Focusing Reflectometry for In-situ Studies

Current trends and future perspectives in neutron reflectometry
08. - 09. 06. 2015, Lillestrøm, Norway
• Lithium Transport Through Thin Silicon Films

• high-intensity specular reflectometry

• the *Selene* guide

• Amor & *Selene*

• reduction of the Li transport data

• *Estia*
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• Estia
example for an \textit{in-situ} study using n reflectometry

\textbf{Lithium Transport Through Thin Silicon Films}

in cooperation with E. Hüger, F. Strauß and H. Schmidt, TU Clausthal

\textbf{technological motivation:}
\begin{itemize}
  \item Si layers can be used in Li batteries to prevent oxidation of the electrodes
  \item Si films can be used as electrodes in Li batteries
\end{itemize}

⇒ How fast does Li diffuse through thin amorphous Si films?
⇒ What is the solubility of Li in Si?
⇒ What is the influence of the Si:O:Li interface layer?

multilayer structure using the different densities of $^{6}\text{Li}$ and $^{7}\text{Li}$
**in-situ** furnace

- $T \in [25^\circ C, 500^\circ C]$
- $\dot{T} = 50 \text{ Ks}^{-1}$ for heating
- $\dot{T} = 12 \text{ Ks}^{-1}$ for cooling

**time-structure**

- interval
  - (measurements at RT in between annealing periods)
- **continuous measurement**

**instrument:**

Amor — a TOF reflectometer at PSI, Switzerland
reflectivity during annealing at 240°C
measurement time 1.5 min
reflectivity during annealing measurement time 1.5 min after various times

ml is chemically stable
Li contrast is vanishing
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specular reflectometry

\[ q_z = 4\pi \frac{\sin \theta}{\lambda} \]

\[ k \propto \frac{1}{\lambda} \]
angle-dispersive reflectometry

\[ \lambda \]

\[ \theta \]
high-intensity reflectometry

angle-dispersive reflectometry

energy-dispersive reflectometry
high-intensity reflectometry

angle-dispersive reflectometry

ergy-dispersive reflectometry

angle- and energy-dispersive reflectometry
reflective focusing optics

parabolic
parallel to convergent

elliptic
divergent to convergent
reflective focusing optics

elliptic
reflective focusing optics

elliptic
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point-to-point focusing

with

2 subsequent elliptical reflectors

for

horizontal and vertical direction

Selene picture:
ceiling painting in the Ny Carlsberg Glyptotek, København
light-field-diaphragm
countrol of footprint

uncorrected, inverted image

aperture
defines divergence

image
sample
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prototype guide on Amor@PSI

slit = virtual source
polariser

1\textsuperscript{st} segment
spin flipper

2\textsuperscript{nd} segment
sample stage

flight tube
detector

optical bench, 8 m long
prototype guide on Amor@PSI

- total length = 4 m
- max spot size ≈ 2 × 2 mm²
- divergence ≈ 1.8° × 1.8°
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reduction of Li-transport data

raw data

$\theta$ deg

$log_{10} I(\lambda, \theta)$

$^{6}\text{Li}/\text{Si}/^{7}\text{Li}/\text{Si}$ multilayer

counting time 1.5 min
reduction of Li-transport data

$\log_{10} I(\lambda, \theta)$

$\log_{10} I^0(\lambda, \theta)$

$R(\lambda, \theta)$

$\log_{10} R(\lambda, \theta)$

$^6\text{Li}/\text{Si}^7\text{Li}/\text{Si}$ multilayer

$\theta$ deg

$\theta$ deg

$\theta$ deg

$m = 5$ supermirror

quotient

counting time 1.5 min

$m = 5$ supermirror

quotient
reduction of Li-transport data

$^6\text{Li}/\text{Si}/^7\text{Li}/\text{Si}$ multilayer annealed at $240^\circ\text{C}$
continuously measured with 1.5 min resolution

$\theta$ deg

$R(\lambda, \theta)$
preliminary results

• 10 nm amorphous Si

permeability of Li in Si \( P = (1.28 \pm 0.25) \cdot 10^{-16} \text{cm}^2\text{s}^{-1} \)

• \( P \) and \( D \) depend strongly on Si film thickness

\[
\begin{array}{l|cccccc}
\text{\( d_{\text{Si}} \)/nm} & 4 & 7 & 9 & 11 & 15 & 20 \\
\hline
\text{full intermixing after \( t \)/s} & <30 & 180 & 4800 & 9000 & 54000 & >90000 \\
\end{array}
\]

• The Li-O-Si interface has no significant influence

  ○ data analysis in progress

  ○ further experiments are planned
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Estia  
*a polarised focusing reflectometer*

*for small samples*

for the investigation of chemical and magnetic depth-profiles near surfaces and of lateral correlations and structures

- functional devices: *spin-valves, spintronics*
- diffusion processes: *Li batteries, corrosion protection*
- multifunctional materials: *interface-coupled electric and magnetic properties*
- towards real materials: *raster-scanning of bent, faceted or multi-domain surfaces*

**loupe-like neutron guide**
- new operation modes
- low background
- high flexibility
  - decoupling of beam size and divergence

**pushing the limits**
by 2 to 3 orders of magnitude for
- tiny samples (< 1 mm²)
- fast measurements (< 0.1 sec)
- in-situ studies during growth or manipulation
TOF reflectometer for the ESS

- horizontal scattering plane
- sample size $< 10 \times 50$ mm$^2$
- divergence $1.5^\circ \times 1.5^\circ$
- $\lambda \in [4, 10]$ Å
**comparison** for high-intensity mode to *Selene* prototype on Amor

<table>
<thead>
<tr>
<th>factor</th>
<th>source flux</th>
<th>guide transmission</th>
<th>footprint</th>
</tr>
</thead>
<tbody>
<tr>
<td>ESS / SINQ</td>
<td>150</td>
<td>Selene / Amor + Selene</td>
<td>4</td>
</tr>
<tr>
<td>4</td>
<td>10</td>
<td>(sample size 1 cm$^2$)</td>
<td>10</td>
</tr>
<tr>
<td>6 000</td>
<td>in total</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

⇒ $q_z$-range 0.005 Å$^{-1}$ to 0.05 Å$^{-1}$ in one shot (0.07 s)

alternative use of flux:
- smaller samples ($< 1$ mm$^2$)
- higher $q_z$ (smaller structures)
- off-specular scattering (lateral structures)
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Thank you!