Parte 4

TEXTURE ANALYSIS OF METALS IN MICRO-VERSION : THE ELECTRON BACKSCATTER DIFFRACTION TECHNIQUE

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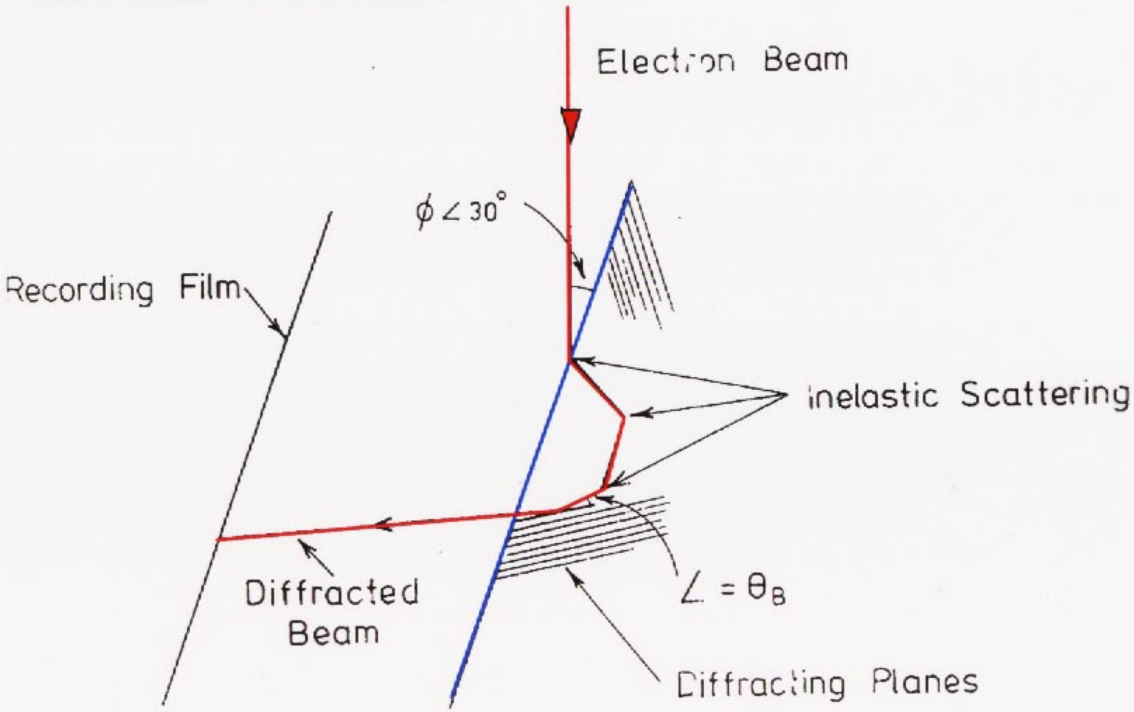
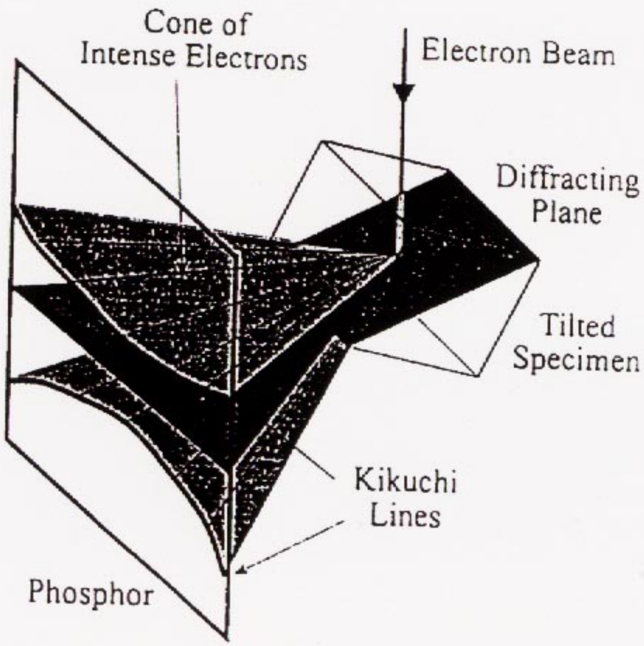
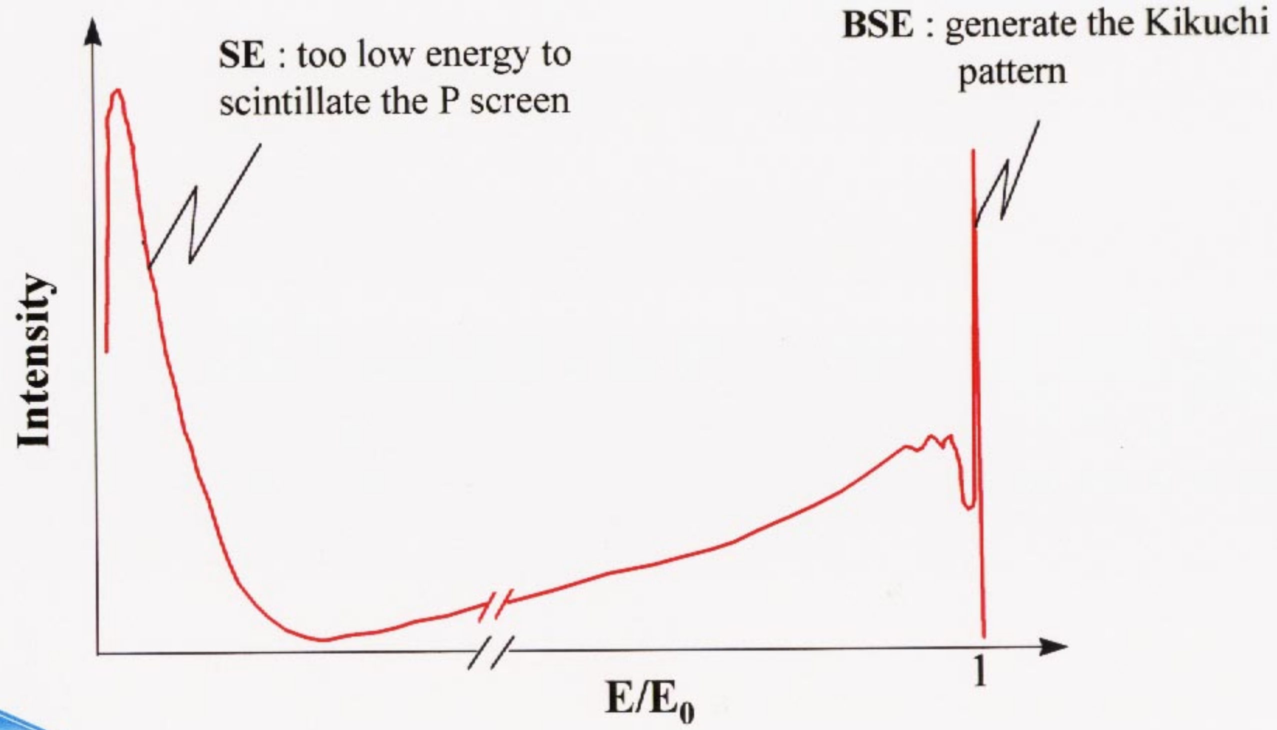
The Kikuchi Pattern → Electron BackScattering Diffraction (EBSD)



Kikuchi Lines = Image of crystal planes





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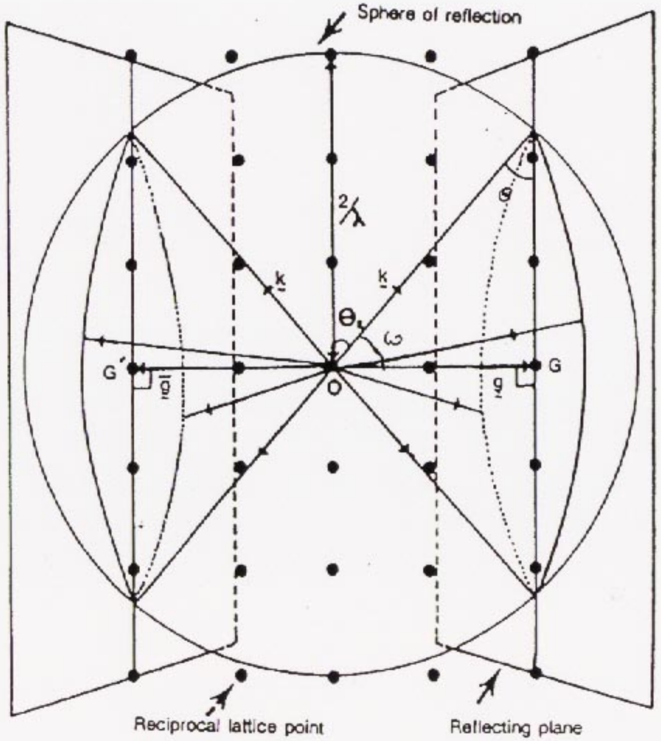
The formation of Kikuchi Lines



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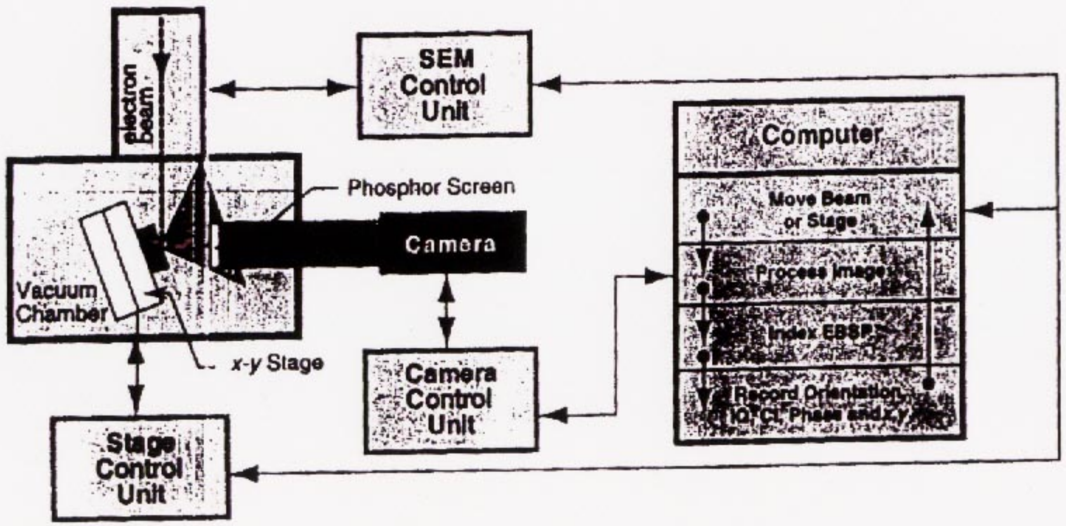
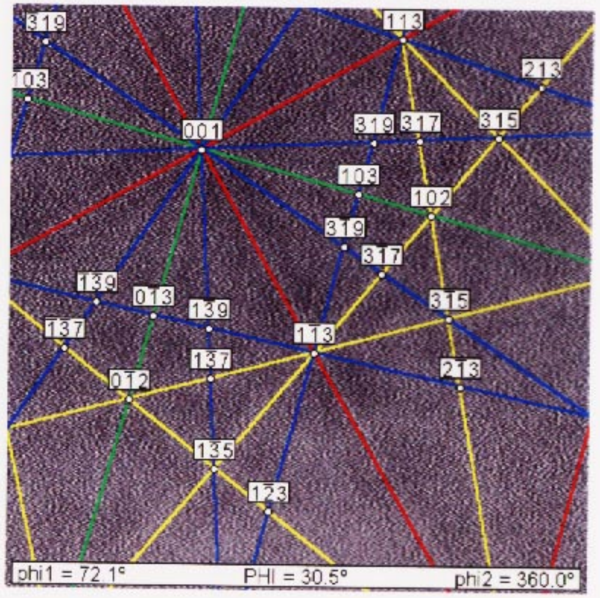
Orientation Imaging Microscopy

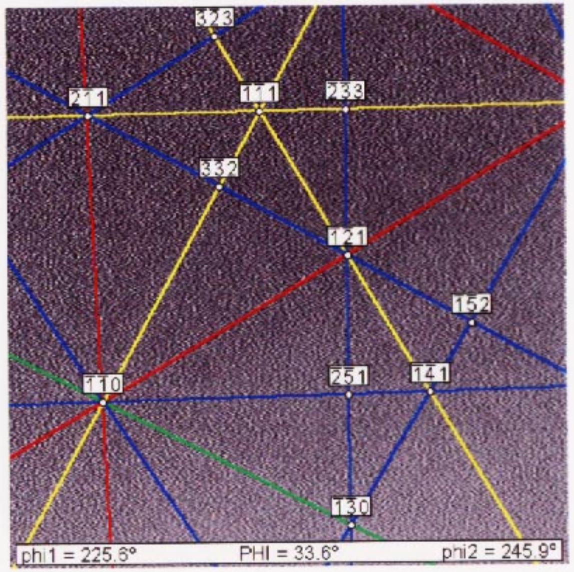
= Automated EBSD acquisition + post processing of data



Example of experimental EBSD pattern (BCC steel)

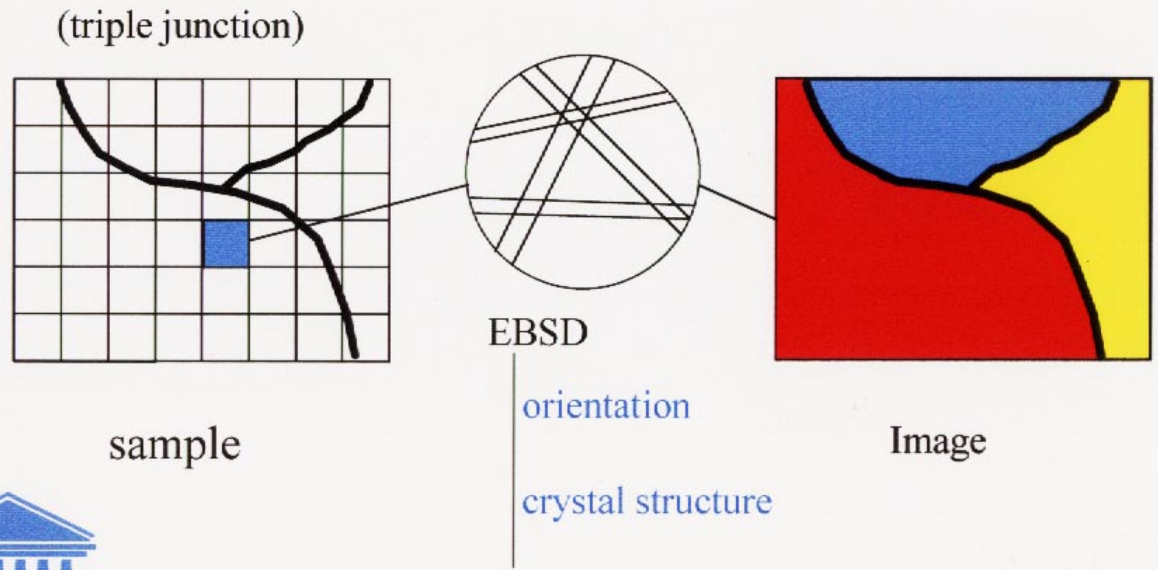


Example of experimental EBSD pattern
(residual austenite in TRIP steel)



***O**rientation **I**maging **M**icroscopy (OIM)*

= automated scanning version of *Electron Backscattering Diffraction (EBSD)*



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OIM image contains crystallographic information of sample surface

- crystal structure (bcc, fcc, hcp)
- crystal orientation
- crystal defects: dislocaton substructure
  residual stresses
  grain boundaries

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Image Quality Factor (IQ)

- Quantifies the band contrast of the EBSD pattern
- Depends on sample preparation and operation mode
- Also depends on material parameters :
  crystal and structural defects !

Confidence Index (CI)

- Quantifies the reliability of the pattern indexing
- $0 < CI < 1$
- $CI = \dfrac{V_1 - V_2}{V_{total}}$ with V1 = 1st solution
  V2 = 2nd solution
  Vtot = total number of solution

Operational mode of microscope for OIM

AV = 20 kV
Specimen tilt = 70deg
Spot size = 5-7
Dynamic Focus
(Specimen Tilt Correction)

Hardware parts of OIM system

Phosphor screen + camera
Image processor (frame integration, background correction)
Beam Controler
PC (>200 MHz)

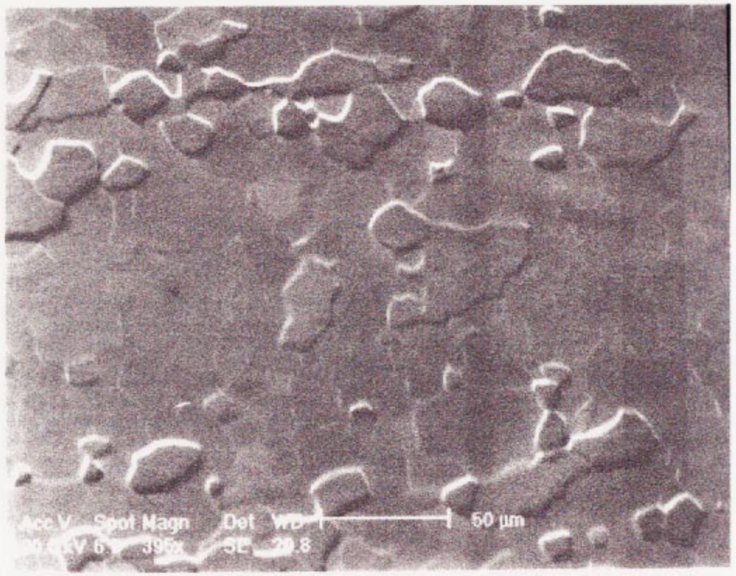
Acquisition Time

Quality of Diffraction Pattern
Sample preparation
Numbers of Integrated Frames
Crystal Structure(s) present in sample

E.g.: Steel sample (fully recrystallized, single phase)
$\approx$ 0.5 - 1.0 s per pixel
Typical scan $\approx$40.000 pixels $\longrightarrow$ 6 - 12 h

ULC steel (CEIT) : 90% cold rolled + annealed

(ferrite single phase polycrystal structure)



SE image on ESEM XL30

OIM image of same sample site

Greyscale defined by image quality factor of diffraction pattern

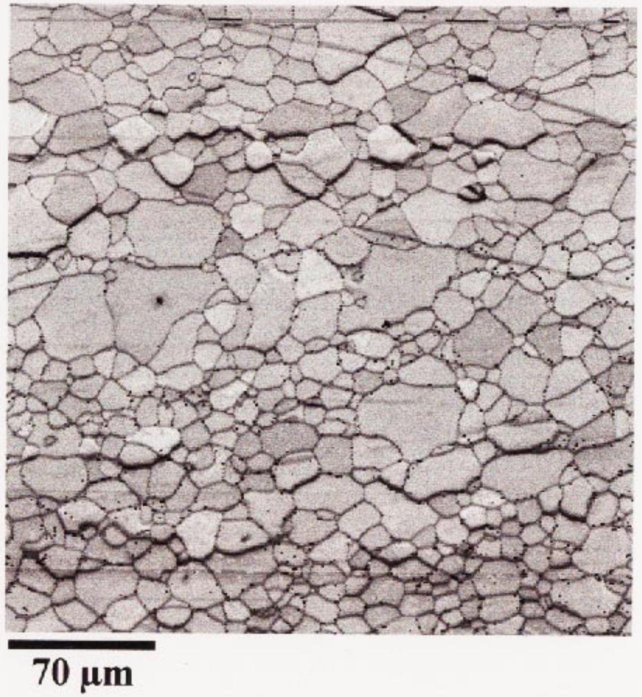


Image Quality
1.74 - 174.20

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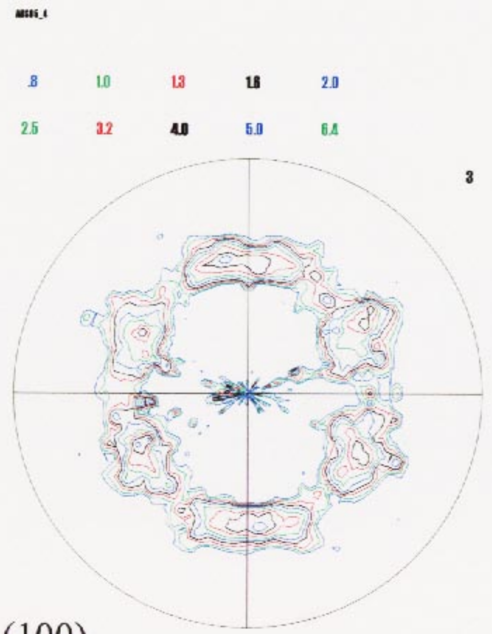
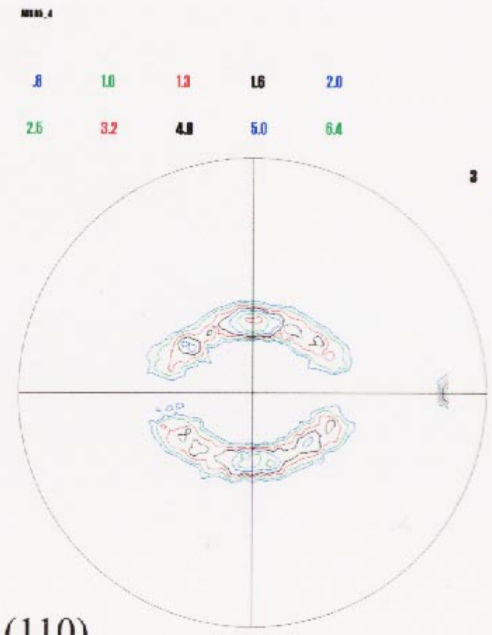
Pole figures obtained by X-ray diffraction



(100)



(110)

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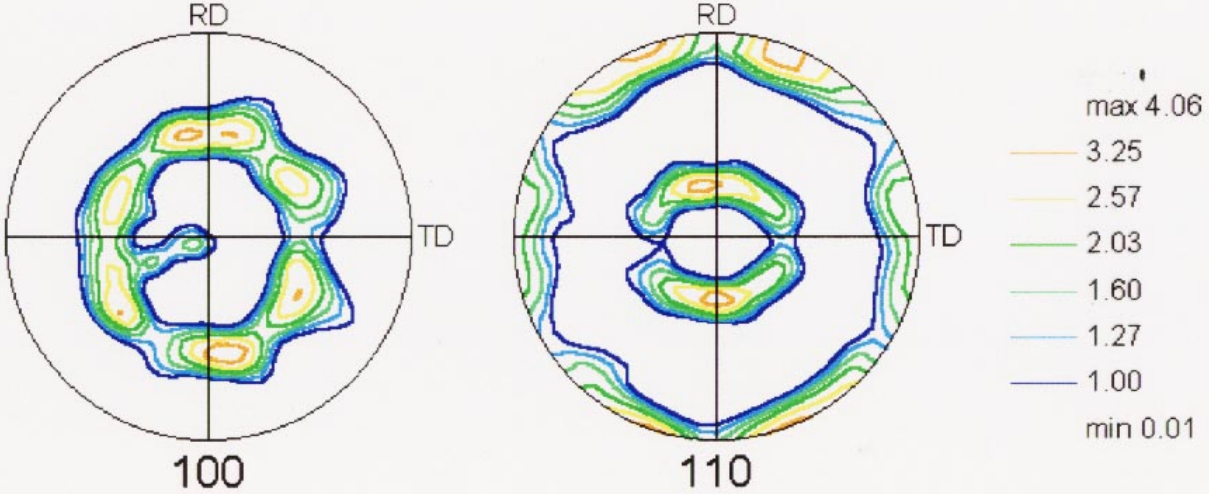
Pole figures obtained by OIM



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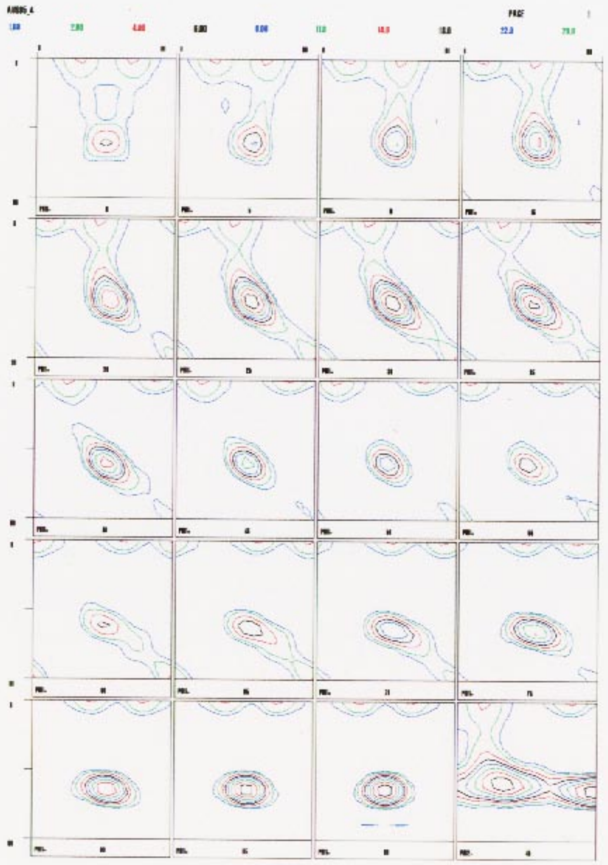

ODF obtained from X-ray diffraction measurement

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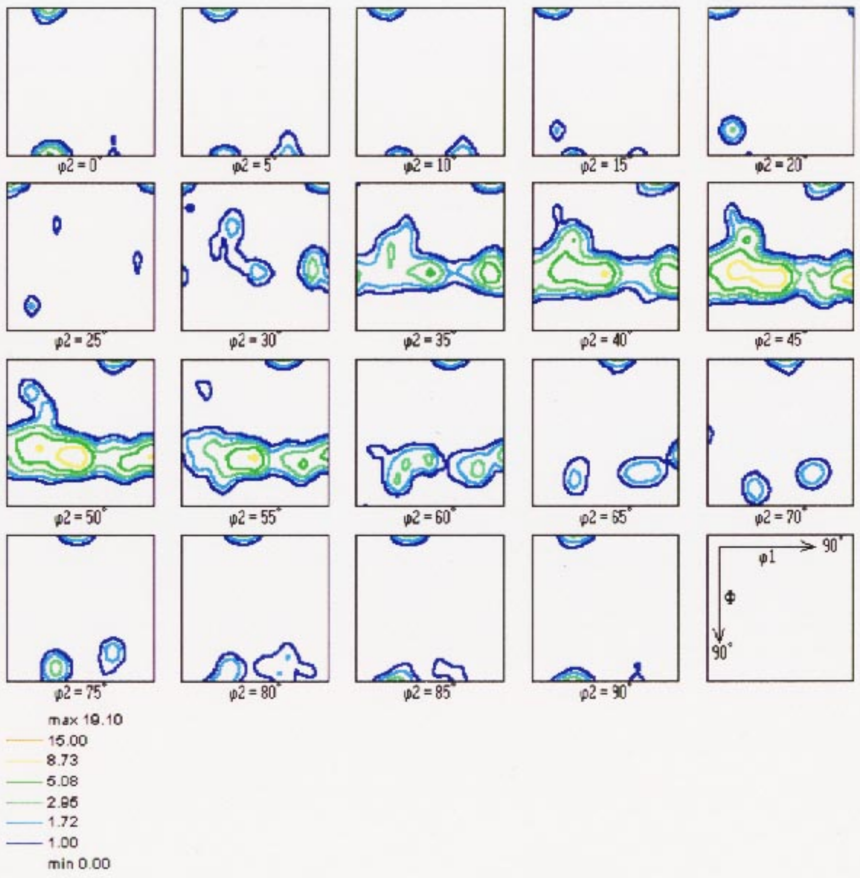
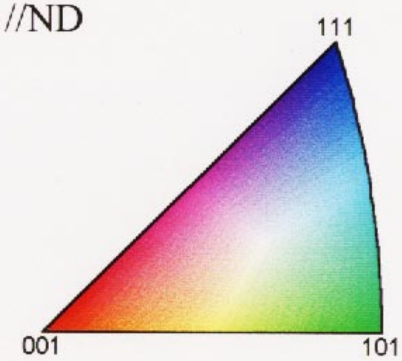


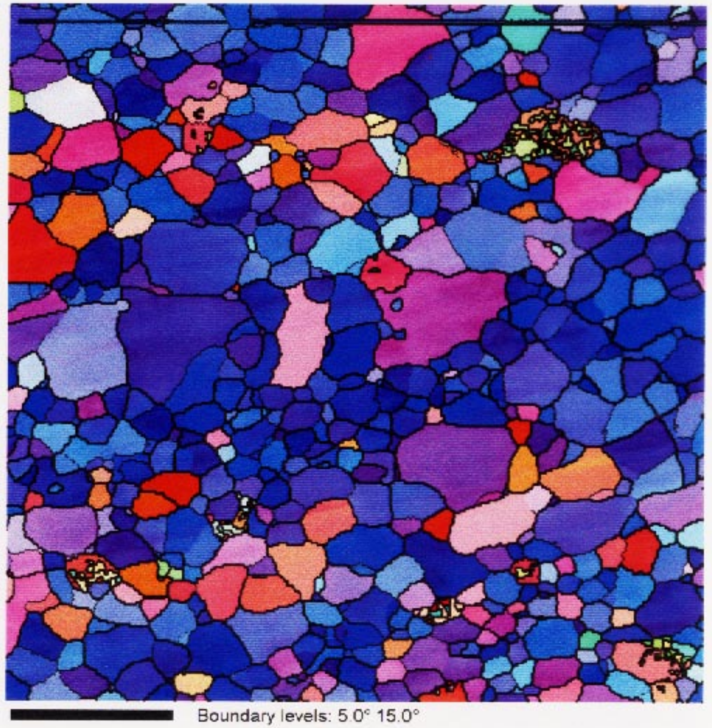
ODF obtained from OIM measurement as calculated by TSL software

OIM image of same sample site

Colors defined by orientation of crystal direction // ND

//ND





Boundary levels: 5.0° 15.0°

70 µm

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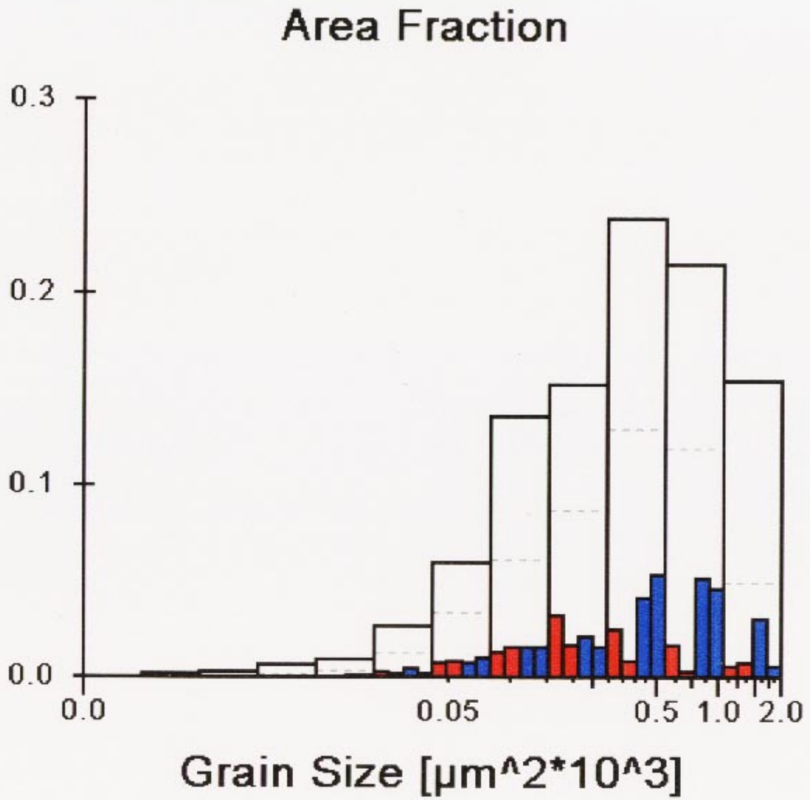
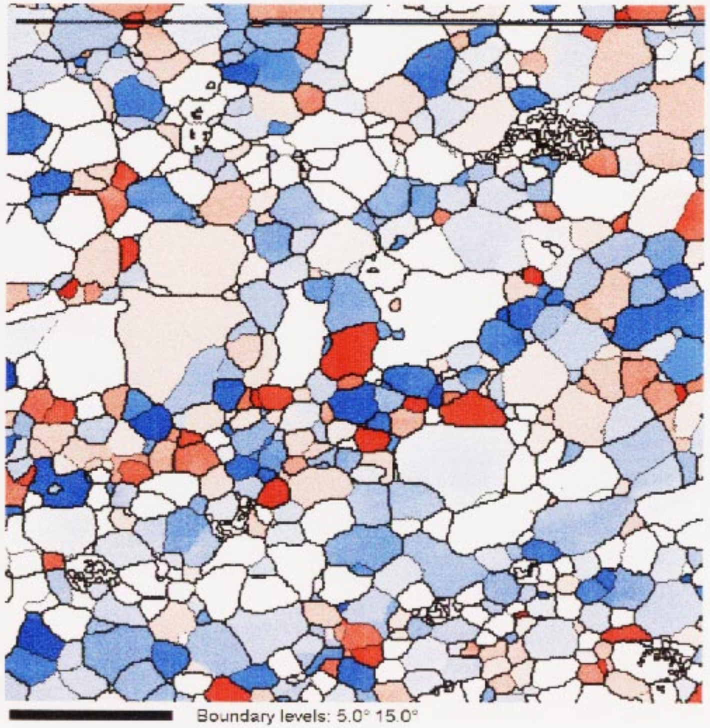
Grain Size Distribution



blue = {111}<211>

red = {111}<110>

Presence of Specific Orientations in the Microstructure

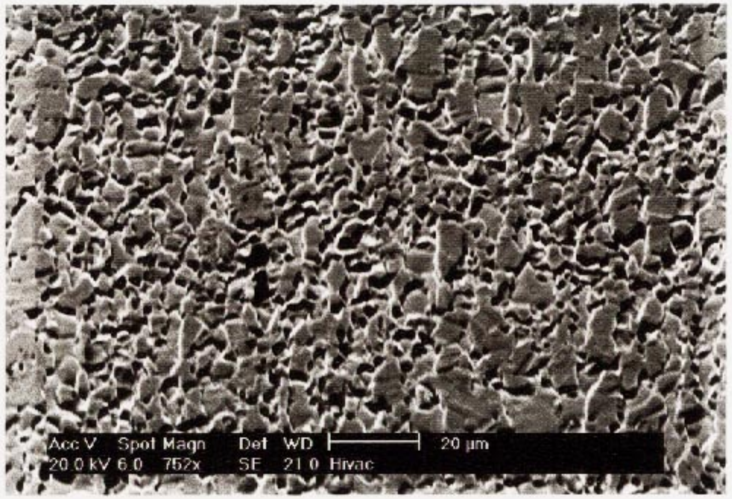


70 µm

blue = {111}<211>
(43% volume fraction)

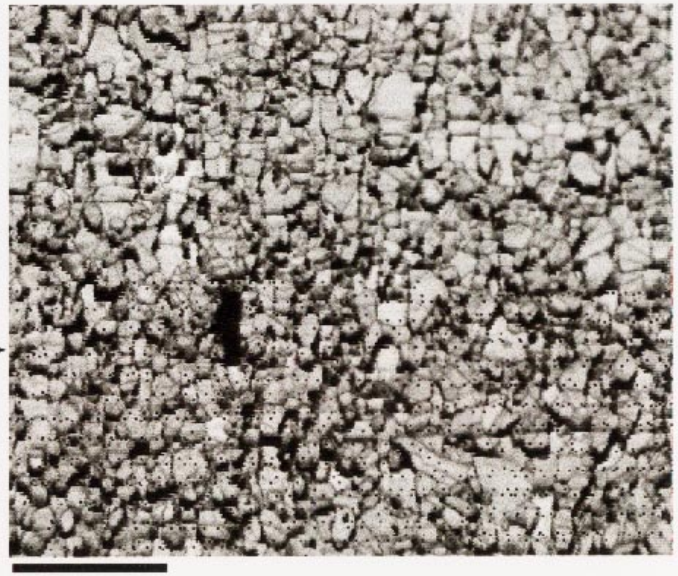
red = {111}<110>
(27% volume fraction)

Duplex Stainless Steel (Ferrite + Austenite)



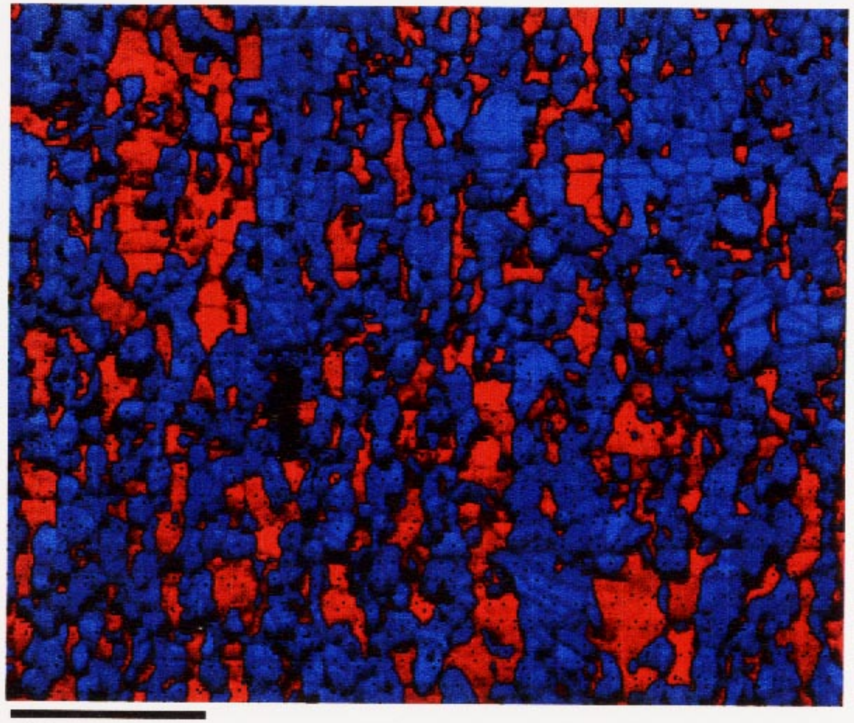
← SE image in SEM

OIM image
Greyscale = Image Quality factor →



35 µm

Phase Distribution in Duplex Stainless Steel



blue = austenite
(71% volume fraction)

red = ferrite
(29% volume fraction)

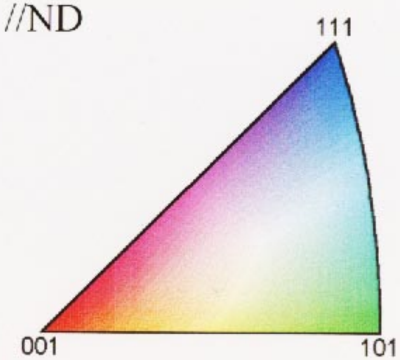
Texture of Ferrite Phase

35 µm

—— $5° < \omega < 15°$

—— $15° < \omega < 180°$

//ND



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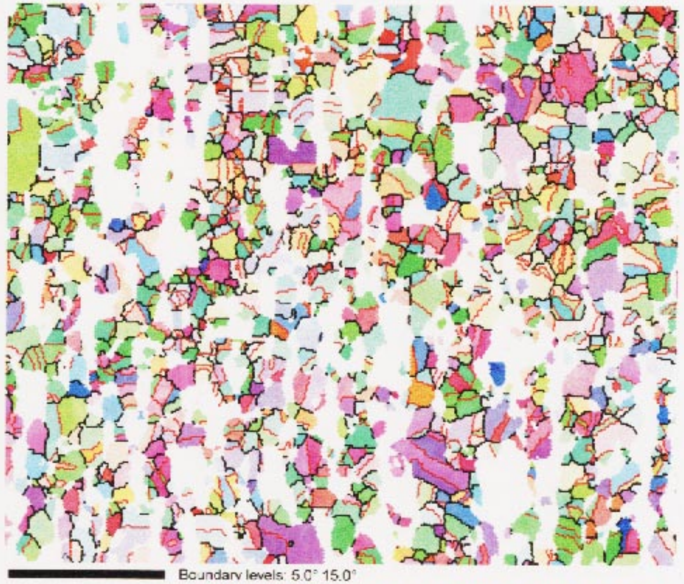
Texture of Austenite Phase



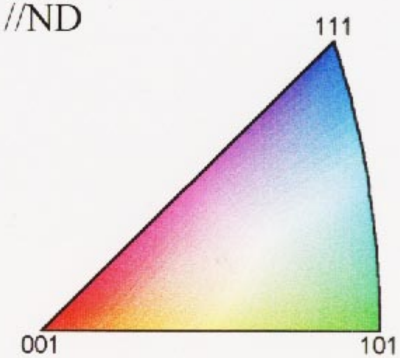
35 µm

—— $5° < \omega < 15°$

—— $15° < \omega < 180°$

—— twin misorientation

//ND



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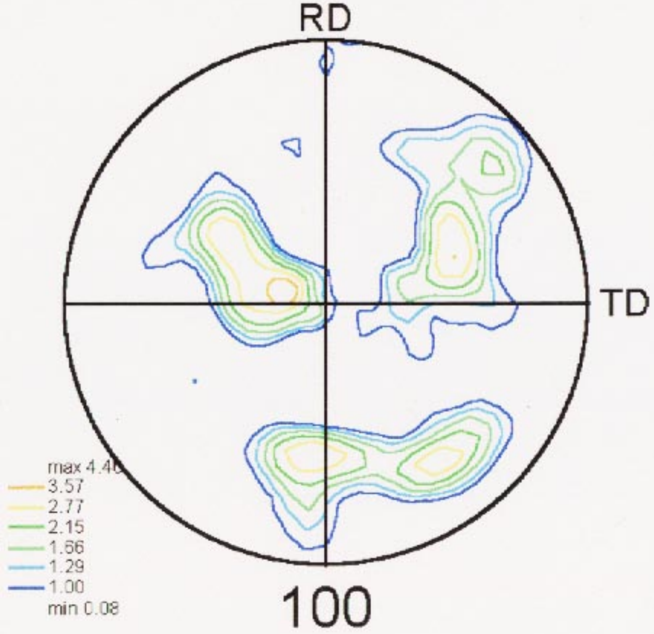
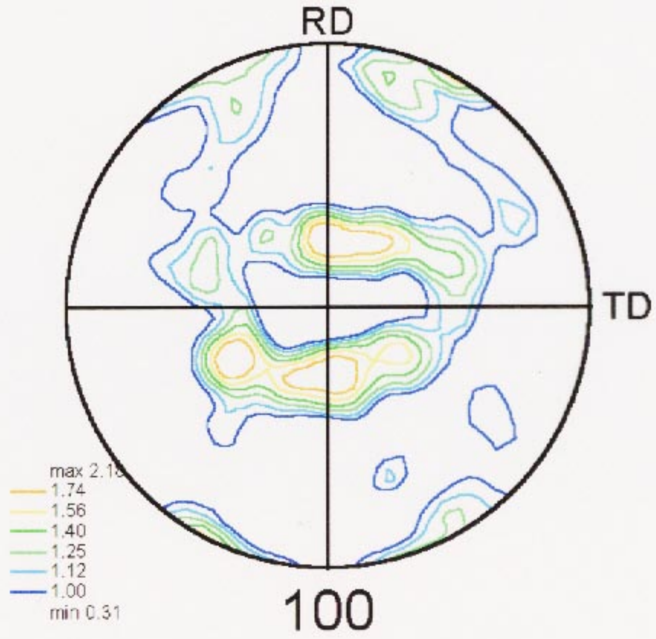
Comparison of Austenite and Ferrite Texture

Ferrite

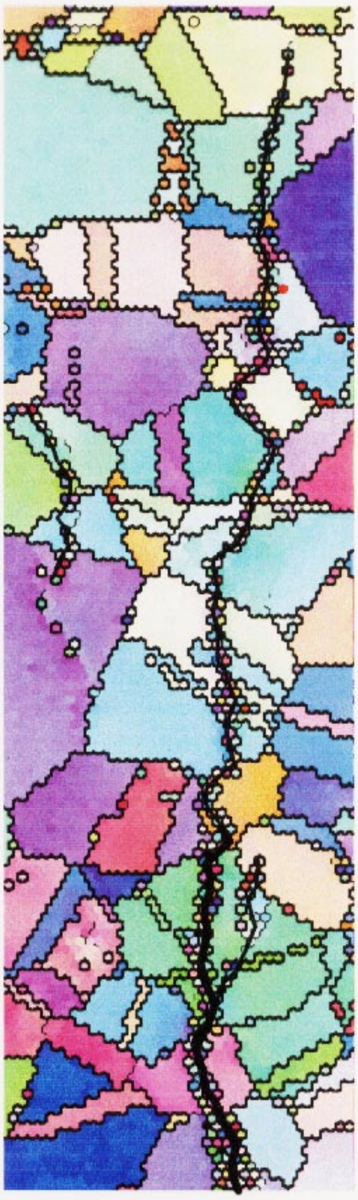


Austenite



Microcrack in Aluminium

(courtesy: TexSem ltd.)



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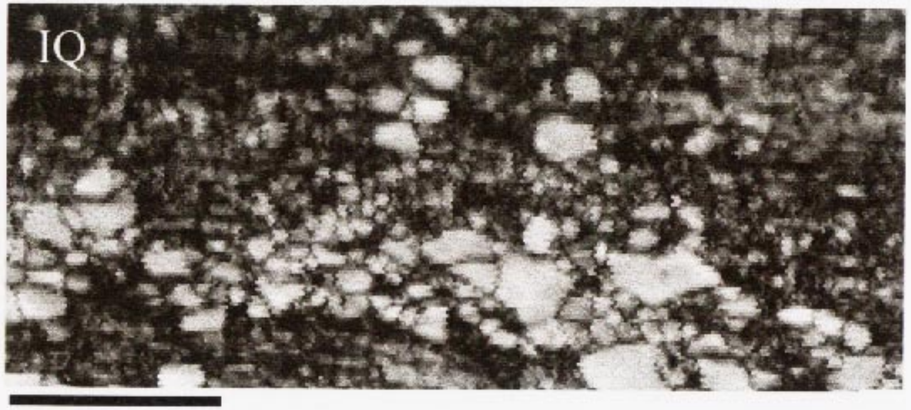
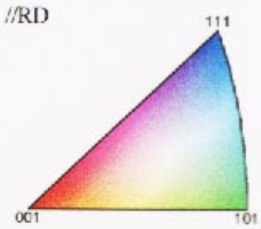
Partially recrystallized steel sample

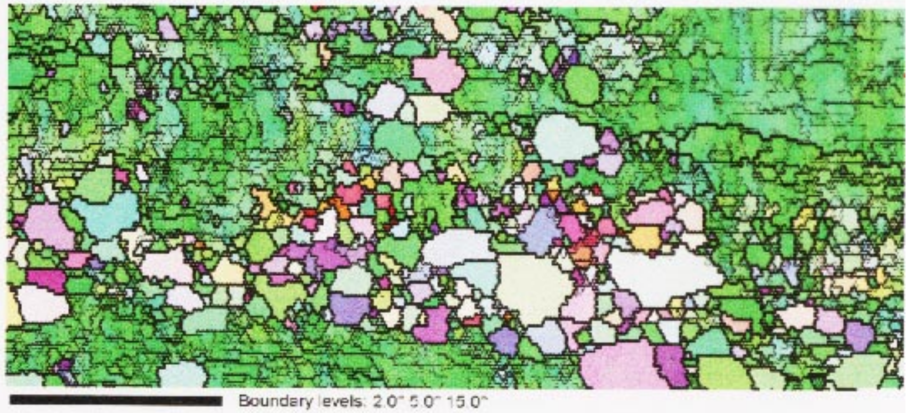


20 μm

RD

ND

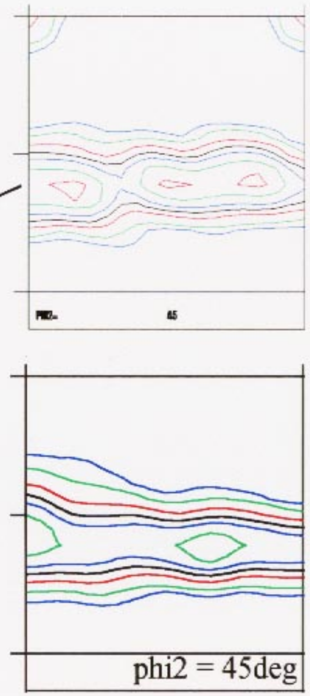




OIM scan of Early Nucleation Structure

RD

TD



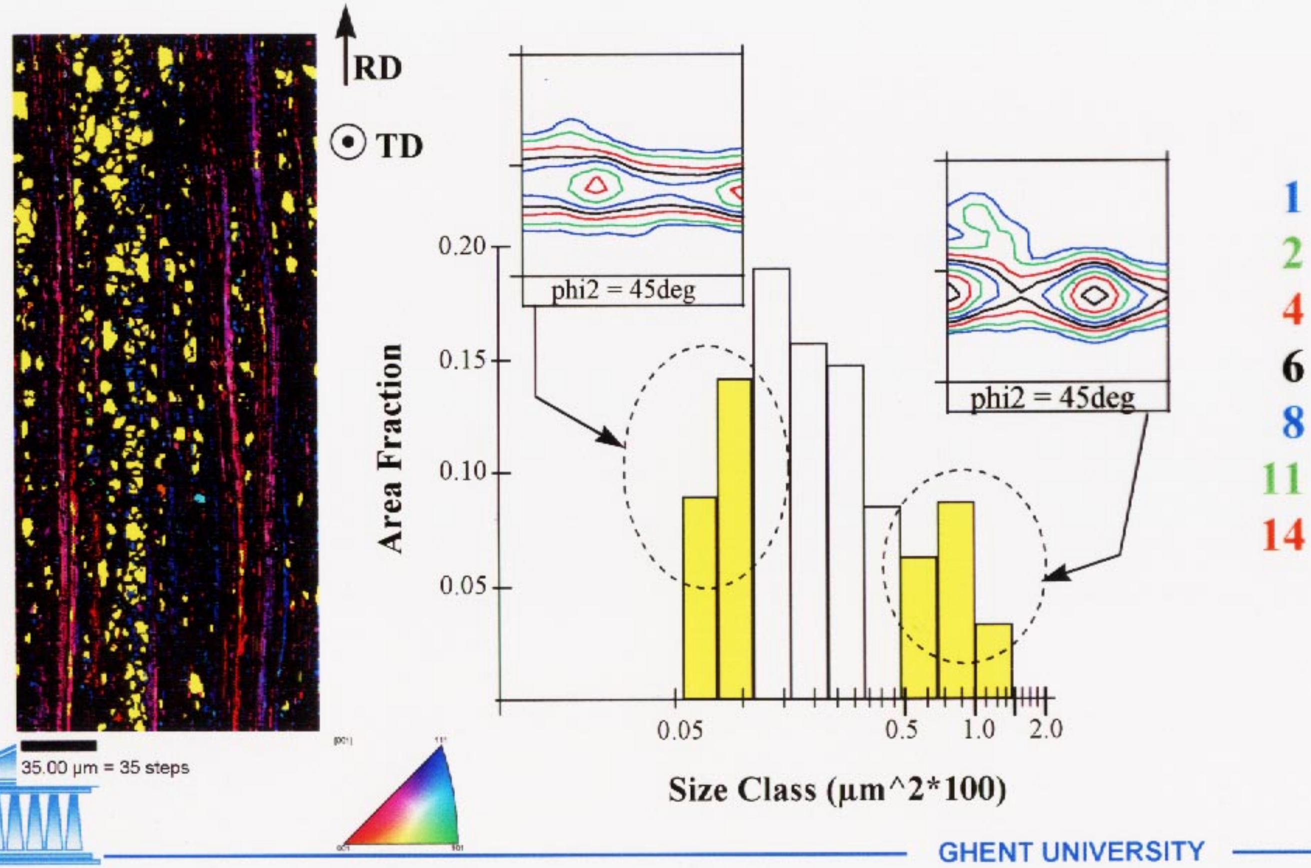
Nucleation Texture and corresponding microstructure

Average of 4 different OIM scans

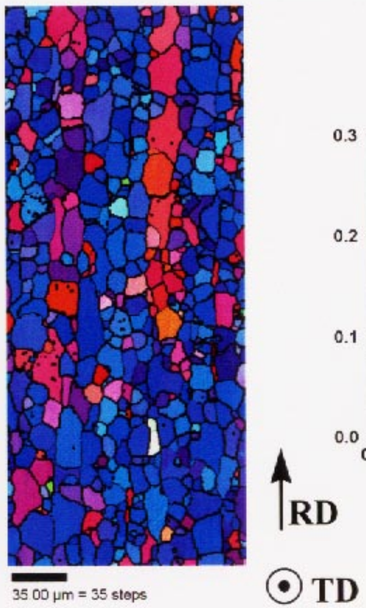
1
2
4
6
8
11
14

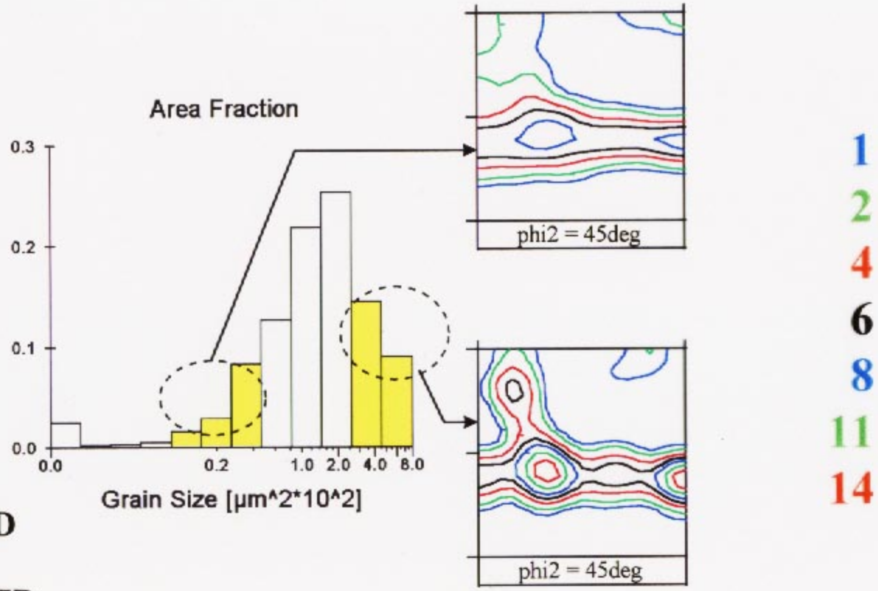
No clear evidence for {554}<225> nucleation

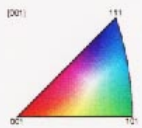
Intermediate Recrystallization Stage



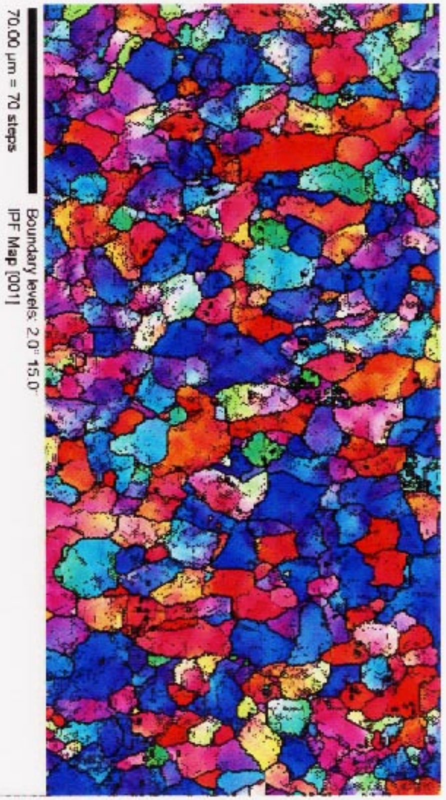
OIM scan of Fully Recrystallized Structure
Quenched from 720°C



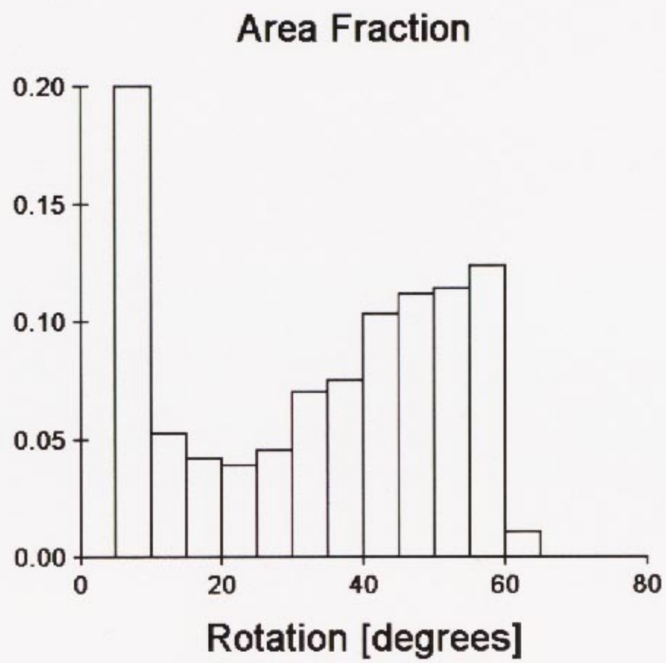




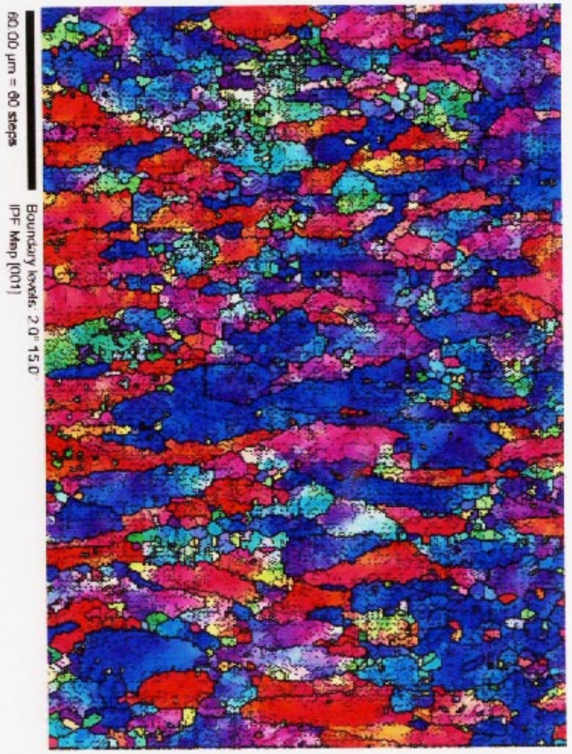
Deformation (sub-)structure : ~~45~~ 10% cold rolled steel sheet



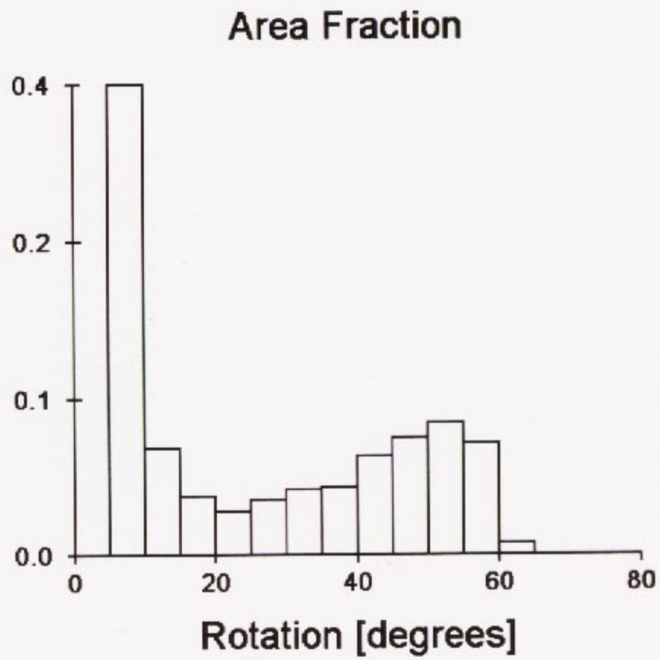
→ RD



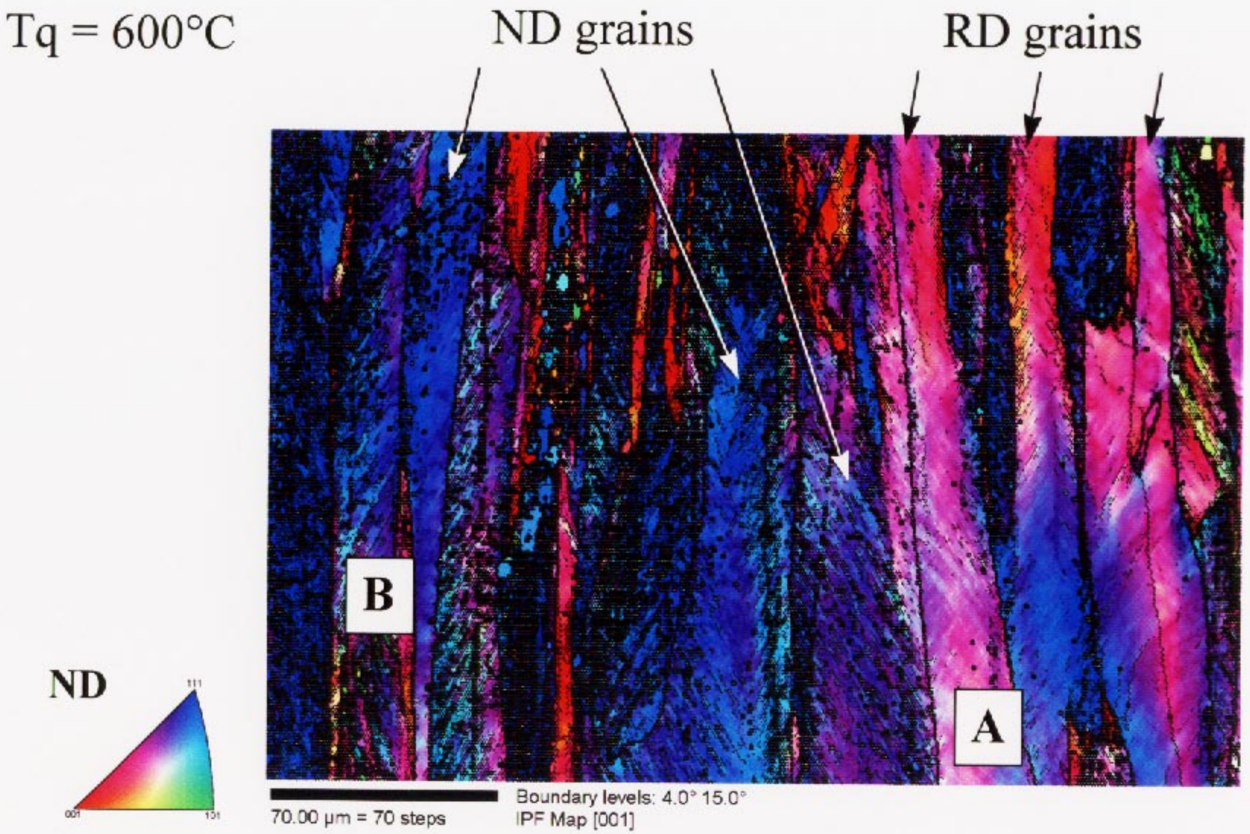
Deformation (sub-)structure : 45% cold rolled steel sheet

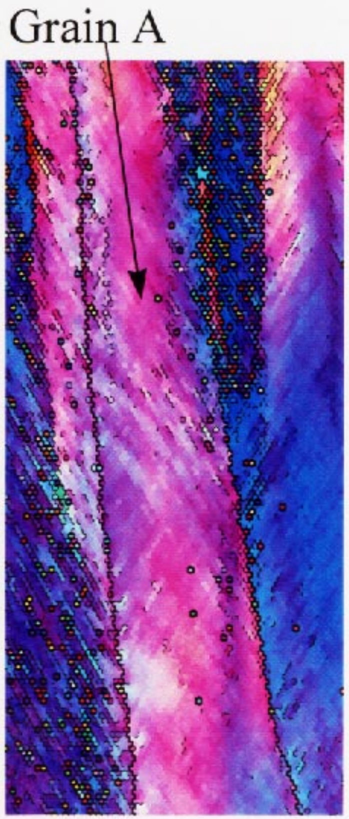


→ RD

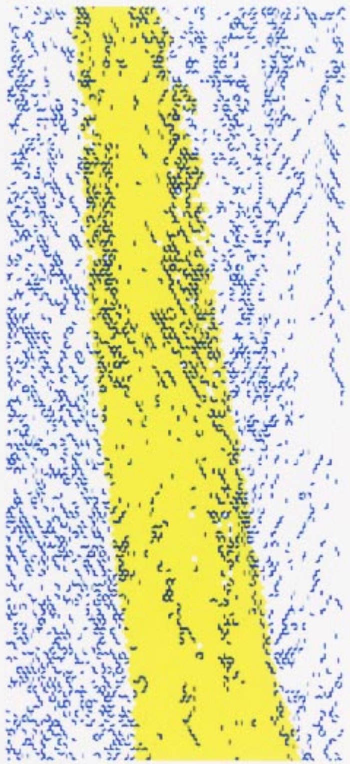


Observation of in-grain shear bands in 70% CR + recovered structure

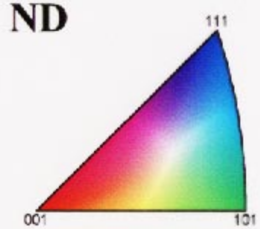


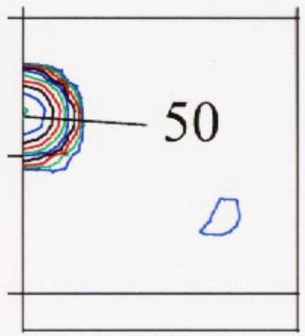


15.00 µm = 15 steps

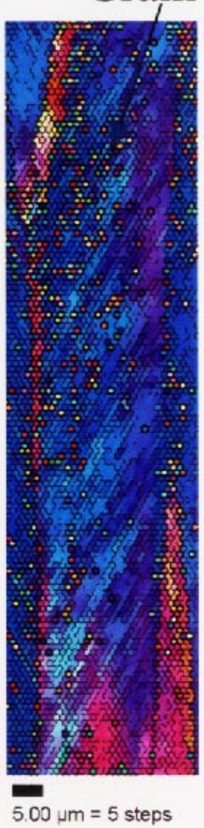


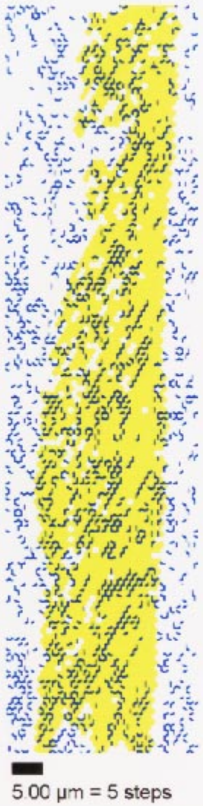
15.00 µm = 15 steps

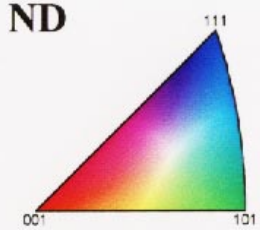


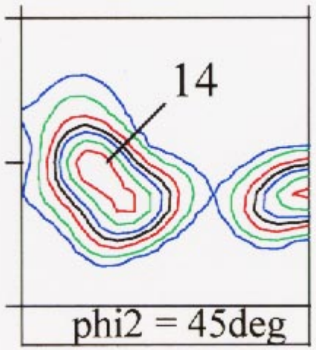


Blue GB = <110>4° ; Δ =2.5°





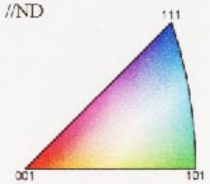


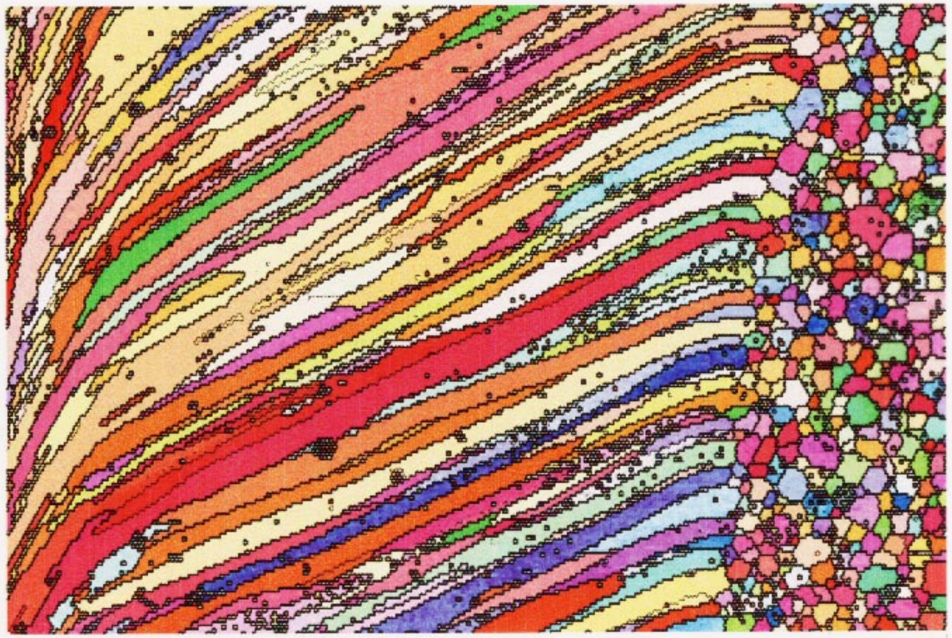


Blue GBs = <110>6deg

Δ = 6deg

Electron Beam Weld in Beryllium





(courtesy: TexSem Ltd.)

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Conclusion

•Originally the ***E**lectron* ***B**ack**S**cattering* ***D**iffraction* technique was mainly used for local orientation studies in texture related research.

•***O**rientation* ***I**maging* ***M**icroscopy* has fully developed into a new branch of microscopy.

•The main advantages of this technique are:

☺ allows to produce microscope images based on crystallographic contrast
☺ wide variety of materials
☺ relatively friendly operation mode.

•Thus, potential of scanning electron microscopyis extend into a field which was hitherto mainly accessible by X-ray techniques.

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ESEM studiedag

ORIENTATION IMAGING MICROSCOPY

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14/10/1998

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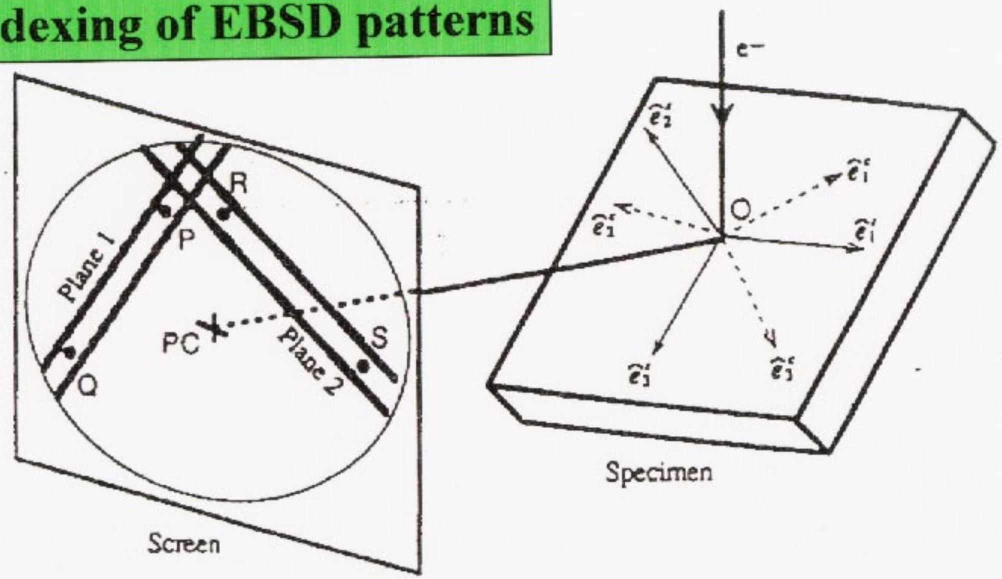
Indexing of EBSD patterns

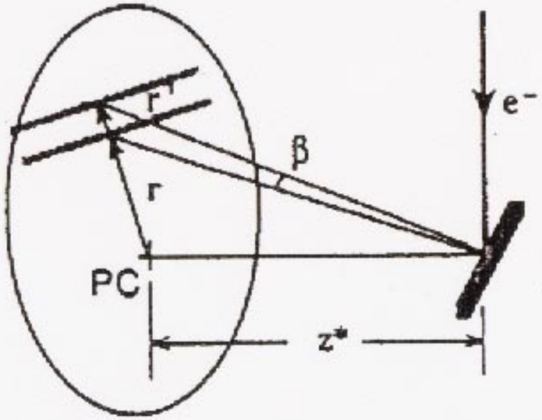


Interplanar angle between planes: $\cos\gamma = \left|\bar{n}_1 . \bar{n}_2\right|$

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

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

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: $\bar{e}_i^{c}$

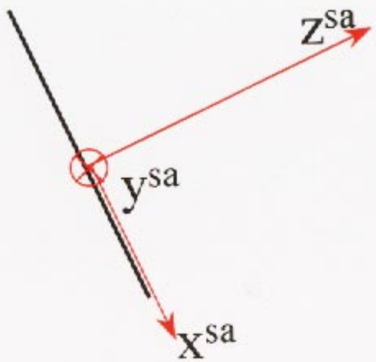
   (<100> axes for a cubic crystal)

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

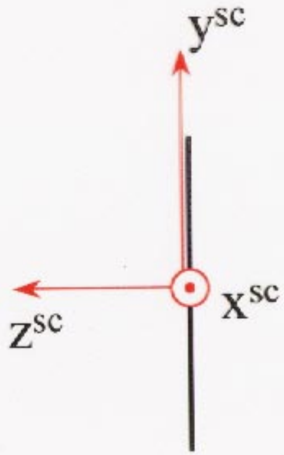
   (RD, TD, ND axes for rolled sheet)

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

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



sample

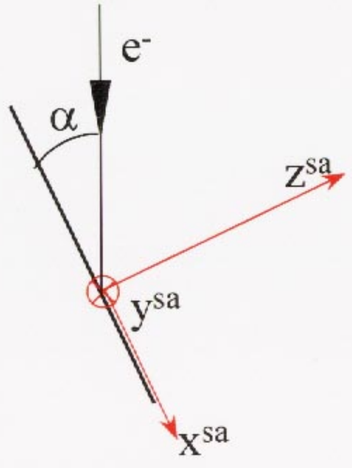


screen

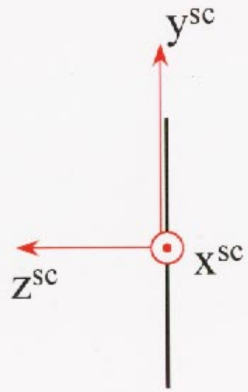
Transformation from pattern to screen coordinates:

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

Transformation from screen to sample coordinates: $g''_{ij} = \bar{e}_i^{sc} . \bar{e}_j^{sa}$



sample



screen

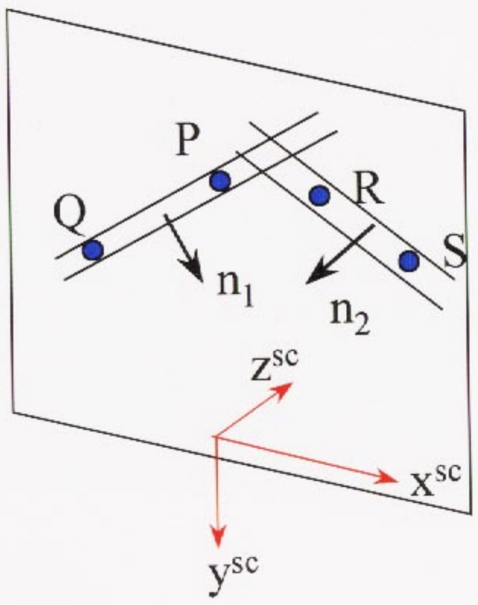
$$\bar{e}_1^{sc} = -\bar{e}_2^{sa}$$

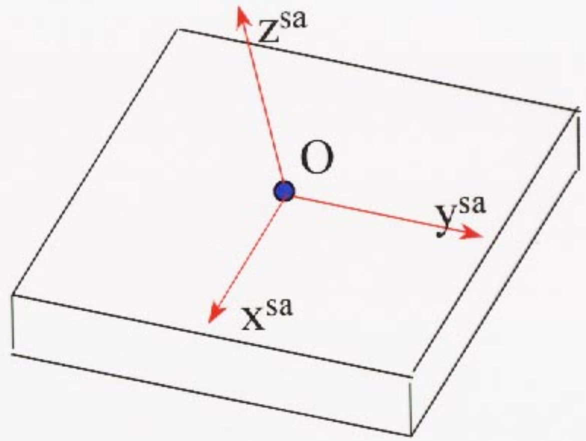
$$\bar{e}_2^{sc} = \bar{e}_1^{sa} \sin\alpha - \bar{e}_3^{sa} \cos\alpha$$

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

Construction of pattern reference system

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





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

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

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

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

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

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Transformation from crystal to pattern coordinates:

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

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

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

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

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

crystal to pattern ↓ — pattern to screen ↓ — screen to sample ↓

Orientation matrix: $g_{ij} = g'''_{ik} \; g'_{kl} \; g''_{lj}$

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