In situ X-Ray Reflectivity measurements during Sputtering of Vanadium Carbide thin films

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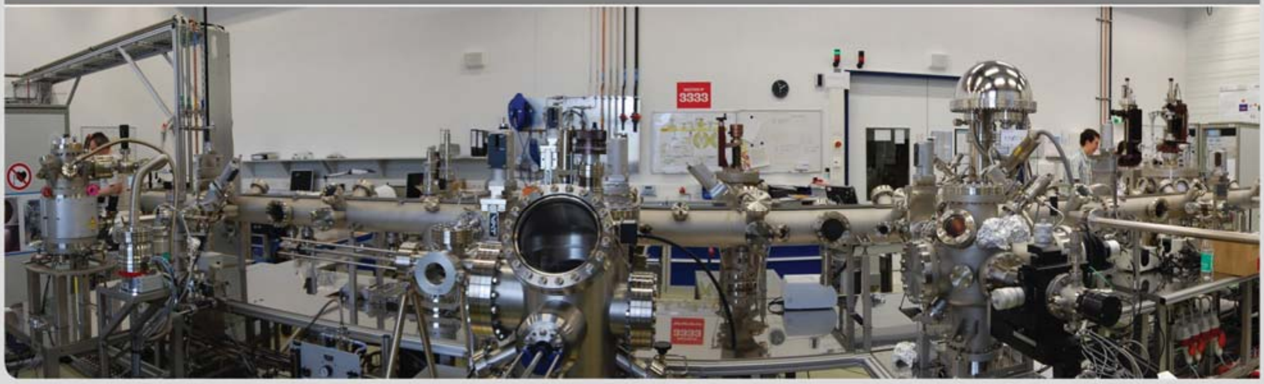

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- Motivation
- *In situ* X-Ray Reflectivity
- Three Examples:
  - *In situ* XRR at different DC Power
  - *In situ* XRR at different Growth Temperatures
  - Interruption of Deposition
- Summary & Outlook

Motivation

Vanadium Carbide (VC_{1-x})

- Growth of thin films by Sputtering
- Hard coating material for tools

deposition conditions and **microstructure formation** define **mechanical properties**

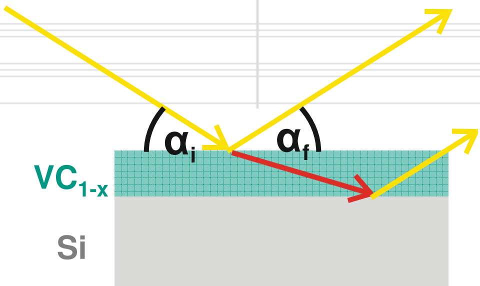
→ **Understand growth process depending on sputtering conditions**

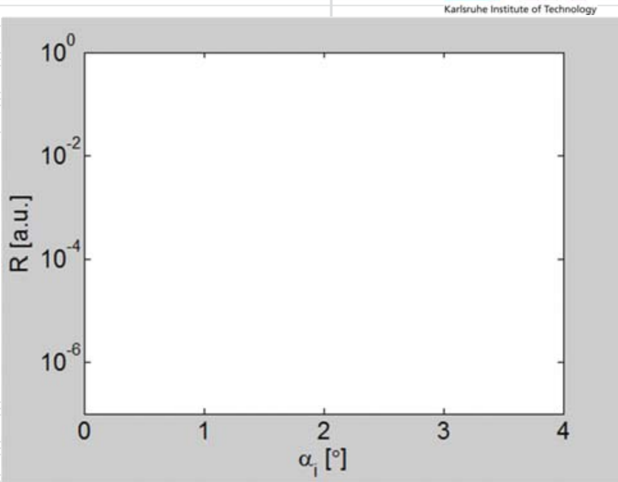
→ **Investigation needs suitable methods**

- **nondestructive** monitoring of growth process
- resolution in **sub-nanometer scale**
- compatibility with the **gas atmosphere**
- investigation of
  - **polycrystalline** material
  - **high deposition rates** (0.22 nm/s @ DC Power 200 W)

➡ ***In situ* X-Ray Reflectivity**

Basics of X-Ray Reflectivity





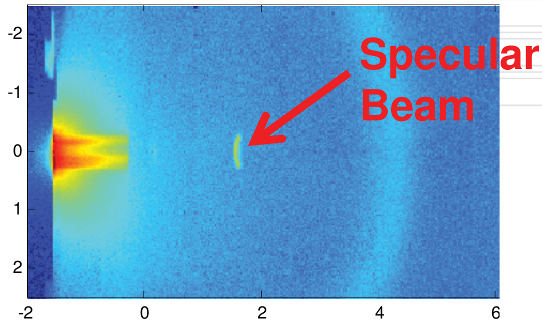
- Electron density ('Critical Angle')
- Thickness ('Kiessig fringes')
- Roughness ('Slope') [1]
- Description by Parratt-Algorithm [2]
  - Fully dynamical description of XRR

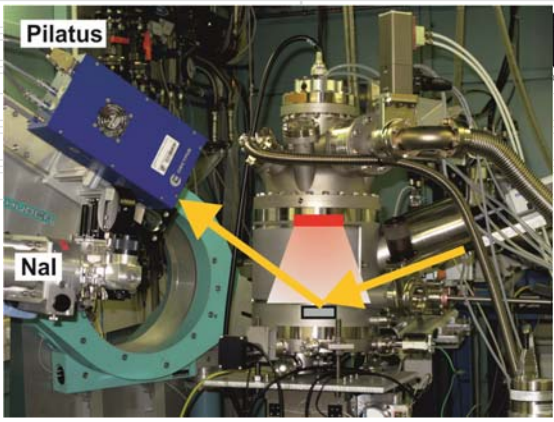
➡ **Two options** to measure *in situ* XRR

1. Full angular range XRR
2. XRR at a fixed angular position

[1] Pietsch, Holy, Baumbach, *High Resolution X-Ray Scattering from thin films and lateral Nanostructures*, Springer 2004
[4] Parratt, *Phys. Rev. 95*, **2**, p. 359-369,(1954)

Experimental Setup





- **Setup @ MPI-Beamline:**
  - Energy: 10 keV
  - Beamsize: 300µm x 200µm
  - Optics
    - Resolution in q_z: ~ 0.005 $Å^{-1}$
  - Detector: Pilatus 1K
    - Resolution in time: ~1.1-2.3 s

- **Sputter conditions** [1]:
  - Target: VC_{1-x}
  - Substrate: Si(100) with natural oxide
  - Target-substrate Distance: 10 cm
  - Argon Pressure: 2 x 10^{-3} mbar
  - Deposition rate 0.22 nm/s@ 200 W

[1] Krause , Kaufholz et al., J. Synchrotron Rad. (2012),**19**, 216-222

In situ X-Ray Reflectivity: "full angular range"

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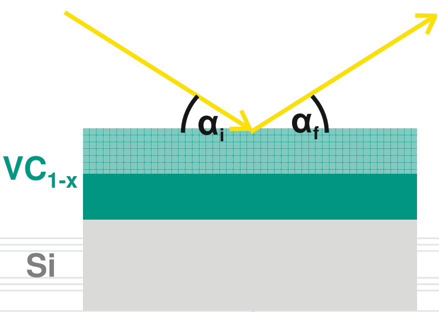
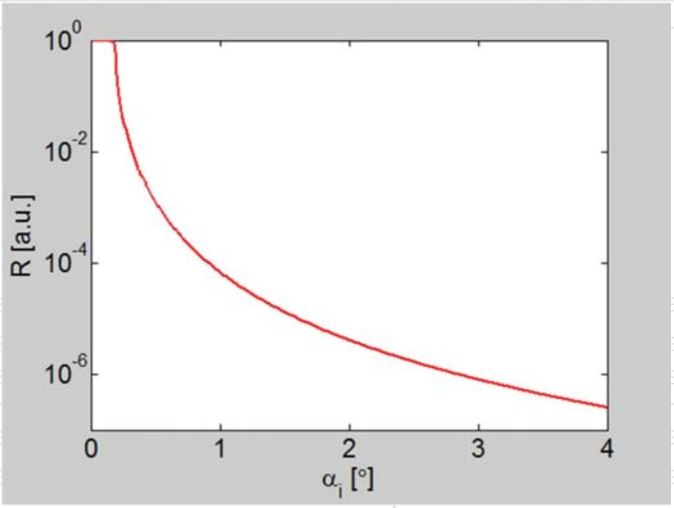
- Measure **full angular range**



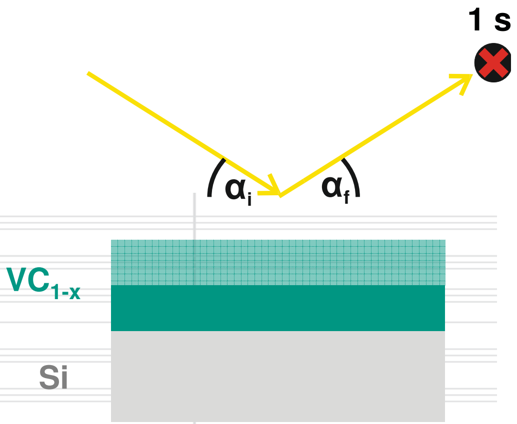


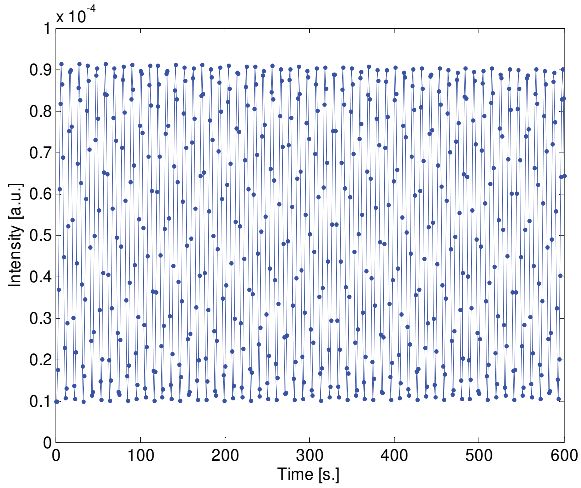
- High deposition rate of 0.22 nm @ 200 W → ~90nm deposition/XRR
- Possible electron density and roughness changes

→ Interpretation of XRR curve difficult

In situ X-Ray Reflectivity: "fixed angular position"

- Detector and sample are at a **fixed angular position**
- Measuring Pre- and Post-growth full angular range XRR





Simulation Input:
DC Power: 200 W → Deposition Rate: 0.217 nm/s
$\alpha_i = 1.6°$

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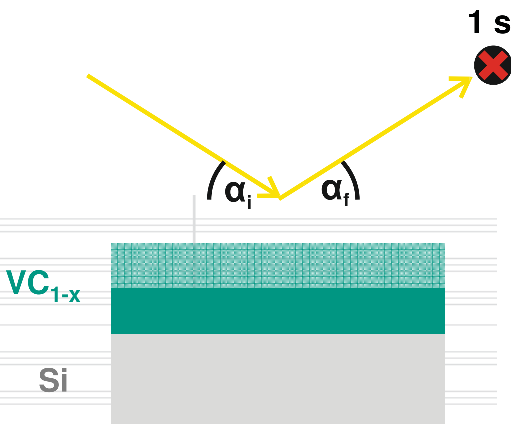

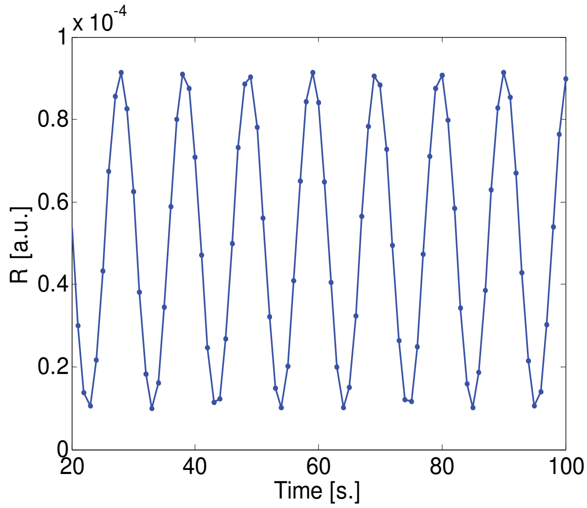

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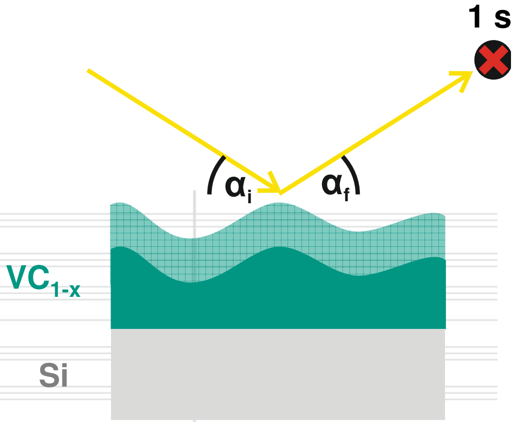

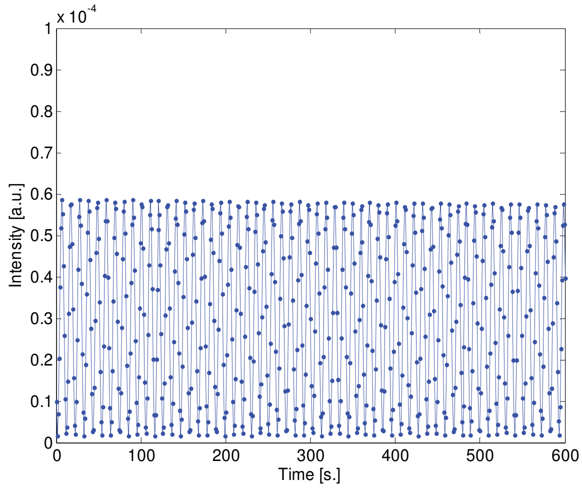

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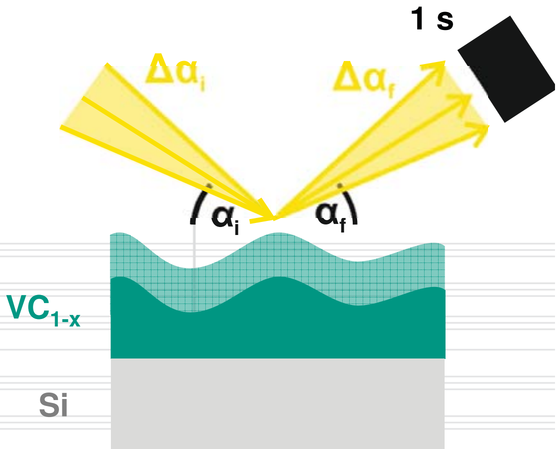

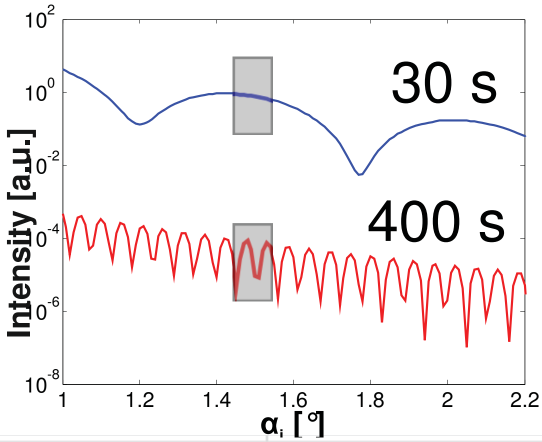

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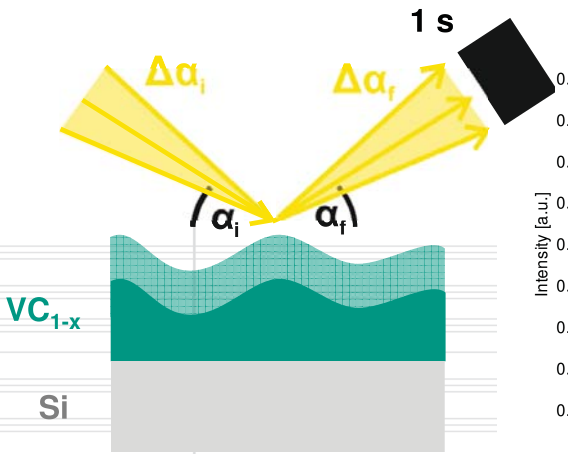

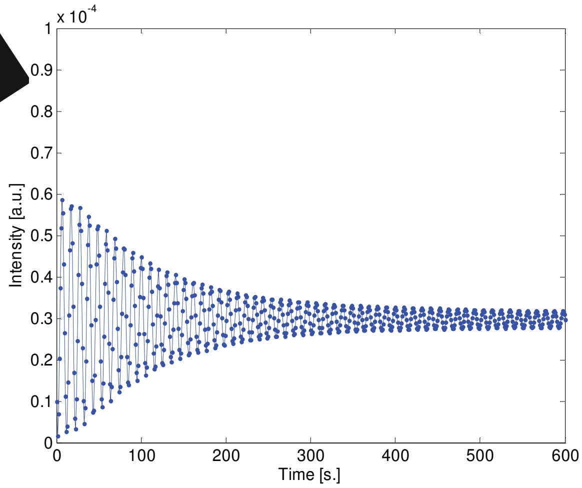

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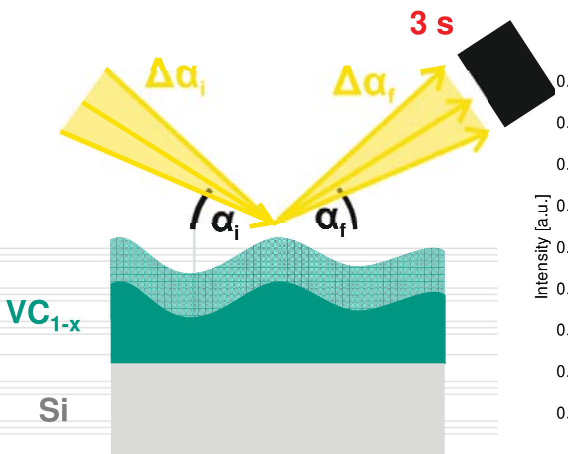
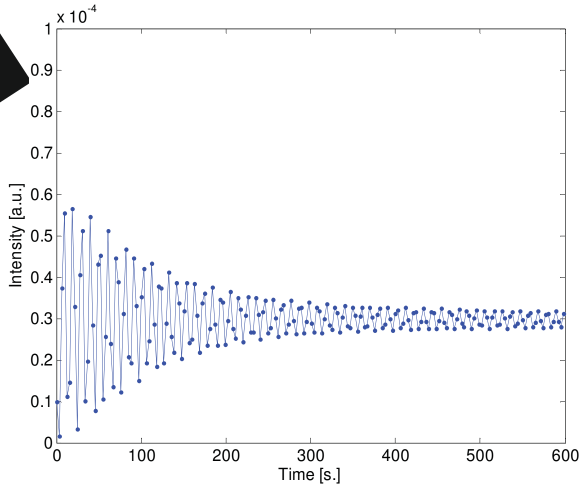
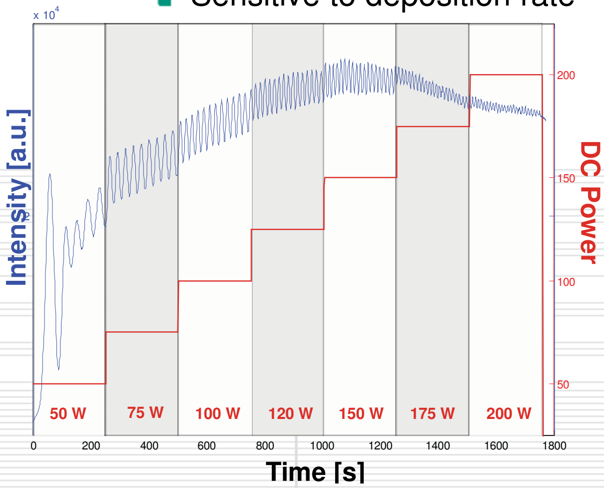


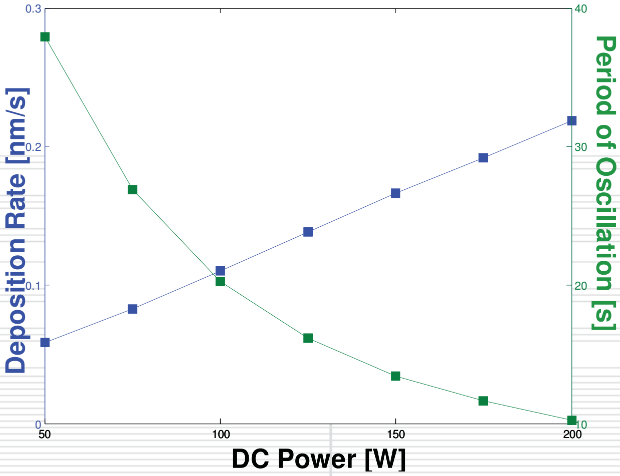


Simulation Input:
DC Power: 200 W → Deposition Rate: 0.217 nm/s
$\alpha_i = 1.6°$

Example 1: Determination of Deposition Rate depending on DC Power at RT

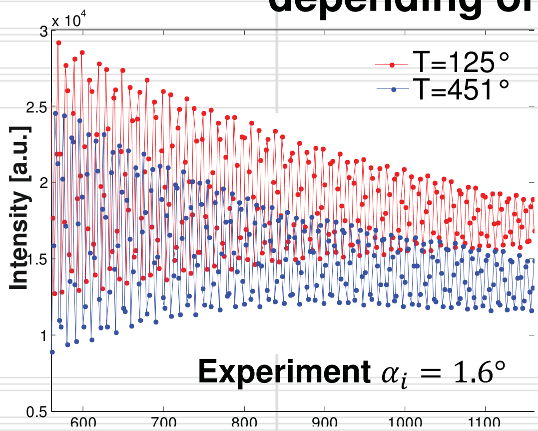
- Increase of DC Power by ΔP = 25W every 250s
- $\alpha_i = 1.6°$:
  - Error due to changes in electron density <1%
  - Sensitive to deposition rate

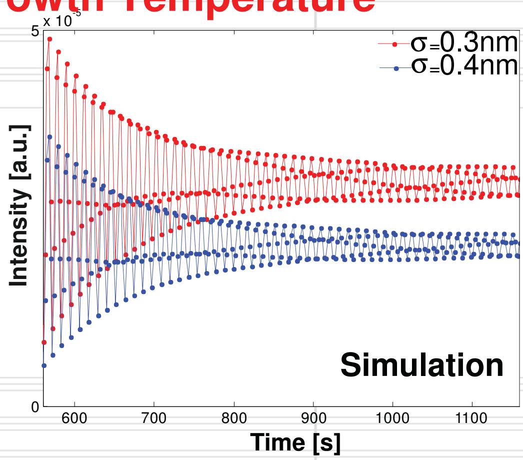




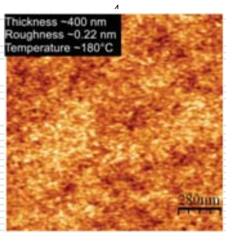
→ Deposition Rate ~ DC Power

Example 2: Monitoring of Roughness depending on Growth Temperature

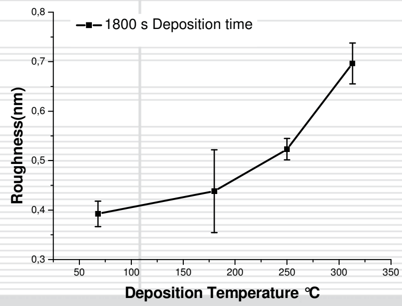




- **Increase of Temperature** leads to **increase of Roughness**
- Consistent with ex situ AFM

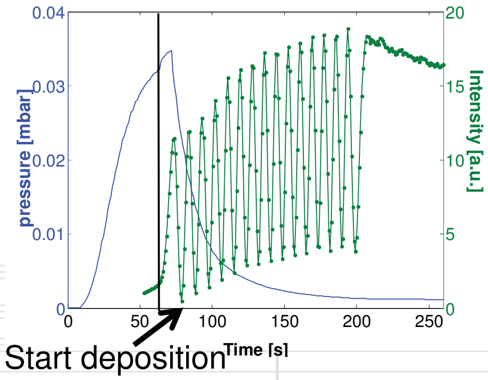


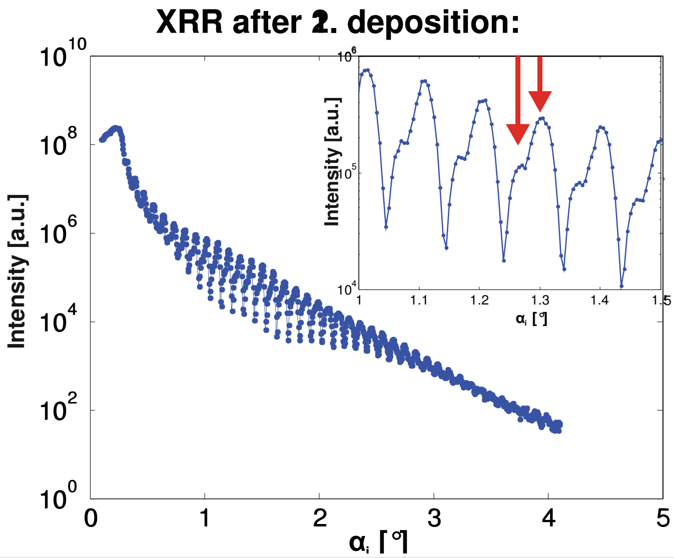
Ex situ: AFM



Example 3: Different Electron Densities due to Interruption of Deposition

- Interruption of deposition after 200s @ RT and DC Power of 200 W





Multilayer of **one material**

Summary

- *In situ* X-Ray Reflectivity is suitable for investigation of VC_{1-x}
  - Sensitive to
    - → **Deposition Rate**
    - → **Roughness**
    - → **Density**
- Sensitive to different sputtering conditions

Outlook

- Simulation of *in situ* XRR curves
  - Growth Model (Scaling law)
  - Include diffuse scattering
  - Limits of method
- Combining with other methods for a better understanding
  - *In situ* & *ex situ* X-Ray Diffraction and Absorption Spectroscopy
  - XPS, AFM, TEM, … (in UHV conditions)
  - Measuring Hardness via Nano-/Microindentation

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Thank You for Your Attention !