Efficiency and mechanism of dislocation density reduction during heteroepitaxial growth of diamond for detector applications

2nd ADAMAS Workshop @ GSI
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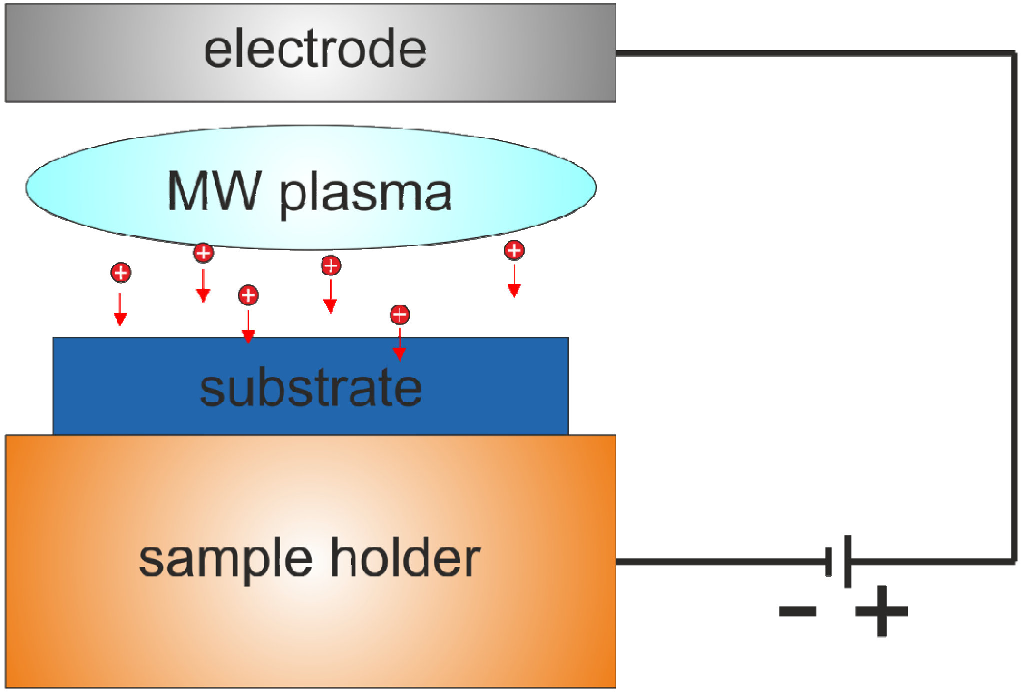
Michael Mayr, Christian Stehl, Martin Fischer,
Stefan Gsell, Matthias Schreck

Outline

- Growth and nucleation of heteroepitaxial diamond layers on Ir(001)
- Defects in crystals
- Dislocation density reduction by increasing thickness
- Raman and PL measurements on diamond cross sections
- Epitaxial lateral overgrowth (ELO)

Bias enhanced nucleation (BEN)



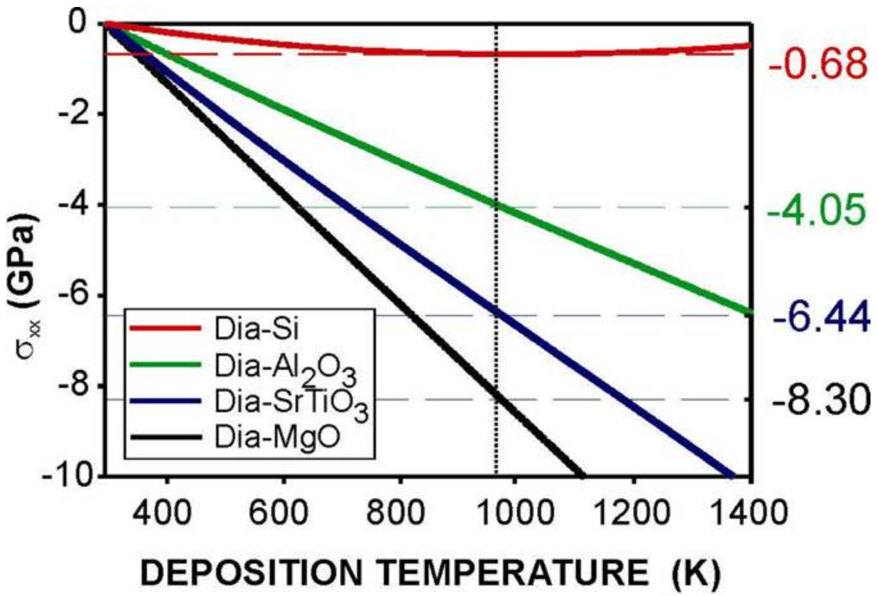
- bombardment of substrate surface with positively charged ions from a hydrogen / methane plasma
- electric field ~ several kV/cm

→ nucleation density reaches

<u>10^{11} cm^{-2}</u>

Multi-layer-system Ir/YSZ/Si(001)

Si: + high quality crystals
+ large size
+ low price



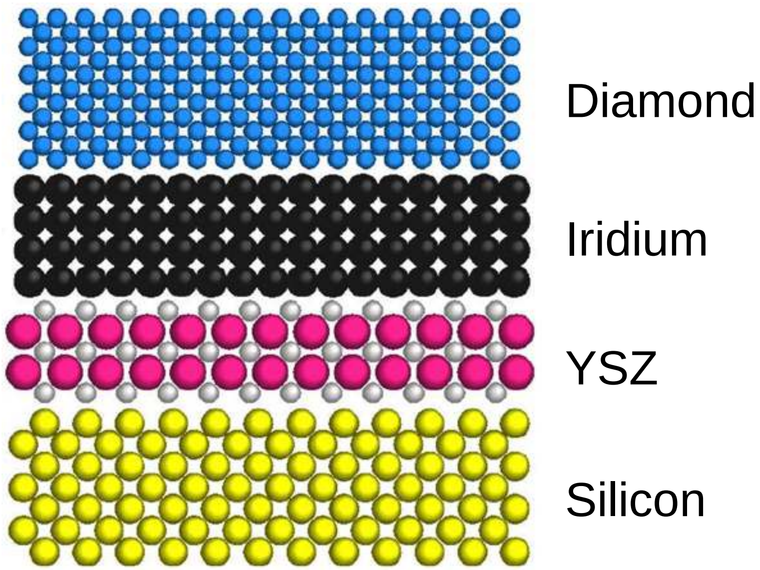
(Gsell, Bauer, Goldfuß, Schreck & Stritzker, 2004)

→ Low thermal stress!

but: diamond directly on silicon never reached single crystal quality

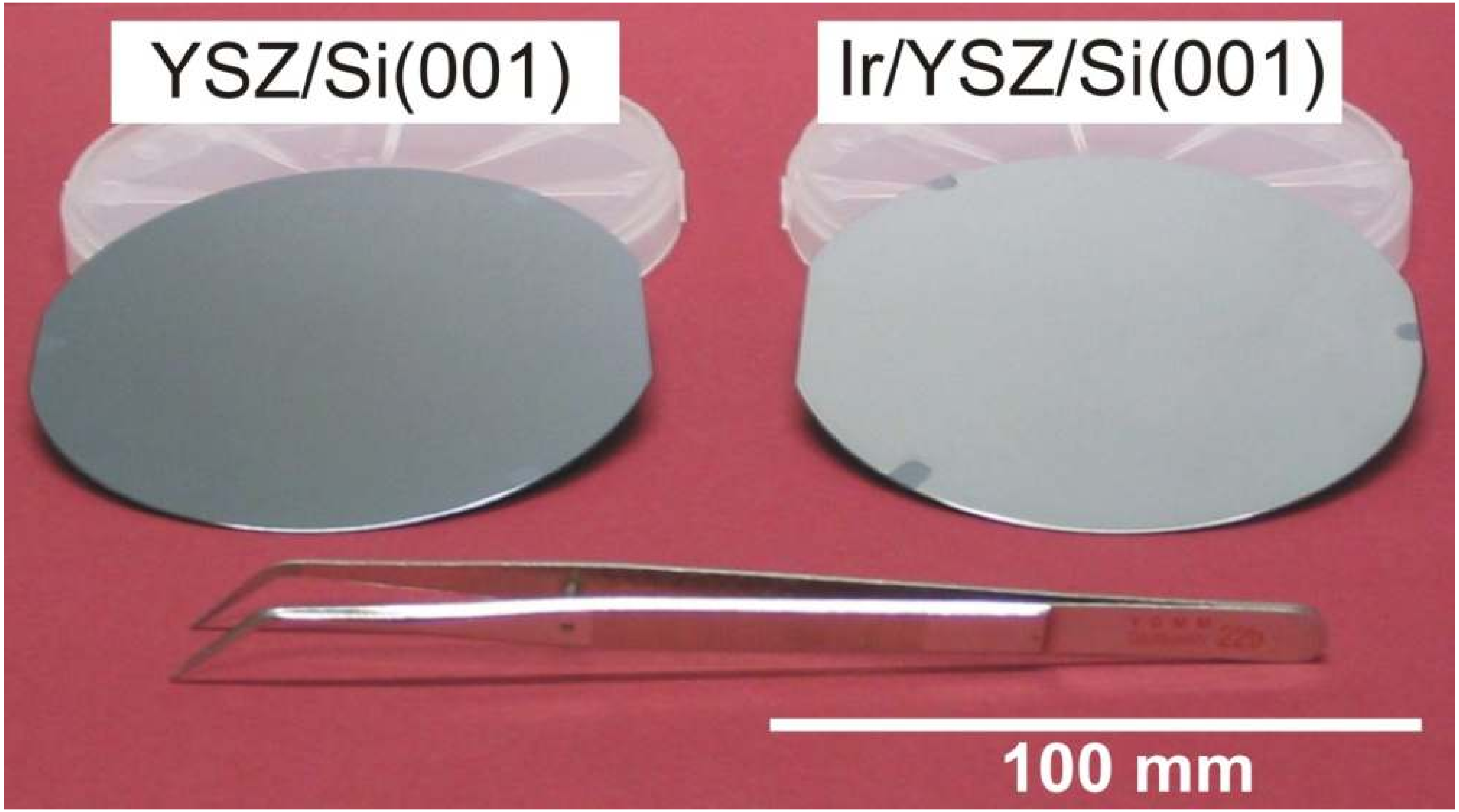
<u>Multi-layer-system (Ir/YSZ/Si(001))</u>

YSZ: yttria (Y_2O_3) - stabilized zirconia (ZrO_2)

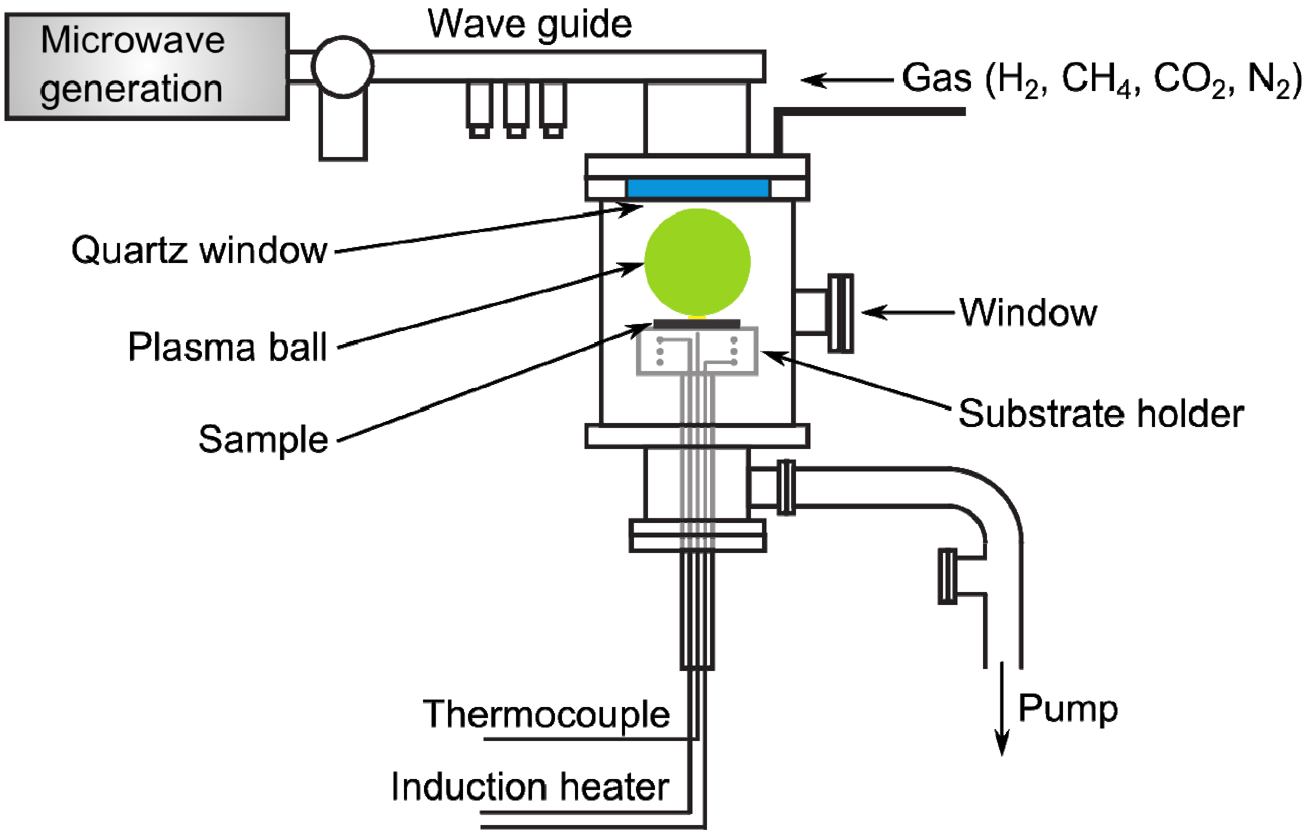


Lattice misfit related to diamond
~34,3% for Si and **~7,6%** for Ir

Diamond on Ir:
+ extremely high density of oriented nuclei
+ low mosaic spread of grains


YSZ/Si(001)
Ir/YSZ/Si(001)
100 mm

Growth by „Microwave enhanced plasma chemical vapour deposition“ (MWPCVD)



Schematic view and photograph of a CVD reactor.

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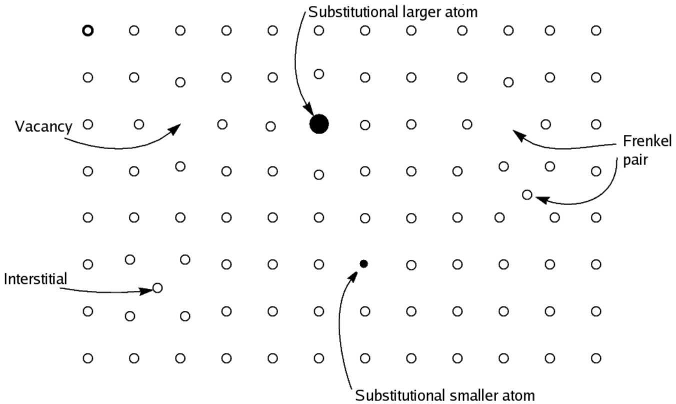
Defects in crystals

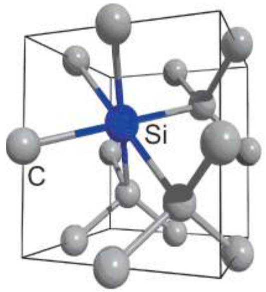
Universität Augsburg University

Point defects (0D)

Vacancies, interstitials, antisites, …



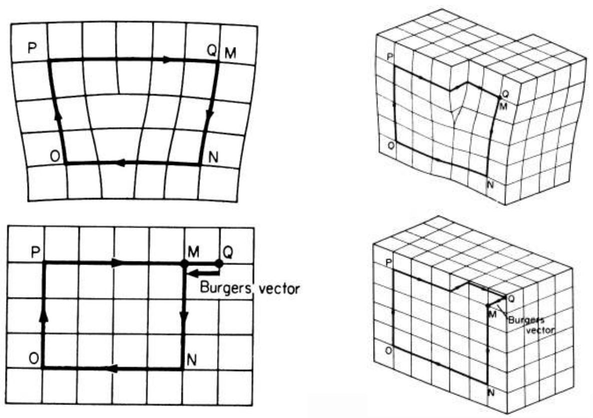
http://en.wikipedia.org/wiki/Crystallographic_defect



Neu et al., New J. Phys. 13 (2011) 025012

Line defects (1D)

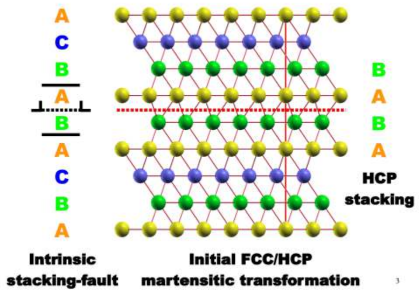
Edge/screw dislocations, mixed types



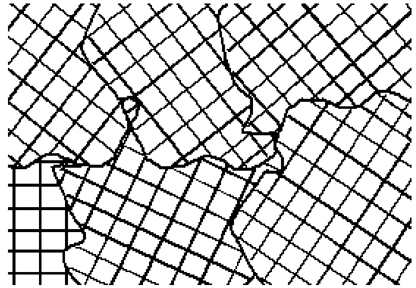
Hull & Bacon, Introduction to dislocations, 1984, Pergamon

Planar defects (2D)

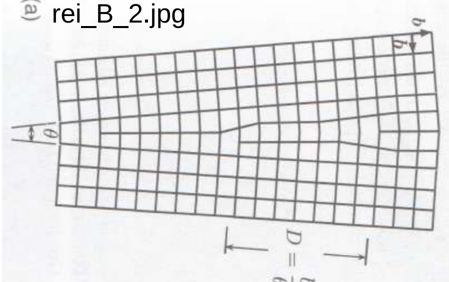
Stacking faults, grain boundaries, …



http://amadm.unileoben.ac.at/ReyesHuamantinco_Andrei_B_2.jpg



http://www.tf.uni-kiel.de/matwis/amat/mw_for_et/kap_4/illustr/korngrenze1.gif



C. Kittel: Einführung in die Festkörperphysik, 14th ed. (2006)

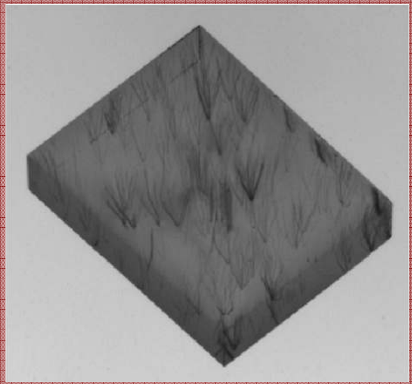
Symmetric small angle tilt boundary in cubic lattice (1 degree of freedom, in general: 5)

Bulk defects (3D)

Voids, precipitates, …

Imaging of dislocations

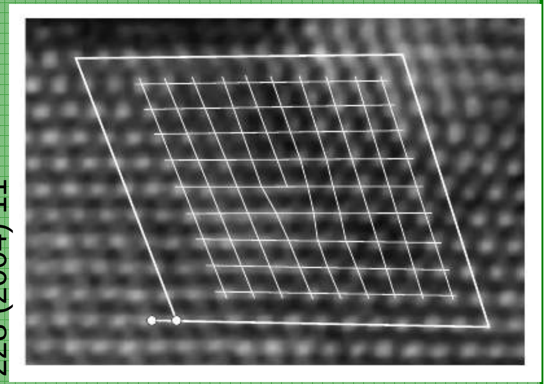
X-ray topography



M. Gaukroger et al., DRM 17 (2008) 262

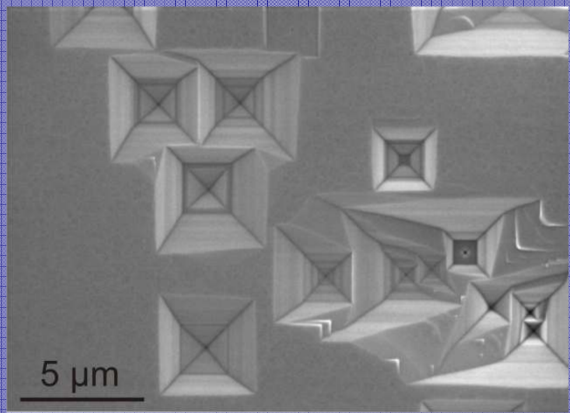
TEM

A.Blumenau et al., Defects and Diffusion, Forum 226-228 (2004) 11



0 | 10^5 | 10^8 | Dislocation density (cm^{-2}) | 10^{13}

Etching: generation of etch-pits



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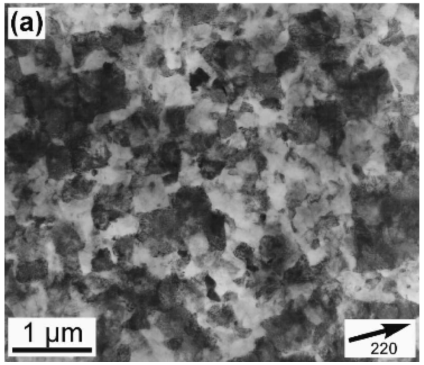
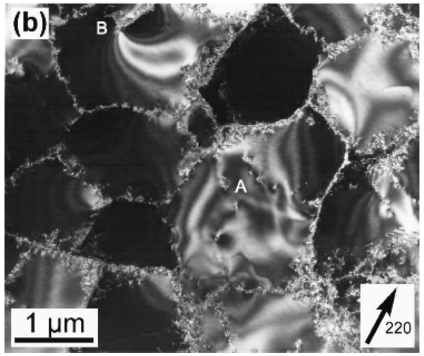
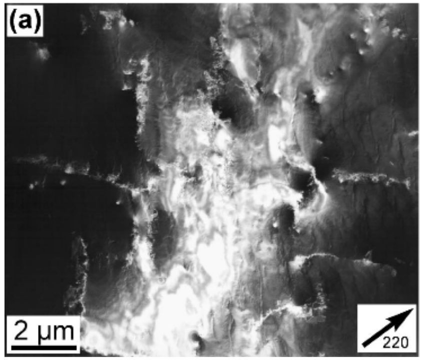
Change during growth process



0.6 µm



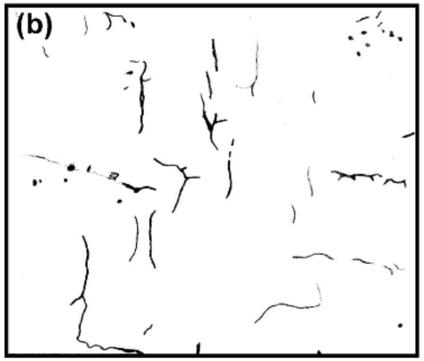
8 µm



34 µm

M. Schreck et. al.
Journal of Applied Physics **91**, 2002

Plan-view TEM images of epitaxial diamond layers grown on Ir/$SrTiO_3$(001) with increasing thickness.

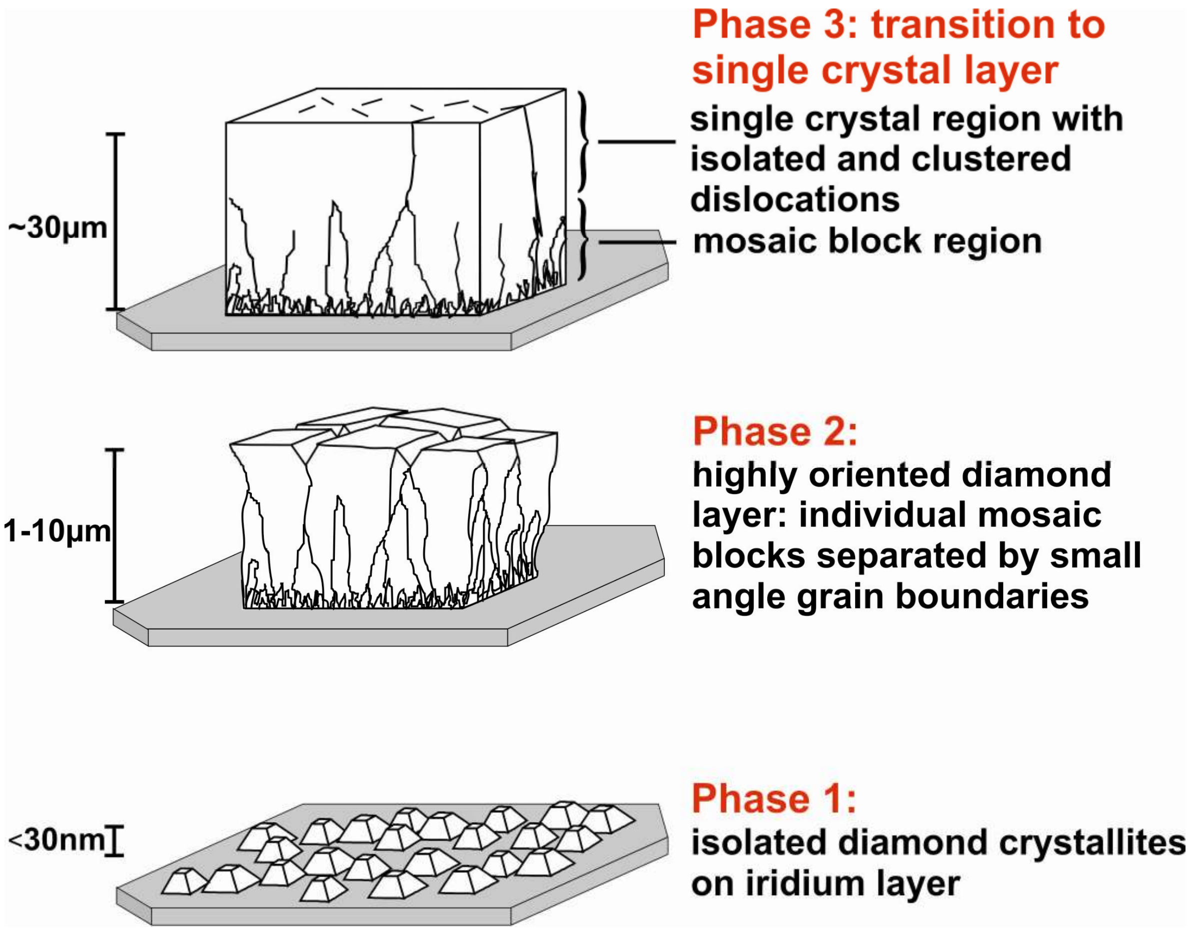


Drawing of the defect lines for 34 µm.

→ no longer isolated mosaic blocks bounded by polygonized network of grain boundaries

Highly oriented mosaic crystal becomes defective single crystal!

Variation of the defect structure with film thickness

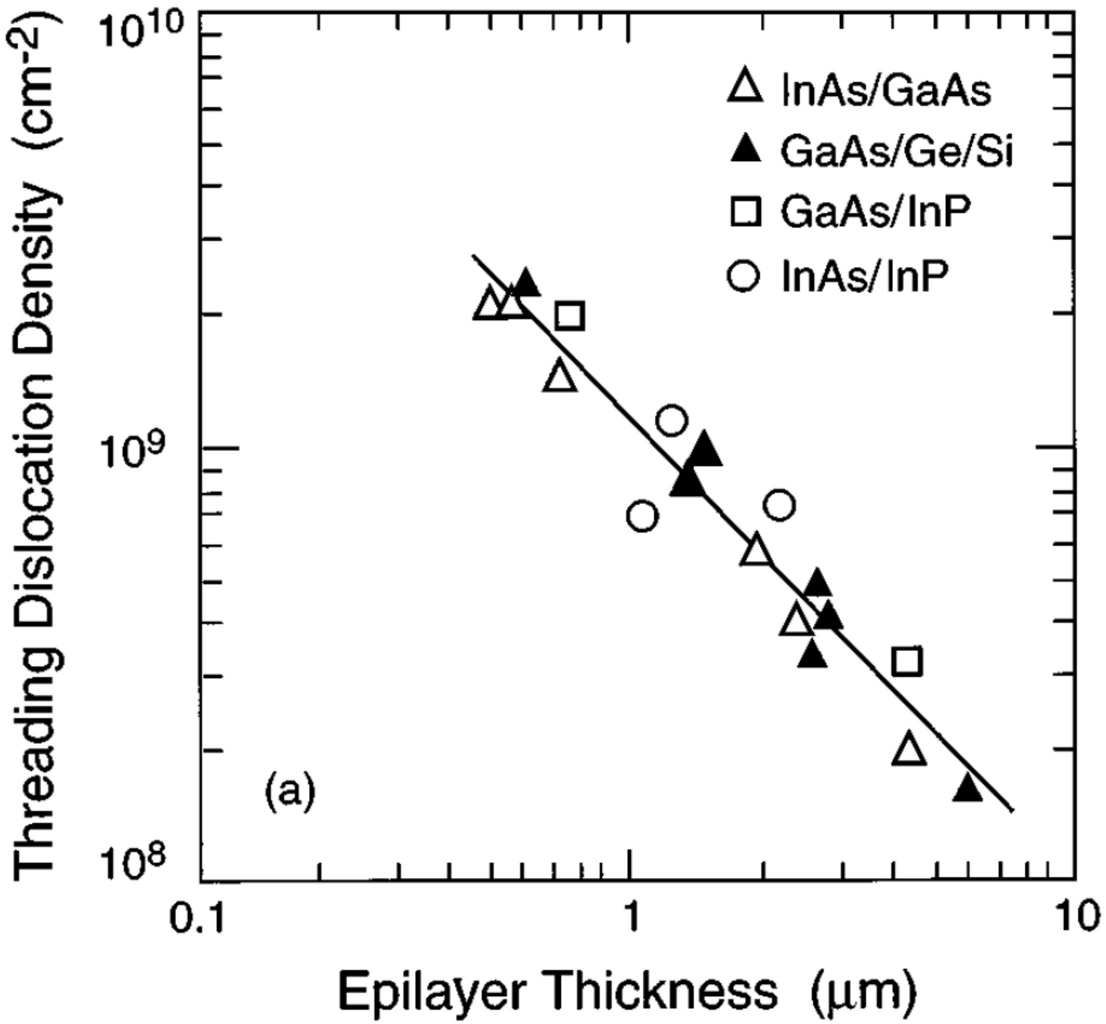


During growth, dislocations can interact and reduce their number by fusion or annihilation!

→ possible only if approach distance between two TDs with different Burgers vectors reaches a critical value r_a.

Changing distance can happen by means of **glide**, **climb** or **cross slip** of dislocations.
No real movement of dislocations but **„effective“ climb, glide** … during growth

Comparing to various semiconductor crystals



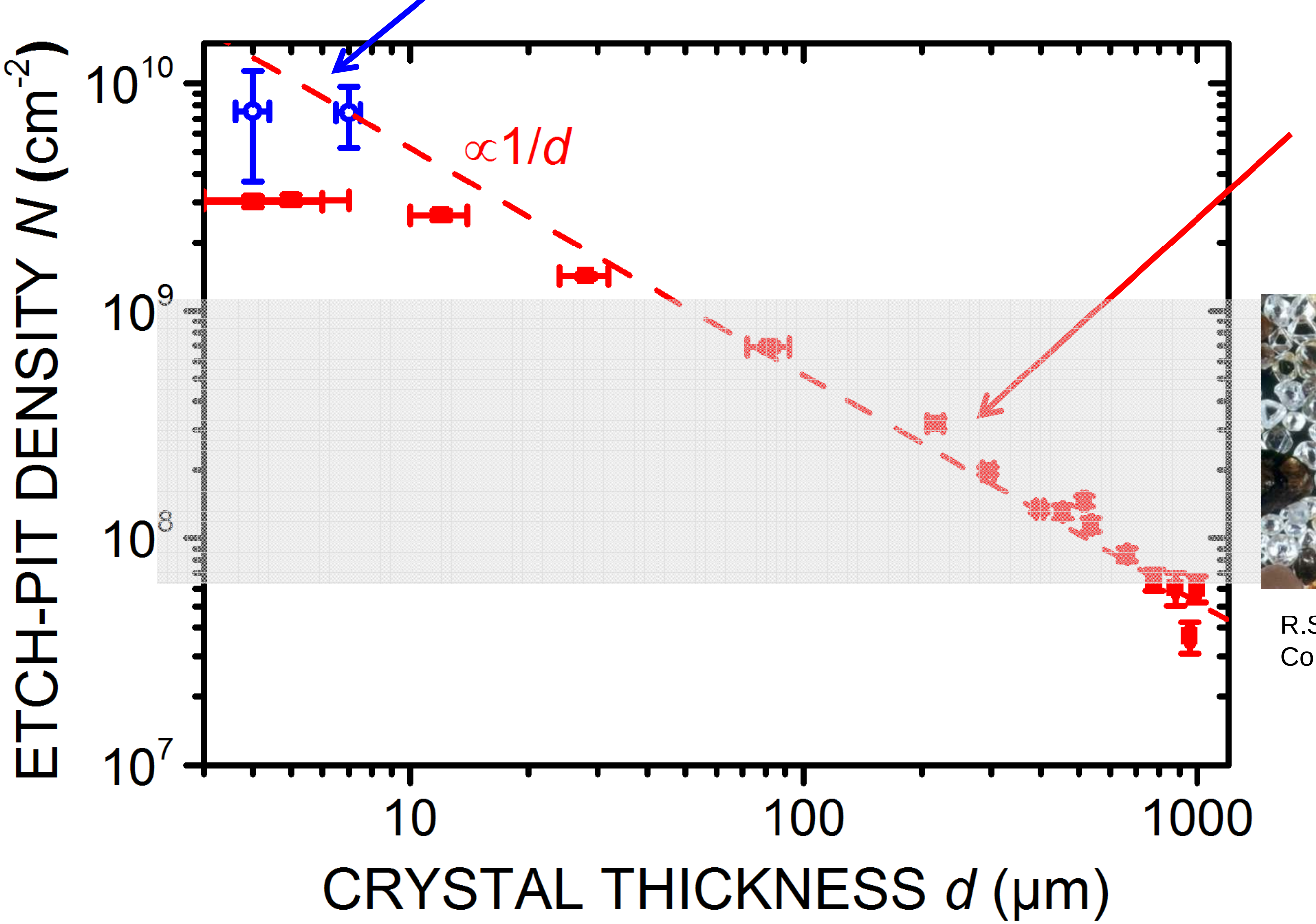
A. Romanov et al., PSS(B) 198 (1996) 599

Mathematical modeling yields a **1/d** dependance!

Experimentally proven for numerous semiconductor material crystals.

Variation of etch-pit density with crystal thickness

Data points from TEM measurements



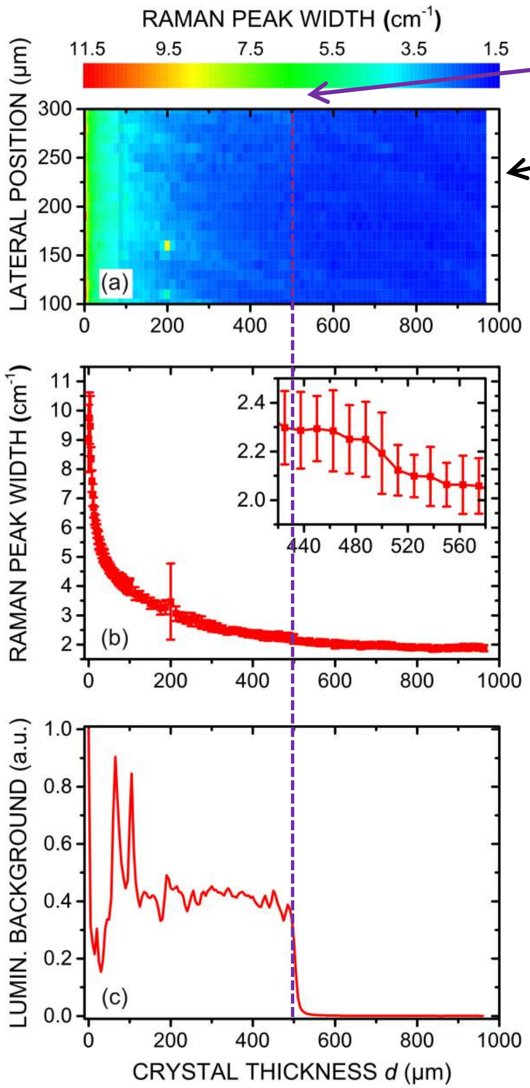
Data points from etching experiments

Natural IIa, brown - colourless

R.S. Balmer et al. J. Phys.: Condens. Matter **21** (2009) 364221.

C. Stehl et al. APL **103**, 151905 (2013)

Variation of the Raman and luminescence signal with crystal thickness



Nitrogen is switched off at d=500 µm

growth side

Drop in Raman FWHM, when nitrogen flux is switched off

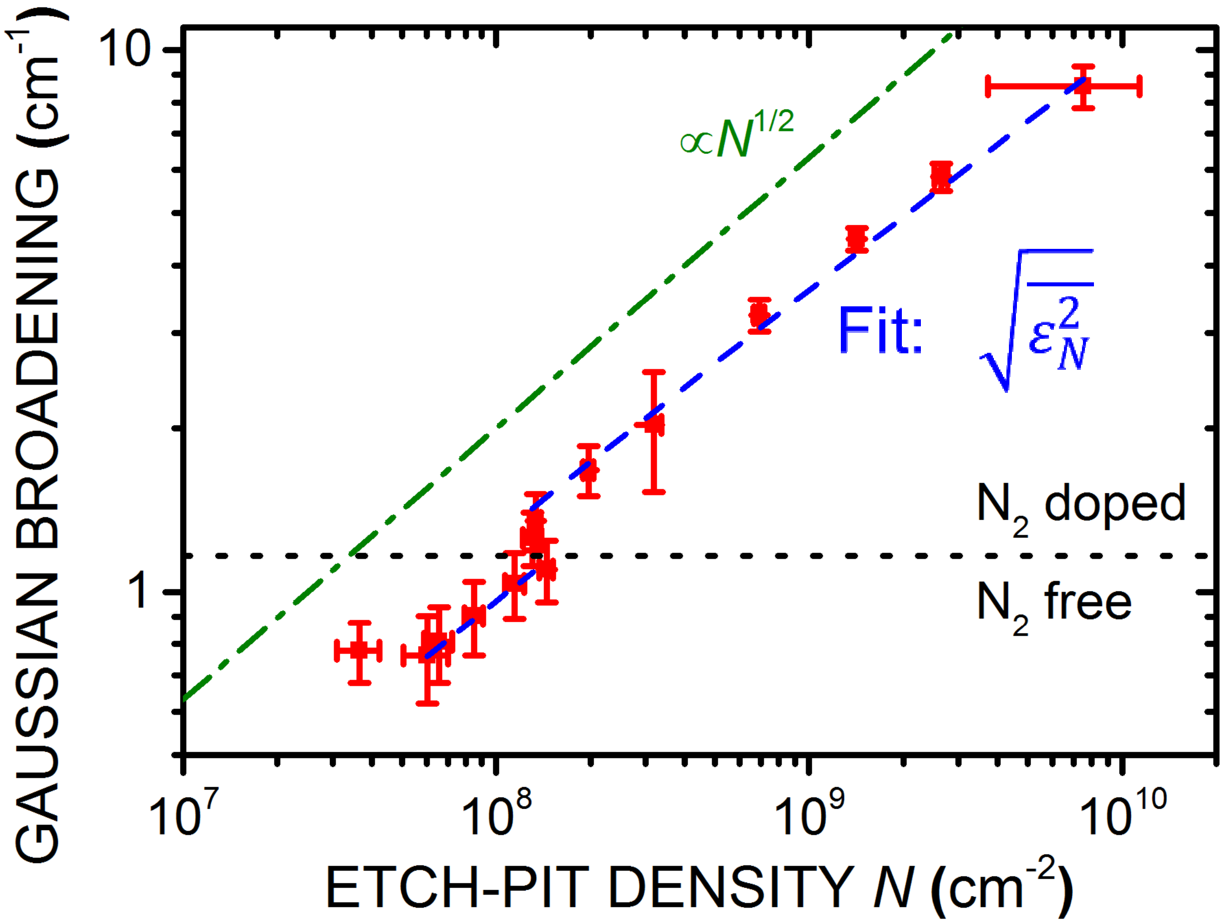
→ Broadening caused by point defects generated by adding nitrogen?

But also: Decrease of Raman FWHM with increasing thickness of the sample!

Possibility of creating a „calibration curve“ of Gaussian broadening vs. etch-pit density with previous data?

C. Stehl et al. APL **103**, 151905 (2013)

Correlation of Gaussian broadening and etch-pit density



C. Stehl et al. APL **103**, 151905 (2013)

Gaussian broadening predominantly caused by strain due to dislocations?

mean square strain in XRD analysis*:

$$\overline{\varepsilon_N^2} \propto N \times ln \frac{10^7}{2\sqrt{N}}$$

dislocation density N $\propto$ etch-pit density

(*) Hordon & Averbach, Acta Metall. 9, 237 (1961)

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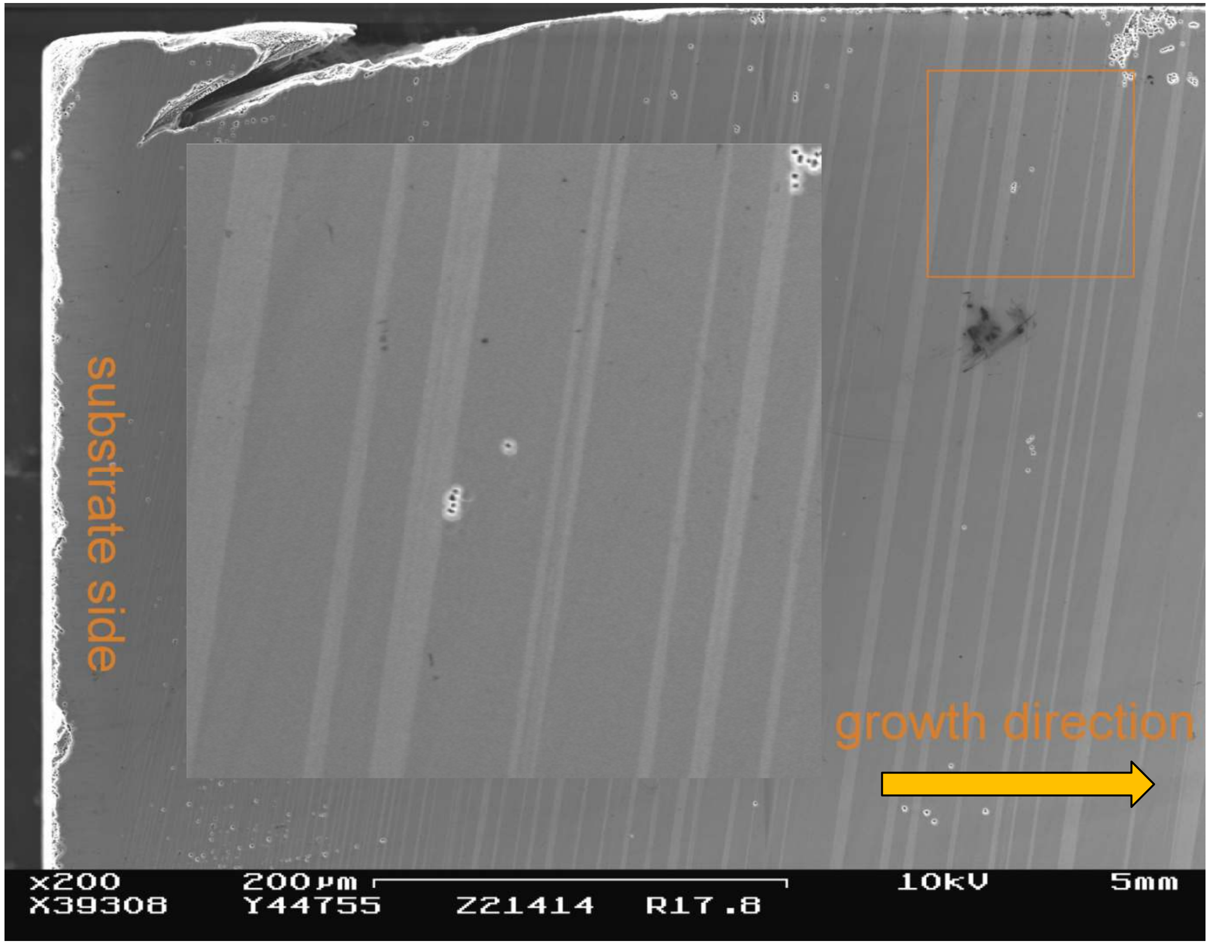
Raman- and PL-measurements on diamond cross sections



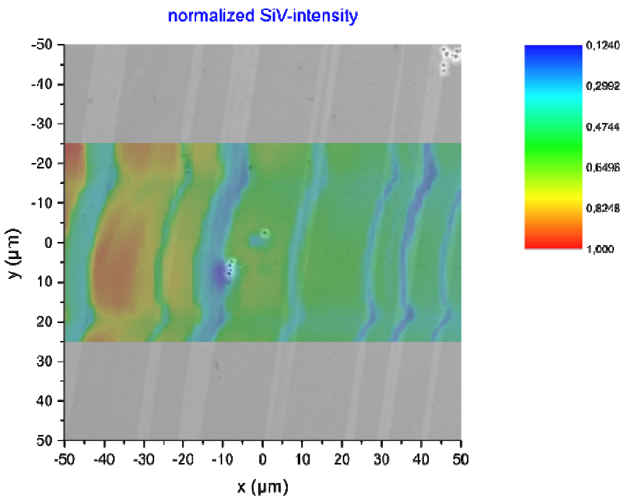
Measurements performed by C. Stehl

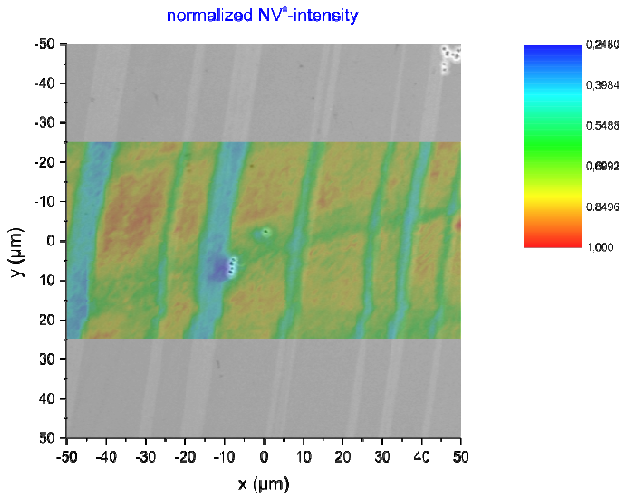
SEM-image of the cross section of a (001)-diamond layer

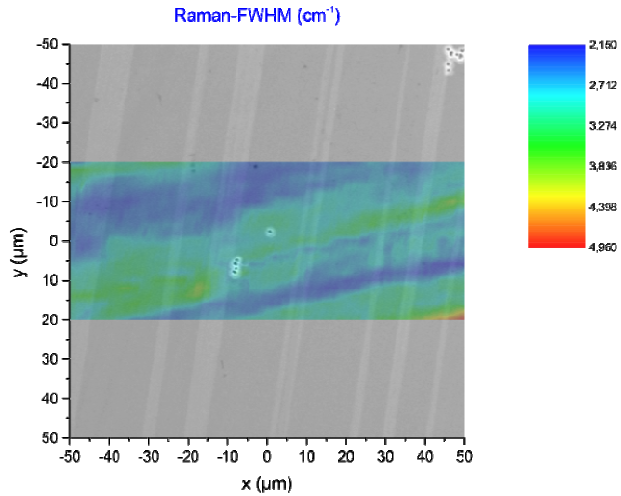
→ bright and dark stripes are risers/terraces

→ PL-/Raman-mapping of the orange marked section

Raman- and PL-measurements on diamond cross sections







Measurements performed by C. Stehl

→ Enhanced SiV-intensity in dark stripes

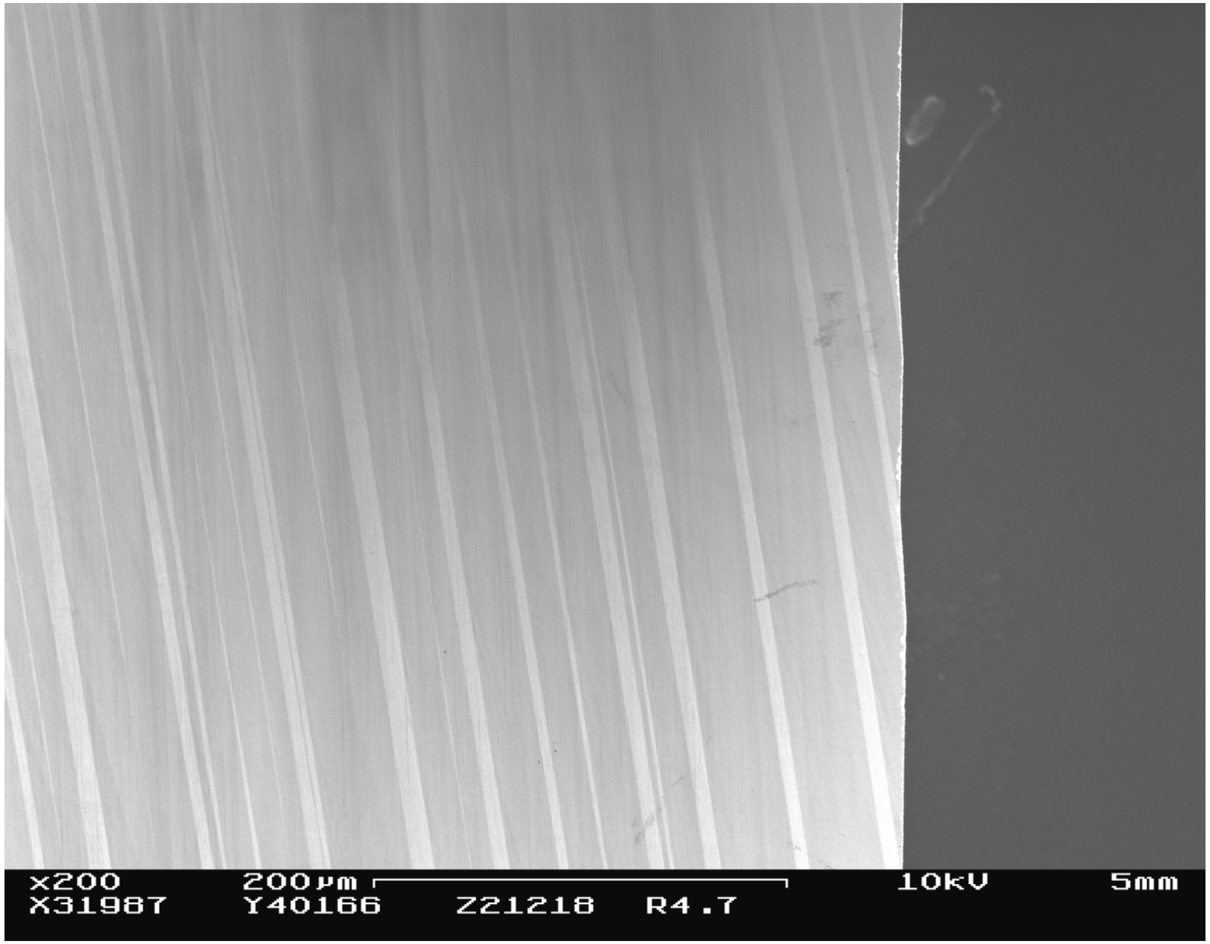
→ Enhanced NV^0-intensity in dark stripes

→ defect bands approximately in growth direction (tilt of ~12°)

- Identification of dark/bright stripes as risers/terraces?

- Correlation of Raman-FWHM broadening with etch-pit/dislocation density
  → Application of formerly obtained correlation data

Identification of risers / terraces



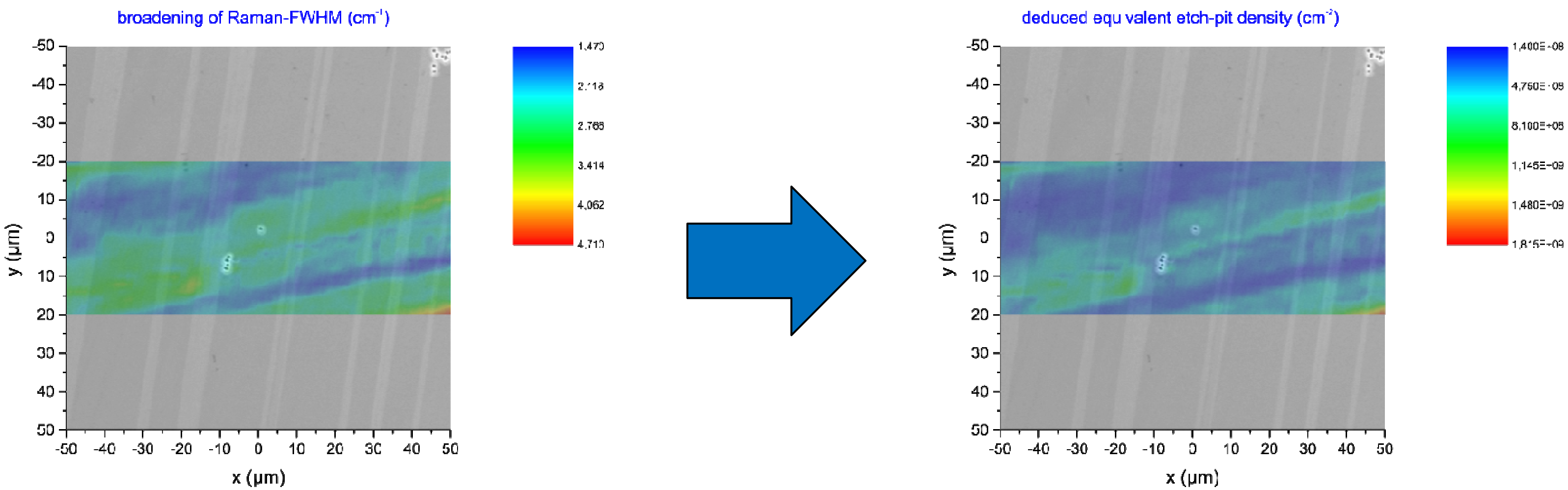
→ Difficulty of distinguishing risers from terraces.

Further investigation needed!

Measurements performed by C. Stehl

Dislocation network

Correlation of gaussian broadening with etch-pit density (using formerly obtained curve for calibration):



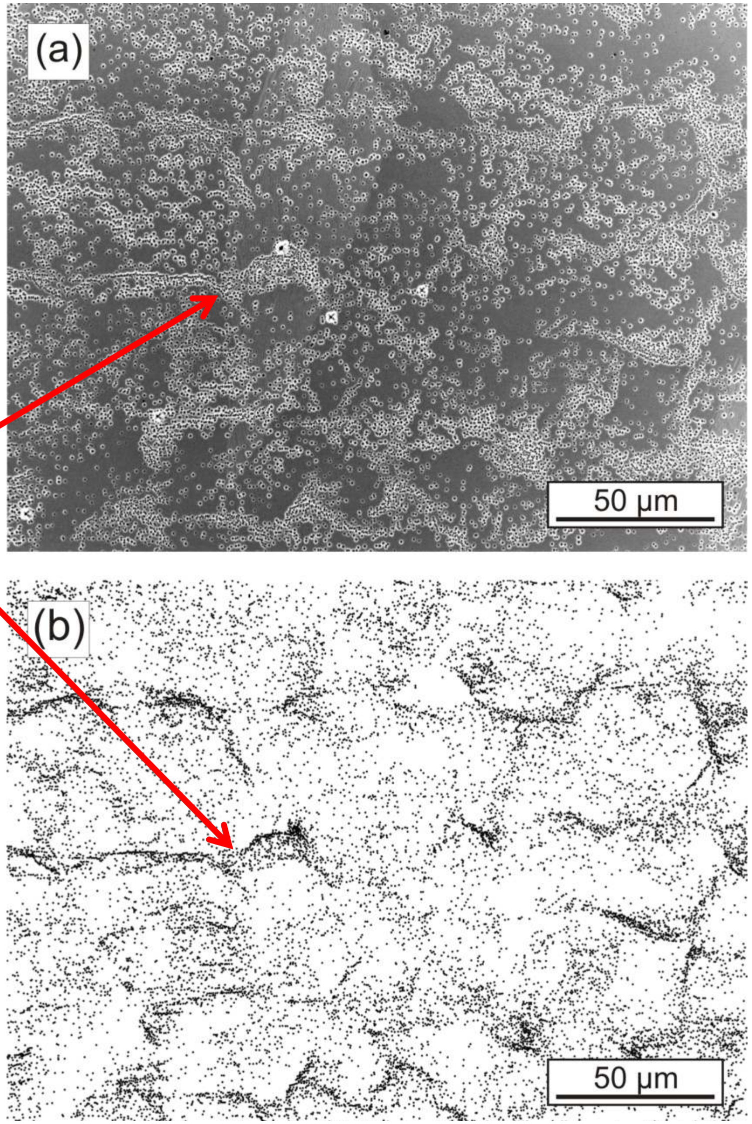
The estimated deduced equivalent etch-pit density varies by an order of magnitude over the measured cross section!

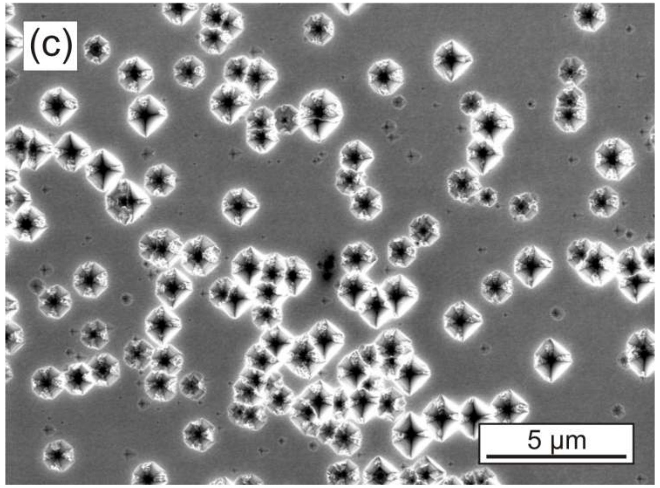
→ Variation caused by dislocation networks which develop out of grain boundaries with extended growth.

Dislocation network

Image of growth surface:

Clustering of dislocations in bands





M. Fischer et al., Diamond Relat. Mater. 37, 41 (2013)

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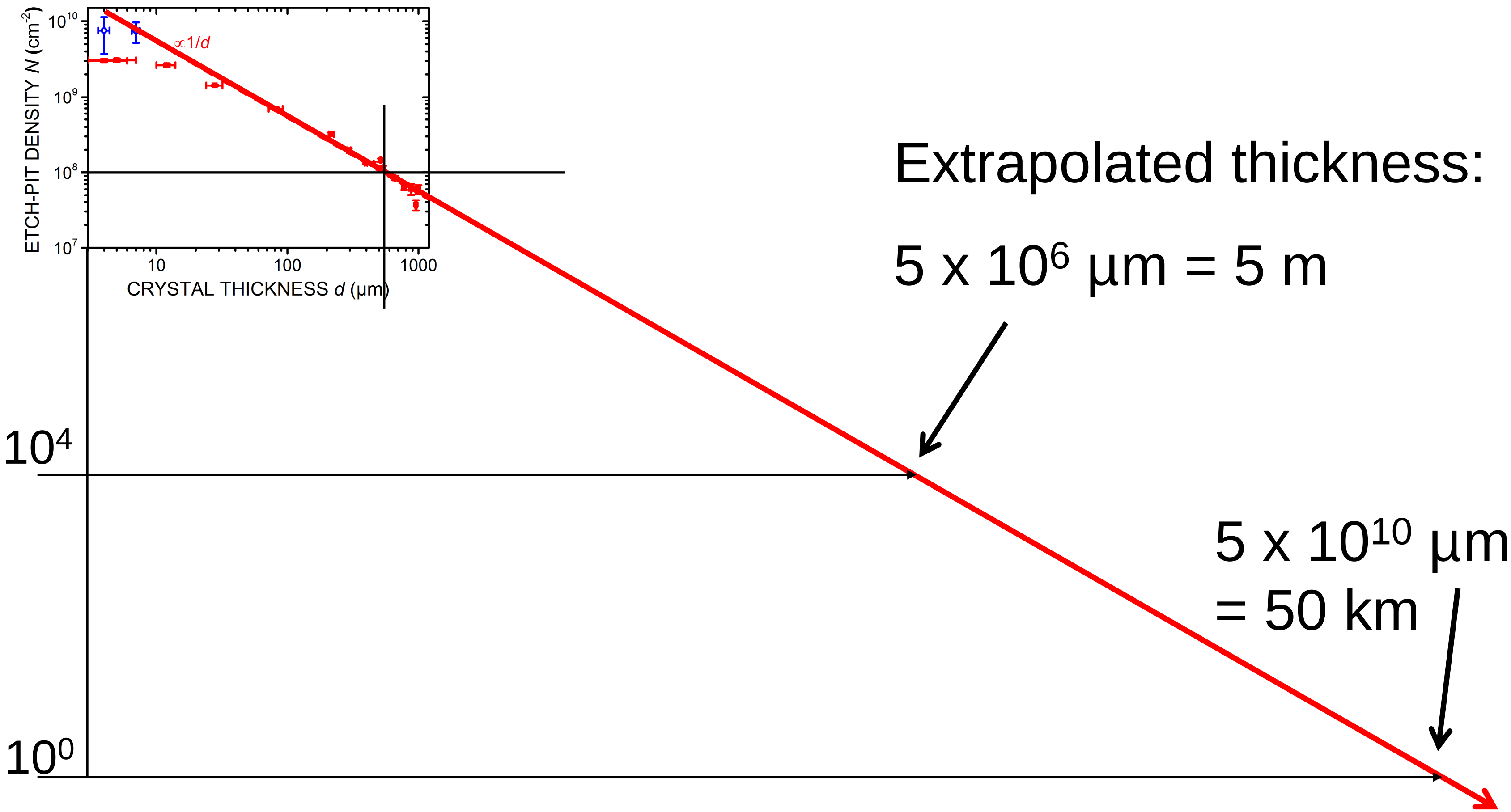
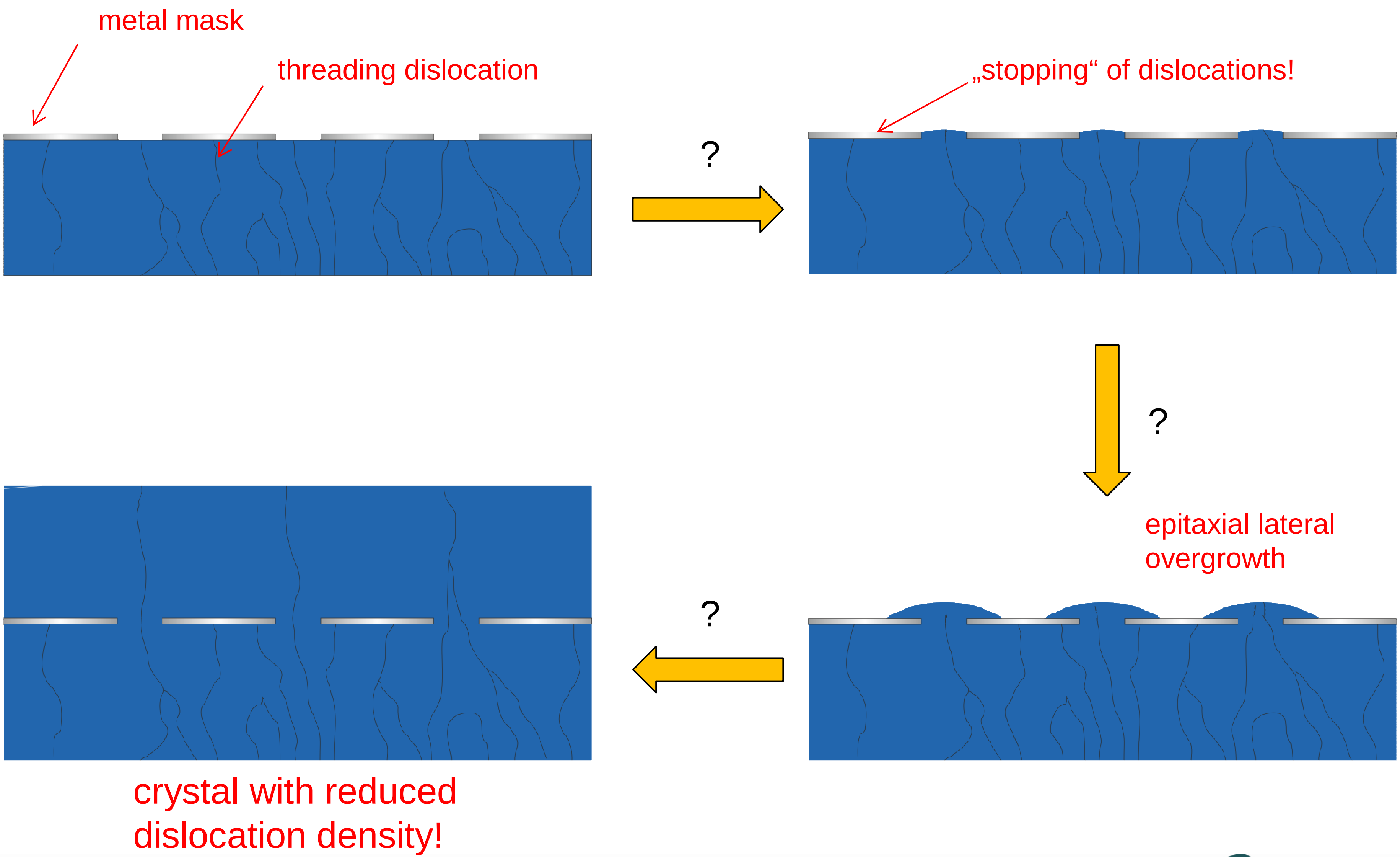
Extrapolation of etch-pit density



Further extended growth not practical → change of strategy needed!

Epitaxial lateral overgrowth (ELO)



Thank you for your attention!

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