TEM study of dislocations in diamond-on-iridium and first results of ELO experiments

5th ADAMAS Diamond Workshop 2016

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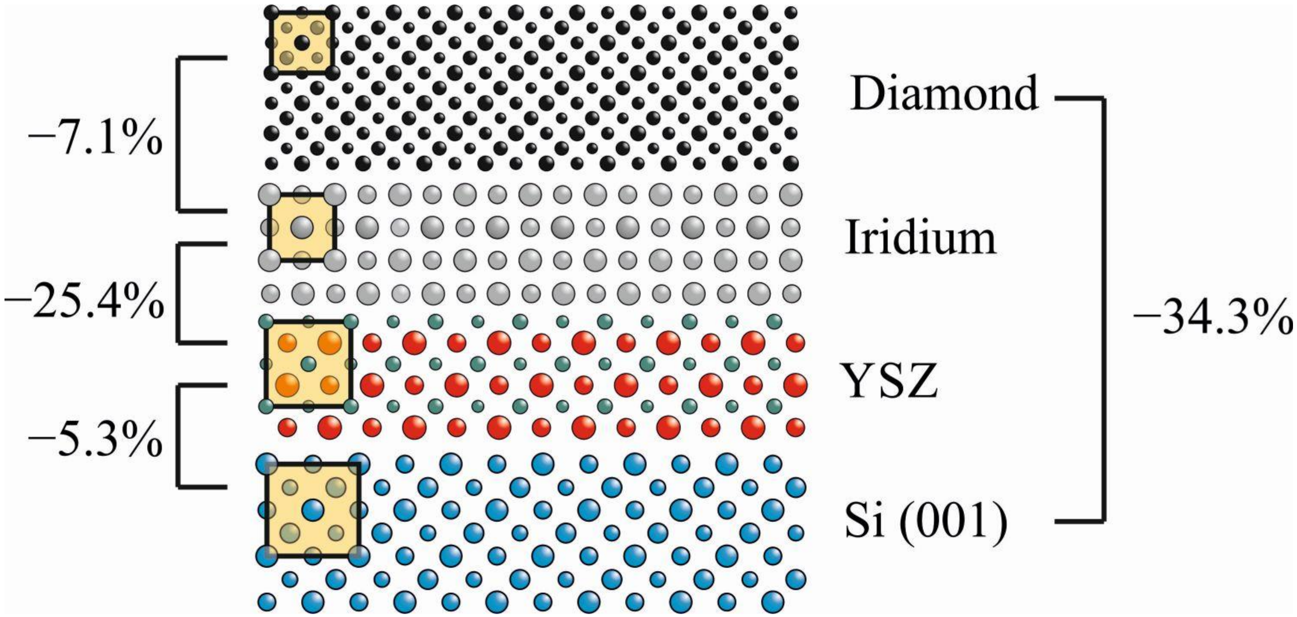
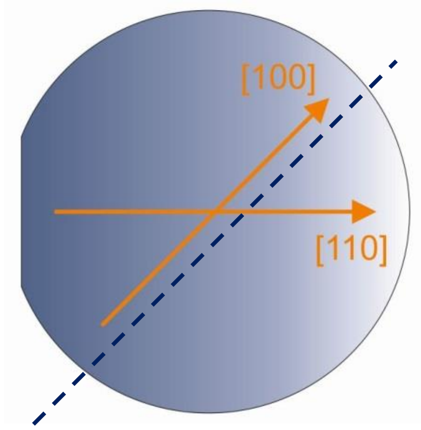
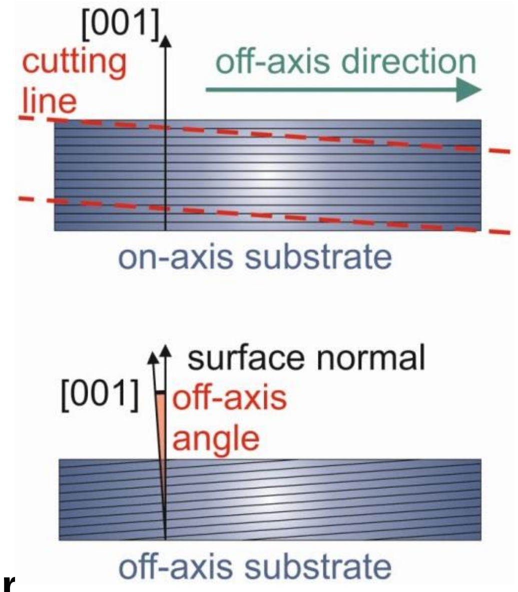
Heteroepitaxial diamond growth



off-axis growth:





2 off-axis directions:

- (001) off-axis towards [100]
- (001) off-axis towards [110]

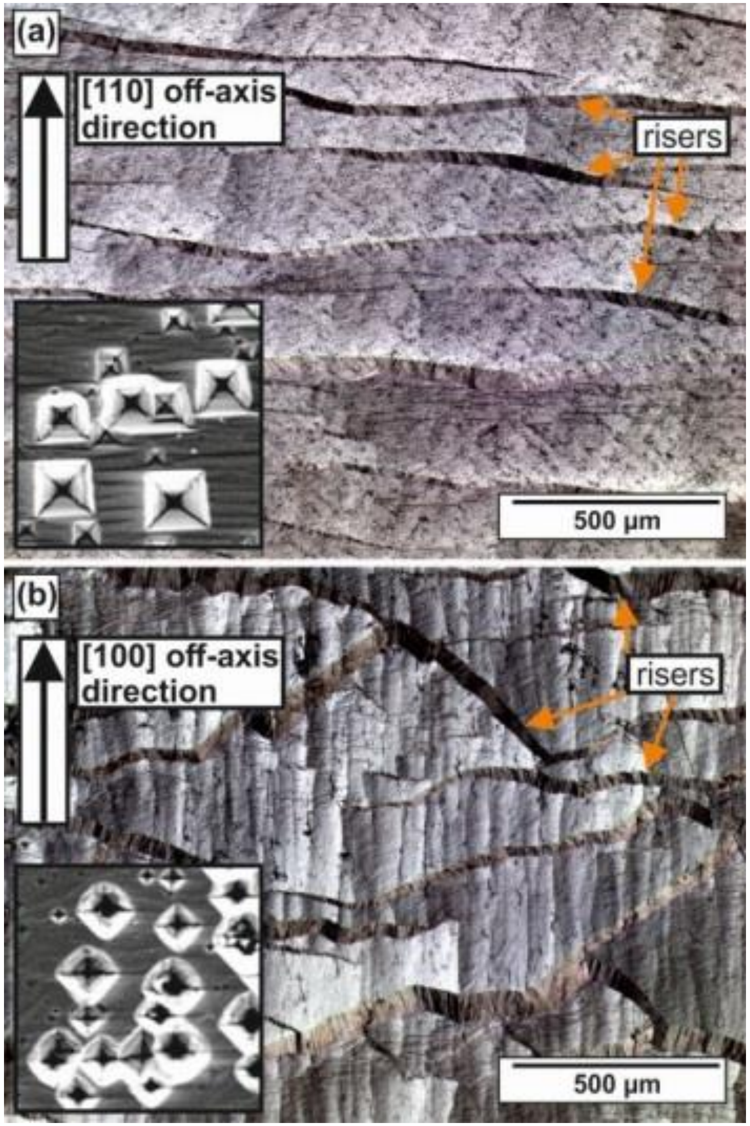
realized by **miscut of Si-substrate**

off-axis growth:

→ stabilization of growth surface
→ growth rate acceleration
→ suppression of non-epitaxial grains

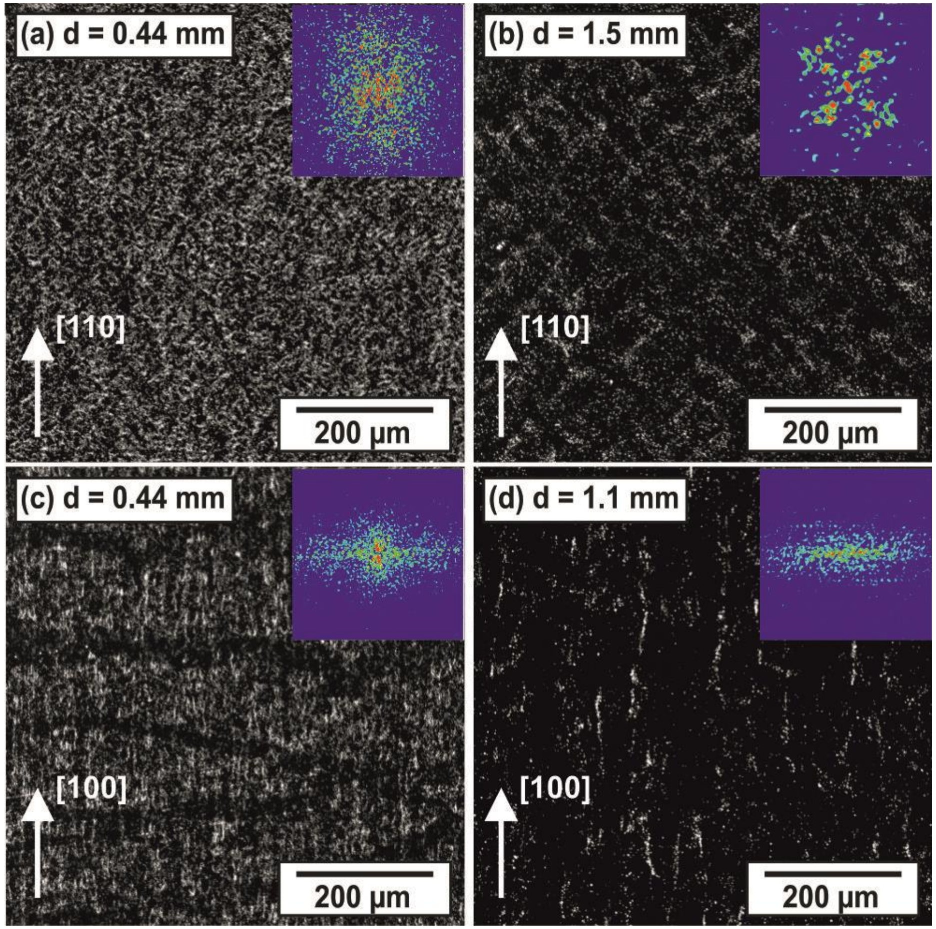
substrate:
4 inch Si(001) wafer

Etch-pit pattern reorganization and growth surface



DIC optical micrographs surface (inset: SEM)

Dark-field optical micrograph of etch-pit patterns (inset: FFT)

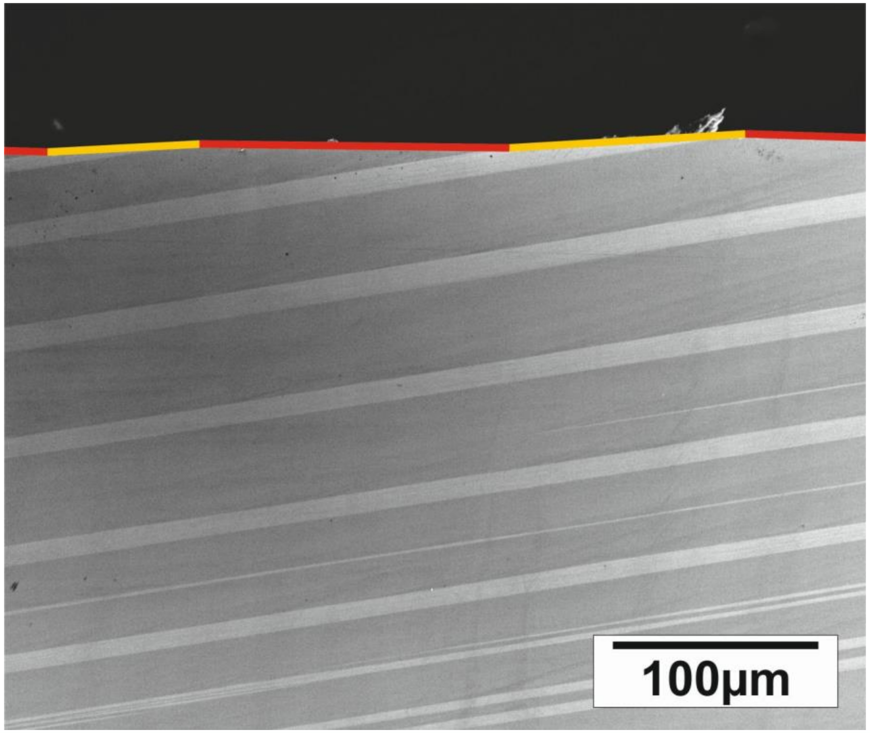


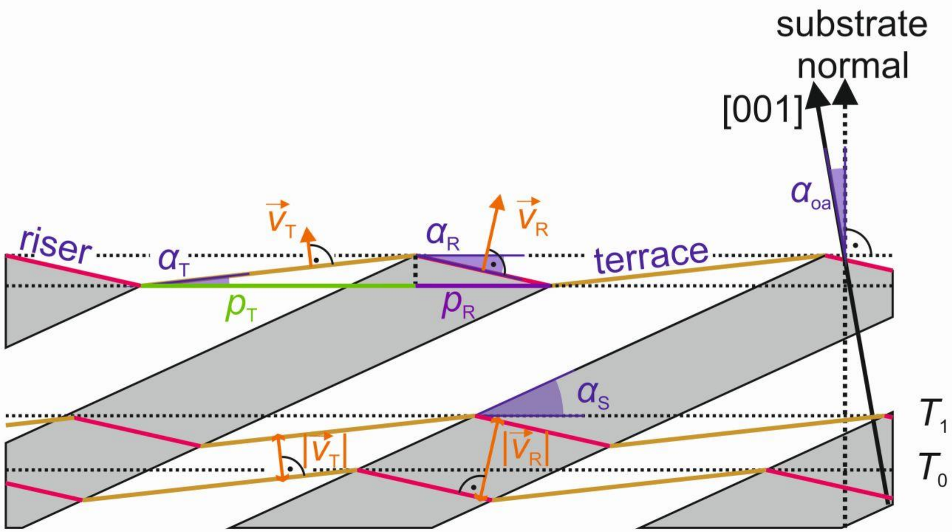
Motivation

During growth:

- formation of **distinct surface pattern**
- lateral **redistribution of dislocations**
- **striations** visible in cross-section SEM

Cross-section SEM
(surface marked in color)





As shown in:

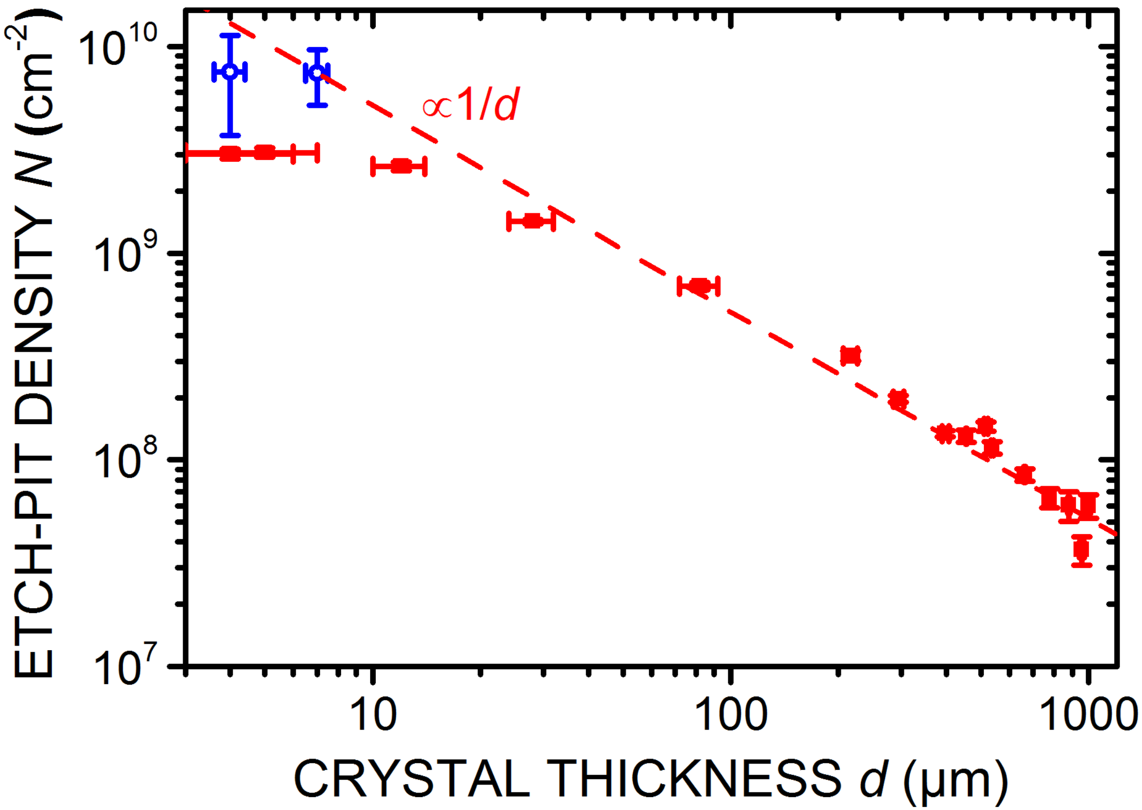
[13] M. Mayr et al., Phys. Status Solidi A 211, 2257 (2014).
[14] M. Mayr et al., Phys. Status Solidi A 212, 2480 (2015).

IMPORTANT QUESTIONS:

- How are dislocations tilted/redistributed?
- Interaction with surface features?
- Consequences of dislocational tilt?

Dislocation density reduction by extended growth

Dislocation density reduction by extended growth:



TEM (facilitates direct information about dislocation type and dynamics).

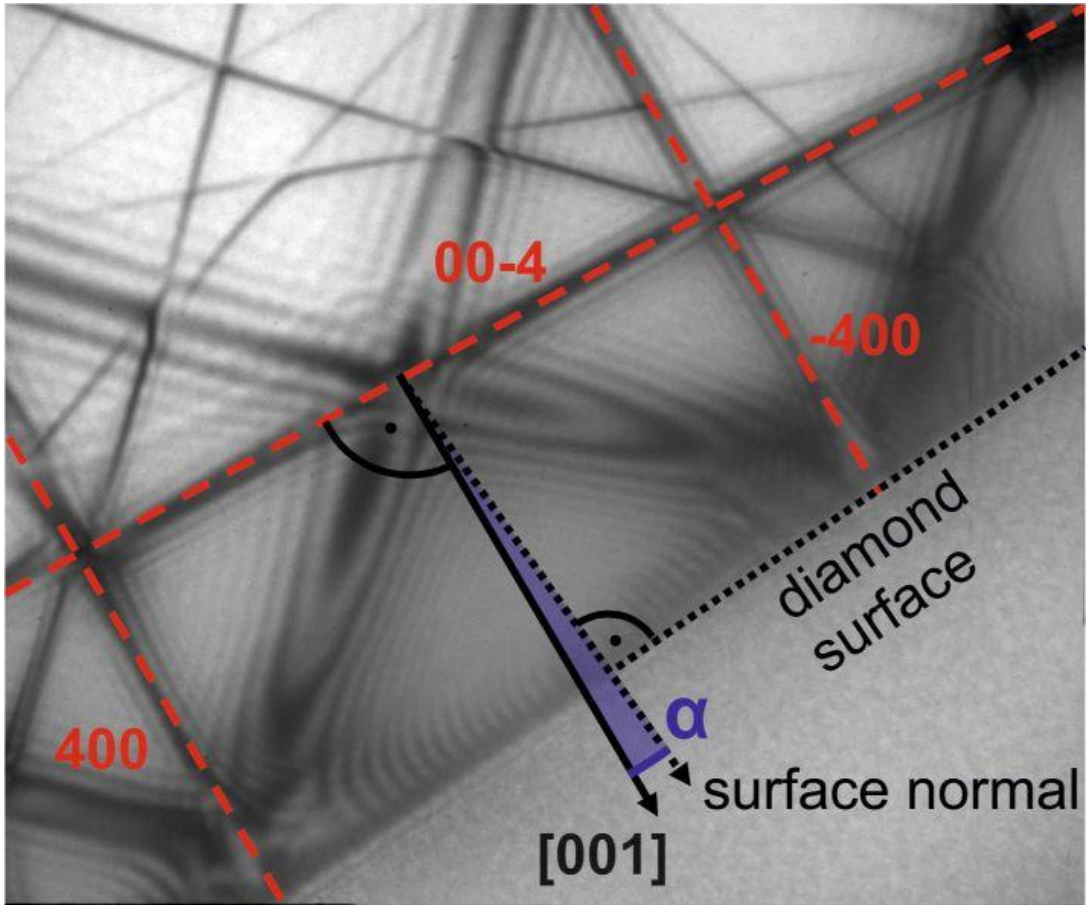
→ suitable density of dislocations necessary!

➔ **Suitable sample thickness: 12 – 34 µm**

As shown in:

C. Stehl et al. Appl. Phys. Lett. **103**, 151905 (2013)

TEM methods – LACBED



Large angle convergent beam electron diffraction (**LACBED**): overlap of reciprocal with direct image

⇩

exact estimation of **surface angle** with respect to the **lattice:**
ZOLZ Kikuchi lines (red dashed)

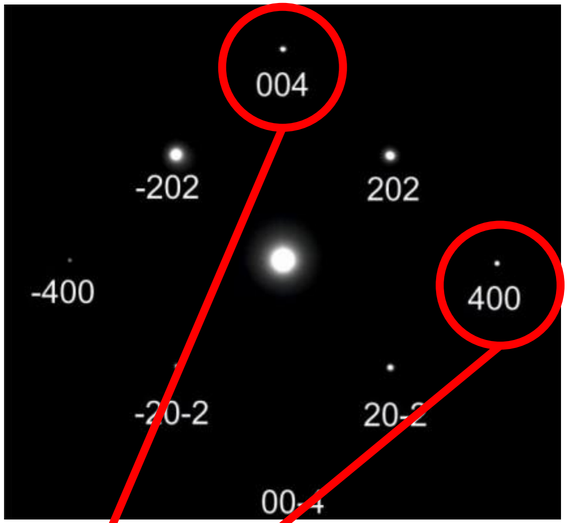
TEM methods – WBDF
Burgers vector analysis

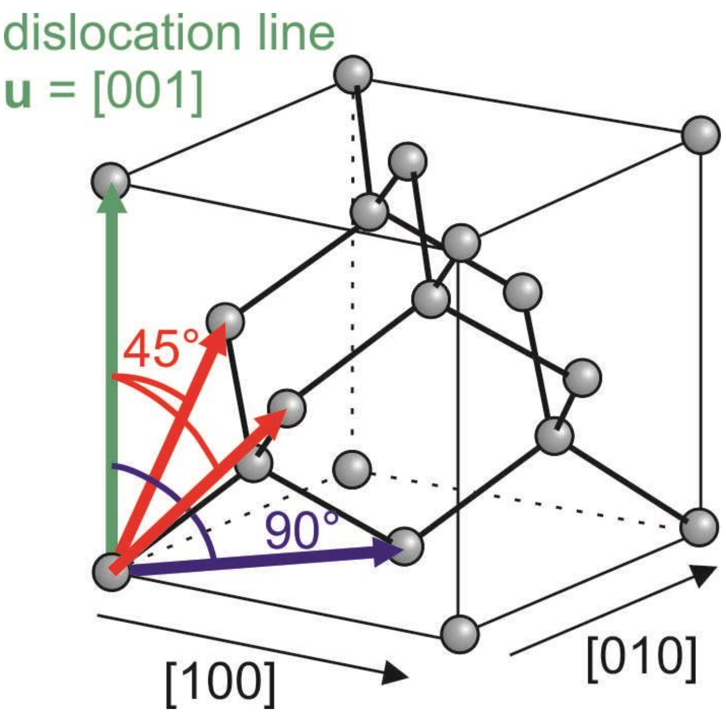
Weak beam dark field (**WBDF**)

⇩

g•b-criterion enables Burgers vector analysis

Selected area diffraction (SAD) pattern of diamond from the [010] zone axis.



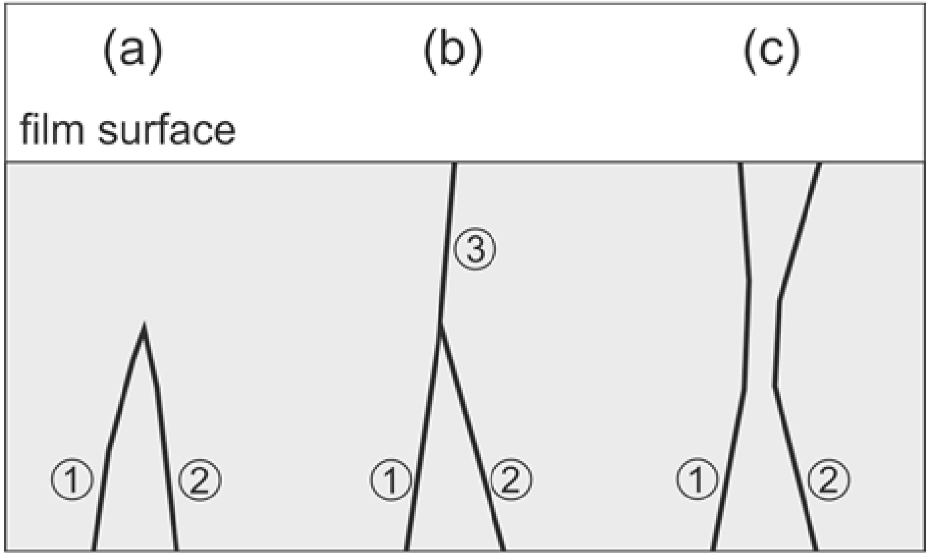


Product of possible perfect dislocation Burgers vectors **b** and **g**-reflections in the [010] zone-axis.

Perfect dislocations: **b** given by ½<110>

b \ **g**	004	400	type
±½ [110]	0	±2	**90° dislocation**
±½ [-110]	0	∓2	
±½ [101]	±2	±2	**45° dislocation**
±½ [011]	±2	0	
±½ [-101]	±2	∓2	
±½ [0-11]	±2	0	

Possible interaction mechanisms



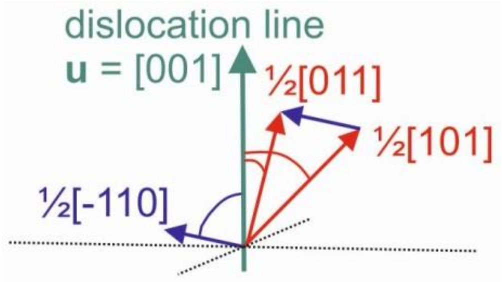
Reduction mechanism:

(a) $b_1 = -b_2$ Annihilation

(b) $b_3^2 < b_1^2 + b_2^2$ Fusion

(c) $b_3^2 > b_1^2 + b_2^2$ Scattering

Fusion: In principle all interactions within ½<110> (including resulting vector!) possible; otherwise: **scattering**.

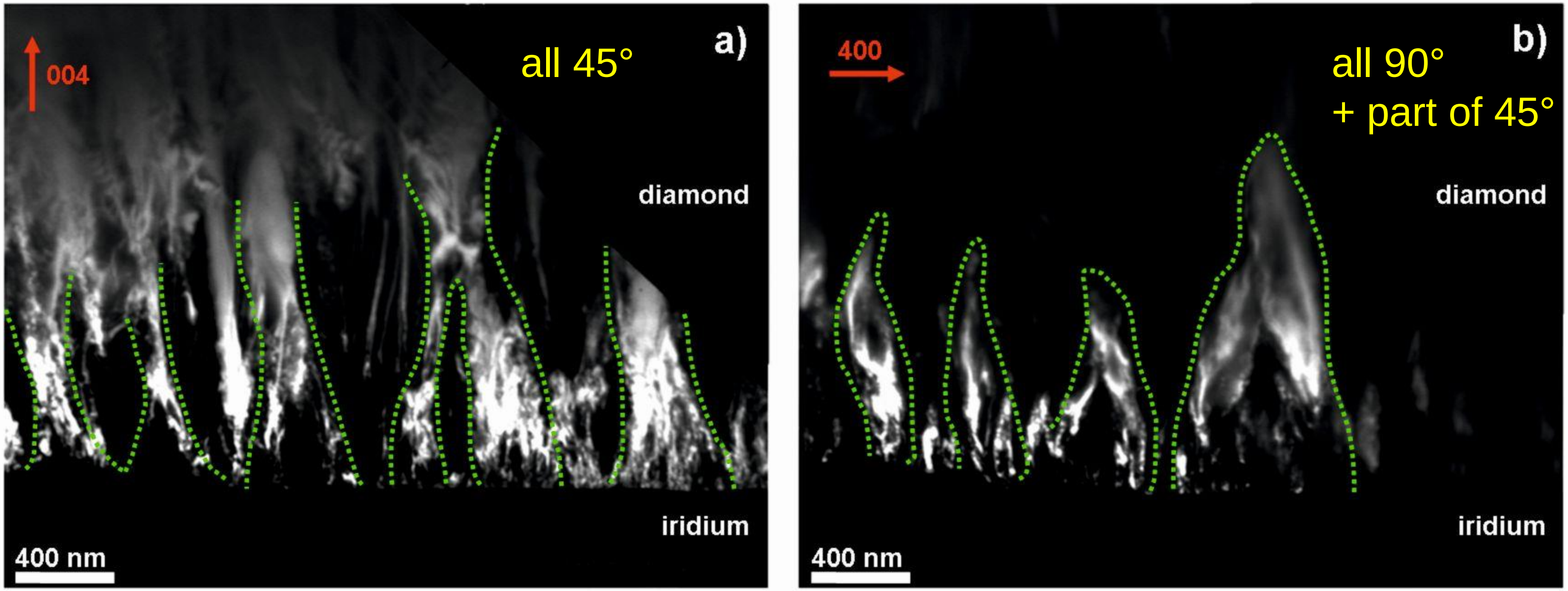


Fusion of dislocations (examples):

- 45° + 90° → 45° dislocation
  i.e. ½[101] + ½[-110] → ½[011]
- 45° + 45° → 90°
  i.e. ½[101] + ½[01-1] → ½[110]

Defect development – early growth stage

WBDF *cross-section* images close to iridium surface
identical sample area
004 (a) and 400 (b) reflection.

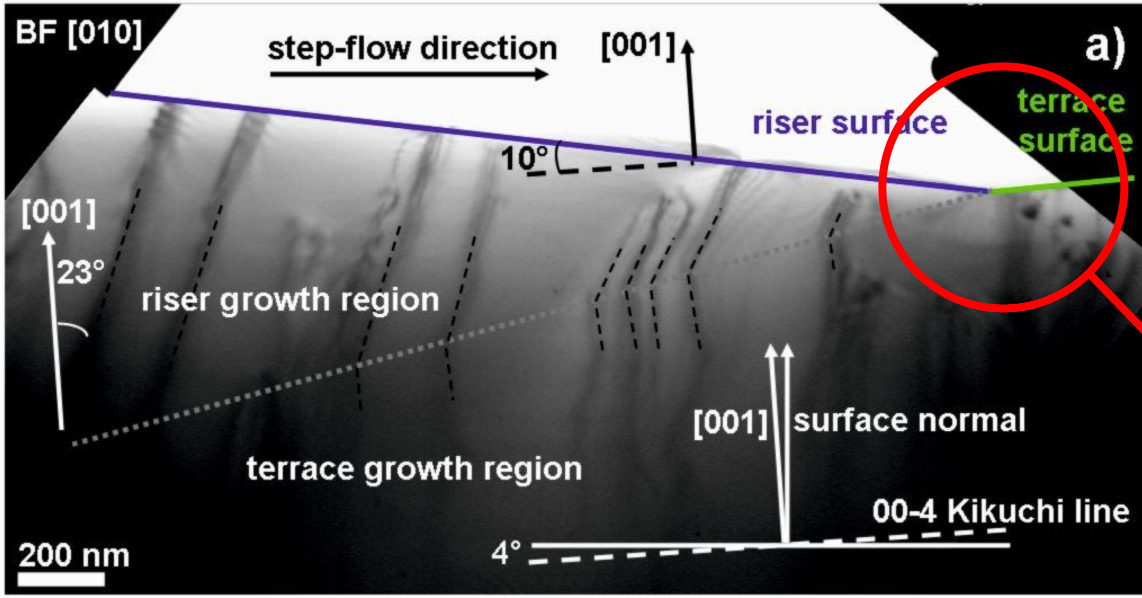


- ❖ Formation of bundles (green markings).
- ❖ Higher density of 45° dislocations (statistically: 2x).
- ❖ Highly effective reduction mechanism directly after nucleation.
- ❖ Higher efficiency for 90° (pure edge type) dislocation reduction.

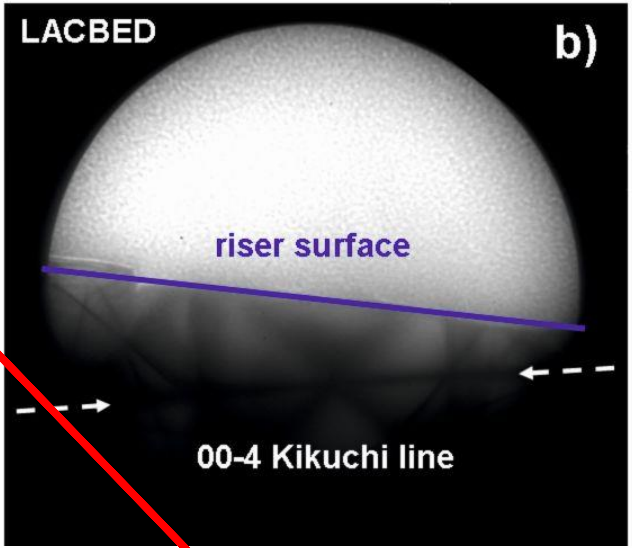
Dislocations at growth surface

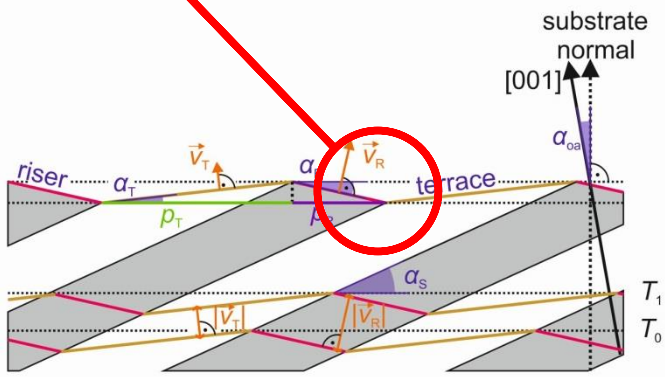
34µm growth (100ppm N_2 in the gas phase → step-bunching with **terrace/riser** structure).

Bright-field image (cross-section)



LACBED pattern

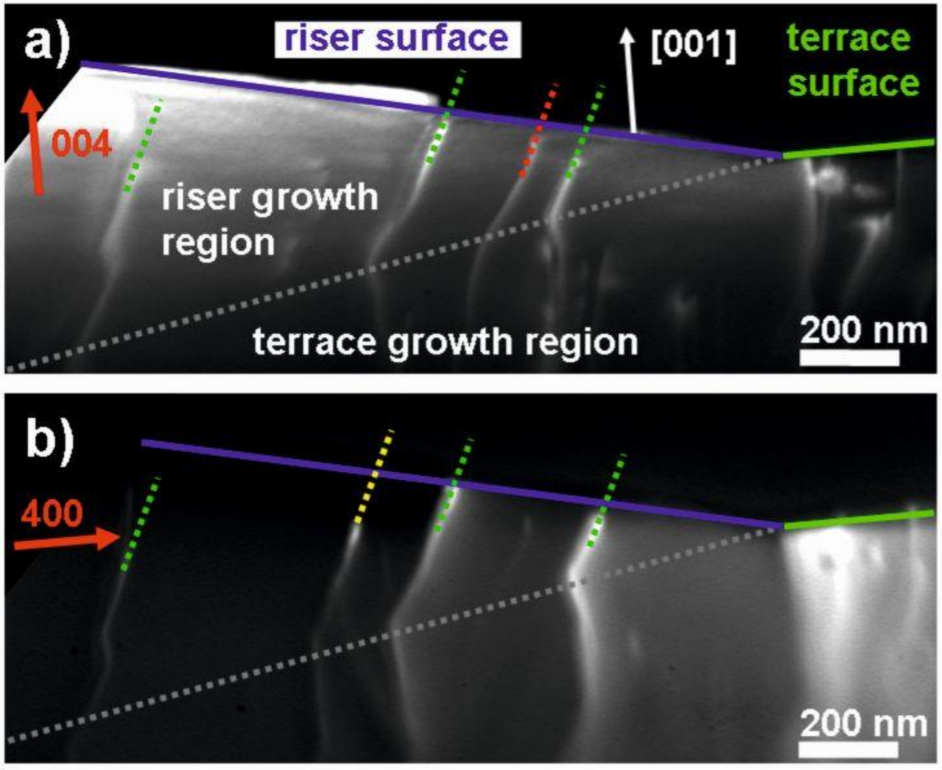




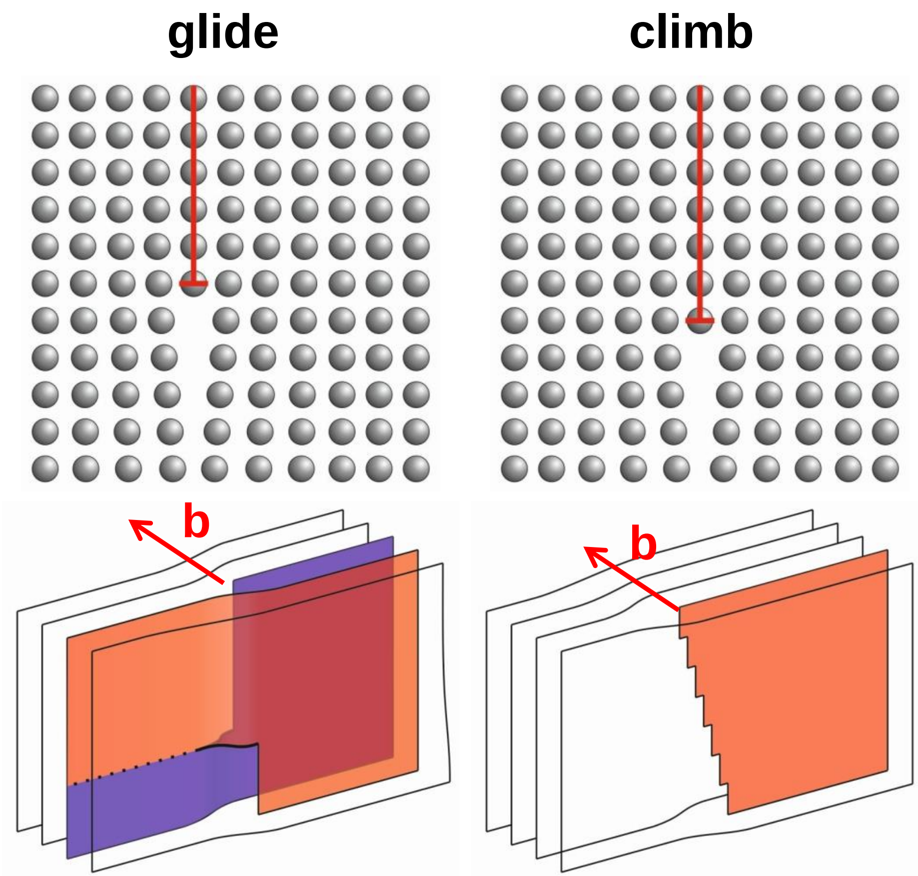
- ❖ bending of dislocations in step-flow direction (at grey dashed line)
- ❖ high inclination angle (23° away from [001]!).

WBDF analysis (riser area)

WBDF images (same spot)
004 (a) and 400 (b) reflection.



green: ½[±10±1] (45°)
red: ½[0±1±1] (45°)
yellow: ½[±1±10] (90°)



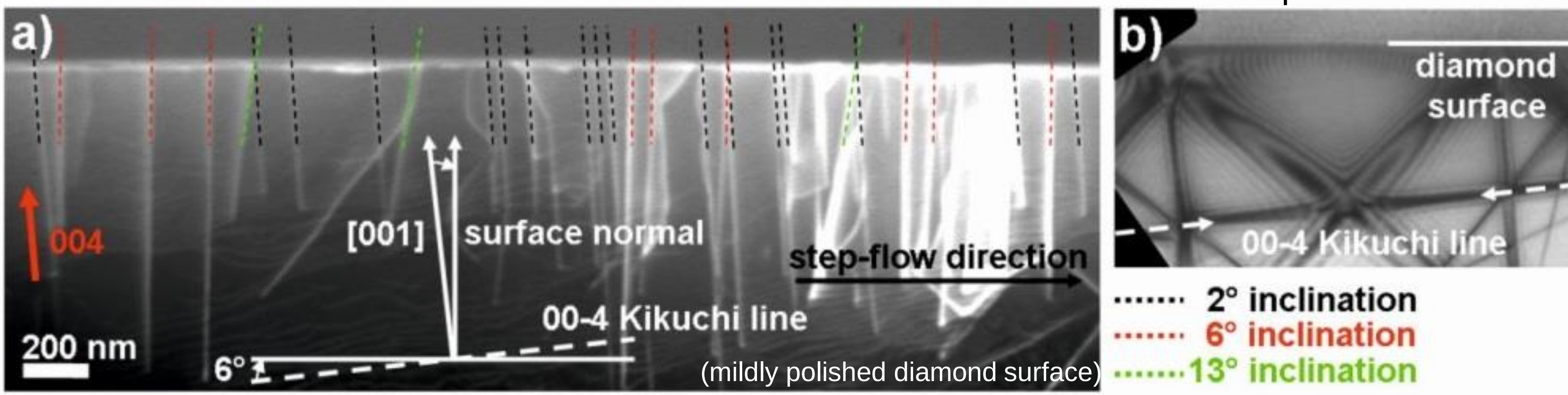
- Dislocations of all types are present.
- All tilt angles are identical; **no dependency** on accompanying **glide** or **climb** process.

Nitrogen-free growth

12µm growth (**no** N_2 in the gas phase → lack of terrace/riser structure, **smooth surface**).

WBDF image (cross-section)
004 reflection → all 45° dislocations visible.

LACBED pattern

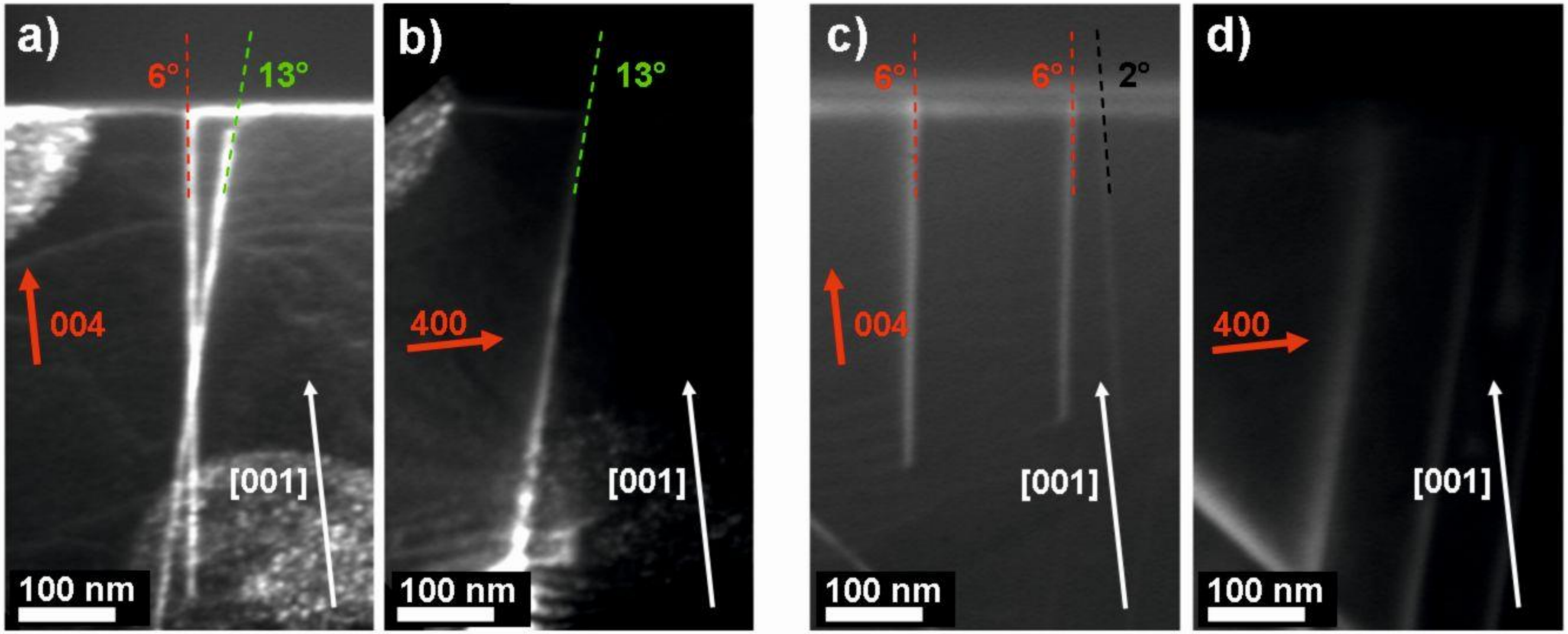


- General tilt towards off-axis direction again, but much smaller (2° - 13°).
- Three groups with different but discrete (!) tilting angles observed (2°, 6° and 13°).

QUESTION: DEPENDENCE ON BURGERS VECTOR?

Nitrogen-free growth (WBDF analysis)

WBDF analysis
004 (a,c) and 400 (b,d) reflection.



- All and only 45° dislocations with **b** = ½[±10±1] show high tilt angle of **13°**
  → effective **glide** process.
- Dislocations with **b** = ½[0±1±1] perform effective **climb**, lower tilt angles,
  → change towards compressive / tensile **stress** (sign of Burgers vector).
- Two different tilt angles for effective climb.
  → suggestion: different barriers for tensile or compressive stress?
  (final decision not possible with the present TEM technique)

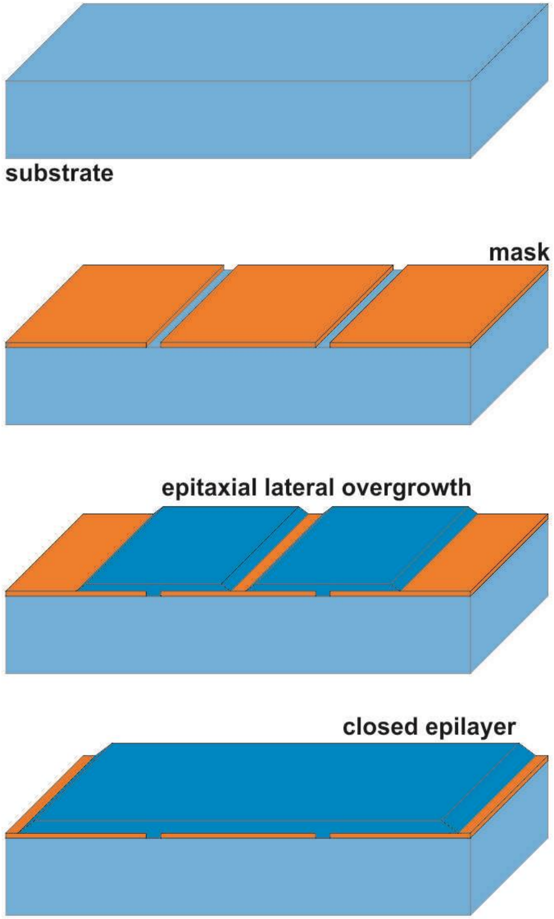
M. Fischer et al., Appl. Phys. Lett. **100**, 041906 (2012)

Summary (TEM)

- In **early growth stage** efficient reduction of dislocations by **annihilation** or **fusion.**
- Growth with **N_2** → step-bunching with terrace/riser growth areas; discrete bending by a high tilt angle (up to 20°) towards step-flow direction in riser areas; tilt angle shows **no dependence** on the dislocation type.
- Diamond grown **without N_2** → no terrace/riser formation; **discrete tilt** angles for 3 different groups of 45° dislocations.
- 45° dislocations with the **highest tilt** angle are characterized by an **effective glide** movement; all the **other** dislocations‘ tilting corresponds to an **effective climb**, connected with generation of **intrinsic stress** during growth.
- **N_2** in the process gas is suspected to level out the differences for dislocation tilting.

Epitaxial Lateral Overgrowth (ELO)

- Growth phenomenon known since 1965 (Tausch, Lapierre)
- Application for synthesis of **high-quality** epitaxial films



Method used in III-V semiconductor growth, i.e. by **Shuji Nakamura** for the production of blue light-emitting diodes with extended lifetime

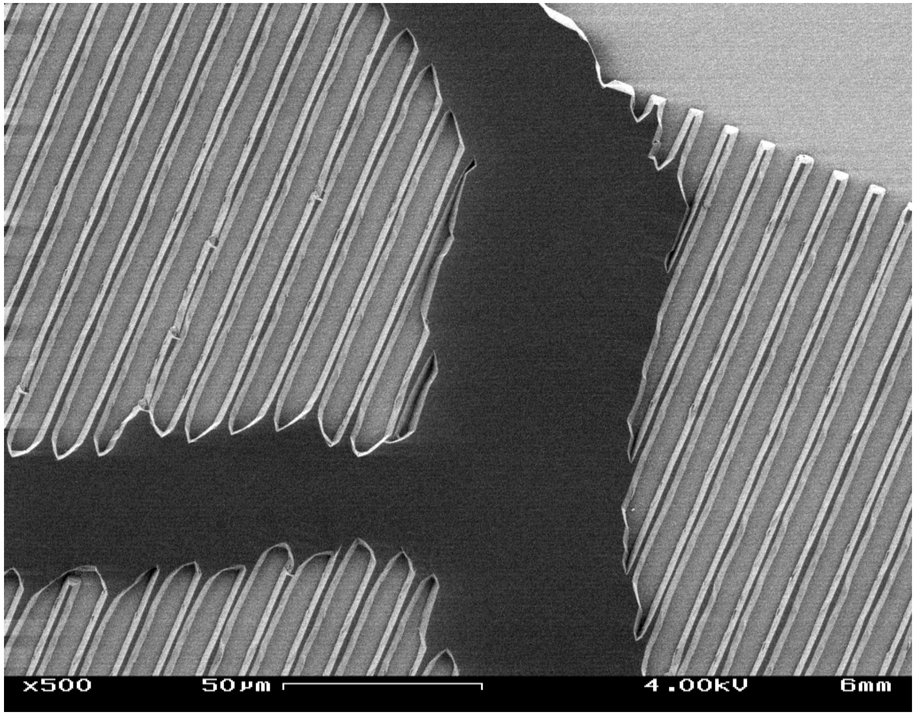
(dislocation density reduction of up to 4 orders of magnitude)

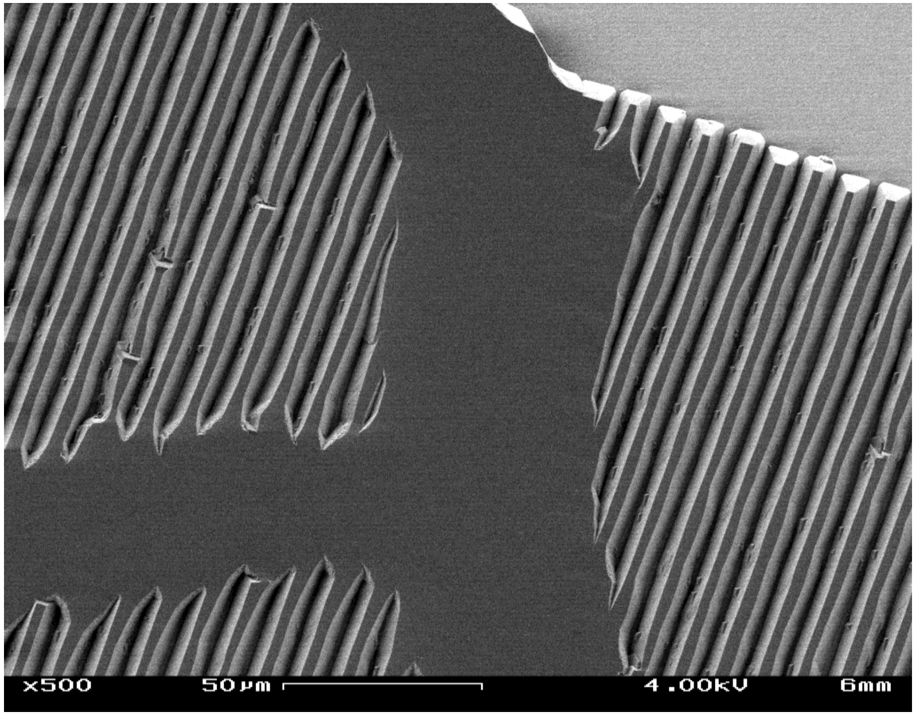
Shuji Nakamura
(physics nobel prize 2014)

photograph by: Ladislav Markuš

ELO – formation of a closed layer

Under construction
preliminary results





sequence of an ELO process on a heteroepitaxially grown diamond sample; etching reveals resulting defects

appearance of defects in a very early growth stage

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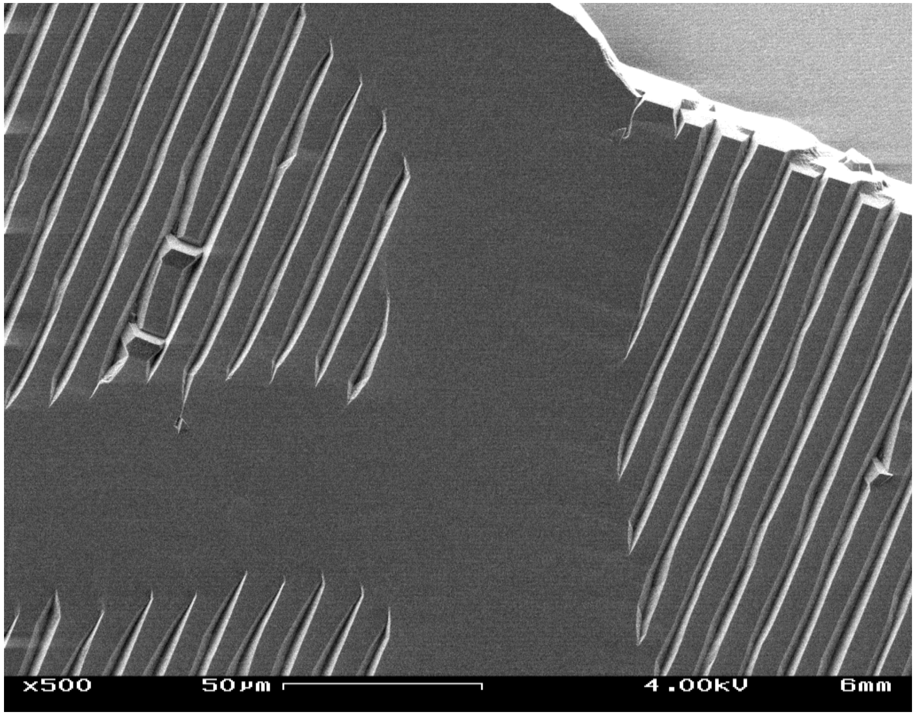

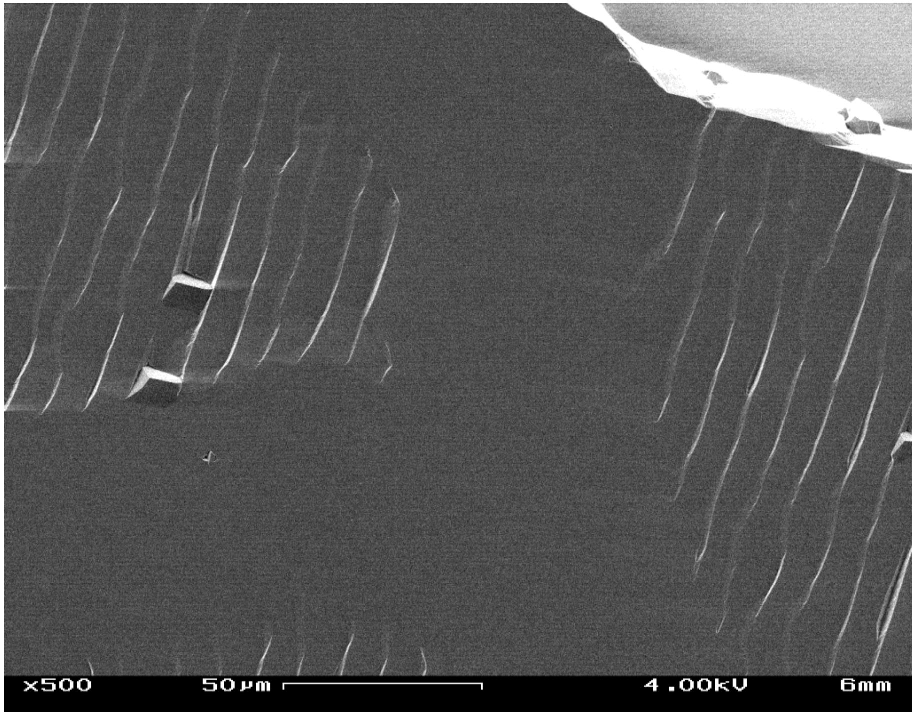

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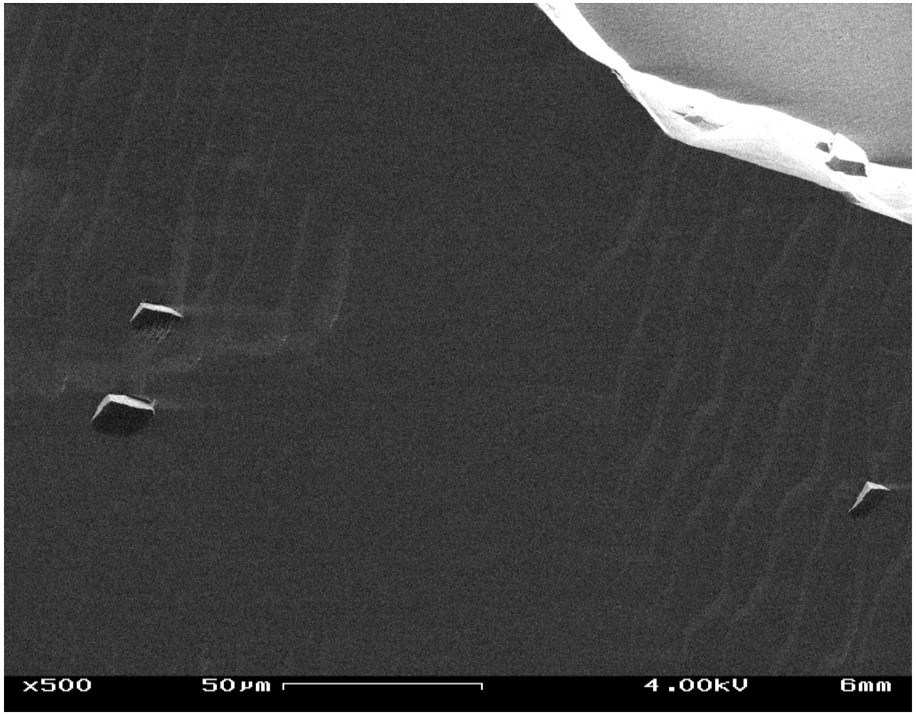
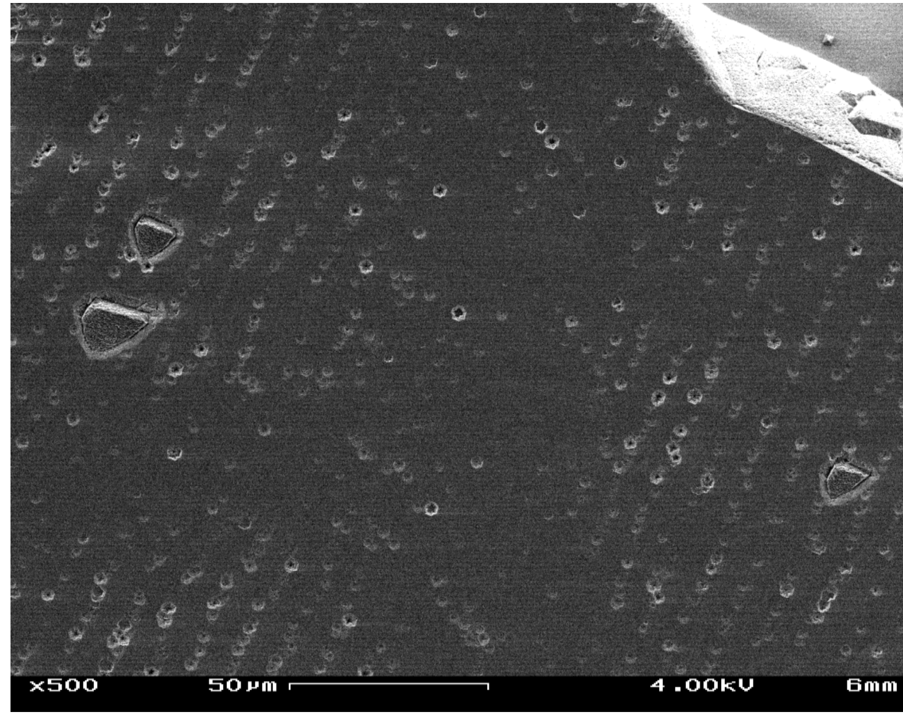




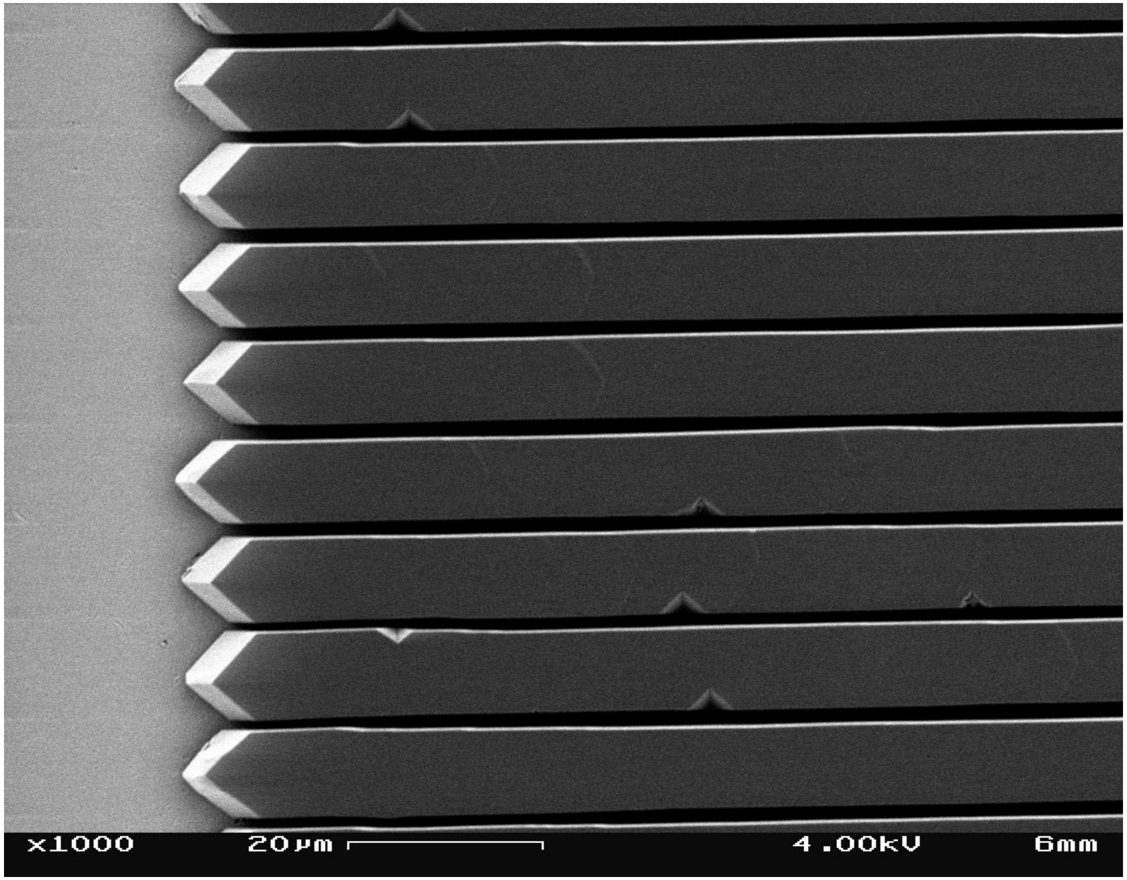
sequence of an ELO process on a heteroepitaxially grown diamond sample; etching reveals resulting defects

appearance of defects in a very early growth stage

Early ELO growth experiments

Under construction
preliminary results

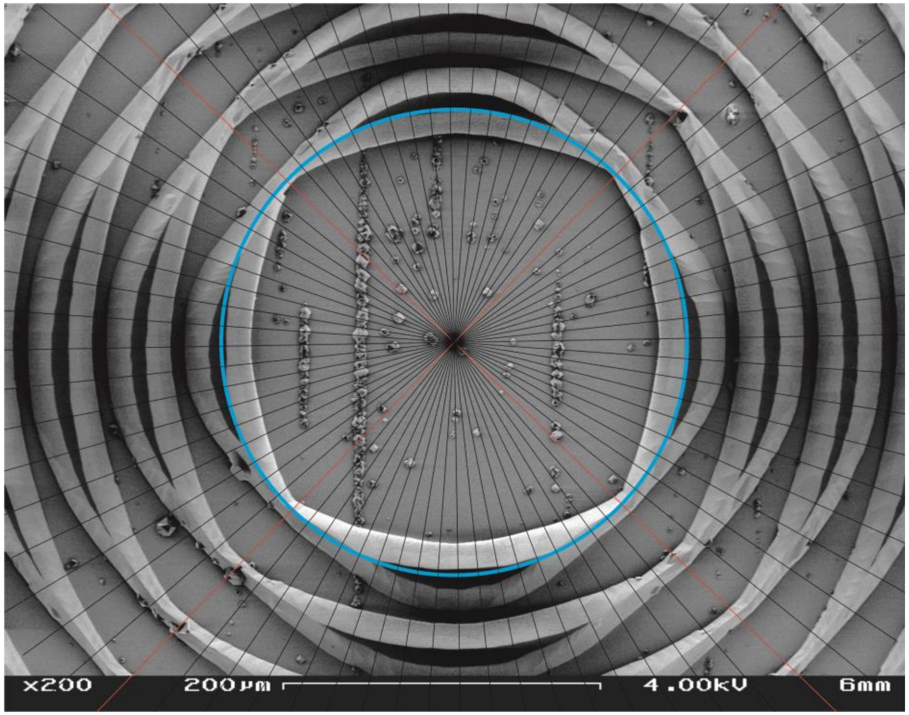
Testing of different growth parameters and stripe-directions:

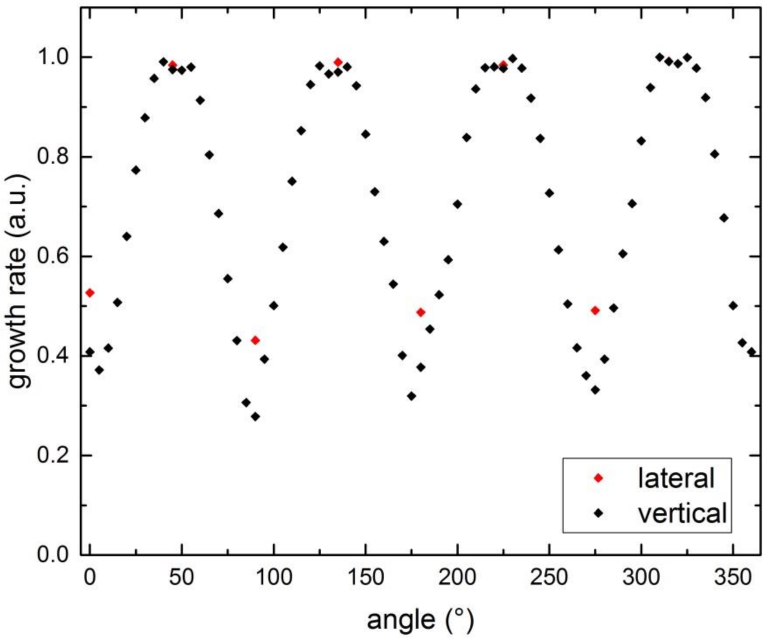


patterned growth experiment

Evaluation of stripe direction for optimal overgrowth

Under construction
preliminary results





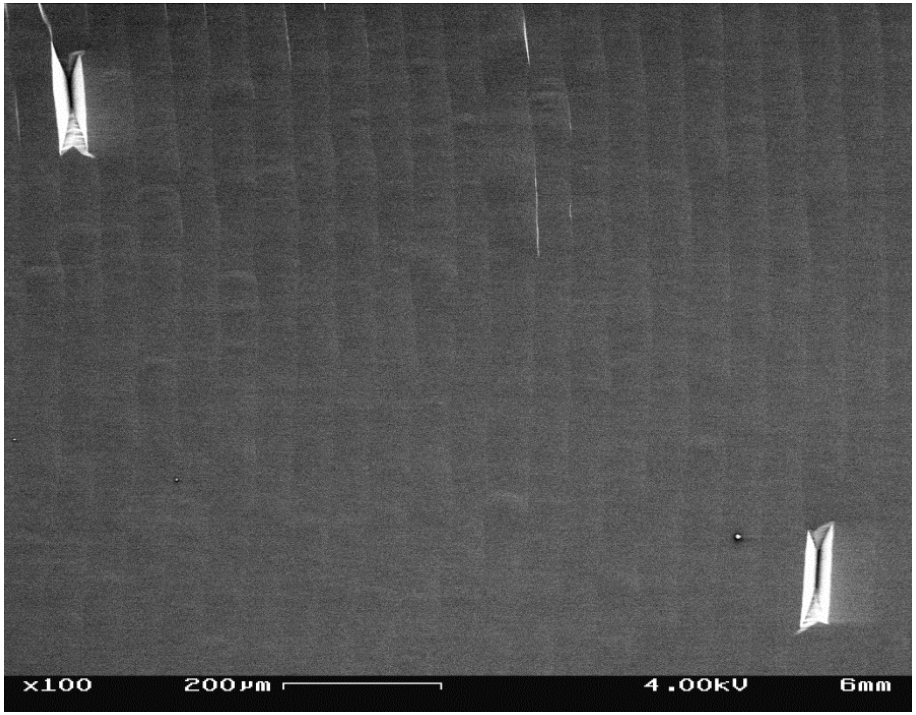
Measuring of stripe width and height shows distinct maxima and minima.

→ important for further experiments!

Scaling up –
50 µm stripe distance

Under construction
preliminary results

Is it possible to grow a closed layer with ~ 50 µm lateral growth?



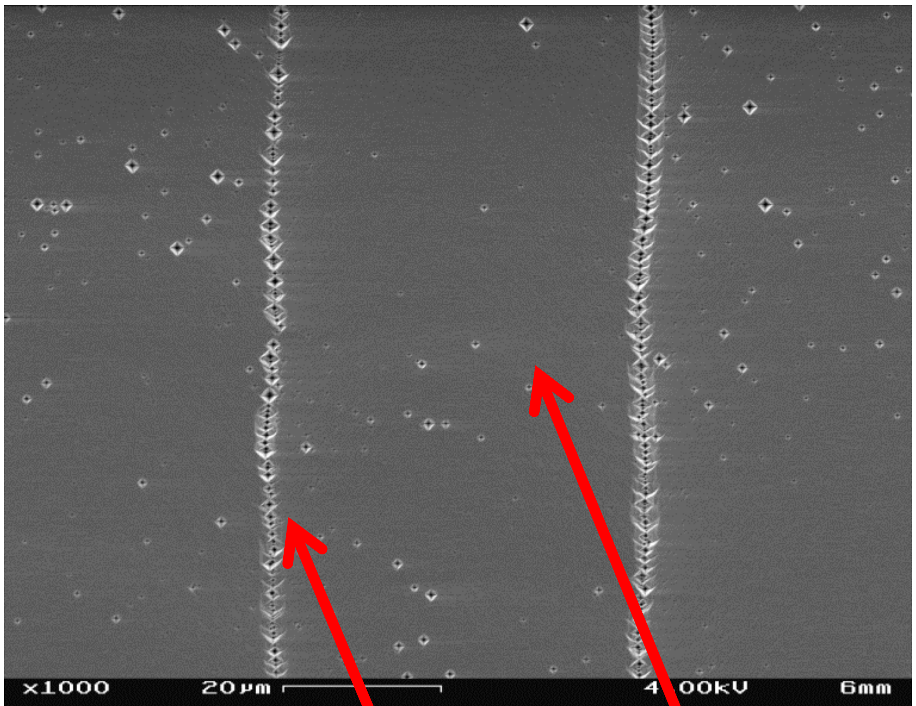
Yes!

Dislocation densities?

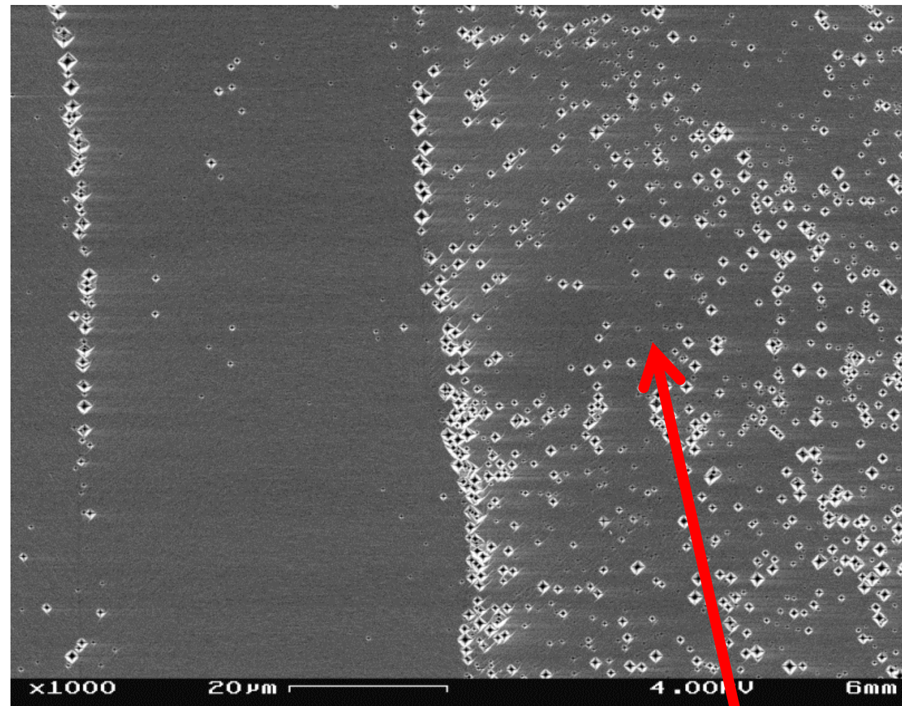
Under construction
preliminary results

Threading dislocations revealed by preferential etching:

structured area



border structured/non-structured area



lateral growth area

coalescence boundary

non-structured area

Summary (ELO)

What have we succeeded in?

- ✓ Demonstration of closed layer formation for stripe distance ~ 50 µm
- ✓ Non-epitaxial crystallites suppression
- ✓ Parasitic nucleation kept low
- ✓ Proof of dislocation manipulation

What‘s next?

- Estimation of dislocation reduction factor
- Growth of thick detector grade samples
- Electrical characterisation → detector behaviour?

How good can we get?

Thank you for your attention!

Acknowledgements:

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travel and lodging expenses sponsored by EMMI