

As was pointed out in Note (8) of Section 3.3.2.4 and in part (iii) of Section 3.3.4.4, there exist twin axes with noncrystallographic multiplicities $n = 5, 7, 8$ *etc.* Twins with five- or tenfold rotations are frequent in intermetallic compounds. As an example, $FeAl_4$ is treated here (Ellner & Burkhardt, 1993; Ellner, 1995). This compound is orthorhombic, $2/m\,2/m\,2/m$, with an axial ratio close to $c/a = \tan 72°$, corresponding to a pseudo-fivefold axis along [010] and equivalent twin mirror planes (101) and $(\bar{1}01)$, which are about 36° apart. In an ideal intergrowth, this leads to a cyclic pseudo-pentagonal or pseudo-decagonal sector twin (Fig. 3.3.6.8). All features of this twinning are analogous to those of pseudo-hexagonal aragonite, treated in Section 3.3.2.3.2, and of K_2SO_4, described above as Example 3.3.6.7.

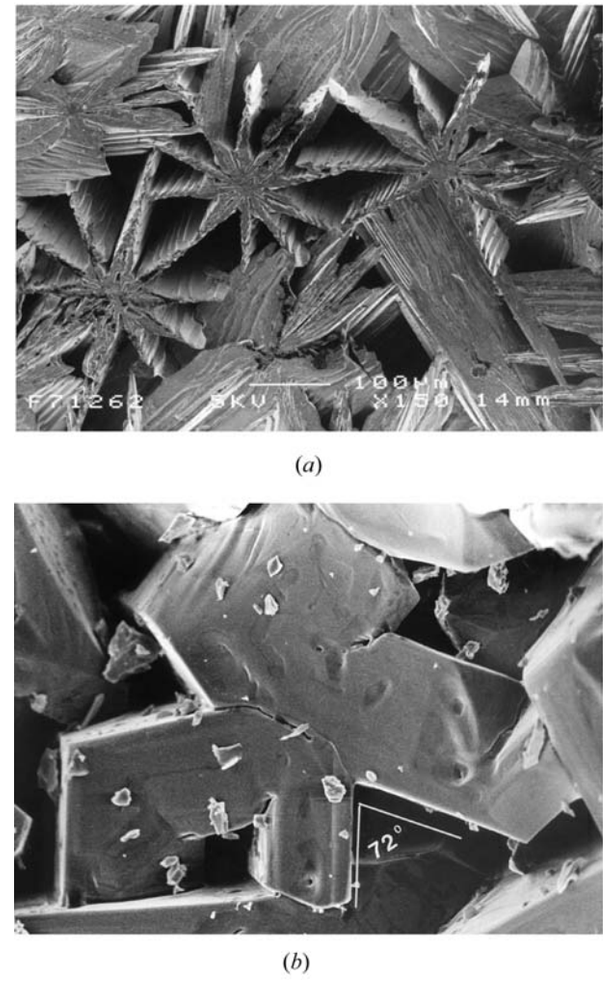

Fig. 3.3.6.8. Pentagonal–decagonal twins. (*a*) Decagonal twins in the shape of tenfold stars on the surface of a bulk alloy, formed during the solidification of a melt of composition $Ru_8Ni_{15}Al_{77}$. Scanning electron microscopy picture. Typical diameter of stars *ca.* 200 µm. The arms of the stars show parallel intergrowth. (*b*) Pentagonal twin aggregate of Fe_4Al_{13} with morphology as grown in the orthorhombic high-temperature phase, showing several typical 72° angles between neighbouring twin partners (diameter of aggregate *ca.* 200 µm). Orthorhombic lattice parameters $a = 7.7510$, $b = 4.0336$, $c = 23.771$ Å, space group *Bmmm*. The parameters c and a approximate the relation $c/a = \tan 72°$; the pseudo-pentagonal twin axis is [010]. On cooling, the monoclinic low-temperature phase is obtained. The twin reflection planes in the orthorhombic unit cell are (101) and $(10\bar{1})$, in the monoclinic unit cell (100) and $(\bar{2}01)$; *cf.* Ellner & Burkhardt (1993, Fig. 10), Ellner (1995). Both parts courtesy of M. Ellner, Stuttgart.

The intersection symmetry of all twin partners is $\mathcal{H}^* = 12/m1$; the reduced composite symmetry $\mathcal{K}^*$ of a domain pair in contact is $2'/m\,2/m\,2'/m$. The extended composite symmetry of the ideal pentagonal sector twin is $\mathcal{K}(10) = \mathcal{K}(5) = 10(2)/m\,2/m\,2/m$.

3.3.6.9. *Multiple twins of rutile*

Rutile with *eigensymmetry* $4/m\,2/m\,2/m$ develops growth twins with coinciding twin reflection and composition plane {011}. Owing to its axial ratio $a/c = \tan 57.2°$, the tetragonal c axes of the two twin partners form an angle of 114.4°. The intersection symmetry of the two domains is $\mathcal{H}^* = 2/m$ along the common direction [100]. The reduced composite symmetry of the domain pair is $\mathcal{K}^* = 2/m\,2'/m'\,2'/m'$, with the primed twin elements parallel and normal to the plane (011). A twin of this type, consisting of two domains, is called an 'elbow twin' or a 'knee twin', and is shown in Fig. 3.3.6.9(*a*).

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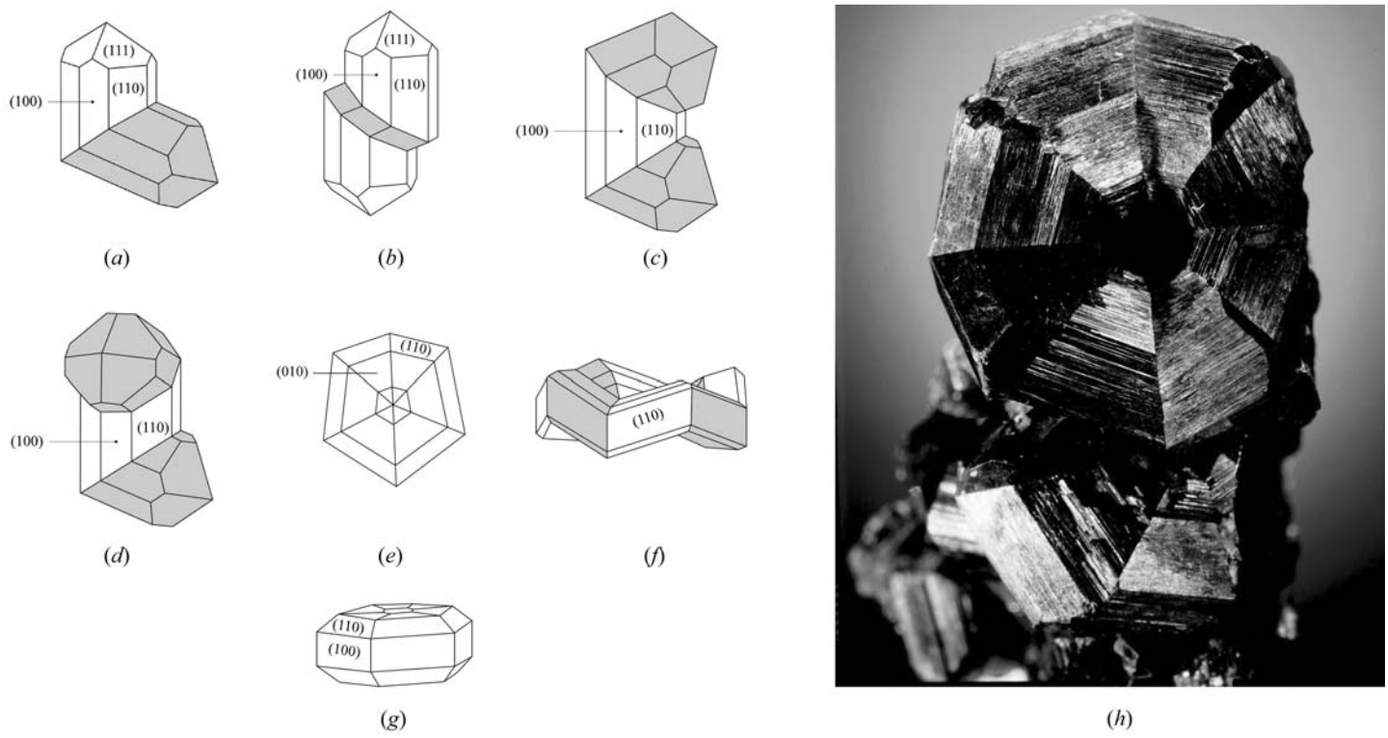

Fig. 3.3.6.9. Various forms of rutile (TiO_2) twins, with one or several equivalent twin reflection planes {011}. (*a*) Elbow twin (two orientation states). (*b*) Twin with two orientation states. One component has the form of an inserted lamella. (*c*) Triple twin (three orientation states) with twin reflection planes (011) and $(0\bar{1}1)$. (*d*) Triple twin with twin reflection planes (011) and (101). (*e*) Cyclic sixfold twin with six orientation states. Two sectors appear strongly distorted due to the large angular excess of 35.6°. (*f*) Cyclic eightfold twin with eight orientation states. (*g*) Perspective view of the cyclic twin of (*e*). (*h*) Photograph of a rutile eightling (*ca.* 15 mm diameter) from Magnet Cove, Arkansas (Geologisk Museum, Kopenhagen). Parts (*a*) to (*e*) courtesy of H. Strunz, Unterwössen, *cf.* Ramdohr & Strunz, 1967, p. 512. Photograph (*h*) courtesy of M. Medenbach, Bochum.

In point group $4/m\,2/m\,2/m$, there exist four equivalent twin reflection planes {011} (four different twin laws) with angles of 65.6° between (001) and $(0\bar{1}1)$ and 45° between (011) and (101), leading to a variety of multiple twins. They may be linear polysynthetic or multiple elbow twins, or any combination thereof (Fig. 3.3.6.9). Very rare are complete cyclic sixfold twins with a large angular excess of $6 \times 5.6° = 33.6°$ (corresponding formally to a '5.5-fold' twin axis) and extended composite pseudosymmetry $\mathcal{K}(6) = 6(2)/m\,2/m\,2/m$, or cyclic eightfold twins with a nearly exact fit of the sectors and a morphological pseudo-$\bar{8}$ twin axis. In the 'sixling', the tetragonal axes of the twin components are coplanar, whereas in the 'eightling' they alternate 'up and down', exhibiting in ideal development the morphological symmetry $\bar{8}2m$ of the twin aggregate. The extended composite symmetry is $\mathcal{K}(8) = 8(1)/m\,2/m\,2/m$ with eight twin components, each of different orientation state. These cyclic twins are depicted in Figs. 3.3.6.9(*e*), (*f*), (*g*) and (*h*).

The sketch of the 'eightling' in Fig. 3.3.6.9(*f*) suggests a hole in the centre of the ring, a fact which would pose great problems for the interpretation of the origin of the twin: how do the members of the ring 'know' when to turn and close the ring without an offset? Fig. 3.3.6.9(*h*) suggests that the ring is covered at the back, *i.e.* originates from a common point (nucleus). This was confirmed by a special investigation of another 'eightling' from Magnet Cove (Arkansas) by Lieber (2002): the 'eightling' started to grow from the nucleus and developed into the shape of a funnel with an opening of increasing diameter in the centre. This proves the nucleation growth of the ring (*cf.* Section 3.3.7.1.1).

3.3.6.10. *Variety of twinning in gibbsite, $Al(OH)_3$*

Gibbsite (older name: hydrargillite) forms a pronounced layer structure with a perfect cleavage plane (001). It is monoclinic with *eigensymmetry* $\mathcal{H} = 12/m1$, but strongly pseudo-hexagonal with an axial ratio $b/a = \tan 30.4°$. In contrast to most other pseudo-hexagonal crystals, the twofold *eigensymmetry* axis b is not parallel but normal to the pseudo-hexagonal c axis. The normal to the cleavage plane (001) is inclined by $\beta - 90° = 4.5°$ against [001]. Owing to the pseudo-hexagonal metrics of the plane (001), the lattice planes (110) and $(\bar{1}10)$, equivalent with respect to the *eigensymmetry* $\mathcal{H} = 2/m$, form an angle of 60.8°.

The following four significant twin laws have been observed by Brögger (1890):

(i) *(001) reflection twin*: the cleavage plane (001) acts both as twin mirror and composition plane. The pseudo-hexagonal axes [001] of both partners are inclined to each other by 9.0°. This twin law is quite common in natural and synthetic gibbsite.

(ii) *(100) reflection twin*: the twin mirror plane (100) is also the composition plane. The angle between the (001) planes of both partners is 9.0°, as in (i); the pseudo-hexagonal axes [001] of both partners are parallel. This twin law is not common.

(iii) *(110) reflection twin*: again, twin mirror plane and composition plane coincide. The two (001) planes span an angle of 4.6°. This twin law is very rare in nature, but is often observed in synthetic materials. A sixfold sector twin of synthetic gibbsite, formed by cyclic repetition of {110} twin reflection planes 60.8° apart, is shown in Fig. 3.3.6.10. The pseudo-hexagonal axis [001] is common to all domains. Since the (001) plane is inclined towards this axis at 94.5°, the six (001) facets of the twinned crystal form a kind of 'umbrella' with [001] as umbrella axis (Fig. 3.3.6.10*a*). This (001) umbrella faceting was recently observed in twinned synthetic gibbsite crystals by Sweegers *et al.* (1999).

In contrast to orthorhombic aragonite with only three pseudo-hexagonal orientation states, these gibbsite twins exhibit *six* different orientation states. This is due to the absence of any *eigensymmetry* element along the pseudo-hexagonal axis [001]. The intersection symmetry of all orientation states is $\bar{1}$. The reduced composite symmetry of a domain pair is $\mathcal{K}^* = 12'/m'1$, with m' the twin mirror plane (110).

references