Faculty of Electrical and Computer Engineering - Institute of Semiconductors and Microsystems

NanoZEIT seminar @ SEMICON 2011

In-situ Monitoring of Atomic Layer Deposition Processes

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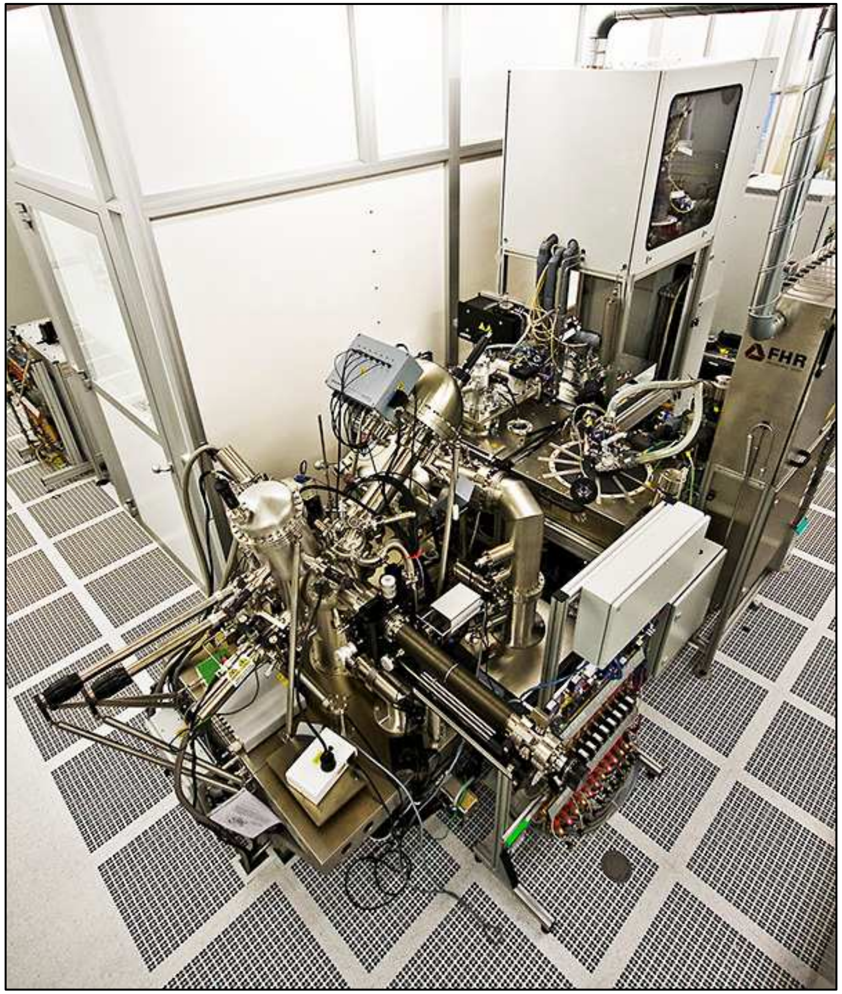
Outline

In-situ analytics for ALD processes

- Motivation for ALD processes
- Principle of atomic layer deposition
- Motivation for in-situ analytics
- ALD tool and available analytics
- Process development
- Process characterization
- Process monitoring and control



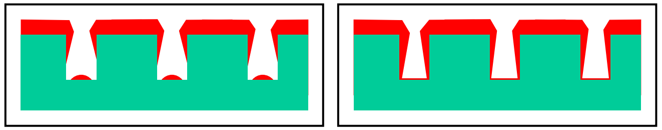
1. Motivation for ALD prozesses

Deposition of ultra thin, highly conformal films with precise thickness and composition control on large substrates, 3D structures or porous materials.

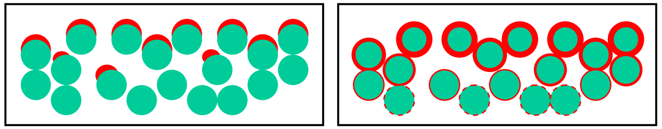
Problems using PVD/CVD

PVD CVD

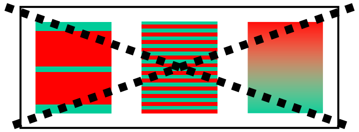
- Conformity in high aspect ratio structures



- No deposition in porous materials possible



- Composition and thickness control of ultra thin films, laminates and graded layers (thickness < 10 nm)



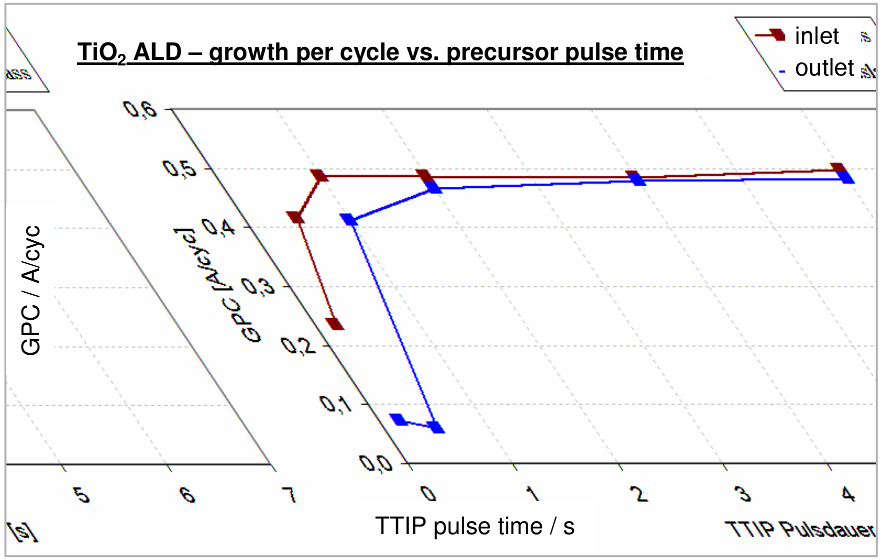
2. Principle of atomic layer deposition

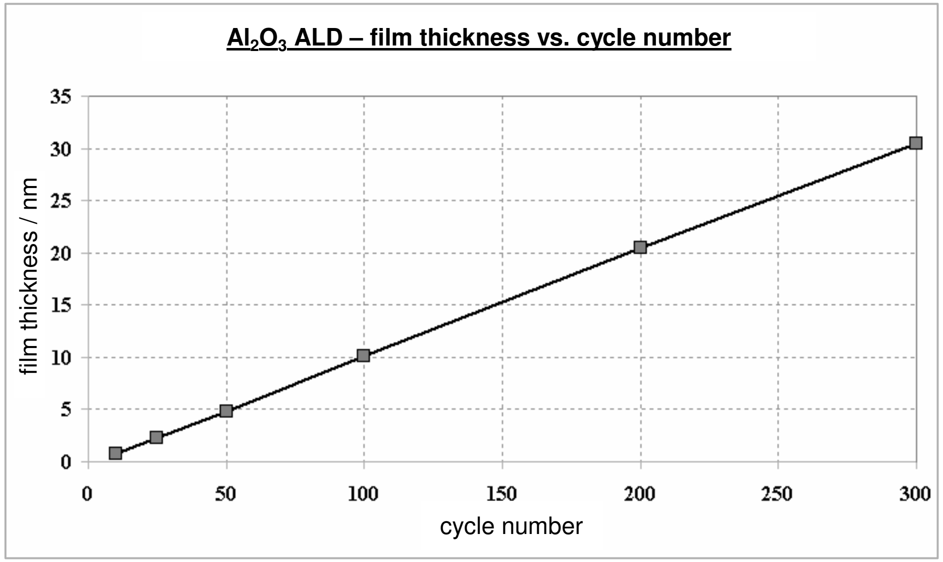
→ Sequential CVD process with temporal or spatial separation of reactants

→ Self-limiting film growth (surface saturation)

→ Cyclic deposition

→ Thickness control by the number of ALD cycles

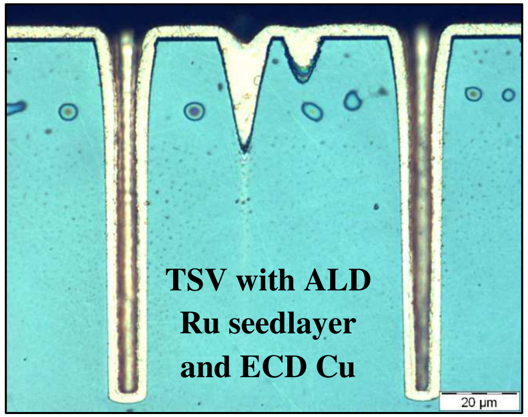


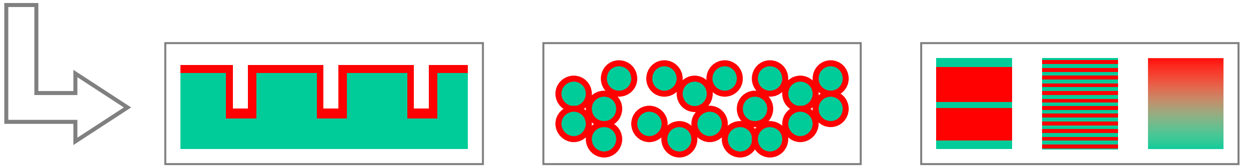


2. Principle of atomic layer deposition

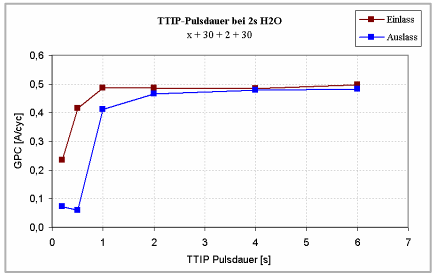
Advantages

- Ultra thin films with high conformity
- Precise control of thickness and composition
- Deposition on large area substrates, in high aspect ratio structure and in porous materials
- Large number of materials and processes
- Low process temperatures



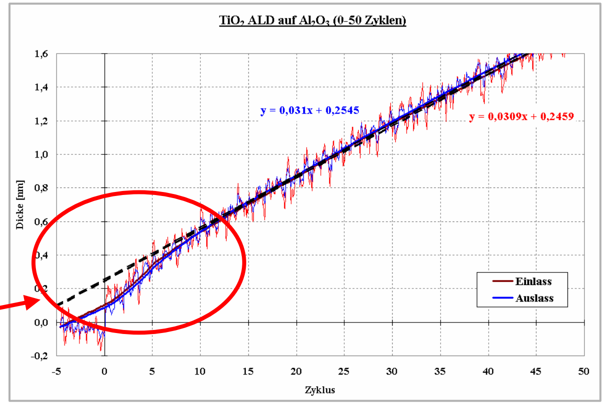


3. Motivation for in-situ analytics



1. **Extensive process development**
- Large number of process and tool parameters
- Interaction of parameters
- Long process times and expensive precursors

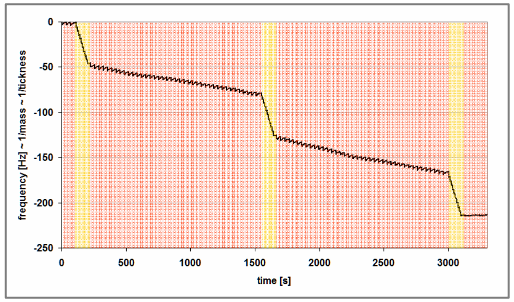
→ Faster and more detailed process development



2. **Film growth characterization**

→ Reaction mechanism studies

→ Substrate effects (during first ALD cycles)



3. **Process monitoring and control**

→ Monitoring of thickness and composition

→ Controlled production of composites and film stacks

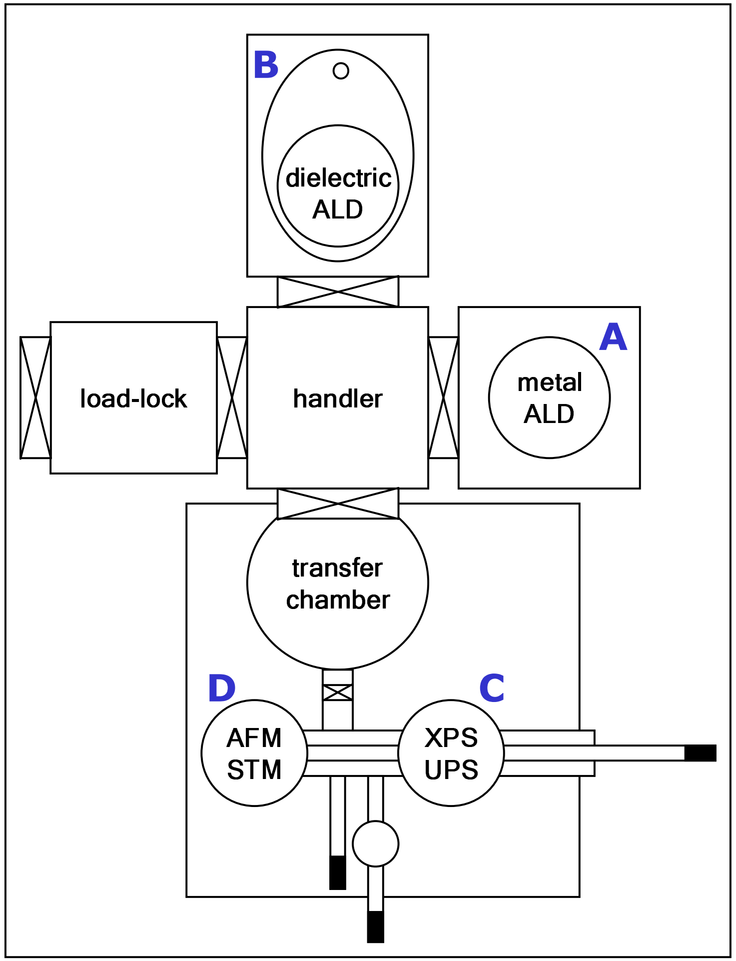
4. ALD tool and available in-situ analytics

ALD tool

- *FHR Anlagenbau* multi-chamber research tool for ALD of metal (**A**) and dielectric (**B**) films
- connected analytics tool (**C** + **D**) for film characterization without vacuum break
- in-situ RTP lamps (> 1000 °C)
- current processes: Al_2O_3, TiO_2, TaC, TaN, AlN, Ru, RuO_x

in-situ analytics

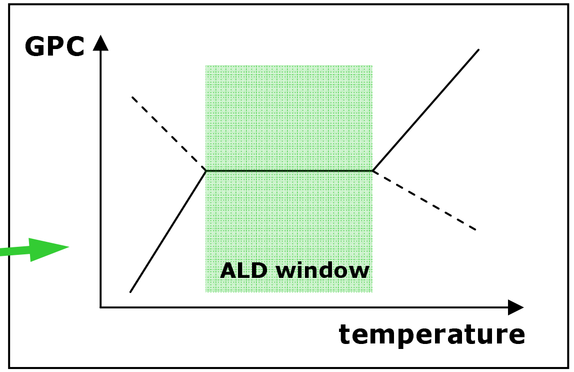
- Spectroscopic Ellipsometry (SE - **A**)
- Quadrupole Mass Spectrometer (QMS – **A/B**)
- Quartz Crystal Microbalances (QCM - **B**)
- X-Ray Photoelectron Spectroscopy (XPS - **C**)
- UV Photoelectron Spectroscopy (UPS - **C**)
- Scanning Probe Microscopy (AFM/STM - **D**)



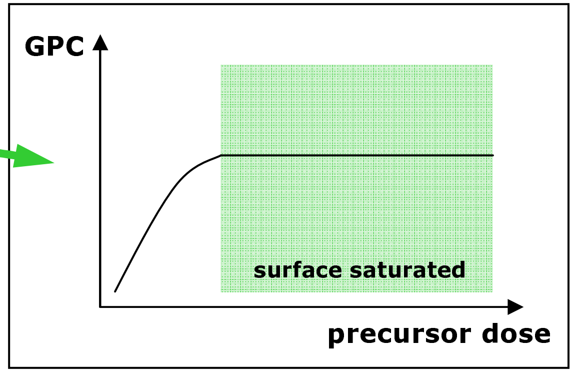
5. Process development

Important parameters for ALD process development:

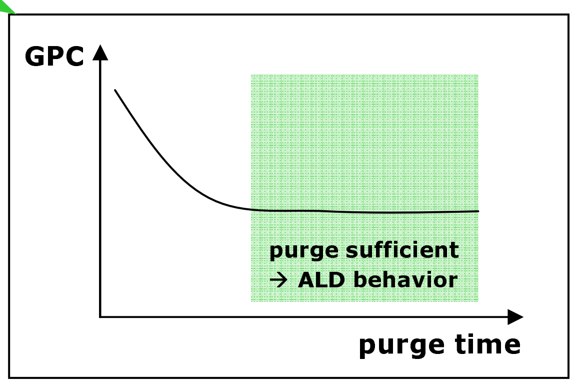
- **Substrate temperature**
  → defined by substrate, precursor, application or desired film properties



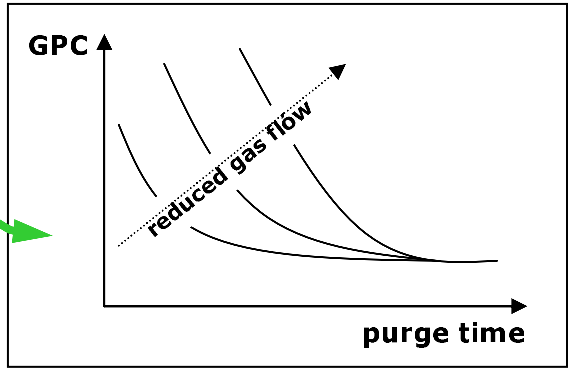
- **Precursor and reactant doses**
  → as low as possible to save time and money but as high as needed for saturation



- Sufficient **purge times** to avoid CVD
  → as short as possible to save time



- **Gas flow** and **pressure**
  → tool and application dependent
  → affecting other parameters



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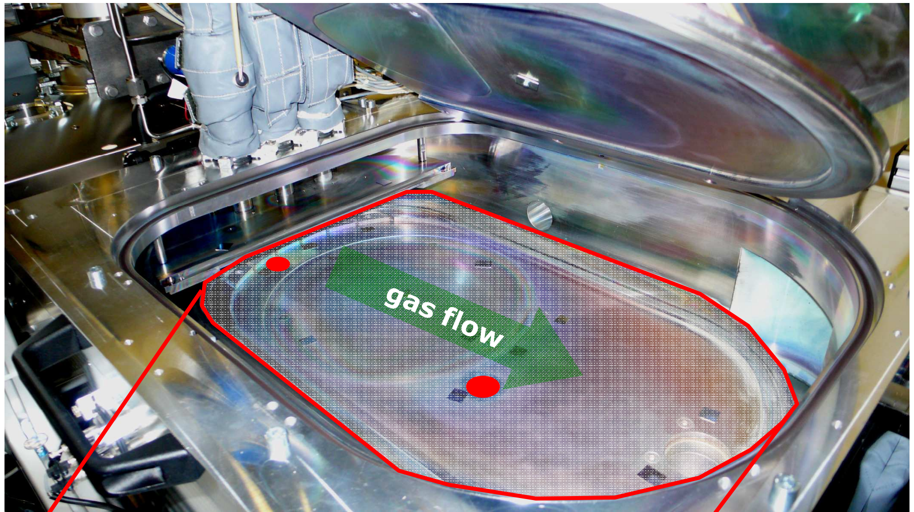
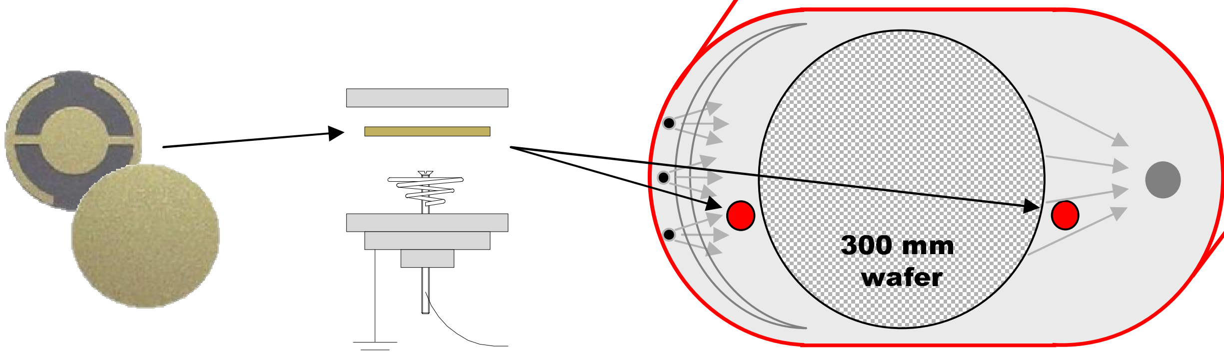
5. Process development

- cross-flow ALD reactor
  with heated inner chamber
  for 300 mm wafer

- 2 self-build sensor heads
  for Quartz crystal microbalances
  near gas inlet and exhaust





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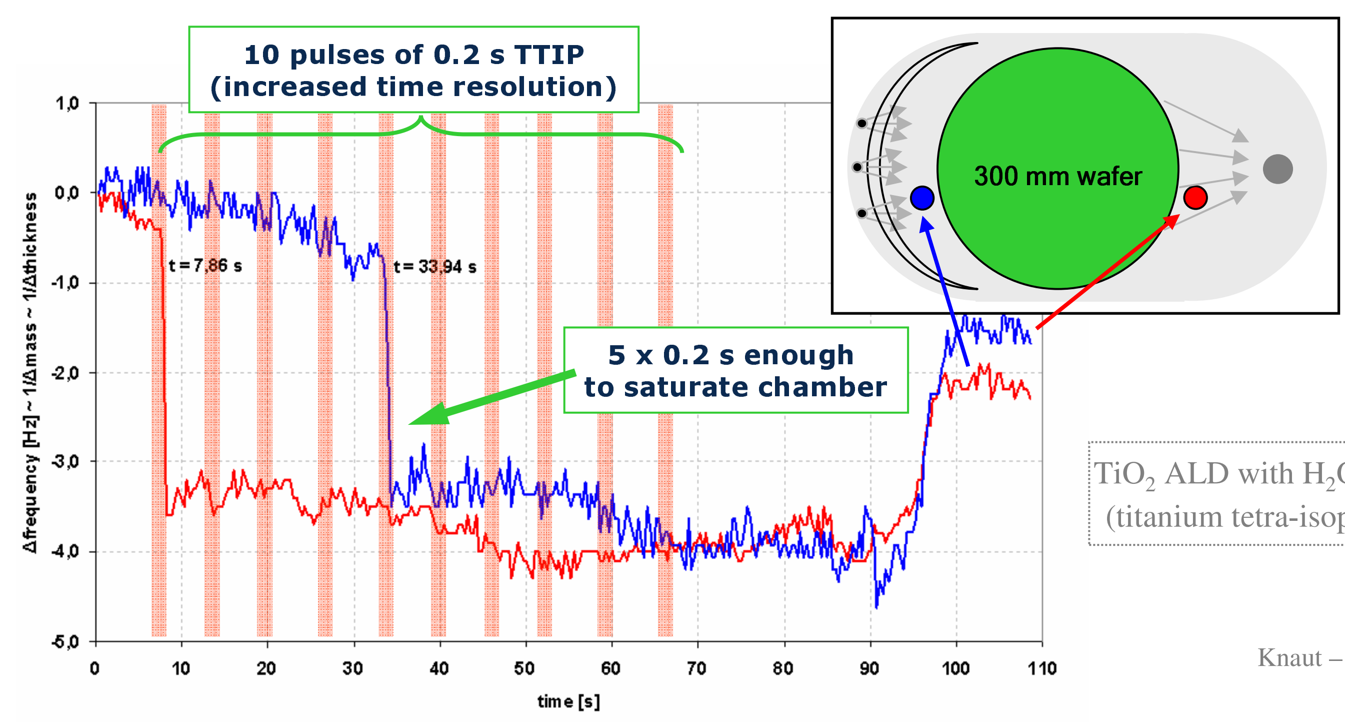
5. Process development

QCMs for precursor dosing tests

→ Comparison between QCM near gas inlet and exhaust

→ Estimating precursor doses → starting points for faster process development



TiO_2 ALD with H_2O and TTIP (titanium tetra-isopropoxide)

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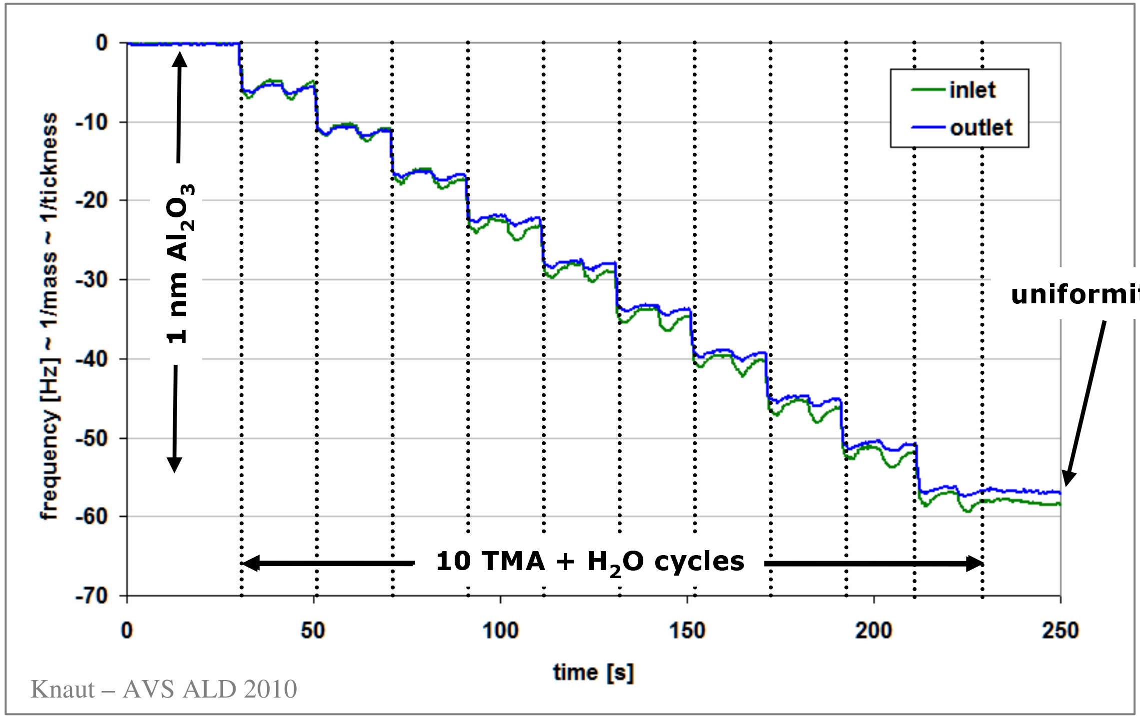
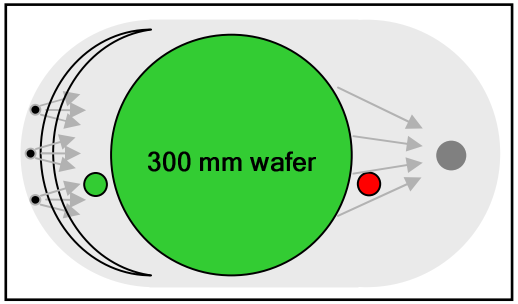
5. Process development

Combination of two sensors

→ Real-time thickness and uniformity monitoring possible

→ Very high sensitivity





Uniformity values comparable to reference measurements by Spectroscopic Ellipsometry

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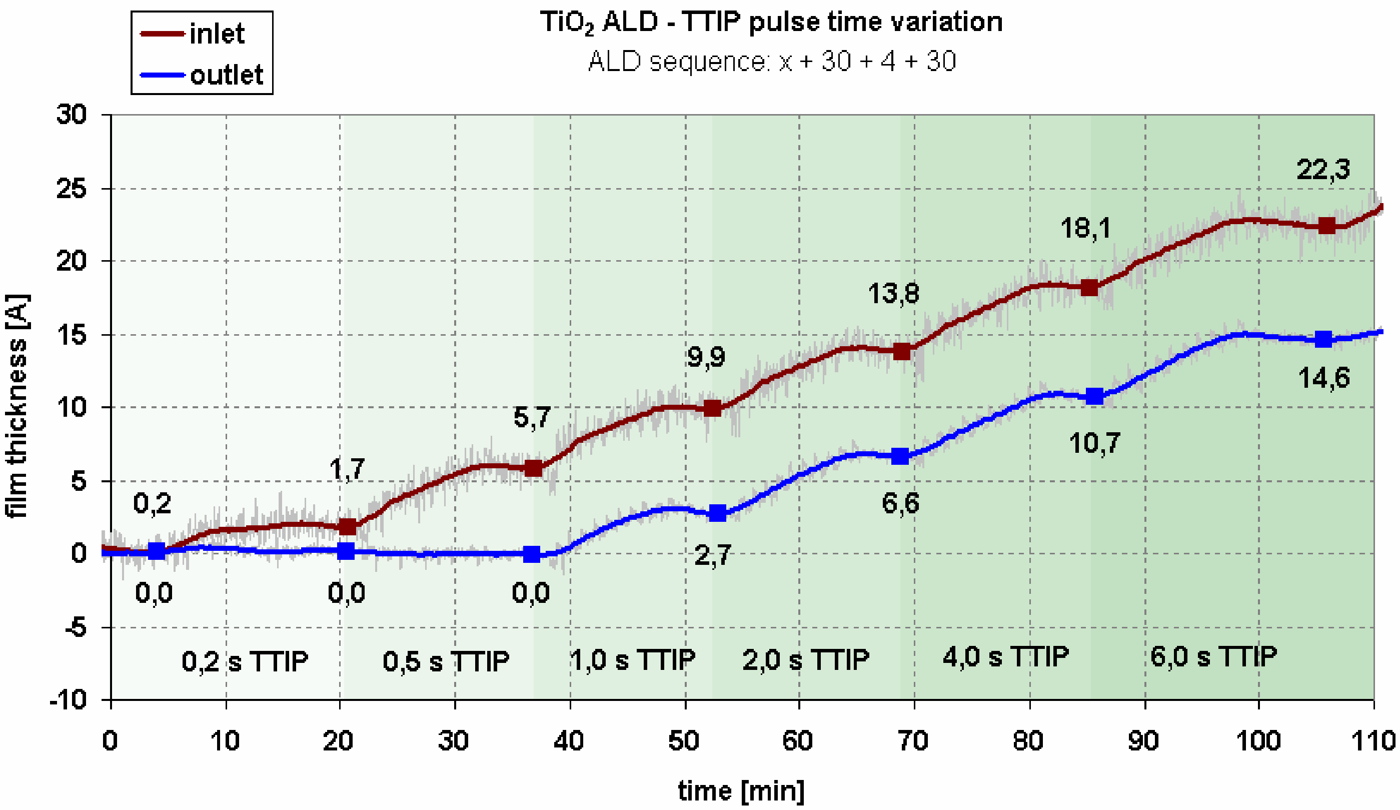
5. Process development

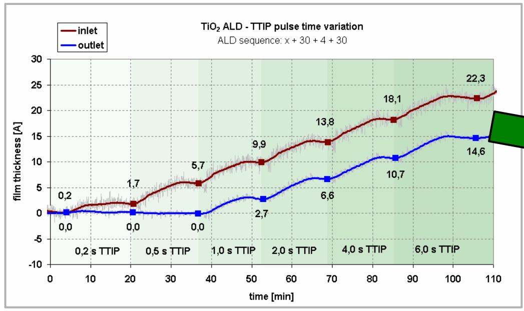
Parameter variation and GPC calculation from QCM frequency

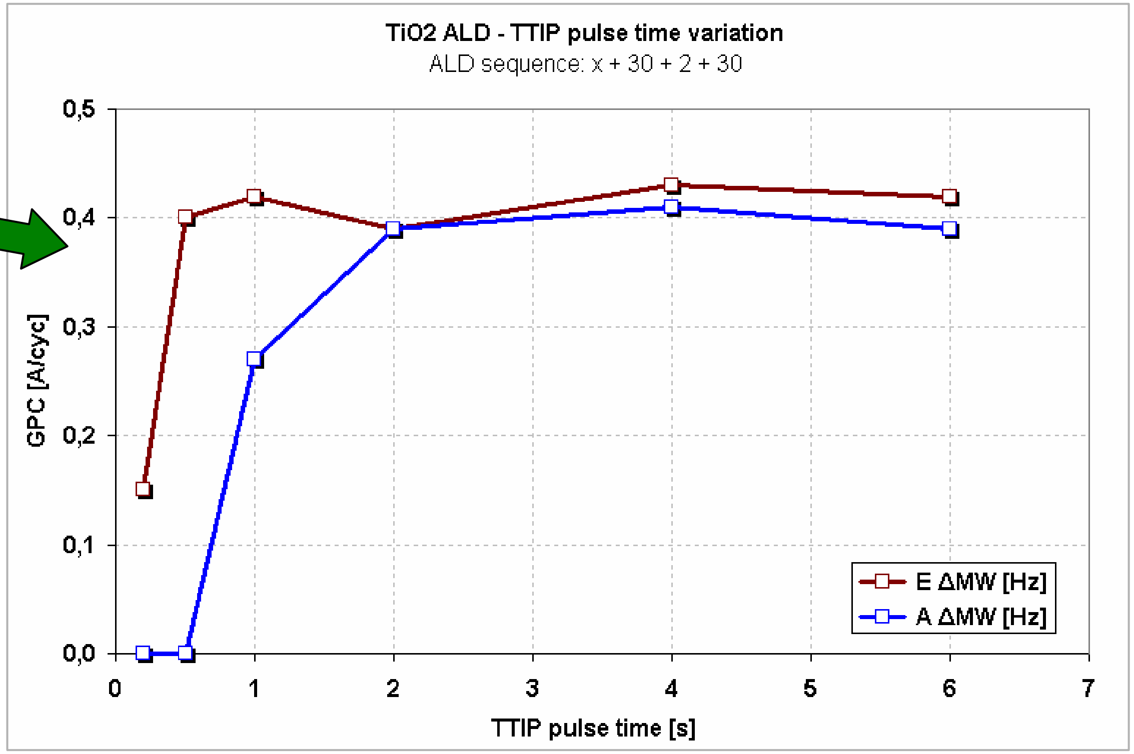


Short sub-processes of only a few cycles to characterize a parameter set

5. Process development

Parameter variation and GPC calculation from QCM frequency





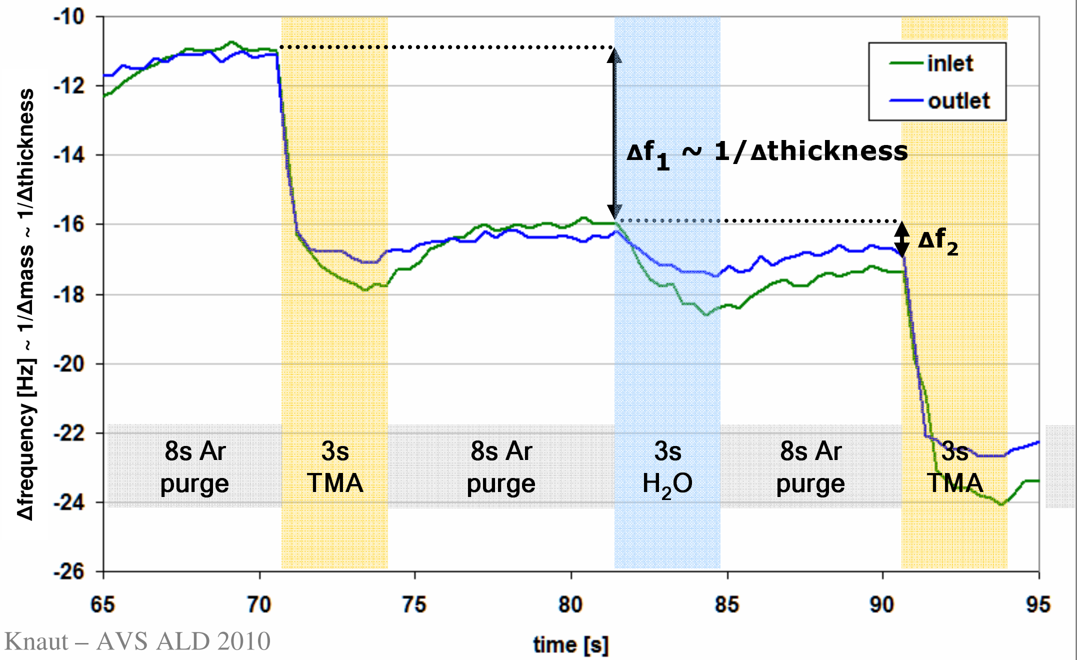
Inlet and outlet saturation curves obtained from varying TTIP pulse time in 10 cycles sub-processes → 100 min (not days) for GPC and uniformity information

6. Process characterization

Observing half-reactions and film growth using QCMs

→ Possibility to compare processes with theory of chemistry and film growth

→ Reactions during single ALD cycles observable



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Al_2O_3 ALD - chemical reaction:

$Al(CH_3)_3 + 3H_2O \rightarrow Al_2O_3 + 6CH_4$

Precursor step (Δf_1)

TMA chemisorption = **+ 92 amu**

Reactant step (Δf_2)

Ligand exchange = **+ 6 amu**

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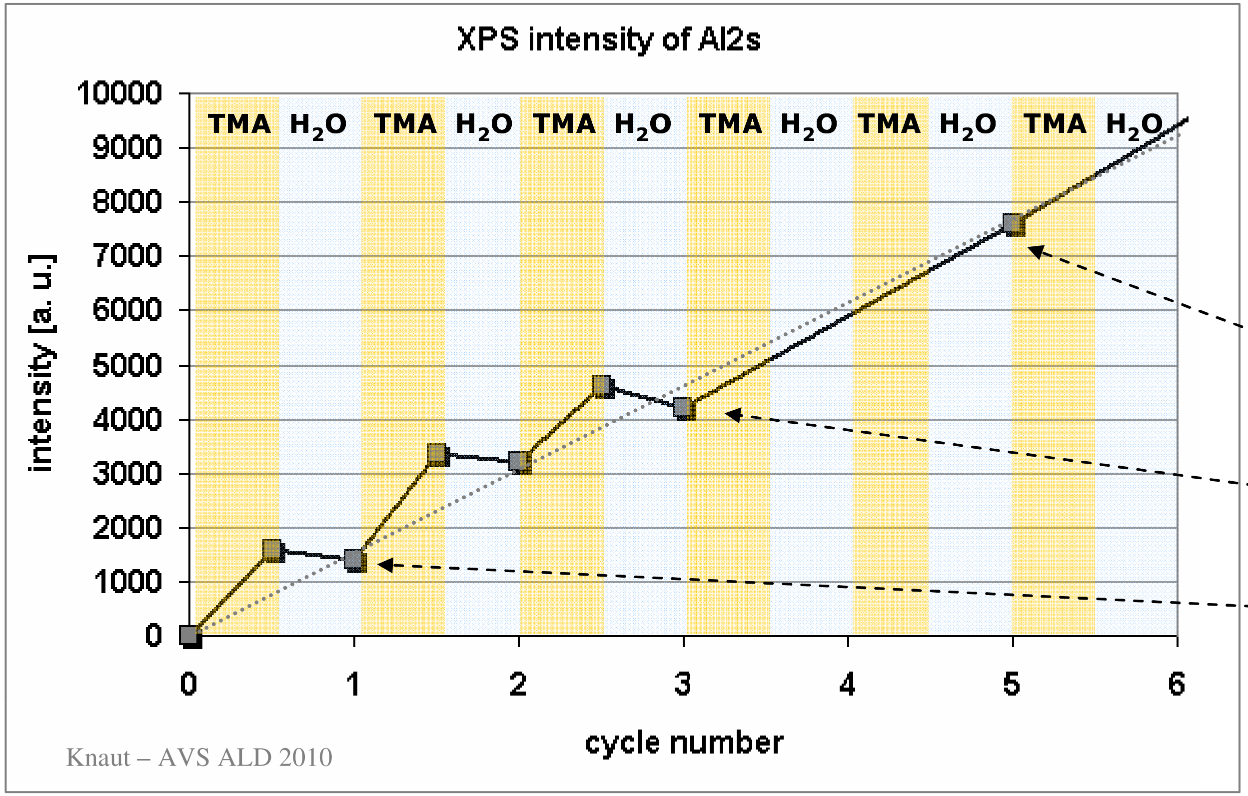
6. Process characterization

XPS study without vacuum break to analyze film growth during first ALD cycles

→ alternating ALD deposition and XPS measurement steps



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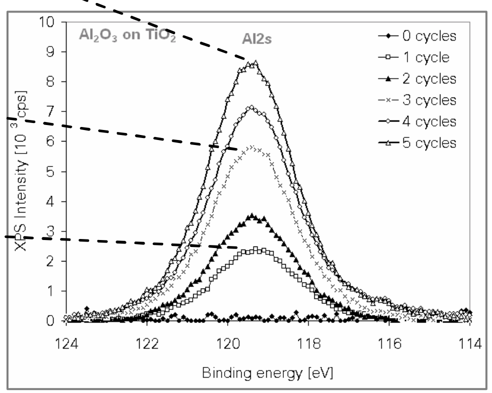
Al_2O_3 ALD with H_2O and TMA (trimethylaluminium) on TiO_2

→ Ideal ALD growth during first Al_2O_3 ALD cycles on TiO_2

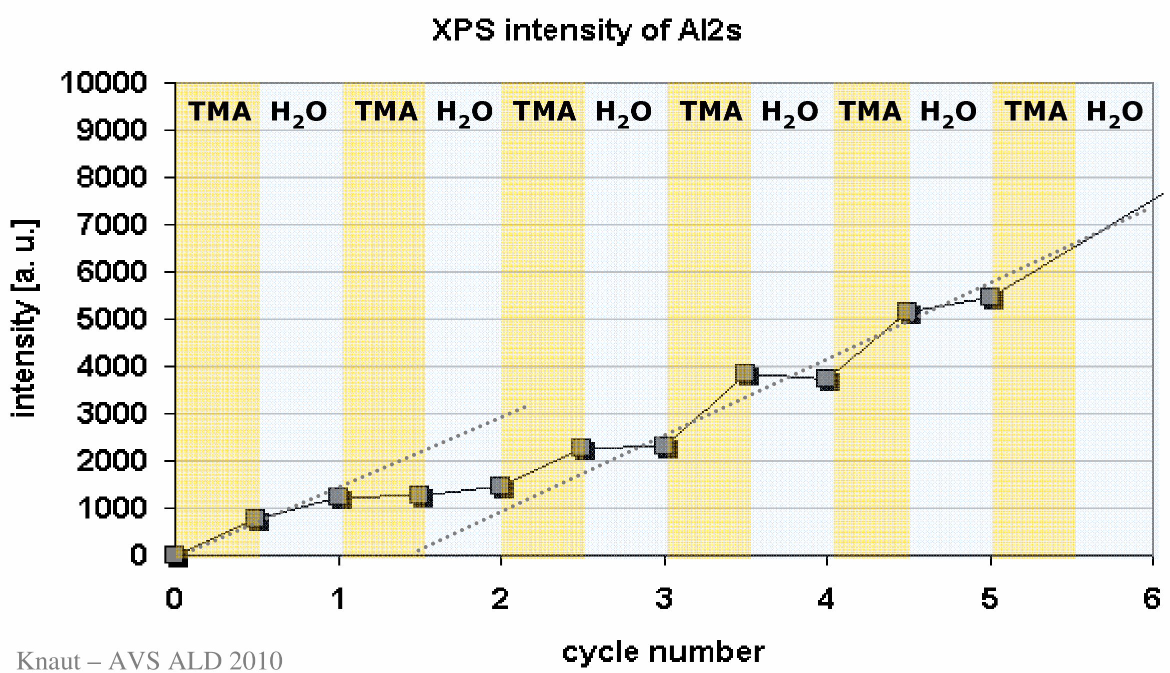


6. Process characterization

XPS study without vacuum break to analyze film growth during first ALD cycles

→ already small film growth deviations visible

→ coverage and film thickness calculable



Al_2O_3 ALD with H_2O and TMA (trimethylaluminium) on ALD Ru

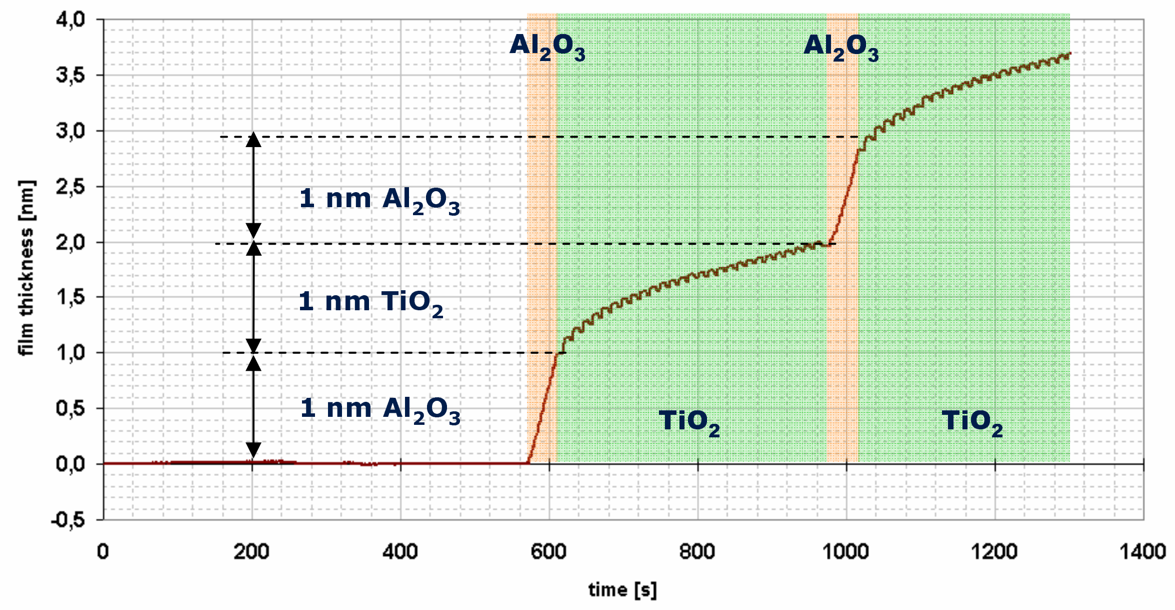
→ slightly inhibited growth during first Al_2O_3 ALD cycles on Ru

7. Process monitoring and control

QCM measurements during ALD processes

→ Monitoring process interactions, drifts and cross-contaminations

→ Controlling composition of film stacks, laminates and graded films



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laminate of 1 nm ALD films:

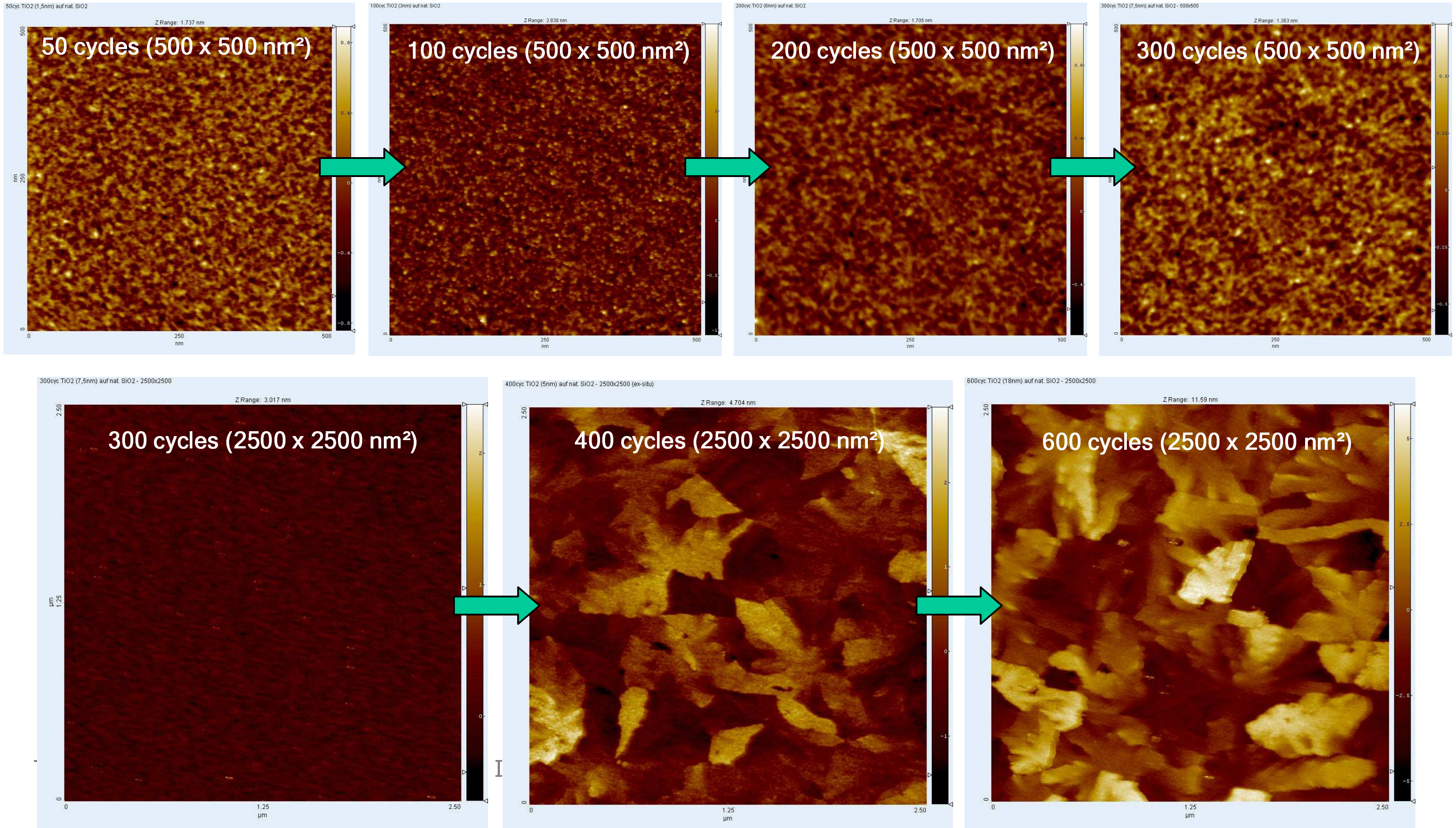
Al_2O_3 ALD with H_2O and TMA (trimethylaluminium)
+
TiO_2 ALD with H_2O and TTIP (titanium tetra-isopropoxide)

→ optimized processes but interaction during first TiO_2 cycles

→ thicknesses obtainable from QCM frequency

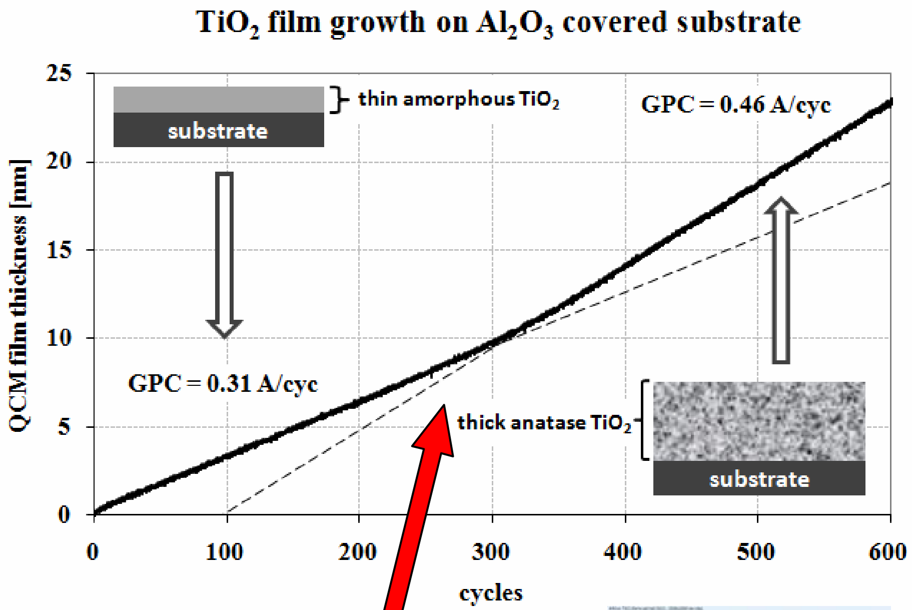
7. Process monitoring and control

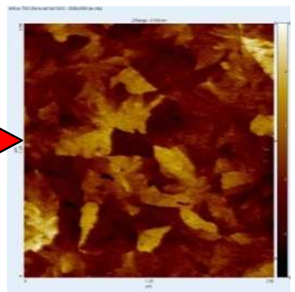
Control and optimization of film properties (ALD of TiO_2)



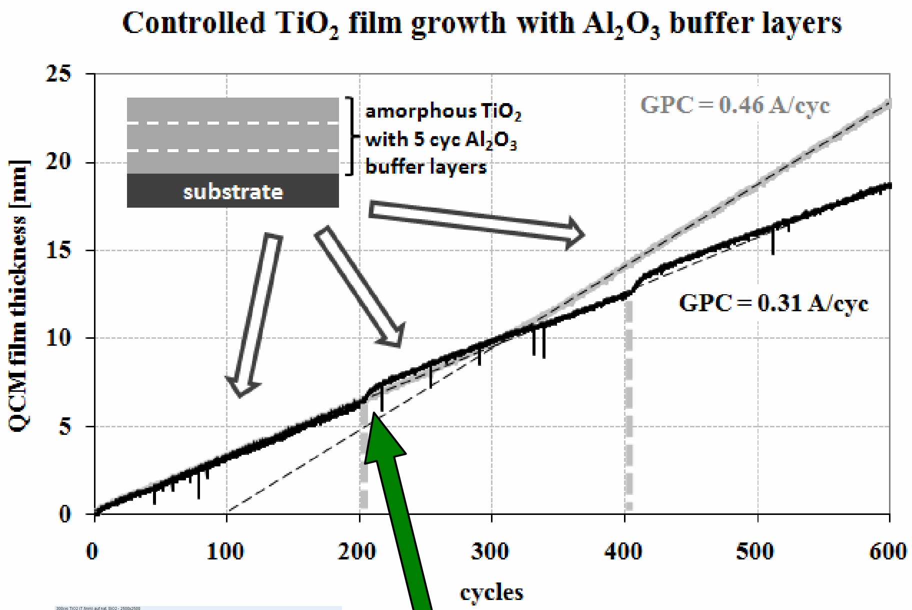
7. Process monitoring and control

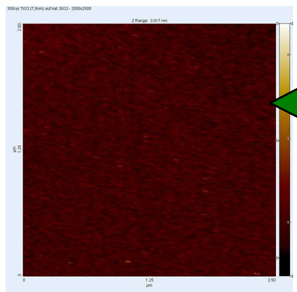
Control and optimization of film properties





pure TiO_2:
growth changes
after 300 cyc
→ rough films





TiO_2-Al_2O_3-laminate:
constant growth rate,
no crystallization,
smooth films

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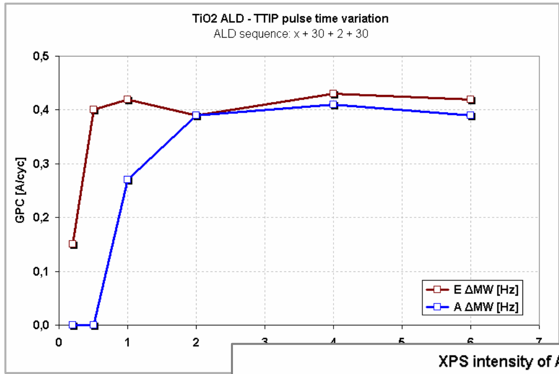
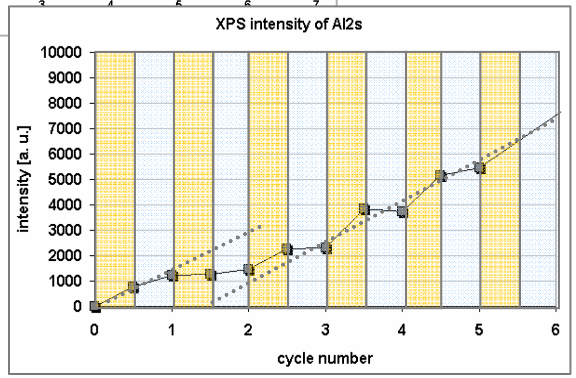
8. Summary

In-situ Monitoring of ALD processes for …



… improved process development

- Dosing test to estimate starting points
- Faster testing of parameter sets

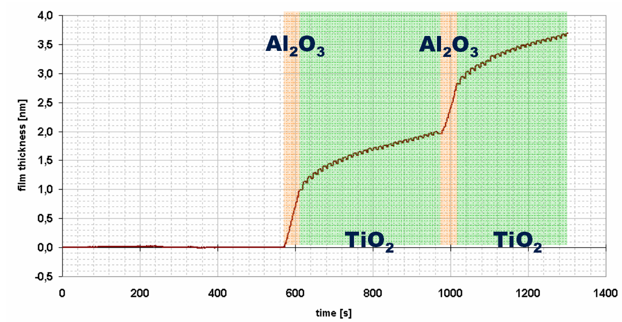


… characterization of film growth and substrate effects

- Possibility to analyze initial film growth during first cycles
- Sensitivity to single gas pulses

… controlled deposition of ALD films, stacks and laminates

- Monitoring of complex ALD processes
- Controlling film properties



»Wissen schafft Brücken.«