Electron Microscopy I
Lecture 08

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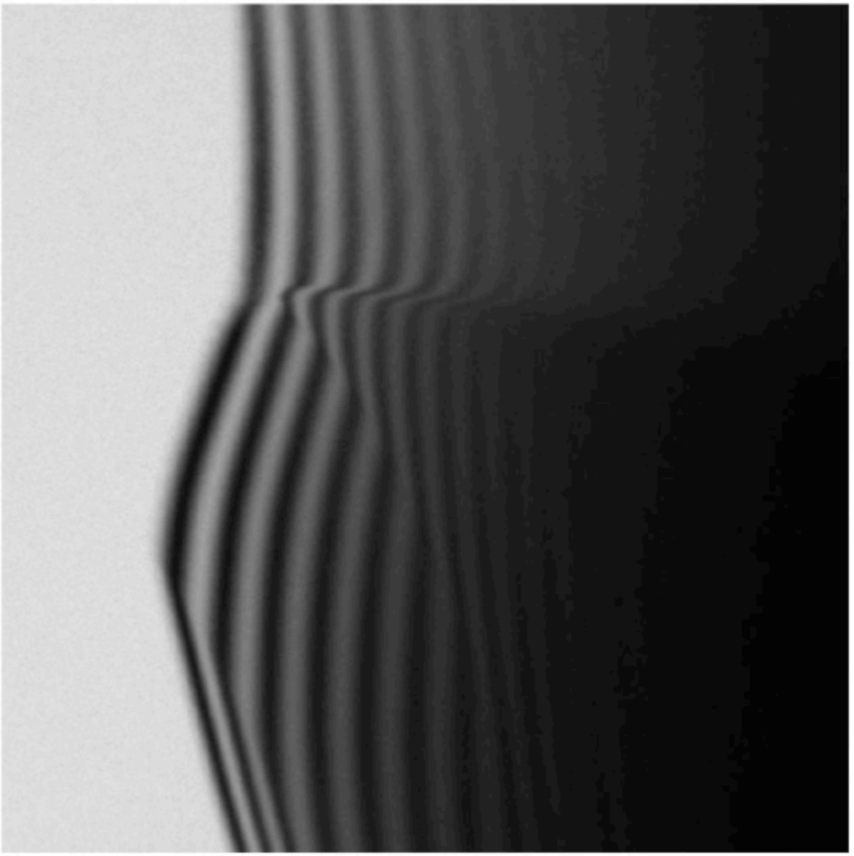
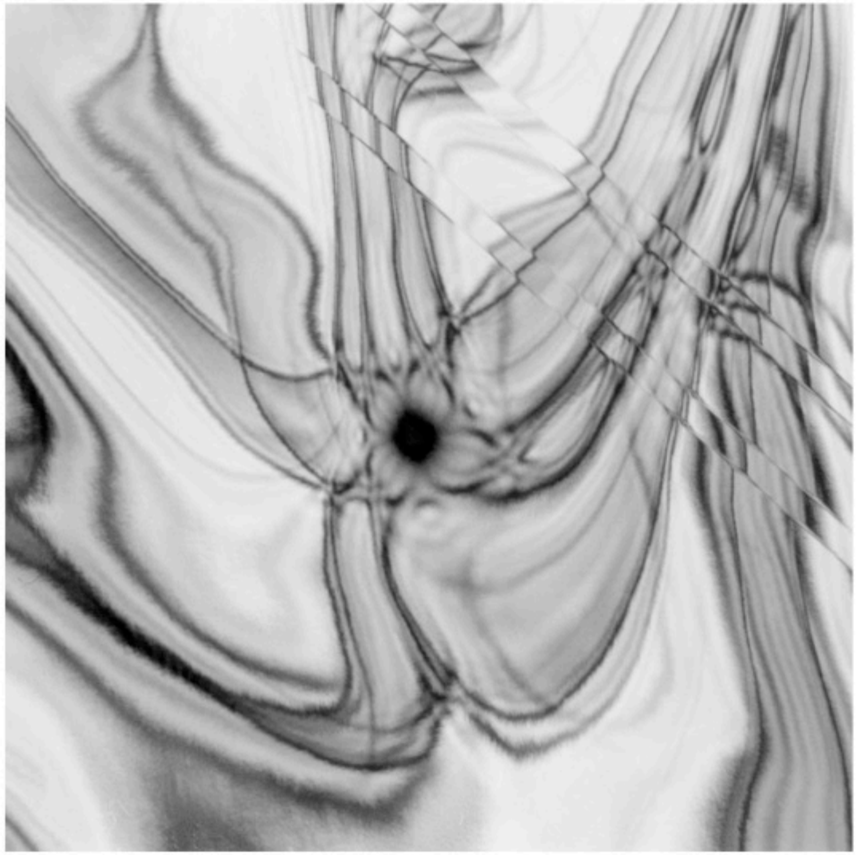
Electron microscopy I

4.3 Contrasts in perfect (single) crystals

Contrasts in the perfect crystal: thickness and bending contours



Thickness contours in Si crystal

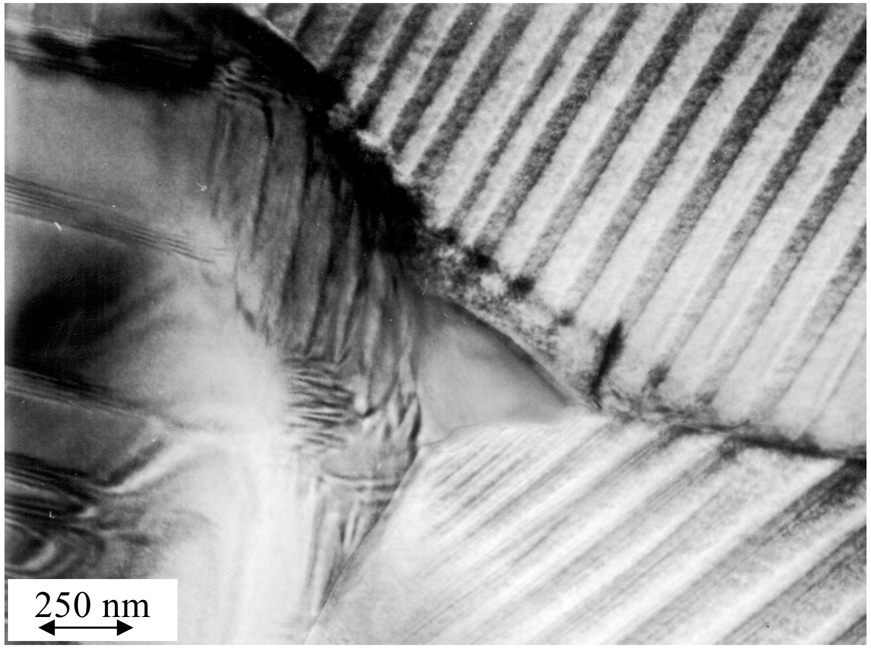


Bending contours in a Si crystal

Determination of the local specimen thickness using thickness contours

4.4 Contrasts in crystalline samples with lattice defects

TEM bright field image of a $BaTiO_3$ -functional ceramic: Grains with ferroelectric domains and reaction phase at the triple point



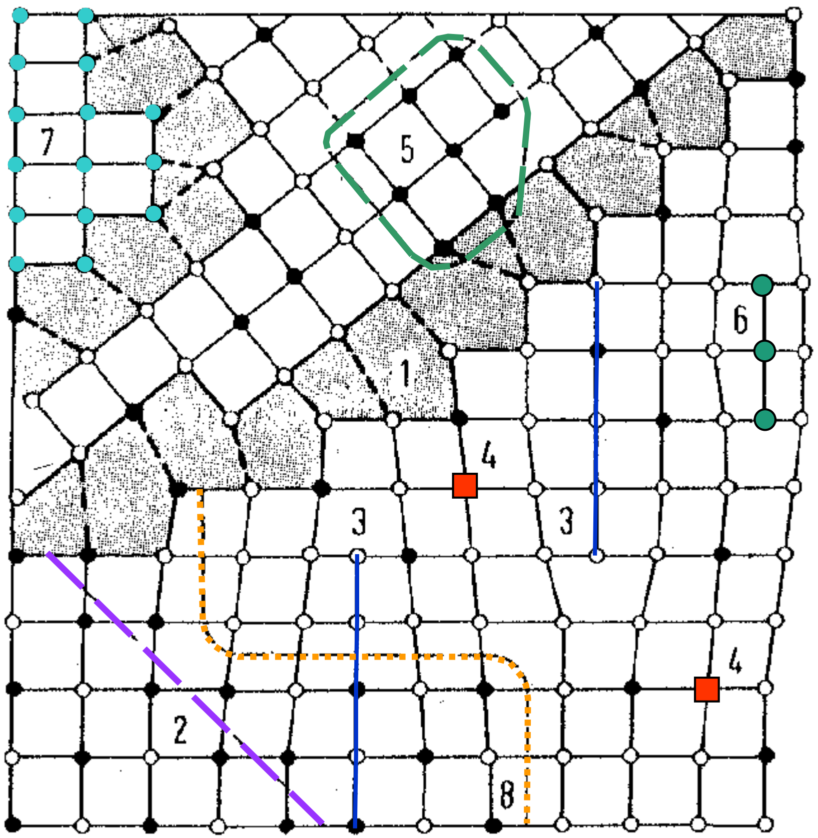
Solids/materials

- *Microstructure* - entirety of defects
- Defect structure strongly influenced by the (in)homogeneity of the chemical composition

<u>Possibilities for chemical composition:</u>

1. Chemically pure: only one element present (however, impurities are always present)
2. Alloy: two or more elements with a <u>spatially homogeneous</u> composition
3. alloy with <u>spatially inhomogeneous</u> composition (*matrix, precipitates*)

4.4 Contrasts in crystalline samples with lattice defects



Hornbogen, Skrotzki, "Werkstoffmikroskopie", p.2

Classification of defects

- Point defects (zero-dimensional) of a structural nature (vacancies, interstitial atoms) and chemical nature (impurities, dopants)
- Linear defects (dislocations)
- Two-dimensional (planar) defects, e.g. stacking faults (individual intercalated lattice planes), antiphase interfaces, grain boundaries
- with inhomogeneous chemical composition: Particles/particles/precipitates (three-dimensional defects), i.e. areas with a different chemical composition than their surroundings and often also a different crystal structure

Schematic representation of the structure of a material:
1. grain boundary 2. antiphase boundary 3. step dislocation 4. vacancy 5. coherent particle 6. partially coherent particle 7. non-coherent particle 8. boundary between ordered and disordered phase

4.4 Contrasts in crystalline samples with lattice defects

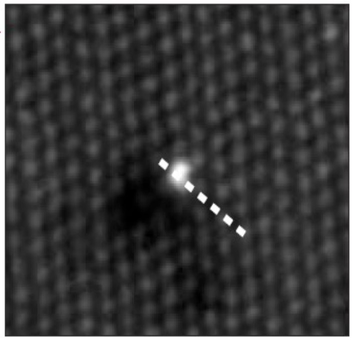
Silicon single crystal

Defects in crystals

- All crystals contain defects in different concentrations
- Extreme case: Si single crystal
  (1 - 2 m length, 40 cm diameter)

  only point defects (vacancies, interstitial atoms, impurity atoms, dopants), but no dislocations or 2-dimensional defects

<u>Point defects in crystals</u>
Cannot be imaged using standard TEM techniques
Exception: High-angle annular dark-field scanning TEM
(HAADF STEM)



4-5 erbium atoms in SiC

U. Kaiser et al., Nature Materials 2002

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Calculation of the scattering amplitude F in crystals with defects

- Defect characterization: Replace all vectors $\vec{r}_{pi}$ with $\vec{r}_{pi} + \vec{R}$ for $\overrightarrow{|R|}$ << lattice plane spacing.
- $\vec{R}$ indicates the displacement of an atom from its regular lattice position.
- Structure factor F_s in good approximation unaffected

⟶ Displacement vector $\vec{R}$ only affects lattice amplitude

$$F = F_S \sum_{\text{All Atoms in column}} \exp\left(2\pi i[\vec{g}+\vec{s}]\left[\vec{r}_{pi}+\vec{R}\right]\right)$$

$$F = F_S \int_0^t \exp\left(2\pi i\left[\vec{g}+\vec{s}\right]\left[\vec{r}+\vec{R}\right]\right) dz$$

$\vec{g} \cdot \vec{r} = integer$ ⟶ $\exp(2\pi i \vec{g}\vec{r}) = 1$ and $\vec{s}\vec{R} << \vec{g}\vec{R}$

$$F = F_S \int_0^t \exp\left(2\pi i\left[z s_z + \vec{R}\vec{g}\right]\right) dz$$

Special case: $\vec{g} \cdot \vec{R} = 0$ Defect shows no contrast if $\vec{g} \perp \vec{R}$

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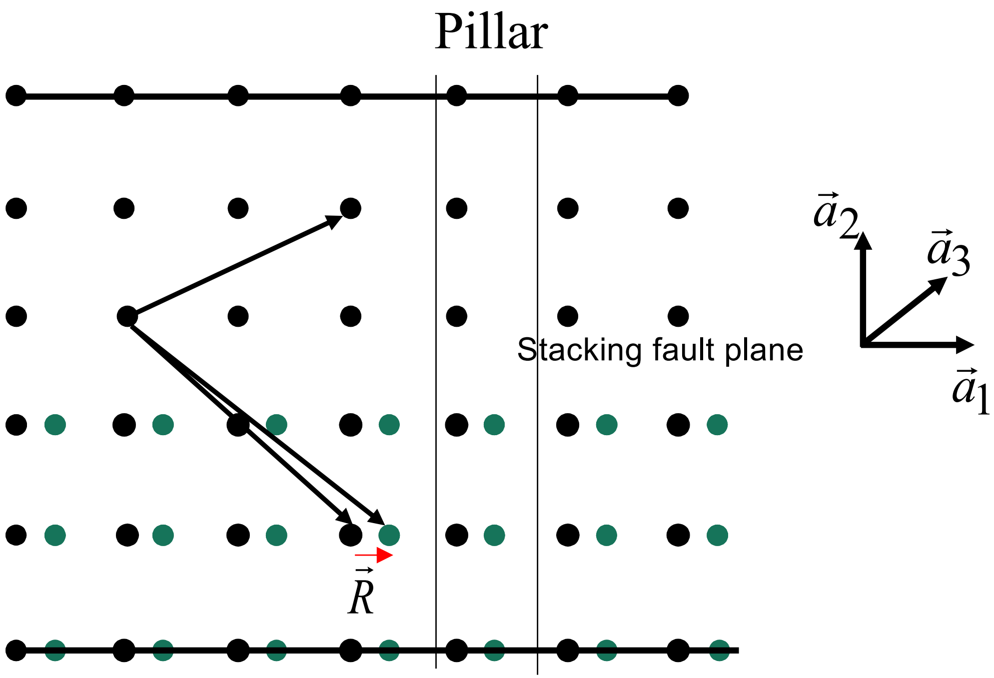
Stacking fault

Schematic representation of a stacking fault in the crystal lattice parallel to the sample surface.



Formal description: atoms no longer on their regular lattice sites, but shifted by vector $\vec{R}$

$$\vec{r}_{pi} \rightarrow \vec{r}_{pi} + \vec{R}$$

$|\vec{R}|$ < interatomic distances

$$\vec{r}_{pi} = u\vec{a}_1 + v\vec{a}_2 + w\vec{a}_3 \qquad \vec{R} = \frac{1}{2}\vec{a}_1$$

$$\vec{r}_{pi} + \vec{R} = \left(u + \frac{1}{2}\right)\vec{a}_1 + v\vec{a}_2 + w\vec{a}_3$$

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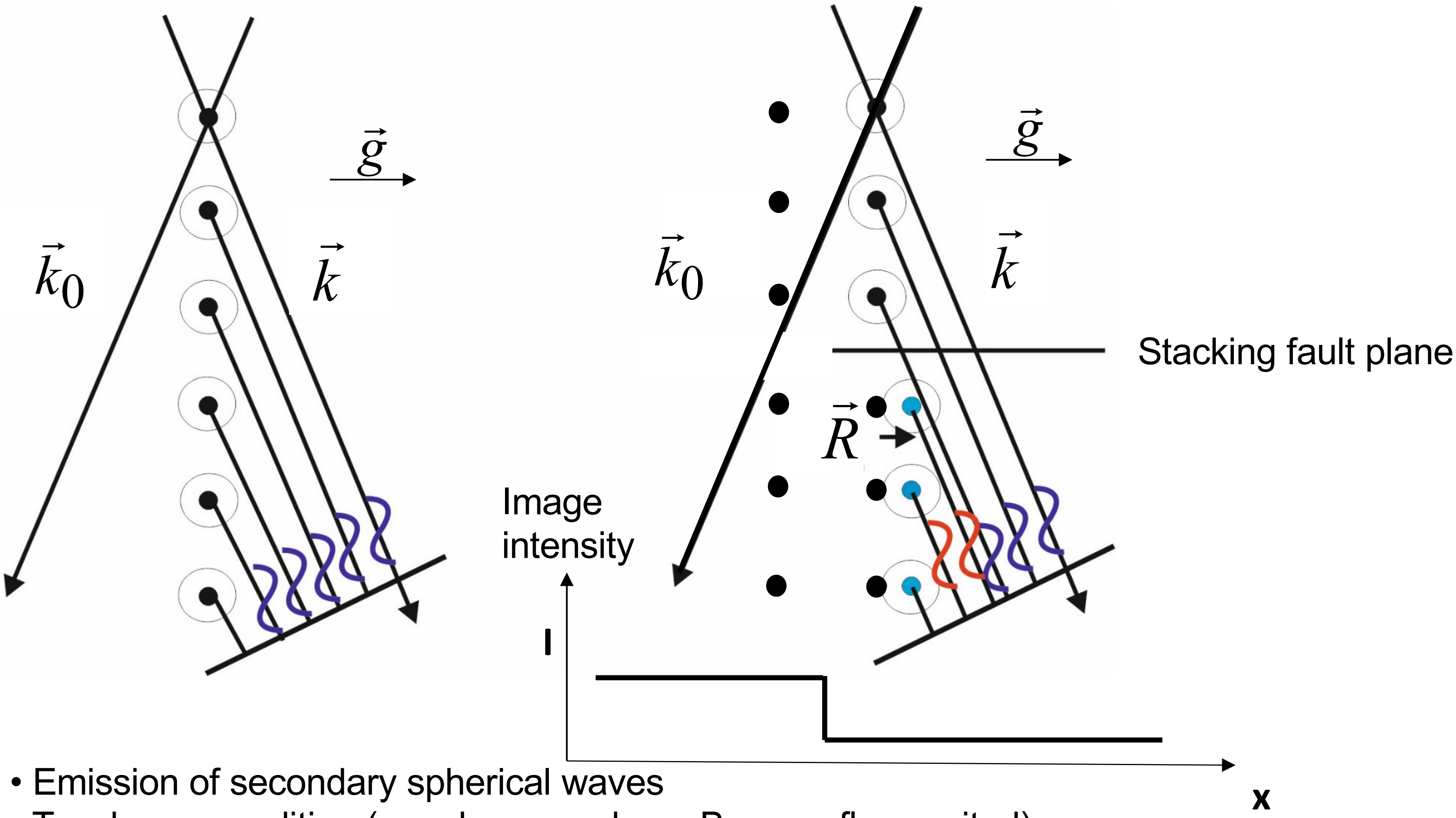
Contrast of a stacking fault (column approximation)



- Emission of secondary spherical waves
- Two-beam condition (zero beam and one Bragg reflex excited)
- Image intensity depends on the position of the stacking fault in the sample

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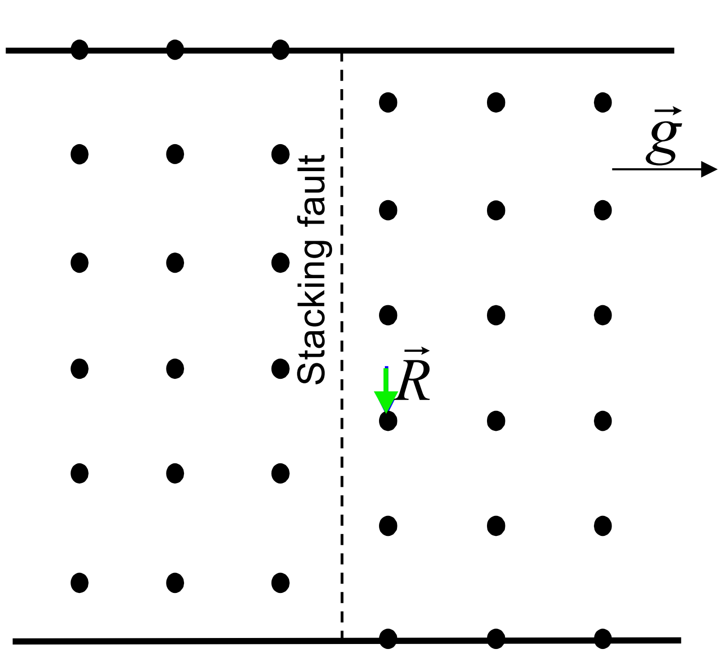
Contrast of a stacking fault

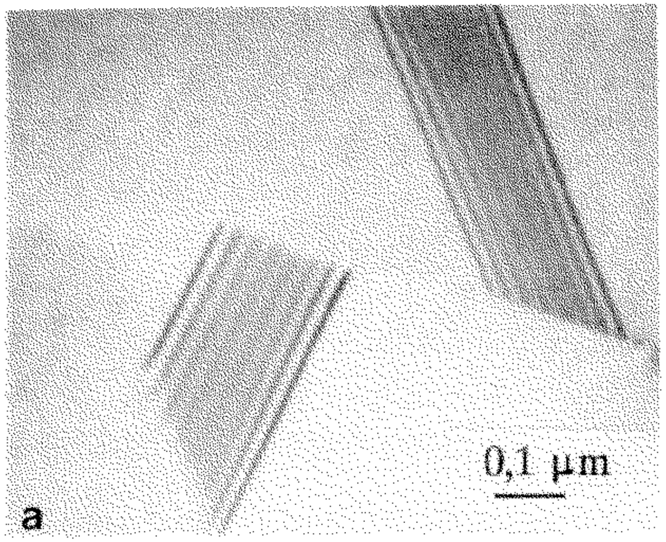


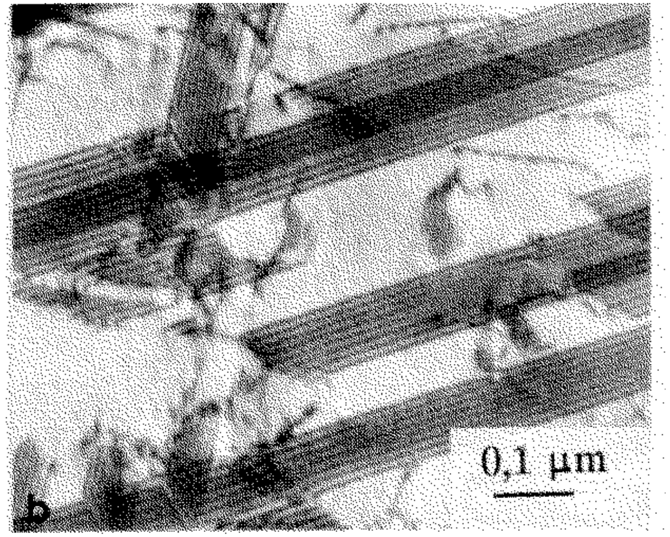
$$F = F_S \int_0^t \exp\left(2\pi i\left[z s_z + \vec{R}\vec{g}\right]\right) dz$$

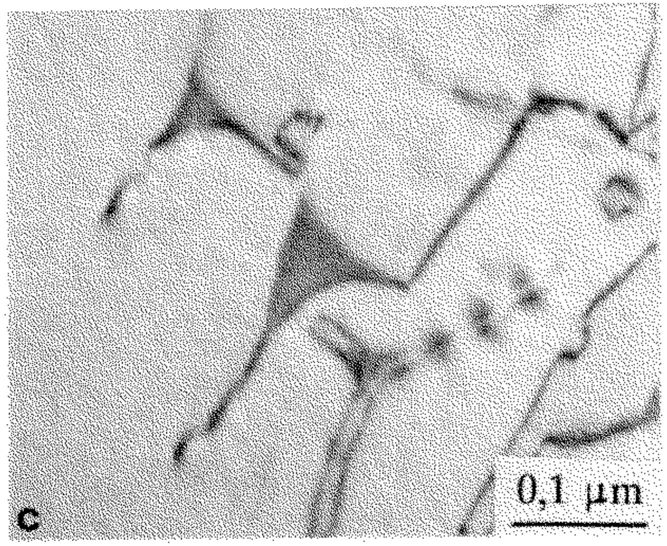
How do the lattice planes lie with the shown reciprocal lattice vector?

What contrast does a stacking fault create with the shown displacement vector and the shown reciprocal lattice vector?

4.4 Contrasts in crystalline samples with lattice defects







Brightfield TEM images of stacking faults

(E. Hornbogen, B. Skrotzki, "Werkstoffmikroskopie", Fig. 5.3)

a) $AlSi_{0.2}$ Sample with stacking fault running diagonally through the sample → Stripe contrast (identical intensity of a stacking fault at the same sample depth)

b) Multiple stacking faults on top of each other in steel

c) Stacking fault between partial dislocations with large splitting width

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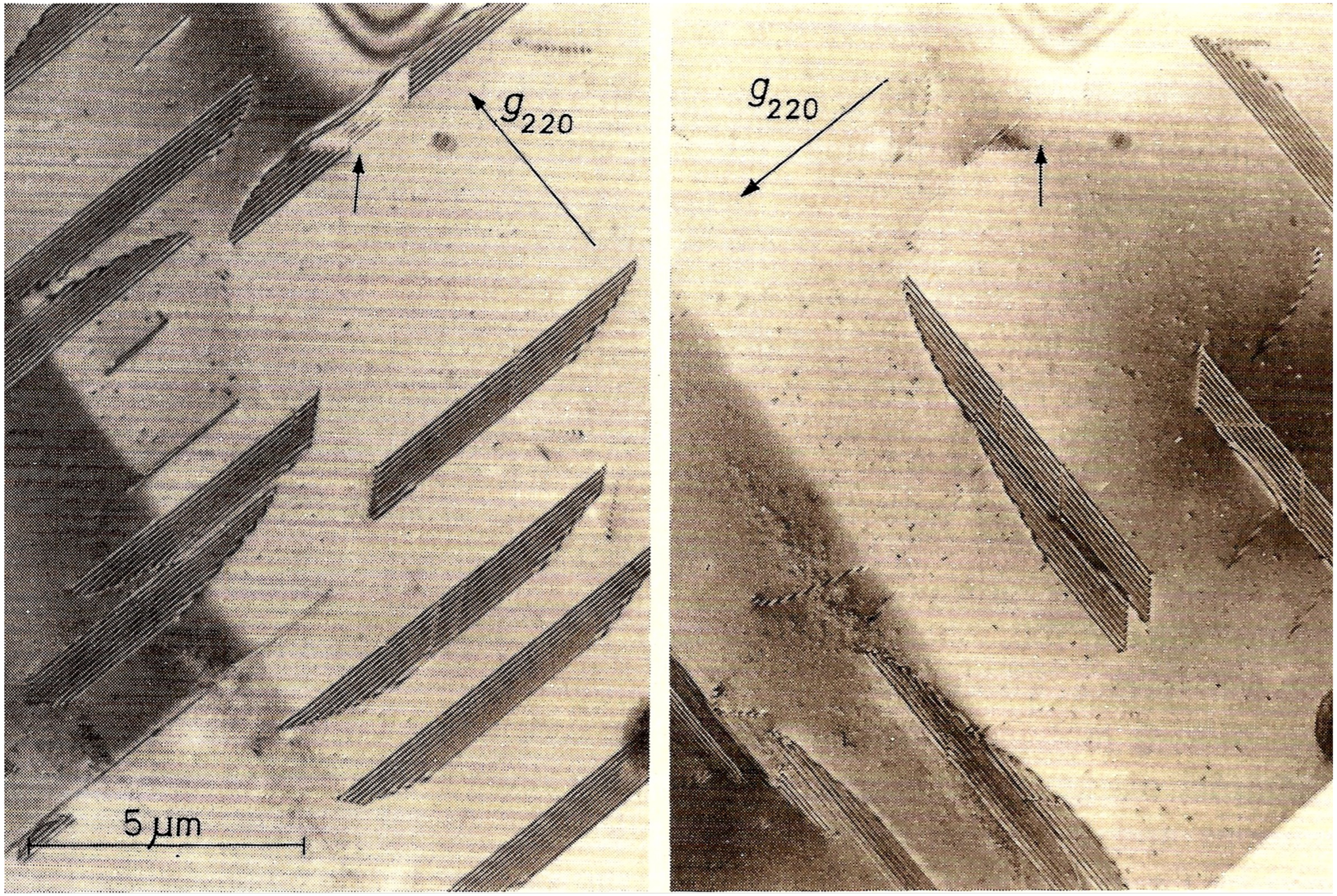
- **Criterion as a basis for the characterization of defects**



Stacking fault network in P-doped (001)Si with different -vectors
(Bethge, Heidenreich, "EM in Solid State Physics", p. 308)

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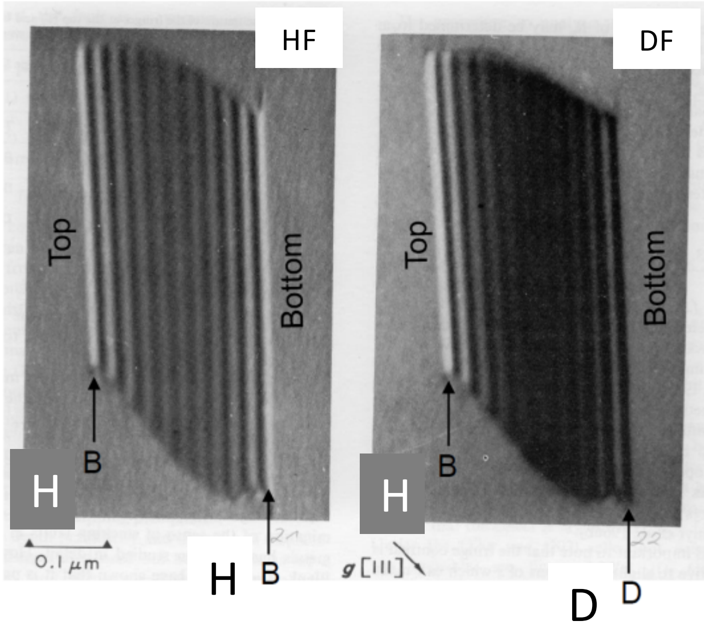
HF/DF - image contrast of stacking faults

<u>Characteristics of SF-kontrast</u>

1. BF mapping is symmetrical , DF mapping is asymmetrical
2. Absorption dampens the fringe contrast inside the film, see BF/ DF illustration
3. The first fringe shows the same contrast in the BF and DF
4. First fringe bright (H)
   First fringe dark (D) →
5. Stacking faults are invisible if $\vec{g} \cdot \vec{R} = 0$
6. As the sample thickness changes, more fringes are added from the center of the sample. The outer fringes remain unchanged.
7. In the Bragg condition s=0, the number n of dark fringes is given by: $(n-1) \cdot \xi_g$ = t (t is the sample thickness).
   But if s≠0 then the number of fringes does not easily correlate with the sample thickness.
8. The minimum value $\vec{g} \cdot \vec{R}$, at which the fringe contrast for a general stacking fault can still be observed is 0.02.



J.W.Edington: Pratical Electron Microscopy i.. ...erials Sciences, Fig. 3.30

(4) → $\vec{g} \cdot \vec{R} > 0$

(7) → $t \sim 9 \cdot \xi_{111}$

4.4 Contrasts in crystalline samples with lattice defects

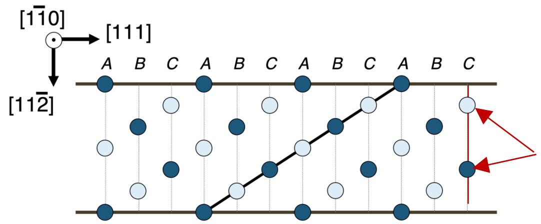
Stacking faults in fcc materials lie on the most densely packed {111} lattice planes with the stacking sequence ABCABC.

Different types of stacking faults can occur, e.g:

Extrinsic stacking faults: Plane added (B): ABCBABC

Intrinsic stacking faults: Plane removed (A): ABCBC

<110> Projection of an ordered fcc lattice: ABCABC

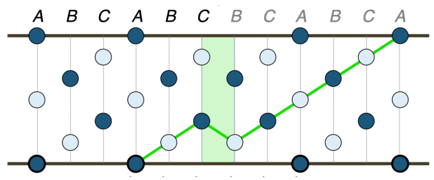


(atoms on the **C** stacking sequence of a (111) plane in the fcc crystal)

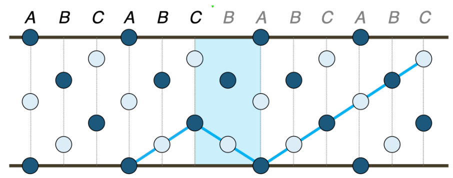
<u>Method for determining the stacking fault nature</u>:

von Gevers, Art and Amelinckx (1963) uses only dark-field images for fcc samples:

Intrinsic stacking faults



Extrinsic stacking faults

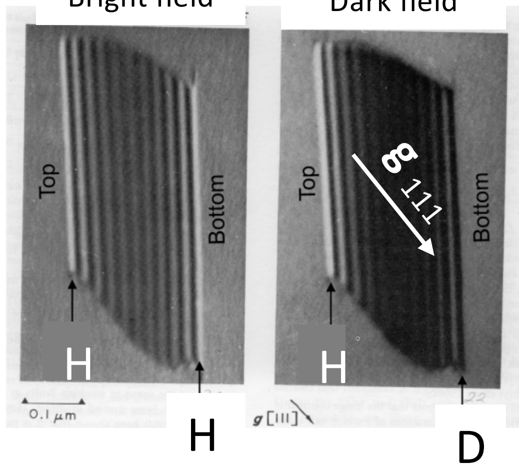


The vector is placed in the center of the stacking fault contours / strips:

points away from the bright stripe	**g={111}**, {220} or {400}	intrinsic SF
	g={200}, {222} or {440}	extrinsic SF
points to the bright stripe	**g={111}**, {220} or {400}	intrinsic SF
	g={200}, {222} or {440}	extrinsic SF

Caution: Sometimes the outer stripes are not clearly identifiable!

Bright field　　Dark field



intrinsic SF

J.W.Edington: Pratical Electron Microscopy in Materials Sciences, Fig. 3.30

4.4 Contrasts in crystalline samples with lattice defects

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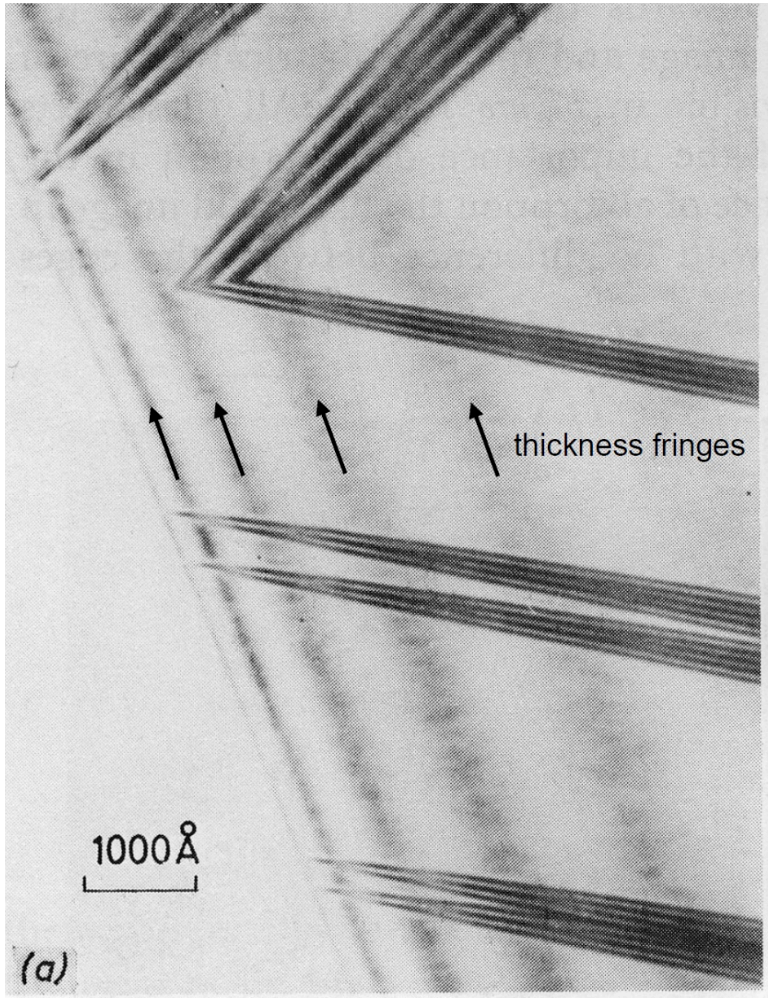
Influence of the sample thickness:

(6) As the sample thickness changes, more fringes are added from the center of the sample. The outer fringes remain unchanged.



Sir P. Hirsch et al. : Electron Microscopy of Thin Crystals Fig.: 10.11, a

Stacking fault in Cobalt.

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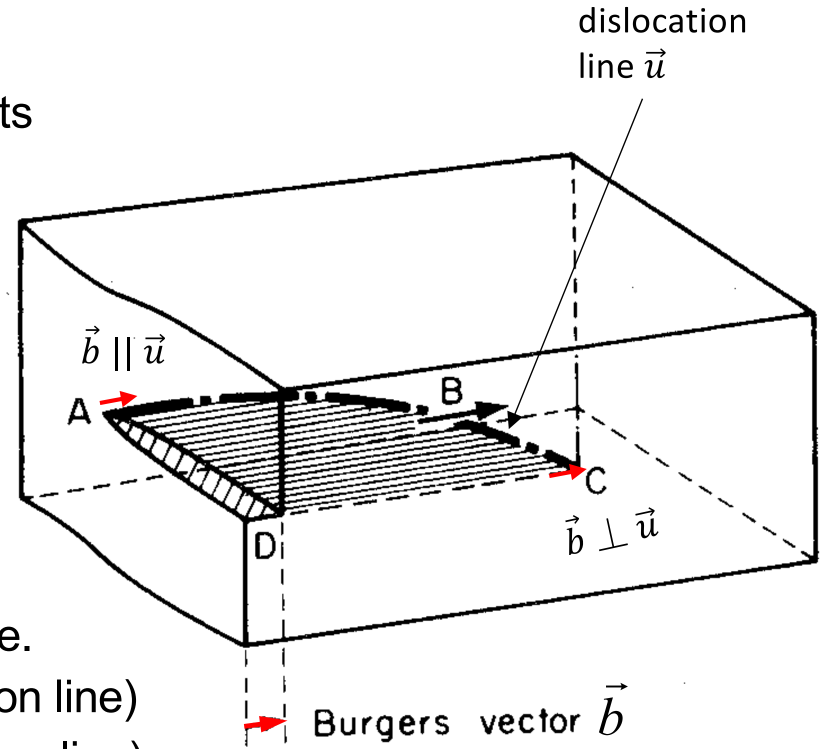
Dislocations

Supplementary literature:

D. Hull, Introduction to Dislocations, Pergamon Press
P.;. Anderson,J.P. Hirth, J.Lothe, Theory of dislocations
P. Haasen, Physical Metallurgy, Springer Verlag

- Plastic properties of a crystal are decisively determined by the properties of dislocations (density, *structure and mobility*)
  ⟶ Analysis of dislocations in the TEM
- Dislocations: one-dimensional, i.e. line-like defects
- Displacement of the upper half of a crystal compared to the lower one by one **vector** $\vec{b}$
  Dividing line = dislocation
- Displacement vector $\vec{b}$ (Burgers vector),
  Direction and length dependent on crystal structure and direction of the acting force
- Different types of dislocations, characterized by angle between Burgers vector and dislocation line.
  Borderline cases: *Edge dislocation* ($\vec{b} \perp$ Dislocation line)
  Screw dislocation ($\vec{b} \parallel$ Dislocation line)



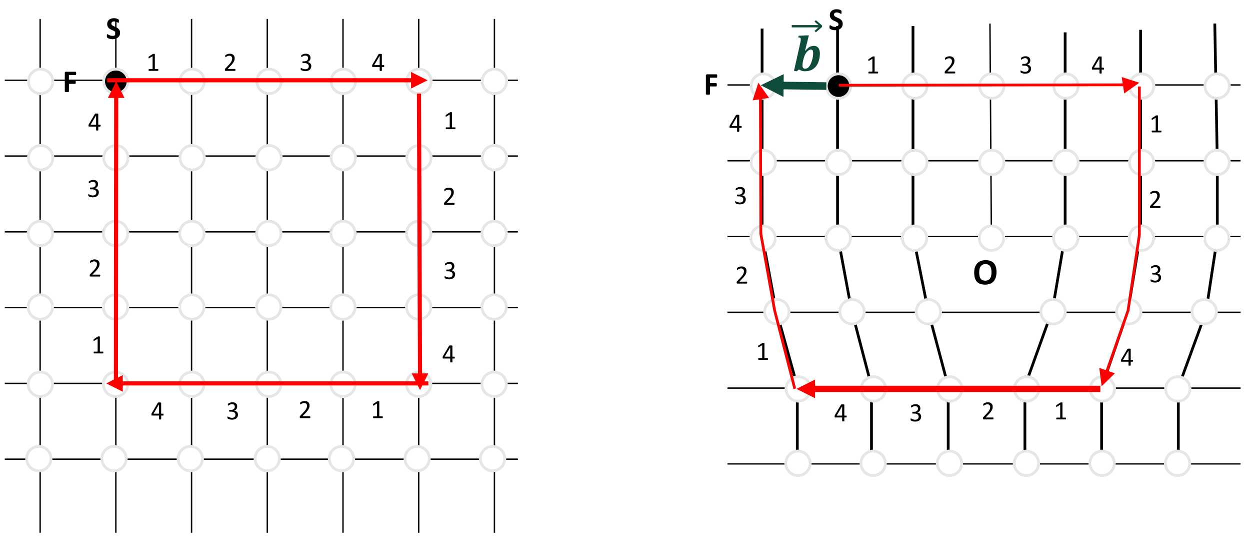
Determination of the Burgers vector

Burgers circuit - closed circuit in the perfect crystal is replicated and repeated in the crystal disturbed by the dislocation. The exact same number of steps in each direction is repeated.

↷ Due to the disruption of the crystal lattice, the start and end points of the circuit in the disrupted crystal are not identical.

- Burgers vector: Vector connecting the start and end points of the Burgers circuit.

Introduction of the convention: SF/RH = Start to Finish / Right Hand convention



Step offset with inserted lattice plane

<u>Perfect</u> dislocation: Burgers vector is lattice translation vector
in contrast to <u>partial dislocation</u>, which does not transfer the lattice into itself

4.4 Contrasts in crystalline samples with lattice defects

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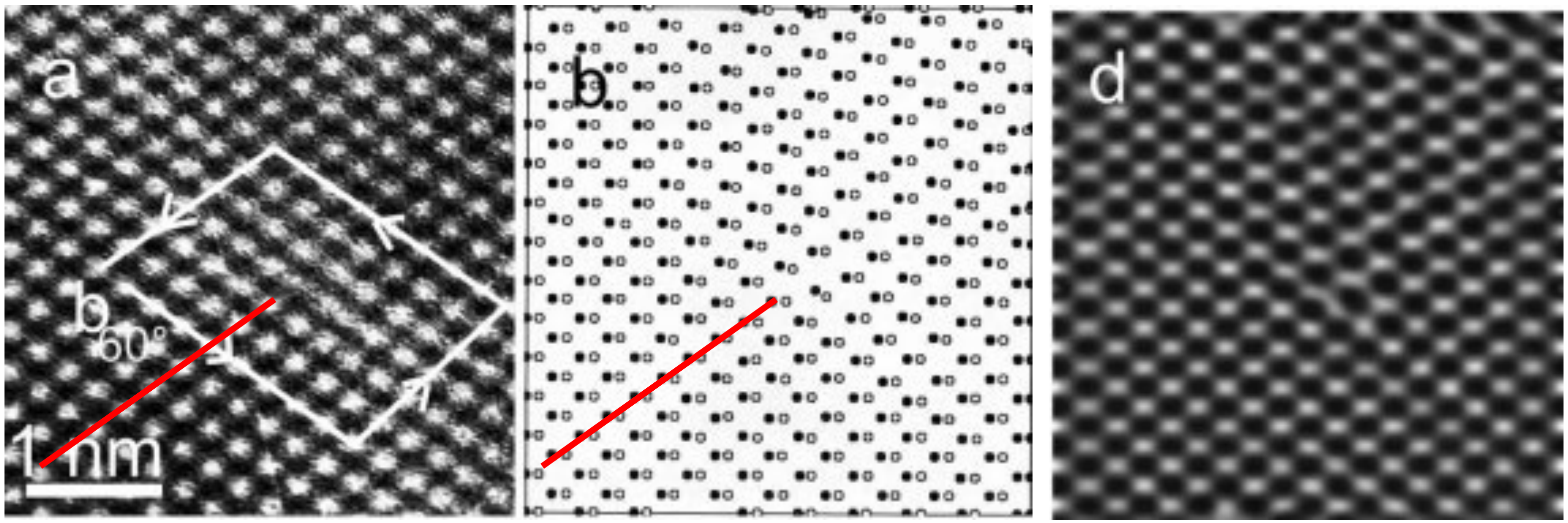
Aim of dislocation analysis with *conventional* TEM

- Determination of: 1. Burgers vector (direction and length)

  2. line direction of the offset and thus the sliding plane
  (*dislocation line direction and Burgers vector result in dislocation type and sliding plane*)

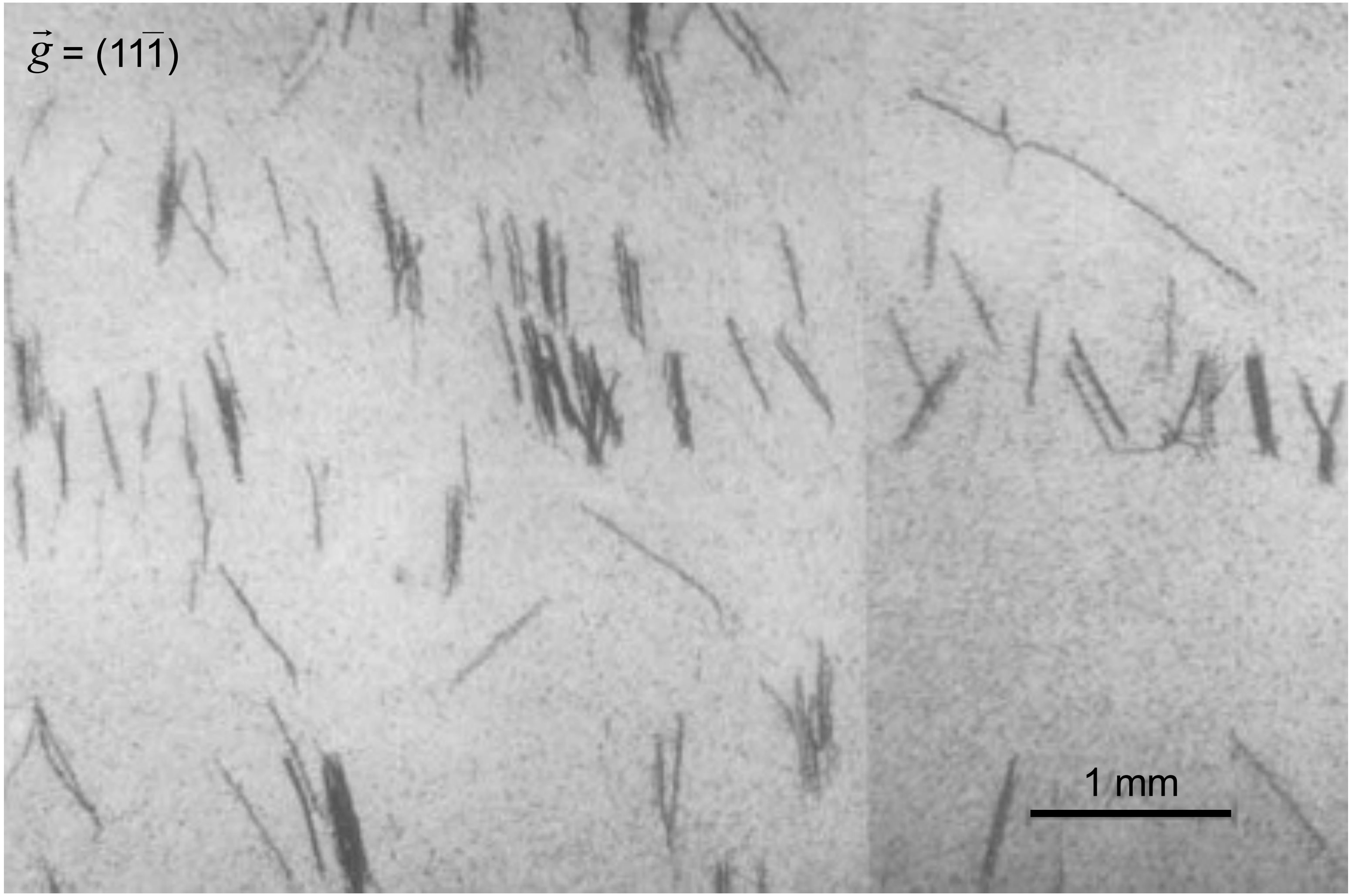
  3. dislocation density (see e.g. U. Essmann, Phys. Stat. Sol. **17**, 725 (1966))
- No analysis of the atomic arrangement around the dislocation nucleus → High-resolution TEM



Experimental image | Model | Image simulation

Nistor et al, HRTEM studies of dislocations in cubic BN, phys. Stat. sol. (a) 201, 2578 (2004)

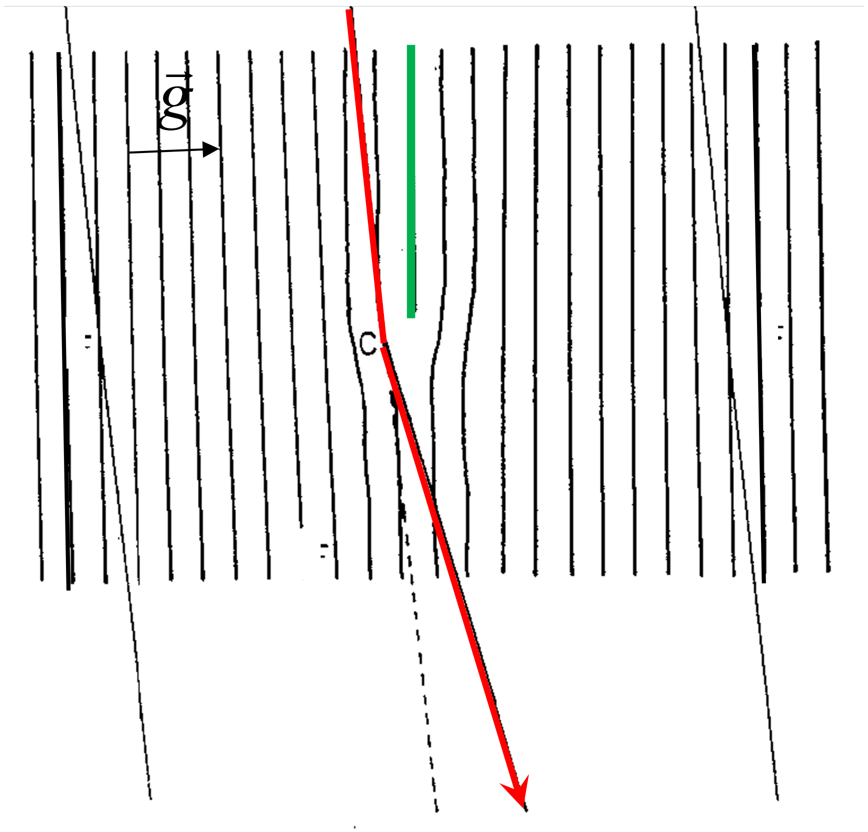
4.4 Contrasts in crystalline samples with lattice defects



TEM bright field image of dislocations lying obliquely in a copper single crystal.

U. Essmann, phys. stat. sol. 17, 725 (1966)

Contrasts of dislocations: simple qualitative explanation



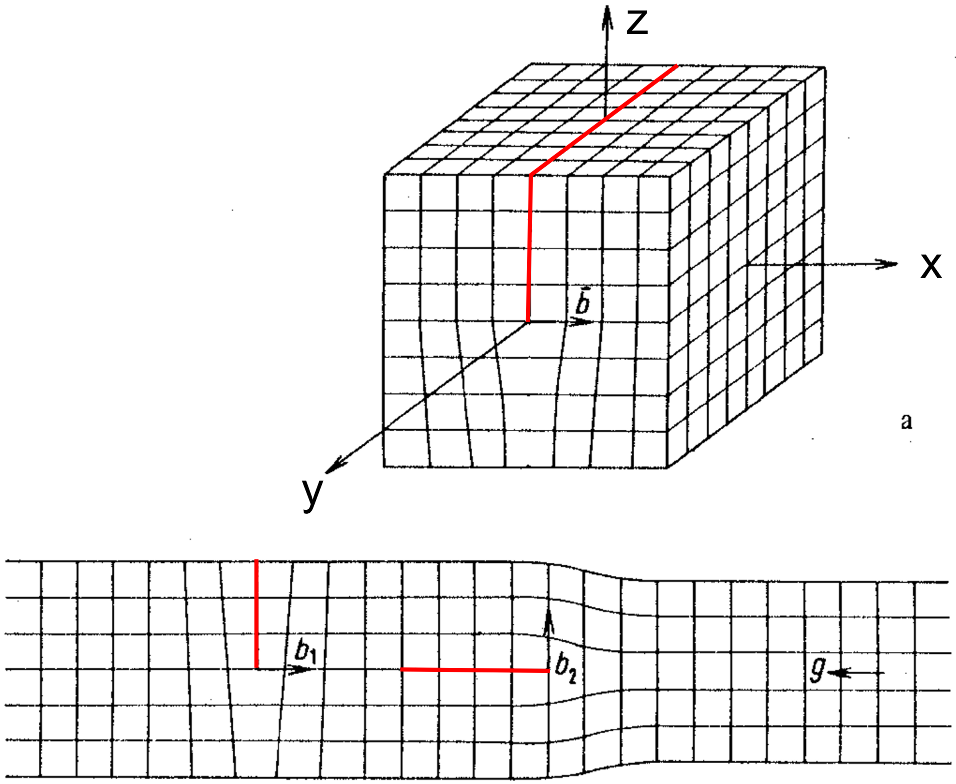
Goodhew, Humphreys, Electron microscopy, Fig. 4.23

- Bending of the lattice planes in the vicinity of the inserted half plane (*Displacement line points vertically out of the image plane*)
- undisturbed crystal is oriented in such a way that Bragg's condition is approximately fulfilled (*small excitation error* s_z) for the lattice planes shown with $\vec{g}$
- by curving the lattice planes around the dislocation core

  → exact fulfillment of the Bragg Condition local at position C

  → strong inflection - Dark dislocation contrast

$\vec{g}\cdot\vec{R}=0$ - **Invisibility criterion for dislocations** → $\vec{g}\cdot\vec{b}=0$



Hornbogen, Skrotzki, "Werkstoffmikroskopie", p.105

Example: Step dislocation in a cubic primitive lattice

- with the *diffraction vector* $\vec{g}$ only visibility of the dislocation with Burgers vector $\vec{b}_1$
- No curvature of the grid planes used for mapping when offset with $\vec{b}_2$

  ⟶ no contrast

- Unambiguous determination of the Burgers vector (*simple crystal systems*)

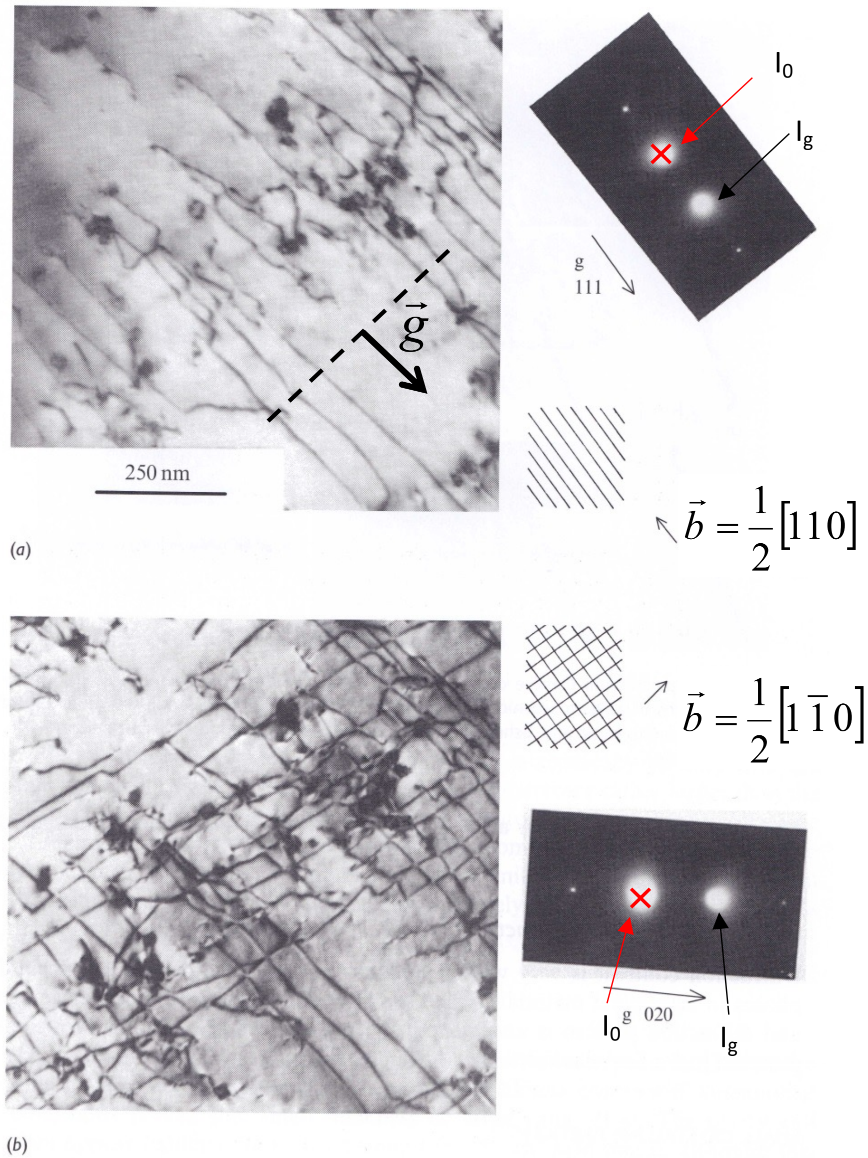
  ⟶ at least two extinction conditions

$$\vec{b}=\vec{g}_1\times\vec{g}_2$$

4.4 Contrasts in crystalline samples with lattice defects

Bright field images of a dislocation network

Diffraction images from the applied two-beam condition



Nb-stabilized steel sample

face-centered cubic structure:

a/2<110>-type Burgers vectors on {111} planes

Bottom: $\vec{g}$ = (020)
complete dislocation network,
Two vertical oriented sets of multiple dislocations

Top: $\vec{g}$ = (111)
a set of dislocation extinct

$\rightarrow \quad \vec{g} \cdot \vec{b} = 0$

further extinction with $\vec{g}$ = (002)
(*not shown*)

$\rightarrow \vec{b} = \frac{a}{2}[1\bar{1}0]$

P. J. Goodhew, J. Humphreys, R. Beanland, "Electron Microscopy and Analysis, Fig. 4.29

Displacement analysis

- Application of the $\vec{g} \cdot \vec{b} = 0$ criterion with two extinctions for $\vec{g}_1$ and $\vec{g}_2$ to determine the direction of $\vec{b} = \vec{g}_1 \times \vec{g}_2$
  (determination of the length more complex, see e.g. Morniroli et al., Phil. Mag. 86, 4883 (2006))

- Complete/unambiguous extinction only if the step component of the dislocation $\vec{b}_e$ and the line direction $\vec{u}$ are also perpendicular to $\vec{g}$, as the dislocation has a complex displacement field $\vec{R}$ in reality ($\vec{b}$ must be replaced by $\vec{R}$)

$$\vec{R}_{(r,\varphi)} = \frac{1}{2\pi}\left\{\vec{b}\varphi + \vec{b}_e \frac{\sin 2\varphi}{4(1-\nu)} + \vec{b} \times \vec{u}\left(\frac{1-2\nu}{2(1-\nu)} \ln\frac{r}{r_o} + \frac{\cos 2\varphi}{4(1-\nu)}\right)\right\}$$

- In the case of complex crystal structures and elastically highly anisotropic materials, complete/unambiguous extinction is also often not observed

- Calculation of dislocation contrasts using the displacement field $\vec{R}(r,\varphi)$ with the aid of the column approximation most successful but at the same time more complex

  $\rightarrow$ Simulation of different dislocation configurations and comparison of simulated Image contrasts with experimental images
  (see e.g. M. De Graef, Introduction to conventional transmission electron microscopy, Cambridge University Press)

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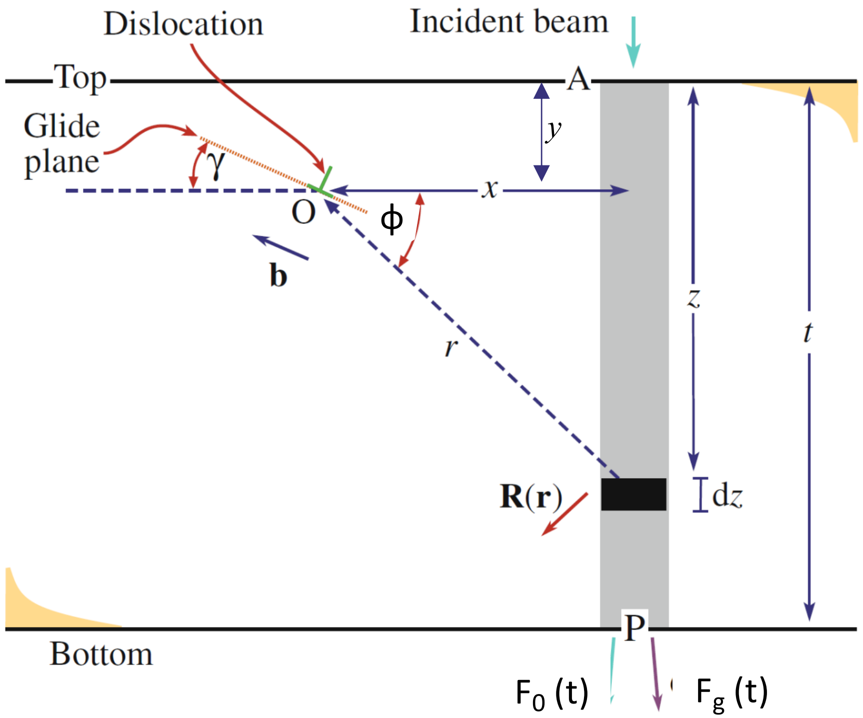
Simulation of dislocation contrasts (column approximation): "Fine structure" of dislocation contrasts



Carter, Williams, Transmission Electron Microscopy, Fig.: 26.2.

- Transfer to depth *y*, *x* = lateral distance of the column from the dislocation core
- Displacement field $\vec{R}$ as a function of the distance *r* from the dislocation kernel and the angle φ for any dislocation (from elasticity theory)
- Calculation of F_g and F_0 by kinematic or dynamic diffraction theory

$$\vec{R}_{(r,\varphi)} = \frac{1}{2\pi}\left\{\vec{b}\,\varphi + \vec{b}_e \frac{\sin 2\varphi}{4(1-\nu)} + \vec{b}\times\vec{u}\left(\frac{1-2\nu}{2(1-\nu)}\ln\frac{r}{r_o} + \frac{\cos 2\varphi}{4(1-\nu)}\right)\right\}$$

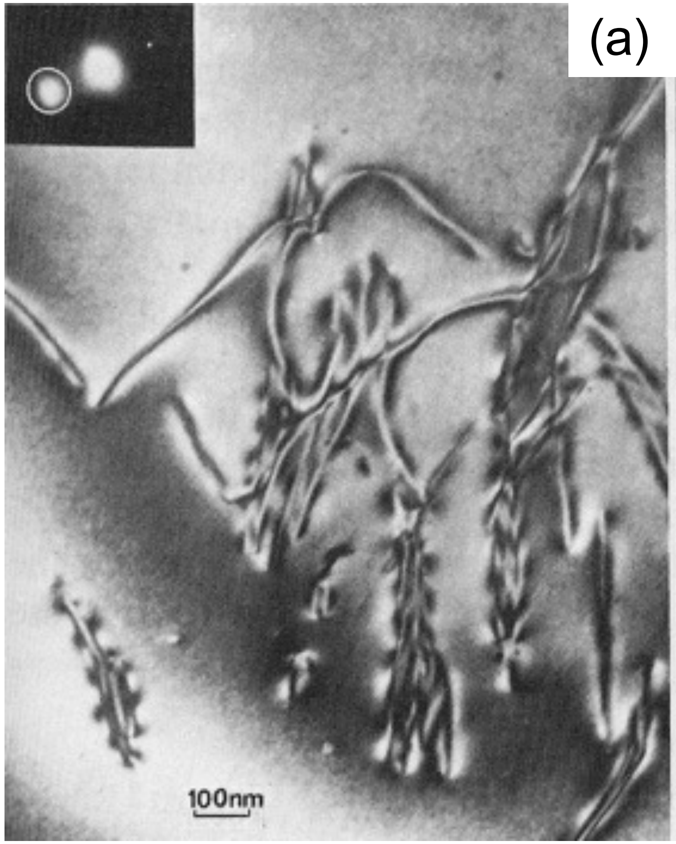
r_o : inner cut-off radius of the displacement field $\vec{R}$
ν: Poisson's ratio
$\vec{b}_e$: Step component of the Burgers vector, φ, *r* : Definition see figure
$\vec{u}$: Unit vector of the dislocation line direction

4.4 Contrasts in crystalline samples with lattice defects

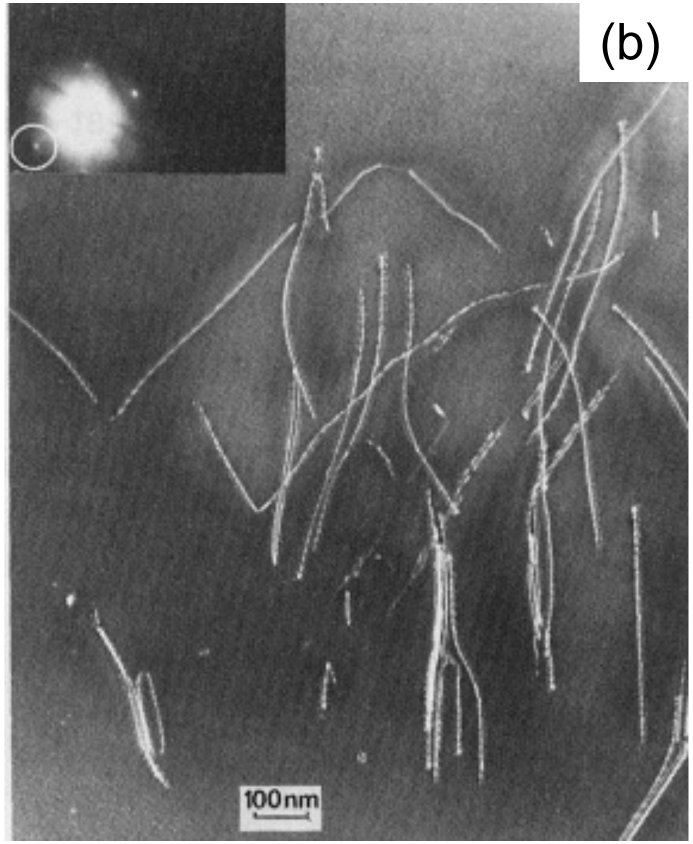
Bright field image with s=0

"weak beam" dark field (WBDF) image with s>>0

(a)



(b)



L. Reimer, "Transmission Electron Microscopy", Fig. 8.29

Dark-field TEM images of dislocations in deformed Si with (a) strongly excited $(\bar{2}20)$–reflex. (b) $(\bar{2}20)$ weak-beam dark-field imaging.

Simulation of dislocation contrasts

Calculated intensities for a screw displacement with
Displacement kernel at $x/\xi_g = 0$
with $\vec{g} \cdot \vec{b} = 2$

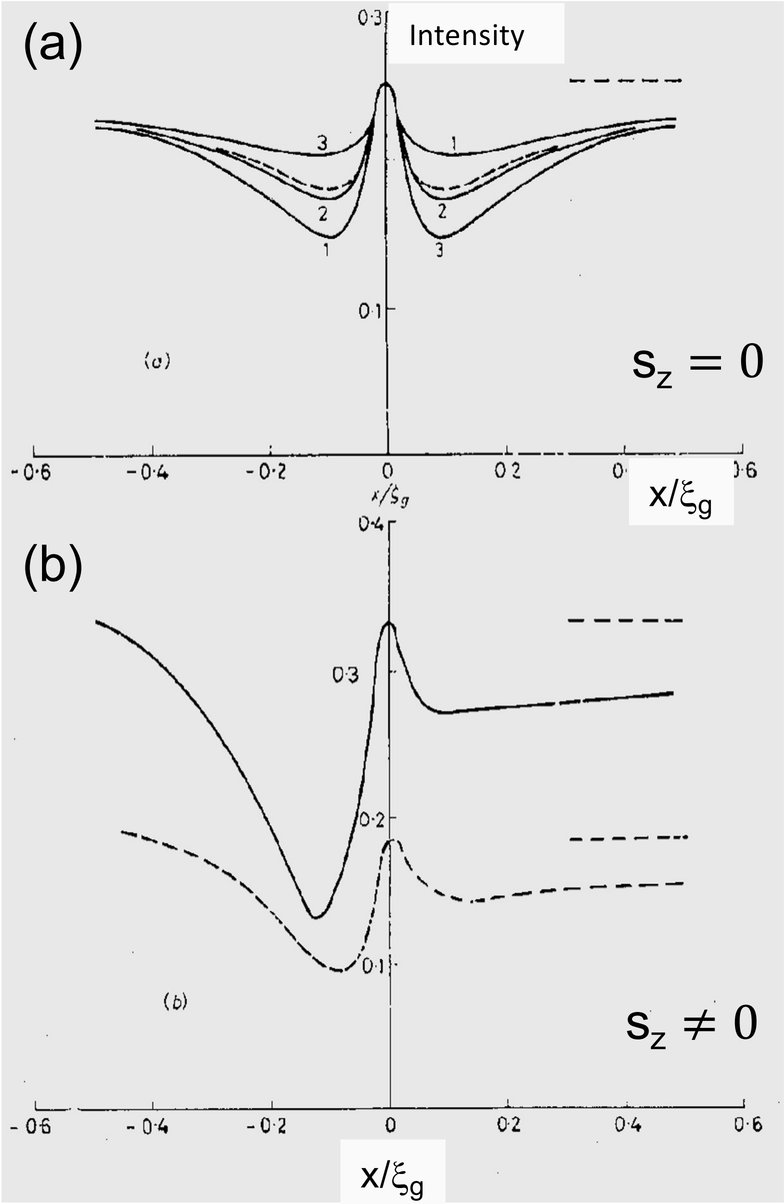
with a sample thickness t = 8 ξ_g for
(a) w = 0, $s_z = 0$
Curves 1-3 show dislocations at depths y/ξ_g =4, 4.25 and 4.5
(b) w = 0, 3, $s_z \neq 0$

ξ_g: Extinction length of the object to be mapped
used Bragg Reflexes

$$w = s_z \xi_g$$

Solid lines: Brightfield image
Broken lines: Dark field imaging

P. Hirsch, A. Howie, R.B. Nicholson, D.W. Pashley, J. Wheelan, Electron microscopy of thin crystals, p.254



4.4 Contrasts in crystalline samples with lattice defects

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Information from simulation of dislocation contrasts

1. two contrast lines per offset possible (see simulation on slide 25 and Fig.(a) on slide 26 for *w* = 0)

   for exactly fulfilled Bragg condition (s_z =0) and $\vec{g} \cdot \vec{b} = 2$

→ s_z =0 unfavorable for the mapping of dislocations (defects in general)

2. In case of deviation from the Bragg case $s_z \neq 0 \rightarrow$ Intensity minimum that is shifted to one side of the dislocation
   (*the sign of s_z determines the direction of the shift in the center of contrast*)

3. Dependence of the contrast width of an offset on extinction length

→ the smaller the extinction length, the narrower the contrast (*better separation of the dislocations at small distances*)

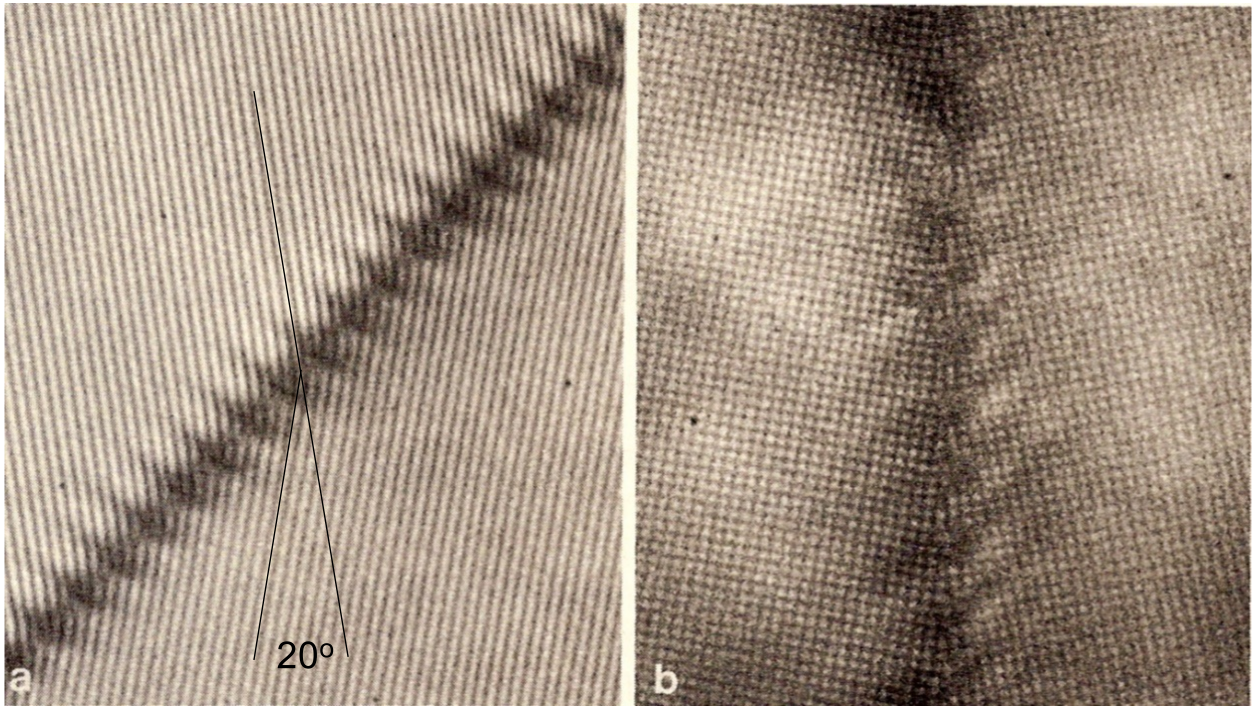
   Example: ξ_g = 50 nm $\rightarrow$ typical contrast width ξ_g /5 $\approx$ 10 nm

4. dislocation contrast depending on the depth of the dislocation in the sample.

4.4 Contrasts in crystalline samples with lattice defects

Grain boundaries

- Planar defects in polycrystalline samples
- Separation of differently oriented monocrystalline areas (grains)
- "Thickness" of grain boundaries only in the order of a few atomic layers (approx. 1 nm)



Reimer, "Transmission Electron Microscopy", Fig. 8.11

High resolution TEM images of a 20 degree grain boundary in gold,
a) {200} grating plane with 0.204 nm grating plane spacing
b) Crossed grid planes at a grain boundary in [100] zone axis

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Grain boundaries

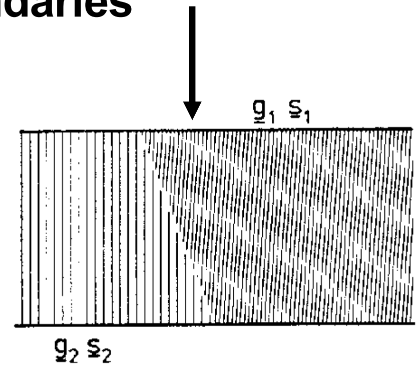
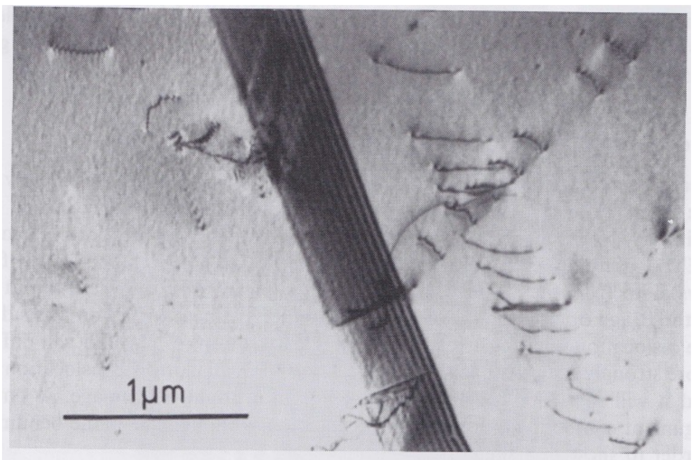


Abb. 4.20 Beugungsbedingungen bei einer Korngrenze. Wenn das obere Korn (rechts) so orientiert ist, daß der Beugungsvektor **g** mit Abweichung s_1 angeregt ist, dann wird wahrscheinlich ein anderer Vektor (g_2) in dem unteren Korn (links) mit anderem und möglicherweise wesentlich größerem s_2 angeregt.

P.J. Goodhew, J. Humphreys, R. Beanland, Electron Microscopy and Analysis, Fig. 4.23



P.J. Goodhew, J. Humphreys, R. Beanland, Electron Microscopy and Analysis, Fig. 4.24

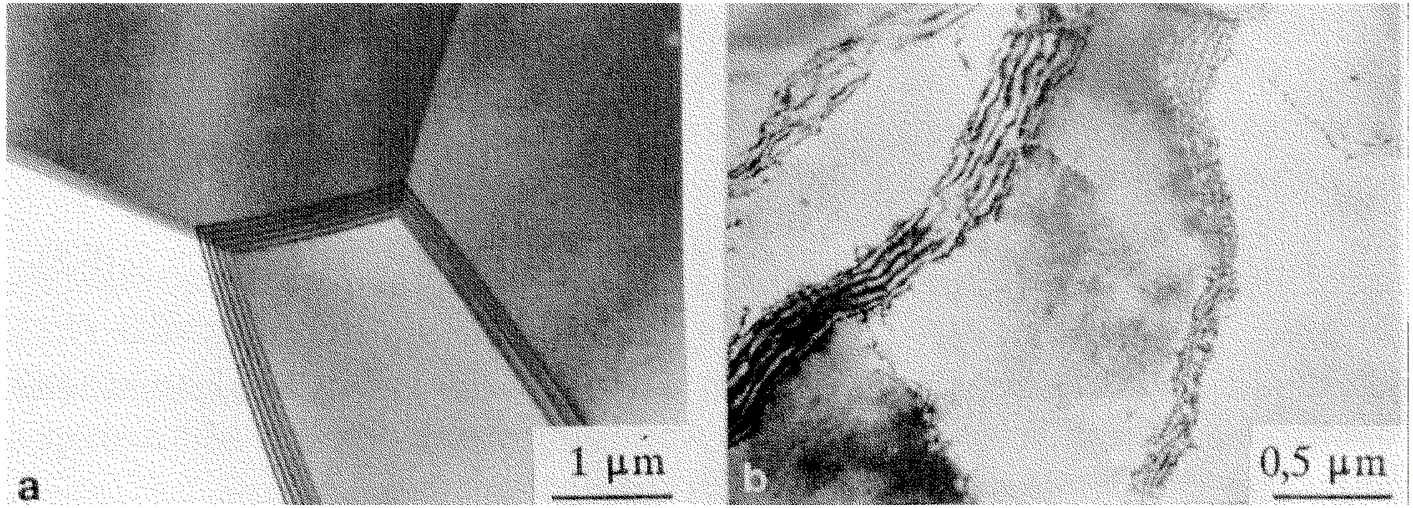
Contrast dependent on excitation of Bragg reflexes in neighboring Grains

Different mapping vectors ($\vec{g}_1$, $\vec{g}_2$) and excitation error (s_1, s_2) in neighboring grains

Thickness contours at the grain boundary, if two-beam condition only is present in one grain and in the 2nd grain. No Bragg reflex is strongly stimulated

→ Thickness contours for strongly bending grain

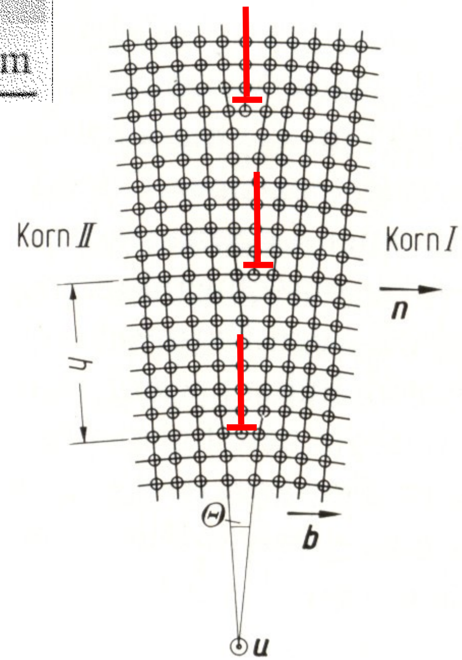
Grain boundaries in aluminum



E. Hornbogen, B. Skrotzki, Materials microscopy, Fig. 5.6

Left: Large angle grain boundaries with clear contrast differentiate between grains by
Bragg reflexes stimulated to varying degrees

Right: Small angle grain boundaries from regular ordered transfers



P. Haasen, Physical Metallurgy, Fig. 3.3

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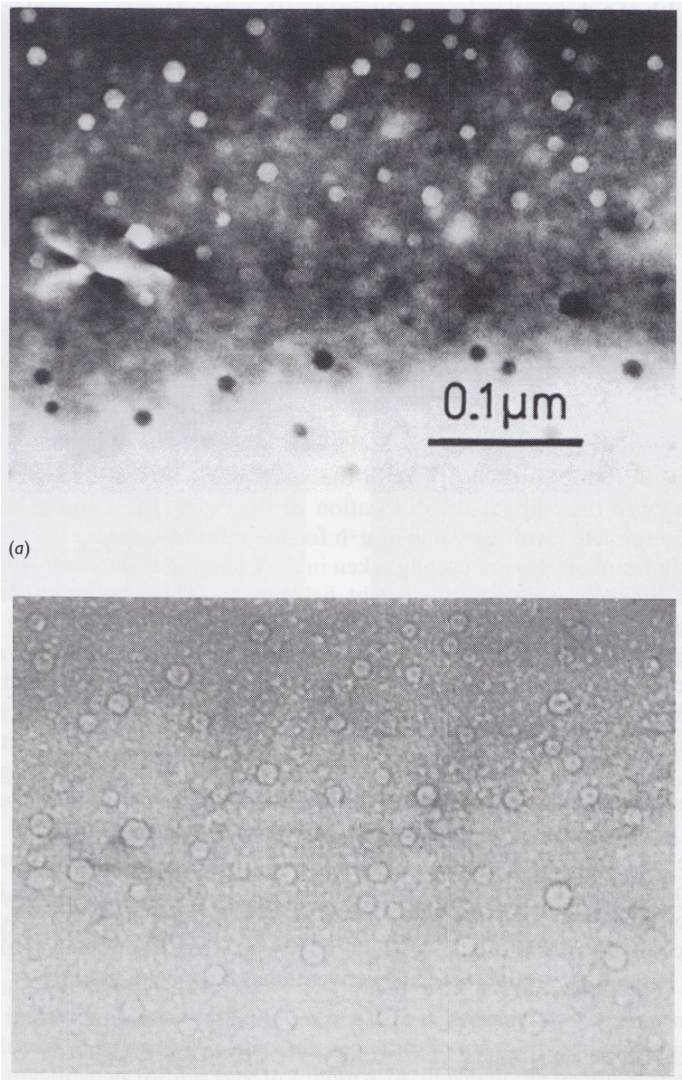
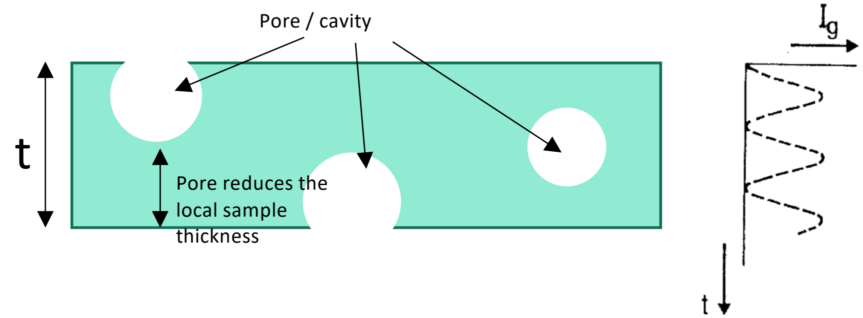
Contrasts of (gas-filled) cavities in aluminum



Bright field image with strongly excited $\vec{g}$:
Contrast inversion through locally reduced
Sample thickness in relation to the electron beam.



Kinematic diffraction (no strong Bragg reflex) suggested):

- Cavities with brighter contrast due to local Reduced sample thickness (mass thickness contrast)
- Fresnel diffraction (dark edges) due to ab-rupten refractive index jump with defocusing

P.J. Goodhew, J. Humphreys, R. Beanland, Electron Microscopy and Analysis, Fig. 4.33

Summary: Lecture 8

Laboratory for Electron Microscopy

- Contrasts in crystalline samples are created by lattice defects that disrupt the ordered lattice structure of materials. Classification: Point defects, line-like defects, two-dimensional defects, three-dimensional defects.
- To calculate the scattering amplitude: Let's replace all vectors $\overrightarrow{r_{pi}}$ with $\overrightarrow{r_{pi}} + \vec{R}$ and introduce a displacement vector $\vec{R}$. Here $\vec{R}$, is **much, much smaller** than the lattice plane spacing. $\vec{R}$ indicates the displacement of an atom from its regular lattice position and only affects the lattice amplitude. (the structure factor F_s remains unaffected to a good approximation)
- Structure amplitude with displacement vector $\vec{R}$: $\mathrm{F} = F_s \int_0^t \exp(2\pi i[z s_z + \vec{R}\,\vec{g}_{hkl}])dz$
- Special case: If the reciprocal lattice vector $\vec{g}_{hkl}$ is perpendicular to $\vec{R}$ then $\vec{g}_{hkl} \perp \vec{R}$. In this case $\vec{g}_{hkl} \cdot \vec{R} = 0$ and the defect shows no contrast. This special case is used to determine the displacement vector $\vec{R}$**. Two excitation conditions with different reciprocal lattice vectors** are found where $\vec{g}_{hkl} \cdot \vec{R} = 0$. The cross product results in $\vec{R} = \vec{g}_{h_1k_1l_1} \times \vec{g}_{h_2k_2l_2}$.
- Stacking faults are two-dimensional planar defects that occur in crystalline materials and disrupt the stacking sequence (ABCABC) of the most densely packed planes (ABCBABC). The beginning and end of a stacking fault are characterized by partial dislocations.
- Stacking fault contrast is caused by a disturbance of the previously constructively interfering waves. At the location of the stacking fault, the waves are no longer in phase, which causes a reduction in intensity in the bright field image. Stacking faults lying obliquely in the sample generate an oscillating fringe contrast that changes depending on the excitation condition and the imaging condition (HF/DF). These systematic changes are used for stacking fault characterization.
- Dislocations are the carriers of plastic deformation. By displacing the upper half of a crystal relative to the lower half of a crystal by a displacement vector (Burgers vector) $\vec{b}$, a dividing line is created, which is called a dislocation. The Burgers vector has a length and a direction that depends on the crystal structure and the direction of the acting force. Different types of dislocations are characterized by the angle between the Burgers vector $\vec{b}$ and the dislocation line $\vec{u}$. Borderline cases are the screw dislocation ($\vec{b}$ II $\vec{u}$) and the step dislocation ($\vec{b} \perp \vec{u}$).
- The dislocation is described by an extra half-plane in the grid. The grid planes bend at the offset core (the end of the half plane). The Bragg condition can be fulfilled locally at the bent grating planes and contrast is created in the image. If **two excitation conditions are** found $\vec{g}_{hkl} \cdot \vec{b} = 0$ and the dislocation thus becomes "invisible", the Burgers vector $\vec{b}$ of the dislocation can be determined via the cross product $\vec{g}_{h_1k_1l_1} \times \vec{g}_{h_2k_2l_2} = \vec{b}$.
- Simulation of dislocation contrasts via column approximation. The "fine structure" of dislocation contrasts is simulated here. It becomes clear that the dislocation contrast depends on the position of the dislocation in the sample depth and on the set excitation error s_z . The larger s_z , the clearer the dislocation contrast.
- **Grain boundaries** are surface-like defects in polycrystalline samples and separate differently oriented monocrystalline areas (grains). Due to the different orientations of the grains, different excitation conditions are fulfilled, which lead to different intensities in the image in an RF or DF image.