In situ study of multilayers reflectivity upon heat treatment under synchrotron radiation

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Outline

Aim: Pretreatment of ESRF multilayer optics

- Multilayer description
- Multilayers upon annealing under synchrotron radiation
  - Experimental setup
  - Thermal annealing sequence
  - X-ray reflectivity measurements on ESRF beam line BM5
  - Theoretical model
- Summary and outlook
Multilayers Sample Description

- Distributed Electron Cyclotron Resonance (DECR) plasma sputtering

Top view

- Periodic multilayers: [Ru/B₄C] and [W/B₄C]
  - Characterized with x-ray laboratory reflectometer after deposition
  - Period thickness $\Lambda = 4.0$ nm and $\Gamma = 0.5$

- Stable at room temperature
Experimental setup
ESRF Bending Magnet Beamline BM5 20keV

X rays beam 20keV

Molybdenum
heating material

Alumina thermal
insulator

Vacuum
flange

Scintillator

Slits

2 θ

Sample in furnace

Sn Filters

Silicium
Pin diode

Sample holer

Kapton
Windows

Electric plug

Vacuum flange

Scans θ:2θ
- Full spectra 0-2.5 deg
- 3rd Bragg peak

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Thermal annealing sequence

Thermal cycle
Furnace BM5-ESRF

0:2θ Scans
Full spectra: 0-2.5º
3rd Bragg peak

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[Ru/B$_4$C]$_{70}$ Multilayer XRR full spectra at temperature stages

- Conservation of periodic structure
- Reversible and irreversible structural modifications
  - Period $\Lambda$
  - Gamma $\Gamma$

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[Ru/B₄C]₇₀ Multilayer XRR vs. temperature
[Ru/B₄C]₇₀ Multilayer
Period vs. temperature

1-Period growth:
- 3 % from RT up 550°C
- 1.2 % after annealing

2- Offset between results from 8keV and 20 keV
- 1.12 % after annealing from 8 keV
- Limitations of experimental setup
- Absolute values more accurate at 8 keV
[Ru/B₄C]₇₀ Multilayer Model structure of the period

- **Simulation 1**: 2 layers + 2 interfaces
  - \( \Lambda = t_A + t_B \)

- **Simulation 2**: 4 layers + 4 interfaces
  - \( \Lambda = t_{BA} + t_A + t_{AB} + t_B \)
[Ru/B\(_4\)C\(_7\)]\(_{70}\) Multilayer Simulations 1 and 2

Diffusion between materials

Periodic multilayer model with 2 additional interlayers in good agreement with experimental data

E = 20 keV

Theta [deg]

Log [Reflectivity]
[Ru/B4C]$_{70}$ Multilayer Density profile

![Graph showing density profile](image)

<table>
<thead>
<tr>
<th>Model</th>
<th>Density [g/cm$^3$]</th>
<th>t [nm]</th>
<th>$\sigma$ [nm]</th>
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<td>1</td>
<td>Ru</td>
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<td>2.10</td>
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<td>B$_4$C</td>
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<td>B$_4$C/Ru$_2$</td>
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<td>2</td>
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<tr>
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<td>B$_4$C</td>
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Summary

- Periodic multilayers studied during non destructive annealing tests
- Reversible and irreversible structural effects [W/B₄C] and [Ru/B₄C]
- Model with interlayer in the period definition in good agreement with Experimental data

- Stress in layer upon annealing and TEM measurements
- Other multilayer systems
- Pretreatment of ESRF multilayer optics
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