Thermal damage in Mo/Si based multilayers for short-wavelength FELs

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Outline

- Introduction: multilayers for FELs
- Diffusion in multilayers at the sub-picometer scale
- Diffusion through barriers
- Diffusion in single shot FEL experiments
- Summary, outlook
Multilayer optics for FELs

Main advantages...

- No need to stay below critical angle
  - Freedom of selecting periodicity => wavelength / angle
- Wavelength selectivity ~ FEL radiation bandwidth (~1%)
  - Possibility of further monochromatization
- Polarization sensitive
- Can be applied to (focussing) optics

... and challenges

- Photoinduced surface contamination (vac. env.)
  - Carbon deposition
  - Oxidation
- Stress (e.g. membranes / freestanding coatings)
- Thermal damage
  - Apart from “traditional” damage such as thermal deformation...
  - ...also thermal damage on coating should be taken into account!
What is “Thermal damage”?  

Hamburg Weather prediction  : ~22°C, UV Index: 6 (High)  

“Sunburn is caused by UV radiation, either from the sun or from artificial sources … “  


So, what about “sunburn” from FELs? (and what about protection?)  

“The best treatment for sunburn is prevention”  

FEL:  
- Distance to source  
- Grazing incidence  
...  
“what else”?  

What are the exact damage mechanisms?
Death of a ML : Single shot damage @ FLASH

Outside damaged area

Inside damaged area

Change in layered structure is observed (contrast, period, …) : What happened?
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Mo/Si based multilayers for 13.5 nm

7 nm period, normal incidence optics:
- Basic ML structure well characterized.
- “Relatively” low power loads ...
  ... but very tight stability requirements!

⇒ Requires accurate prediction of thermal damage over full lifetime
Thermal damage: Phase transitions

- As-depo structure due to kinetics
- High-T structure due to thermodynamics
Moderate temperatures: Interface diffusion

Diffusion studies by cycles of 48h annealing and structure analysis

- Diffusion leads to additional MoSi$_2$ formation at interfaces

Monitoring diffusion

Experiment: Thermal treatment under protective atmosphere, *in-situ* structure analysis (GIXR, XRD)

- *In-situ* structure analysis enables monitoring of temperature induced structural changes on a *Picometer lengthscale.*
Diffusion mechanism: Temperature scaling

Growth law: Diffusion: $(\Delta \text{interface})^2 \sim Dt$ for diffusion controlled interface growth

Data well described by parabolic growth law.

Arrhenius-type behaviour, activation energy 0.5 eV

In-situ structure analysis allows accurate (<1 pm) characterization of diffusion processes.

Scaling laws allow prediction of thermal damage at “any” time and temperature.
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Diffusion through $B_4C$

Goal: study of single (ex: Si-on-Mo) interface.

High-sensitivity in-situ LEIS @ 500°C:

- Change in the Mo concentration profile reveals Mo diffusion through $B_4C$ barrier.

Fick’s law: Concentration profile yields $D \cdot t$
Acceleration of diffusion

Fick’s law: Concentration profile yields $D \cdot t$

What happens from (1) to (2)?
Accelerated diffusion due to chemical / structural changes?

1. Initial diffusion stage
2. Faster diffusion stage
3. Saturation (no more Mo or Si)

- What happens from (1) to (2)?
  - Accelerated diffusion due to chemical / structural changes?

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Chemical analysis of diffusion stages

XPS results suggest $B_4C$ decomposes long before diffusion accelerates.

No evidence of diffusion speedup due to large chemical composition change.
Structural analysis of diffusion stages

(a) Si + SiO₂
(b) Si + SiO₂
MoSi₂
Mo

(c) Si + SiO₂
MoSi₂
Mo

(d) SiO₂
MoSi₂
Mo

Si wafer 10 nm

As deposited  Before $D$ enhancement  After $D$ enhancement  After full annealing
Structural analysis of diffusion stages

TEM just before diffusion speed-up

TEM just after diffusion speed-up

Crystallization → Grain boundary diffusion → Accelerated diffusion
Stage (1) diffusion constants

Barriers strongly reduce diffusion constants!
(unchanged activation energy suggests same damage mechanism: MoSi formation “through barrier” )
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FLASH beam
\( \lambda = 13.5 \) nm, 10 fs, p-pol, 0.01 – 1 \( \mu \)J/pulse
Microscopy / AFM damage studies

- Damage threshold 45 mJ/cm²

Crater observed:
Sputtering observed above damage threshold?
TEM damage studies

Crater depth = \[ \text{damaged periods} \times \text{period change after phase transformation} \]

=> “traditional” diffusion induced structural changes, but on such short timescales?
Pump-probe damage studies

FLASH beam
\( \lambda = 13.5 \text{ nm, 10 fs} \)
0.01 – 1 \( \mu \text{J/pulse} \)

\( \text{XUV pump – optical probe:} \)

40% reflectance increase
Attributed to change in Si optical constant
(solid \( \rightarrow \) melt)

=> This would explain fast diffusion and observed phase transformation
Summary and outlook

- Large knowledge base on Mo/Si coatings is available from development for EUVL
- GIXR reveals diffusion limited growth of MoSi$_2$ interfaces (300K-600K)
  - MoSi$_2$ interface formation has $E_A=0.5$eV
  - Phase transformation to energetically favorable structure for $T > 600K$.
- LEIS reveals multiple diffusion stages in Mo/B4C barrier/Si at $T=770K$, with diffusion acceleration triggered by crystallization at the interfaces.
  - Diffusion barriers reduce diffusion constants, keeping the same $E_A$
- Damage of Mo/Si with single-shot high-intensity FEL radiation is similar to damage due to thermal annealing: atomic diffusion and phase transitions leading to compaction

It is clear that lessons learned in designing ML’s for “traditional applications” will help greatly in designing ML coatings for FEL applications.
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Thanks for your attention!