
1 Imperfections of the Crystal Structure

1.1 DISLOCATIONS

Dislocations are line defects of the crystal lattice, which border the regions in the crystal interior in which slip has occurred. The measure for the quantity of dislocations in the crystal is given as the *dislocation density*—the total length of all of the dislocation lines per unit volume.

The main characteristic of each dislocation is the *Burgers vector*, which describes the magnitude and direction of slip movement associated with the dislocation. The classification of dislocations is based on the mutual orientation of the dislocation line and the Burgers vector of the dislocation. The direction of an *edge dislocation* is perpendicular to the Burgers vector. In contrast, the direction of a *screw dislocation* line is parallel to the Burgers vector. Most of the dislocations are *mixed dislocations*. They are a combination of screw and edge segments, as well as a large mixed component; that is, the direction of the Burgers vector of such a dislocation changes along the length of the dislocation. Mixed dislocations usually form *dislocation loops*.

When the magnitude of the Burgers vector equals a whole lattice vector, it is referred to as a *unit* or *perfect dislocation*. Such dislocations, when passing through the crystal, do not change the arrangement of atoms in the lattice because a complete lattice translation occurs. Dislocations with a Burgers vector not equal to a whole lattice vector are referred to as *imperfect* or, more often, as *partial dislocations*.

Any perfect (unit) dislocation may have an edge, screw, or mixed nature; each mixed dislocation can be either unit or a partial. Each type of close-packed crystal structure has its own set of unit and partial dislocations.

All dislocation reactions are described by the magnitude and direction of the Burgers vectors. The *dislocation reactions* in real crystals are as follows:

- *Combining* of two or more dislocations
- *Splitting* of unit dislocations into partial dislocations
- *Interaction* of partial dislocations resulting in dislocations of a new type
- *Annihilation* of dislocations with opposite Burgers vectors

A basic physical characteristic of metals and alloys that governs the mode of dislocations movement and the type of dislocation reactions is the *stacking fault energy (SFE)*—the energy necessary to produce a unit area of *stacking fault (SF)* in a perfect crystal. The SF is a planar lattice disorder with a stacking sequence of layers different from the perfect close-packed structure: for example, a layer of ...CACA... stacking sequence characteristic for hexagonal close-packed (hcp) crystal lattice inserted into

the face centered cubic (fcc) lattice with ...ABCABC... stacking sequence results in ...ABCACABC... arrangement, which contains an hpc layer of stacking fault.

The area of the SF is bordered by two *partial dislocations*: a leading one, which disturbs the stacking sequence, and a closing one, which restores the regular atomic arrangement of the matrix. The complex of the two partial dislocations connected by SF ribbon is called *split dislocation*, the equilibrium *width of the split dislocation* (distance between the two partials) determined by the SFE of the crystal. The value of SFE determines also the type of *dislocation substructure* produced in materials by plastic deformation.

The stacking faults are the simplest type of planar defects. When they are inclined to the foil surface, they are visualized in the TEM as ribbons containing parallel white and black fringes resulting from the diffraction contrast arising from the planar imperfection in the periodic arrangement of the atoms.

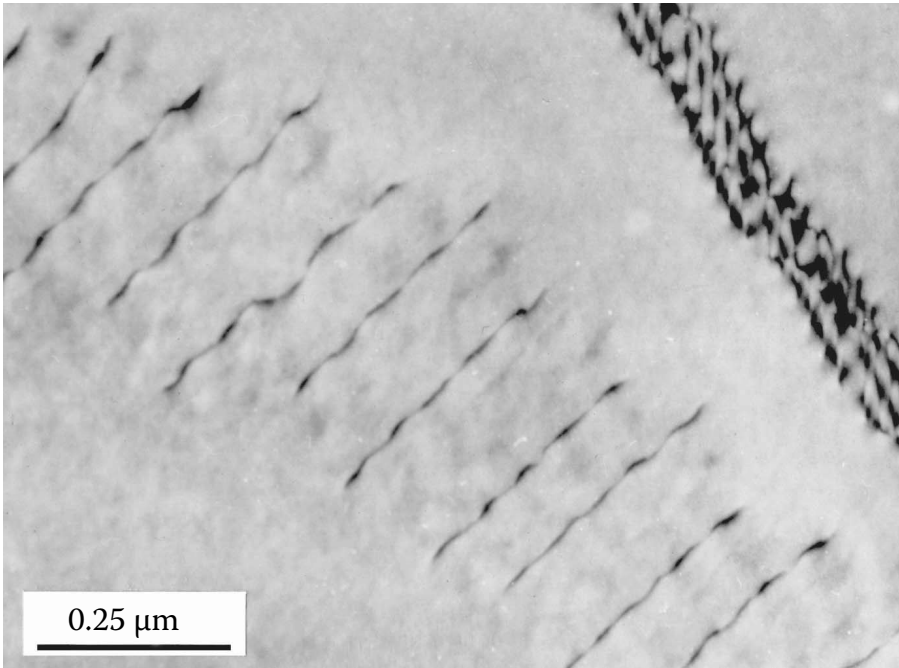


FIGURE 1.1

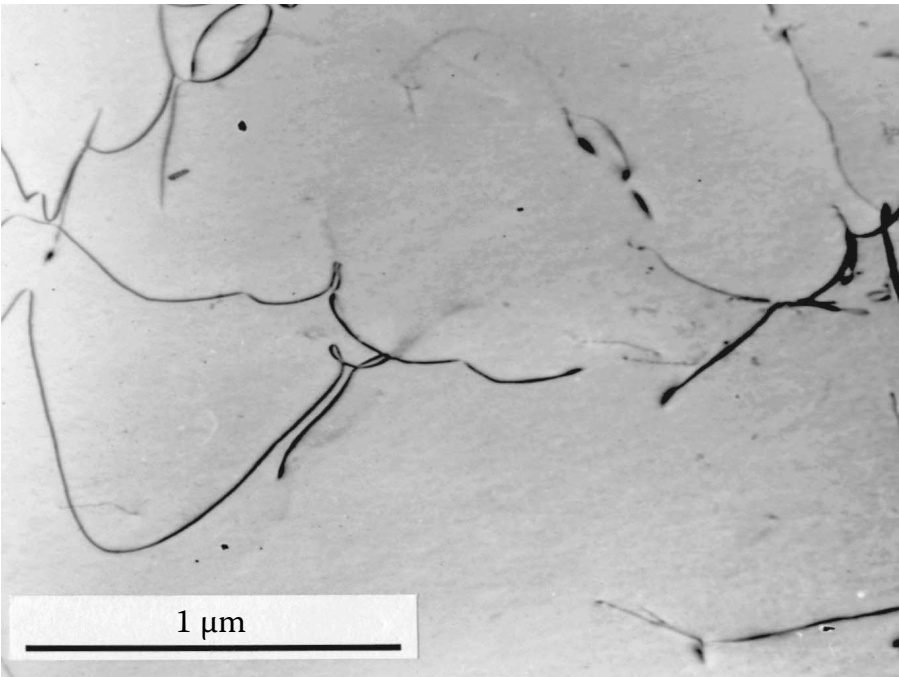


FIGURE 1.2

FIGURE 1.1 Real crystals contain a significant number of dislocations. An annealed crystal will have a dislocation density of approximately 10^7 cm of dislocation length per cubic centimeter of material (or 10^7 cm^{-2}). The smaller the number of dislocations per unit volume, the longer the single dislocation line (a sugar-cube-sized piece of any engineering alloy contains about 10^5 kilometers of dislocation line). The length of a single dislocation line visible in the TEM is much shorter: for example, less than 1 micrometer in the shown micrograph. This is because the dislocations are inclined to and thus cut by the two foil surfaces so that we see only short segments of them.

Quenched stainless austenitic steel. The oscillatory contrast along the shown dislocation segments is caused by the interference between the diffracted and transmitted electron beams near to the surfaces of the thin foil.

FIGURE 1.2 The dislocations in crystals of high SFE are always unit (perfect) dislocations. They can move by slip, not only in the plane of their Burgers vector but also in the intersecting planes, to produce a *three-dimensional* dislocation distribution. This type of distribution is characteristic of many pure metals—nickel, chromium, molybdenum (SFE about $300 \text{ mJ}\cdot\text{m}^{-2}$), aluminum (SFE = $250 \text{ mJ}\cdot\text{m}^{-2}$), magnesium (SFE = $200 \text{ mJ}\cdot\text{m}^{-2}$), titanium (SFE = $150 \text{ mJ}\cdot\text{m}^{-2}$), α -iron (SFE = $140 \text{ mJ}\cdot\text{m}^{-2}$), and zinc (SFE = $100 \text{ mJ}\cdot\text{m}^{-2}$).

Quenched aluminum.

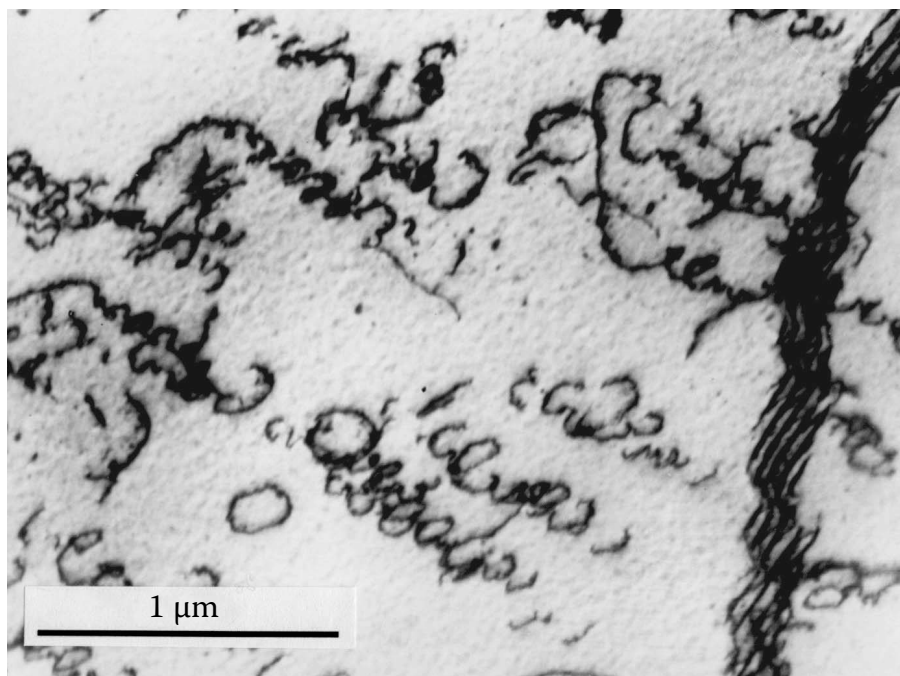


FIGURE 1.3 When a large number of vacancies are present in metals and alloys of high SFE, they produce *helical dislocations*. It is energetically advantageous for vacancies to condense around the axis of screw dislocations. Climbing dislocations with vacancies condensed around them obtain the shape of regular helices with an axis along the Burgers vector of the original screw dislocation. This process can often be observed in aluminum alloys quenched from a high temperature where it is an indicator of a high vacancy supersaturation in the alloy.

Quenched Al-4% Cu.

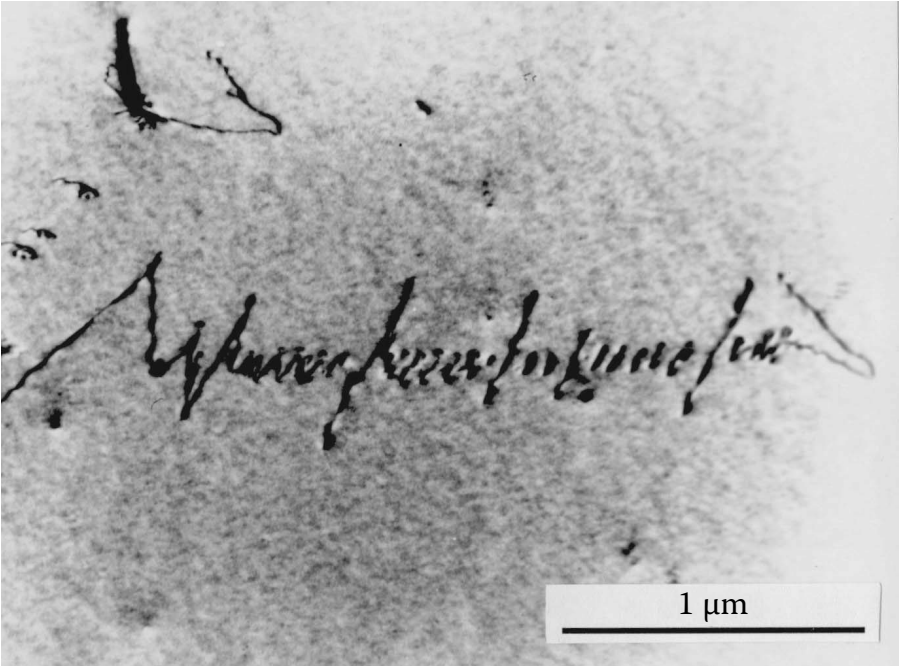


FIGURE 1.4

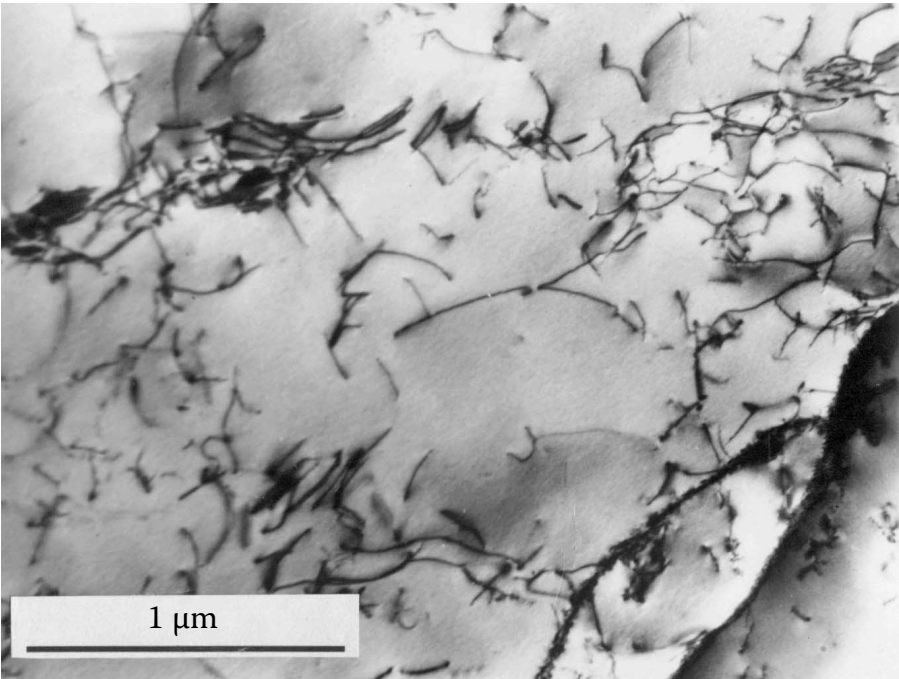


FIGURE 1.5

FIGURE 1.4 The shown image is not an ancient Arabian script, but a helical dislocation.
Quenched Al-4%Cu.

FIGURE 1.5 A lower SFE makes it difficult for dislocations to slip in planes different from the plane containing their Burgers vector. Nevertheless, a three-dimensional distribution of dislocations is still observed in metals of SFE about 30 to 40 $\text{mJ}\cdot\text{m}^{-2}$, for example, in copper or niobium. This is because the *cross-slip* to a plane other than the primary slip plane is still possible as a result of the small separation of the partial dislocations.

Quenched copper.

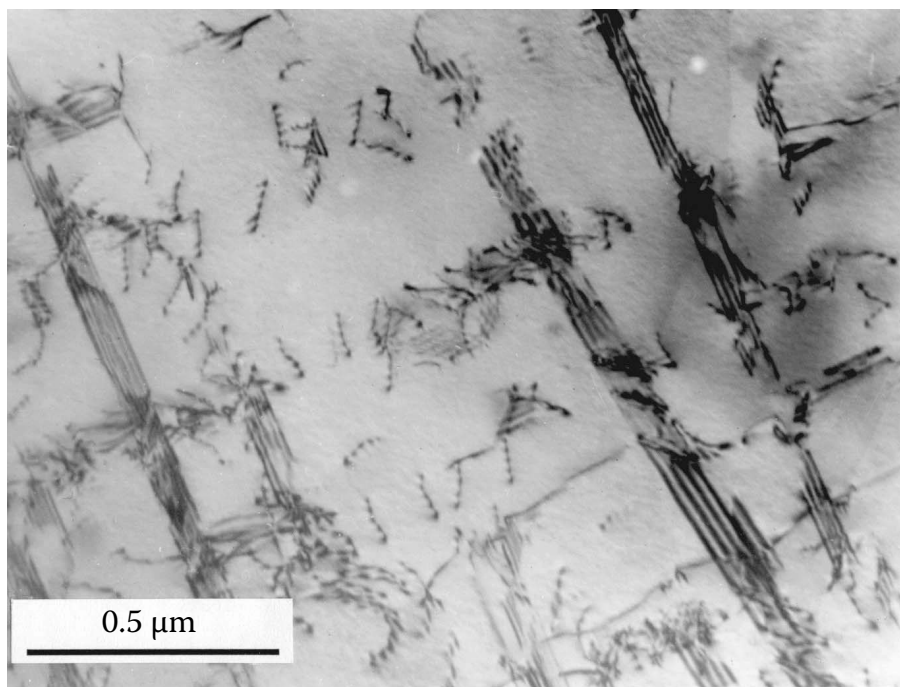


FIGURE 1.6 The distribution of dislocations in materials having an SFE less than 20 $\text{mJ}\cdot\text{m}^{-2}$ is strictly *two-dimensional* or *planar*. A representative pure metal of this type is silver with an SFE of 15 $\text{mJ}\cdot\text{m}^{-2}$. The planar distribution of dislocations is more frequently observed in alloys because alloying usually lowers the SFE. Examples are the stainless steel Fe-18Cr-9Ni (SFE = 16–40 $\text{mJ}\cdot\text{m}^{-2}$), α -brass CuZn30 (SFE = 10 $\text{mJ}\cdot\text{m}^{-2}$), and aluminum bronzes with 4% to 7% Al (SFE = 3–5 $\text{mJ}\cdot\text{m}^{-2}$).

Quenched stainless austenitic steel Fe-18Cr-9Ni.

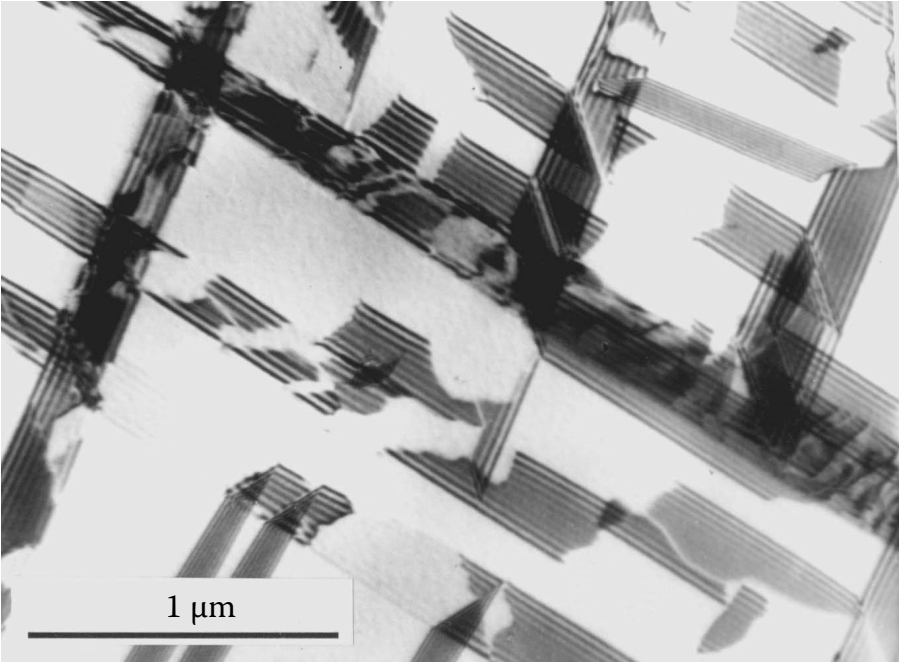


FIGURE 1.7

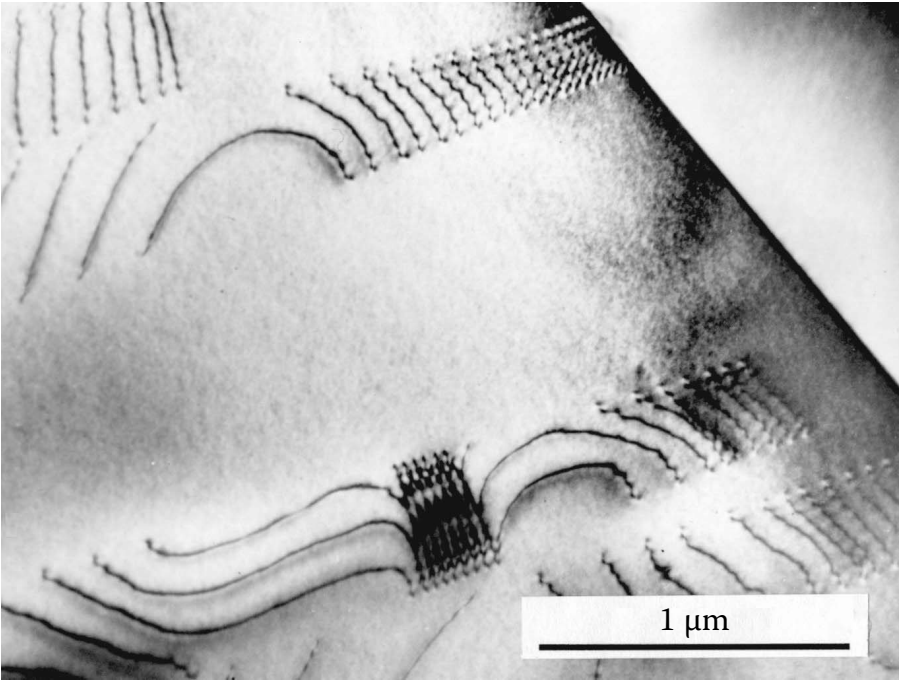


FIGURE 1.8

FIGURE 1.7 When the value of the SFE is very small, it is energetically more advantageous for dislocations to move by splitting into partials separated by SF. The lower the SFE value, the wider the SF layer: in the case of an SFE of about $1 \text{ mJ}\cdot\text{m}^{-2}$, the length of the SF layer can reach several micrometers.

Quenched austenitic nitrogen steel Fe-18Cr-14Mn-0.6N. The change of brightness or the loss of SF image in some areas in the micrograph results from the presence of several overlapping SFs in the foil thickness, which causes a change in the transmitted electrons' intensity.

FIGURE 1.8 By changing the imaging conditions used in [Figure 1.7](#), the parallel black and white fringes typical of a SF image can be made invisible.

Quenched austenitic nitrogen steel Fe-18Cr-14Mn-0.6N. As in [Figure 1.7](#), the partial dislocations in this micrograph are arranged in arrays in the parallel slip planes and actually are connected by SF bands, but the imaging conditions make these bands invisible.

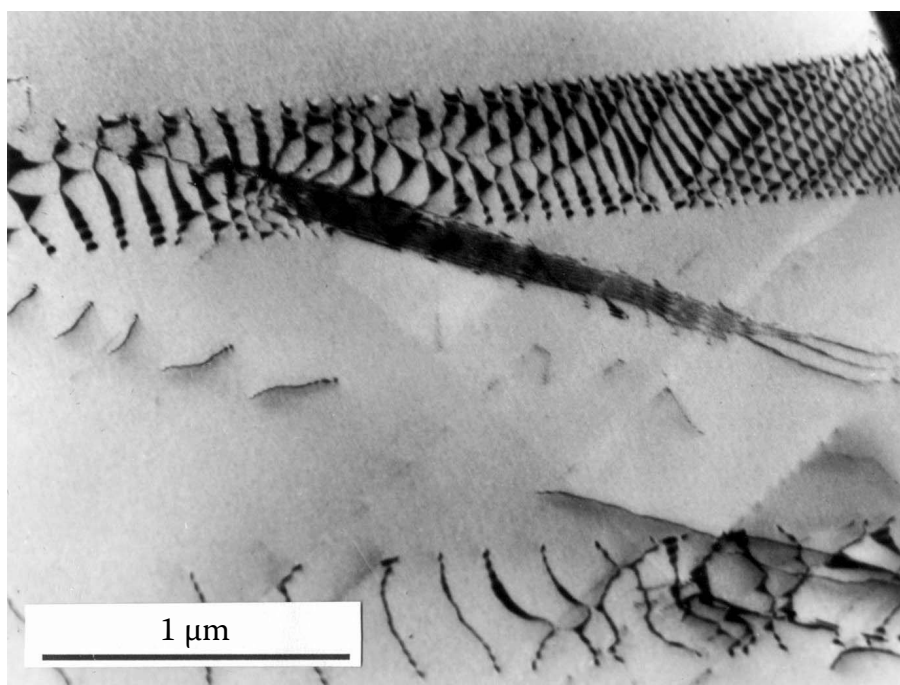


FIGURE 1.9 The intersection of two widely extended dislocations that move on different slip planes in a low-SFE metal produces a *triple dislocation node* containing an SF. Under equilibrium conditions, the area of the node, bordered by partial dislocations, serves as a measure of the SFE value.

Quenched austenitic nitrogen steel Fe-18Cr-14Mn-0.6N. The specific network of alternating expanded and shrunk triple nodes in the shown micrograph is a result of the crossing of two arrays of split dislocations moving on two intersecting planes.

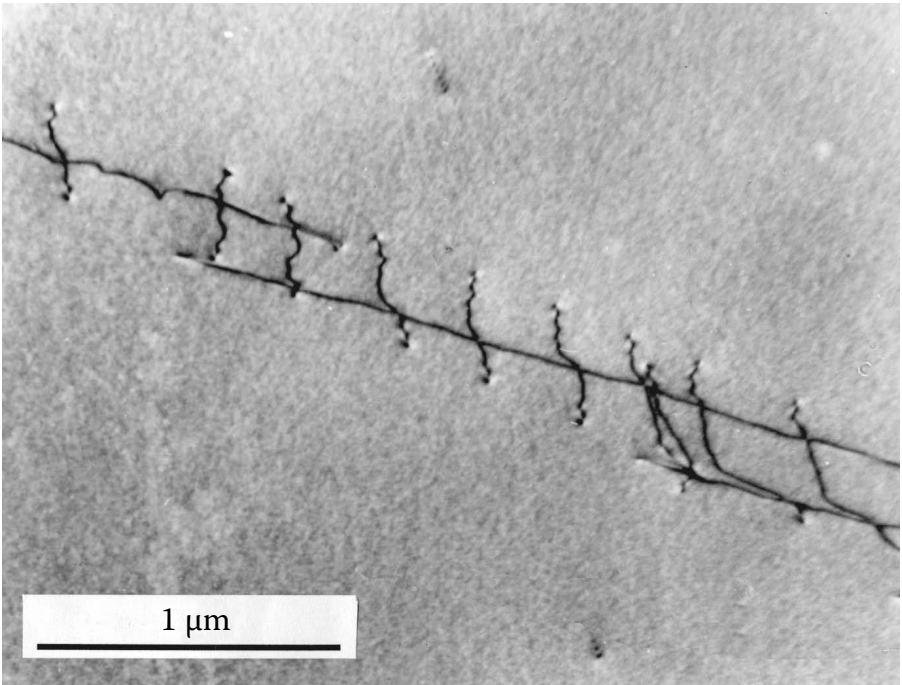


FIGURE 1.10 When a pair of extended dislocations meet at the intersection of two slip planes, the two leading partial dislocations interact and produce a new partial dislocation, which is referred to as a *stair-rod dislocation*. The stair-rod dislocation (named after the rod that holds a stair carpet in place) is practically immobile because of the complex structure of the three dislocations connected by a common wedge-shaped SF. This is the so-called *Lomer-Cottrell barrier* or *sessile dislocation*. It is recognized as one of the most difficult obstacles for dislocations to surpass and thus plays an important role in the work hardening of the fcc metals.

Quenched austenitic nitrogen steel Fe-18Cr-14Mn-0,6N.

1.2 MULTIPLICATION OF DISLOCATIONS

The number of dislocations in a crystal is changed by mechanical processing. The density of dislocations in a well-annealed crystal varies from 10^6 to 10^8 cm^{-2} . After 30 to 40% cold plastic deformation, the dislocation density increases to 10^{11} – 10^{12} cm^{-2} . One of the main sources for the multiplication of dislocations during plastic deformation is a *Frank-Read source*. A Frank-Read source is a region of the crystal with a high density of defects, which is capable of generating dislocations when the shear stress reaches a certain critical level. The process is repeated when a new dislocation breaks out and starts moving away from the source. The source continues to emit dislocations of the same type and can generate an unlimited number of dislocations if the applied stress remains in excess of the critical value.

The dislocations moving through the crystal have to overcome numerous obstacles—point defects and defect clusters, foreign atoms or phases, inhomogeneities and stress fields associated with the solid solution, other dislocations or dislocation complexes, and grain and twin boundaries. If the obstacle is difficult to overcome—for example, a grain boundary, an inclusion interface, or a field of stress concentration—the dislocations stop and form *dislocation pileups*. The stress on the leading dislocation produced by the pileup is proportional to the number of dislocations in the pileup. In many cases, it is sufficient to activate a Frank-Read source.

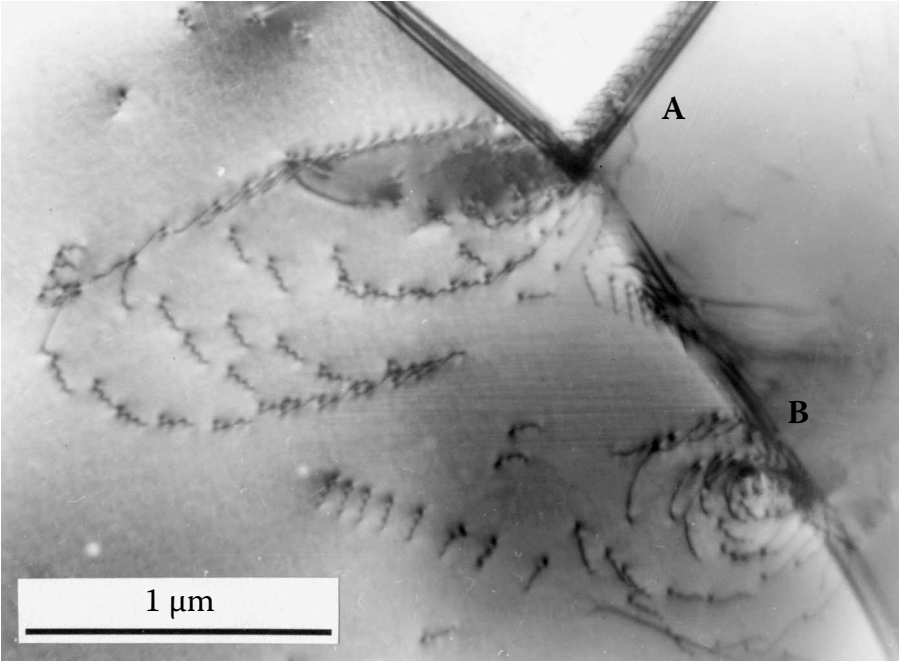


FIGURE 1.11

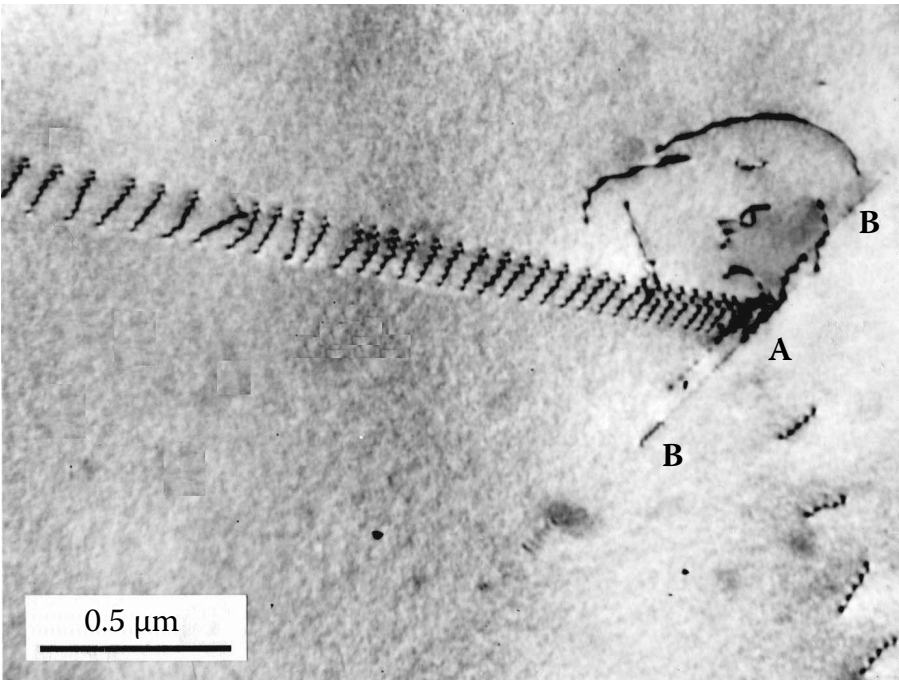


FIGURE 1.12

FIGURE 1.11 Frequently, grain boundaries contain Frank-Read sources.

Quenched austenitic nitrogen steel Fe-18Cr-14Mn-0,6N. Two grain boundary sources are visible in this micrograph: at the triple point A and at point B. Both sources emit dislocations into the grain on the left.

FIGURE 1.12 Dislocation-type barriers contain Frank-Read sources also. Such a barrier is shown at point A in the micrograph. The movement of the large dislocation pileup on the left has been stopped by a plane of high dislocation density intersecting the foil surface at B-B. The stress on the leading dislocation of the large pileup (it contains more than 40 dislocations) has activated a Frank-Read source. The emitted new dislocations glide in two directions—to the top and to the bottom of the micrograph.

Quenched austenitic nitrogen steel Fe-18Cr-14Mn-0,6N.

1.3 VACANCIES

The sites in a crystal lattice that are not occupied by atoms are called *vacancies*. The energy necessary for the formation of a vacancy is very small—about 1 eV—which explains the large concentration of these point defects under thermodynamic equilibrium conditions in metals and alloys. *Sources* for the formation of vacancies are the free surfaces of the crystals and the internal defects (dislocations, grain and subgrain boundaries, phase interfaces).

Vacancies in excess of their equilibrium concentration are generated most often during quenching from high temperatures, during plastic deformation, during ion bombardment, during bombardment by high-energy nuclear particles, or, in some intermetallic compounds, as a result of stoichiometric deviations. They are also produced by the oxidation of some metals, such as Mg, Ni, Cu, Zn, and Cd.

The equilibrium concentration of vacancies increases exponentially with temperature and is very high at the solution treatment temperature. When a solution-treated alloy is rapidly cooled (quenched) from a high temperature, the number of vacancies in the structure remains much higher than the lower-temperature equilibrium concentration. The quenched-in vacancies determine the level of the *vacancy supersaturation*. When the rate of cooling from the solution treatment temperature is lower, vacancies have sufficient time to reach *vacancy sinks* (sites in the lattice where they are annihilated), thus reducing the vacancy supersaturation.

Vacancies play a significant part in nonconservative dislocation movement (climb) and in the processes involving a diffusion transport of atoms—for example, polygonization and recovery. They thus play an important role during the processing of metals and alloys by plastic deformation at elevated temperatures; during solid solution heat treatment, annealing and aging; during creep; as well as in the processes of irradiation damage caused by high-energy particles.

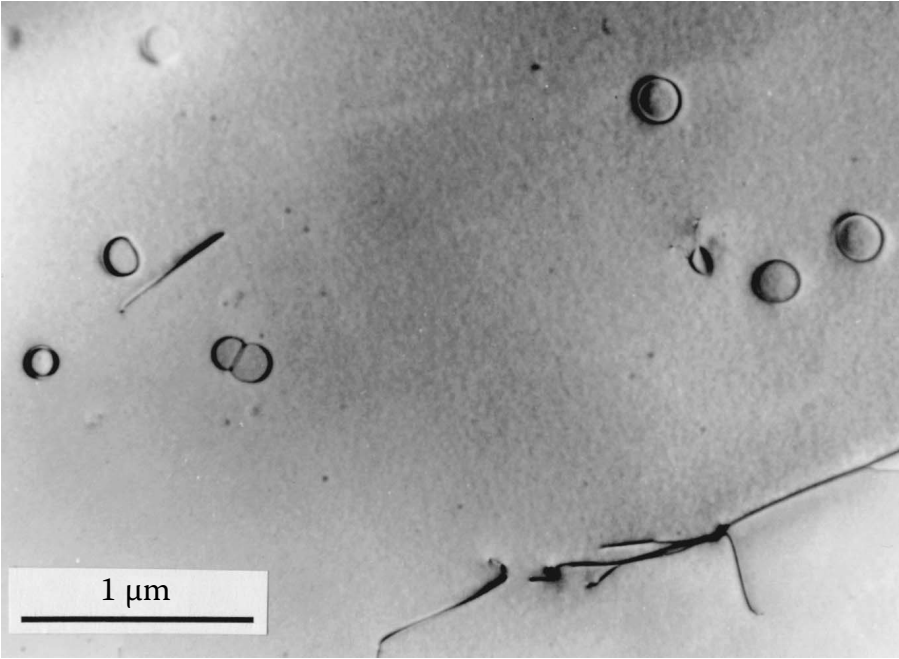


FIGURE 1.13

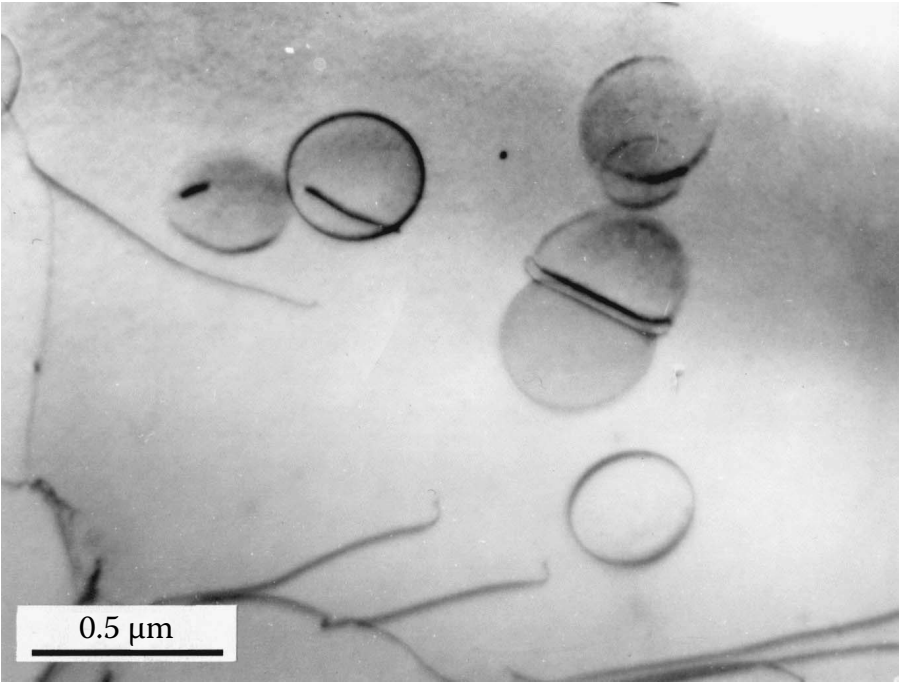


FIGURE 1.14

FIGURE 1.13 Zinc is characterized by its very pronounced ability to form vacancies through surface absorption processes. The vacancies combine into disc-shaped complexes in the basal plane of the hexagonal close packed (hcp) crystal lattice. The separate vacancies are invisible in the TEM because of their small size, but the presence of the *dislocation loops* that border the complexes make the vacancy discs visible.

Quenched zinc.

FIGURE 1.14 The number of vacancies in quenched zinc crystals is high. The bombardment of the thin metal foil by accelerated electrons during TEM observation introduces an additional number of point defects in the surface region and leads to the significant enlargement of the disc-shaped vacancy complexes. The process is similar to that of irradiation-induced damage occurring in industrial nuclear materials.

Quenched zinc after prolonged observation in the TEM. Compare this micrograph to Figure 1.13.

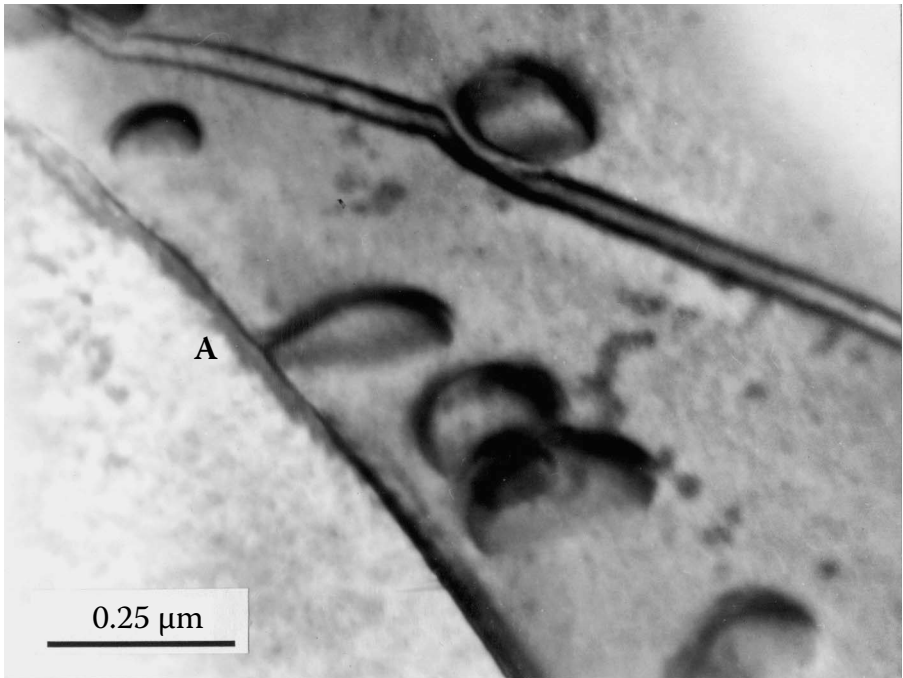


FIGURE 1.15 Besides acting as sources for vacancy formation, the imperfections in the crystal structure (mainly grain boundaries and dislocations) serve as sinks for their annihilation.

Quenched zinc after a prolonged holding in the evacuated column of the TEM. The grain boundary serves as a sink for the vacancies of the discs lying in close proximity to it. One can even see the “flowing out” of the vacancies to the boundary at point A.

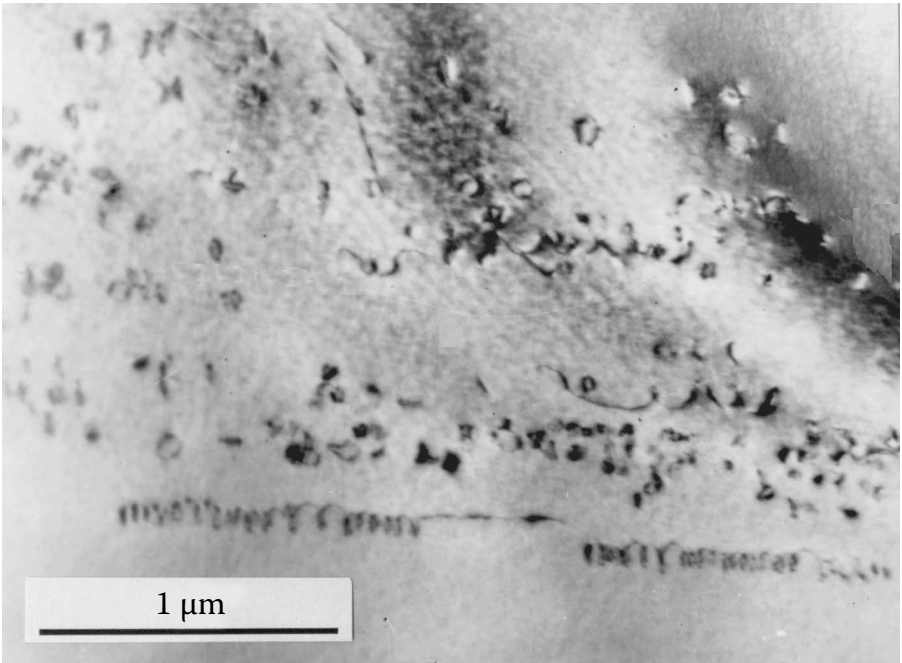


FIGURE 1.16 A high density of vacancy complexes (*vacancy loops*) is a typical component of the microstructure associated with quenched aluminum and aluminum alloys. This is due to their capacity to achieve a high vacancy supersaturation.

Quenched Al-4%Cu.

1.4 GRAIN AND SUBGRAIN BOUNDARIES

The *boundaries* of grains belong to the group of two-dimensional lattice imperfections. They are interfaces that separate regions in the interior of the material at which the crystal lattice changes orientation. The type and nature of the boundaries depend on the *misorientation angle* of the two adjoining grains and on the orientation of the interface boundary plane to them.

A *low-angle boundary (subboundary)* is a wall of tangled dislocations. The misorientation angle across the boundary depends on the number of dislocations that build up the dislocation wall. In a low-angle boundary, the angle does not exceed several degrees.

The misorientation angle of a *high-angle boundary* ranges from several degrees and can reach several tens of degrees. When the angle exceeds 10 to 15 degrees, the boundary is known as a *random high-angle boundary*. This boundary of several interatomic distances in width is in a high state of disorder compared to the matrix crystal structure—the atoms are out of their normal positions, the interatomic bonds are distorted, and consequently the boundary is associated with a higher energy.

There are many models (e.g., dislocation and disclination models, model of the coinciding knots) describing the structure of high-angle boundaries as a function of their misorientation angle, but no common theory has been accepted up to now.

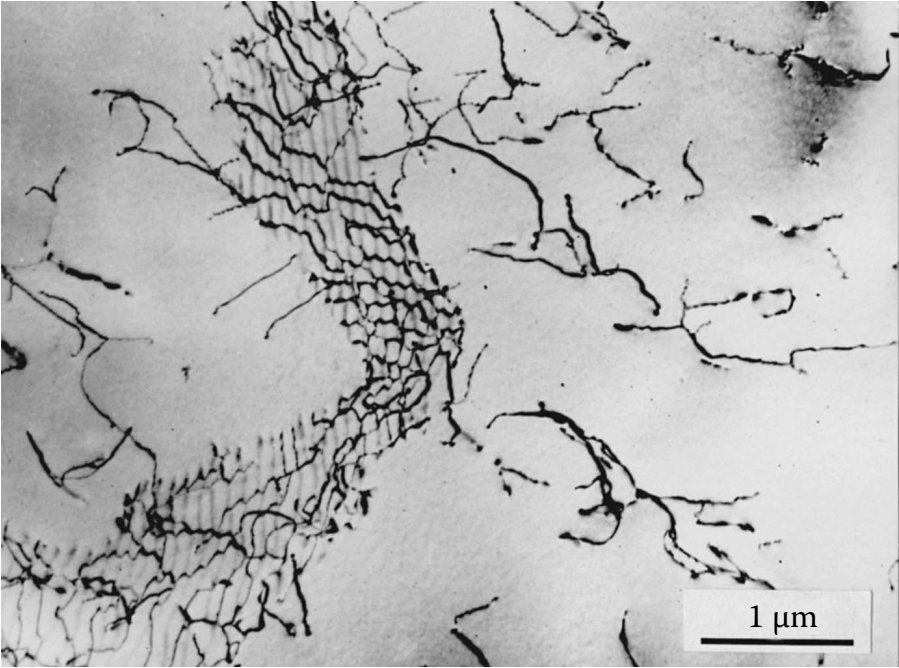


FIGURE 1.17

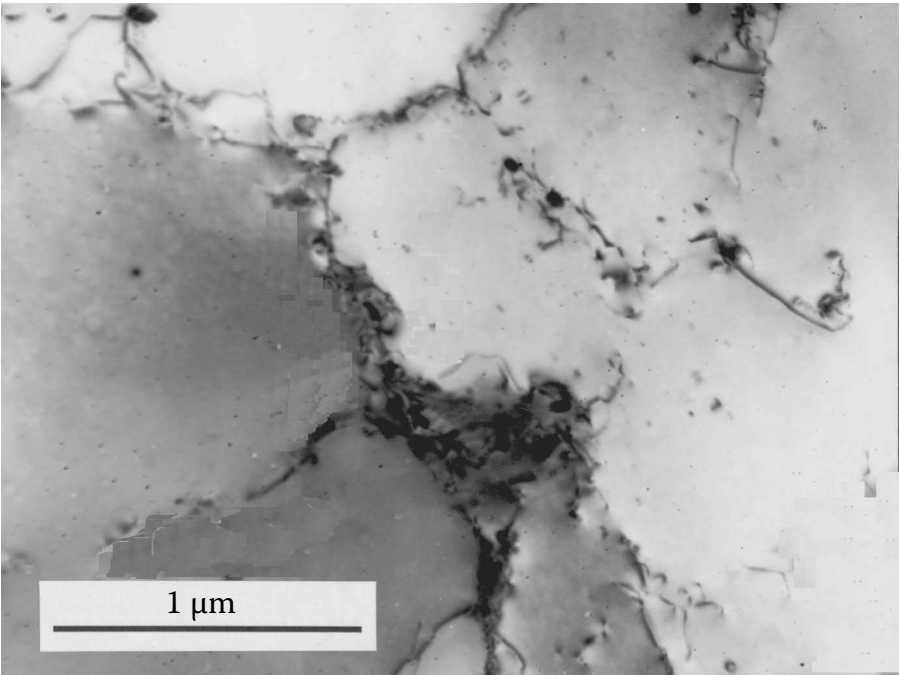


FIGURE 1.18

FIGURE 1.17 The dislocation complexes, called *tangles*, are produced by the interaction of moving dislocations. When the number of tangled dislocations becomes higher, they build a *dislocation wall*.

Annealed aluminum.

FIGURE 1.18 When reaching the dislocation wall, the moving free dislocations interact with the wall dislocations. This interaction results in more regular and stable dislocation complexes—*subboundaries* that separate *subgrains* regions of the crystal with relatively low dislocation density. Each dislocation participating in subboundary formation contributes to the misorientation of the adjacent subgrains by a magnitude depending on its Burgers vector. The amount of misorientation can be estimated in TEM images by the white-black contrast across the boundary: the stronger the contrast, the larger the misorientation angle.

Subgrains in aluminum.

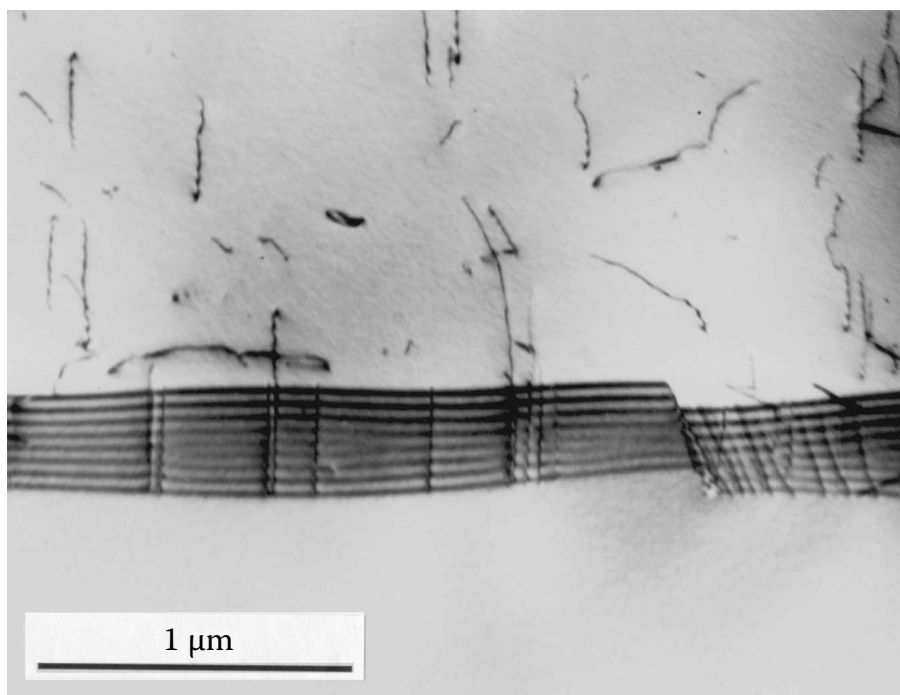


FIGURE 1.19 High-angle boundaries are planar defects in the crystal structure. When the plane of the boundary is inclined toward the foil surface, the boundary is seen in the TEM as a ribbon with parallel sides, the latter being the intersections of the grain boundary with the two foil surfaces. Depending on the TEM imaging conditions, parallel black-white fringes typical for the TEM images of all planar defects can be visible or invisible inside the ribbon.

Quenched austenitic nitrogen steel Fe-18Cr-14Mn-0.6N. Several parallel dislocations appear to be moving inside the boundary in the micrograph, but they are actually moving in a plane either above or below the boundary.

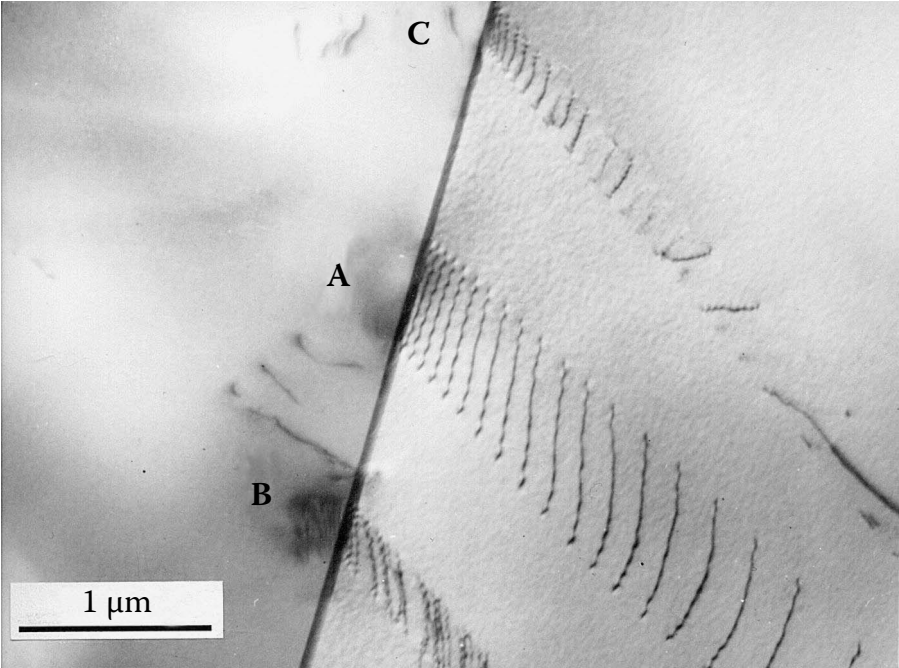


FIGURE 1.20

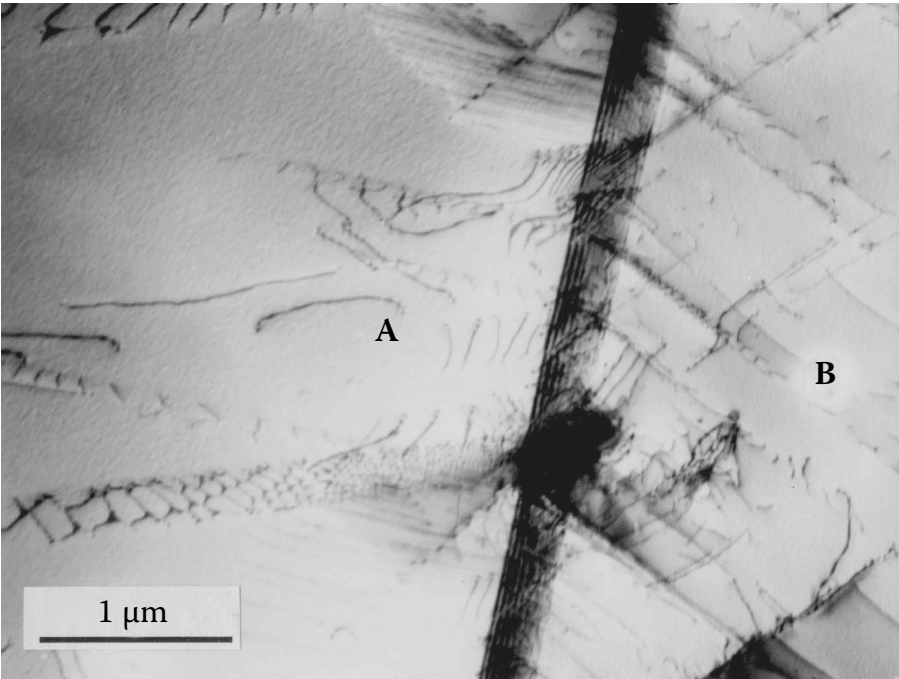


FIGURE 1.21

FIGURE 1.20 The high-angle boundaries are practically impenetrable barriers to moving dislocations.

Quenched austenitic nitrogen steel Fe-18Cr-14Mn-0,6N. The distance between the dislocations in the shown pileups when approaching the boundary diminishes. The stress on the leading dislocations, which can be estimated by the dark shadows at A, B, and C, is very high. When the stress reaches a critical level, the dislocation sources in the boundary will be activated to produce dislocations in the grain on the left.

FIGURE 1.21 The initiation of new dislocations in the next grain is the base mechanism for dislocation transfer across the boundaries during the plastic deformation.

Austenitic nitrogen steel Fe-18Cr-14Mn-0,6N after 2% cold deformation.

1.5 TWINS

A *twin* is a part of the crystal that has a crystal lattice identical to that of the base crystal (*matrix*), but with a crystallographic orientation that is a mirror image of the matrix orientation. Twins are always bordered by a pair of parallel *coherent boundaries* at which is realized the symmetrical tilt between the two twin-related crystals. Because of their extremely low energy due to the perfect fitting of boundary atoms into the lattices of both grains, the coherent boundaries are regarded as special high-angle grain boundaries. The other boundaries of the twin, which have a random orientation to the twinning plane and no geometric relationship to the matrix, are *incoherent boundaries*. The energy of these boundaries is much higher because the boundary atoms do not fit exactly into each grain lattice.

An important feature of twins is the fact that they never cross grain boundaries; they can terminate at grain or twin boundaries or inside the grain interior.

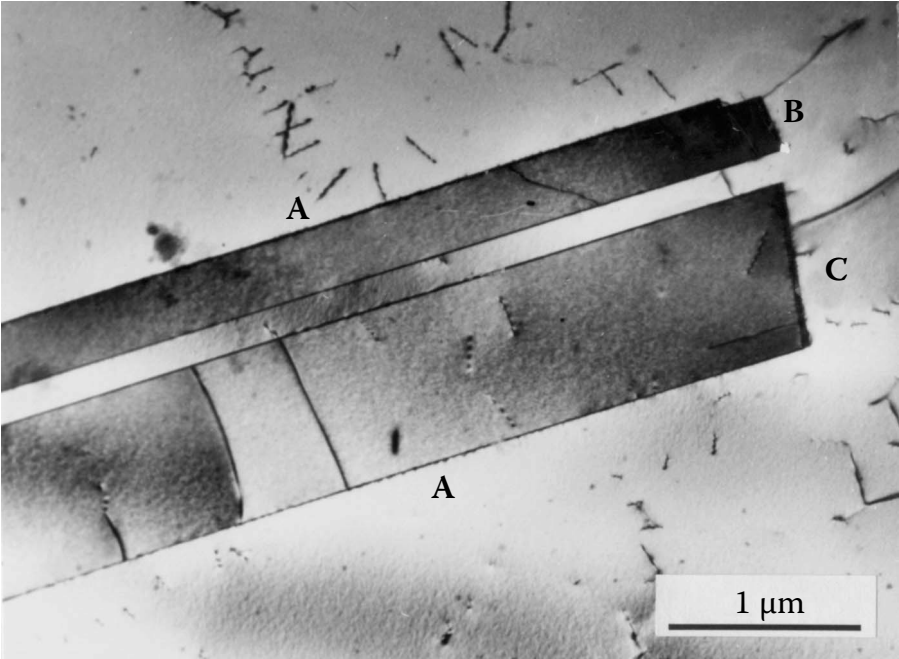


FIGURE 1.22

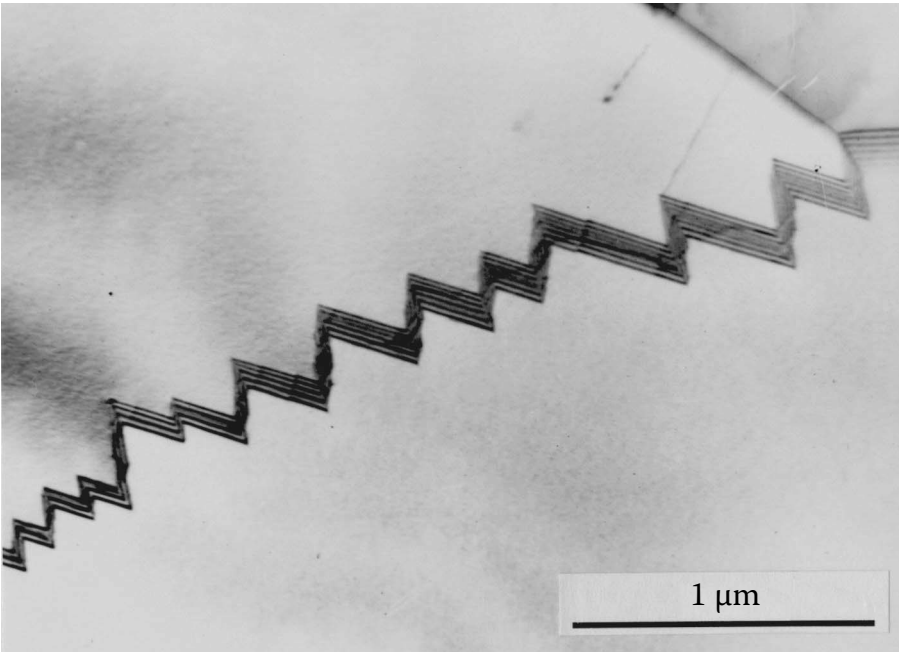


FIGURE 1.23

FIGURE 1.22 The orientation of the different twins in the same grain is usually identical.
Quenched austenitic nitrogen steel Fe-18Cr-14Mn-0,6N. The coherent boundaries (A) of the two twins shown in the micrograph are parallel. The incoherent boundaries (B and C) are randomly oriented to the matrix.

FIGURE 1.23 The grain boundary shown in the micrograph in Figure 1.23 contains coherent (parallel) and incoherent (nonparallel) steps.
Quenched austenitic nitrogen steel Fe-18Cr-14Mn-0,6N.

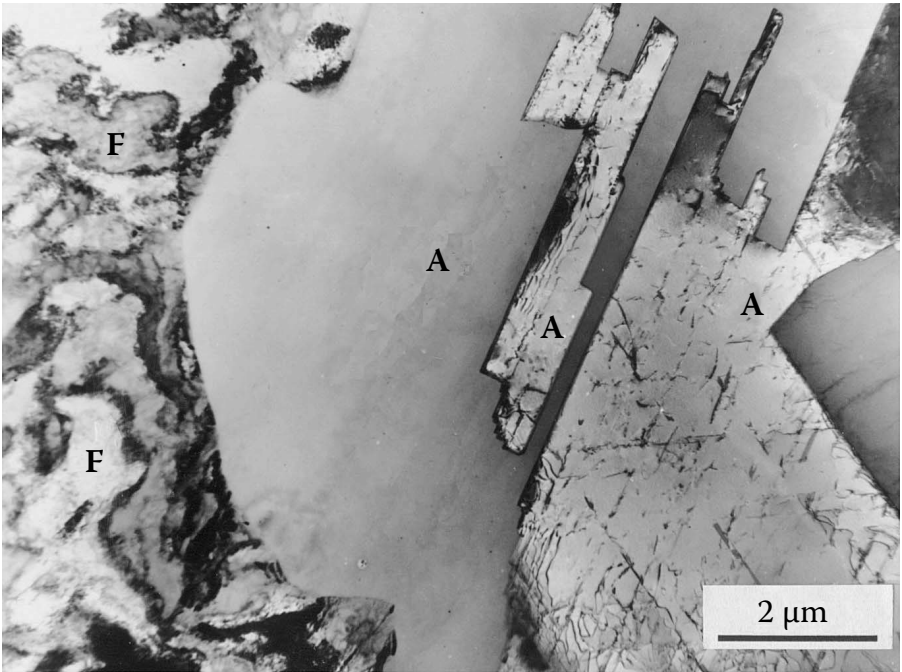


FIGURE 1.24 Twinning is a typical process occurring during recrystallization of low-SFE metals and alloys. These twins are called *annealing twins*. The presence of annealing twins in austenite helps to easily distinguish the austenite (A) from the ferrite (F) grains in a ferrite-austenite (duplex) steel.
Quenched ferrite-austenite steel.

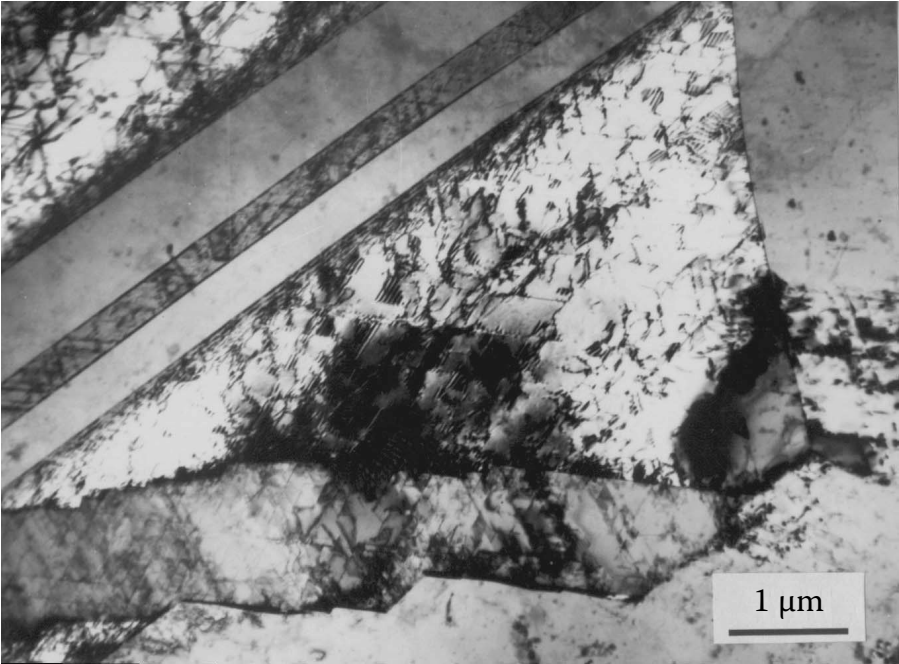


FIGURE 1.25

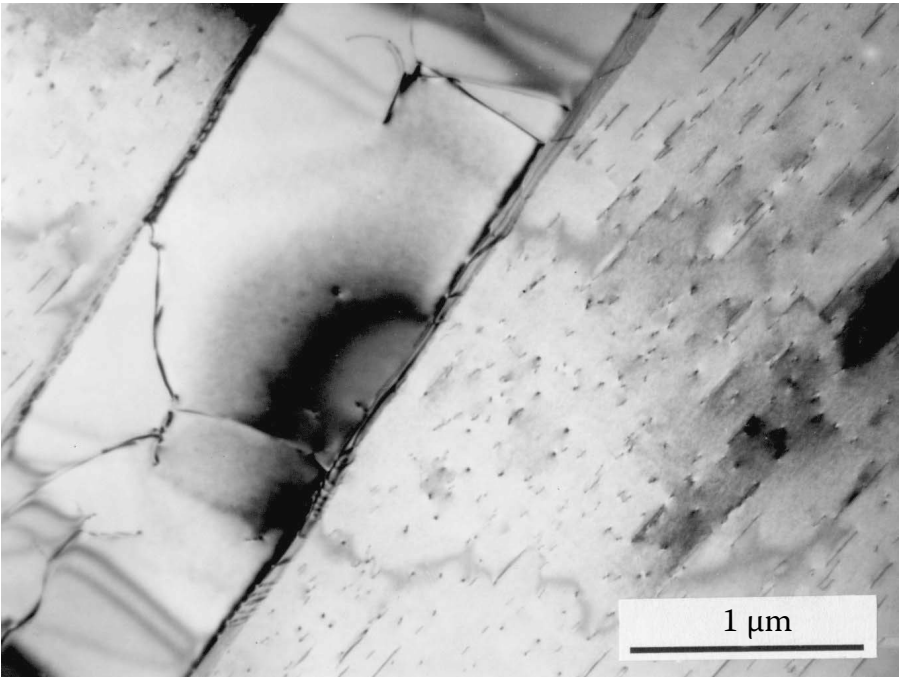


FIGURE 1.26

FIGURE 1.25 The frequency of twin formation helps to roughly assess the SFE value of metals and alloys. Twins can never be found in aluminum (high SFE), but they are a common feature in annealed copper (medium SFE) and are a compulsory element in recrystallized α_{Cu} -grains of brass (very low SFE).

Brass CuZn30.

FIGURE 1.26 *Mechanical twinning* is a principal deformation mechanism in metals having an hcp crystal lattice (Mg, Zn, Cd). This is due to the restricted slip, which can occur only in the basal plane. The twinning process in some of these metals is accompanied by energy release in the form of sound—for example, the well-known “metal yield” accompanying the bending of a tin rod due to the rapid formation of numerous twins.

Zinc after 2% deformation.

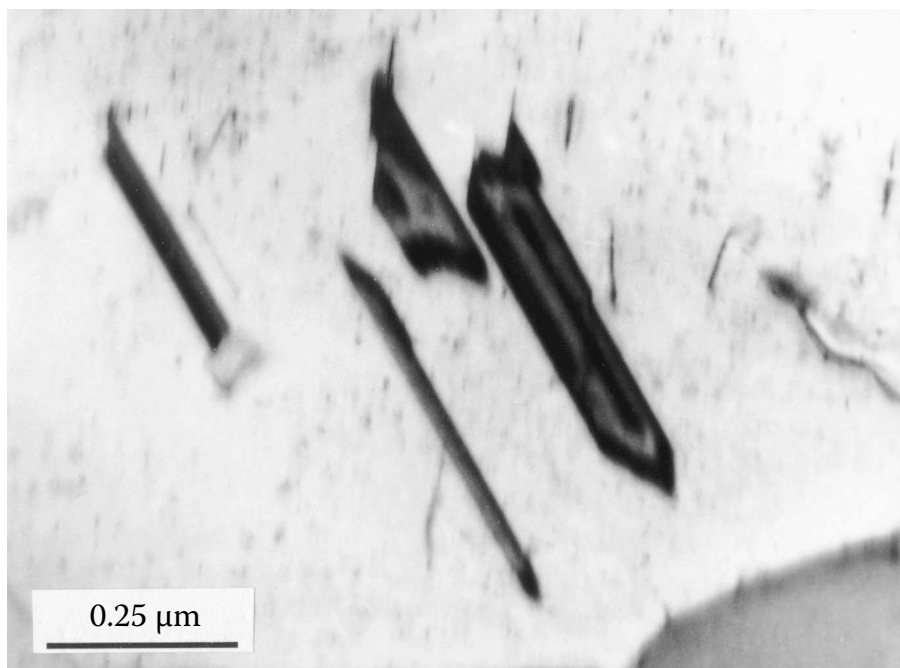


FIGURE 1.27 Twinning is a comparatively rare process in body-centered cubic (bcc) crystals. Only TEM observations can reveal the extremely narrow *microtwins* formed in some low-alloyed steels.

Ferritic steel containing 10%Mn, 2%Si, 0.1%V, and 0.1%N.

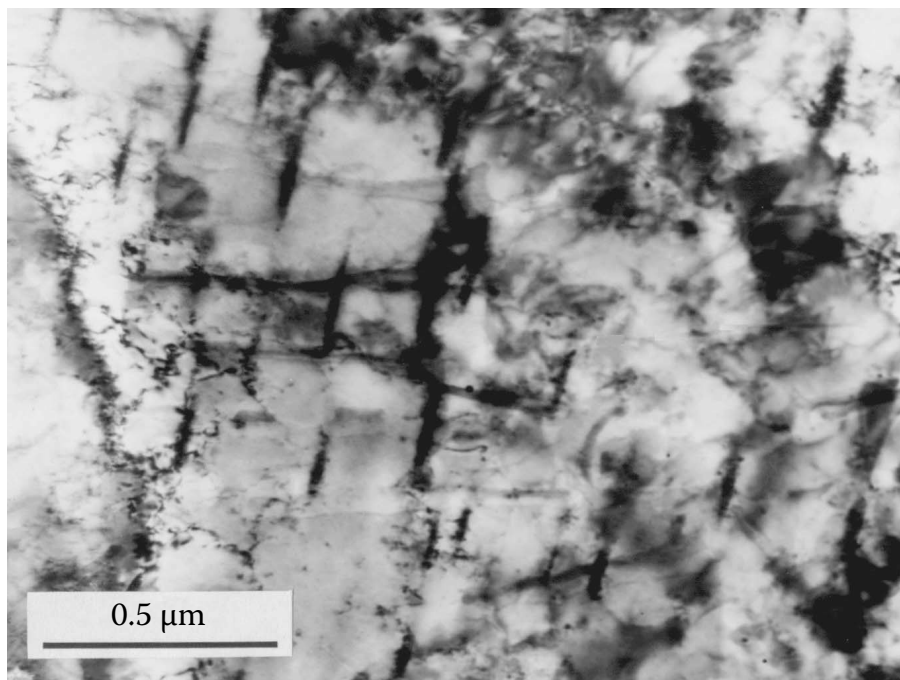


FIGURE 1.28 *Deformation twins* can be produced in bcc crystals by plastic deformation, especially through dynamic loading. These mechanical twins are usually very thin and are referred to as microtwins.

Ferritic steel containing 10%Mn, 2%Si, 0.1%V, and 0.1%N after high-velocity plastic deformation.