INSTITUT
D'ÉLECTRONIQUE
ET DES SYSTÈMES

UMR 5214 CNRS-UM

Electron Channeling Contrast Imaging for epi-layer structural defect characterization

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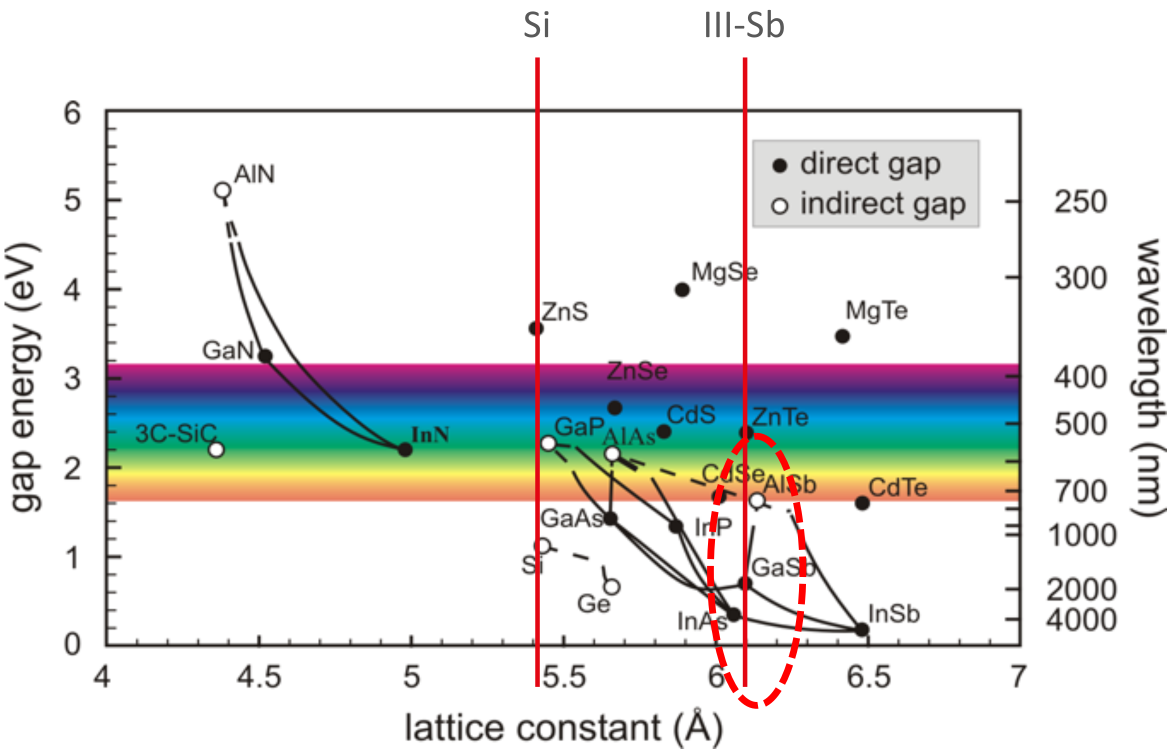
Acknowledgements : DFG / ANR (FILTER, ANR-20-CE92-0045), Equipex+ HYBAT, ANR-21-ESRE-0026

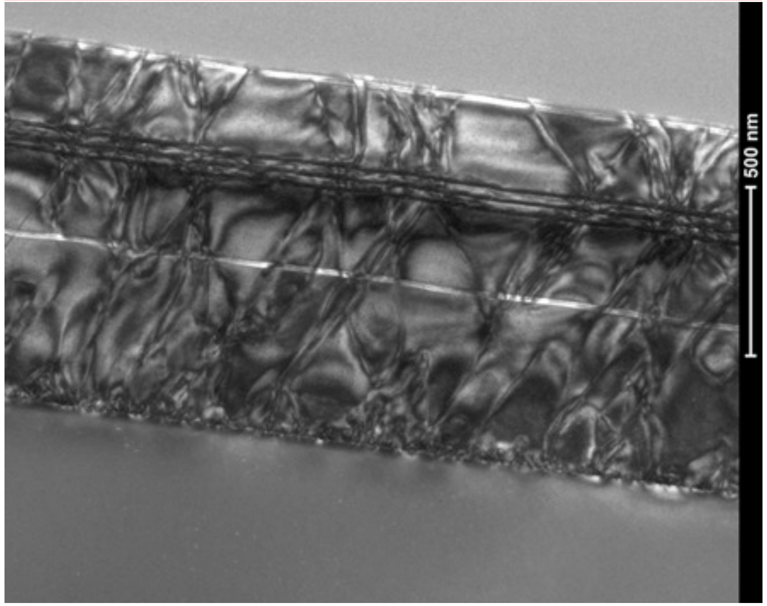
Context : epitaxy of III-Sb on Silicon substrates



Ultra-large lattice-mismatch

$\Delta a/_{a} \approx 12\%$

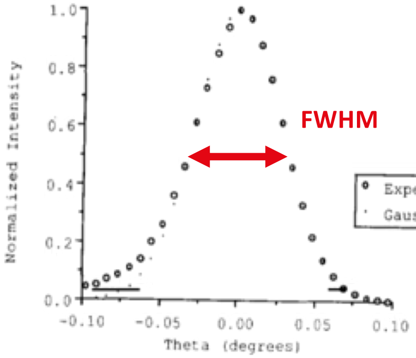
Large dislocation densities

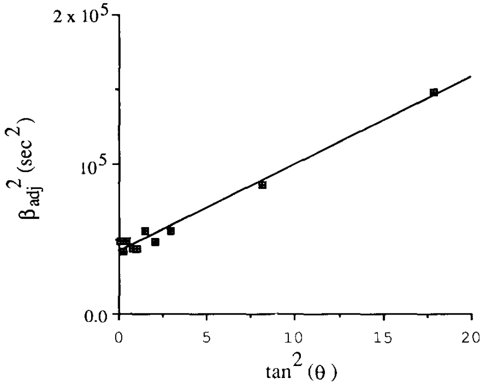


Need for an effective dislocation counting technique

Threading dislocations counting techniques

X-ray diffraction



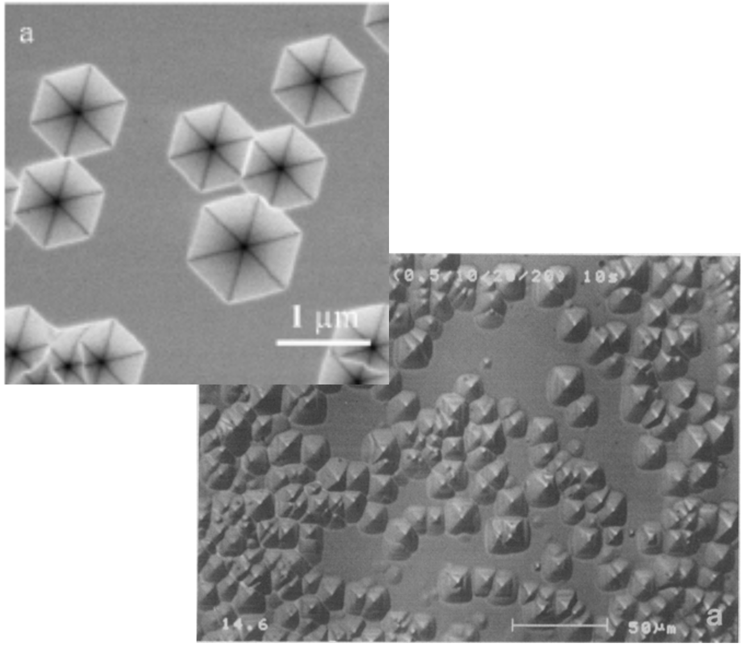


$$TDD = K_\alpha / 4.36 . b^2$$

J.E. Ayers, Journal of Crystal Growth 135 (1994) 71—77

Etch-Pit Density (EPD)

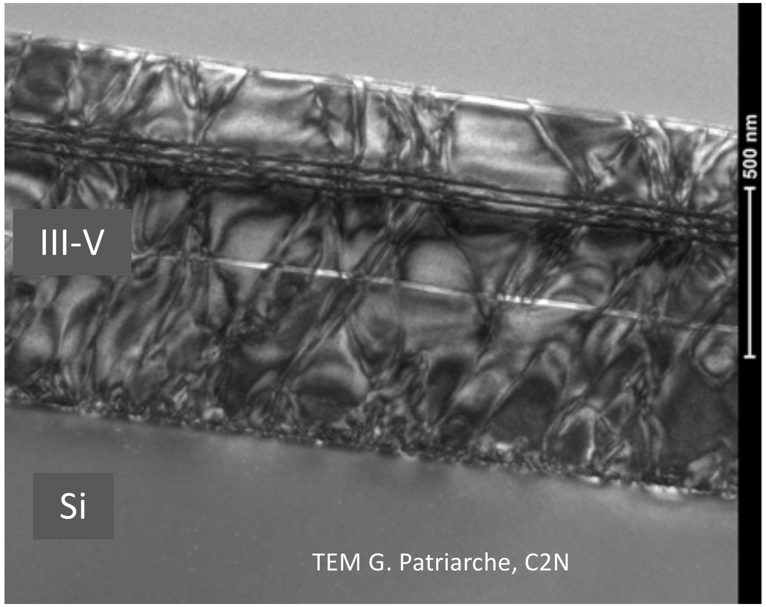
EPD in GaN,
G. Kamler et al., Eur. Phys. J. Appl. Phys. 27, 247–249 (2004)



EPD in InP,
E.Peiner and A. Schlachetzki, Journal of El. Mat., Vol, 21, No. 9, 1992

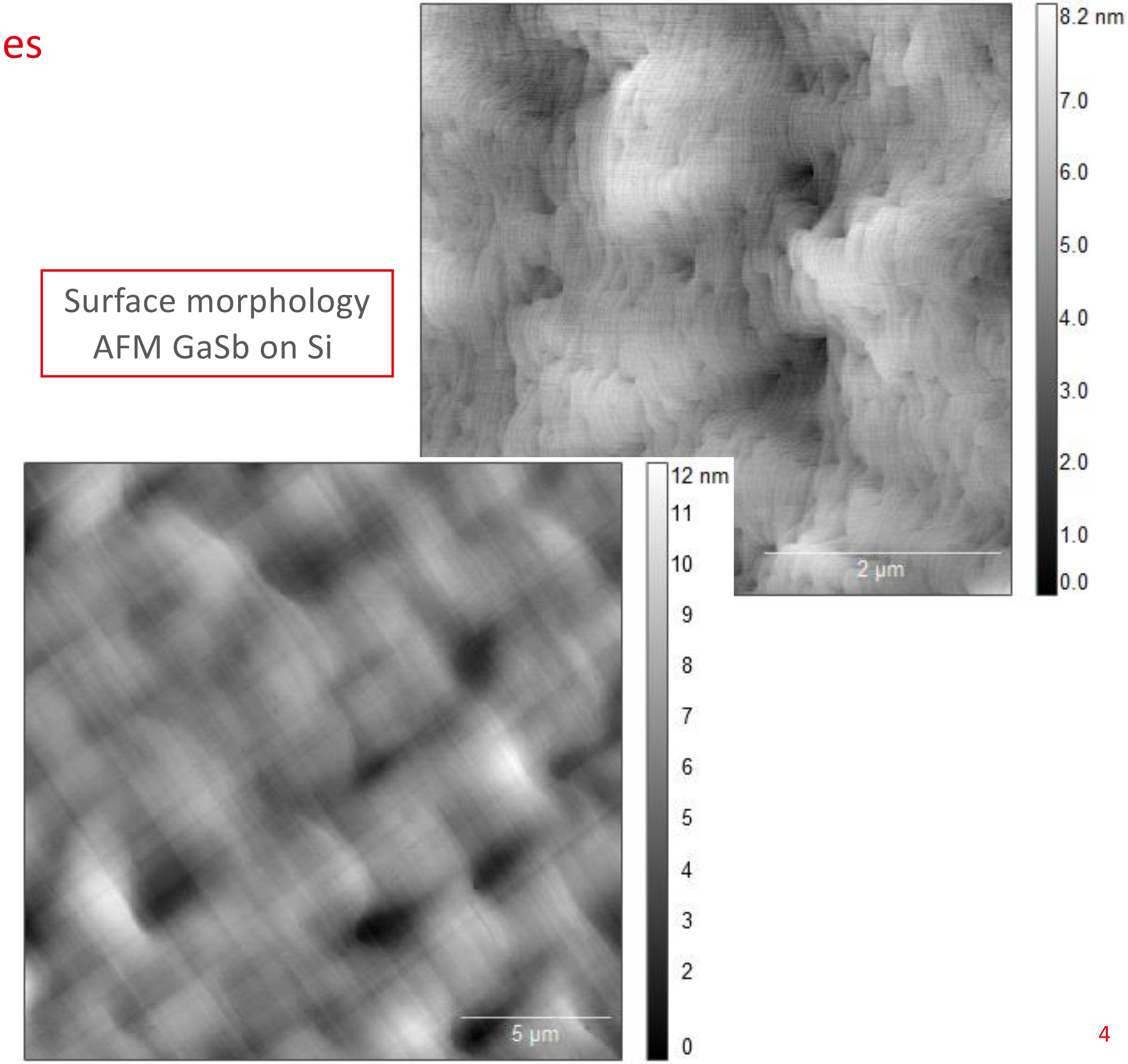
Threading dislocations counting techniques

Cross section TEM



TEM G. Patriarche, C2N

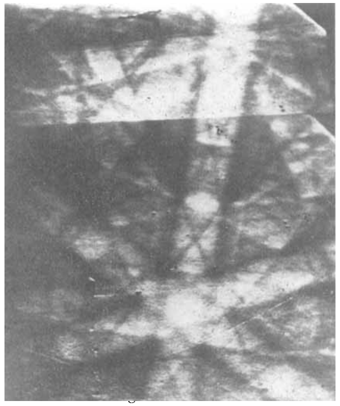
Surface morphology
AFM GaSb on Si

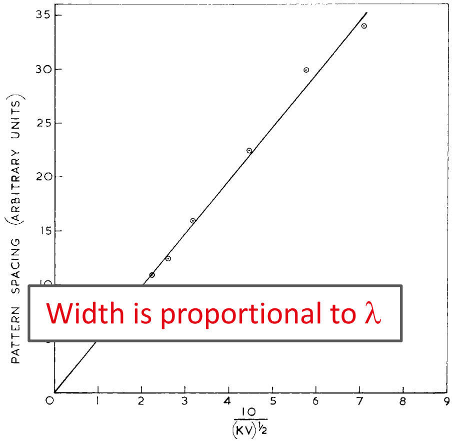


Historical perspective

- **Observation of “Kikuchi-like” bands** : Coates, 1967
  - Access to the crystal orientation in a SEM
- **Explanation = “anomalous absorption” at Bragg angle** : Booker *et al.*, 1967
  - ECP ≠ Kikuchi lines because not related to diffraction
  - **Imaging dislocations is possible because the bending of the lattice planes changes the absorption**
  - Access to the **Burgers vector**
- Dynamical **theory** calculation : Clarke and Howie 1971
  - Need high currents and high accelerating voltages to observe defects ➔ FEG SEM, STEM
- **First attempt in Scanning TEM** : Clarke *et al.*, 1971
  - 80-100 kV STEM, thin foil
- **First attempts in SEM** : Pitaval et al., 1977, Morin et al., 1979
  - **FEG SEM**, large tilt (50-70°), high-energy filter, side-mounted BSED
  - ➔ dislocations in Si and invisibility criteria validated
- **More convenient config**. : Ng *et al.*, 1998, Simkin and Crimp, 1999
  - Standard 4-quadrant Si diode detector on the pole piece ➔ small tilts

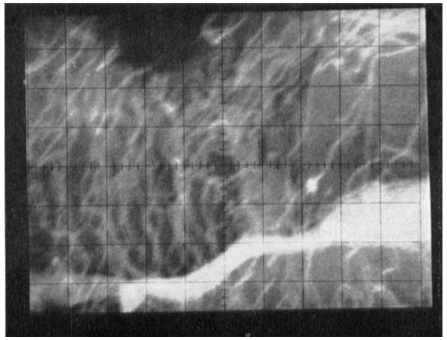
ECP Ge sample
Coates, Phil Mag 16:1179, 1967

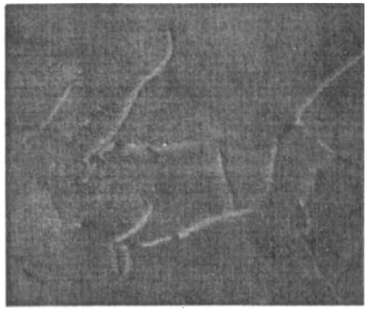




Width is proportional to λ

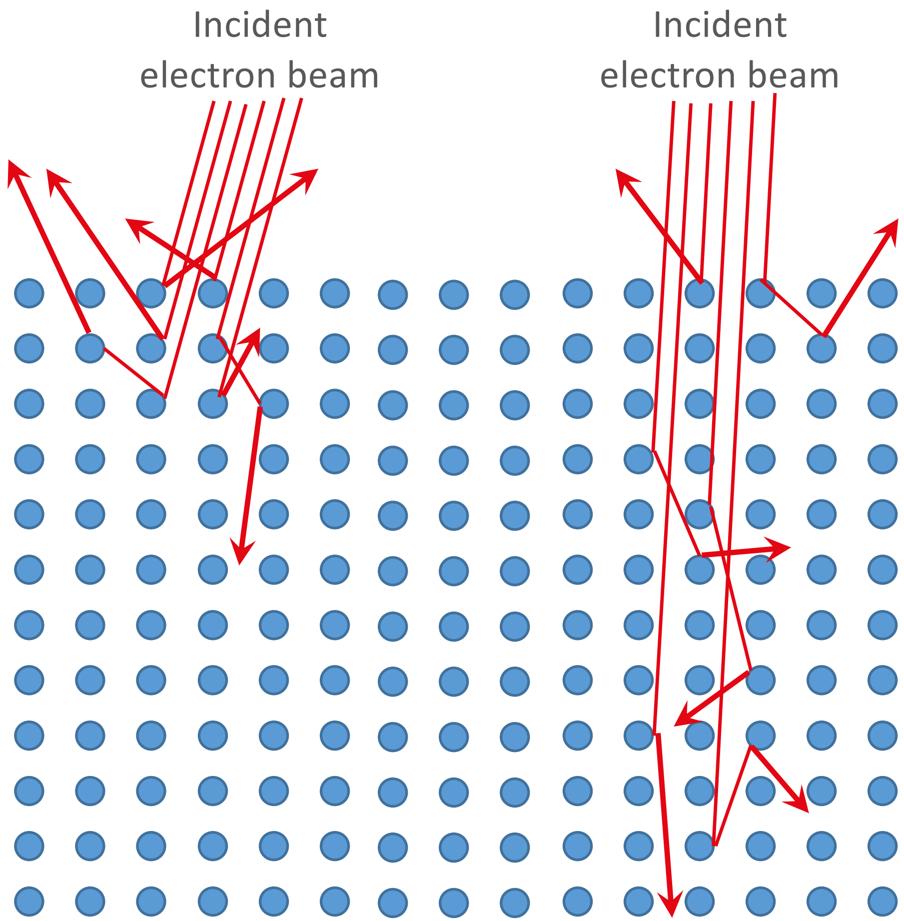
STEM ECCI Cu-8% Al
Clarke et al., Phil Mag, 24:190, 973, 1971





ECCI Si
Morin et al., Phil Mag A, 40:4, 511-524, 1977

Back scattered signal from a crystal : influence of the incidence angle



When the incident
beam gets **parallel to the crystal planes**:

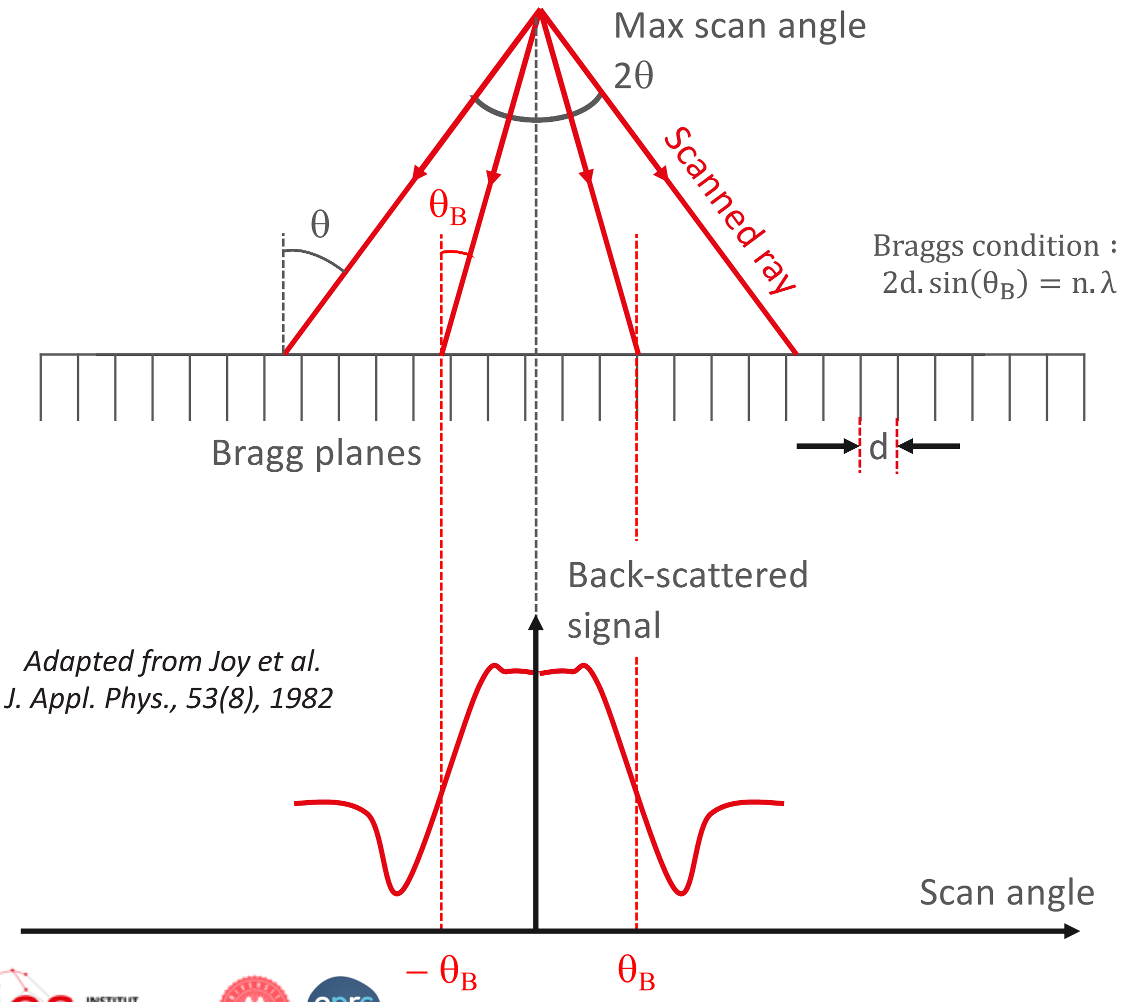
➔ Beam **channeling** through the crystal

➔ Lower **back-scattered electron** (BSE) emission

How can we orientate the
sample wrt the e^- beam?

Adapted from Joy et al., J. Appl. Phys., 53(8), 1982

Low-magnification : contrast related to the crystal structure

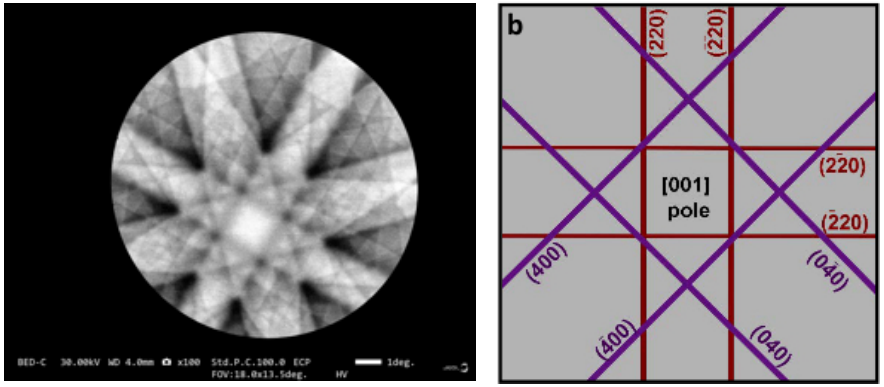


Braggs condition :
$2d.\sin(\theta_B) = n.\lambda$

Adapted from Joy et al.
J. Appl. Phys., 53(8), 1982

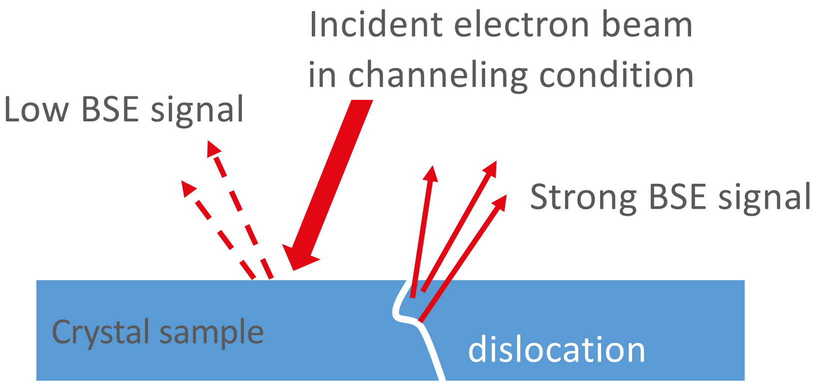
- Enhanced back-scattered signal when $|\theta| < \theta_B$
- Interpreted as the superposition of two Block wave functions
- True for many lattice-planes

➔ Electron Channeling Pattern



Allows to orientate (tilt and rotation) the sample to set the channeling direction of interest

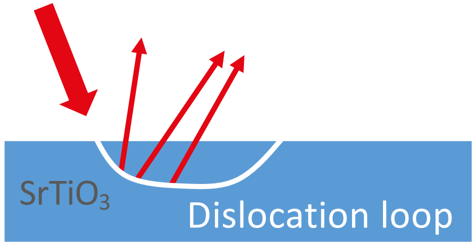
Back scattered signal from a crystal : structural defects

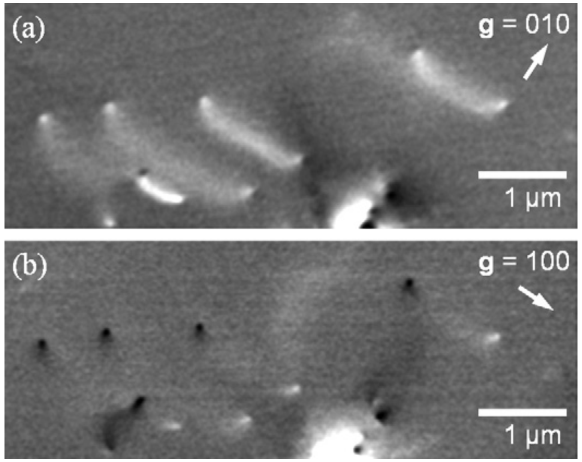


Structural defects such as dislocations have a strong BSE contrast in channeling condition

➔ **ECCI (Electron Channeling Contrast Imagery)**

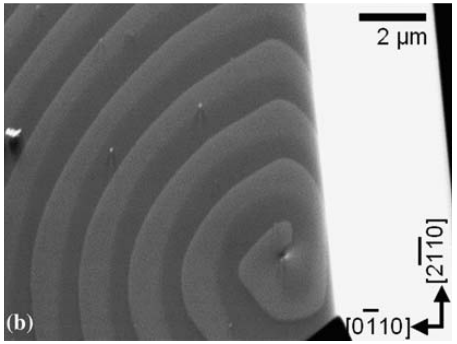
Some examples from the literature

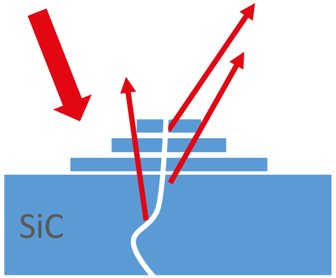




$SrTiO_3$ (001) substrate

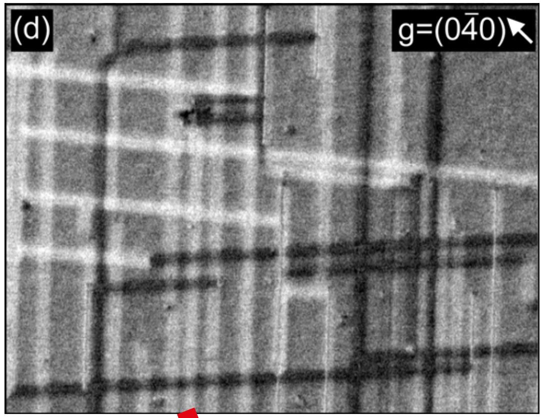
Y.N.Picard et al., Micr. Today, 2012
doi:10.1017/S155192951200007

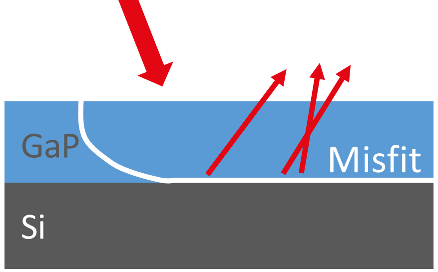




Screw dislocation SiC / 4HSiC

Y.N.Picard et al., JEM, 2007
doi: 10.1007/s11664-007-0308-0



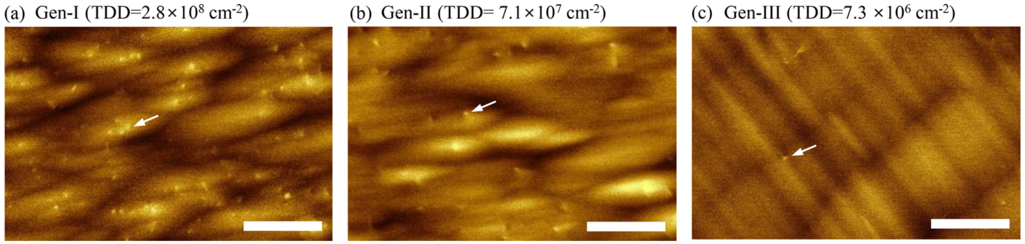


MD network, GaP / Si

Carnevale et al., Appl. Phys. Lett. 104, 232111 (2014)
10.1063/1.4883371

GaAs / Si

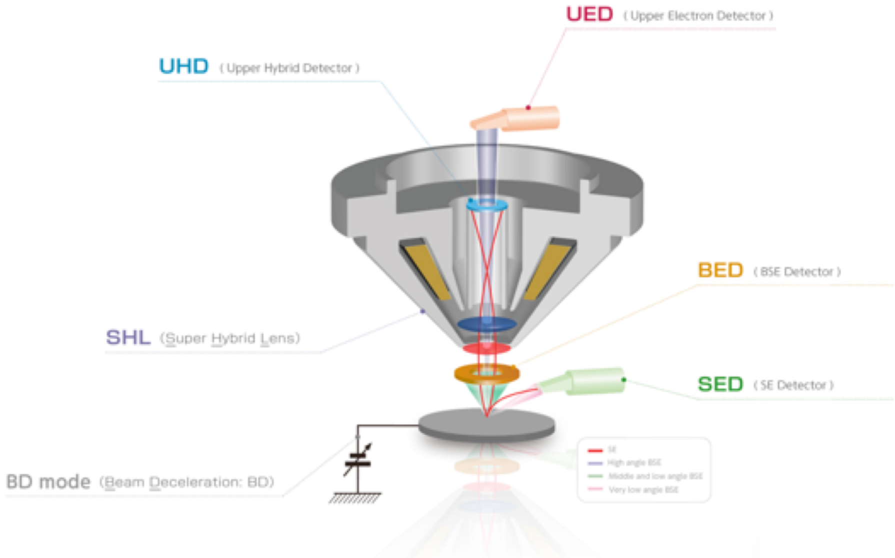
Jung et al., Appl. Phys. Lett. 112, 153507 (2018)
doi: /10.1063/1.5026147


(a) Gen-I (TDD=2.8×10^8 cm^{-2}) (b) Gen-II (TDD= 7.1×10^7 cm^{-2}) (c) Gen-III (TDD=7.3×10^6 cm^{-2})

SEM-FEG JEOL JSM-IT800 HL

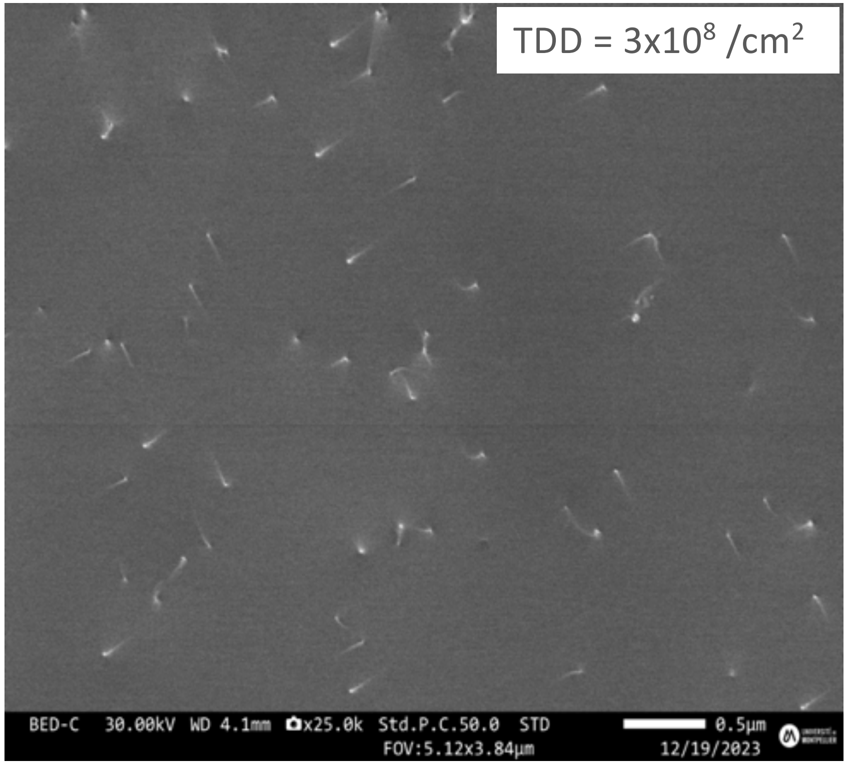
EQUIPEX+ HYBAT
ANR-21-ESRE-0026

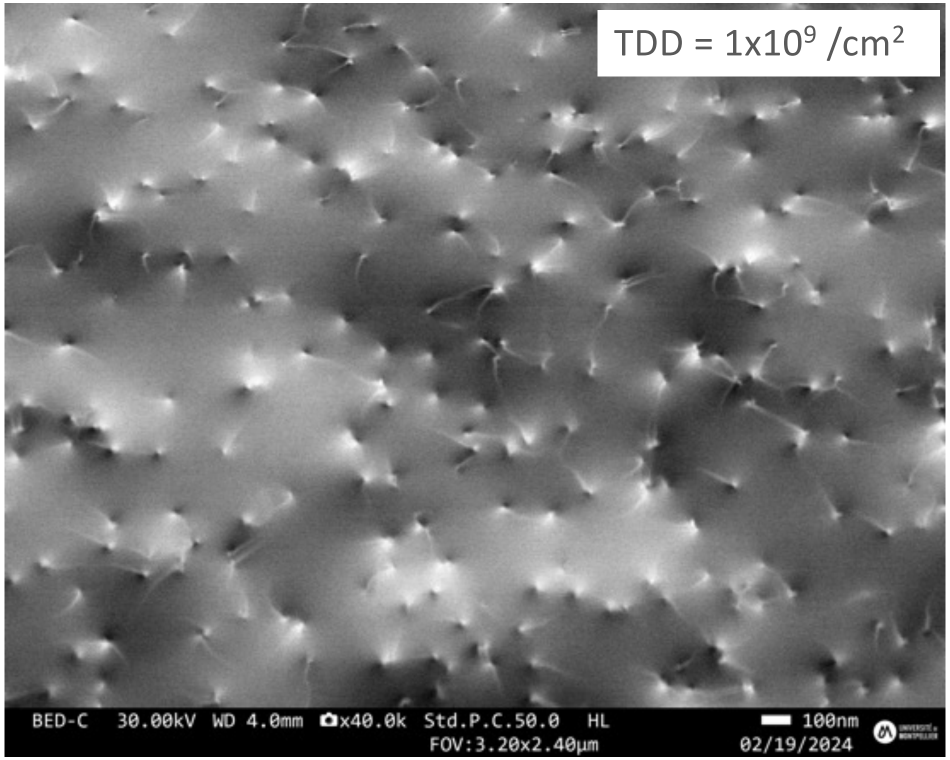
FRANCE 2030



- Installed summer 2023
- Field emission electron gun
- BSE detector
- Full eucentric goniometer stage

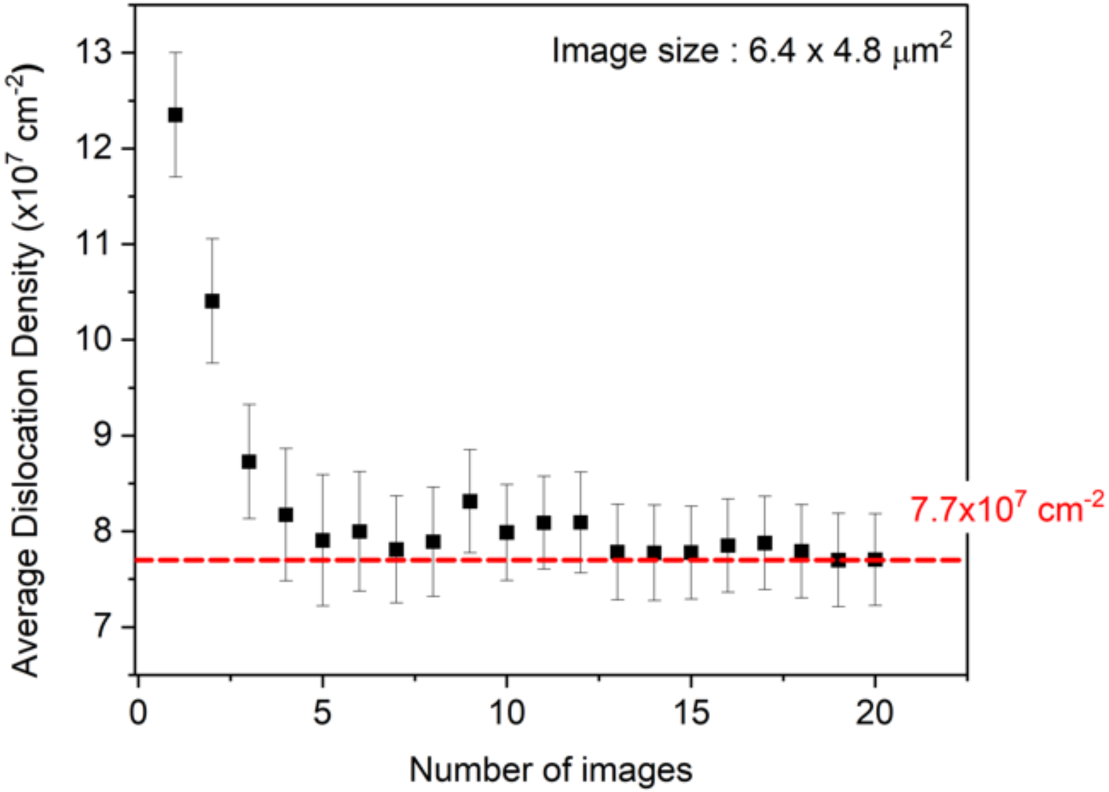
GaSb on Si (001)



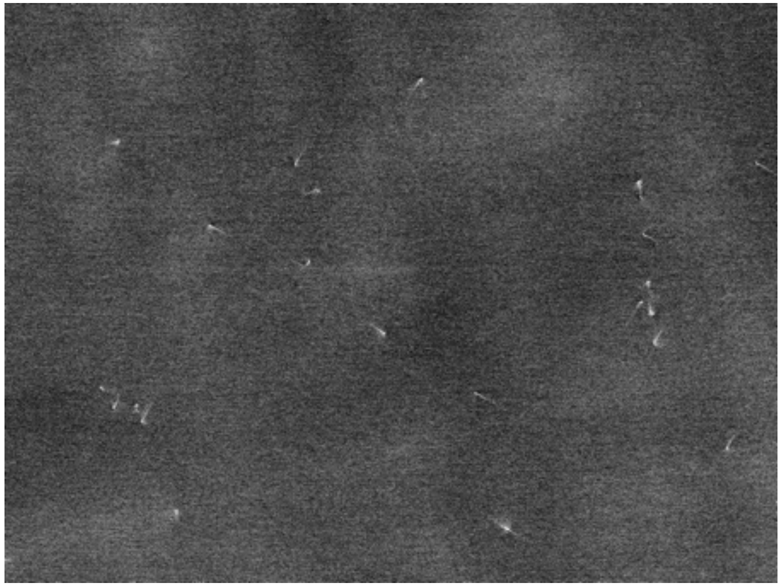


A wide range of dislo. densities can be measured accurately

GaSb on Si (001) : Dislocation Density

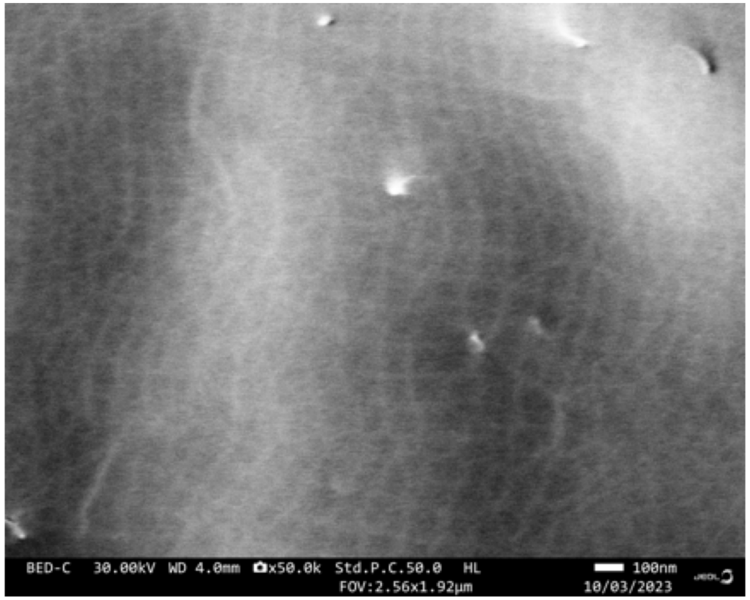


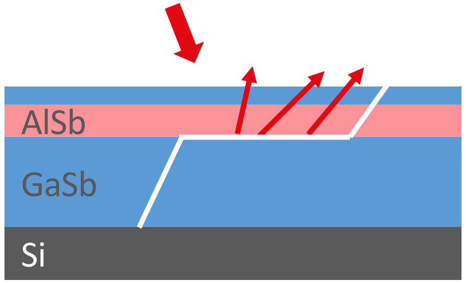
Statistic counting on sample C2345



Low threading dislocation densities
→ inhomogeneous distribution

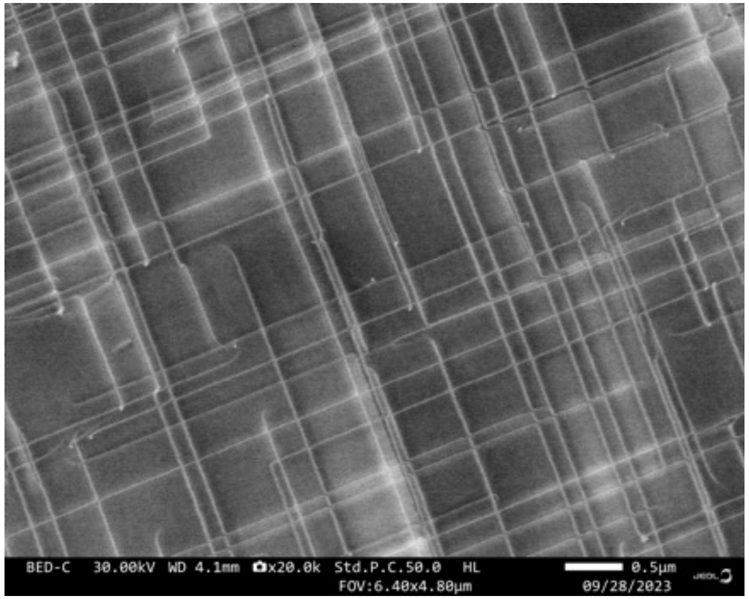
GaSb on Si (001) : Misfit dislocation arrays

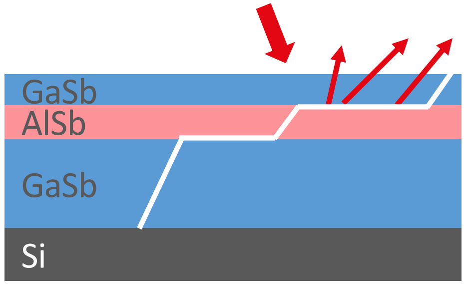




AlSb strain relaxed by the formation of an array of dislocation

Thicker GaSb cap layer → second relaxation step





Conclusion

ECCI is a powerful tool to study defects in epi layers :

- Non-destructive
- Relatively simple and quick (5 min to 1 hour)
- Very accurate dislocation density measurements
- Sensitive to the burger’s vector (invisibility criterion) → to be explored !