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

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  - In situ XRR at different DC Power
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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

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**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: VC₁₋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

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**In situ X-Ray Reflectivity: “full angular range”**

- 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
- $\alpha_i = 1.6^\circ$

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

- Increase of DC Power by $\Delta P = 25\,\text{W}$ every 250s
- $\alpha_i = 1.6^\circ$:
  - Error due to changes in electron density <1%
  - Sensitive to deposition rate

$\text{Deposition Rate} \sim \text{DC Power}$

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

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!