Karlsruhe Institute of Technology

In situ control of the structure formation of magnetron sputtered Vanadium Carbide coatings: periodic modulation of the microstructure

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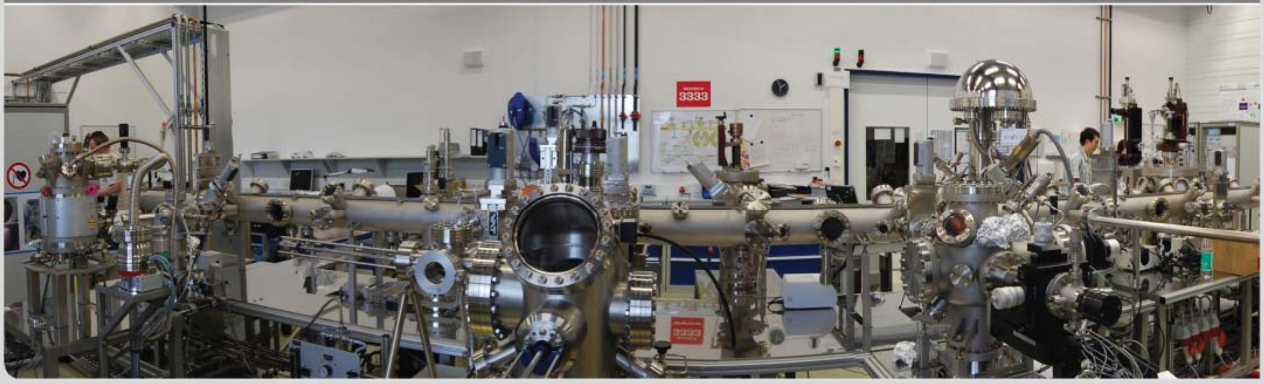
Institute for Photon Science and Synchrotron Radiation (IPS)



Motivation

- Hard coating materials
  - Enhance life time of tools, artificial medical implants …
- Multilayer systems
  - Tailoring of e.g. optical, tribological, mechanical properties
  - Alternating layers of **two** materials (e.g. **two** different average densities)

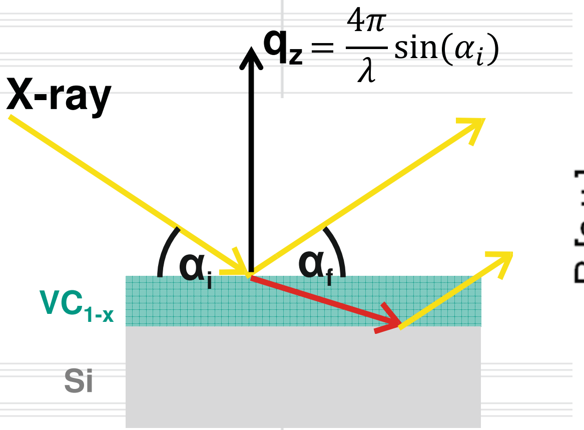
New approach:

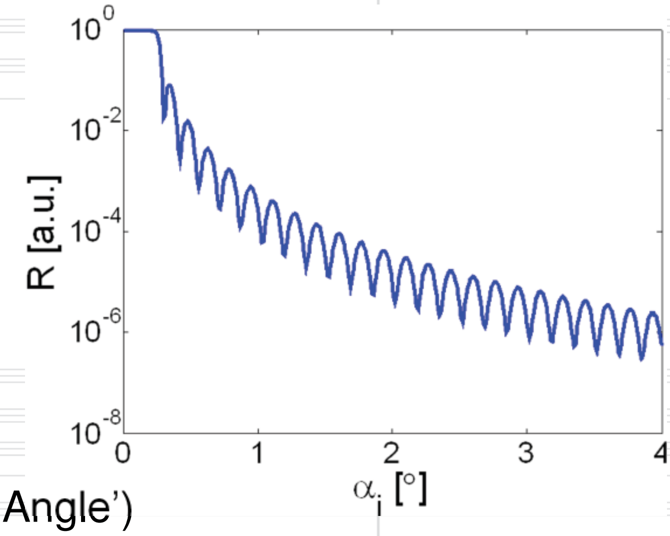
Growth conditions can influence the average density of a single layer

Is it possible to grow a "one-material" multilayer?

→ *In situ* X-ray reflectivity study during growth

Basics of X-Ray Reflectivity

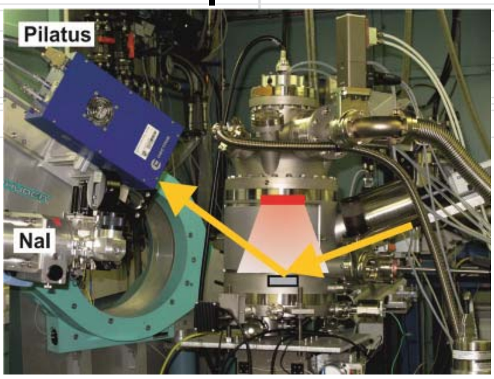


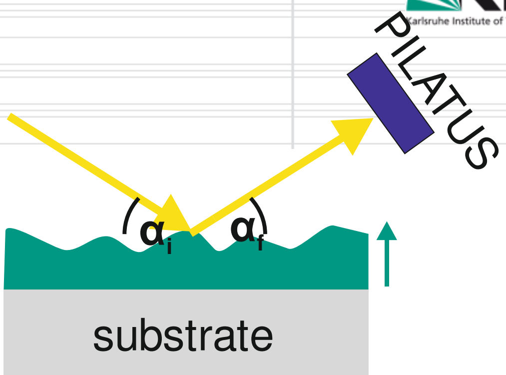


- Electron density ('Critical Angle')
- Thickness ('Kiessig fringes'): $D = \frac{2\pi}{\Delta q_z}$
- Roughness ('Slope')

[1] Pietsch, Holy, Baumbach, *High Resolution X-Ray Scattering from thin films and lateral Nanostructures*, Springer 2004

In situ: Experimental Setup



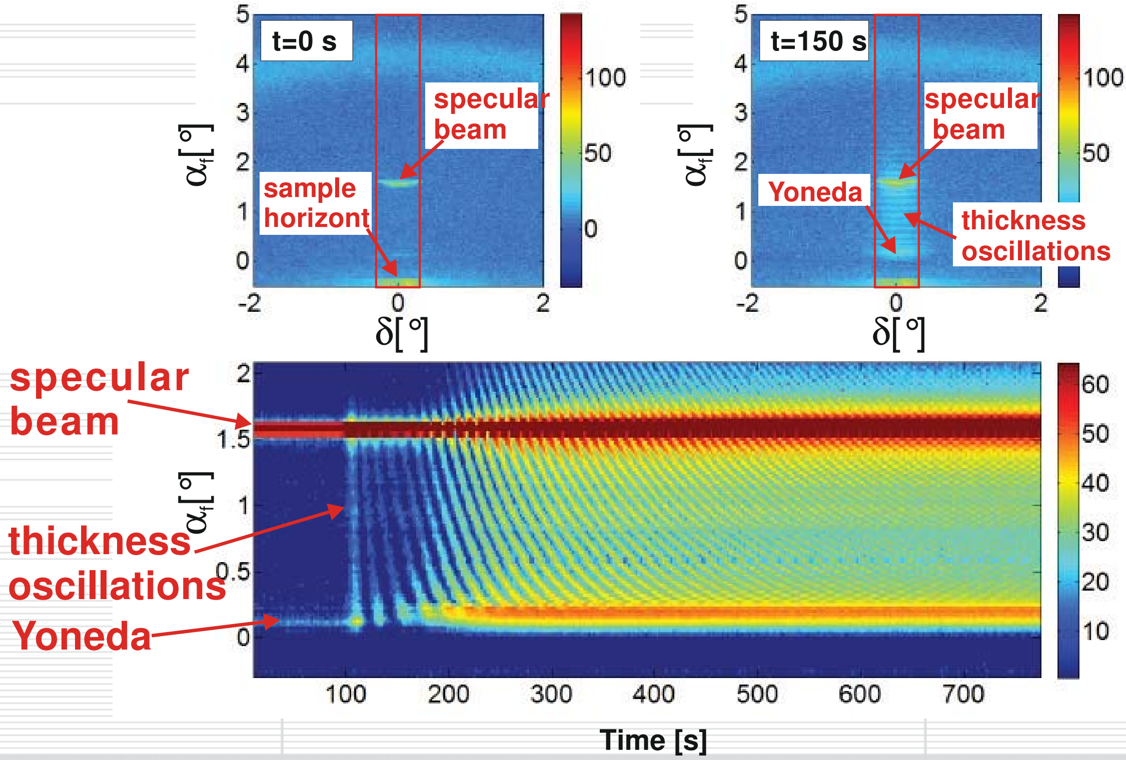


- Sputter conditions: [1]
  - Target: Vanadium Carbide
  - Substrate: Si(100) with natural oxide
  - Target-substrate Distance: 10 cm
  - Argon Pressure: 2 x 10^{-3} mbar
  - Fully automized sputtering process
  - 0.22 nm/s at DC Power 200 W
- Setup @ MPI-Beamline:
  - Energy: 10 keV
  - Beamsize: 300µm x 200µm
  - XRR measurements at fixed angular position:

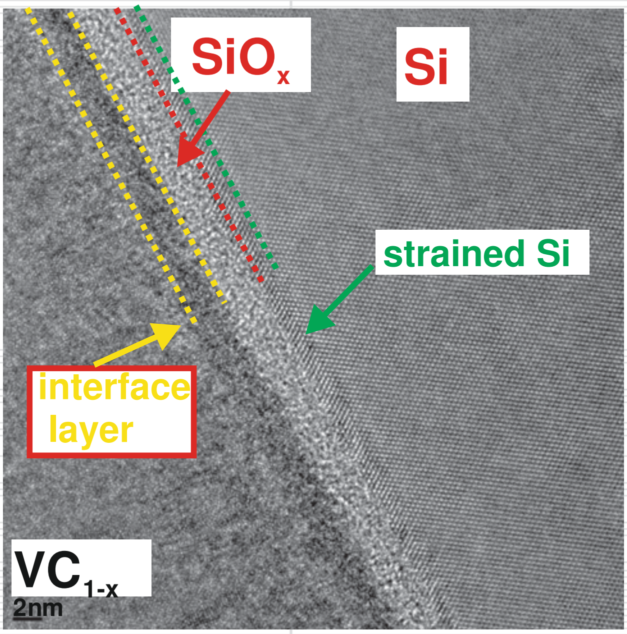
    $$\alpha_i = \alpha_f = 1.6°$$
  - Detector: Pilatus 100K

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

Pilatus measurements at $\alpha_i = \alpha_f = 1.6°$



TEM Analysis *after growth*



- Vanadium Carbide on Silicon
- DC Power: 50 W
- Room Temperature

- Bilayer system:

→ **Growth of dense interface layer**

- *In situ* XRR measurements:

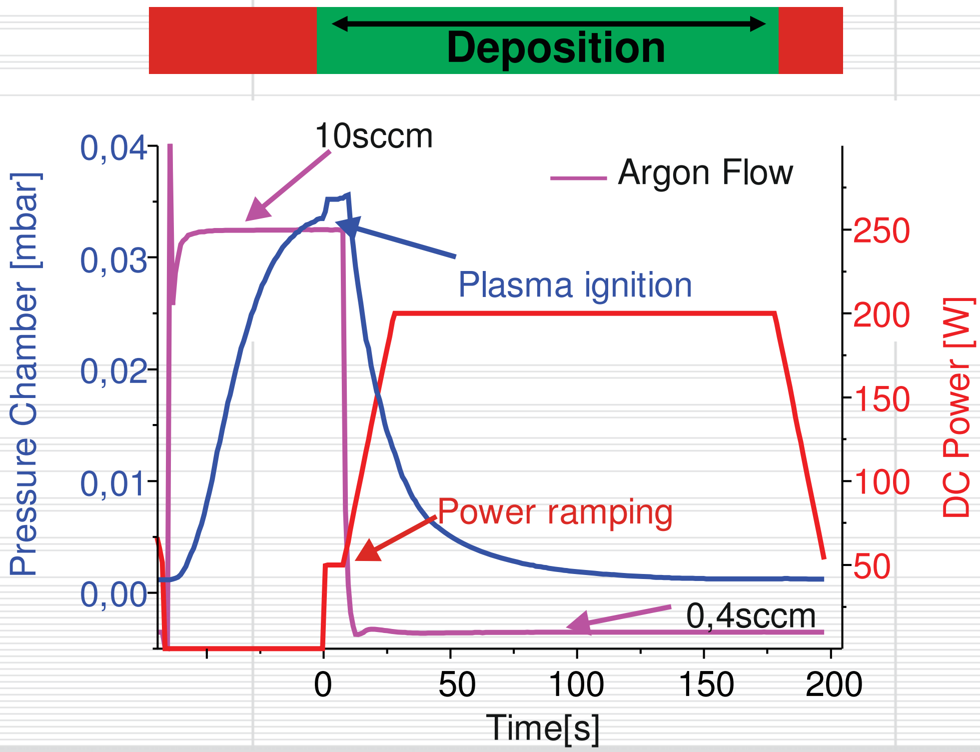
→ High pressure leads to dense growth

Possibility
to grow multilayer of one material!

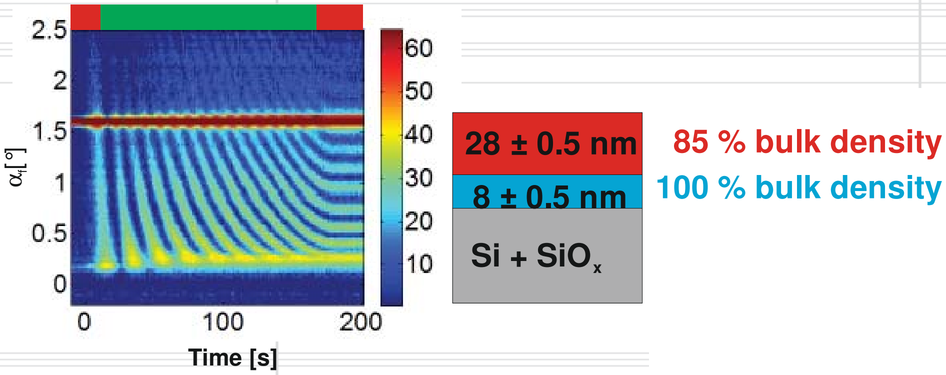
- **Idea:**
  - Grow bilayer by pressure variation (high/low pressure)
  - Thickness of Bilayer: 35 nm
  - Deposition Time: 180s @ DC Power: 200W

➡ Repeating this process: Achieve a multilayer structure

Multilayer by interrupted deposition: Sputtering conditions

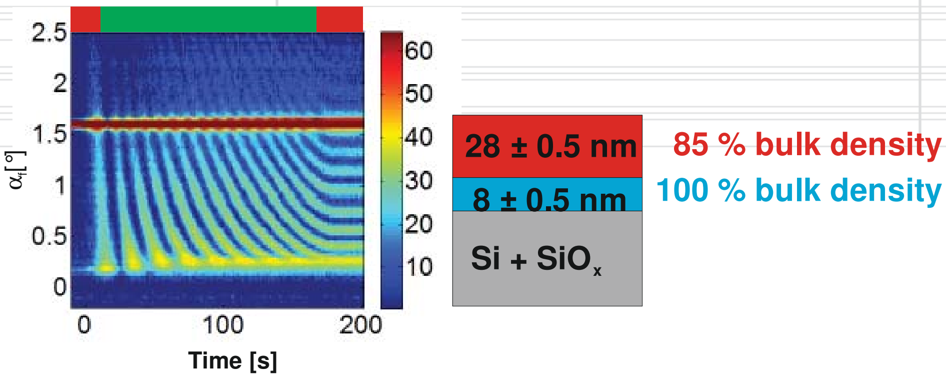


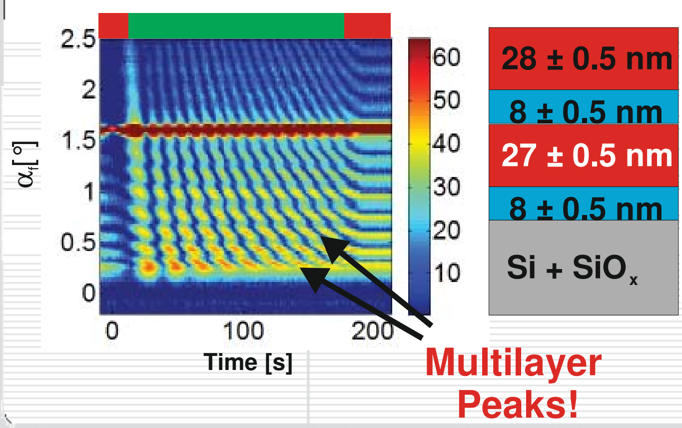
In situ Reflectivity: Period 1



- XRR map like “normal” deposition
- Detailed Analysis: Growth of bilayer system!

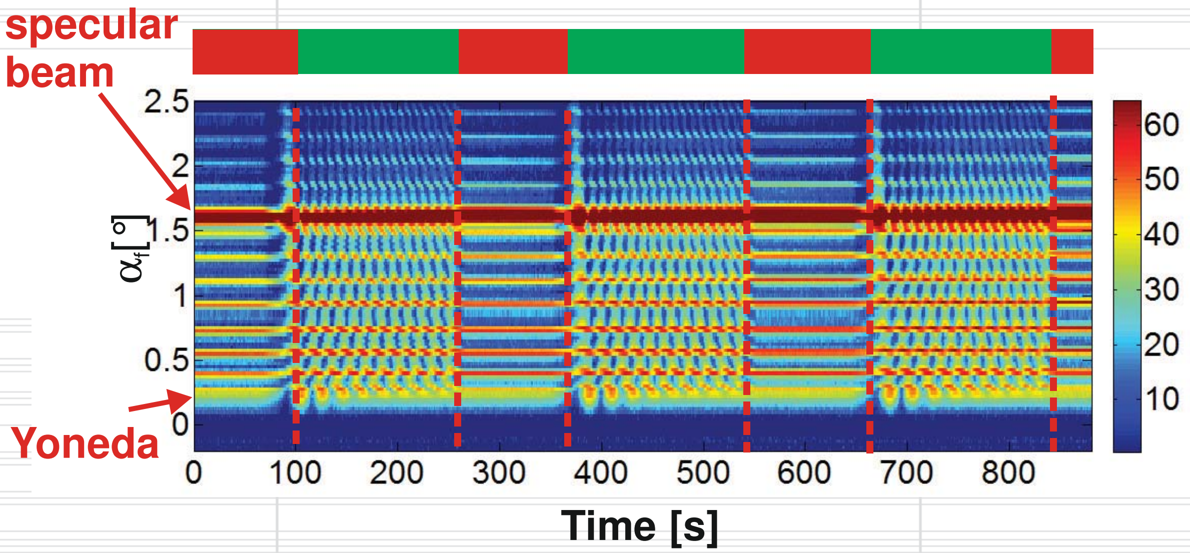
In situ Reflectivity: Period 1 and 2





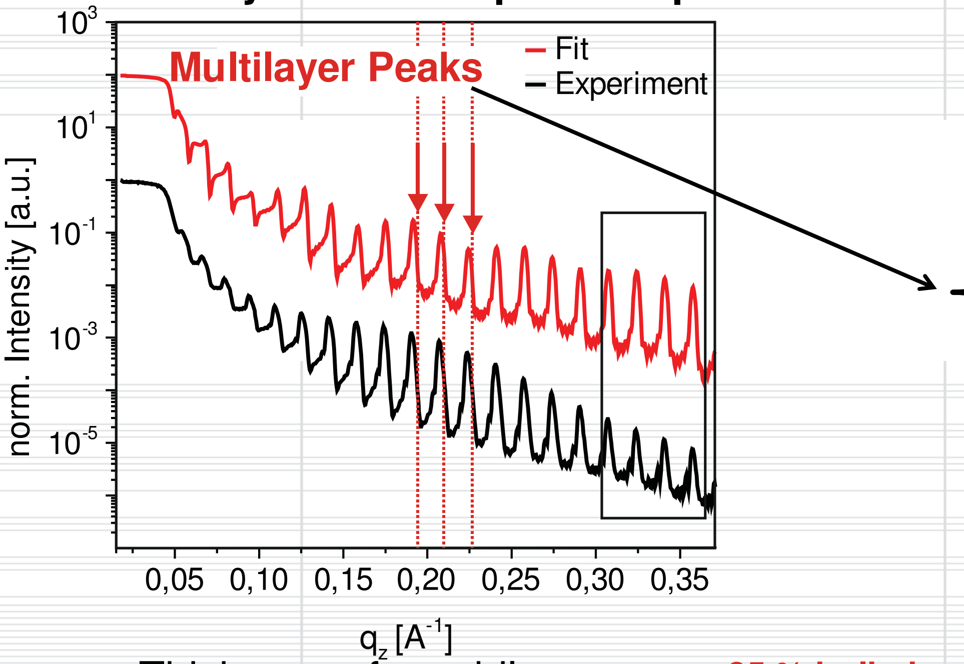
- XRR map shows additional peaks
- Slight reduction of thickness of “buried” bilayer

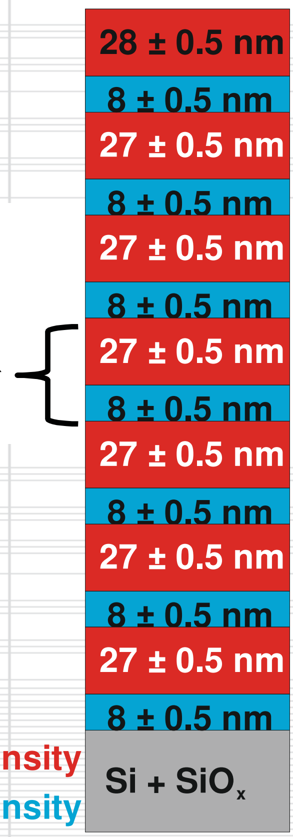
Multilayer by interrupted deposition: *in situ* Reflectivity Periods 5-7



Growth of periodic structure!

Multilayer by interrupted deposition: Reflectivity after 7th deposition period

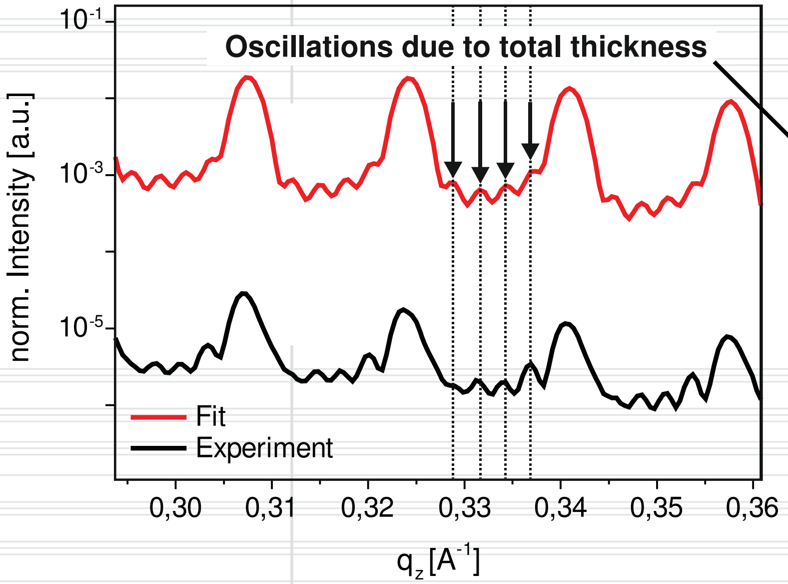


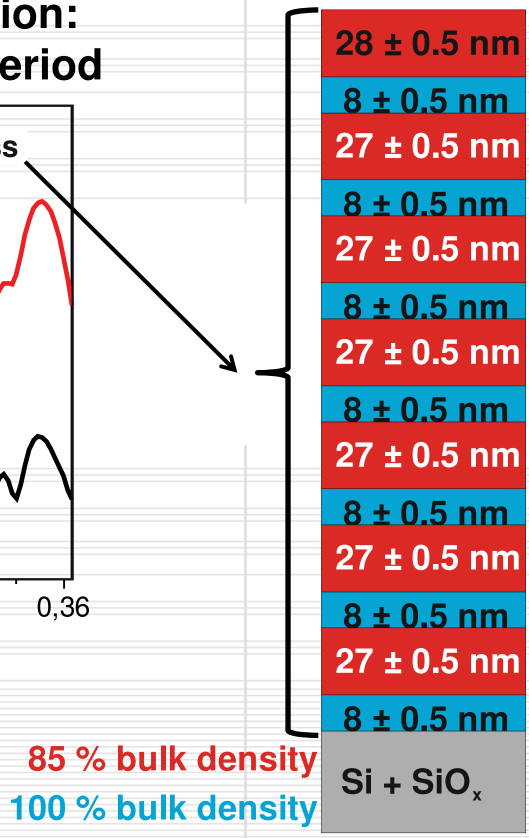


Thickness of one bilayer:
35±0.75 nm

85 % bulk density
100 % bulk density

Multilayer by interrupted deposition: Reflectivity after 7th deposition period





Total thickness
after deposition of 7 bilayers:
245±1 nm

Summary

- Successful growth of multilayer systems of one material by variation of the gas pressure

→ Periodical modulation of the microstructure of single material

- Monitoring of the multilayer formation by *in situ* X-ray reflectivity measurements

→ Non-destructive investigation of the average density of the microstructure during growth

→ Sensitive to temporal changes of buried layers during deposition

- Multilayer system growth by a simple deposition process

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Thank You for Your Attention !