Contamination and Degradation Testing

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Some Terms

- **Ageing:**
  With time or after use it is different than before
  (usually: different = worse)

- **Contamination:**
  Initially it was clean, but now it’s dirty
  (removable! (?)

- **Degradation:**
  After usage it is damaged now
  (can not be recovered)
Outline

- Dirt
- Dirt and Radiation
  - optical surfaces
  - photodetectors
- \((D + R) - D = R\)
- Prospects
Synchrotron radiation available at PTB

Berlin-Adlershof

PTB monochromator beamlines:

BESSY

MLS

Berlin-Adlershof
What you (usually) want from PTB

Calibration & characterisation of

**optical components:**
- filters
- spectral transmission
- mirrors
- spectral reflectance
- gratings
- diffraction efficiency

**detectors:**
- photodiodes
- photoemissive/photocathodes
- photoconductive
- imagers (CCD, APS)
- spectral responsivity

**spectrometers:**
- dispersive type (grating)
- filter radiometer type
- ...
- spectral responsivity
- irradiance sensitivity
- ...

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What you (usually) get from PTB

- Measurement value + uncertainty
  - Uncertainty budget is for measurement conditions at PTB calibration
  - Does **not** include different measurement conditions and ageing

<table>
<thead>
<tr>
<th>&quot;true&quot; value ?</th>
<th>vs.</th>
<th>&quot;real value&quot; ?</th>
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<tbody>
<tr>
<td>Ideal conditions</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Determination of material parameters</td>
<td></td>
<td></td>
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<tr>
<td>Comparison to theoretical models</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Real conditions</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Determination of performance parameters</td>
<td></td>
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<td>Comparison to other measurements</td>
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difficult to realise (but PTB attempts)

Conditions during calibration the same as in your apparatus?

**How big is the discrepancy?**

Our experience: typically < 5% without (!) aging
How to approach the “true“ value

Sample with initial adsorbates from:
- processing
- transport
- pumpdown

Cleaned sample for measurement:
- reflectance
- transmission
- efficiency

surface cleaning:
- in-air UV-ozone cleaning
- in-vacuum heating / bakeout
- in-vacuum plasma cleaning (RF, DC; Ar, O₂, H₂)

Sample with initial adsorbates from:
"Simple" example: quartz transmission standard

sample 1 (uncleaned) at air

Transmittance vs. Wavelength (nm)

- Transmission (y-axis)
- Wavelength (nm) (x-axis)

Transmittance values for sample 1 at different wavelengths.

- 0.86
- 0.88
- 0.90
- 0.92
- 0.94

Sample 1 (uncleaned) comparison at air.
"Simple" example: quartz transmission standard

Sample 1 (uncleaned) at air
Sample 2 (UV cleaned) at air

Difference cleaned/uncleaned: up to 4 %!
“Simple” example: quartz transmission standard

sample 1 (uncleaned) at air
sample 1 (uncleaned) at vacuum
sample 2 (UV cleaned) at air

uncleaned sample (1):
NO difference air - vacuum

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“Simple” example: quartz transmission standard

sample 1 (uncleaned) at air
sample 1 (uncleaned) at vacuum
sample 2 (UV cleaned) at air
sample 2 (UV cleaned) at vacuum

uncleaned sample (1):
NO difference air - vacuum
UV cleaned sample (2):
up to 2 % difference!
Change by irradiation

- mirror
- filter
- grating
- detector
- surface adsorbates
  - residual gas molecules
- HE particles
- surface contamination
- interface change/damage
- bulk damage
Typical measurement sequence

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<table>
<thead>
<tr>
<th>sample prep.</th>
<th>pre meas.</th>
<th>Sample irrad.</th>
<th>after meas.</th>
<th>further analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>&quot;elsewhere&quot;</td>
<td>reflectometer @PTB</td>
<td>irrad./contam. chamber @PTB or &quot;elsewhere&quot;</td>
<td>reflectometer @PTB</td>
<td>&quot;elsewhere&quot;</td>
</tr>
</tbody>
</table>
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Problems:
- ambient air exposure during transport
- inadequate conditions e.g. during irradiation
The EUVL experience (from 2001 on)

... on Mo/Si multilayers

photons
13 nm
(92 eV)

Oxidation

contamination layer: $C_xH_y$, $H_2O$,...
passivation layer, cap layer, oxide

- Si + $H_2O$ + slow electron = oxidation enhancement
- Oxidation is irreversible
- Mitigation of oxidation in the presence of EtOH
- Formation of self-terminating C-layer

⇒ Initial capping with C-layer prevents oxidation

but: hydrocarbons will let the C-layer grow

The EUVL experience (from 2001 on)

Carbon growth
(similar on metal surfaces)

- Carbon layer growth depends on:
  - absolute amount of hydrocarbons
  - type (mass number) of hydrocarbons
  - energy of photons
  - number of photons (irradiation)
  - (surface material)

- can be removed and initial values can be restored

Further experiences (in VUV)

Carbon growth

- photons
  - 5 eV to
    - 10 eV

contamination layer
passivation layer, oxide

Almost no carbon growth with low (< 10 eV) photon energies, but:

- self-terminating (?) condensation in vacuum
- depending on surface (material, quality, cleanliness)
- few % transmission losses already by very thin (mono?-) layers of condensates
- Very fast (1 min during pumping)
- In-situ removal partially possible (heating,...)
Why wavelengths matters...

- half-value layer thickness of representative materials

- Very thin layers can significantly contribute
- absorbed energy is deposited in surface layer
Example: Pt mirror

![Graph showing the reflectance of a Pt mirror across different wavelengths.](image)
Example: Pt mirror

\[ \text{new} \]
\[ \text{irradiated} = \text{contaminated} \]
Example: Pt mirror

- New irradiated = contaminated
- Thick carbon coating (reference sample)

- Thin carbon different than thick carbon
- Depending on structure
Example: Pt mirror

- cleaning possible
- but not perfect

DC plasma cleaned (O₂)
Example: C-coated Si Mirror

- exposed to GW EUV pulses (DESY FEL)
- reconstruction of C configuration observed ("needles")
- massive plasma cleaning for re-coating
- non-uniform residuals remaining
- multiple analysis performed, here: VUV reflectometry

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Contamination and Degradation Testing
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**Contamination and Degradation Testing**
Example: C-coated Si Mirror

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EUV/VUV reflectometry:
- modeling (layer material and thickness) is possible if opt. constants are known
- surface sensitive (few nm)
- no buried layer analysis
- no speciation or chemical structure analysis

Further analysis required to investigate specific processes!
Detectors

„typical“ Si photodiode

- surface contamination
- interface change/damage
- bulk damage

change of spectral responsivity
Primary effects in radiation detection

- **ideal case**
  - Surface reflection
  - (photo)electron loss
  - dead layer absorption
  - recombination

- **loss mechanisms**
  - additional absorption
  - local effects!
  - photochemistry (carbon growth)

- **contamination**
  - surface charge
  - interface oxidation
  - trap state creation (CCE change)

- **unavoidable** vs. **removable**
  - unavoidable (cannot be minimized by detector design)
  - removable

- **ageing & damage**
  - can be minimized by detector design
Irradiation Ageing

- signal degradation
- includes possible carbon growth
- (almost) identical conditions, so carbon should be the same for all.

121 nm

rel. change in responsivity

radiant exposure /μJ cm⁻²
Example for ageing: Si photodiode

AXUV-100G photodiode 10 mm x 10 mm

Relative spectral responsivity map:

- **SiO₂ transparent**
  - weak effect of 193 nm irradiation

- **SiO₂ absorbing**
  - massive effect of 121 nm irradiation
  - irreversible damage of the SiO₂ passivation layer cannot be removed by surface cleaning
Example for ageing: Si photodiode

**AXUV-100G photodiode** 10 mm x 10 mm

change of spectral responsivity with wavelength after 121 nm irradiation

(measured in irradiated spot)

- Surface charge effect (photoelectron emission)
- Damage
- SiO₂ transmission edge
Outlook: Surface analysis to understand processes

Electron spectroscopy (XPS/UPS):
chemical speciation possible
learn about surface processes

Further Methods at PTB in the (soft-)X-ray range:
spectroscopic ellipsometry, XRF, XRR, NEXAFS

Example: XPS spectra of different Si oxidation states

Si 2p XPS

- Si(Bulk) at $h\nu = 280$ eV (bulk sensitive)
- Si-SiO$_2$-Interface (Suboxide) at $h\nu = 130$ eV (surface sensitive)

Change of probing photon energy:
- Element sensitivity (cross sections)
- Probing depths (mean free path)

E. Darlatt
Conclusions

1. Contamination can lead to deviating calibration results even at low irradiation levels.

2. *In situ* sample cleaning can (at least partially) remove contamination effect, *ex situ* sample cleaning might even enhance (later) contamination!

3. Irradiation (UV or shorter wavelength) can result in:
   - carbon growth
   - interlayer change (oxidation, ...)
   - structural (bulk) damage

4. Differentiation between contamination and (other) degradation is not always possible.

5. Prediction of effects (and inclusion into the uncertainty budget) is difficult since:
   - environment parameters for contamination are difficult to control
   - processes are still not well understood (⇒ *further research work needed*).

6. ▪ avoid ambient air (humidity, hydrocarbons)
   - if it is unavoidable, define reproducible cleaning and measuring procedures