

# *In situ* X-ray studies of the early stage of ZnO Atomic Layer Deposition on InGaAs

Evgeniy Skopin\*

L. Rapenne, J.L. Deschanvres, E. Blanquet, L. Pithan, G. Ciatto, D. D. Fong, M.-I. Richard, and H. Renevier

\*PostDoc, LMGP, Grenoble INP & CNRS, Grenoble, France



# Collaborators

**H. Renevier, J.-L. Deschanvres, L. Rapenne, C. Jimenez, M. Jouvert, S. Quessada, I. Gelard, H. Roussel & A. Claudel (NÉEL Institute, Grenoble)**

LMGP, UGA & CNRS, Grenoble, France



**E. Blanquet, A. Crisci, T. Encinas, S. Coindeau**

SIMAP, UGA & CNRS, Grenoble, France



**M.-I. Richard**

Aix-Marseille Univ., IM2NP-CNRS, Marseille, France



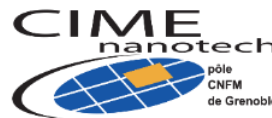
**D. D. Fong**

Argonne National Laboratory, Argonne, USA



**V. Bolcato**

CIME, UGA & CNRS, Grenoble, France



**C. Gomez**

PTA, Grenoble, France



**G. Ciatto, N. Aubert, P. Fontaine**

Synchrotron SOLEIL, Saint Aubin, France



ID3 (ESRF) staff : **F. Carla, H. Isern, T. Dufresne, L. Pithan**



**X. Mescot**

IMEP-LAHC, Grenoble, France



**P. Rodriguez, M. Bertrand**

Leti, technology research institute, Grenoble (France)



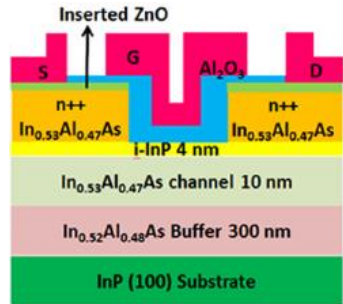
**Funders:**

**MINOS Labex**



# Talk outline

Motivation:  
ZnO for MIS  
junctions, IPL

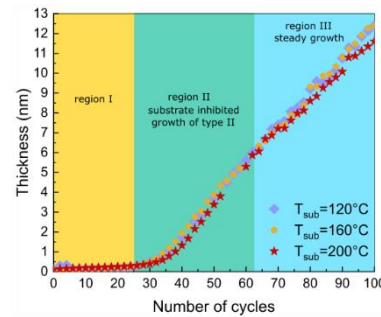


MOON reactor  
for *in situ* ZnO  
ALD analysis

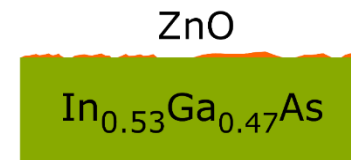


ZnO ALD early stages  
on  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$

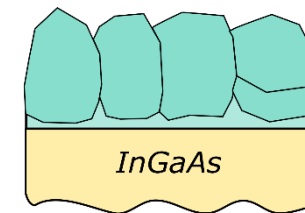
Substrate  
temperature  
effect



Transient region



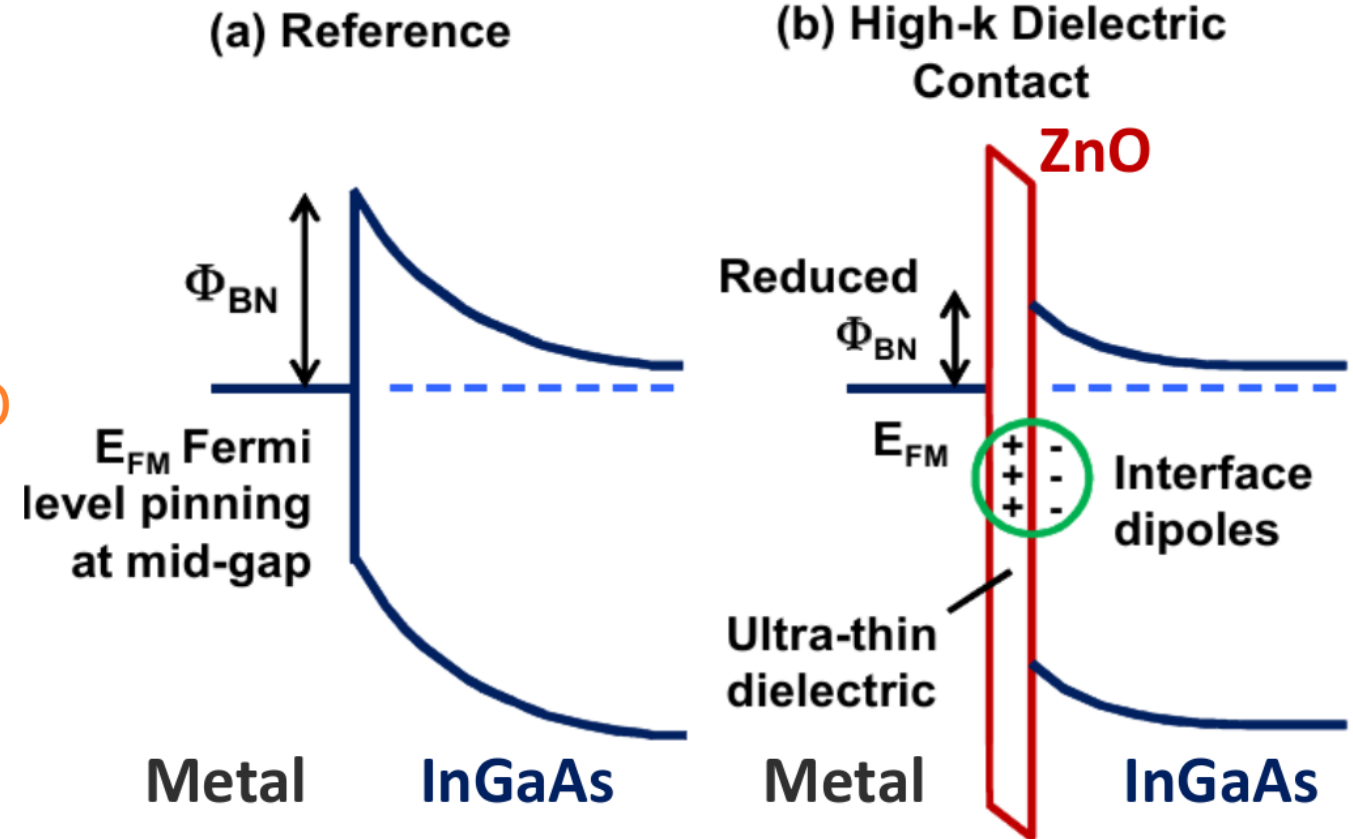
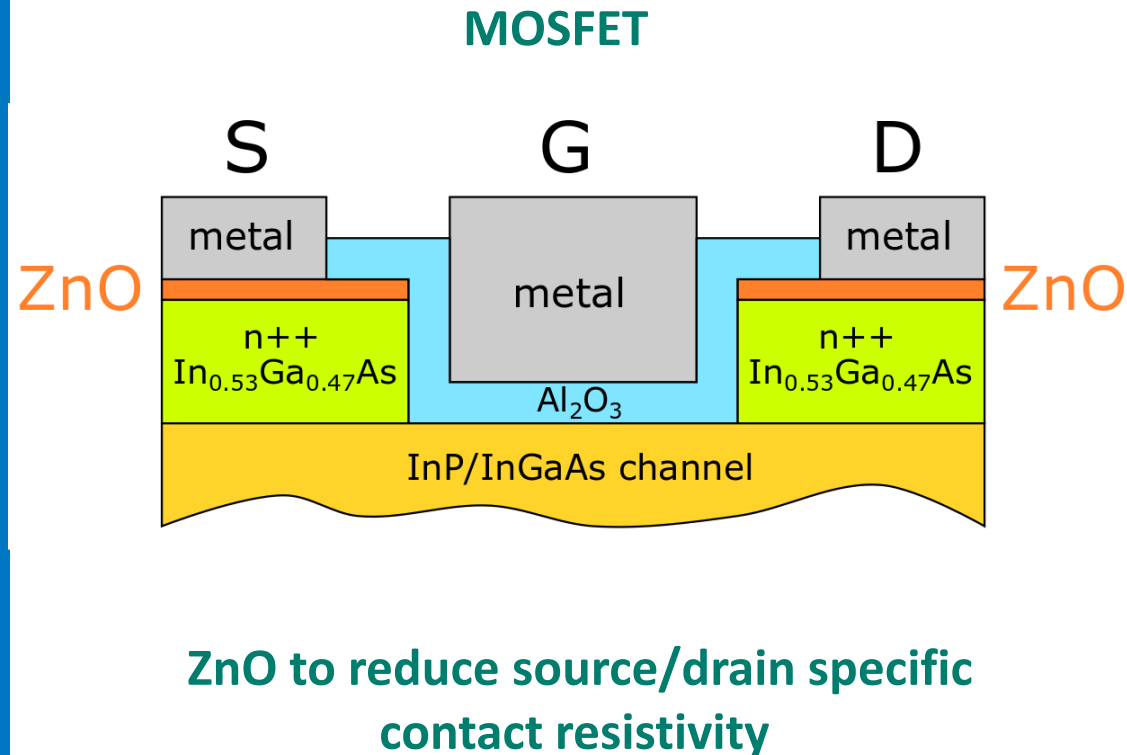
ZnO cristallisation



Conclusion



# Source/Drain specific contact resistivity

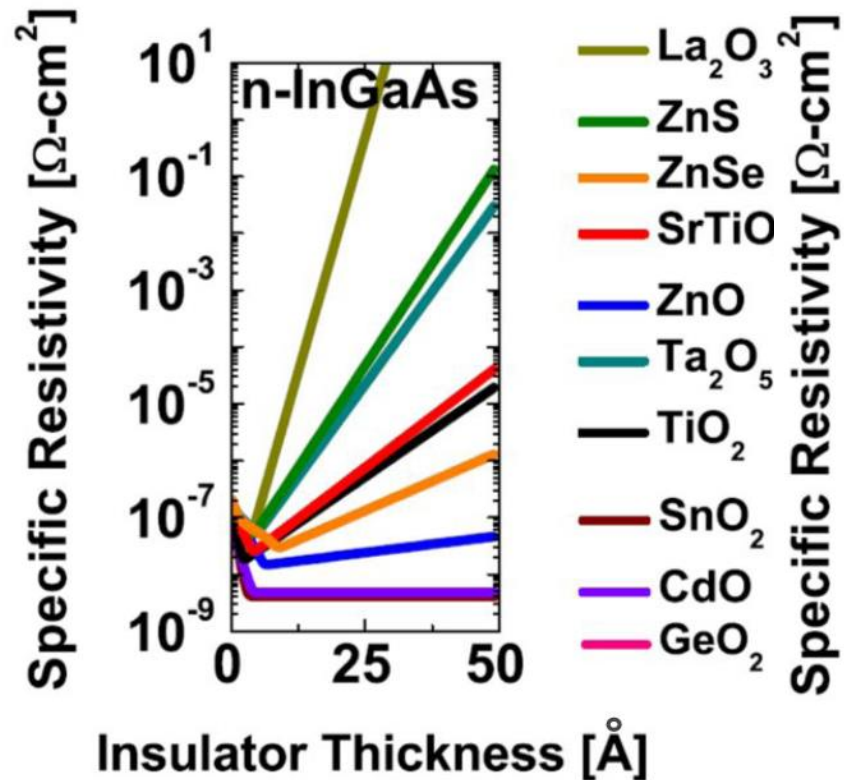


*Liao et al., Appl. Phys. Lett. 103, 072102 (2013)*

*Ang et al., IEDM (2012) 18.6.1*

# Optimal ZnO thickness

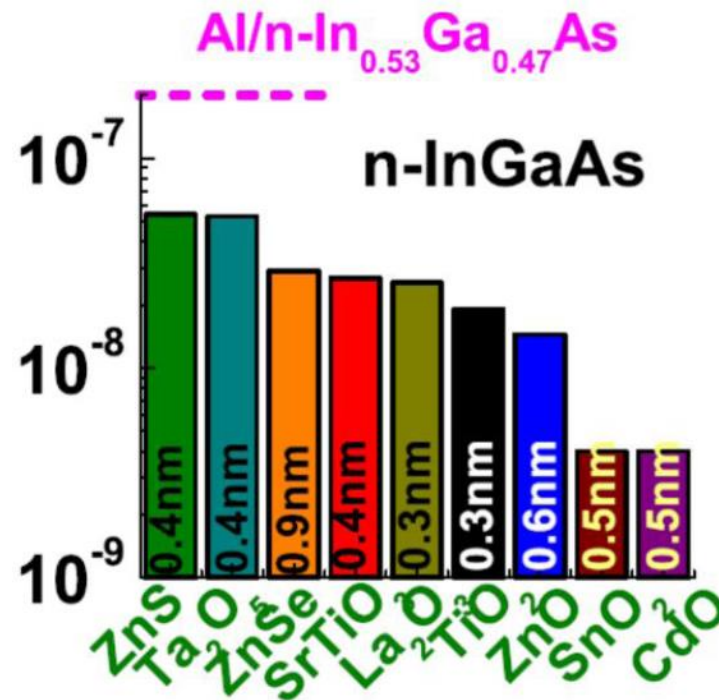
## Al/Insulator/n-InGaAs



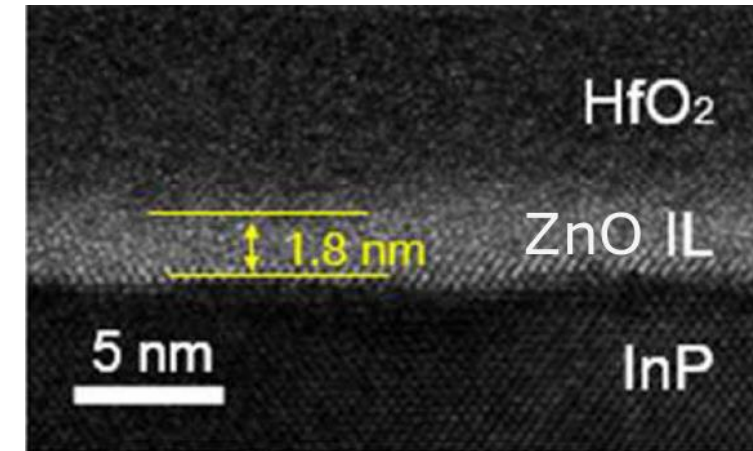
calculations

A. Agrawal et al. APL 101 042108 (2012)

## Optimal thickness



## ZnO interfacial passivation layer (IPL)

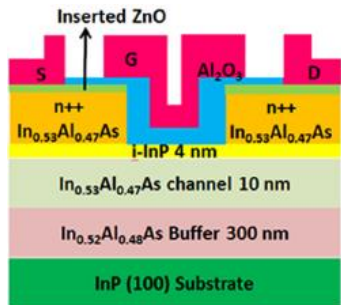


- ✓ suppresses film crystallization
- ✓ reduces interface state density

S.H. Kim et al. ACS App. Mat. & Int. 8.32 (2016)

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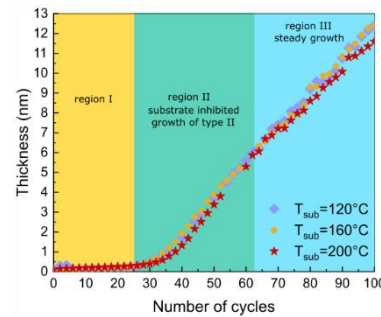


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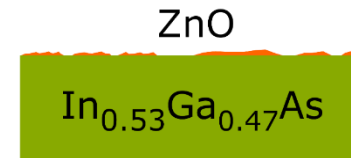


ZnO ALD early stages  
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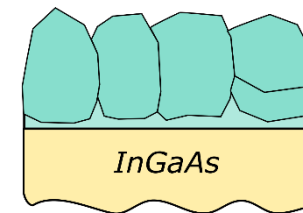
Substrate  
temperature  
effect



Transient region



ZnO cristallisation



Conclusion



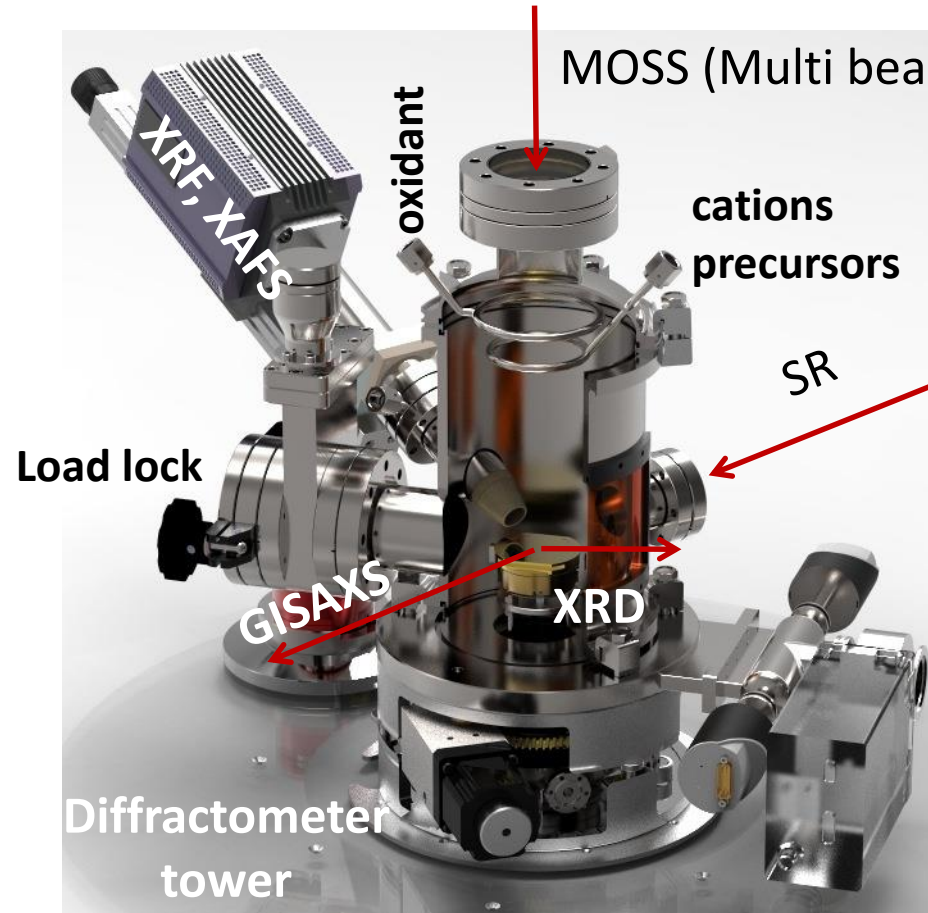


# *In situ* growth study of ZnO ALD

## Thermal ALD

### Allows:

- ✓ pressures from atmosphere to vacuum
- ✓ Thermal ALD: temperatures from room to 800°C
- ✓ Counter-rotating flange



## Synchrotron probes

- ✓ Fluorescence, spectroscopy (XAFS)
- ✓ Reflectivity vs angle, energy, thickness
- ✓ Grazing Incidence XRD
- ✓ Surface diffraction
- ✓ Anomalous diffraction, DAFS spectroscopy

## In house probes

- ✓ Substrate curvature (MOSS)
- ✓ PL (Photoluminescence)
- ✓ Ellipsometry (ready to start)
- ✓ Residual Gaz Analyzer (near future)

*Designed and built under the guidance of D. De Barros*

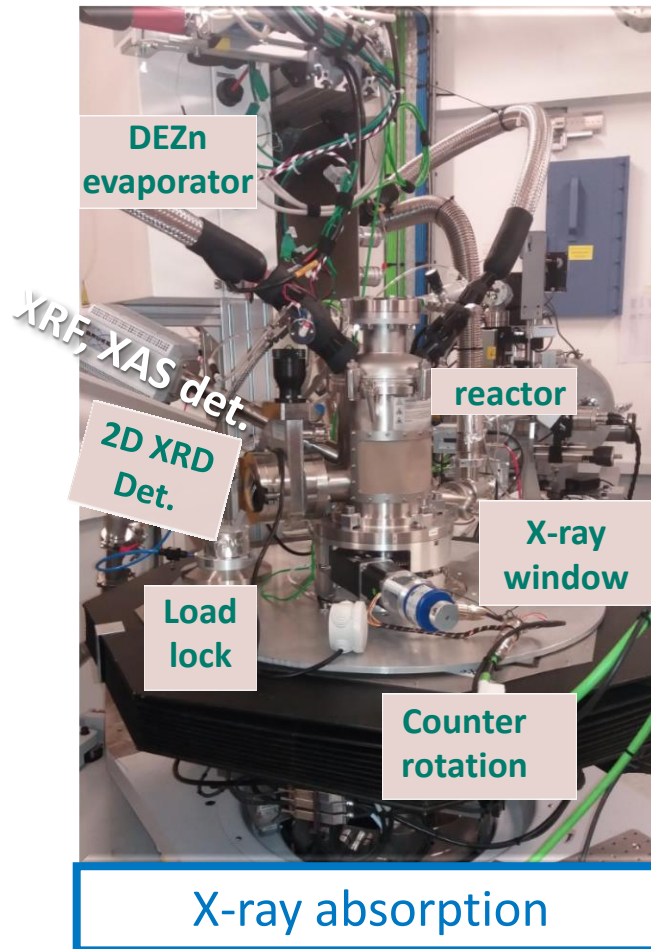


*Chem. Mat.* 28 592 (2016)

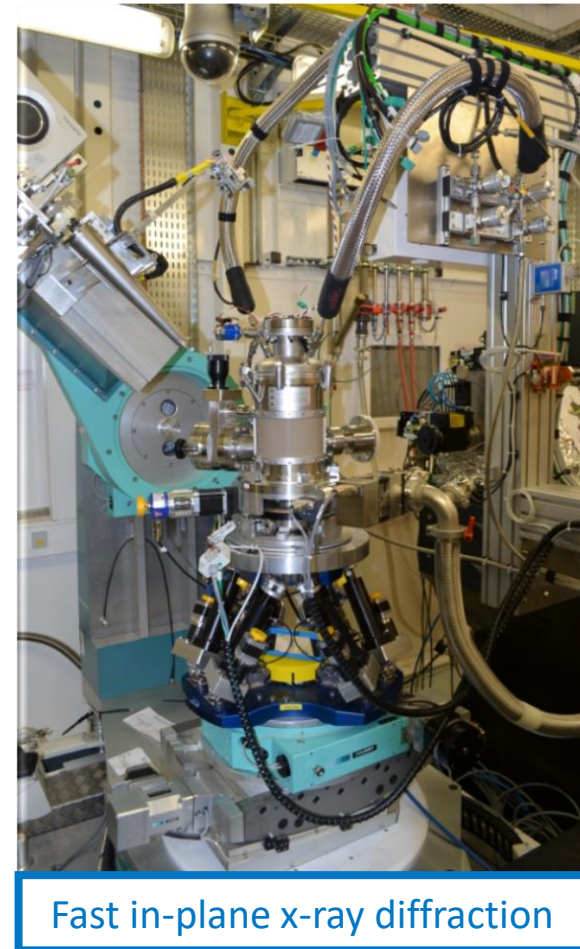
*Cryst. Growth Des.* 16 5339 (2016)

# *In situ* growth study of ZnO ALD

ALD setup @ SOLEIL  
SIRIUS beamline



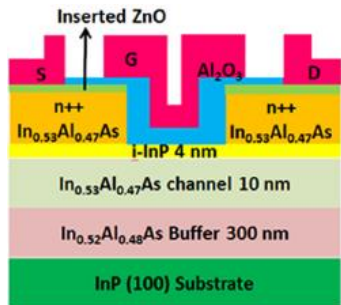
ALD setup @ ESRF  
ID3 beamline





# Talk outline

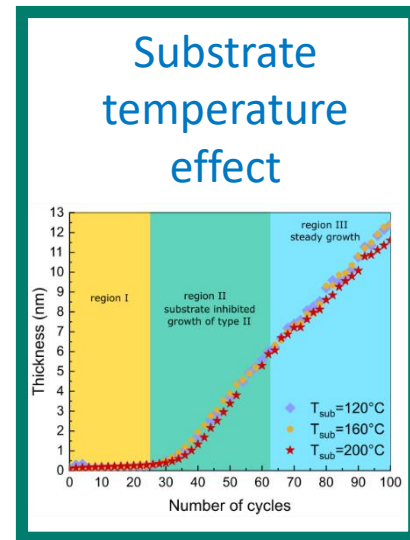
Motivation:  
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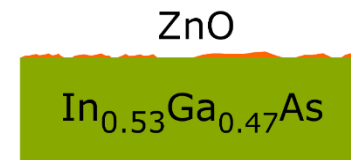
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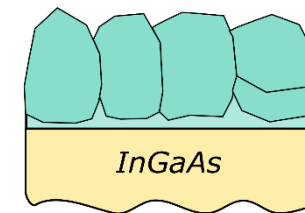
ZnO ALD early stages  
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Transient region



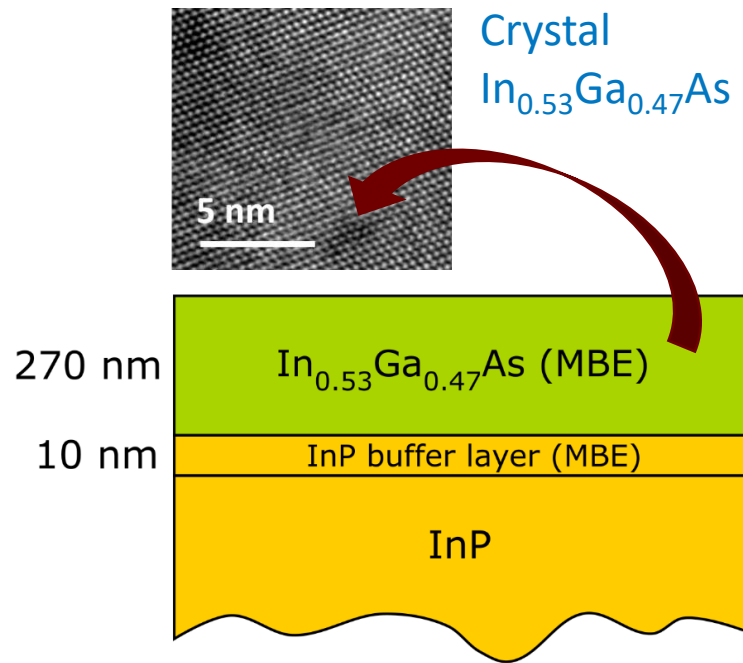
ZnO cristallisation



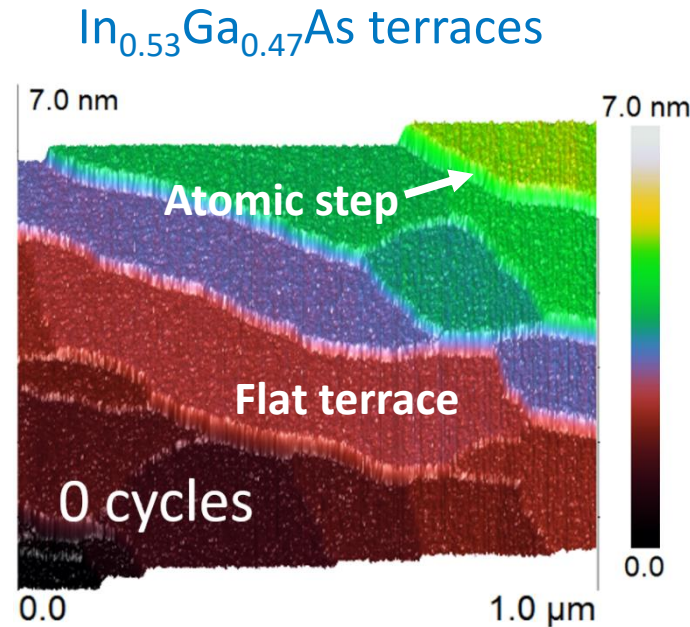
Conclusion



# ZnO ALD on $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ substrate



n-InGaAs & p-InGaAs provider : III-V Lab™



3D AFM image

**ZnO ALD**  
**Precursors:**  
**DEZn ( $\text{Zn}(\text{C}_2\text{H}_5)_2$ )**  
 **$\text{H}_2\text{O}$**

**$\text{DEZn}/\text{N}_2/\text{H}_2\text{O}/\text{N}_2$**

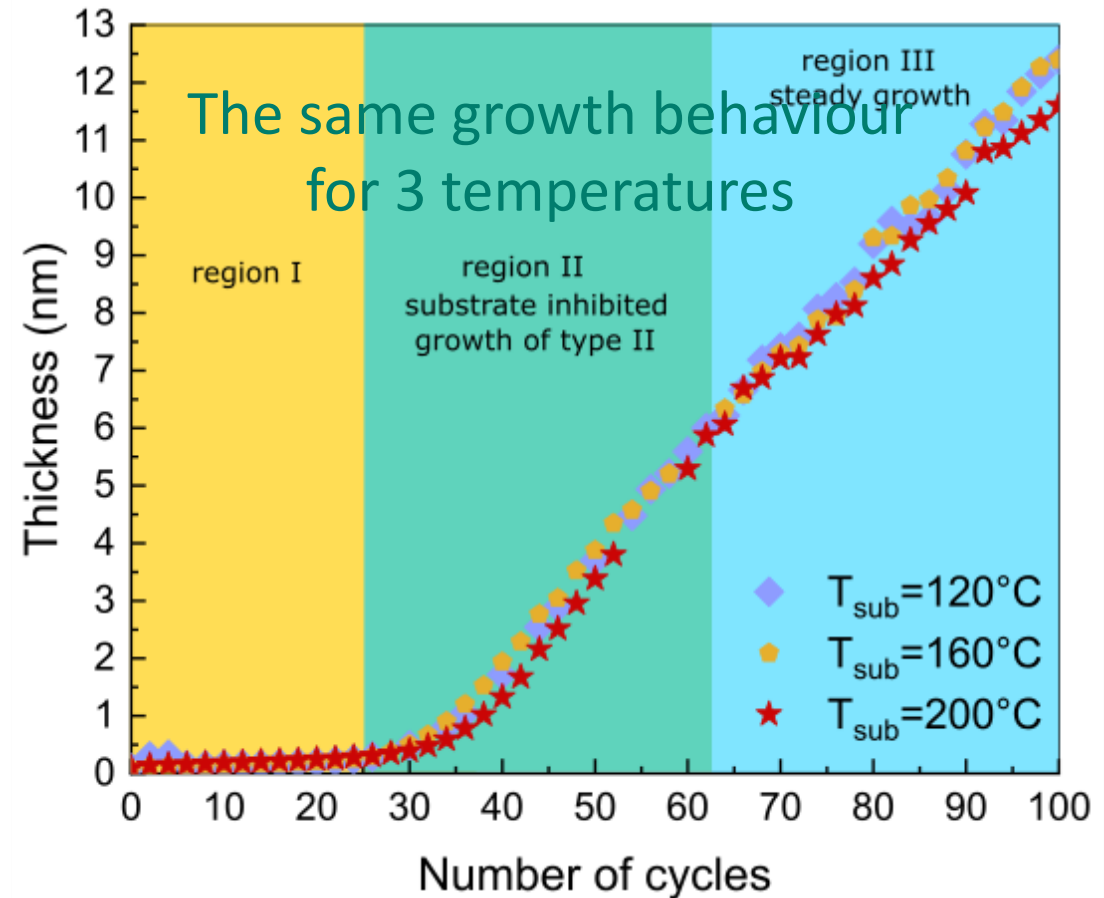
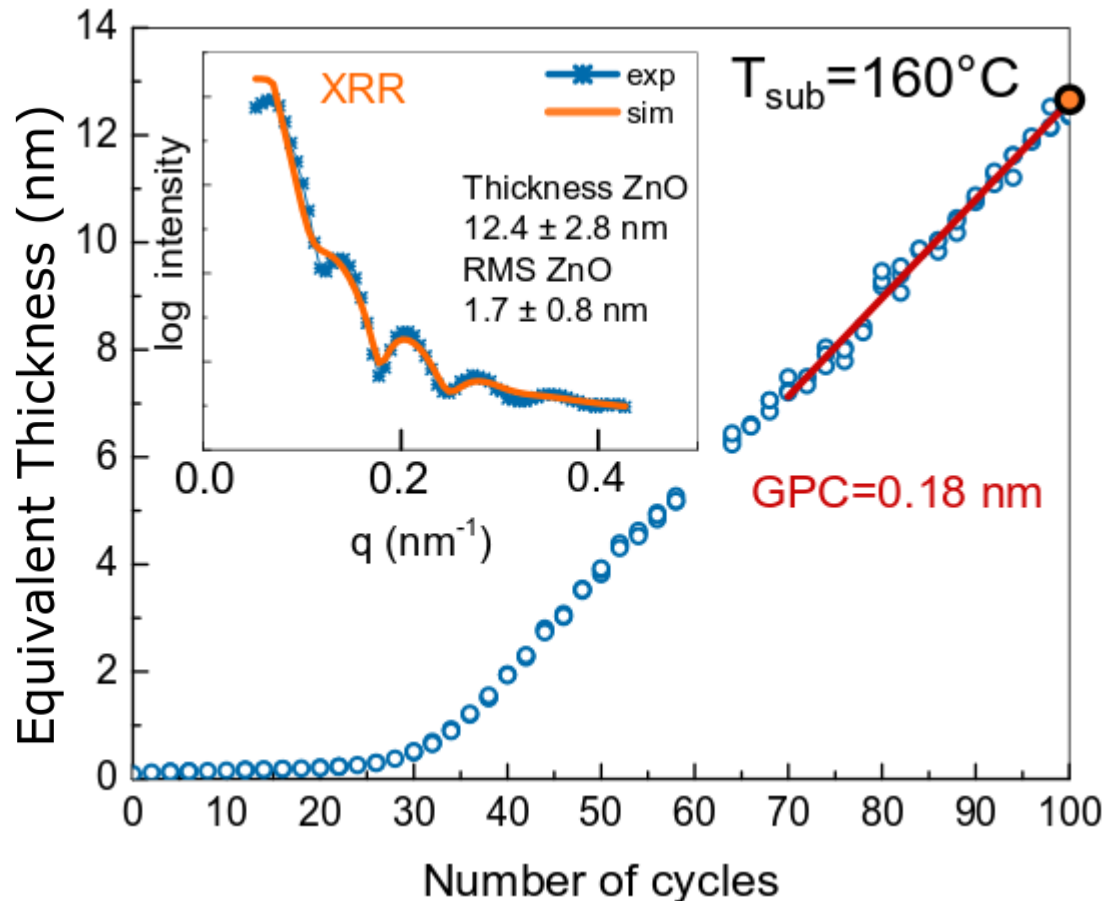
**Etched 5 min in a 4M HCl solution**

*APL 93, 194103 (2008)*

# Substrate temperature effect

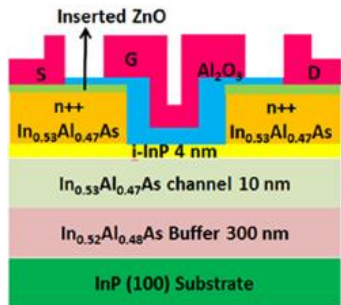
- Substrate temperature = 120°C, 160°C, 200°C
- Number of cycles = 100

- DEZn/ H<sub>2</sub>O/ N<sub>2</sub> flow = 5sccm/2.6sccm/1000sccm
- DEZn/ H<sub>2</sub>O/ N<sub>2</sub> inj. or purge time = 5s/40s/45s



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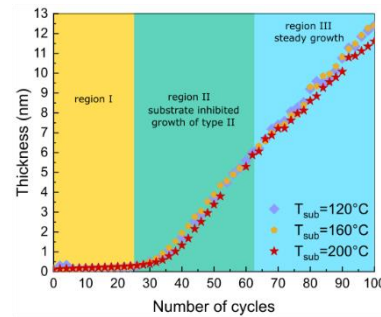


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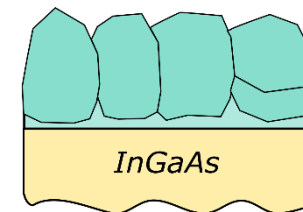
Substrate  
temperature  
effect



Transient region

ZnO  
 $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$

ZnO cristallisation



Conclusion



# ZnO transient growth on InGaAs

Nanoscale

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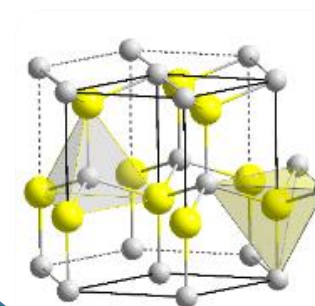
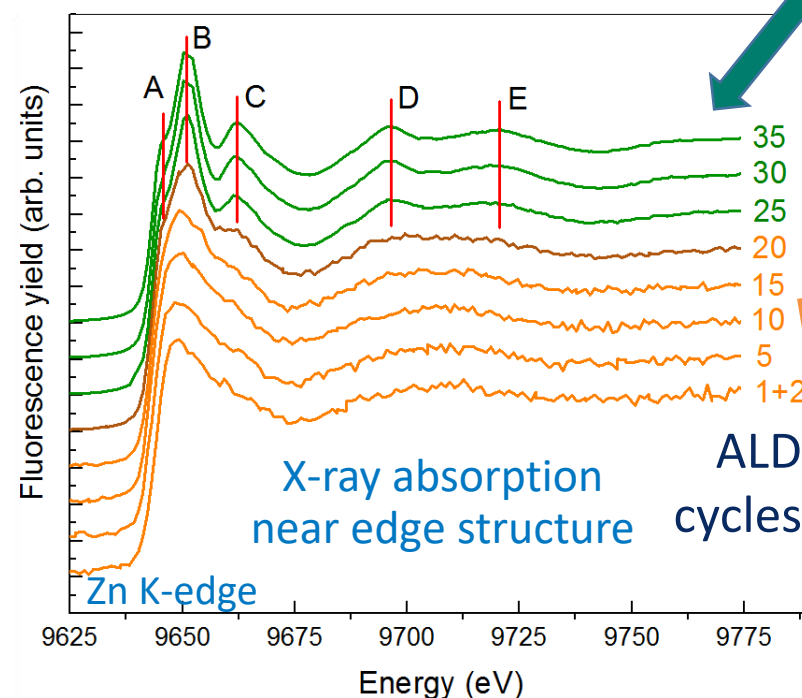
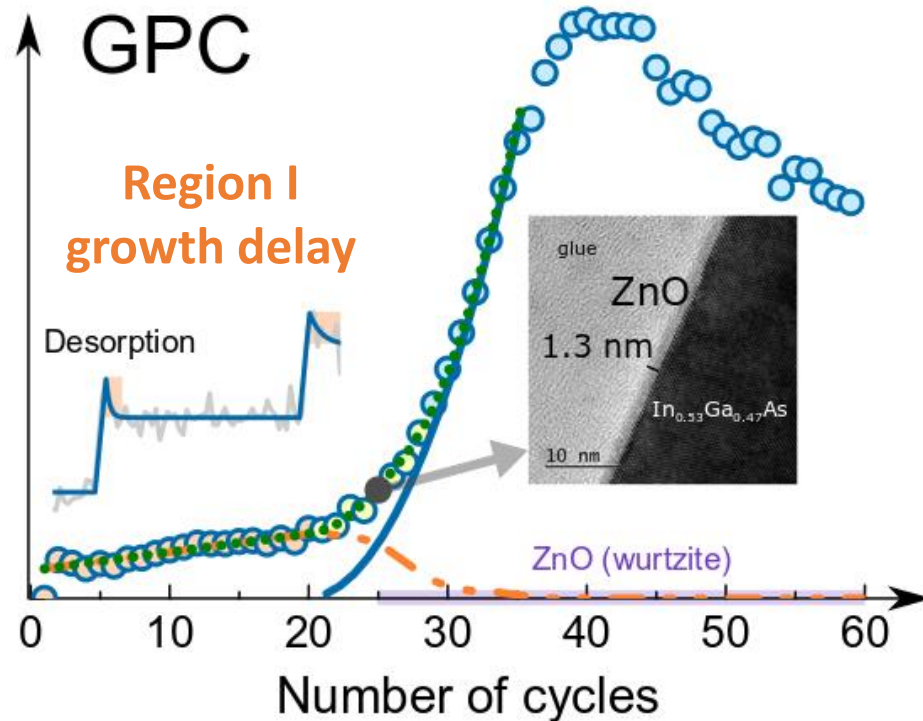


Cite this: *Nanoscale*, 2018, **10**, 11585

## The initial stages of ZnO atomic layer deposition on atomically flat $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ substrates†

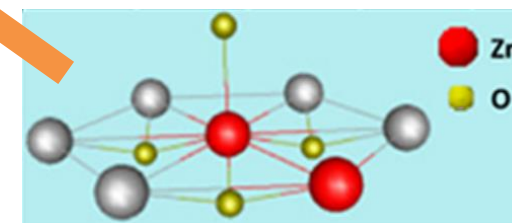
Evgeniy V. Skopin,<sup>a</sup> Laetitia Rapenne,<sup>a</sup> Hervé Roussel,<sup>a</sup> Jean-Luc Deschanvres,<sup>id a</sup> Elisabeth Blanquet,<sup>b</sup> Gianluca Ciatto,<sup>c</sup> Dillon D. Fong,<sup>d</sup> Marie-Ingrid Richard<sup>id e,f</sup> and Hubert Renevier<sup>id \*a</sup>

**Region II**  
**S-shape growth**



Wurtzite structure

*Cryst. Growth Des.* 2016, **16**, 5339–5348

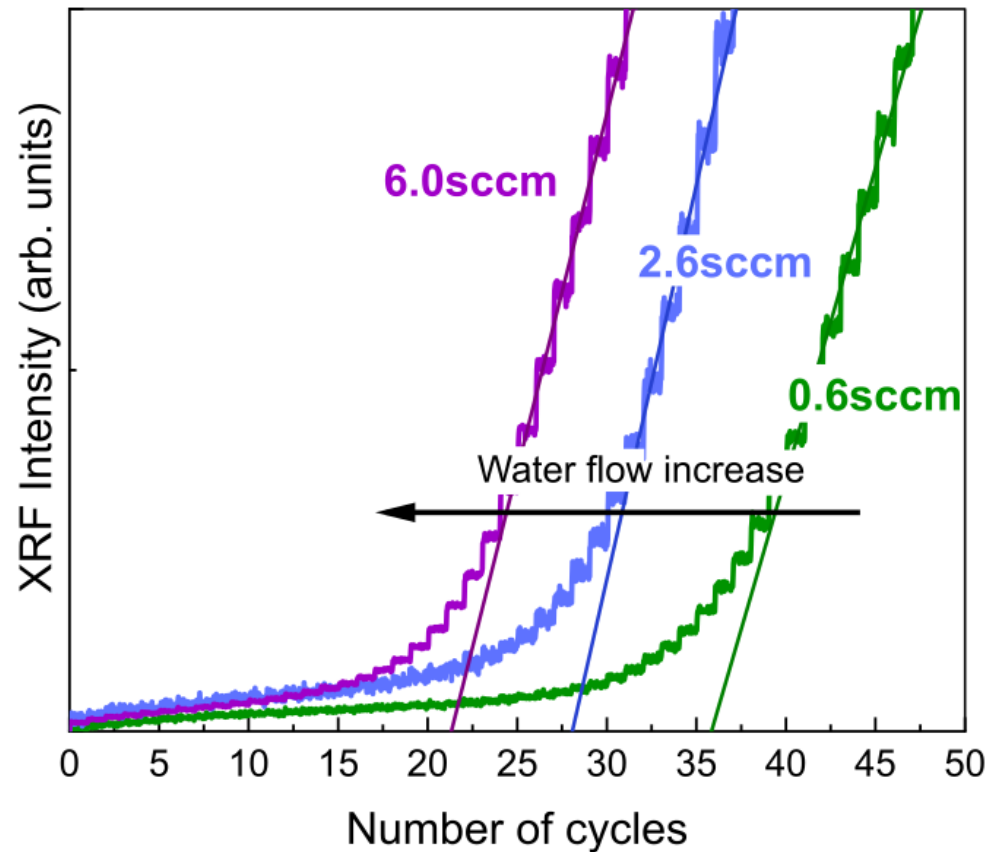


Disordered Zn local environment/ small islands

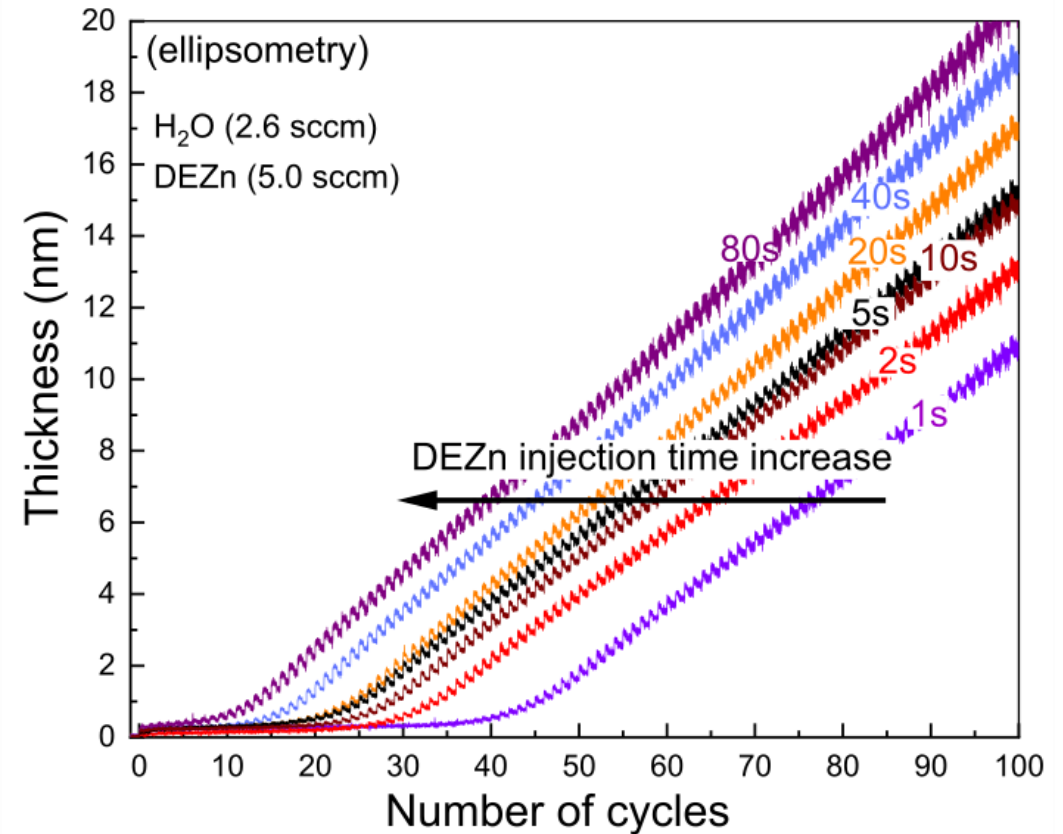


# Precursor flow/inj.time effect on growth delay (regime I)

## X-Ray Fluorescence



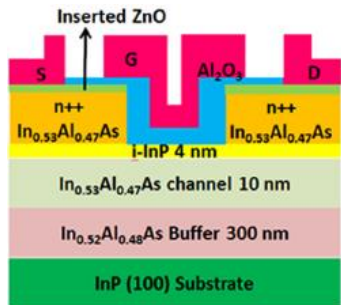
## Ellipsometry *in situ*



DEZn/ H<sub>2</sub>O/ N<sub>2</sub> flow = 5sccm/2.6sccm/1000sccm; DEZn/ H<sub>2</sub>O/ N<sub>2</sub> inj. or purge time = 5s/40s/45s

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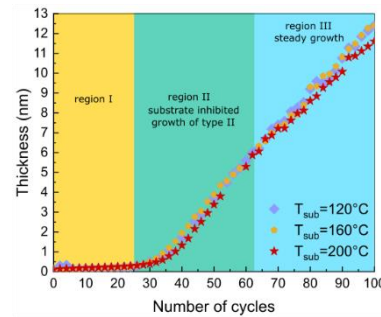


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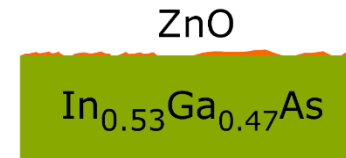


ZnO ALD early stages  
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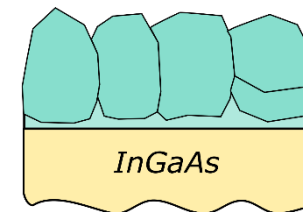
Substrate  
temperature  
effect



Transient region



ZnO cristallisation



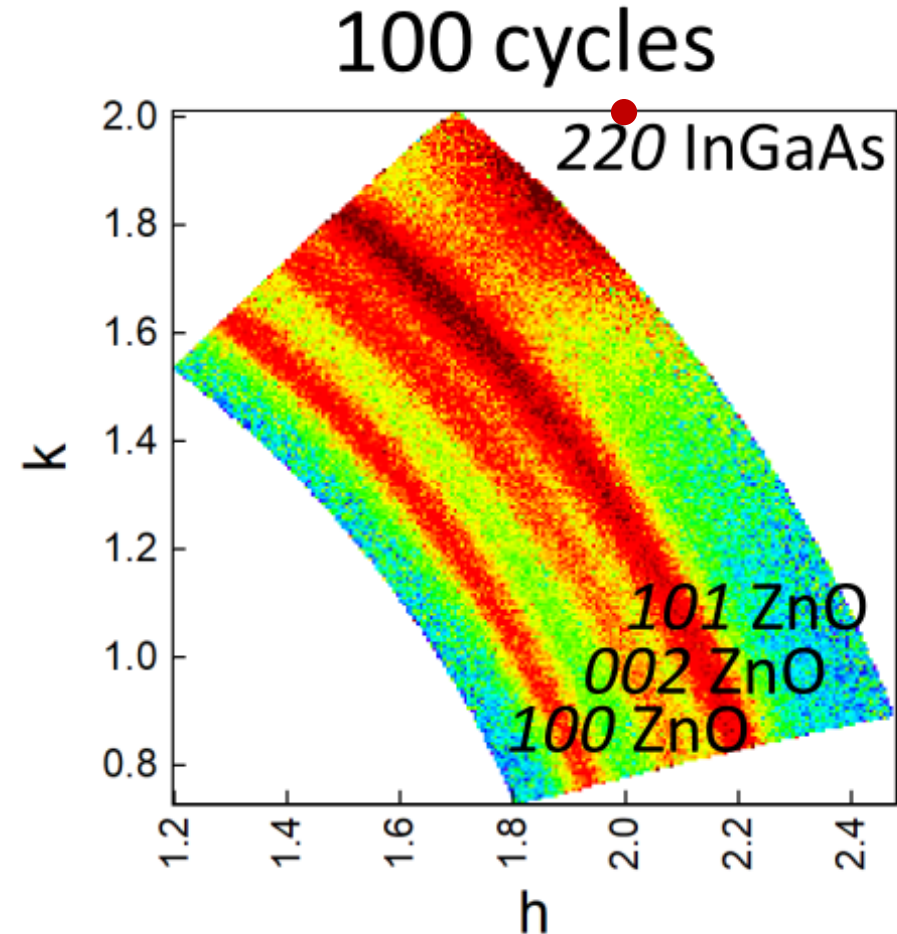
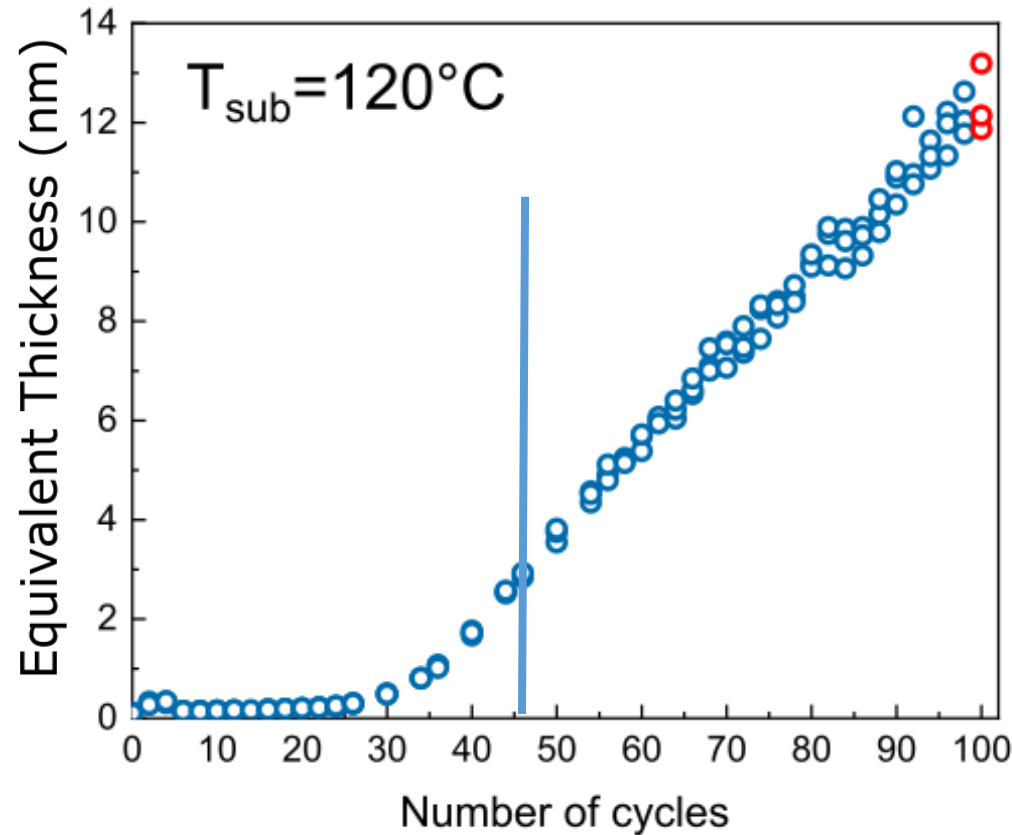
Conclusion



# In-plane RSM vs ZnO film thickness



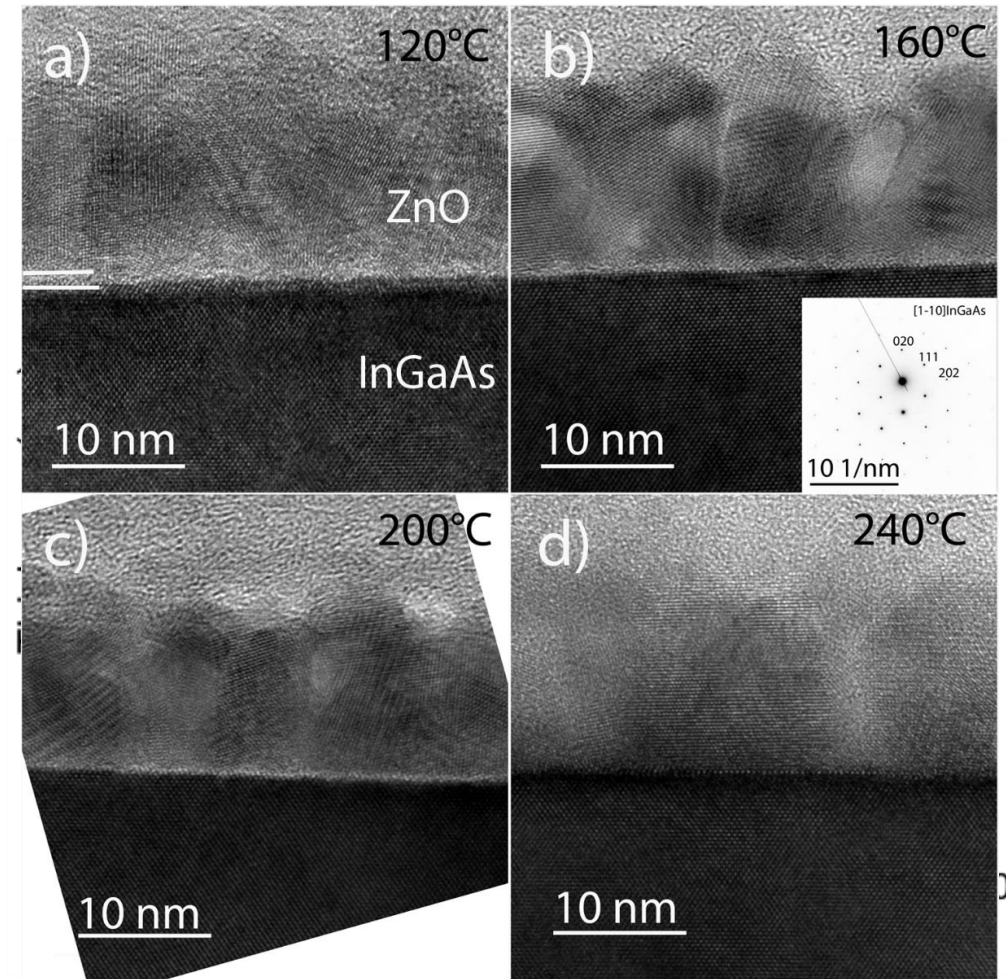
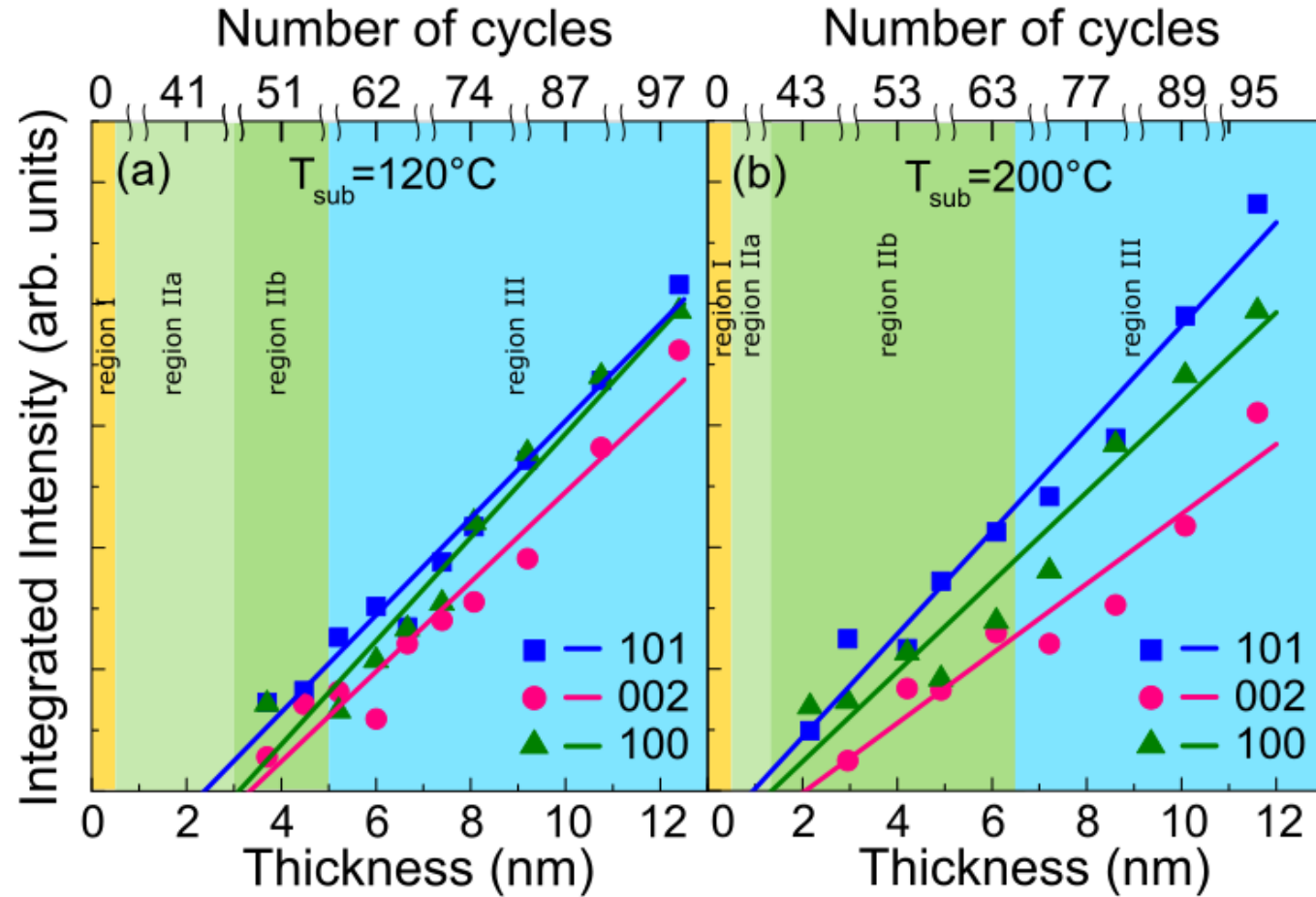
## Fast in-plane diffraction



RSM : reciprocal space map

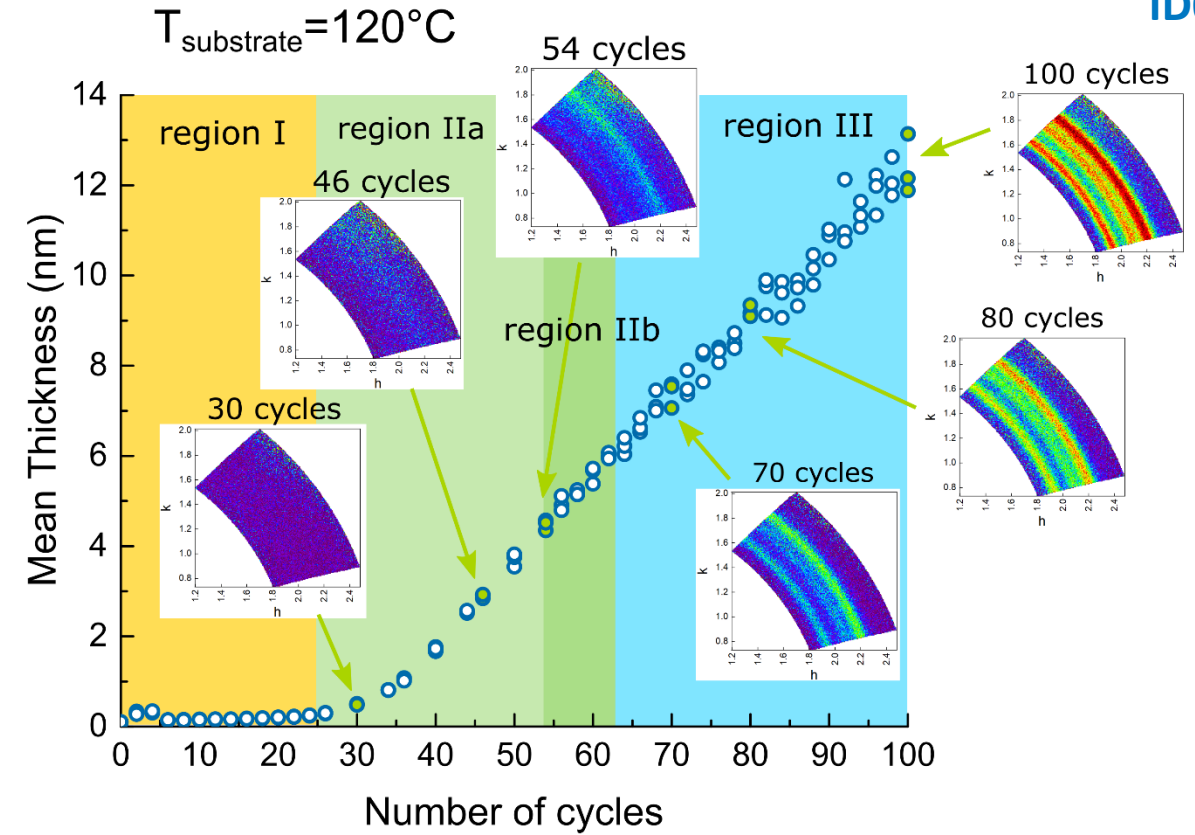
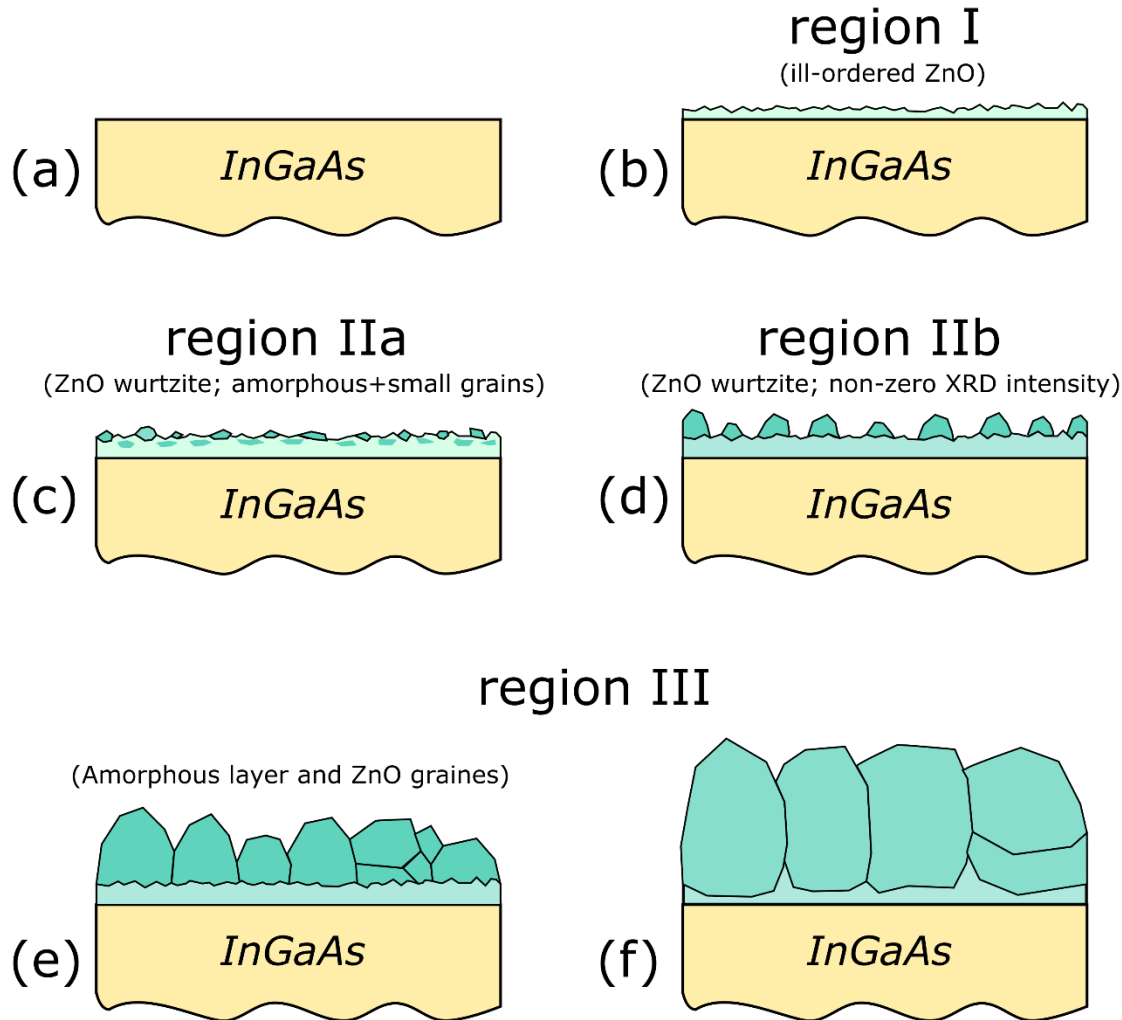
No in-plane texture

# ZnO crystallization during growth





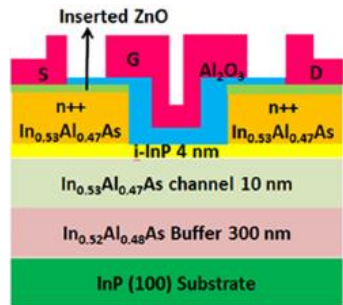
# ZnO cristallisation on InGaAs





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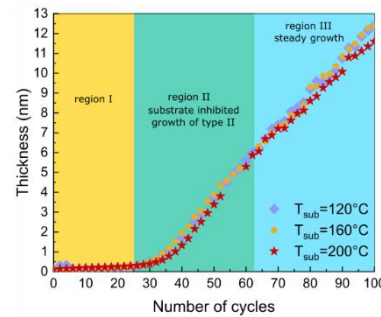


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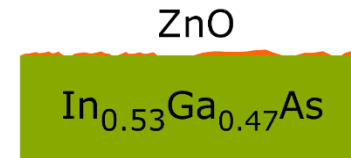


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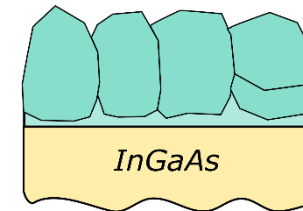
Substrate  
temperature  
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Transient region



ZnO cristallisation

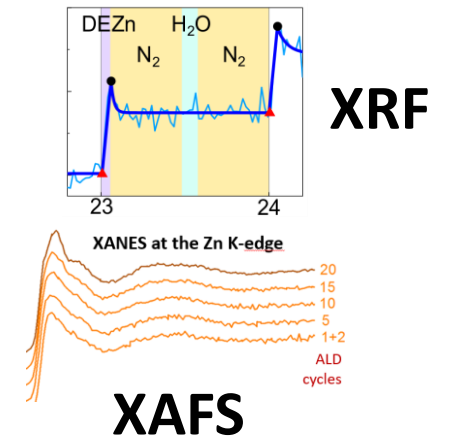
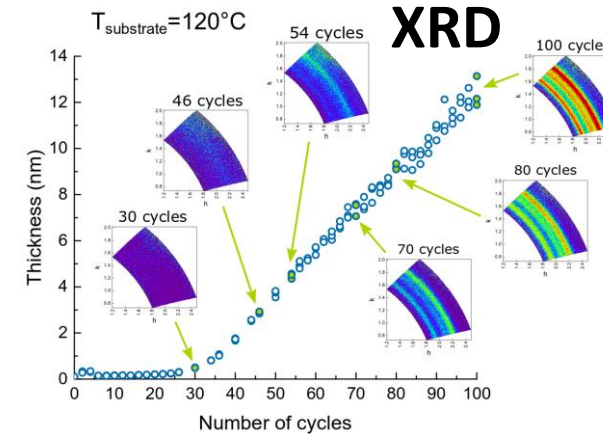
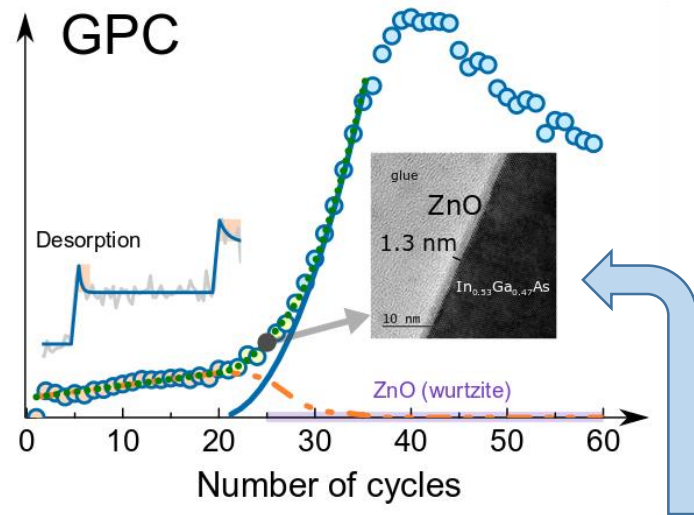


Conclusion



# Conclusion

- ✓ GPC of ZnO ALD on InGaAs  $\sim 0.2\text{nm}\cdot\text{cy}^{-1}$  in steady growth regime & ALD temperature window
- ✓ Evidenced a transient growth regime (prior to steady growth)
- ✓ Further demonstrated the interest of *in situ* synchrotron experiments for studying the incipient growth of ALD



- ✓ 1-2 nm thick, continuous, ill-ordered ZnO film

E. V. Skopin et al. *Nanoscale* 10 11585 (2018)

E. V. Skopin et al., in preparation (2019)

EuroCVD 22 Baltic ALD 16 | 2019  
24-28.06.2019 | Luxembourg



Thank you for your attention!