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This chapter presents a review of the typical growth defects of crystals fully grown on (planar) habit faces, i.e. of crystals grown in all kinds of solutions, in supercooled melt (mainly low-melting organics) and in the vapour phase. To a smaller extent also growth on rounded faces from the melt is considered when it seemed to be adequate to bring out analogies or discuss results in a more general context. The origins and the typical configurations of defects developing during growth and after growth are illustrated by a series of selected x-ray diffraction topographs (Lang technique) and, in a few cases, by optical photographs.

After the introduction (Sect. 4.1) the review starts with the formation of inclusions (Sect. 4.2) which are the main origin of other growth defects such as dislocations and twins. Three kinds of inclusions are treated: foreign particles, liquid inclusions (of nutrient solution) and solute precipitates. Particular attention is directed to the regeneration of seed crystals into a fully facetted shape (capping), and inclusion formation due to improper hydrodynamics in the solution (especially for KDP).

Section 4.3 deals shortly with striations (treated in more detail in another chapter) and more comprehensive with the different kinds of crystal regions grown on different growth faces: growth sectors, vicinal sectors, facet sectors. These regions are usually differently perfect and possess more or less different physical properties, and the boundaries between them frequently are faulted internal surfaces of the crystal. Two subsections treat the optical anomalies of growth and vicinal sectors and the determination of the relative growth rates of neighboured growth faces from the orientation of their common sector boundary.

In Sect. 4.4 distinction is made between dislocations connected to and propagating with the growth interface (growth dislocations), and dislocations generated behind the growth front by plastic glide due to stress relaxation. The main sources of both types of dislocations are inclusions. In crystals grown on planar faces, growth dislocations are usually straight-lined and follow (frequently non-crystallographic) preferred directions depending on the Burgers vector, on the growth direction and the elastic constants of the crystal. These directions are explained by a minimum of the dislocation line energy per growth length or, equivalently, by zero force exerted by the growth surface on the dislocation. Calculations based on anisotropic linear elasticity of a continuum confirm this approach. The influence of the discrete lattice structure and core energy on dislocation directions is discussed. Further subsections deal with the Burgers vector determination by preferred directions, with the post-growth movement of grown-in dislocations, with the generation of post-growth dislocations, and with the growth-promoting effect of edge dislocations.

In Sect. 4.5, Twinning, the main characteristics of twins and their boundaries, their generation by nucleation and by inclusions, their propagation with the growth front and their growth promoting effect are presented. The post-growth formation of twins by phase transitions and ferroelastic (mechanical) switching is shortly outlined. The last Sect. 4.6 compares the perfection of crystals (KDP and ADP) slowly and rapidly grown from solutions, that the optical and structural quality of rapidly grown crystals is not inferior to that of slowly grown crystals, if particular precautions and growth conditions are met.

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4.1 Overview
The present chapter mainly deals with growth defects in crystals fully grown on planar habit faces. To a smaller extent also crystals grown on rounded faces from the melt are considered when it seemed to be adequate to bring out analogies or discuss results in a more general context. Crystals grow on habit faces in solutions, in supercooled melts and in the vapour. A special feature of this growth method is that there is practically no temperature gradient inside the crystal, provided that facet growth occurs freely on the whole surface of the crystal (without contact to a container wall). This is also the case for growth in the supercooled melt: the crystallization heat released at the growing habit faces keeps these on crystallization temperature – or at least close to it [4.1]. The absence of a temperature gradient and, thus, of thermal stress inside the crystal allows the development of defect structures according to first thermodynamical principles and to preserve them in their as-grown geometries, unless thermal gradients are introduced by improper cooling to room temperature after growth. This particularly concerns dislocations in crystals growing in their plastic state. Dislocations are the essential elements of stress relaxation by plastic glide: they are generated, moved and multiplied by stress. Thus – in the presence of thermal stress – it makes an essential difference whether crystals are grown in their brittle or their plastic state. From solution, crystals grow in the brittle or in the plastic state (dependent on the specific mechanical properties at growth temperature); from the melt, however, crystals always grow in the plastic state, because each material has a more or less extended plastic zone below its melting point. It will be shown that growth dislocations develop the same geometrical features in crystals grown on habit faces from solution in the brittle state and from supercooled melts in the plastic state, provided thermal gradients are absent.

In this review the generation of defects at the interface and the propagation with the advancing growth front are separately considered. This is because certain defects formed by a growth disturbance (e.g. by inclusions) may heal out and do not continue into the further growing crystal, whereas other defects (dislocations, twins, grain boundaries), once initiated, are forced to proceed with the interface despite growth under optimal conditions. These defects can only be eliminated by growing out at the sides of the crystal, e.g., during Czochralski pulling on interfaces which are convex toward the melt. Moreover, distinction is made between defects always connected to the interface (growth defects, esp. growth dislocations) and defects generated ‘behind’ the growth front (post-growth defects). The latter defects may be formed already during the growth run, either by thermal stress or by precipitation. Furthermore, defect configurations may be preserved in their as-grown geometry or changed after growth (e.g., post-growth movement of dislocations).

Many experimental results and the majority of photographs presented in this review were obtained by growth experiments and x-ray topographic studies...
Generation and Propagation of Defects During Crystal Growth

4.2 Inclusions

Two categories of inclusions are distinguished according to their genetic origin [4.12, 13]: primary inclusions are associated with the growth front, i.e. they arise during growth, whereas secondary inclusions are formed after growth. Primary inclusions are key defects because they are the source of other defects (dislocations, twins) which propagate with the growth front into the further growing crystal. Inclusions of both categories may form stress centres which give rise to dislocation loops or half-loops by plastic glide (stress relaxation). Among primary inclusions there are three kinds of inclusions distinguished:

- Foreign particles
- Solvent (liquid) inclusions in crystals grown from solutions
- Solute precipitations in crystals grown from impure or doped melts

Secondary inclusions are precipitates of solute impurities (dopants) formed after growth in the solid state during slow cooling, annealing or processing of crystals which are grown at high temperatures. They are due to the supersaturation of solutes at temperatures below the temperature at which the crystal was grown. These solutes precipitate if their diffusion mobility is sufficiently high and not frozen-in (as is usually the case at room temperature). In the same way vacancies and self-interstitials may condense into dislocation loops and stacking faults, e.g. during processing silicon crystals for electronic applications (swirl defects, e.g. [4,14]).

Here we treat only primary inclusions. A very detailed theoretical and experimental treatment of the capture of inclusions during crystal growth is presented by Chernov and Temkin [4.15]. A similar study with particular consideration of crystallization pressure is reported by Khainov-Mal’kov [4.16].

4.2.1 Foreign Particles

Foreign particles pre-existing in the nutrient (solution, melt) increase the risk of (heterogeneous) nucleation. Their incorporation into the growing crystal, however, is often considered as not very critical due to the crystallisation pressure [4.16] (disjoining force after Chernov and Temkin [4.15]) which repulses foreign particles from the growth interface. Nevertheless, particles coming into contact with the growth face may be incorporated, depending on the size and chemical/physical nature of the particles and on growth conditions such as stirring, growth rate, supersaturation [4.15]. For example, potassium alum crystals can be grown inclusion-free (as assessed by optical inspection and x-ray topography) from old (i.e. repeatedly used) unfiltered aqueous solutions containing many floating dust particles, provided that growth conditions (temperature control, stirring) are stable enough to avoid the formation of liquid inclusions (see below).

On the other hand in crystals of benzil grown in old (repeatedly filled up) supercooled melts ($T_m = 96{°}C$), flocks of solid decomposition products floating in the melt are quite readily incorporated (unpublished observation by the author). In contrast to the solution growth of potassium alum, such benzil melts were not stirred, but thermal convection occurred due to the release of crystallization heat at the crystal surface [4.1]. In the latter case the incorporation seems to be favoured by the higher viscosity and the lower agitation of the nutrient phase, and probably also by the chemical similarity (carbon) of the particles to the growing crystal.

Foreign solid inclusions are very common in minerals. In laboratory and industrial crystal growth they usually play a minor role because they are easily avoided by filtering of the nutrient before growth. If solid inclusions appear during the growth run, e.g.,
as abrasives of the stirring device, continuous filtering is advised. This has been demonstrated by Zaitseva et al. [4.17] for the rapid growth of huge KDP and DKDP crystals with linear sizes up to 55 cm: continuous filtering during the whole growth run considerably increased the optical quality and laser damage threshold of these crystals.

The intentional incorporation of particle inclusions for the study of the generation of dislocations is reported in Sect. 4.4.2. The intentional inclusion of oil drops during crystallization from solutions was studied by Kliia and Sokolova [4.18].

4.2.2 Solvent Inclusions

Solvent inclusions are very common in crystals grown by all variants of solution growth (aqueous and organic solvents, flux). Two origins are distinguished.

Facetting (Capping) of Rounded Surfaces

In general crystals grow from solutions with planar faces (habit faces), whereby faces with low surface energy grow slowly and determine the final morphology of the crystal (Wulff theorem [4.19]). If surfaces are rounded, (e.g., of the seed crystal or after redissolution), during first growth, facets of habit faces and (between them) terraces of these faces are formed. The facets become larger and the terraced regions grow out until a single edge between the two habit faces engaged is formed, as shown in Fig. 4.1 (Theorem of Herring [4.20–23], see also growth on spheres [4.24][4.25, p.130]). The healed-out regions often have the shape of caps (capping region). The growth on terraced surfaces favours the entrapment of solvent inclusions which may lead in extreme cases to a spongy structure of the capping region. This usually happens during first growth on seed crystals which were rounded during a final etching

A conspicuous example of capping is provided by potassium dihydrogen phosphate (KDP) grown in aqueous solution on (001) seed plates (Fig. 4.2). KDP develops habit faces {100} (tetragonal prism) and {101} (tetragonal dipyramid), but {001} is not a habit face. Thus in the first stage of growth on a (001) seed plate a spongy capping zone in the form of a tetragonal pyramid {101} over the seed plate as basis is formed, followed by clear further growth on {101} pyramid faces (Fig. 4.2). Detailed descriptions of this (001) capping process in KDP and ADP (ammonium dihydrogen phosphate) crystal growth are presented by Zerfoss and Slawson [4.12] and Janssen-van Rosmalen et al. [4.26].

Fluctuation of Growth Conditions (Growth Accidents)

A sufficiently strong change of growth condition (e.g., of supersaturation, stirring rate, stirring direction) may introduce – due to local variations of supersaturation – a (temporary) instability of growth faces: regions of retarded and promoted growth occur, leading to elevations and depressions on the growth face. Overhanging layers
and, thus, to changes of the impurity incorporation. As a rule, they affect the whole growth front and thus form inhomogeneity layers parallel to the interface. The term striations is usually applied when the impurity layers appear in a (quasi)-periodic sequence (Fig. 4.8a). If there are isolated layers, due to sporadic changes of growth conditions, often the term growth bands is used. In mineralogy, the notation growth zoning is common. In crystals grown under rotation, strictly periodic rotational striations, correlated with the rotation rate, may occur. They are due to a nonuniform radial temperature distribution around the rotation axis, leading to slight changes of growth conditions (even with remelting) within a rotation period.

The impurities may be contaminants of the solution or of the melt, or incorporated solvent components. Striations are also formed by dopants intentionally introduced with the aim to tailor specific physical properties of the crystals. The rate of incorporation depends on the species of impurities (dopants) and is governed by their distribution coefficient with regard to the crystal to be grown (see elsewhere in this Volume).

The regions of different impurities/dopants form layers coinciding with the instantaneous growth front. In crystals grown on habit faces (from solution or supercooled melt) they are planar as is shown in Figs. 4.8a and 4.9. The intensity of the striations, i.e., the concentration of impurities, may be considerably different in distinct growth sectors (see following Sect. 4.3.2). This is due to different surface structures of different growth faces which may facilitate or impede the impurity capture. Symmetrically equivalent growth sectors show the same intensity of striations unless the growth conditions (e.g., solution flow) at the corresponding growth faces are different. Conspicuous examples are the so-called hourglass growth patterns of crystals stained with organic dyes. Staining of crystals has been thoroughly studied by Kahr and Guerney [4.29], and Kahr and Vasquez [4.30]. Striations are often modified by growth hillocks (vicinal pyramids) as is shown below (Sect. 4.3.3). An x-ray topographic study of the striation formation in the presence of vicinal pyramids in rapidly grown KDP crystals is presented by Smolsky et al. [4.48].

**Fig. 4.8** (a) Growth striations and growth-sector boundary in a crystal grown on planar (habit) faces. The sector boundary is an internal surface formed by the movement of the edge joining the two faces during growth. It separates regions of different growth directions. (b) Division of a fully faceted crystal into growth sectors. The vectors \( \mathbf{n}_i \) indicate the growth directions. Dashed lines: contours of the crystal at different stages of growth. One of the growth sectors has grown out.

**Fig. 4.9** X-ray topograph of a (0001) plate (about 8 mm diameter, 0.35 mm thick) cut out of a quartz-homeotypic gallium phosphate GaPO\(_4\) crystal grown from high-temperature solution in phosphoric/sulphuric acid [4.47]. It shows the triangular arrangement of growth sectors with pronounced striations. Growth-sector boundaries are visible by topographic contrast or by the bends of the striations. Diffraction vector \( g(1010), AgK\alpha \) radiation.
Crystals grown on rounded interfaces exhibit curved striations. An example of striations accompanied by tiny gas bubbles in a Czochralski crystal is shown in Fig. 4.10. Facets formed on rounded interfaces lead to regions (facet sectors, Sect. 4.3.4 below) with planar striations. The occurrence and intensity of these striations may be quite different from striations formed along curved interfaces. This is due to distinct growth modes with different distribution coefficients for rough growth on curved interfaces and growth on facets from supercooled melt.

In general, growth striations lead to local changes of physical properties (e.g., electric conductivity, optical birefringence). This is a major problem in the growth of doped crystals for sophisticated electronic and optical solid-state devices. It can be encountered by the suppression of melt convection, e.g., by growth under microgravity [4.49] and by growth in magnetic fields [4.50], which are treated elsewhere in this Handbook. An extensive treatment of the origin of striations and of recipes to largely avoid them is presented by Scheel [4.51].

4.3.2 Growth Sectors

Bulk crystals grow in all directions of space. Due to their structural and physical anisotropy, the types, distribution and geometry of growth defects are distinct for different growth directions. This is pronounced in crystals, grown from solutions and supercooled melts, which develop planar growth (habit) faces and, thus, consist of regions (growth sectors) grown in discrete directions defined by the normals of the growth faces involved (Fig. 4.8b). Among all habit faces that are possible in principle, the final crystal usually exhibits only those faces which possess low surface (attachment) energies and thus – according to Wulff’s theorem [4.19–21] – have low growth velocities. Fast faces with higher attachment energy grow out and vanish from the external morphology (see Wulff–Herring construction [4.20–23]). Thus the crystal may contain more growth sectors, usually in close neighbourhood of the seed crystal, than are recognized from its final outward morphology (Fig. 4.8b).

Growth sectors are separated by growth-sector boundaries. These boundaries are internal surfaces over which the edges between neighboured faces have swept during growth. They are surfaces generated by the parallel movement of a straight line. When projected parallel to the edge (zone axis) of the two faces 1 and 2 involved, the boundary appears as a straight or somewhat curved line, the (local) direction of which depends on the (instantaneous) relative growth velocity \( v_1/v_2 \) of these faces (Sect. 4.3.6 and Fig. 4.14). If \( v_1/v_2 \) is constant, the line is straight (i.e., the boundary is planar), if \( v_1/v_2 \) fluctuates, the line is irregular, often zigzag-like as sketched in Fig. 4.8a (i.e., the boundary is an irregularly waved surface).

Growth-sector boundaries and their surroundings may be perfect crystal regions. In many cases, however, they are fault surfaces which can be observed by etching, optical birefringence and x-ray diffraction topography. The fault may be due to increased local impurity incorporation when growth layers on neighboured faces meet at their common edge, or to slightly different lattice parameters in both sectors. The latter lead to a transition zone along the boundary with lattice distortions which can be detected by the meth-
ods just mentioned. An example for this case is shown in Fig. 4.11. Lattice distortions preferentially occur along boundaries between symmetrically nonequivalent faces, due to different incorporation of impurities (see previous Sect. 4.2.1) which leads to slight differences of their $d$-values. Boundaries between symmetrically equivalent sectors are often strain-free, but may be visible by the sharp bends of growth striations (if present), see Fig. 4.9. An illustrative example of the extraordinarily rich growth sectoring of natural beryl, revealed by x-ray topographic imaging of sector boundaries and striations, is presented by Herres and Lang [4.52]. For the x-ray topographic characterisation of faulted growth-sector boundaries as shift or tilt boundaries, see Klapper [4.7, 8, 53].

The different incorporation of additives in different growth sectors is strikingly demonstrated in the so-called dyeing of crystals which goes back to Sénarmont (1808–1862) and was in the last two decades extensively studied by Kahr and coworkers (e.g. [4.29, 30]). They grew crystals from solutions with organic dye molecules as additives. The distinct incorporation of these molecules on different growth faces is conspicuously apparent from the different colouring of their growth sectors (see also Sect. 4.3.5 Optical Anomalies below). A similar study of colouring the growth sectors of KDP with organic dyes is reported by Maeda et al. [4.54].

### 4.3.3 Vicinal Sectors

Another, less pronounced kind of sectoring frequently arises within the growth sectors, treated above, by growth hillocks (growth pyramids). These very flat vicinal pyramids, which are caused by dislocations emerging at their apex, often exhibit facets (vicinal facets) deviating by only very small angles from the main growth face. The facets are formed by terraces of growth layers, and their slopes depend on the step height and the widths of the terraces. On facets with different slope angles the incorporation of impurities is different. This leads to slightly distinct $d$-values of the regions grown on different vicinal facets (vicinal sectors). In analogy to growths sectors, the ridges of the vicinal pyramids form the vicinal-sector boundaries, which may be faulted surfaces. This also holds for the valleys between neighboured vicinal pyramids.

A detailed x-ray topographic study of vicinal sectors and their boundaries, formed on {101} dipyramid faces and {100} prism faces of KDP and ADP was published by Smolsky et al. [4.55] and Smolsky and Zaitseva [4.56], who also coined the term vicinal sector. An atomic-force microscope in-situ investigation of the step structures of vicinal hillocks in relation to the Burgers vectors of unit and multiple unit height of the dislocations generating the hillocks is presented by De Yoreo et al. [4.57]. Pronounced triangular vicinal pyramids are observed on {111} octahedron faces of potassium alum (cf. the optical and x-ray topographic study by Stukenberg et al. [4.58], and Klapper et al. [4.59]). Tetragonal vicinal pyramids generating faulted sector boundaries on {001} faces of tetragonal nickel sulphate hexahydrate have been studied by van Enckevort and Klapper [4.60]. The impurity incorporation on different slopes of vicinal hillocks on {111}
boundary in the corresponding growth stage. Thus, the relative growth rates are easily reconstructed if the growth-sector boundary is visible.

An illustrative example, showing the strong changes of the growth velocities (growth-rate dispersion) due to fluctuations of growth conditions and defects is presented in Fig. 4.15a,b [4.59, 75]. It shows x-ray topographs of a (110) plate cut from a potassium alum crystal (grown from aqueous solution by temperature lowering) which was subjected to a temporary re-dissolution by a temporary temperature increase of about 1 °C [4.27]. The two boundaries between the central cube sector (001) and the two neighboured octahedron sectors (111) and (111) are clearly depicted by kinematical contrast due to lattice distortions. (For the contrast variations in different x-ray reflections, see Chap. XX by Dudley in this Handbook.) Figure 4.15c outlines the shape of the crystal in different stages of growth, reconstructed from the course of the growth-sector boundaries (dotted line). Four regions (1)–(4) of different relative growth rates are distinguished. In the first period, after seeding-in, the crystal was grown by continuous temperature decrease of about 0.3 °C/day until it reached the shape outlined by A-A-A-A. At this stage the temperature of the growth chamber was increased in one step by 1 °C. Due to the slow transfer of the temperature jump into the solution, redissolutions started about half an hour later, recognized by the rounding of the crystal edges. Now the previous temperature and decrease rate was restored and growth continued as before. By this disturbance a layer of liquid inclusions covering a part of the (001) facet was formed, and its growth rate, relative to the neighboured {111} faces (the growth rate of which remained constant during the whole experiment), was strongly increased in region (2). In regions (3) and (4) it decreased again. From the angles $\beta_1$, $\beta_2$ ($\alpha = 35.26^\circ$) the relative growth rates were determined to

\[
v(001)/v(111) = 1.0/5.6/1.7/0.8
\]

in growth intervals (1)/(2)/(3)/(4) (averaged over the nearly equal left and right-hand sides). The drastic increase in period (2) is obviously due to the dislocations originating from the inclusions,
4.4 Dislocations

4.4.1 Growth Dislocations and Post-Growth Dislocations

Dislocations are generated during crystal growth, by plastic deformation and by the condensation of self-interstitials and vacancies. In the study of crystal growth defects it is useful to distinguish between two categories of dislocations:

1. Dislocations, which are connected with the growth front and proceed with it during growth (growth dislocations or grown-in dislocations); and
2. Dislocations, which are generated behind the growth front, either still during the growth run or during cooling to room temperature (post-growth dislocations), or later during processing or by improper handling.

The final arrangement of dislocations in a crystal at room temperature results from growth dislocations, post-growth dislocations and the movement, multiplication and reactions of both after growth. Crystals grown at low temperatures (e.g., from aqueous solution) and in their brittle state usually contain dislocations in their original as-grown configuration; whereas in crystals grown at high temperatures, the original dislocation configurations may be drastically altered by dislocation movement, dislocation multiplication and dislocation reactions. These processes, which may occur during the growth run (behind the growth front), are induced by thermal stress due to temperature gradients and, particularly in crystals grown at very high temperatures, by the absorption of interstitials and vacancies (dislocation climb).

In this chapter the formation and propagation of dislocations in crystals grown at low temperatures (below 100 °C) under zero or only low thermal gradients are treated. The development of dislocation configurations during growth from melt under high thermal gradients or during processing at elevated temperatures has been experimentally and theoretically studied by various authors (e.g., [4.79–82]) and is reviewed by Rudolph in Chap. XX of this Handbook.

4.4.2 Sources of Growth Dislocations

For topological reasons dislocations lines cannot start or end in the interior of a perfect crystal. They either form closed loops, or they start from external and internal surfaces (e.g., grain boundaries), or from other defects with a break of the crystal lattice. In crystal growth, such defects may arise from all kinds of inclusions (e.g., foreign particles, liquid inclusions, bubbles, solute precipitates). When inclusions are overgrown and closed by growth layers, lattice closure errors may occur. These errors are the origin of growth dislocations which are connected to the growth front and propagate with it during further growth.

It is a very common observation that inclusions are the source of growth dislocations. Examples are shown in Figs. 4.3, 4.5, 4.10, etc. The appearance of dislocations behind an inclusion (viewed in the direction of growth) is correlated with its size: small inclusions emit only a few dislocations or are often dislocation-free. Large inclusions (> 50 μm) usually emit bundles of dislocations. In some cases, however, large inclusions (several millimeters in diameter) of mother solutions without dislocation generation have been observed (e.g., in the capping zone of KDP [4.83, 84]).

The generation of growth dislocations by foreign-particle inclusions has been experimentally studied by Neuroth [4.85] on crystals growing in aqueous solution (potassium alum) and in supercooled melt (benzophenone (C6H5)2CO, \(T_m = 48°C\); salol \(C_{13}H_{10}O_3\), \(T_m = 42°C\)). A seed crystal is fixed to a support in such orientation that a dominant growth face (octa-
hedron (111) for cubic potassium alum, prism (110) for orthorhombic benzophenone, pinacoid (100) for orthorhombic salol) develops horizontally. After a sufficiently long distance of (visually) perfect growth, a small ball of solder (0.3–0.5 mm) is placed on the horizontal growth face, and growth continued without change of conditions. During the whole experiment the growth surface was observed with a microscope (long focal distance) in reflected light or by Michelson interferometry, both with video tape recording. After the deposition of the ball the face grows slowly as before without additional surface features as long as the ball is not covered by growth layers. During this period the crystal seems to sink into the growing crystal. In the moment when the ball is covered by growth layers, a sudden hectic criss-cross movement of growth steps is observed, which after some ten minutes calms down and develops into several slowly growing spiral hillocks. During further growth the number of hillocks is reduced to a few dominating growth hills which have overrun other hills.

For the study of the dislocations associated with the inclusions, a plate containing the ball and the region behind it was cut out of the crystal and subjected to x-ray topography. Figure 4.16 shows that numerous dislocations, propagating with the growth front, originate from the back side of the ball. Their density is partially too high to be resolved by this imaging method.

Similar experiments have been performed with mechanical in-situ violation (puncturing, scratching) of an interface perfectly and steadily growing in solution, supercooled melt and by Czochralski pulling [4.85]. Again, bundles of dislocations originate from these damages, which in solution growth usually give rise to liquid inclusions. In plastic crystals (always the case in melt growth) the mechanical impact generates glide dislocations which emerge at the growth front and continue as growth dislocations. Similar experiments are reported by Forty [4.86, esp. p. 23]. His review presents a rich collection of photographs of growth spirals and other surface patterns on growth faces of various crystals.

The formation of screw dislocations in thin plates of organic crystals during growth from solution and from the vapour has been studied in-situ by Russian authors using (polarised) light microscopy with film recording [4.87–89]. Screw dislocations arise at re-entrant corners between branches of dendrites [4.87] and by growth around intentionally introduced particles [4.88, 89]. In these cases the dislocations run through the lamellae and do not end inside the crystal. The mechanism of the formation of lattice closure errors and of dislocations behind an inclusion on nanometer scale is not yet fully understood although simple models have been derived. An example is presented by Dudley et al. [4.90].

As has been pointed out (Fluctuation of Growth Conditions (Growth Accidents)) paragraphs have no number. So they can’t be referenced by number. Please confirm this change., in habit-face crystals inclusions preferentially arise in the regeneration zone of growth on rounded interfaces, in particular in the zone of first growth on a seed crystal. Moreover, dislocations and other defects (grain boundaries, twins) pre-existing in the seed will continue into the growing crystal. Thus, the perfection of the seed as well as the seeding-in process are most crucial for the growth of perfect crystals. This holds for all methods of seeded growth, not only for habit-face crystals. That the regeneration zone around the seed crystal is the main source of dislocations is apparent from several topographs shown in this review. It is stressed that inclusions and dislocations can largely avoided by very slow (and thus time-consuming)

![Fig. 4.16 X-ray topograph of a (010) plate (about 1.5 mm thick, width 21 mm) cut from a crystal of orthorhombic salol grown in supercooled melt. It contains a solder ball (diameter about 0.4 mm) dropped on a perfectly growing (100) facet (directed upward). Numerous dislocations were generated behind the ball. Dislocations of the fan propagating to the left are pure screw and exactly parallel to one of the prominent ⟨101⟩ edges which dominate the shape of the crystal (cf. Deviations from Calculated Directions (i) and Fig. 4.22). The (not resolved) dislocations of the vertical bundle have Burgers vectors [100] and [001]. Diffraction vector g(200), CuKα1]
growth during the regeneration period of first growth on a perfect seed.

Finally it is emphasized that inclusions can also block already existing dislocations. This has several times been observed by the author and reported in the literature. It frequently happens to dislocations in the seed crystal which are blocked by inclusions formed in the regeneration zone (capping) of first growth and do not enter the growing crystal [4.83, 84]. Thus provoking a capping zone by an intentionally introduced deviation of the seed surface from a habit face may be helpful for reducing the number of dislocations coming from the seed, but it implies also a considerable risk of generating new dislocations behind the inclusions. The blocking of growth dislocations by closed inclusions must obey the conservation law of Burgers vectors as discussed in the following Sect. 4.4.3.

4.4.3 Burgers Vectors, Dislocation Dipoles

The sum of the Burgers vectors of all dislocations originating from a closed inclusion embedded in an otherwise perfect crystal is zero [4.83]. This directly follows from Frank’s conservation law of Burgers vectors (see text books on dislocations, e.g. [4.91–93]) which states that the sum of Burgers vectors \( b_i \) of all dislocation lines going into a dislocation node (i.e., with line direction into the node) is zero \( \sum b_i = 0 \) (analogous to Kirchhoff’s law of electrical currents). Another proof may be given via the Burgers-circuit definition of Burgers vectors (e.g. [4.91–93]): imagine a Burgers circuit parallel the growth face in the perfect crystal region grown before the inclusion was formed. Now shift the circuit stepwise in growth direction over the inclusion and the dislocation bundle behind it. No closure error of the circuit, which now encircles all dislocations, will arise during this (virtual) procedure: \( \sum b_i = 0 \).

From this it immediately follows that a single dislocation cannot originate from an inclusion. If dislocations are formed, there must be at least two of them, with opposite Burgers vectors. This is often observed when the inclusions are very small. Two slightly diverging dislocation lines emanating from small, x-ray topographically invisible or nearly invisible inclusions were observed in KDP by Fishman [4.83]. Examples are presented in Fig. 4.19 (label A) for salol grown from supercooled melt. A few pairs of slightly diverging dislocations, starting from a point, can also be recognized in Fig. 4.10 of a Czochralski salol specimen. There are, however, many x-ray topographic observations of only one dislocation line arising from an inclusion (e.g., in Fig. 4.3, where only one stronger contrast line comprises the image of more than one dislocation). In these cases the line must represent a pair of two closely neighboured (x-ray topographically not resolved) parallel dislocations with opposite Burgers vectors: a dislocation dipole. Such a dipole can alternatively also be considered as a single dislocation in the shape of a narrow hairpin with its (virtual) bend in the inclusion. In this approach the two branches of the hairpin have the same Burgers vector, but opposite directional sense. Examples of a pure-screw and two pure-edge dislocation dipoles are shown in Fig. 4.17. The two branches of the dipoles attract each other and may annihilate if they come close enough together. This annihilation is possible for screw dislocation dipoles, and for edge dipoles if both edge dislocations of the latter are on the same glide plane. If they are located on different glide planes,
the two dislocations can approach each other to a minimum separation, where they have prismatic character and form the edges of stripes of inserted or missing lattice planes (Fig. 4.17b). A model of the formation of a screw dislocation behind an inclusion is presented by Dudley et al. [4.90].

During x-ray topographic studies of growth dislocations it is often observed that only one contrast line originates from an inclusion, indicating – at first sight – a single dislocation (Fig. 4.3). Here the question arises, how by x-ray topography a dislocation dipole can be distinguished from a single dislocation with the same Burgers vector. If the dipole dislocations are sufficiently separated, they are resolved as two lines or appear as a broader contrast line. However, since the strain fields of the two dislocation have opposite signs and subtract each other, the resultant strain may also be smaller and less extended, if the separation of the two dislocations is small. Thus a dipole may appear on x-ray topographs by similar of even narrower contrast than a single dislocation with the same Burgers vector, and a distinction of both is often not immediately possible. An example is shown in Fig. 11a of van Enckevort and Klapper [4.60], where a single contrast line represents a screw dislocation dipole in nickel sulphate hexahydrate, as is proven by the bulge of one of the two dipole arms. In the same crystal the presence of two closely neighboured etch pits at the apices of growth pyramids and slip traces indicating the escape of one of the two screw dislocations from the hillock center have been observed. Dislocation dipoles are also formed during plastic flow of crystals, when the movement of glide dislocations is locally blocked by obstacles (inclusions, jogs). Examples are given in Sect. 4.4.5 (Figs. 4.25, 4.26).

4.4.4 Propagation of Growth Dislocations

Characteristic Configurations, Theory of Preferred Direction

A dislocation line ending on a growth face will proceed with this face [4.7, 8, 94]. Its direction depends on the shape and the orientation of the growth face and on its Burgers vector. As was shown in Sect. 4.3.2 and Fig. 4.8b, crystal growing on planar (habit) faces consist of growth sectors belonging to different growth faces (different growth directions \( n \)). This leads – under ideal conditions (i.e., stress-free growth) – to a characteristic configuration of growth dislocations which is illustrated in Fig. 4.18. The dislocations start from inclusions and propagate as straight lines with directions \( l \) usually close to, and frequently parallel to, the growth direction of the sector in which they lie. They usually exhibit sharply defined, often noncrystallographic preferred directions \( l_0 \) which depend on the growth direction \( n \) and on the Burgers vector \( b \): \( l_0 = l_0(n, b) \). The dependence
of the preferred direction $l_0$ on the growth direction $n$ becomes strikingly apparent when the dislocations penetrate growth-sector boundaries. This implies an abrupt change of the growth direction: the dislocation lines undergo an abrupt change of their preferred direction $l_0$. An example is shown in Fig. 4.19.

These preferred direction of growth dislocations are explained by two approaches [4.7, 8, 94]:

1. **Minimum-energy theorem** (Fig. 4.20a). The dislocation lines adopt a direction $l$ (unit vector), for which its energy within any growth layer is a minimum. For a growth layer of unit thickness $d = 1$ this can be expressed as

$$E/\cos \alpha = \text{minimum},$$

with $E = E(l, b, c_{ij})$ the elastic energy (strain energy) per unit length of the dislocation line ($c_{ij}$ are the elastic constants of the crystal) and $\alpha$ the angle between $n$ and $l$. The factor $1/\cos \alpha$ accounts for the length of the dislocation line in the layer.

![Fig. 4.20 (a) Derivation of the energy of a straight dislocation line within a layer parallel to the growth face. (b) Illustration of the force $dF$ exerted by the crystal surface upon a line segment $dl$ of a straight dislocation line emerging at the surface (theorem of Lothe [4.95])](image)

2. **Zero-force theorem** (Fig. 4.20b). A dislocation line emerging at the surface experiences a force $dF$ which depends on the angle $\alpha$ between the dislocation direction $l$ and the surface normal $n$. At the surface this force is infinitely large. According to Lothe [4.95] there exists always a direction $l_0$ for which this force is zero for the dislocation line segments at any depth below the surface. It is plausible that a dislocation emerging at the growth face follows during growth this direction of zero force.

Using the formula

$$dF = -\frac{1}{r} \left( \frac{\partial E}{\partial \alpha} + E \tan \alpha \right) dl,$$

(theorem of Lothe [4.95]), it can be shown that both approaches lead to the same preferred directions.

**Verification of the Minimum-Energy Approach**

The strain energy [4.7, 8, 94] per unit length of a straight dislocation line is given by (see textbooks on dislocations, e.g. [4.91–93])

$$E = \frac{Kb^2}{4\pi} \ln \left( \frac{R}{r_0} \right),$$

with $K = K(l, l_b, c_{ij})$ the so-called energy factor of a straight dislocation line, $R$ the outer cut-off radius and $r_0$ the inner cut-off radius, and $b$ the modulus of the Burgers vector $b$. The energy factor $K$ describes the variation of the strain energy with the direction $l$ of the dislocation line. It also depends on the on the Burgers vector direction $l_b$ and on the elastic constants $c_{ij}$ of the crystal. The inner cut-off radius defines the limit until which the linear elasticity theory is applicable, and it corresponds to the radius of the dislocation core.

Since the logarithmic term and the core energy are, in general, not accessible to a numerical calculation, the variation of strain energy $E$ of a given dislocation with Burgers vector $b$ with direction $l$ is – in a certain approximation – considered as proportional to the energy factor $K(l, l_b, c_{ij})$, assuming the logarithmic term as independent of $l$. The energy factor has been calculated using the theory of dislocations in elastically anisotropic crystals developed by Eshelby et al. [4.96]. Since, in general, analytic solutions are not possible, numerical calculations have been performed using a program DISLOC, accounting for elastic anisotropy of any symmetry down to the triclinic case [4.94]. Figures 4.21a,b show the comparison of observed and calculated preferred directions of dislocations with four different Burgers vectors in the (101)
growth sector of KDP [4.97]. The agreement is excellent with deviations of $3^{-6}^\circ$, except for dislocation 4, the observed directions of which scatter by $\pm 5^\circ$ around a direction deviating by about $20^\circ$ from the calculated one. This may be due to the very flat minimum of $K$, which makes the minimum energy directions more subject to other influences like surface features and core-energy variations (see Deviations from Calculated Directions). Similar comparisons have been carried out for various crystals grown on planar faces from solutions and supercooled melts (benzil, $\langle C_6H_5CO \rangle_2$ [4.53, 98]); thiourea, $(NH_2)_2CS$ [4.99], lithium formate monohydrate $\text{HCOOLi} \cdot \text{H}_2\text{O}$ [4.100], ammonium hydrogen oxalate hemihydrate, $\text{NH}_4\text{HC}_2\text{O}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$ [4.101; zinc oxide, ZnO [4.94]. In general, the agreement of observed and calculated directions is satisfactory and confirms the validity of the above theorems. It is pointed out that the preferred directions are independent of the growth method, provided that the growth faces (growth sectors) are the same. This has been demonstrated for benzil grown in solution in xylene and in supercooled melt ([4.1]; [4.7, p. 138 and Fig. 17 therein]). Moreover, basal growth dislocations (Burgers vectors $b = \langle 100 \rangle$) in prism sectors of hydrothermally grown hexagonal (wurtzite-type) ZnO crystals [4.102] show the same minimum-energy configuration as the corresponding growth dislocations in the prism sectors of benzil [4.94]. This similarity is due to the hexagonal lattice, the same prism growth sector and the same Burgers vectors of the dislocation in both cases.

An interesting experimental study of preferred dislocation directions in synthetic quartz is presented by Alter and Voigt [4.103]. They have cut (0001) plates (Z-plates) out of different growth sectors of previously grown highly perfect quartz crystals and used them as seed plates for further growth experiments. The seed plates contained growth dislocations following the preferred directions typical for the sector from which they were cut. Since growth now proceeded on the (0001) face, the dislocations of the seed continued into the growing crystals with preferred directions typical for the Z-sector, exhibiting sharp bends of up to $90^\circ$ at their transition from the seed into the grown crystal. This is instructively shown by x-ray topography [4.103].

On account of the factor $1/\cos \alpha$ in the energy term above, the preferred directions of growth dislocations

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**Fig. 4.21** (a) X-ray topograph ($g(020)$, Ag-K$\alpha$ radiation) of a KDP (100) plate (horizontal width $\approx 28$ mm, thickness $\approx 1.5$ mm), showing bundles of dislocations with noncrystallographic preferred directions emanating from small liquid inclusions (especially in the right-hand (011) growth sector) and from the capping region. The Burgers vectors of these dislocations can be recognized from their preferred directions. In addition, growth bands and features due to vicinal effects are visible. ABC: Boundary between (011) and (011) growth sectors. (b) Plots of calculated energies $E/\cos \alpha$ (arbitrary units) of dislocations with Burgers vectors $b = \langle 001 \rangle$ (1), $b = \langle 100 \rangle$ (2), $b = \langle 011 \rangle$ (3) and $b = \langle 011 \rangle$ (4) in growth sector (011) of KDP (polar coordinates: the energies are given by the length of the radius vector to the curves). The preferred directions of minimum $E/\cos \alpha$ are represented by arrows. The dashed lines are circles with radii equal to the minimum values of $E/\cos \alpha$. Note the close coincidence to observed (a) and calculated (b) dislocation line directions.
are mostly normal or nearly normal to the (local) growth face. In some cases of planar interfaces, however, deviations from the growth normal of up to 30° have been observed in agreement with the calculations. For interfaces with convex curvature (e.g., in Czochralski growth) the dislocation lines diverge and grow out of the crystal boule through its side faces, whereas for concave interfaces the dislocation lines are focussed into the center of the crystal boule (Fig. 4.10). Trajectories of growth dislocations in Czochralski gadolinium gallium garnet (GGG) have been calculated and compared with observed ones by Schmidt and Weiss [4.68]. The curvature of the interface has been taken into account by performing the calculations stepwise in small increments, leading to curved dislocation trajectories. Again the agreement is satisfactory. Moreover, it allowed assigning Burgers vectors to the different dislocation trajectories which were observed optically in polarised light.

In 1997 and following years, preferred dislocations directions and their bending when penetrating a growth sector boundary have been observed by transmission electron microscopy in GaN grown by MOVPE (metal-organic vapour phase epitaxy) using the ELO (epitaxial lateral overgrowth) technique [4.105–109]. The GaN hexagonal pyramids \{1122\} growing through the windows in the mask are in the first stage topped by the (0001) basal plane, which during further growth becomes smaller and finally vanishes. Thus growth dislocations propagating normal to the (0001) facet penetrate the boundary to a \{1122\} sector and are bent by about 90° into the preferred direction in this sector [4.108]. Very recently the bending of dislocations in growth-sector boundaries has also been used for the elimination of threading dislocations by aspect ratio trapping in Ge selectively grown in submicron trenches on Si substrates [4.110]. Similarly, dislocations are eliminated from prism growth sectors of rapidly grown KDP crystals ([4.111, cf. Fig. 4 therein]).

The above theory of preferred dislocation directions does not allow a dislocation line to proceed along a growth-sector boundary, as is sometimes discussed: the line would emerge on the edge constituting the boundary and thus be in a labile position. If, however, the edge is a narrow facet, the dislocation line can lie in its sector and appear to proceed along the boundary, but the probability of breaking out into one of the adjacent sectors would be high. Ester and Halfpenny [4.112] and Ester et al. [4.113] have observed in potassium hydrogen phthalate V-shaped pairs of dislocations originating from growth-sector boundaries, with the two arms of the \(V\), following sharply defined directions, in the two adjacent sectors. This is in accordance with the minimum-energy concept.

**Deviations from Calculated Directions**

Although the agreement of observed and calculated directions of growth dislocations is in general satisfactory, frequently discrepancies are found. The reasons for this may be due to the insufficient approximation by the model on which the calculations are based, or by influences of other defects, or by a particular surface relief. The above calculations are based on linear anisotropic elasticity of the continuum and do not account for the discrete structure of the crystals, the dislocation core energy and, in piezoelectric crystals, for electrical contributions.

The following three causes have been found to affect preferred directions:

i. Discrete lattice structure of the crystals and the neglect of the core energy
benzophenone crystal (temperature of the melt about 1.5°C above \(T_m = 48^\circ\text{C}\), pulling rate 20 mm/day). During pulling the crystal was cooled by blowing air of about 35°C against the rotating crystal in order to obtain a crystal diameter of 25 mm [4.43]. The topograph shows growth dislocations which originate from the zone of first growth on the seed crystal (outside the top of the figure). A few of the dislocations have still their original course (straight or slightly curved lines), but most of them have suffered post-growth movement (which may have occurred already during the pulling process): they consist of a series of bow-shaped segments connected at pinning points (right-hand side of Fig. 4.26). The pinning points are aligned along straight lines marking the original position of the dislocations. On the left-hand side of the Fig. 4.26 the changes are even more drastic: some dislocations have partially annihilated, leaving back only a few large and several small dislocation loops. Post-growth reactions of growth dislocations forming dislocation nodes in

![Fig. 4.25](image)

**Fig. 4.25** Section (about \(4 \times 7 \text{mm}^2\)) of a (110) plate (about 1.5 mm thick) of benzophenone grown from supercooled melt. The *large arrows* mark four growth dislocation in a (111) sector showing sharply defined non-crystallographic preferred directions (predominantly screw character) in their upper, and post-growth movement with pinning stops in their lower parts. Burgers vector \(b = [001]\) vertical. The horizontal segments are pure-edge and form a few dislocation dipoles (*small arrows*). The dislocations emerge through the plate surfaces. \(g(00\bar{2}), \text{CuK}_{\alpha_1}\)

![Fig. 4.26](image)

**Fig. 4.26** Section (3.8 \(\times\) 8.0 \(\text{mm}^2\)) of a plate (thickness about 1.2 mm) cut from a Czochralski crystal (pulling direction upward) of benzophenone \(T_m = 48^\circ\text{C}\), showing growth dislocation after post-growth movement. The pinning points of one of the dislocations at the right-hand side mark its originally straight course. At the left side some dislocations have partially annihilated and form closed loops. \(g(00\bar{2}), \text{CuK}_{\alpha_1}\)

Czochralski salol have been studied by Neuroth and Klapper [4.116]. Interestingly, the dislocations in Czochraski salol of Fig. 4.10 have preserved their growth configuration with straight lines roughly normal to the growth front, despite the thermal stress which is always present in this growth technique. This may be due to the low growth temperature \(T_m = 42^\circ\text{C}\) and the narrow plastic zone of salol below the melting point.

### 4.4.6 Post-Growth Dislocations

Dislocations formed in the interior of the already grown crystal without connection to the growth front or other surfaces must be closed loops [4.7–11]. It is practically impossible to generate closed loops in a perfect crystal by stress, since the stress required for such processes would be extremely high. Inclusions, however, usually represent stress centers and form internal surfaces in the crystal. The stress in the crystal around the inclusions is relieved by the emission of concentric dislocations loops or – more frequently – of disloca-
the x-ray topographs shown in Figs. 4.5, 4.21a, 4.23 and 4.33. The crystals shown in the former three figures have been grown in the 1970s in the laboratory of Bennema (Delft and Nijmegen, NL) in a three-vessel growth system [4.26] at constant temperature of about 30 °C and a supersaturation of about 5.4% with a growth rate about 1 mm/day on the (101) faces. The crystals were mounted on a tree which rotated in the solution. They contain inclusions, inhomogeneous impurity distributions (in the form of striations, growth and vicinal sectors) and dislocations. As was pointed out already by Zerfoss [4.12], Janssen-van Rosmalen et al. [4.26, 32], van Enckevort et al. [4.33], liquid inclusions, in particular the quasi-periodic liquid inclusions in Fig. 4.5, can be avoided by strong stirring.

An x-ray topograph of an ADP crystal rapidly grown in the author’s laboratory is shown in Fig. 4.33 [4.164]. The crystal was grown on a point seed in a sealed 10 l tank by temperature decrease from 55 to about 45 °C with about the same growth rates on {100} prism and {101} pyramid faces in ten days to a width of 110 mm along the ⟨100⟩ edges and a height of about 80 mm. It was rotated in the solution on a platform with about 50 cycles/min and reversal of the rotation every 30 seconds. The rate of temperature decrease was 0.1–1 °C in the generation period and is then stepwise increased to 4 °C/day, dependent on the size of the crystal. It is obvious from Fig. 4.33 that this crystal is much more homogeneous and perfect than the crystals shown in Figs. 4.5, 4.21a, 4.23. Only few dislocations originate from the point seed into the prism and pyramid growth sectors. Due to growth face instabilities large liquid inclusions have formed on the right prism face and but have perfectly healed out during further growth. Interestingly these inclusions block more growth dislocations than they generate. A convincing proof of the homogeneity of this crystal is the appearance of the x-ray pendellösung fringes, which result from the slight thickness decrease toward the edges of the plate. Neigh-
boured (010) crystal plates, not cutting though the seed and through the inclusions, are dislocation-free.

Rapid crystal growth of very large, highly perfect crystals from aqueous solution has been reported only for KDP, its deuterated variant DKDP and for ADP [4.36]. In order to check the applicability of this method to other materials, growth experiments have been carried out with some other compounds usually used in student laboratory courses: pure and mixed potassium and ammonium alums, sodium chlorate and nickel sulphate hexahydrate [4.164]. The growth procedure was essentially the same as reported above for ADP. Apart from some failures due to spontaneous nucleation, large crystals of these materials with edge lengths up to 12 cm could be successfully grown within 7–10 days with growth rates up to 25 mm/day. Optical inspection and x-ray topographic studies indicate that the optical and structural perfection is high and not at all inferior to the quality of corresponding traditionally grown crystals. These results indicate that the rapid-growth method is applicable to many crystals growing from solutions.

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