Micro-texture analysis by electron diffraction

Electrons:

only radiation with electrical charge

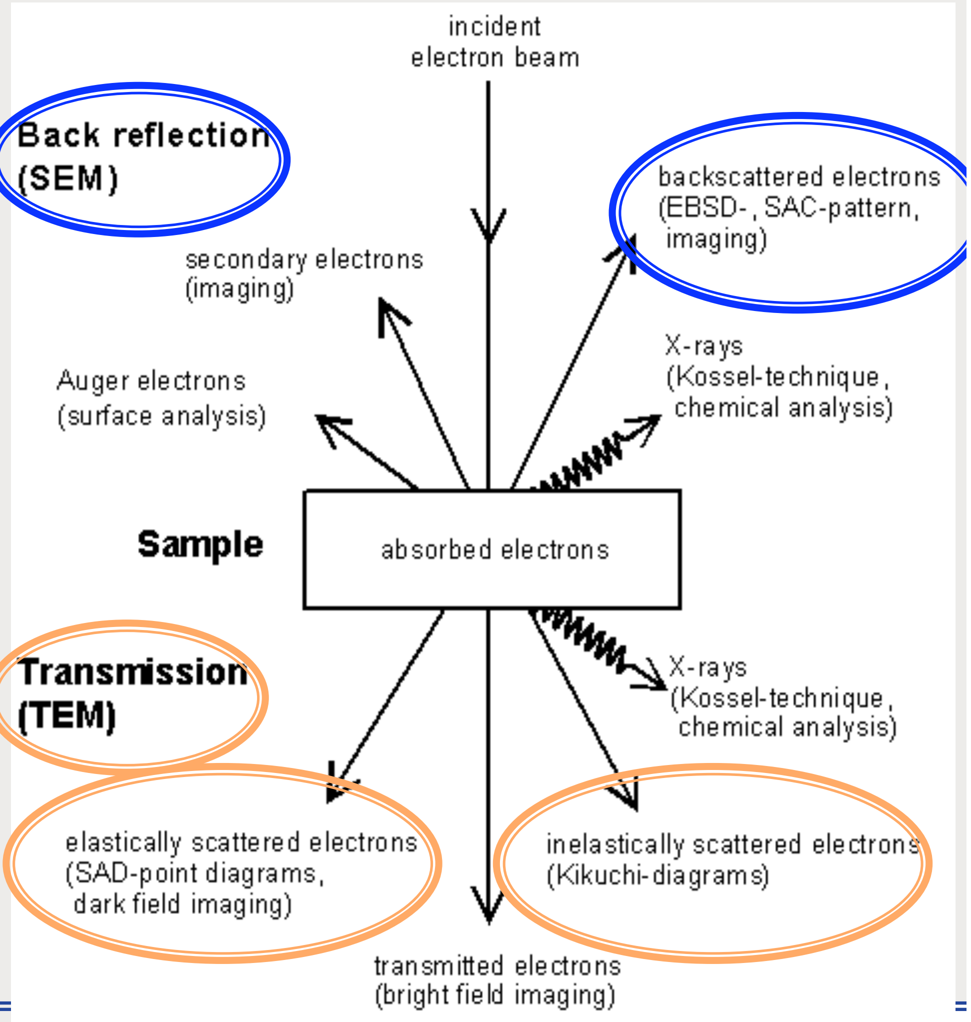
strong interaction with nuclei and shell electrons

vacuum, electron microscope

minimum penetration depth, maximum spatial resolution (0.1µm and 1nm for SEM and TEM, resp.)

wave length

$$\lambda = \frac{h}{\sqrt{2 \cdot m \cdot e \cdot U}} = \frac{h}{\sqrt{2 \cdot m_0 \cdot e \cdot U \cdot \left(1 + e \cdot U / (2 m_0 c^2)\right)}}$$



Selected Area Diffraction, SAD

coherent, elastic scatter of electrons at crystal lattice ⇒ SAD point diagrams

- SEM (20kV) $\lambda = 0.0086$nm
- TEM (200kV) $\lambda = 0.0025$nm

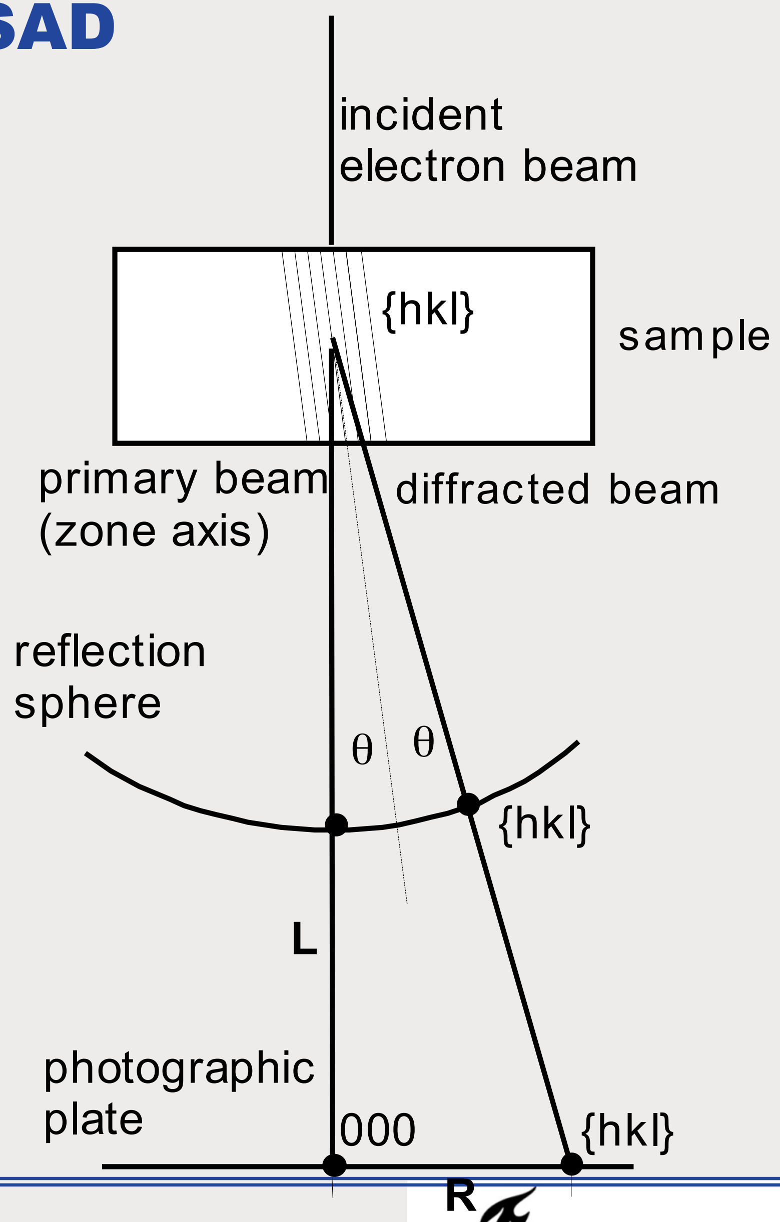
⇒ $\theta < 2°$

⇒ $\sin\theta \approx \theta$

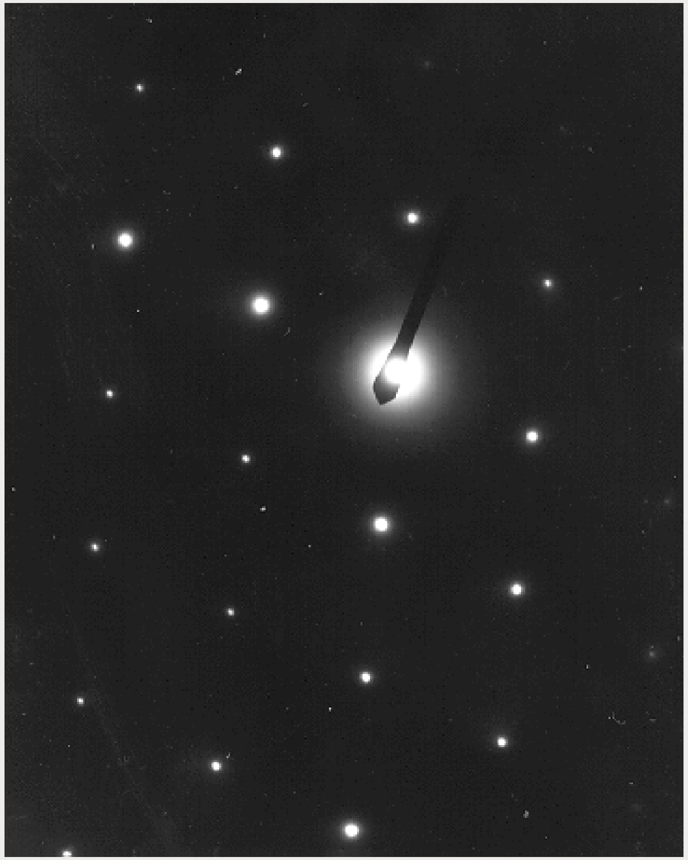
$$n\lambda = 2d\sin\theta \approx 2d\theta = 2dR_{hkl}/2L$$

(with n=1) $\lambda L = R_{hkl}d$ or $R_{hkl} = \lambda L/d$

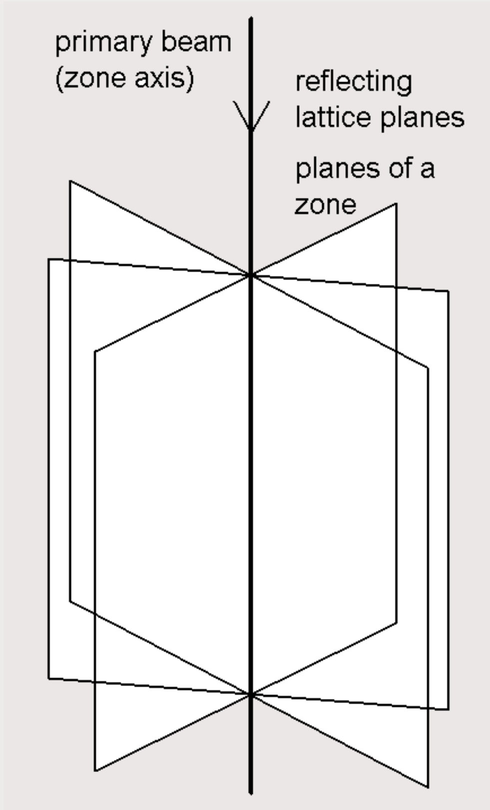
λL: camera constant (magnification)

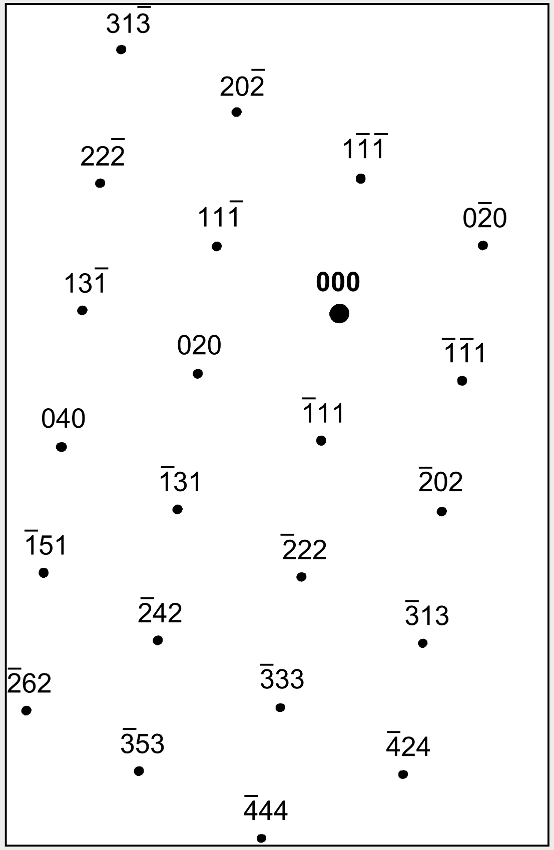


Selected Area Diffraction, SAD



[110]

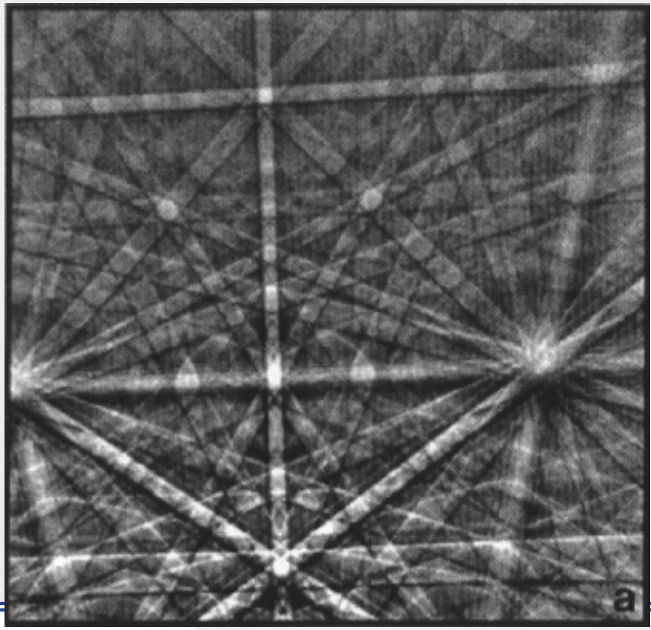




Micro-texture analysis by electron diffraction

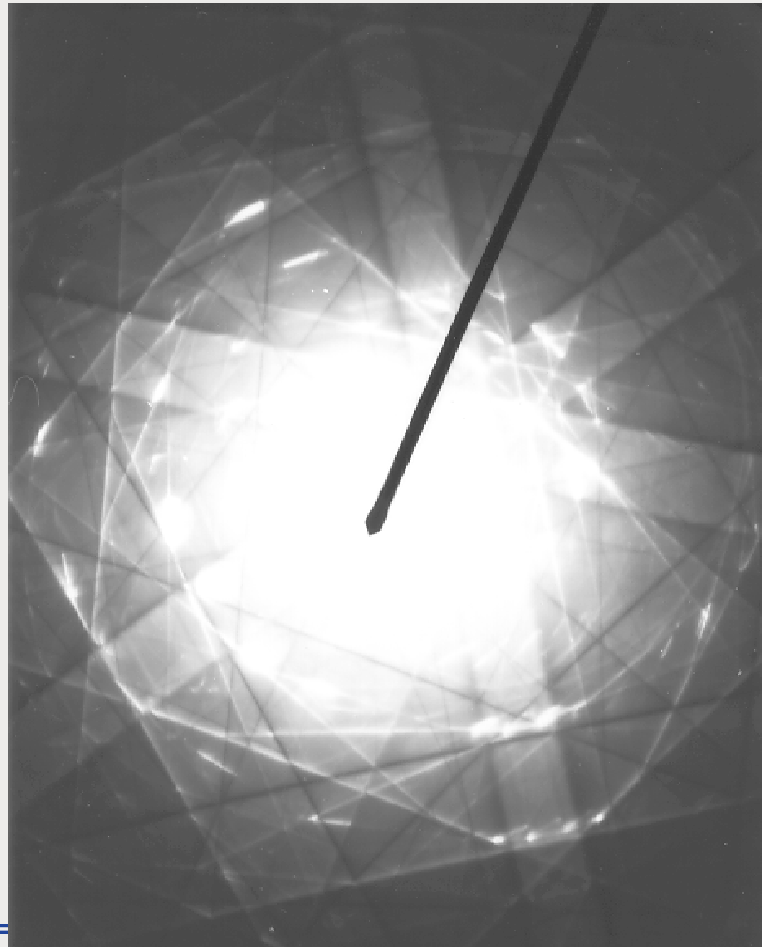
Most techniques for micro-texture analysis by electron diffraction are based on the evaluation of **Kikuchi-patterns**

- TEM (micro diffraction, CBED)
- SEM (EBSD, SAC)

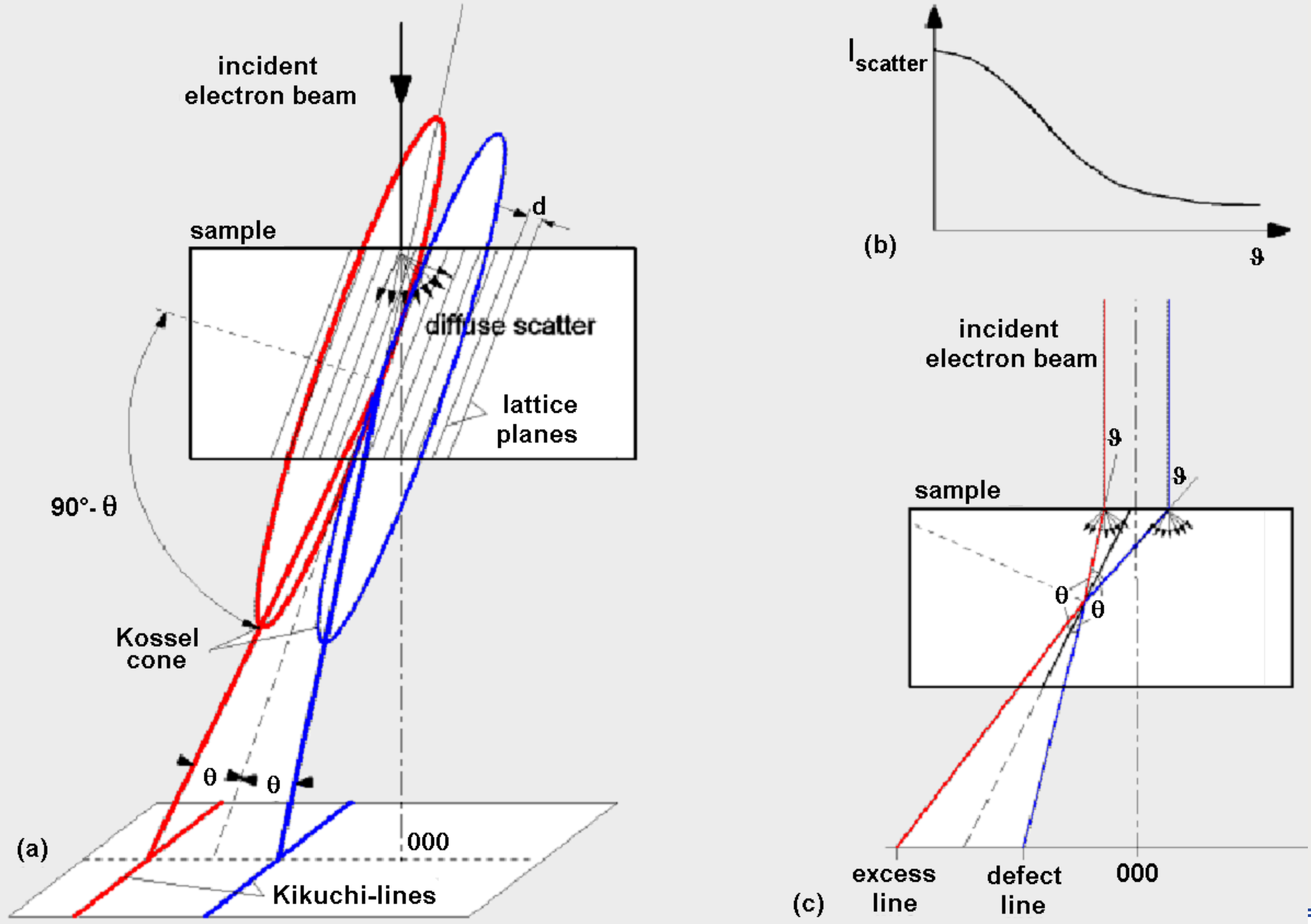


SEM

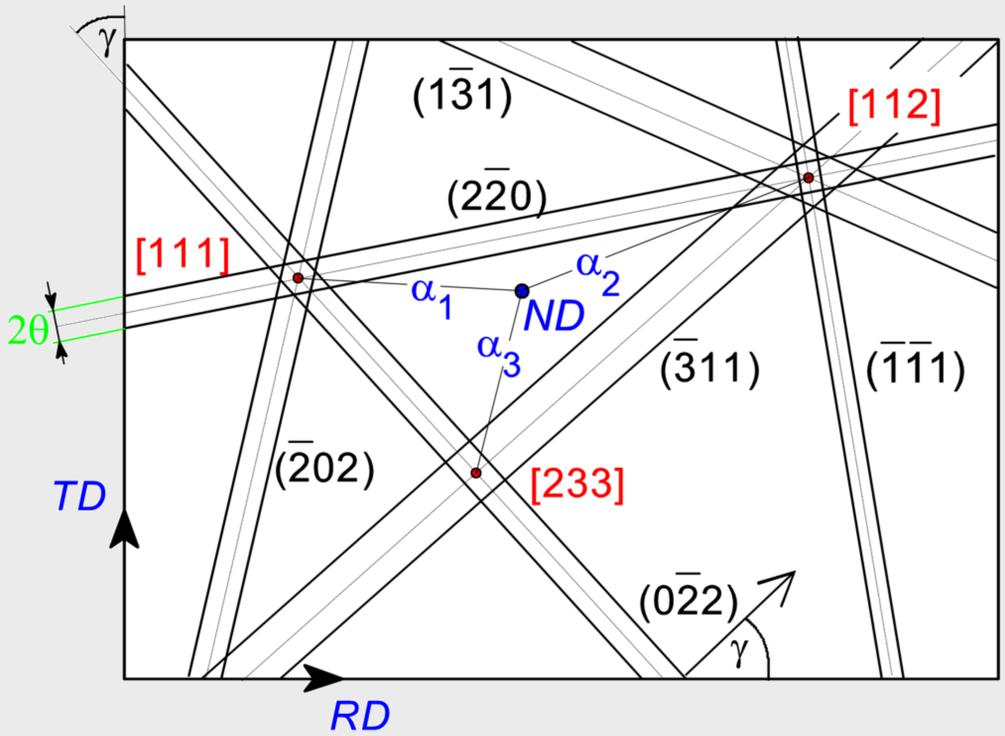
TEM

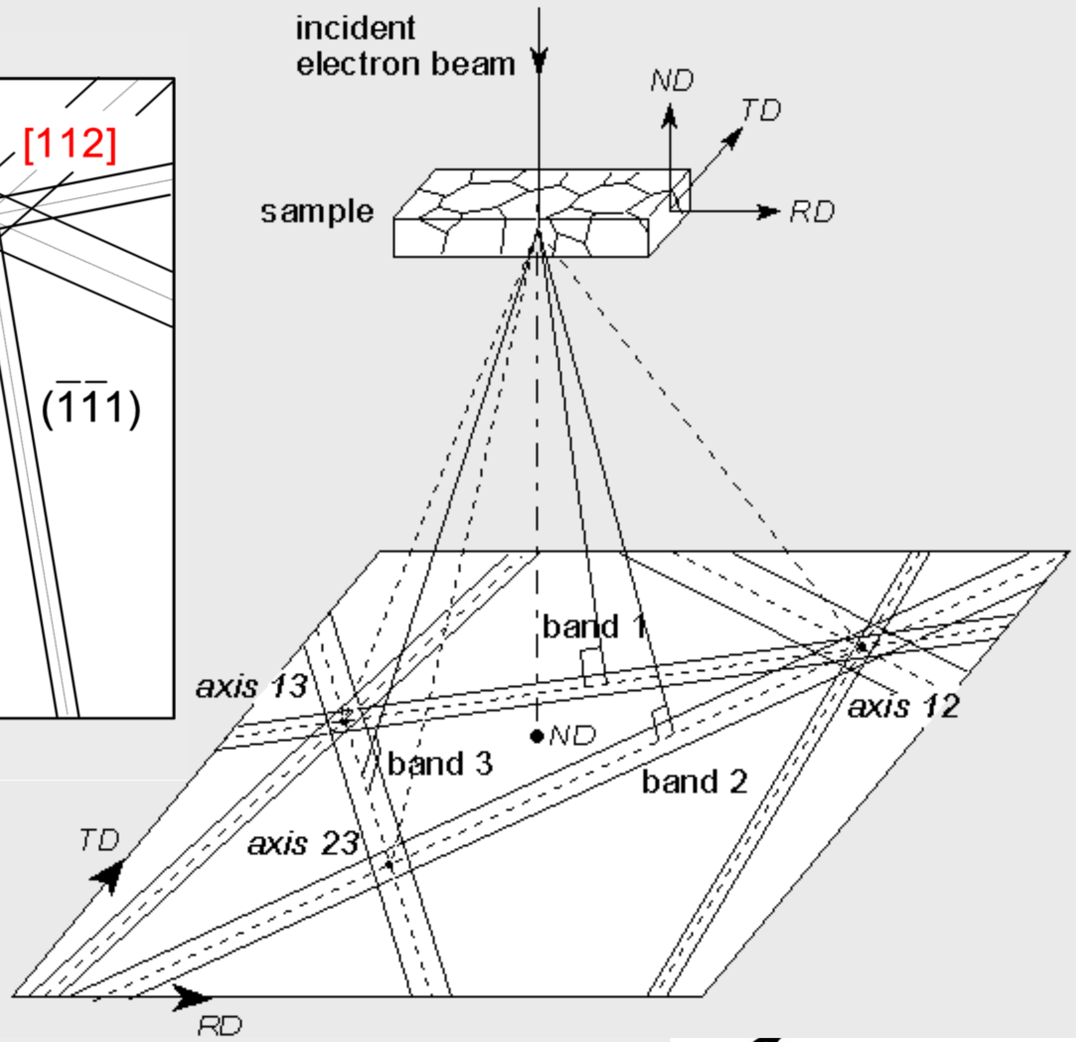


Formation of Kikuchi-Lines

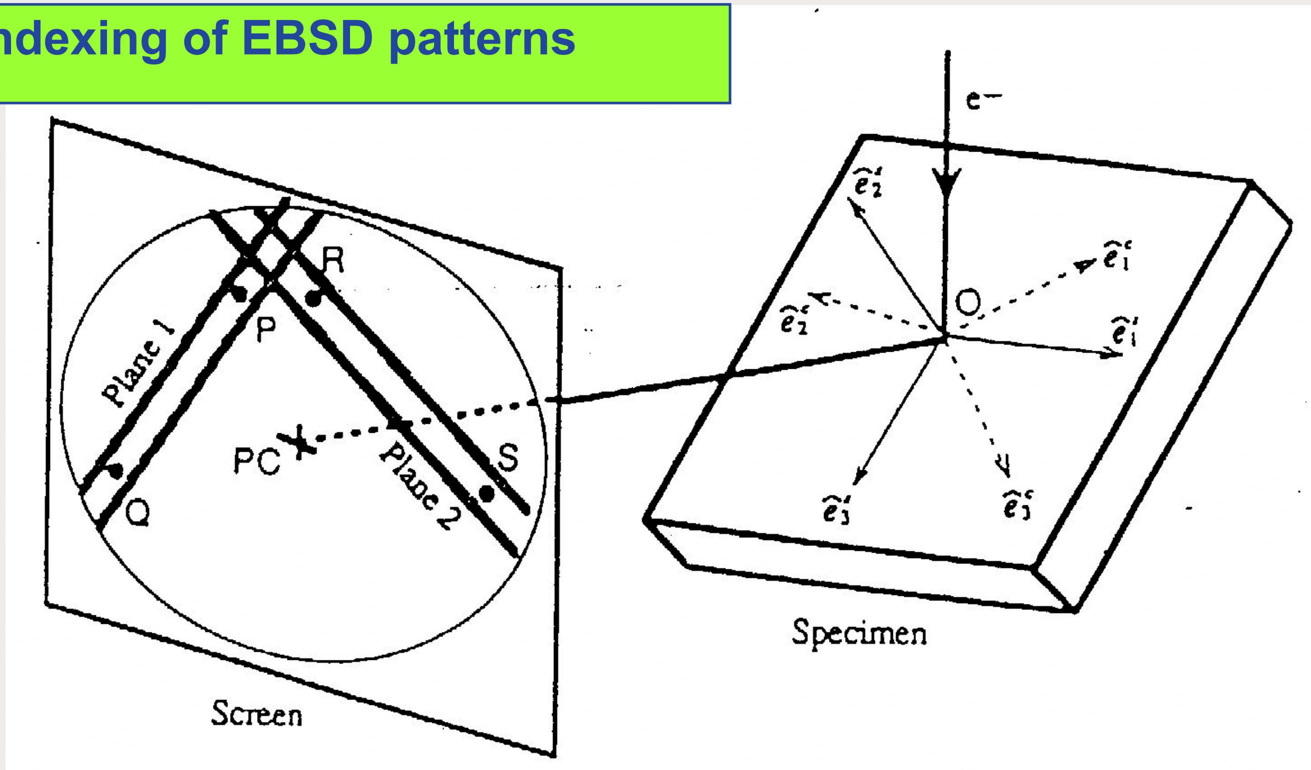


Evaluation of Kikuchi-Patterns





Indexing of EBSD patterns



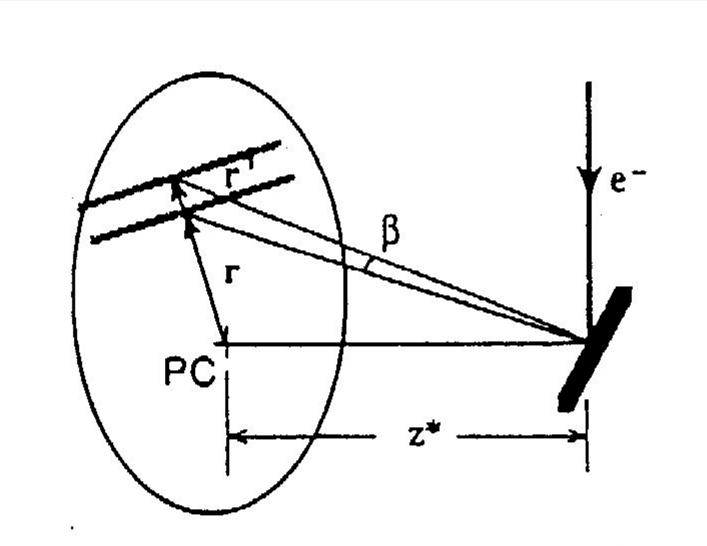
Interplanar angle between planes:

$$\cos\gamma = \left|\overline{n}_1 . \overline{n}_2\right|$$

$$\overline{n}_1 = \frac{\overline{OP} x \overline{OQ}}{\left|\overline{OP} x \overline{OQ}\right|} \qquad \overline{n}_2 = \frac{\overline{OR} x \overline{OS}}{\left|\overline{OR} x \overline{OS}\right|}$$

$$\cos\gamma = \frac{h_1 h_2 + k_1 k_2 + l_1 l_2}{\left(h_1^2 + k_1^2 + l_1^2\right)^{1/2} \left(h_2^2 + k_2^2 + l_2^2\right)^{1/2}}$$

Bandwidth angle β



$$\beta = \tan^{-1}\left(\frac{r'}{z*}\right) - \tan^{-1}\left(\frac{r}{z*}\right)$$

$$\beta = 2\sin^{-1}\left(\frac{\lambda}{2d_{hkl}}\right)$$

Values for γ and β are compared with theoretical values

selection is made on basis of closest match

Calculation of Crystal Orientation

Following reference systems are involved:

1. Crystal reference system: $\bar{e}_i^{c}$

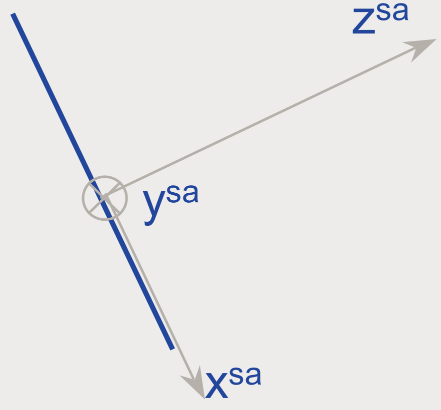
   (<100> axes for a cubic crystal)

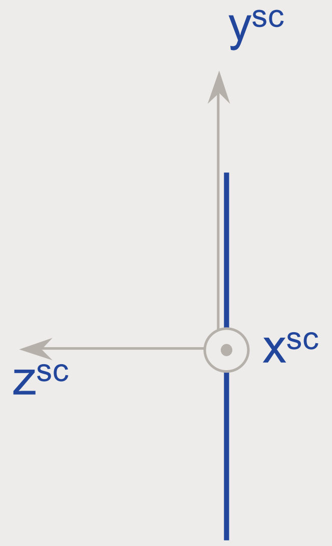
2. Sample reference system: $\bar{e}_i^{sa}$

   (RD, TD, ND axes for rolled sheet)

3. Screen reference system: $\bar{e}_i^{sc}$

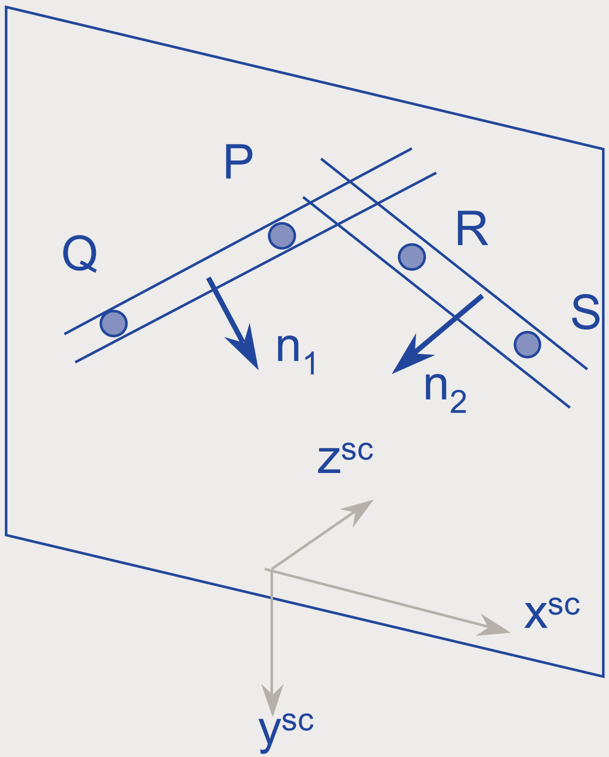
4. Pattern reference system $\bar{e}_i^{p}$

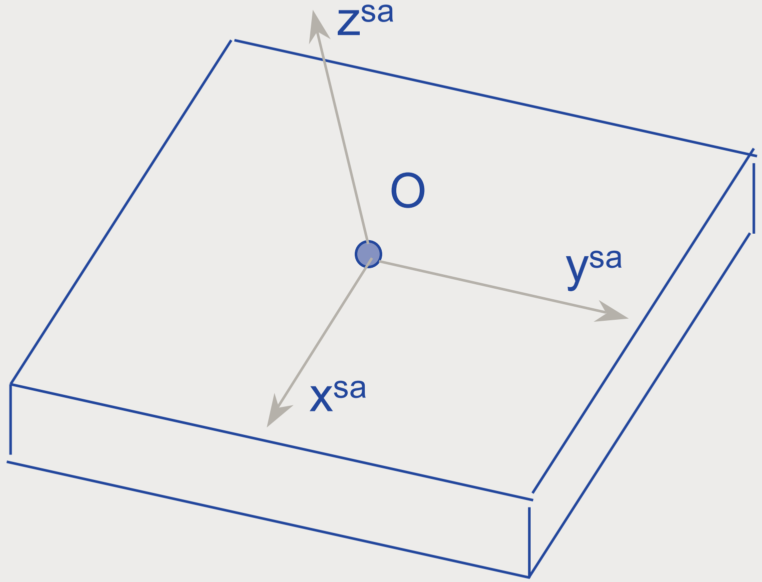




Construction of pattern reference system

base: $\bar{e}_i^p$





$$\bar{n}_1 = \frac{\overline{OP} x \overline{OQ}}{\left|\overline{OP} x \overline{OQ}\right|}$$

$$\bar{n}_2 = \frac{\overline{OR} x \overline{OS}}{\left|\overline{OR} x \overline{OS}\right|}$$

$$\bar{e}_1^p = \bar{n}_1$$

$$\bar{e}_2^p = \bar{n}_1 x \bar{n}_2$$

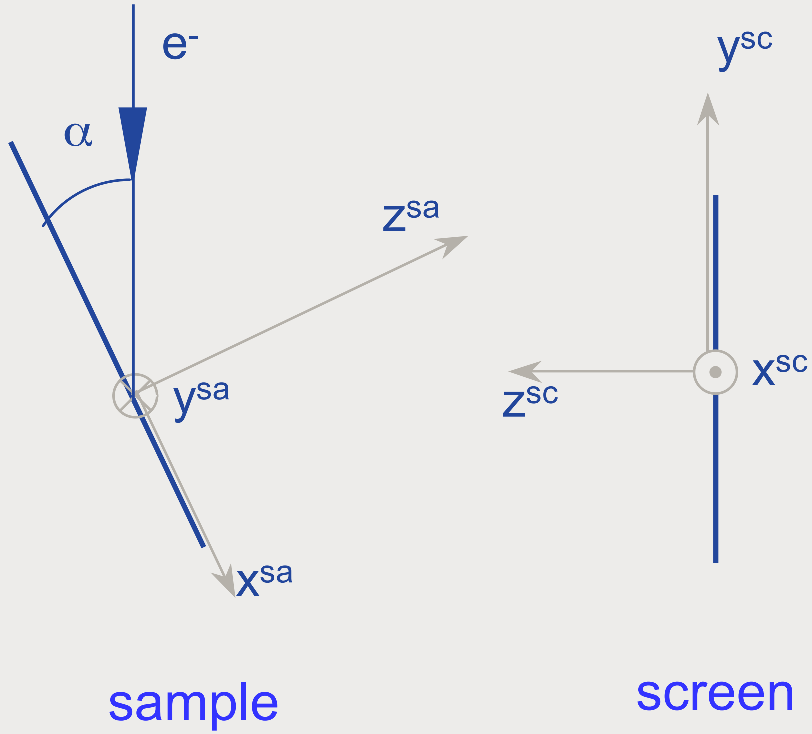
$$\bar{e}_3^p = \bar{e}_1^p x \bar{e}_2^p$$

Transformation from pattern to screen coordinates:

$$g'_{ij} = \bar{e}_i^{\,p} . \bar{e}_j^{\,sc}$$

Transformation from screen to sample coordinates:

$$g''_{ij} = \bar{e}_i^{\,sc} . \bar{e}_j^{\,sa}$$



$$\bar{e}_1^{\,sc} = -\bar{e}_2^{\,sa}$$

$$\bar{e}_2^{\,sc} = \bar{e}_3^{\,sa} \sin\alpha - \bar{e}_1^{\,sa} \cos\alpha$$

$$\bar{e}_3^{\,sc} = -(\bar{e}_1^{\,sa} \sin\alpha + \bar{e}_3^{\,sa} \cos\alpha)$$

Transformation from crystal to pattern coordinates:

$\bar{e}_i^p$ can be expressed in terms of crystal coordinates

$$\bar{e}_1^p = \frac{(hkl)_1}{\left|(hkl)_1\right|}$$

$$\bar{e}_2^p = \frac{(hkl)_1}{\left|(hkl)_1\right|} x \frac{(hkl)_2}{\left|(hkl)_2\right|}$$

$$\bar{e}_3^p = \bar{e}_1^p x \bar{e}_2^p$$

$$g'''_{ij} = \bar{e}_i^c . \bar{e}_j^p$$

pattern to screen

crystal to pattern

screen to sample

Orientation matrix:

$$g_{ij} = g'''_{ik} \quad g'_{kl} \quad g''_{lj}$$

Summary TEM-based Techniques

Advantages

- excellent spatial resolution, down to <1nm
- details of Kikuchi-patterns enable analysis of complex crystal structures
- additional information on substructure

Disadvantages

- tedious sample preparation
- small field of view, poor statistical relevance
- correlation sample region ⇔ macrostructure not straightforward
- automation complicated

Introduction to Texture Analysis
Outline

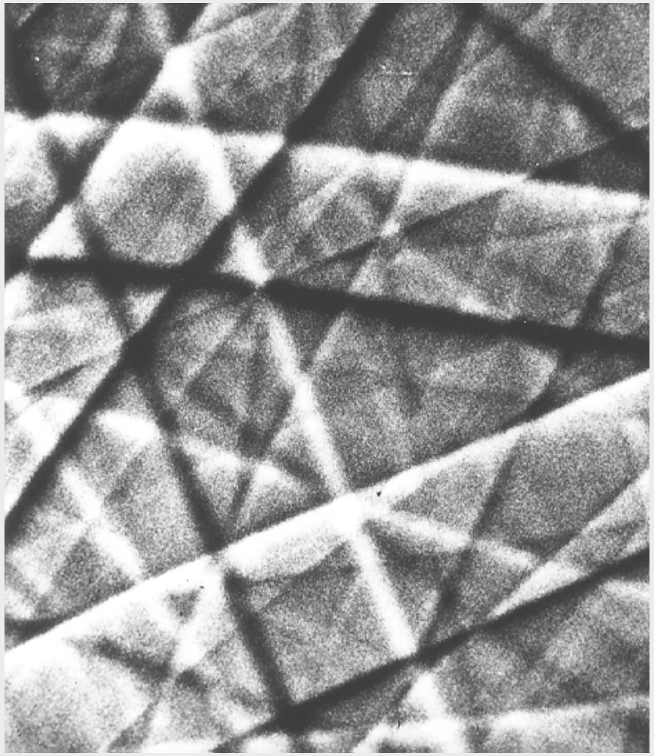
6 Measurements of Microtexture

- **TEM-based techniques, Kikuchi-Patterns**
  formation, interpretation

- **SEM-EBSD**
  principle, development, penetration depth and spatial resolution, calibration, sample preparation

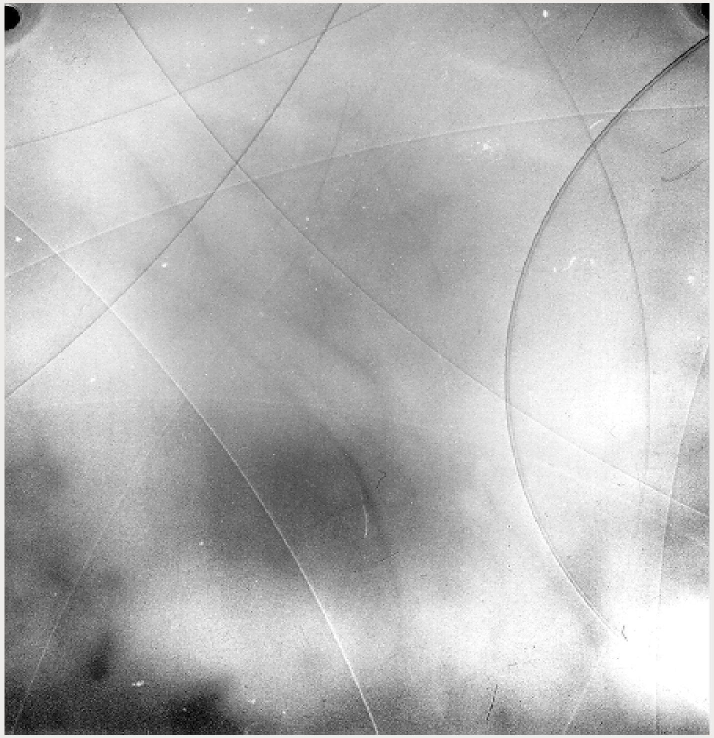
- **Orientation Mapping / Orientation Microscopy**
  experimental orientation microscopy, analysis of orientation maps

SEM-based techniques



Selected Area Channelling (SAC)

rocking of the incident electron beam at constant site: electron channelling patterns (~TEM Kikuchi patterns)



Kossel-Technique

diffraction of characteristic X-rays *that are generated within the sample material*

Electron Back-Scatter Diffraction

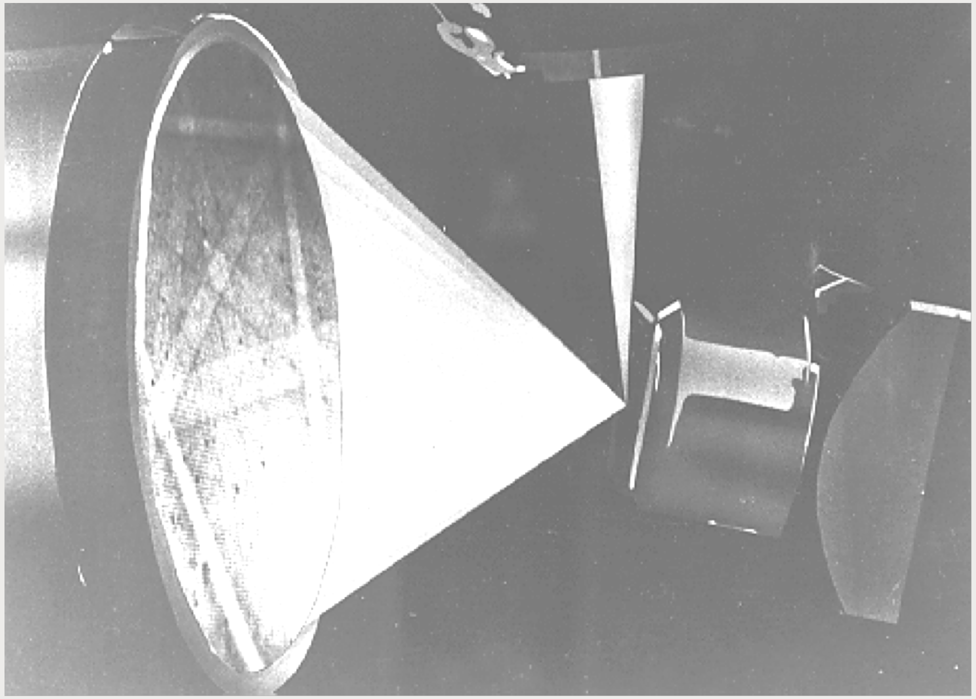
Today, SAC and micro-Kossel virtually obsolete for routine single grain orientation measurements forerunners of **Electron Back-Scatter Diffraction (EBSD)**

Kikuchi-patterns in back-reflection in the SEM

• focused, stationary electron beam

• highly tilted sample (65-70°) to increase gain of back-scattered electrons

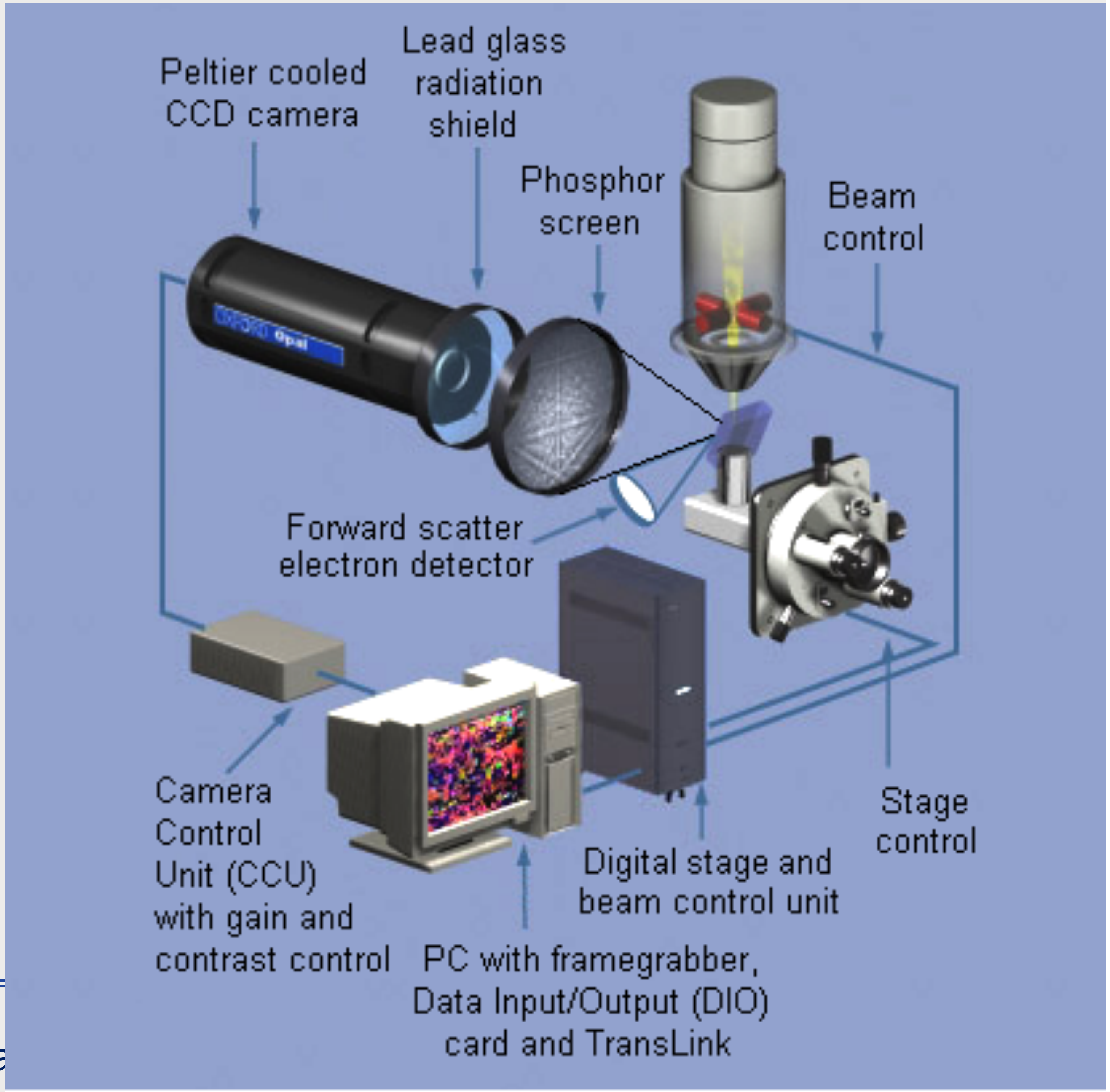
• recording medium (photographic film or phosphor screen + camera) to capture the diffraction patterns



EBSD – Milestones

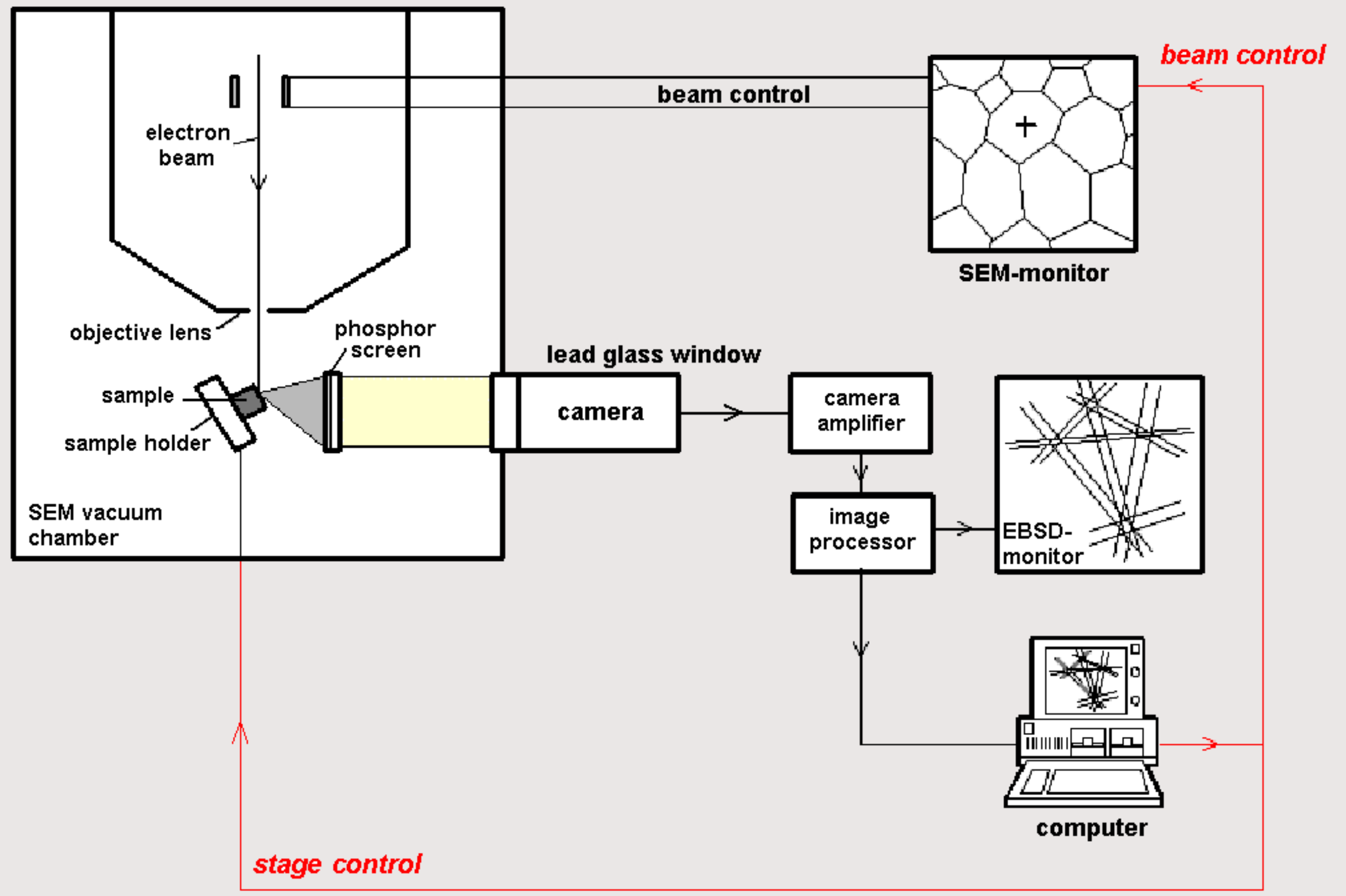
- Meibom and Rupp, 1933; Boersch, 1937: High-angle Kikuchi-Patterns from ”reflected” electrons
- Alam et al., 1954: High-angle Kikuchi-Patterns (-164°) from LiF, KI,
- NaCl, PbS_2 (special-camera)
- Venables & Harland, 1973: SEM, TV-camera ⇒ EBS**P**
- Dingley et al. (1980s): software, Si-calibration
- Hjelen et al. (1990s): commercial hardware, SIT-camera
- Engler and Gottstein (1990s): EBSD at IMM, Aachen
- Schmidt *et al.* (1990s): Hough transform, automatisation of indexing
- Adams et al., 1993: OIM, automatisation of mapping
- recent developments: digital cameras (>25 patterns/s)

Scheme of an EBSD Set-up

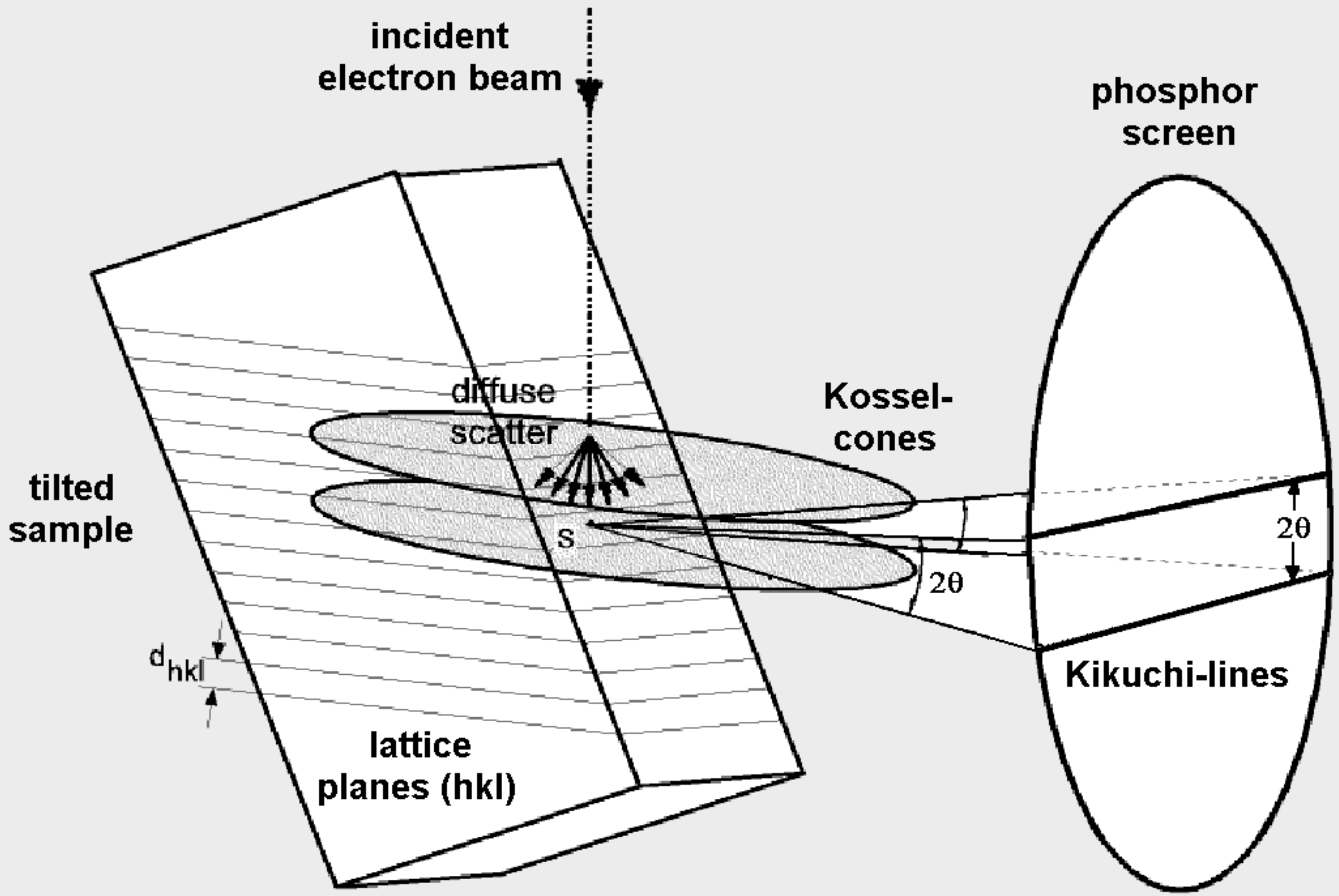


Oxford INCA

Scheme of an EBSD Set-up

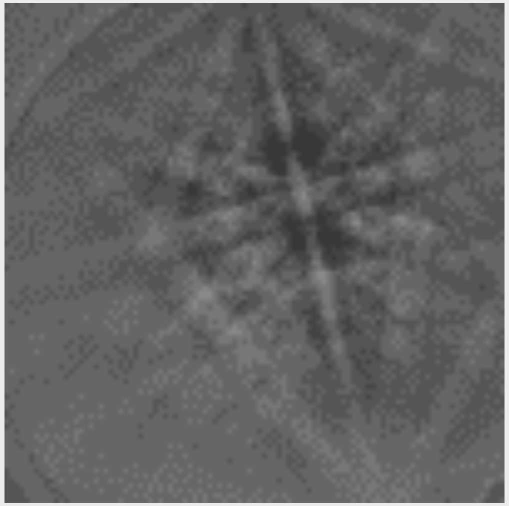


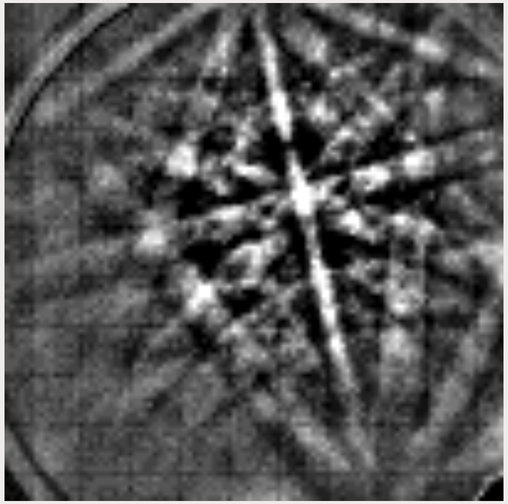
Formation of EBSD Patterns

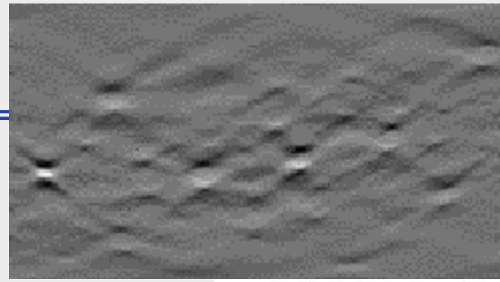


Processing Sequence

- capture EBSD diffraction pattern, digitization
- average *n* frames (1 < n < 16)
- background subtraction
- increase dynamic range
- reduce size (e.g. 512x512 pixels → 128x128 pixels)
- Hough transformation (lines → points)
- evaluate angles between zones
- compare measured set of interzonal angles with known data for crystal structure
- select best fit
- calculate and store orientation

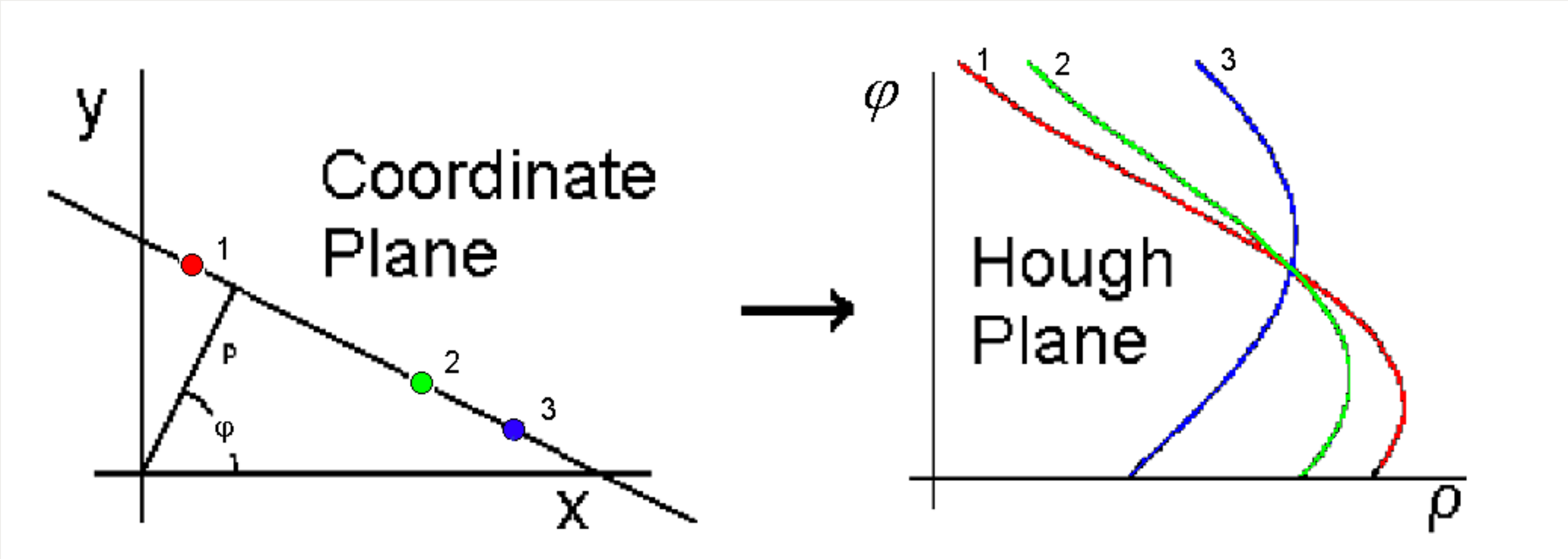






Hough-Transform

$\rho = x \cos \varphi + y \sin \varphi$
ρ: distance from the origin; φ: angle with the normal

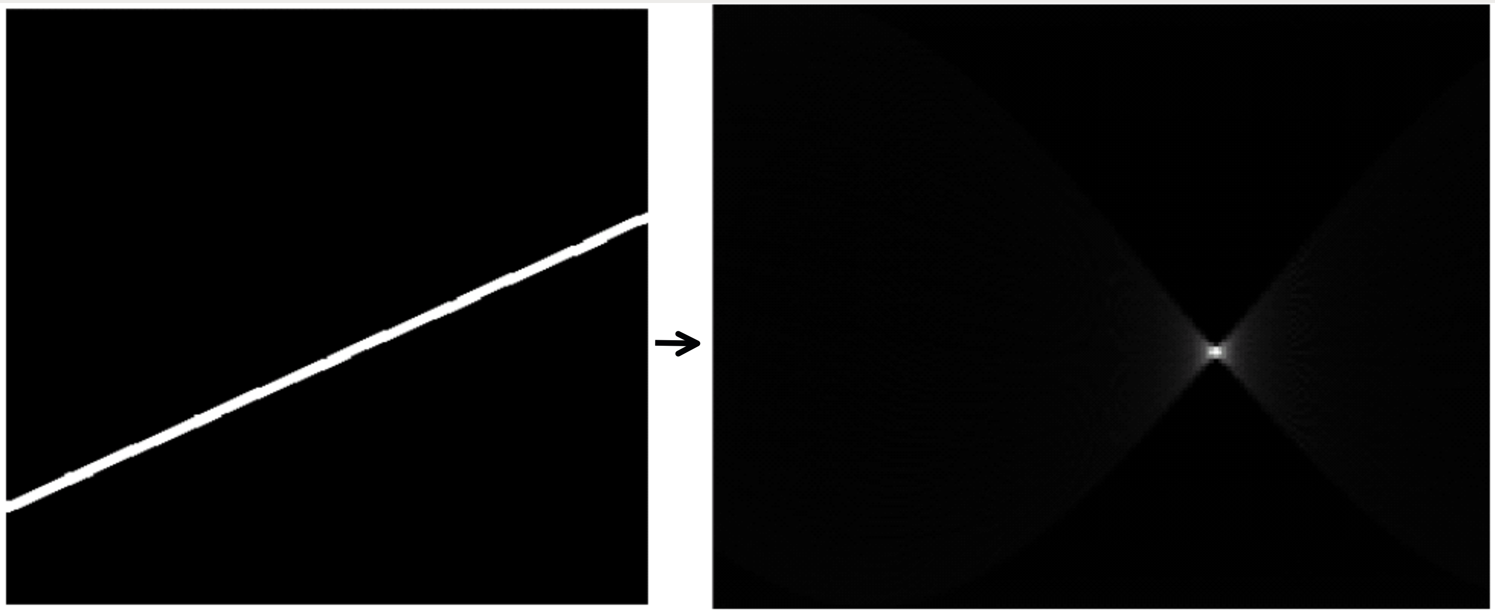


objective: convert the parallel lines / bands found in EBSD patterns into points.
These points can more easily be identified and used in automatic computation.

Hough-Transform

A grey-value image can be transformed by building a Hough Space $H(\rho,\varphi)$ where, for each pixel (x,y) in the original image, all possible ρ values are calculated for all angles φ ranging from 0 to 180° via the equation $\rho = x \cos \varphi + y \sin \varphi$. The intensity value of the pixel at (x,y) is then added to the bin in the array at each corresponding (ρ,φ).

⇒ Radon Transform

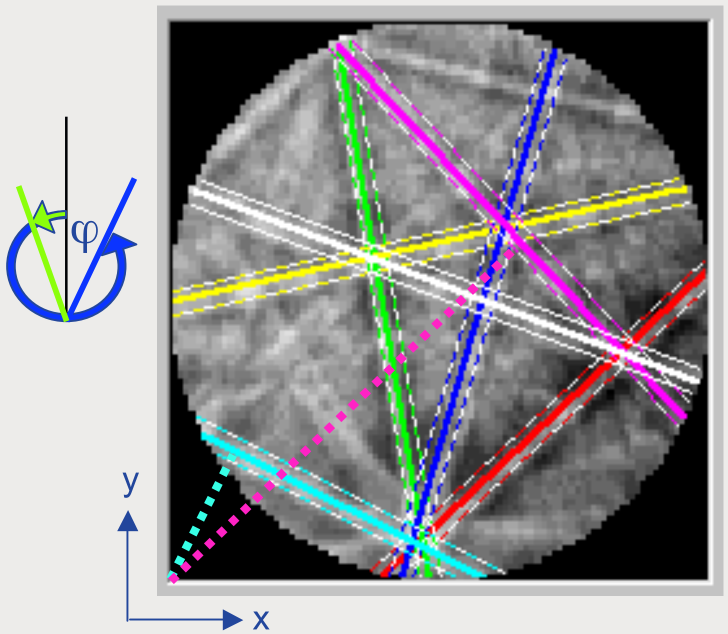


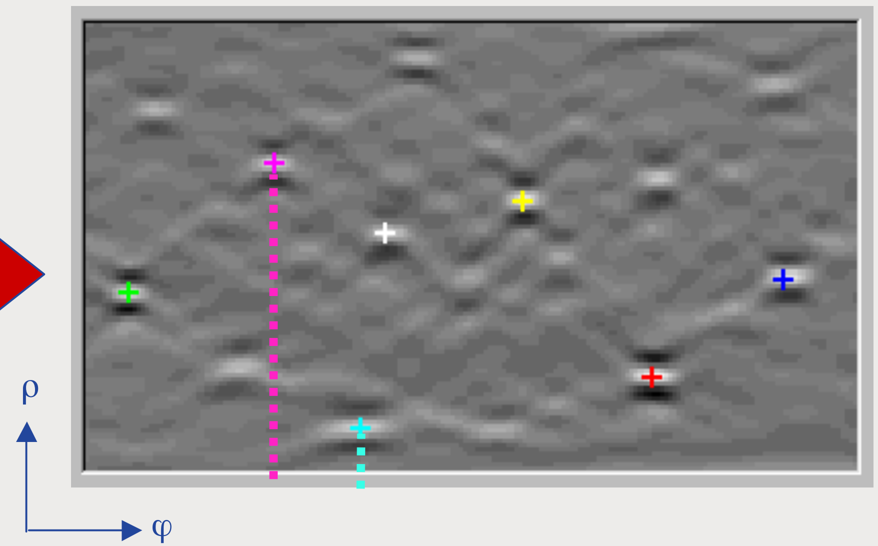
thus, straight lines in the original image (i.e. bands) can be identified by detecting the local maxima in Hough space

Hough-Transform

$\rho = x \cos \varphi + y \sin \varphi$

ρ: distance from the origin; φ: angle with the normal





point / line ⇒ sine-curve / intersection point

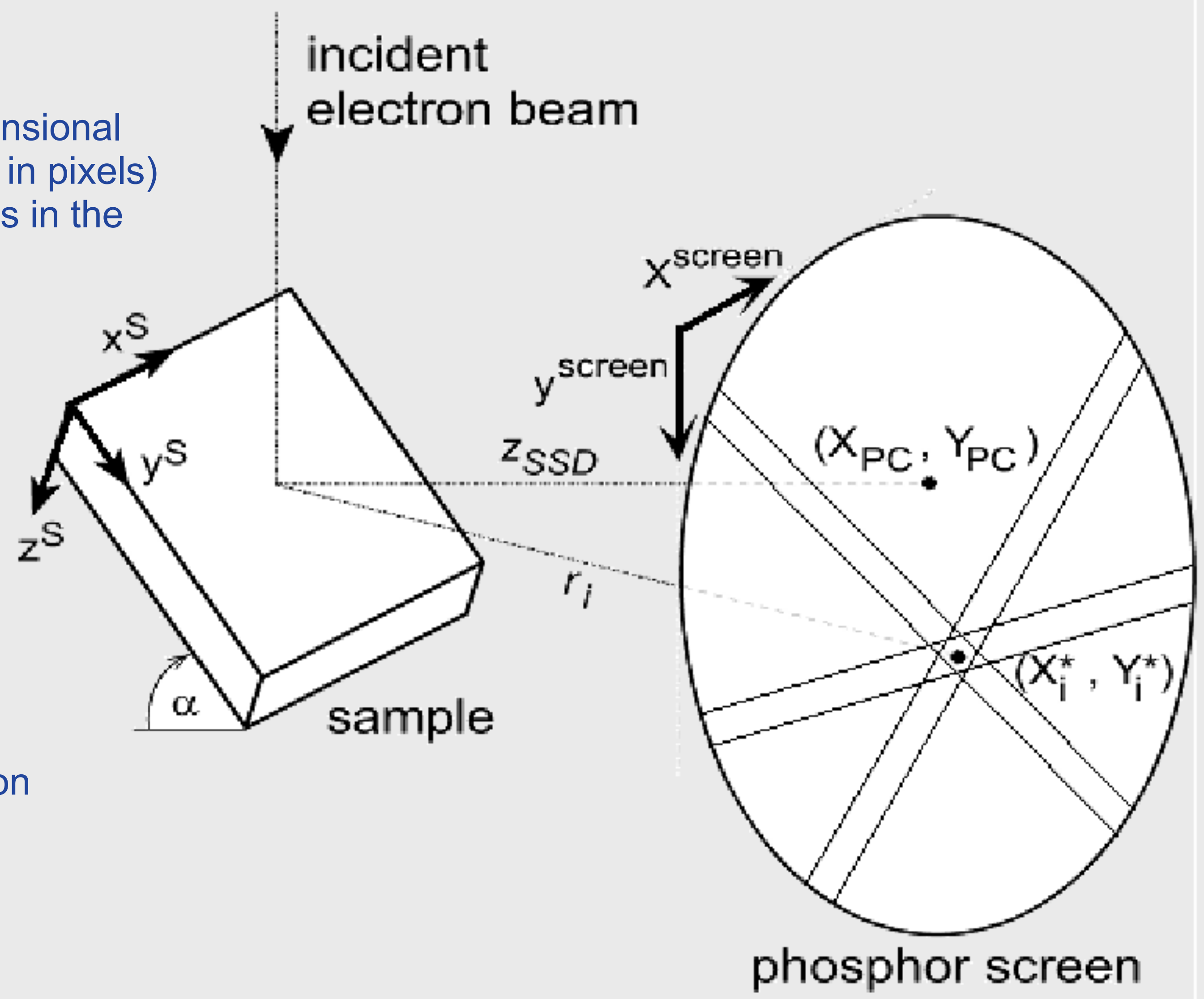
Calibration

transformation of 2-dimensional screen coordinates (e.g. in pixels) into 3-dimensional angles in the specimen

PC: Pattern Centre (X_{PC}, Y_{PC}, Z or SSD)

Methods

- shadow casting (wires, spheres)
- known crystal orientation (Si, GaAs)
- pattern magnification
- iterative pattern fitting



Spatial Resolution of EBSD

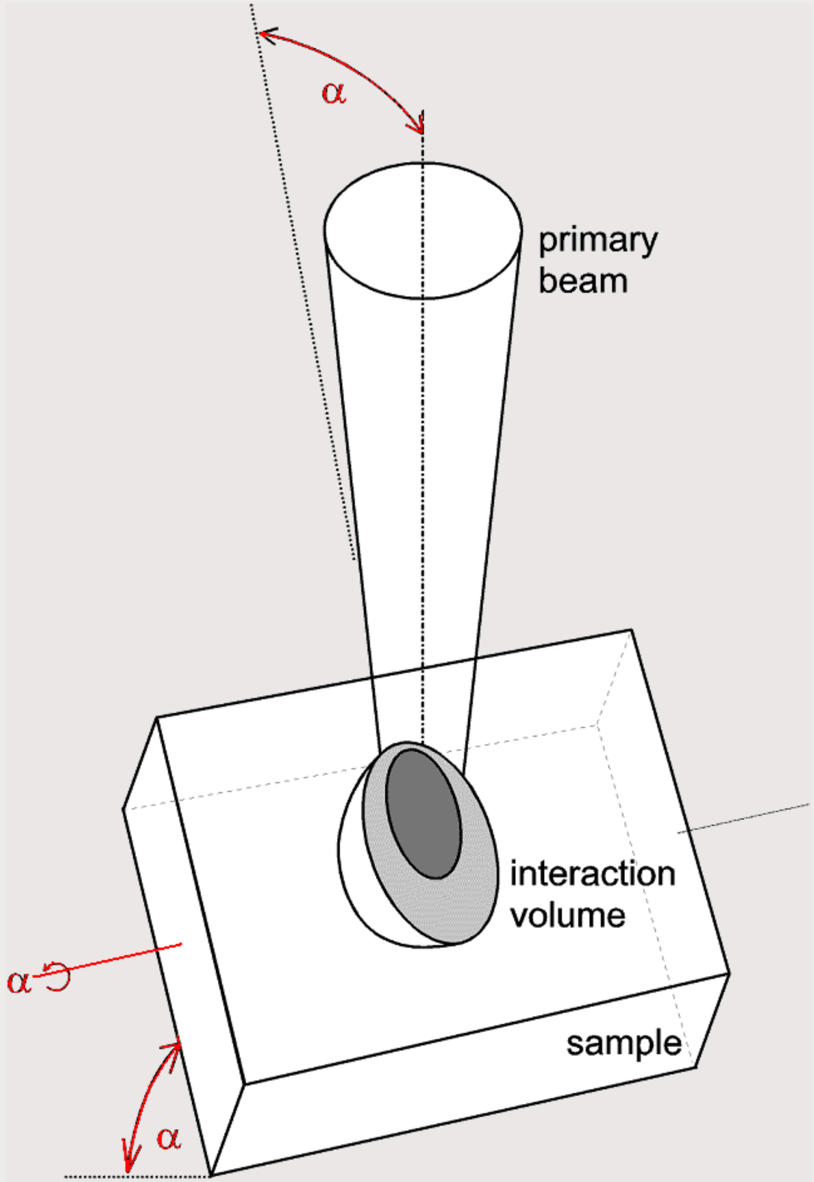
Interaction of back-scattered electrons with sample material

- un-tilted sample: “tear-drop” volume, (size >1µm, dependent on U_{acc}, Z, ...)
- tilted sample: very shallow penetration depth (<20nm)

⇒ dependence on surface quality

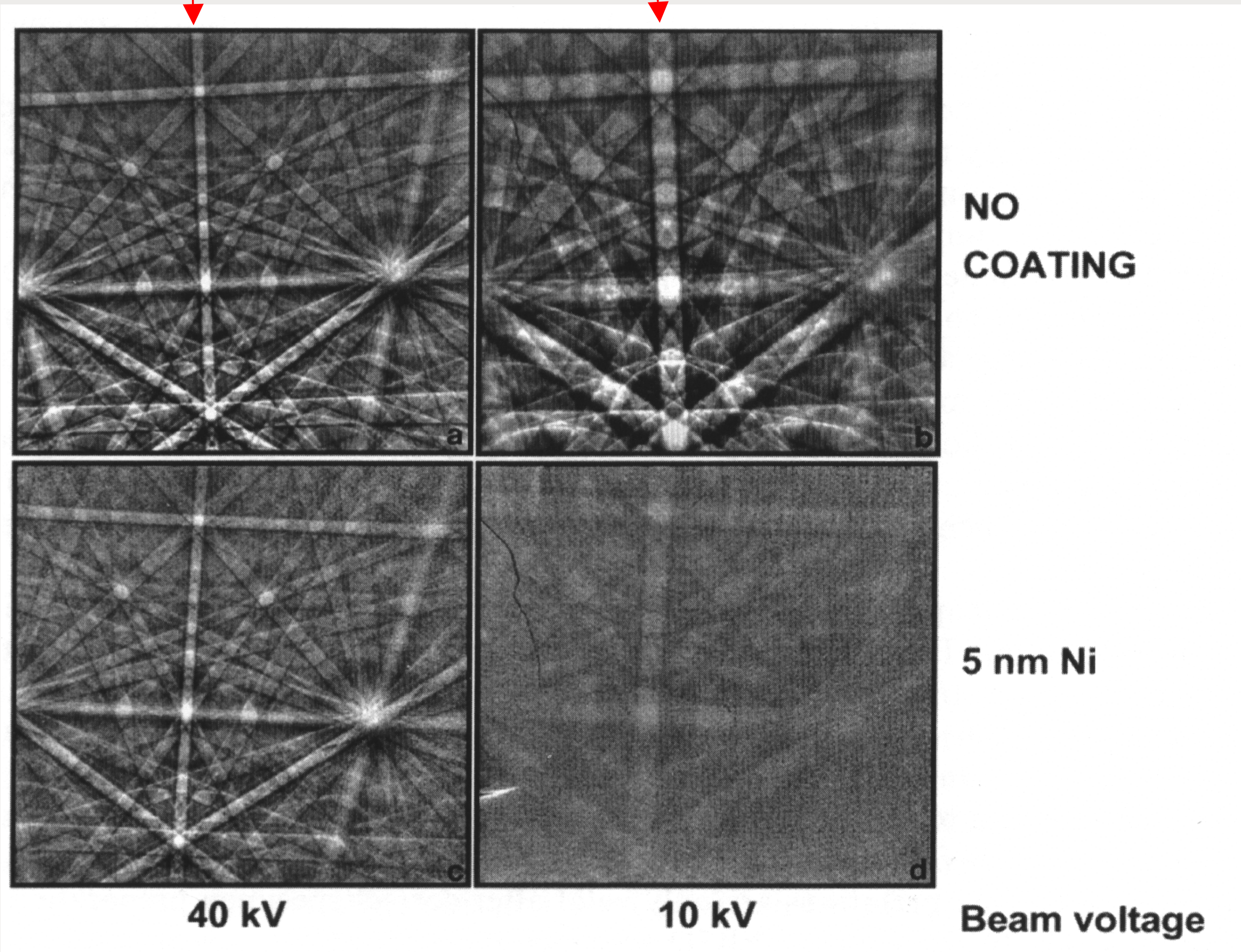
lateral resolution given by spot size: ~100nm

⇒ asymmetry ⊥ vs. || tilting direction (~1:4)



Penetration depth of EBSD

note different band widths



at 10 kV , pattern is almost extinct by 5 nm coating

at 40 kV:

Al: 100 nm

Ni: 20 nm

Au: 10 nm

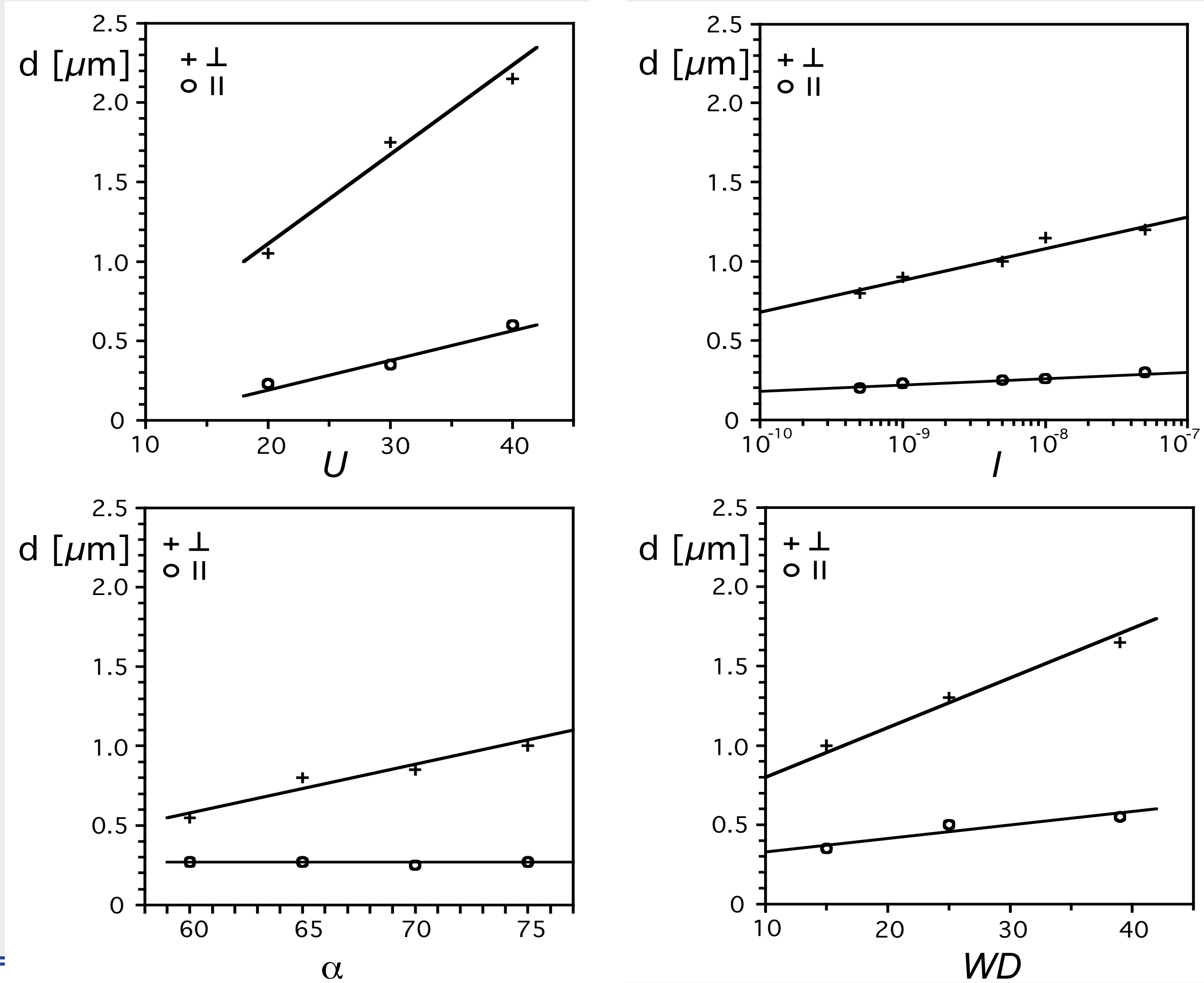
Spatial Resolution of EBSD

- *absolute* spatial resolution: interaction volume / spot size
- *effective* spatial resolution: ability to resolve two patterns from neighbouring crystallites

spot size *vs*. pattern clarity ⇒ minimum for given set of parameters

- Hjelen and Nes, 1990: W gun, Al: **250x700 nm²**
- Humphreys *et al.,* 1999: FEG SEM, Al: **~200 nm**
- Harland *et al.,* 1981 (!): FEG SEM, Ni: **20 x 80 nm²**
- Troost *et al*., 1994: Simulations: **10nm**

Spatial Resolution of EBSD



Typical Parameters

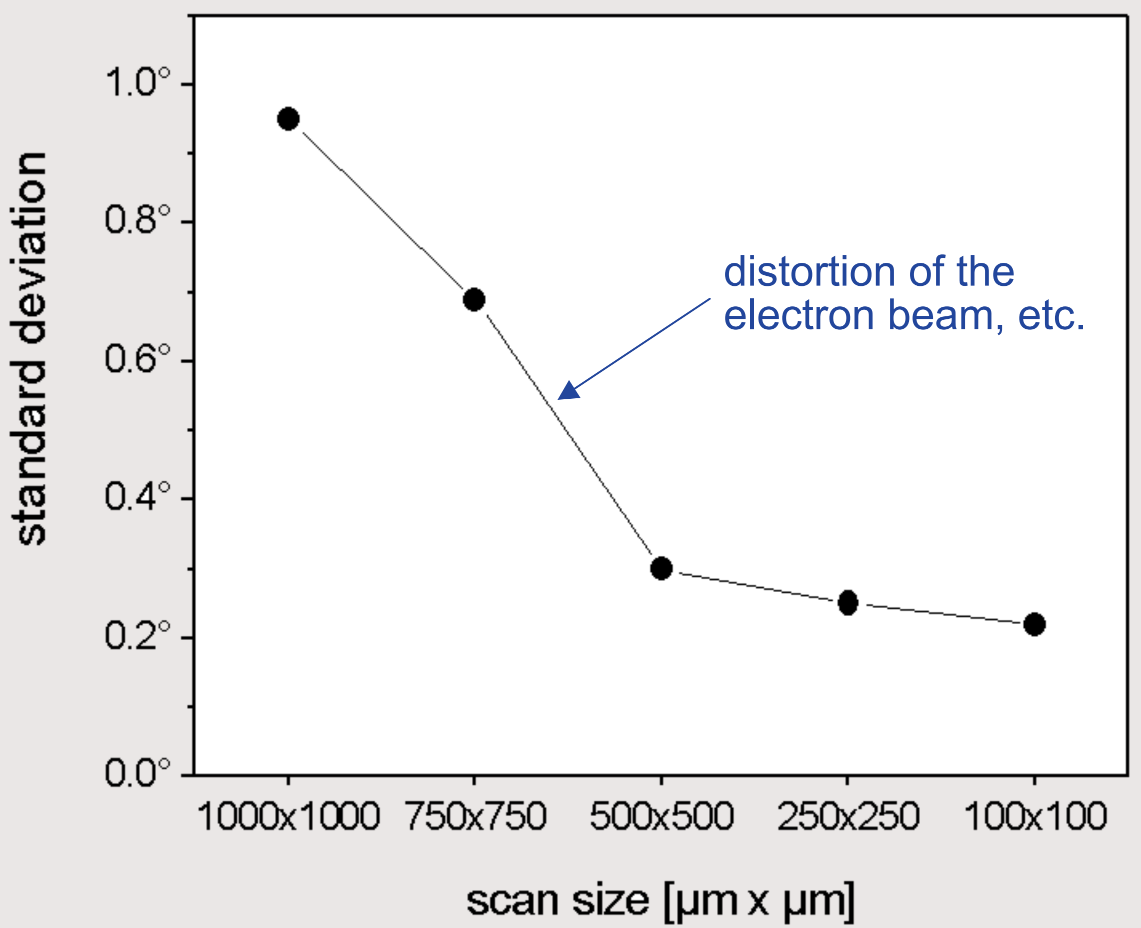
- accelerating voltage
  U: 15-20 kV
- beam current
  I: 1-10 nA
- tilting angle
  α: 65-70°
- working distance
  WD: 15-25 mm

(Hjelen and Nes, 1990)

Angular resolution of EBSD

• *relative accuracy:* the variation in orientations from a highly perfect single crystal is better than 1°

• *absolute accuracy* (including sample mounting, alignment, calibration, etc.): ~5°



EBSD Specimen Preparation

Specimen preparation is usually straightforward, often similar to that for optical microscopy

Main requirement: top 10-50nm of the specimen must be representative of the sample, i.e. no mechanical damage (e.g. grinding), surface layers (e.g. oxides, most coatings), or contamination.

Standard: mounting, grinding and polishing (electropolishing) and/or etching (final polishing in colloidal silica)

etchants

- mild steel: swab with 2% nital for several seconds.
- aluminium-alloys: immerse for several seconds in Keller's reagent, slightly warmed.
- commercially pure aluminium, titanium alloys: electropolish in 5% perchloric acid in ethanol at -25°C (or Struers A2).
- many rocks and minerals: diamond polish block specimens followed by colloidal silica polishing for several hours.
- polysilicon: wash in detergent, immerse for 1 minute in 10% hydrofluoric acid

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- **Orientation Mapping / Orientation Microscopy**
  experimental orientation microscopy, analysis of orientation maps

Orientation Microscopy and Orientation Mapping

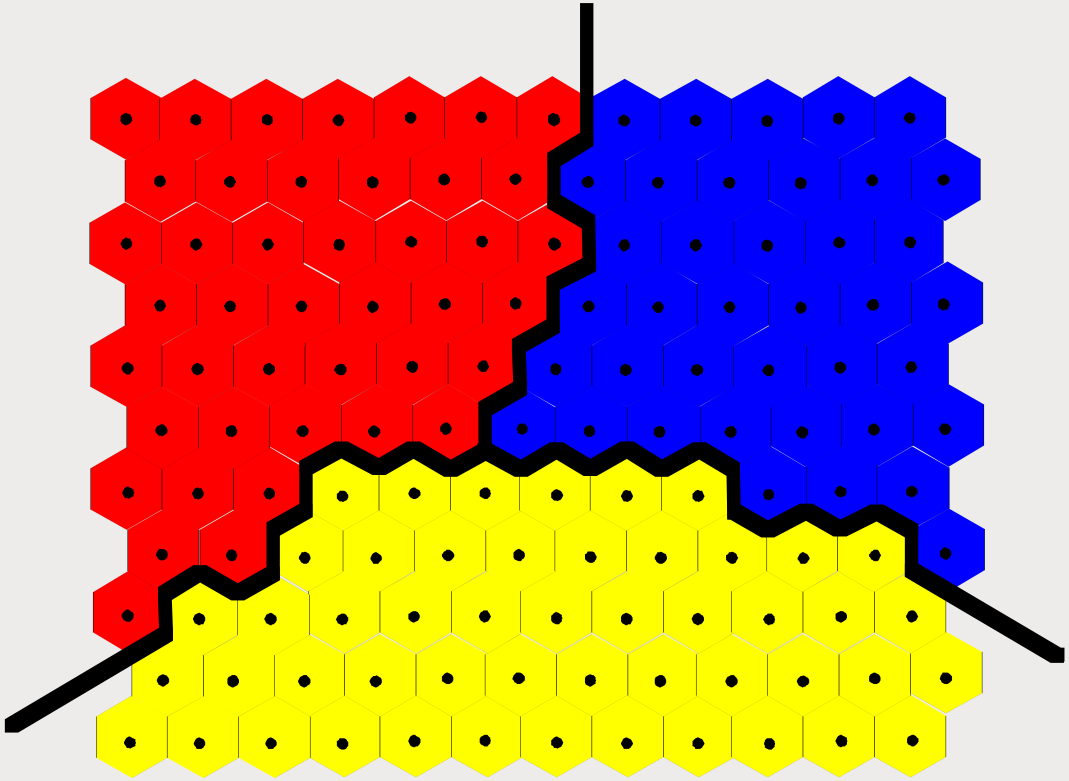
Orientation Microscopy

automated single grain
orientation measurements
in a pre-defined raster

Orientation Mapping

representation of the
orientation topography
as obtained through
orientation microscopy

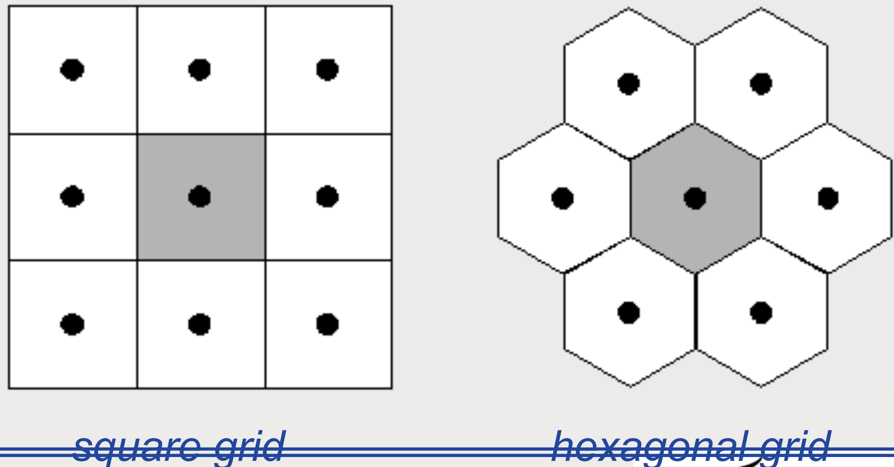
orientation imaging microscopy,
OIM™, Adams et al., 1993



principle

Orientation Microscopy

- *Stage control*: the sample is moved under a fixed electron beam (x/y/z or eucentric x/y-table). Measuring rates up to $1s^{-1}$. Simple, no calibration or focusing problems ⇒ well suited for large samples / low magnifications
- *Beam control*: electron beam is controlled to raster the (stationary) sample. No mechanical movements, precise. Very fast, measuring rates $>10s^{-1}$. Problems with calibration, focusing, etc.
- *Combination*
  "map stitching"
  (Channel 5, HKL Technology)



Scanning speed, step width

Scanning Speed

speed	0.1/s	500 x500=$2.5\cdot10^5$ patterns in 29 days
	1/s	~3 days
	10/s	7 h
	50/s	1:25 h

Step width

minimum: resolution of EBSD, i.e. ~0.1µm

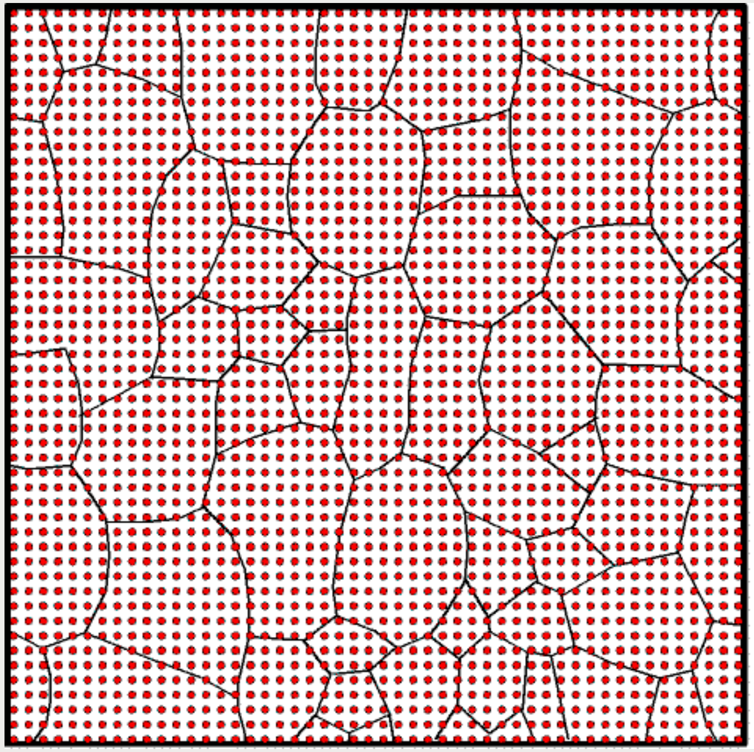
however: dependent on scientific question often too fine, i.e. too long measuring times

good grain maps require 5 - 10 points per grain size, i.e. 25 - 100 points per grain

high resolution of grain boundary details: more points per grain

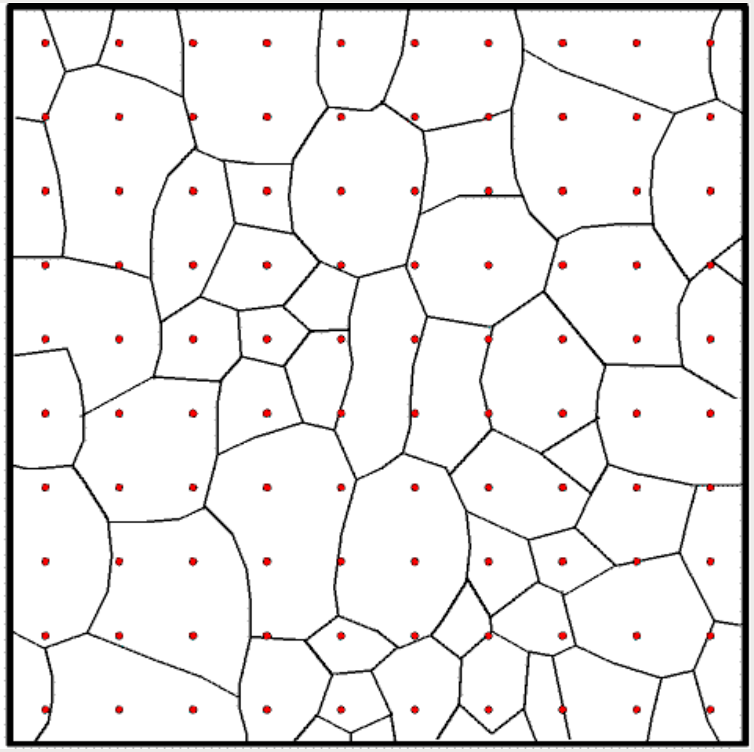
improved grain statistics (texture) : ~1 point per grain

Step width



50 x 50 = 2500 points
i.e. ~ 50 points / grain

detailed maps, yet slow



~ 50 grains

10 x 10 = 100 points
i.e. ~ 2 points / grain

25 times faster, sufficient grain statistics,
yet low resolution map

Orientation Microscopy

Output

- header
(sample, material, calibration data, SEM settings, etc.)

- for each point *i*: position, orientation, pattern quality, reliability data, ...

→ Orientation Mapping

AlLi15U.ang - Editor

Datei Bearbeiten Format ?

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# OIMDirectory           /usr/OIM
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# y-star                 340
# z-star                 338
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# Symmetry               43
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# hklFamilies             1  1  1
# hklFamilies             2  0  0
# hklFamilies             2  2  0
# hklFamilies             3  1  1
#
#   phi1      PHI     phi2        X        Y     IQ     CI
#
  12.566   12.566   12.566    0.000    0.000   43.3  0.000
   6.278    0.309    1.158    1.000    0.000  130.1  0.200
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   0.004    0.310    1.132    3.000    0.000  141.8  0.400
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   0.177    0.548    0.950    5.000    0.000  154.9  0.600
   0.179    0.555    0.937    6.000    0.000  192.4  0.629
   0.180    0.551    0.936    7.000    0.000  190.3  0.686
   0.176    0.552    0.938    8.000    0.000  160.8  0.714
  12.566   12.566   12.566    9.000    0.000   66.6  0.000
   1.075    0.106    6.228   10.000    0.000  113.7  0.143
   1.140    0.110    6.153   11.000    0.000  127.5  0.286
   1.115    0.106    6.180   12.000    0.000  117.4  0.343
  12.566   12.566   12.566   13.000    0.000   82.5  0.000
   3.582    0.718    4.809   14.000    0.000  112.7  0.029
   6.240    0.275    1.100   15.000    0.000  116.2  0.143
   6.257    0.262    1.094   16.000    0.000  144.1  0.486
   6.253    0.262    1.097   17.000    0.000  156.8  0.743
   6.258    0.255    1.086   18.000    0.000  152.4  0.800
   2.054    1.002    0.361   19.000    0.000  107.3  0.200
   2.180    0.411    5.202   20.000    0.000  104.8  0.050
   2.415    0.252    5.767   21.000    0.000  138.9  0.114
   2.425    0.256    5.759   22.000    0.000  177.0  0.343
   2.428    0.259    5.761   23.000    0.000  195.3  0.686
```

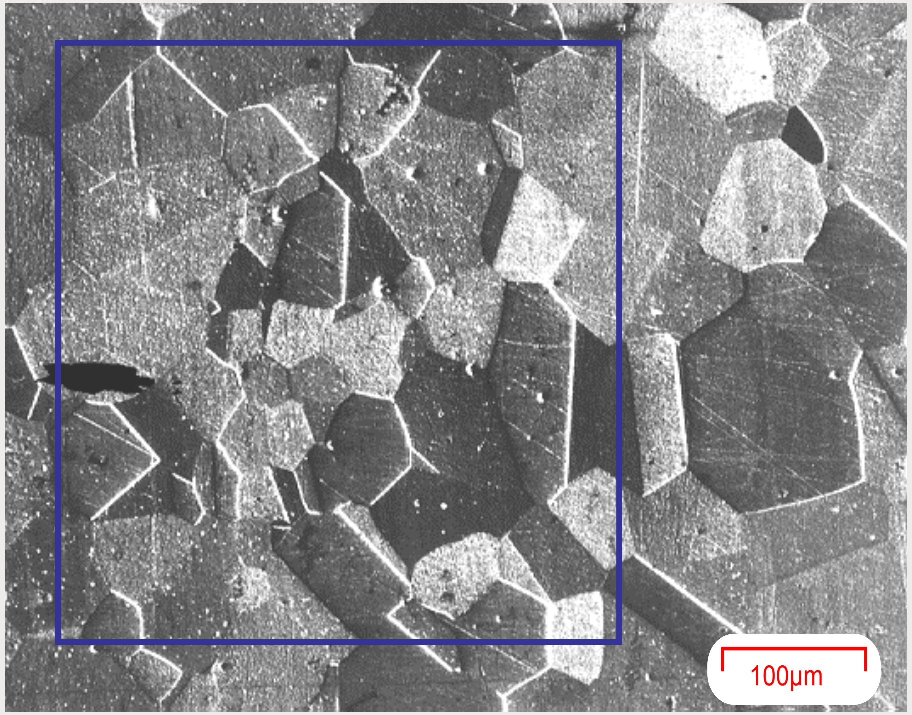
Orientation Microscopy and Orientation Mapping

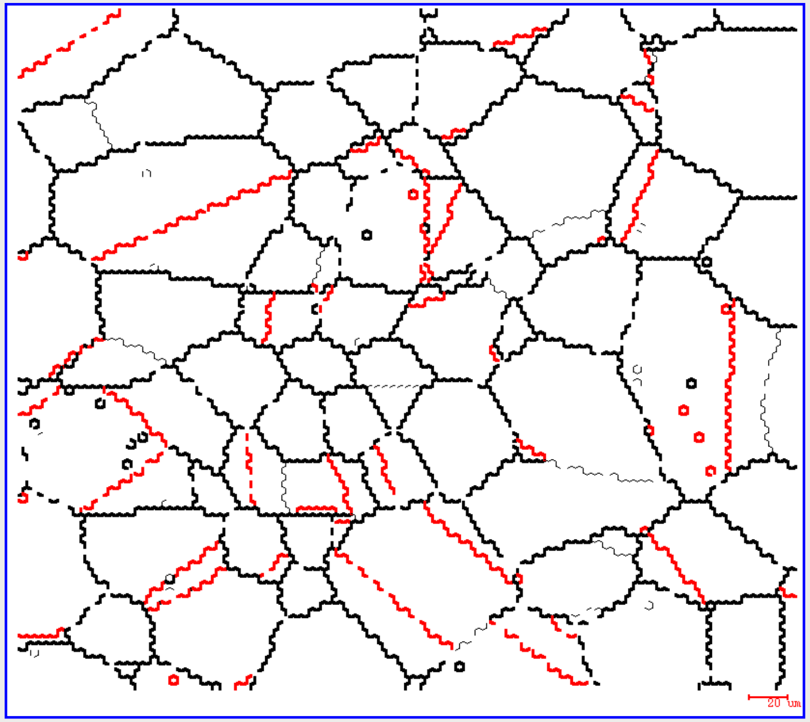
Evaluation Software (Mapping)

- Maps
  orientation, IQ, Taylor-factor, grain size, ...
- boundaries
  HAGB, LAGB, twins, CSL, ...
- orientation distribution
  pole figures, inv. pole figures, ODFs, MODFs, ...
- quantitative evaluation
  histograms, grain size distribution, recrystallized volume fraction, ...

Grain boundary maps

example: (Ni_3Al + B, recrystallised) 400x400 µm², 4µm-steps





high-angle grain boundaries (HAGB): $\omega > 15°$

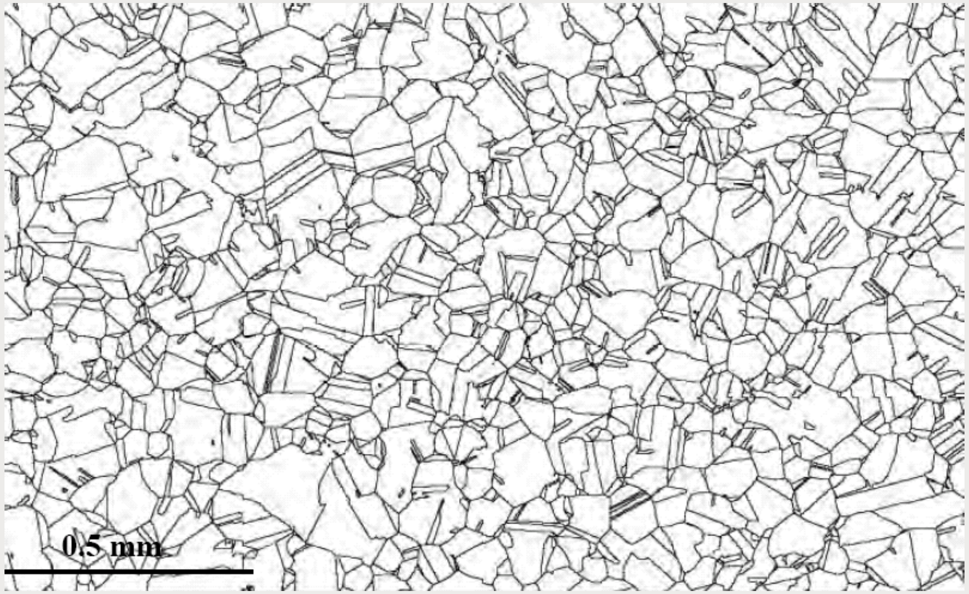
low-angle grain boundaries (LAGB): $5° < \omega < 15°$

twin grain boundaries ($\Sigma3$) : 60°<111>, $\Delta\omega < 5°$

Grain boundary maps

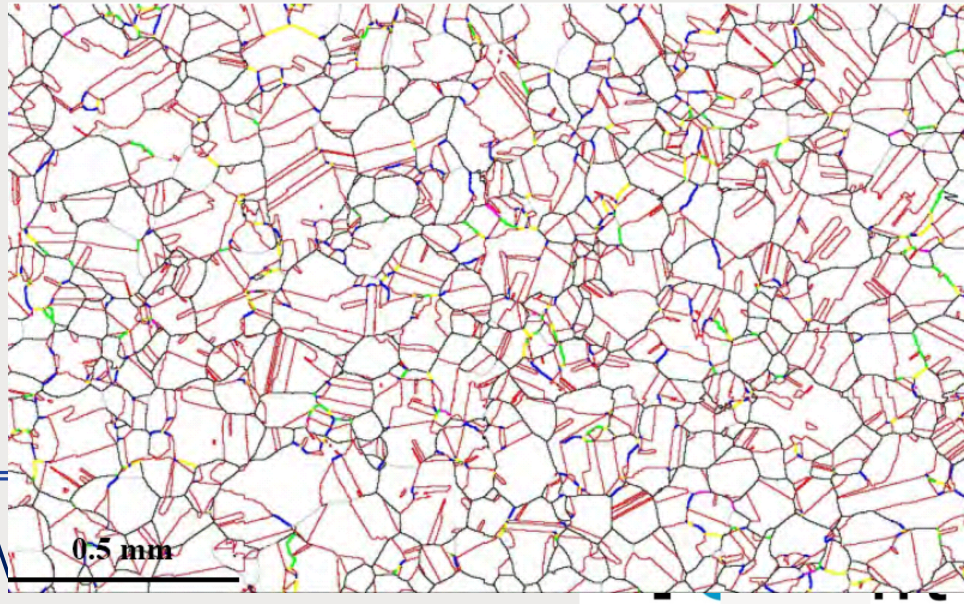
EBSD map of a recrystallized α-brass specimen (Randle 2004)

(a) all interfaces (high-angle grain boundaries) shown as black lines.



(b) special Σ boundaries highlighted

- red: Σ3, 1st gen. twin
- blue: Σ9, 2nd gen. twin
- yellow: Σ27, 3rd gen. twin
- green: Σ81, 4th gen. twin
- black: other boundaries

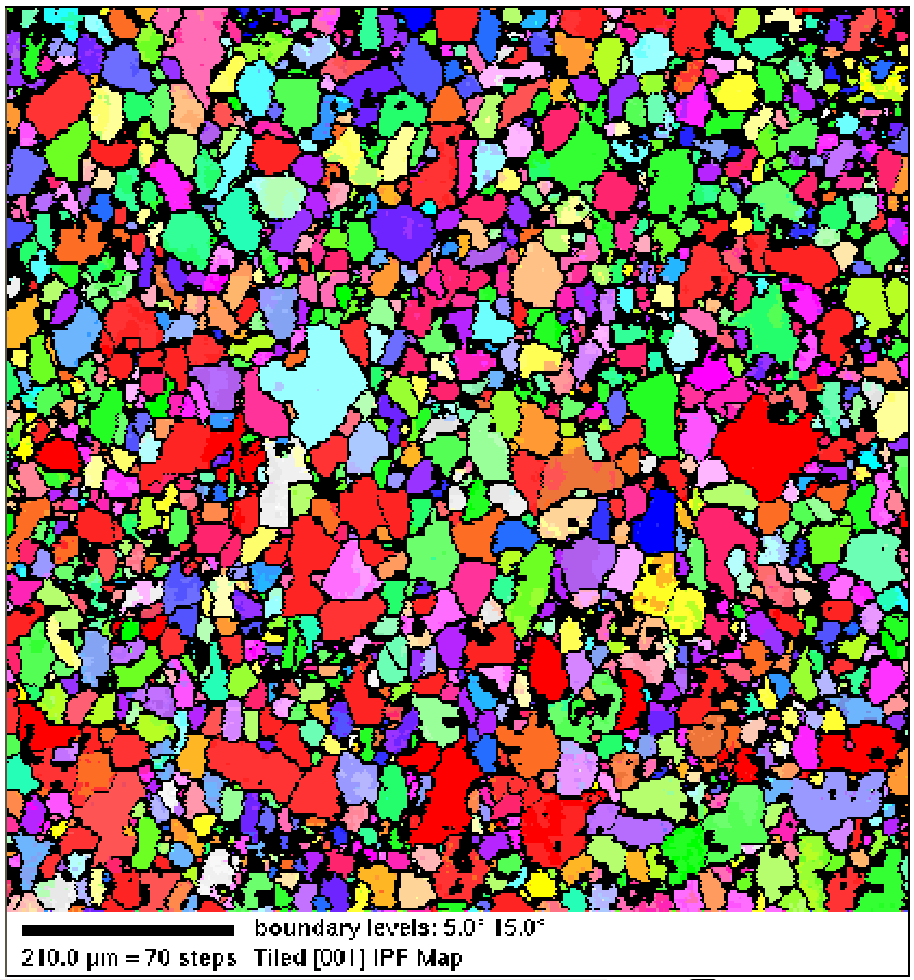


GBE: grain boundary engineering: increase density of "special boundaries" (usually, twins) to improve materials properties (corrosion, strength, ...)

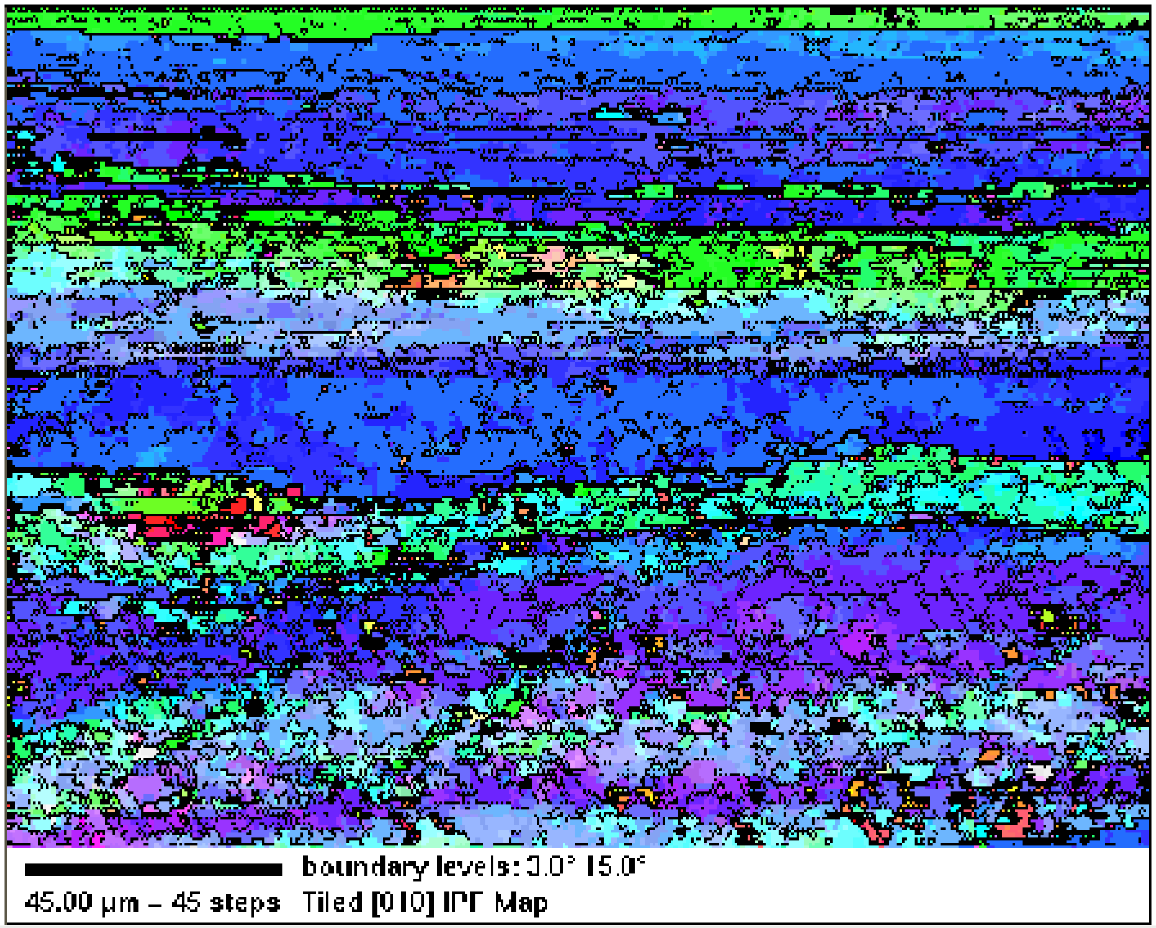
Orientation Maps

random choice of colouring

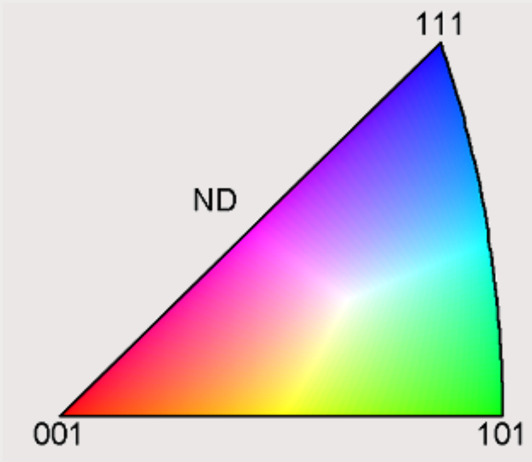
example:
recrystallised Al 1xxx alloy



Orientation Maps

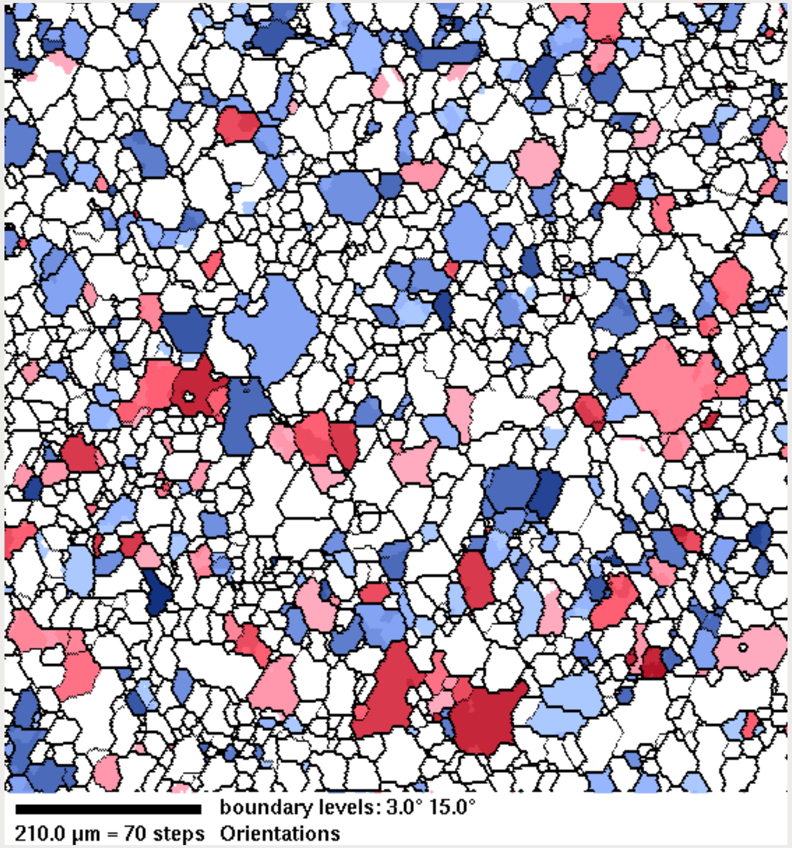


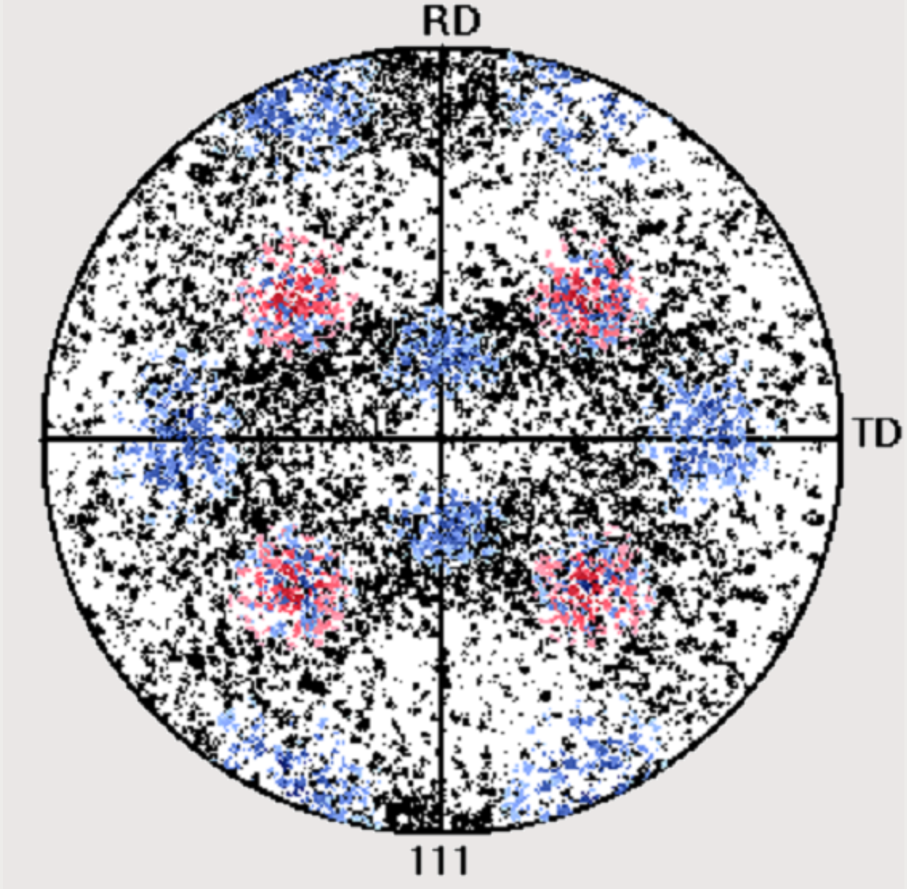
defined colour scheme (stereographic triangle, Euler angles, etc.)



example: cold rolled Al-3%Mg with shear bands

Orientation Maps





highlight specific orientations

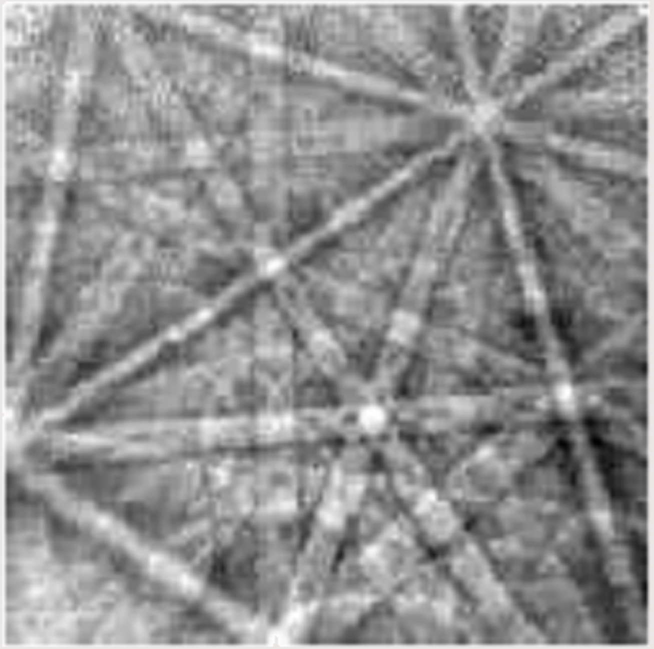
example: recrystallised Al 1xxx alloy, **Cube** and **R** oriented grains

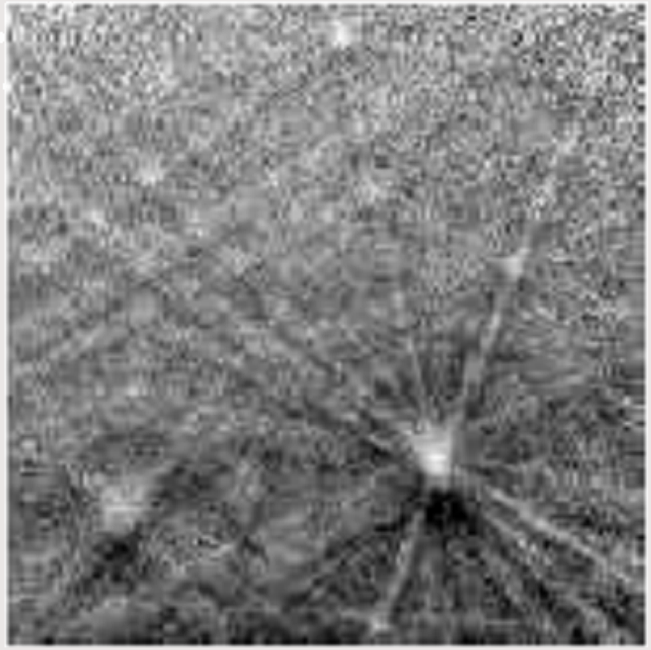
Pattern quality maps

Pattern (*image) quality* parameter, *IQ*: measure of the quality of an EBSD pattern.

IQ is dependent on the material and condition.

However, *IQ* is also a function of the technique and parameters used to index the pattern as well as other factors such as the video processing. Thus, do not use *IQ* as a quantitative measure of strain, etc.

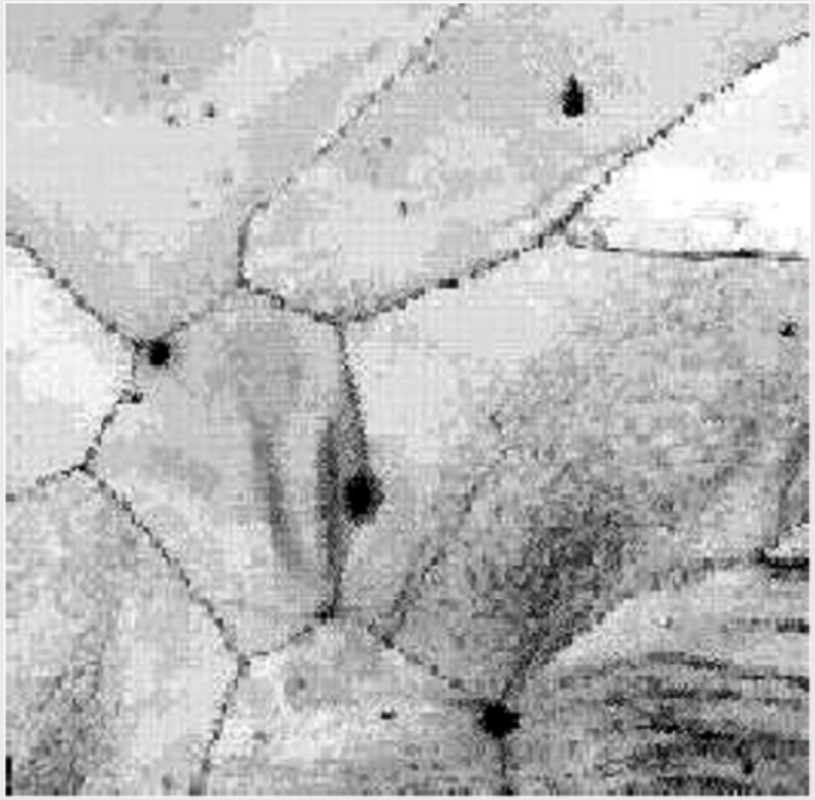




Pattern quality maps

example of an OIM map with *IQ*

pixels are shaded according to IQ
high IQ: bright
low IQ: dark



- **IQ reveals grain boundaries (no or poor patterns)**
- **clusters of non-indexed patterns imply precipitates**
- **gradients in IQ imply dislocation structures**

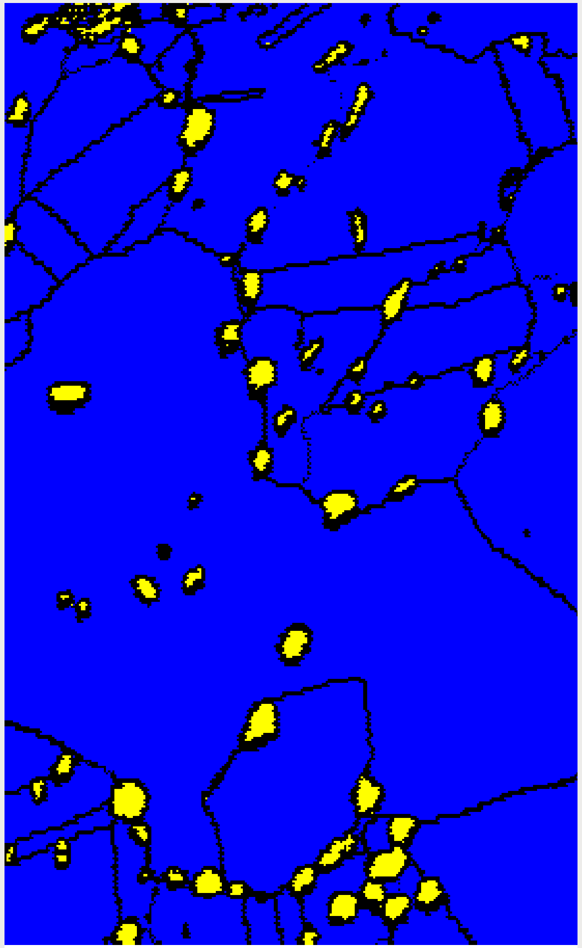
Phase maps, pattern quality maps

phase maps

image quality maps

example:

2-phase Ni-W



phase

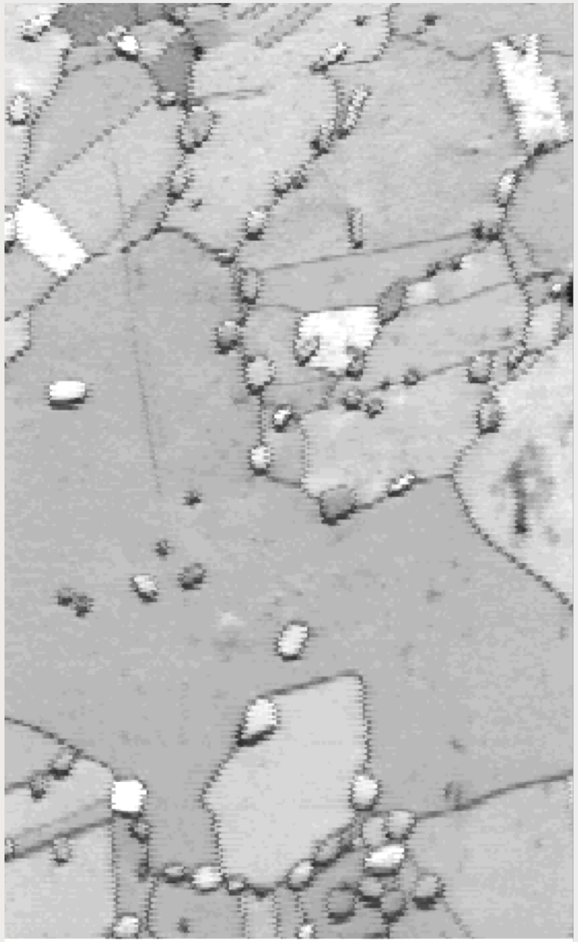


image quality

Measurements of microtexture – Summation

Single grain orientation measurements in the SEM

Kossel / SAC: today virtually obsolete for routine single grain orientation measurements
EBSD: the work-horse technique: fast, versatile, reliable, accurate, easy to use

expanding range of applications:
• improved spatial resolution: → TEM
• improved scanning speed → XRD

Orientation microscopy / orientation mapping

⊕ *visualisation* aspect
⊕ total *quantification* of the orientation aspects
⊕ true grain *morphology* (grain size and shape)

∅ inefficient SEM usage, large amounts of redundant data
∅ black box, problems with data evaluation