

Combined Application of EBSD and ECCI for Crystal Defect Observation in Bulk Samples



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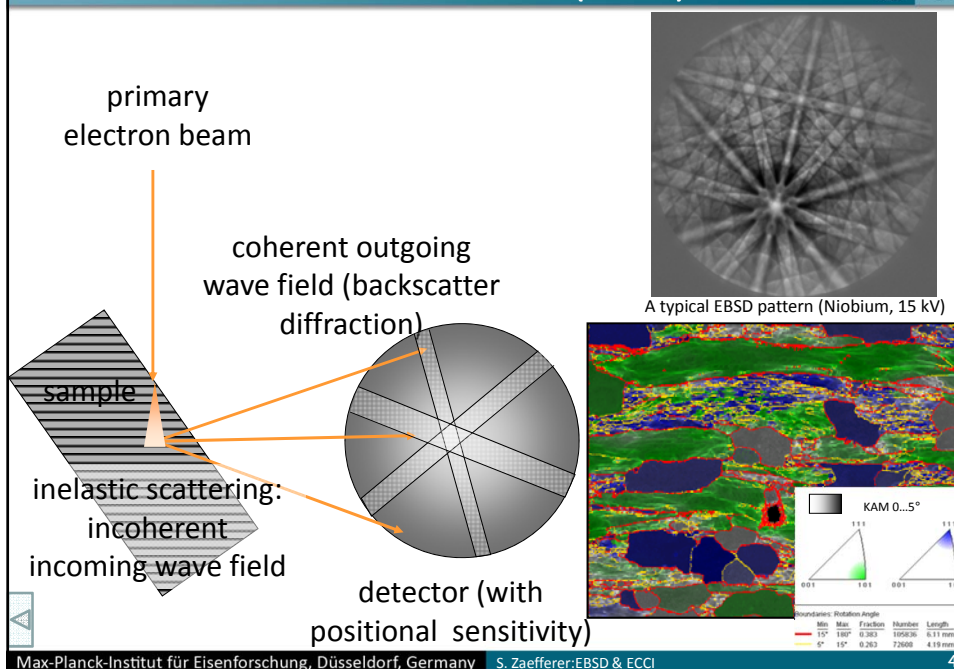
Outline

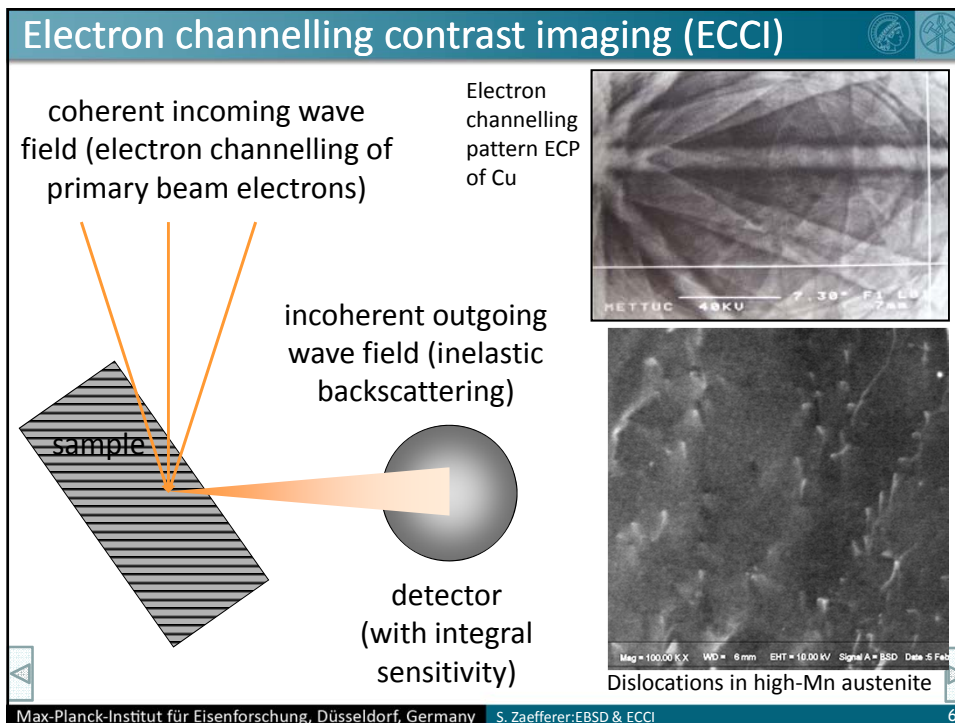
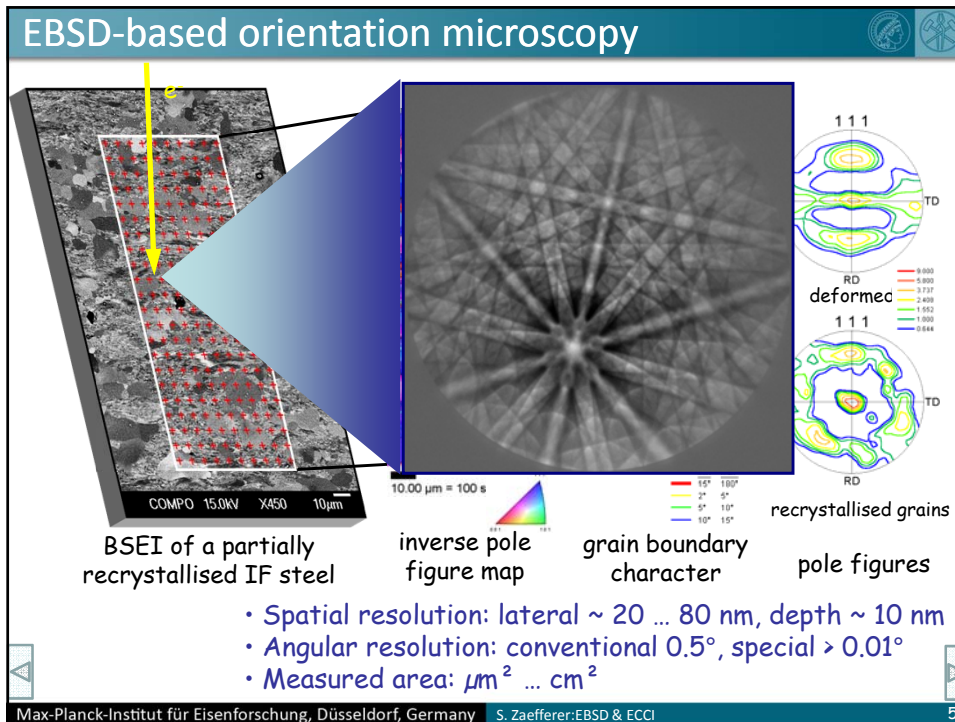
- Introduction:
 - Diffraction techniques in the SEM
- A bit of theory:
 - Electron scattering mechanisms and electron channelling
- Defect observation by ECCI
 - Stacking Faults
 - Dislocations
- Application examples for the ECCI technique
- Conclusions

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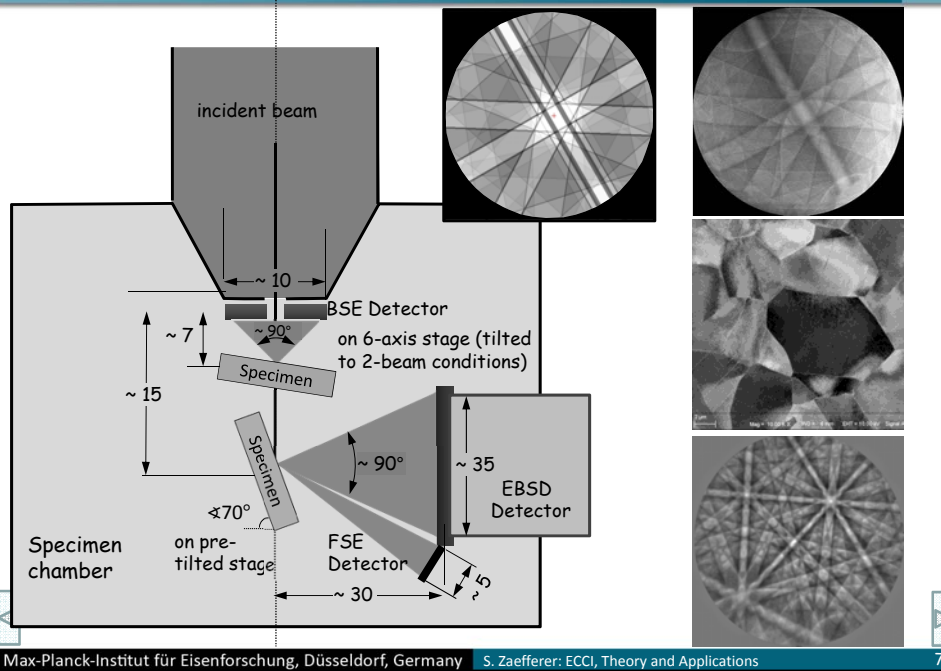
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Electron backscatter diffraction (EBSD)





Diffraction techniques in SEM



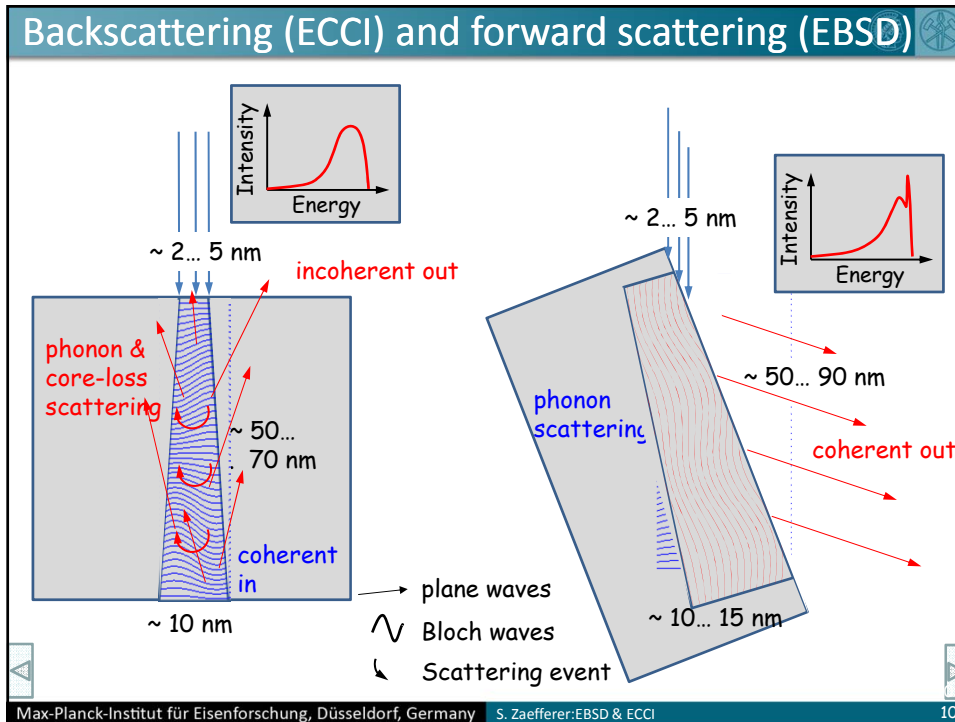
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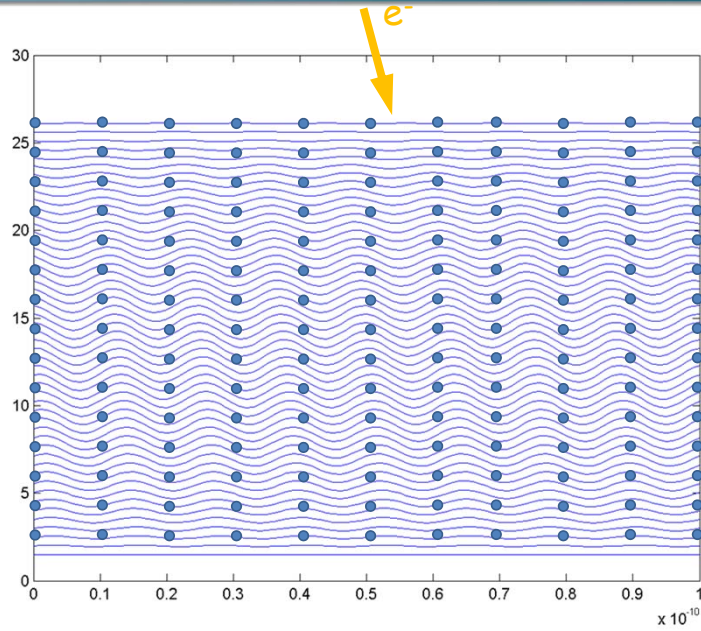
Scattering Mechanisms

	Bragg scattering	Phonon scattering	Core loss scattering	Multiple scattering
Classification	elastic & coherent	quasi-elastic & incoherent	inelastic & incoherent	inelastic & incoherent
Physical phenomenon	coherent Bragg diffraction	forward scattering	formation of electromagnetic radiation (x-rays, light), plasmons, secondary electrons etc.	Combination of elastic and inelastic scattering events
Scattering angle	small (<5°)	medium (< 10°)	Very small (<1°)	For multiple events: large (up to 180°)
Energy loss	0	< 1 eV	several 10 eV to keV	eV to keV
Scattering cross section	$f_e^2 \exp(-2W)$	$f_e^2 [1 - \exp(-2W)]$	Various	Various
Importance for SEM diffraction	EBSD & ECCI	EBSD	ECCI	ECCI

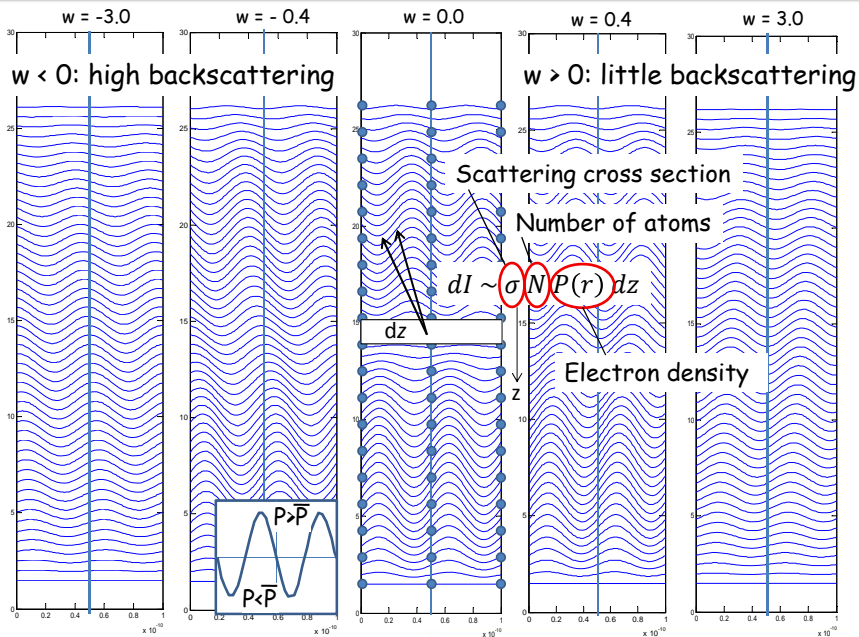
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Total electron density in dependence of excitation



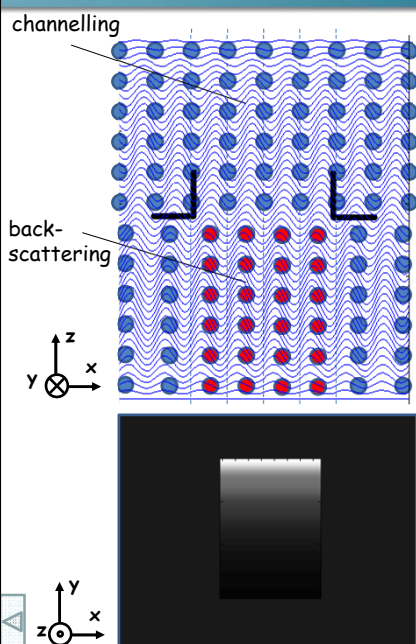
The total electron wave density



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Calculation of defect contrast (stacking fault)

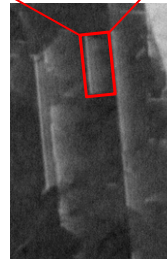
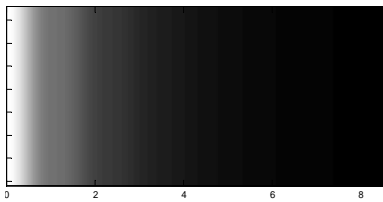
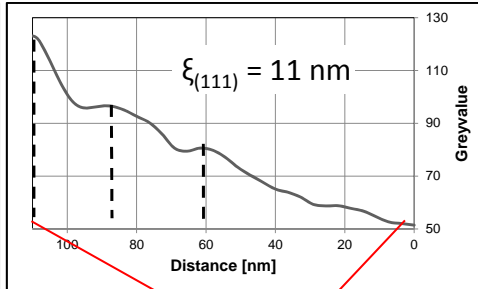
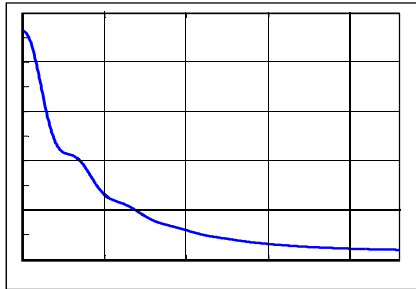


Integration of channelling and backscattering conditions

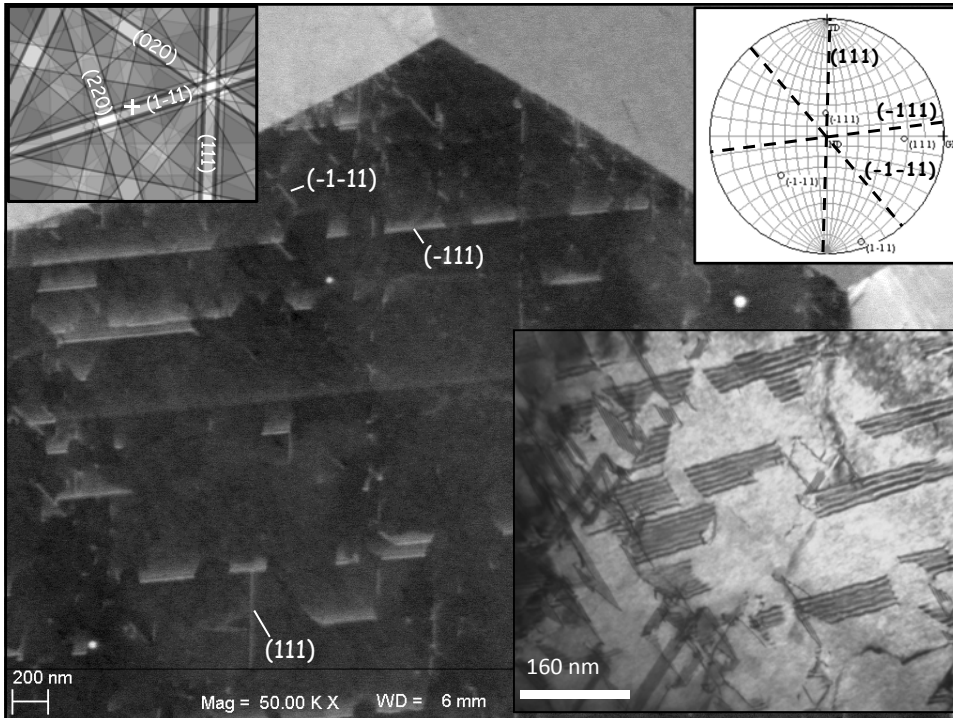
$$\Delta\eta(x_{(z_{SF})}) = \Delta\eta(w_C) \Big|_0^{z_{SF}} + \Delta\eta(w_B) \Big|_{z_{SF}}^{\infty}$$

- Straight bright line at intersection with surface
- Fading contrast on opposite side
- Intensity oscillations with depth

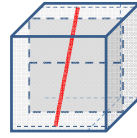
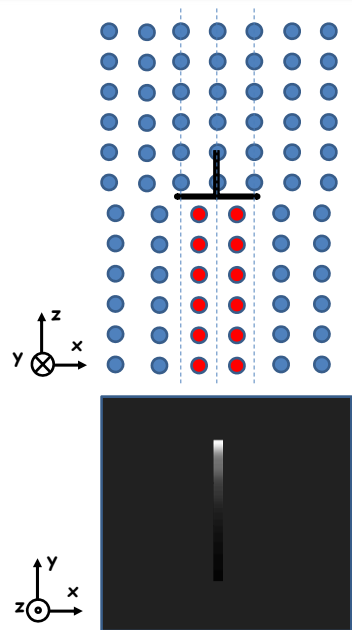
Contrast of stacking faults



Depth of visibility: about $5 \times \xi_g$; about 50 to 60 nm at 15 kV

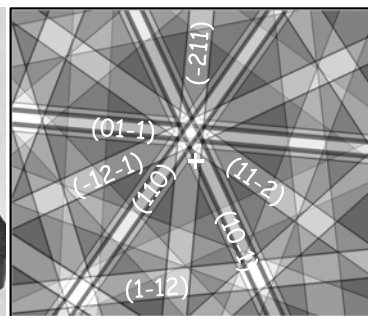
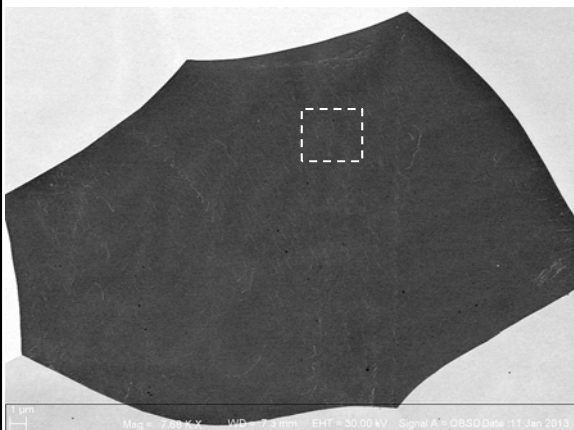


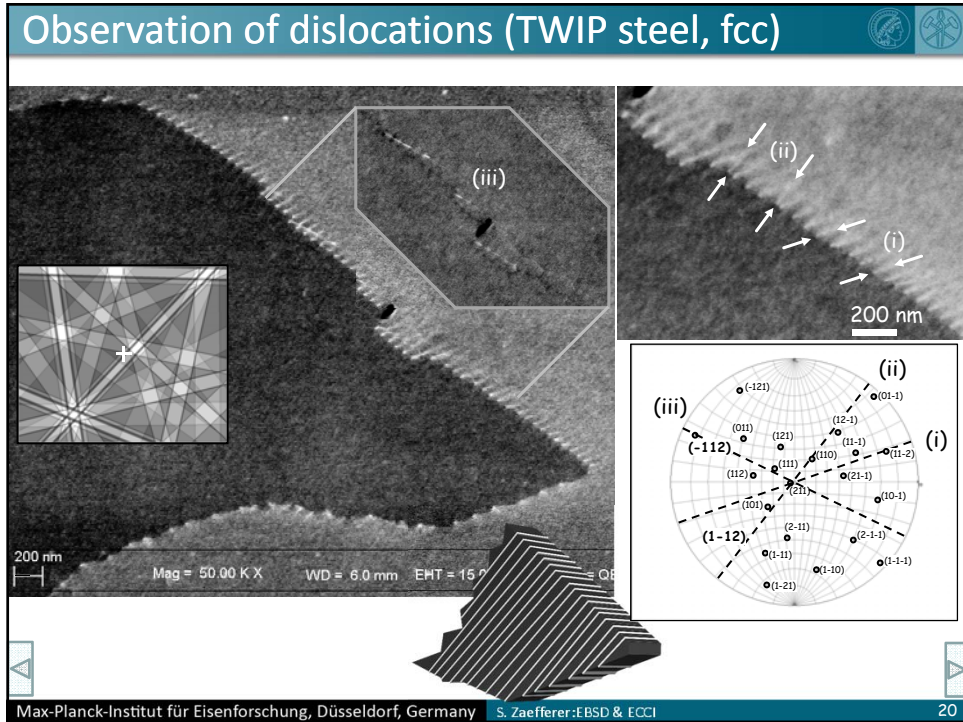
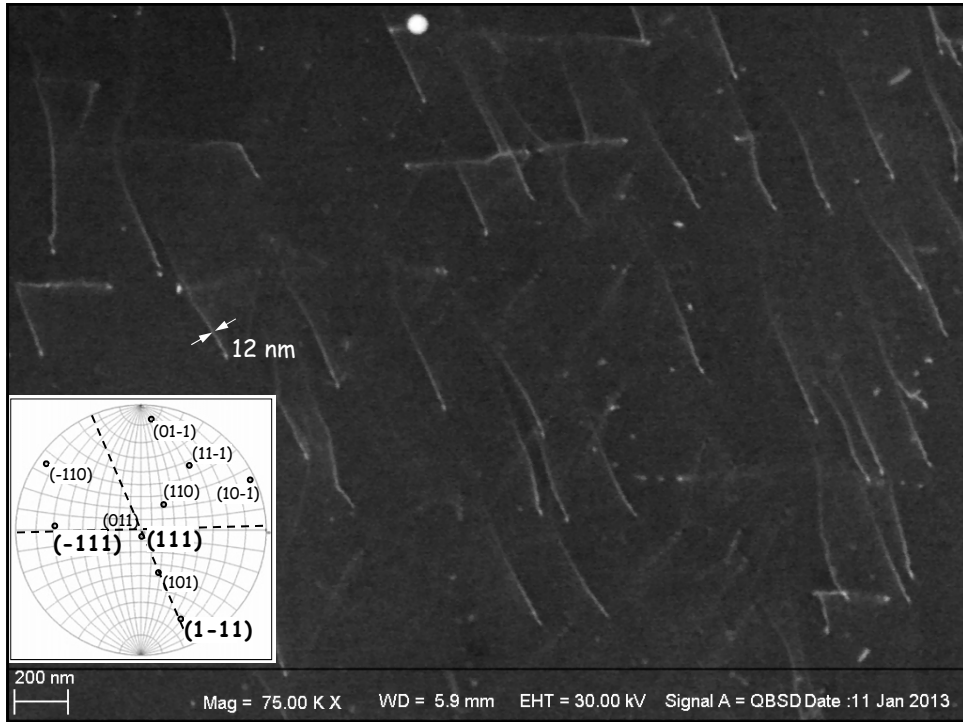
Calculation of defect contrast (edge dislocation)



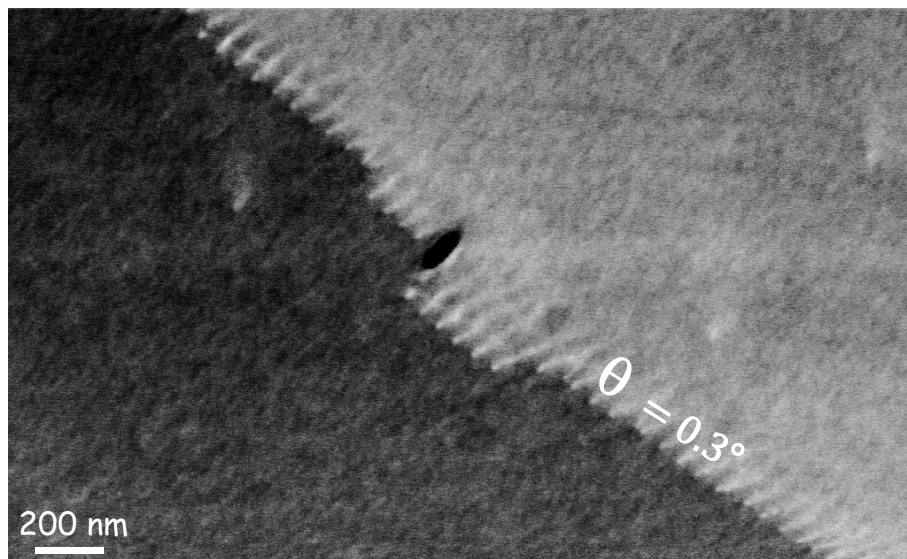
- Bright spot at intersection with surface
- Fading contrast with increasing depth
- Intensity oscillations with depth

Observation of dislocations (Fe-Si alloy, bcc)





Burgers vector determination by cECCI

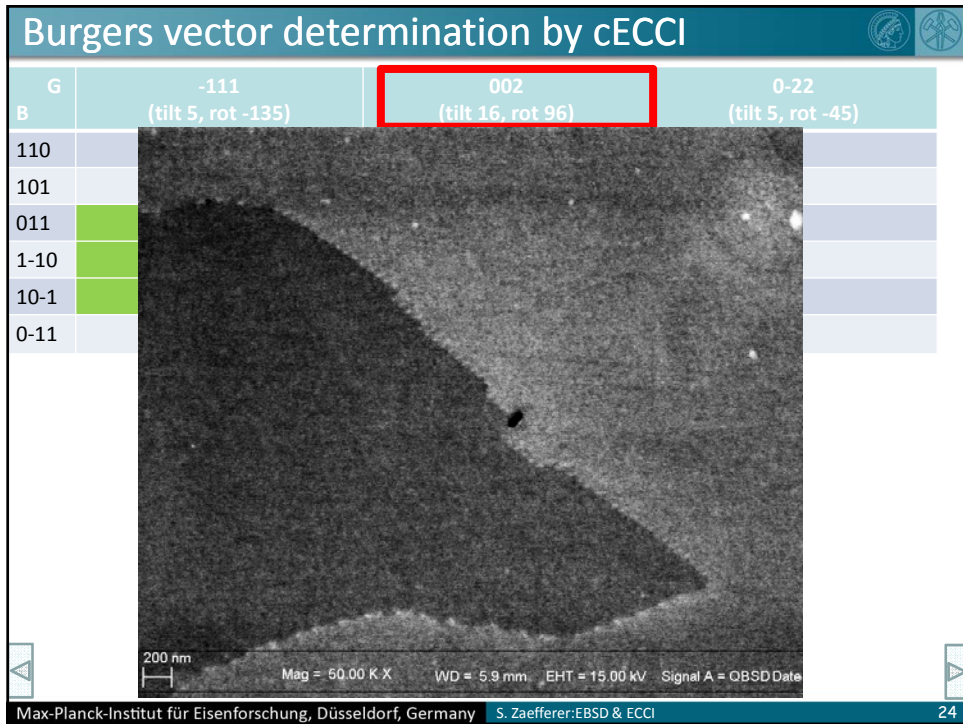
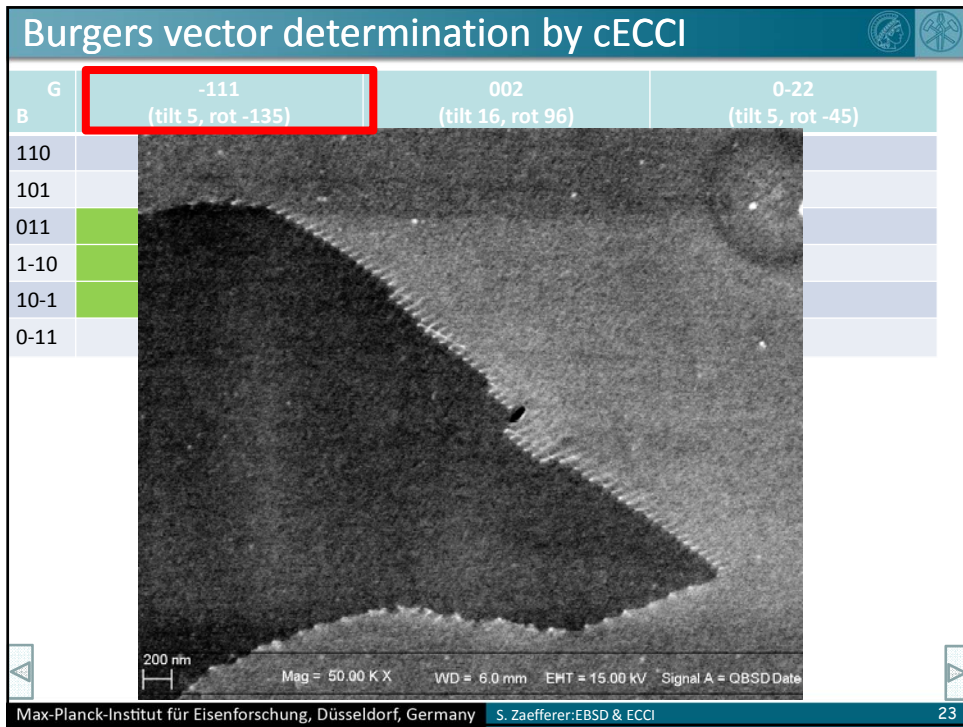


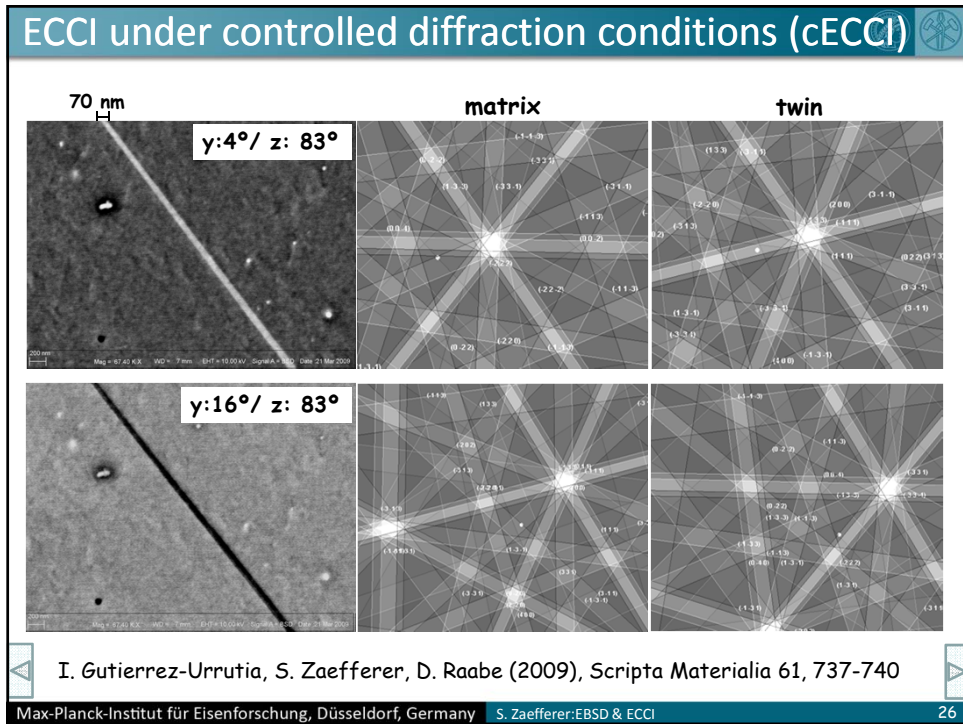
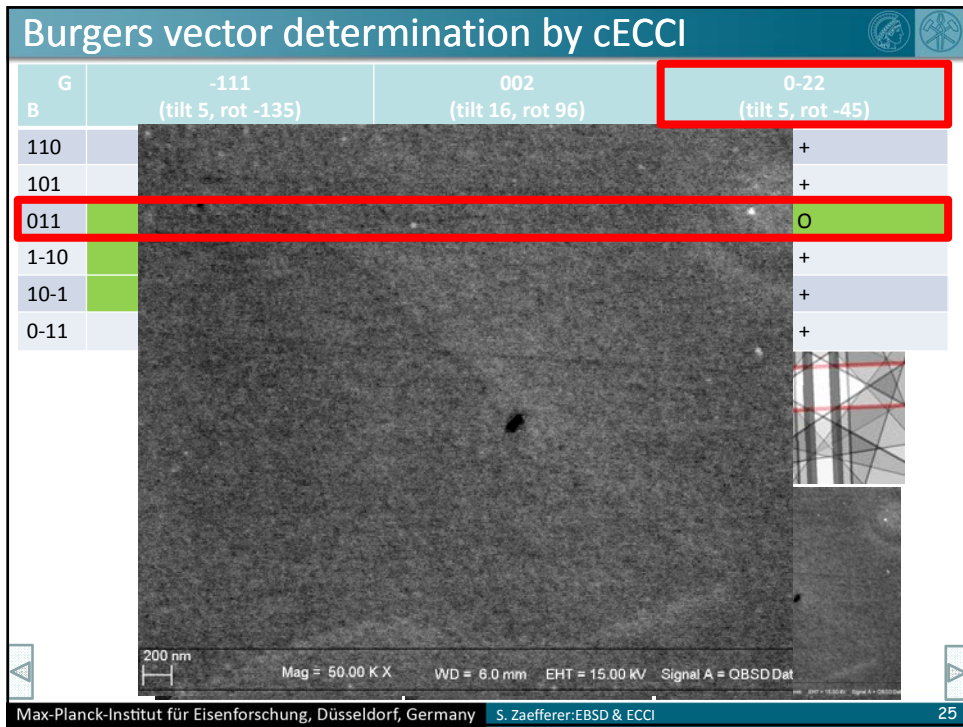
Dislocation spacing, $D \sim 60 \text{ nm}$ $D = |b|/\theta \rightarrow |b| = 0.3 \text{ nm}$

Burgers vector determination by cECCI

G	-111 (tilt 5, rot -135)	002 (tilt 16, rot 96)	0-22 (tilt 5, rot -45)
B 110	O	O	+
101	O	+	+
011	+	+	O
1-10	+	O	+
10-1	+	+	+
0-11	O	+	+



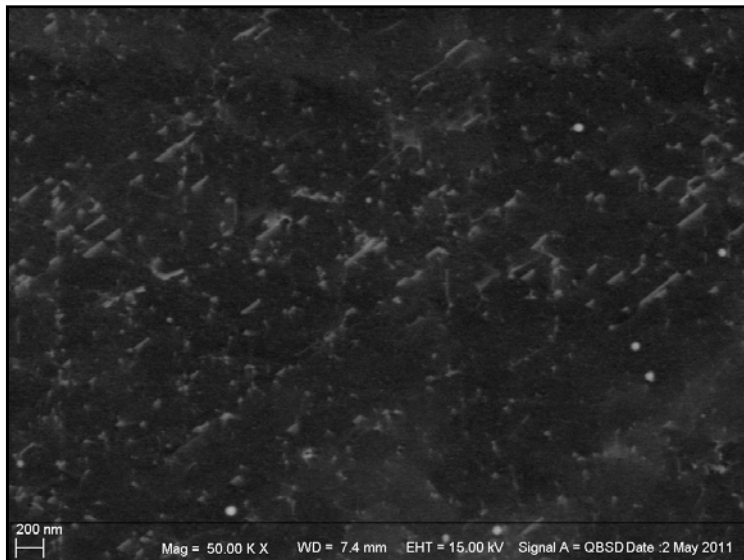




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Dislocation density determination on bulk samples



dislocation density determination
 $\rho : 1.4 \dots 2 \times 10^{13} \text{ m}^{-2}$

Large area defect observatio on bulk samples



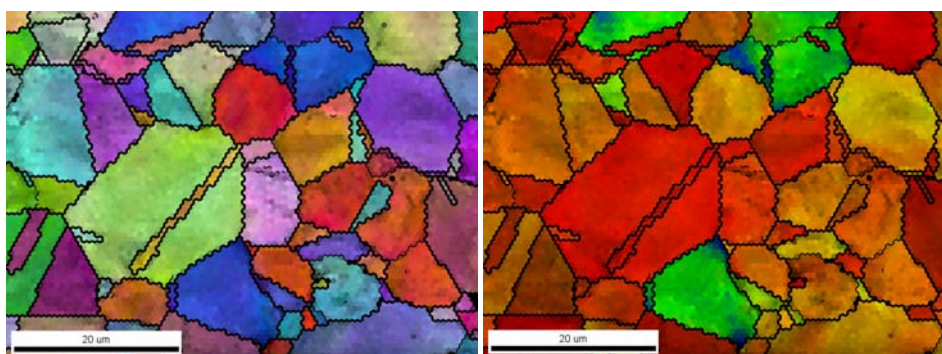
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In-situ deformation experiments

Experiment: uniaxial compression of a predeformed TWIP steel sample

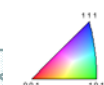


Inverse pole figure map (TD direction)

Gray Scale Map Type: Image Quality
1227.41...5448.7 (1227.41...5448.7)

Color Coded Map Type: Inverse Pole Figure [D10]

Iron - Gamma



Schmid factor map for compression along TD direction

Gray Scale Map Type: Image Quality
1227.41...5448.7 (1227.41...5448.7)

Color Coded Map Type: Schmid Factor

Sigma Values:
0.0 0.0 0.0
0.0 -1.0 0.0
0.0 0.0 0.0

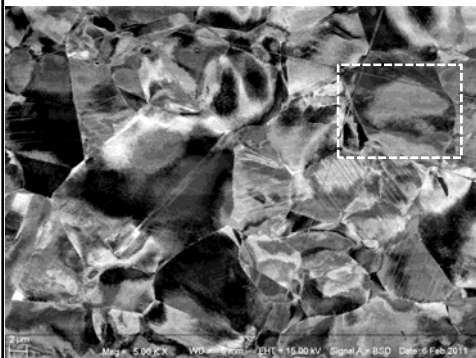
Min	Max	Total Fraction	Part/ton
0.30459	0.499982	0.992	1.000

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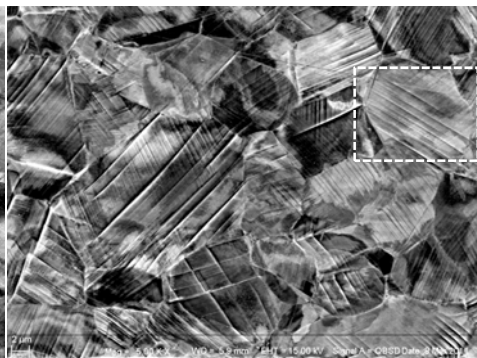
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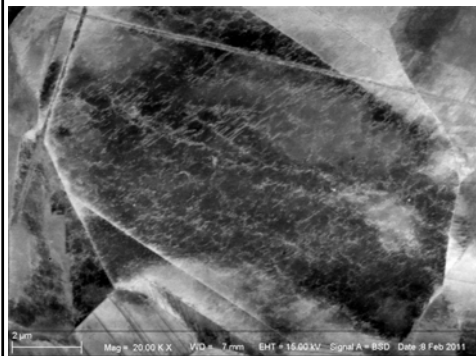


7 % plane strain



+ 1.5 % compression

In-situ deformation experiments



7 % plane strain



+ 1.5 % compression

Conclusions



- cECCI: electron channelling contrast imaging under controlled diffraction conditions
- cECCI enables lattice defect observations at high spatial and angular resolution
- Images appear comparable to classical TEM dark field images but are created from the surface of bulk samples (< 100 nm of visibility)
- Ideal applications: observation of bulk, large samples, in-situ straining experiments

