Physikalisch-Technische Bundesanstalt
Braunschweig und Berlin
Nationales Metrologieinstitut

Contamination and Degradation Testing

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Some Terms

- **Ageing :**

  With time or after use it is different than before

  (ususally: 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

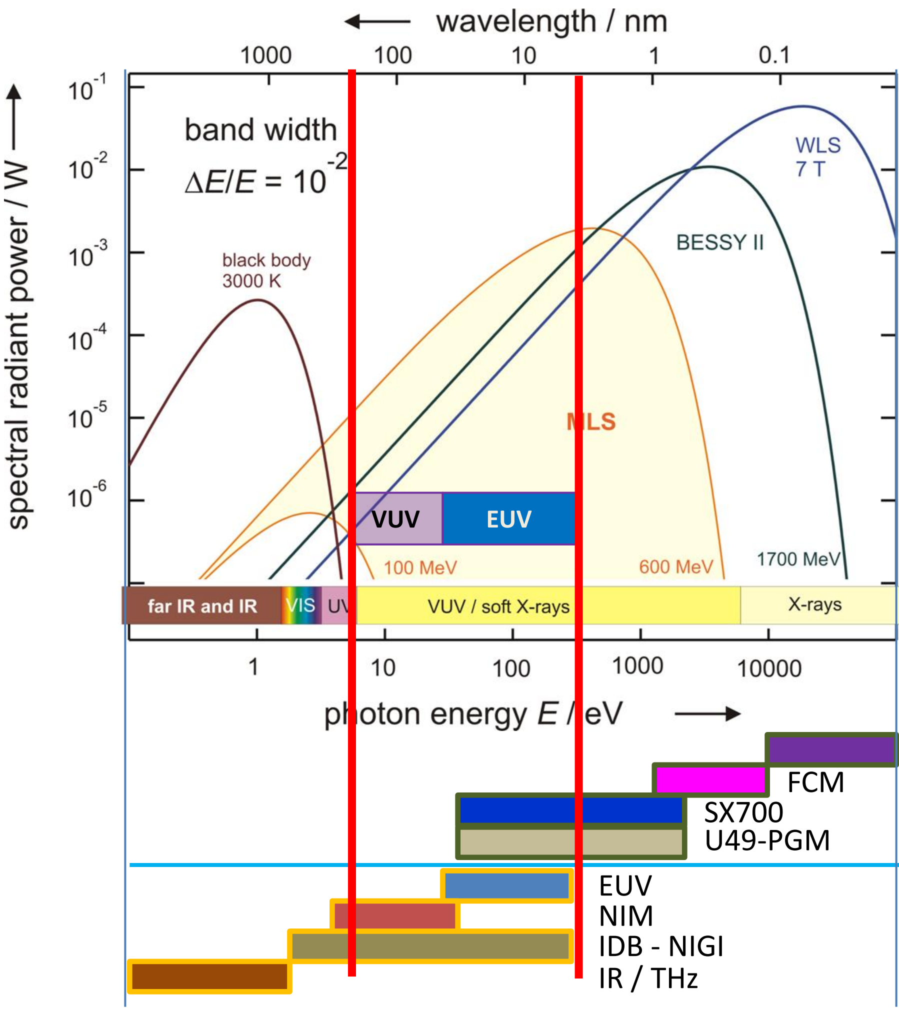
- Dirt =: D
- Dirt and Radiation =: D+R

  optical surfaces

  photodetectors

- (D + R) – D = R ??
- Prospects

Synchrotron radiation available at PTB



Berlin-Adlershof

PTB monochromator beamlines:

WLS-DCM
FCM
SX700
U49-PGM

BESSY (Helmholtz Zentrum Berlin)

EUV
NIM
IDB - NIGI
IR / THz

MLS (PTB)

What you (ususally) 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
...

What you (ususally) get from PTB

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

“true“ value ? **vs.** **“real value“ ?**

- ideal conditions
- determination of material parameters
- comparison to theoretical models

difficult to realise (but PTB attempts)

practical use??

- real conditions
- determination of performance parameters
- comparison to other measurements

Conditions during calibration the same as in your apparatus ?

How big is the discrepancy ?

our experience: typically < 5 % without (!) aging

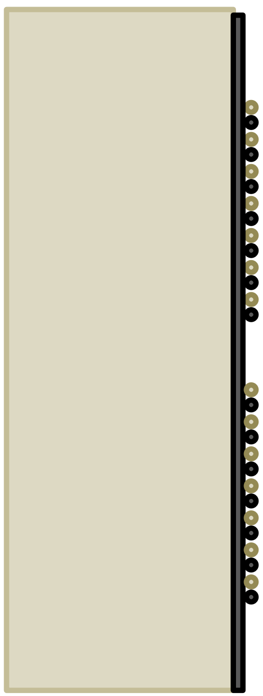
How to approach the “true“ value

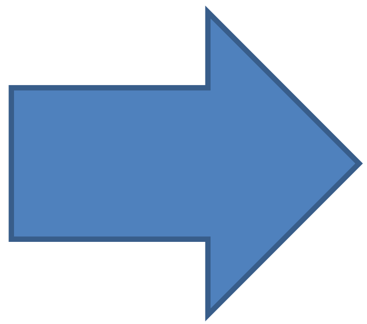
Sample with initial adsorbates from:

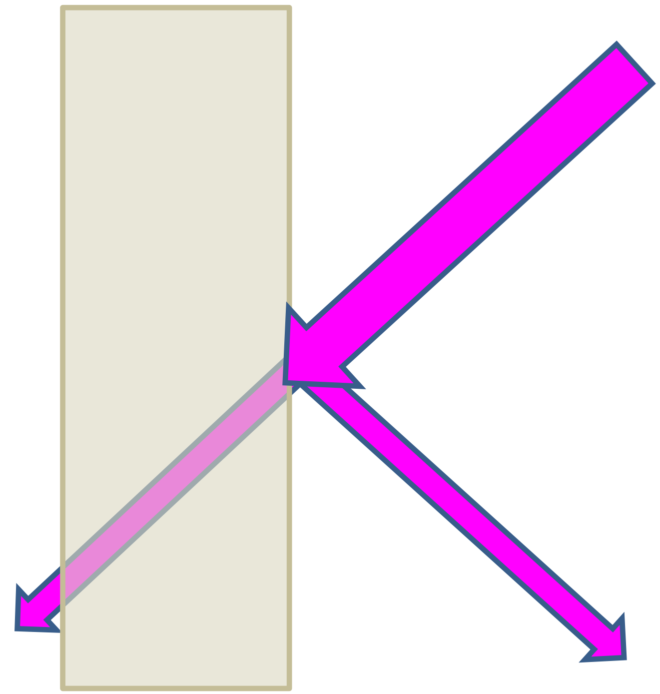
- processing
- transport
- pumpdown

Cleaned sample for measurement

- reflectance
- transmission
- efficiency



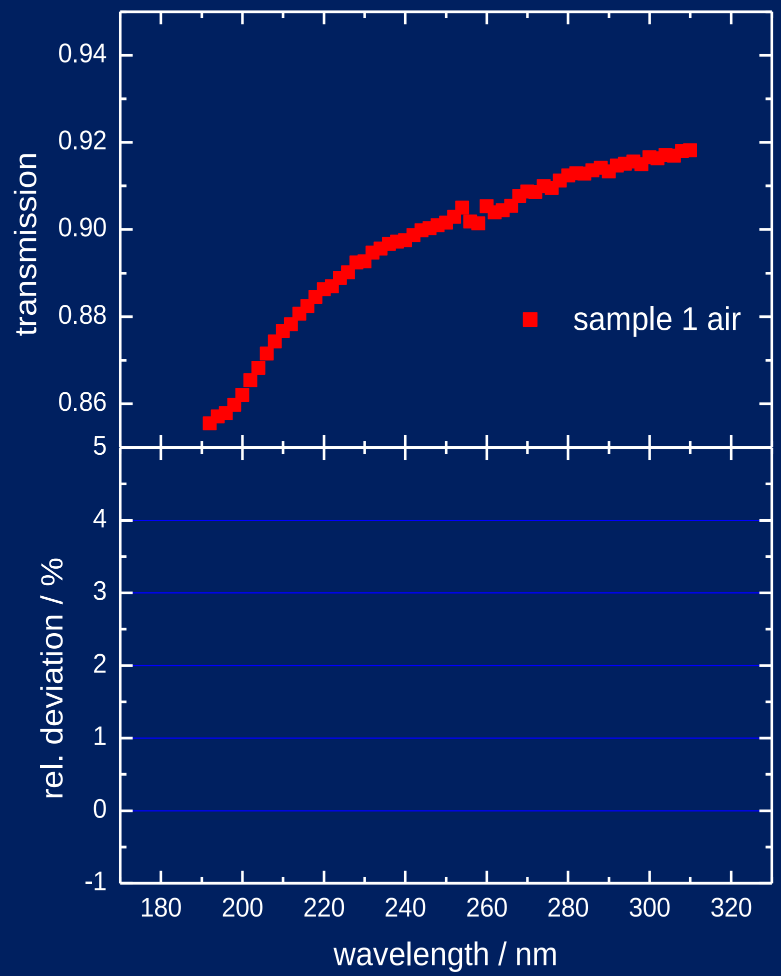




surface cleaning:

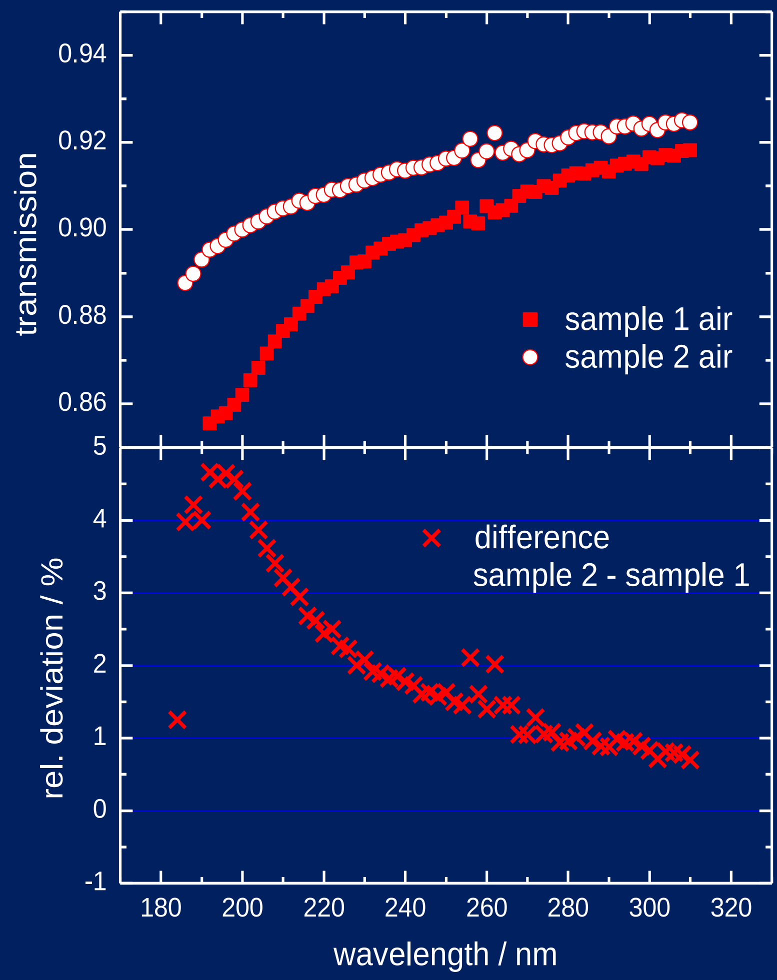
- in-air UV-ozone cleaning
- in-vacuum heating / bakeout
- in-vacuum plasma cleaning (RF, DC; Ar, O_2, H_2)

“Simple“ example: quartz transmission standard



sample 1 (uncleaned) at air

“Simple“ example: quartz transmission standard

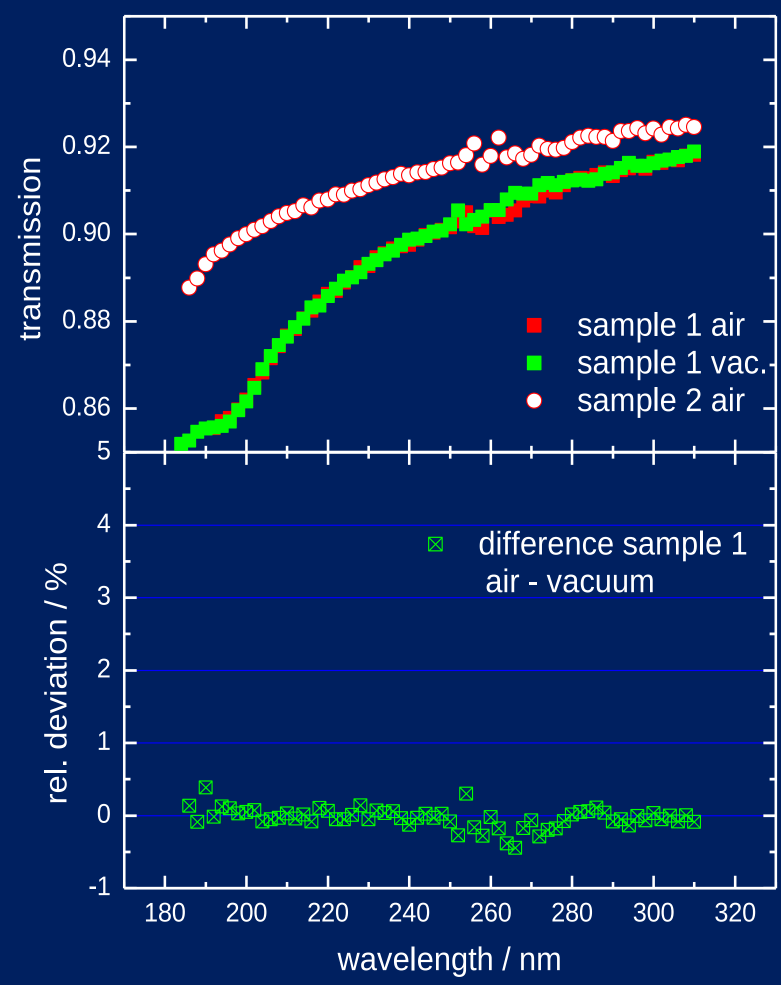


sample 1 (uncleaned) at air

sample 2 (UV cleaned) at air

difference cleaned/uncleaned:
up to 4 % !

“Simple“ example: quartz transmission standard



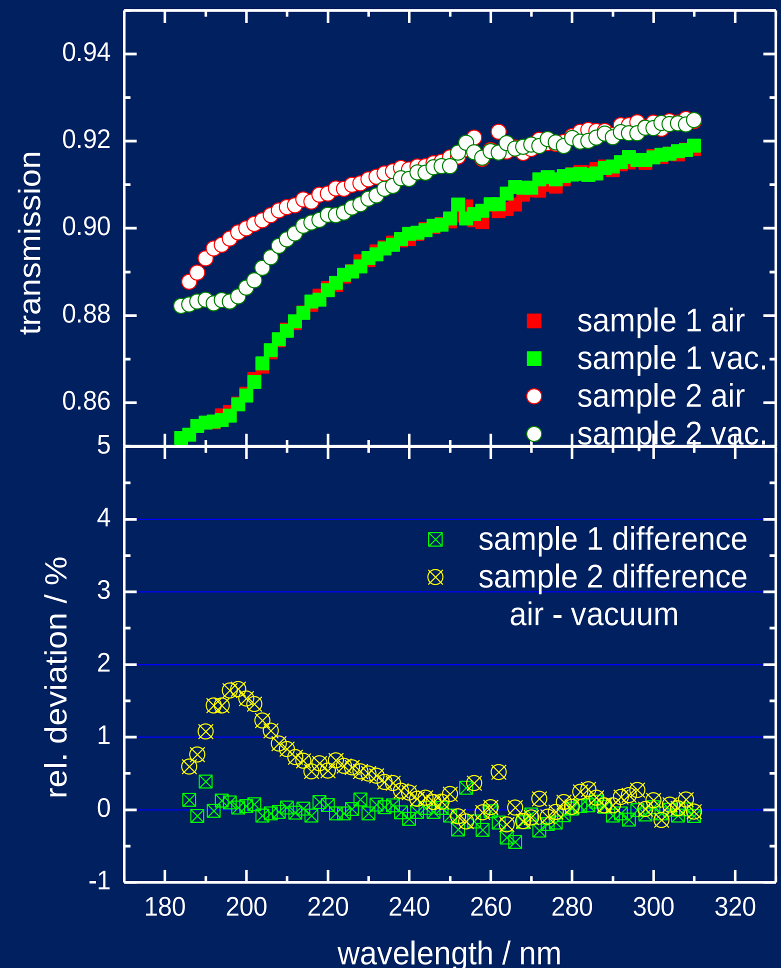
sample 1 (uncleaned) at air

sample 1 (uncleaned) at vacuum

sample 2 (UV cleaned) at air

uncleaned sample (1):
NO difference air - vacuum

"Simple" example: quartz transmission standard



sample 1 (uncleaned) at air

sample 1 (uncleaned) at vacuum

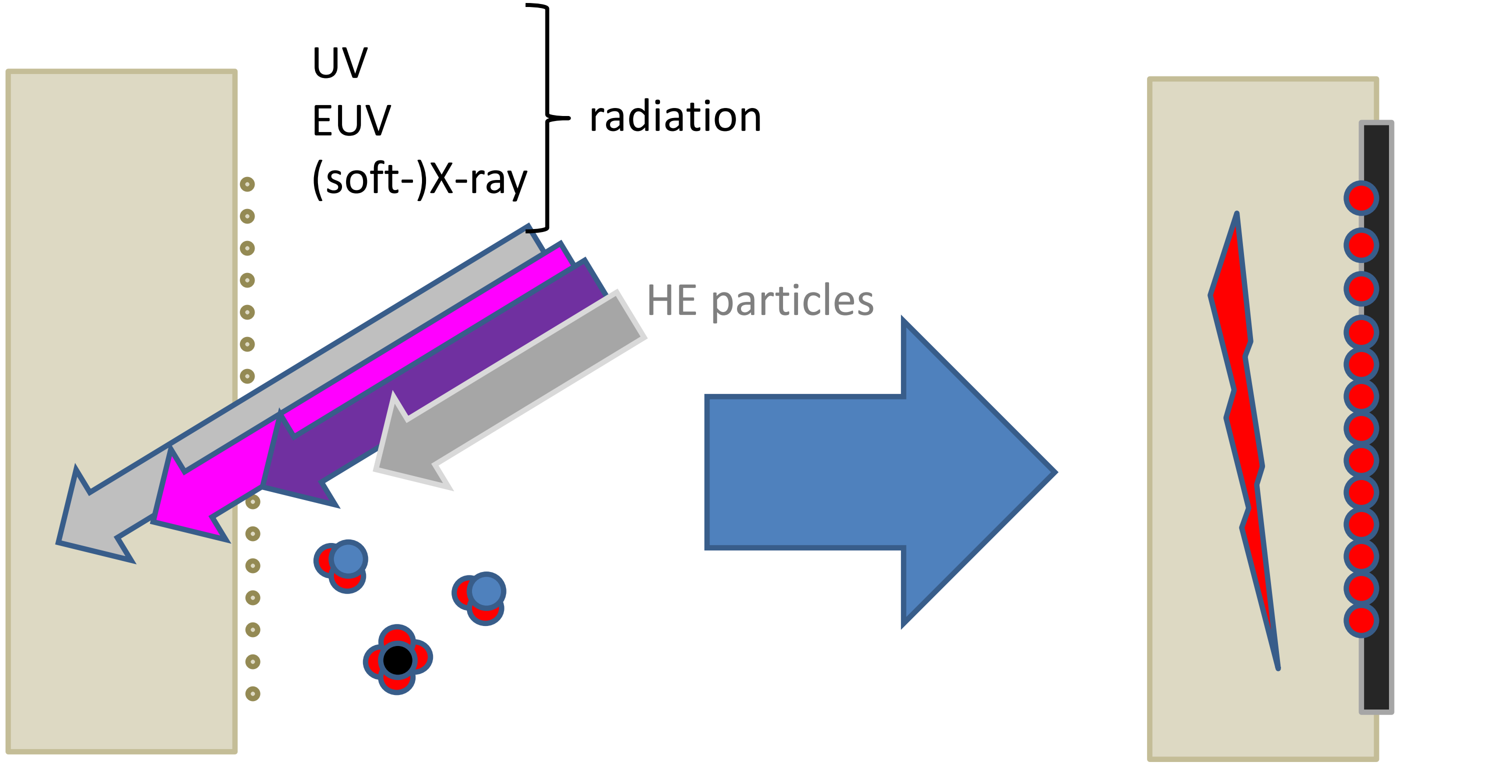
sample 2 (UV cleaned) at air

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

- surface contamination
- interface change/ damage
- bulk damage

Typical measurement sequence

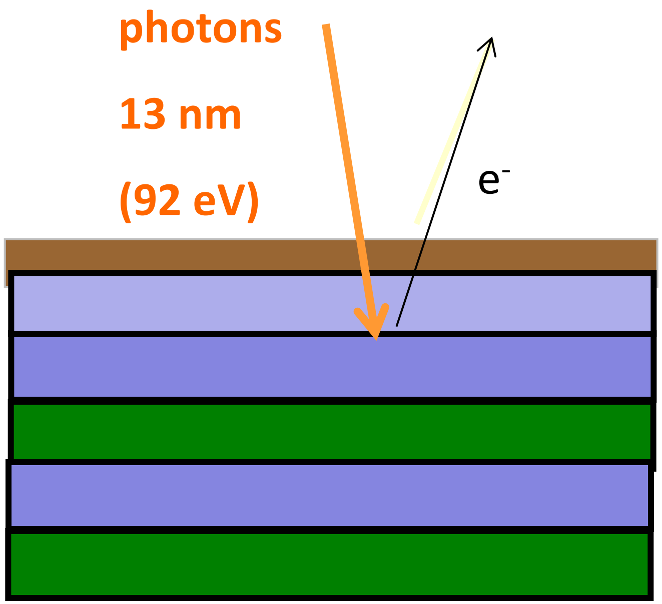
sample prep. → pre meas. → Sample irrad. → after meas. → further analysis

sample prep.	pre meas.	Sample irrad.	after meas.	further analysis
"elsewhere"	reflectometer @PTB	irrad./contam. chamber @PTB or "elsewhere"	reflectometer @PTB	"elsewhere"
PTB possibilities limited		PTB possibilities limited		PTB possibilities limited

Problems:

- **ambient air exposure during transport**
- **inadequate conditions e.g. during irradiation**

The EUVL experience (from 2001 on)



Oxidation

… on Mo/Si multilayers

contamination layer: C_xH_x, 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

Koster et al., Microelectric Engineering 61-62 (2002), 65-76

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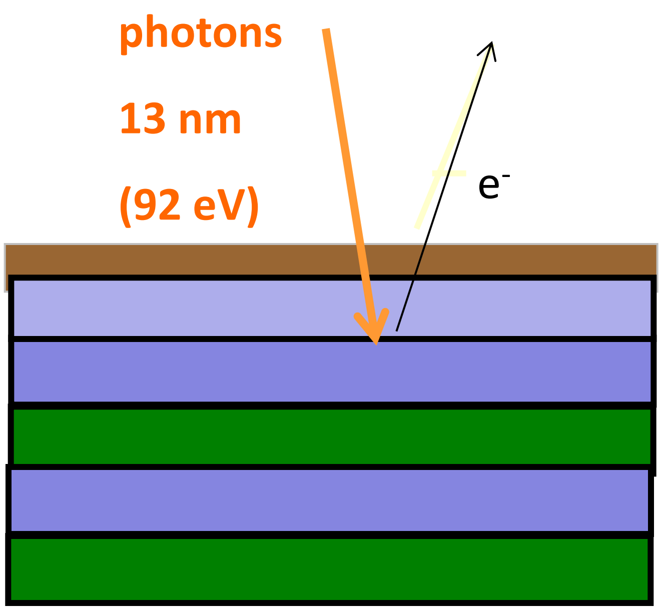


Carbon growth

(similar on metal surfaces)

contamination layer
passivation layer, oxide

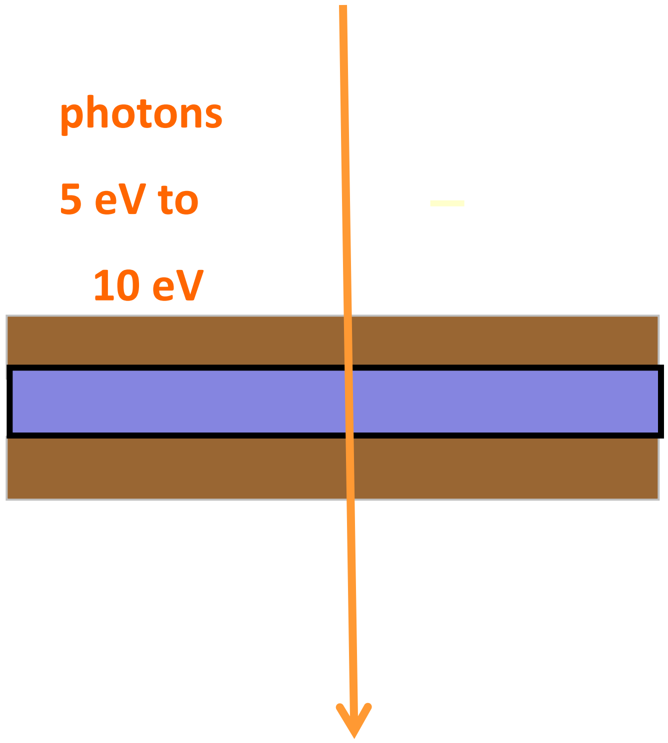
carbon layer growth depends on

- 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

Koster et al., Microelectric Engineering 61-62 (2002), 65-76

Further experiences (in VUV)



Carbon growth

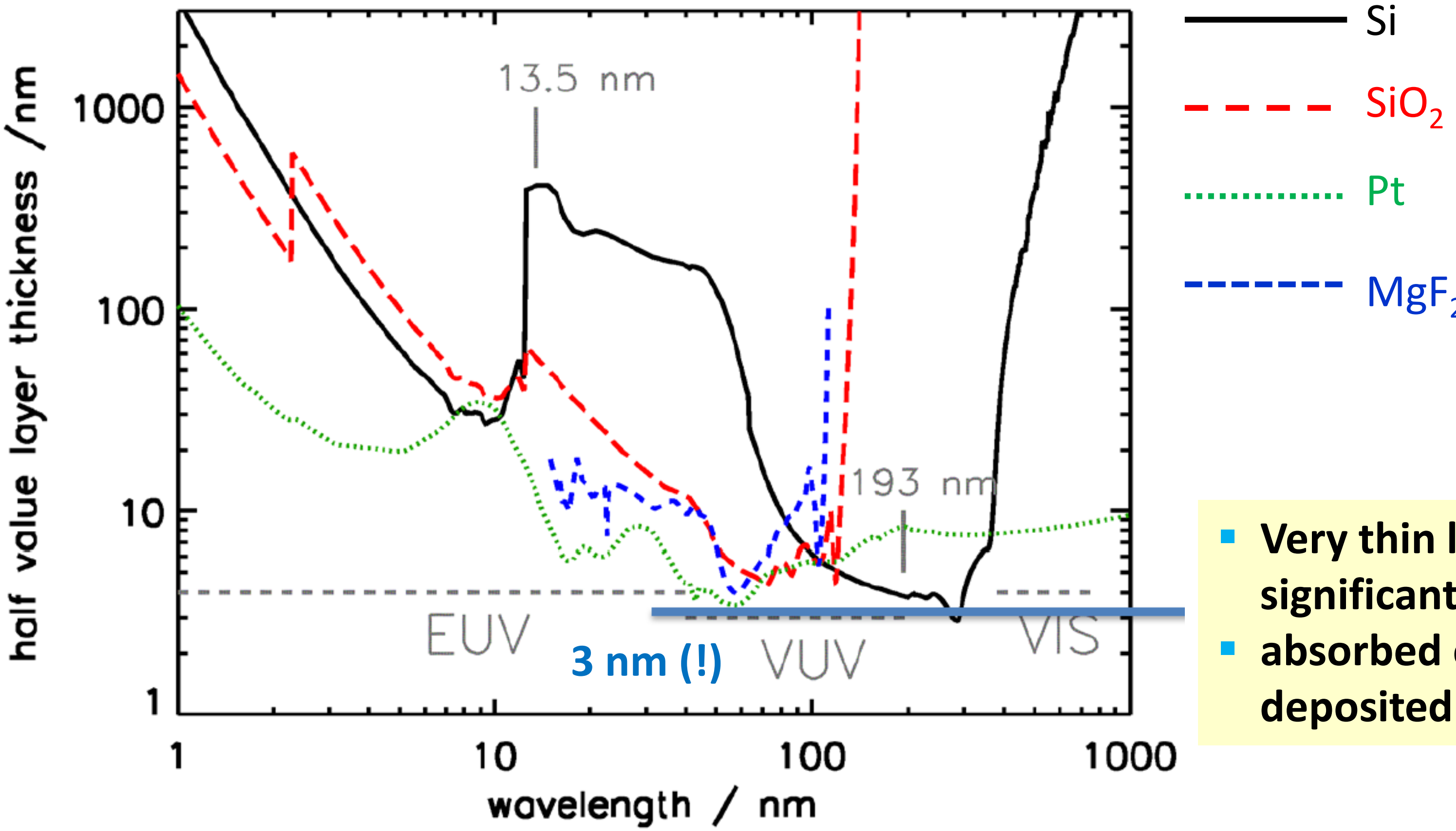
contamination layer
passivation layer, oxide

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

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

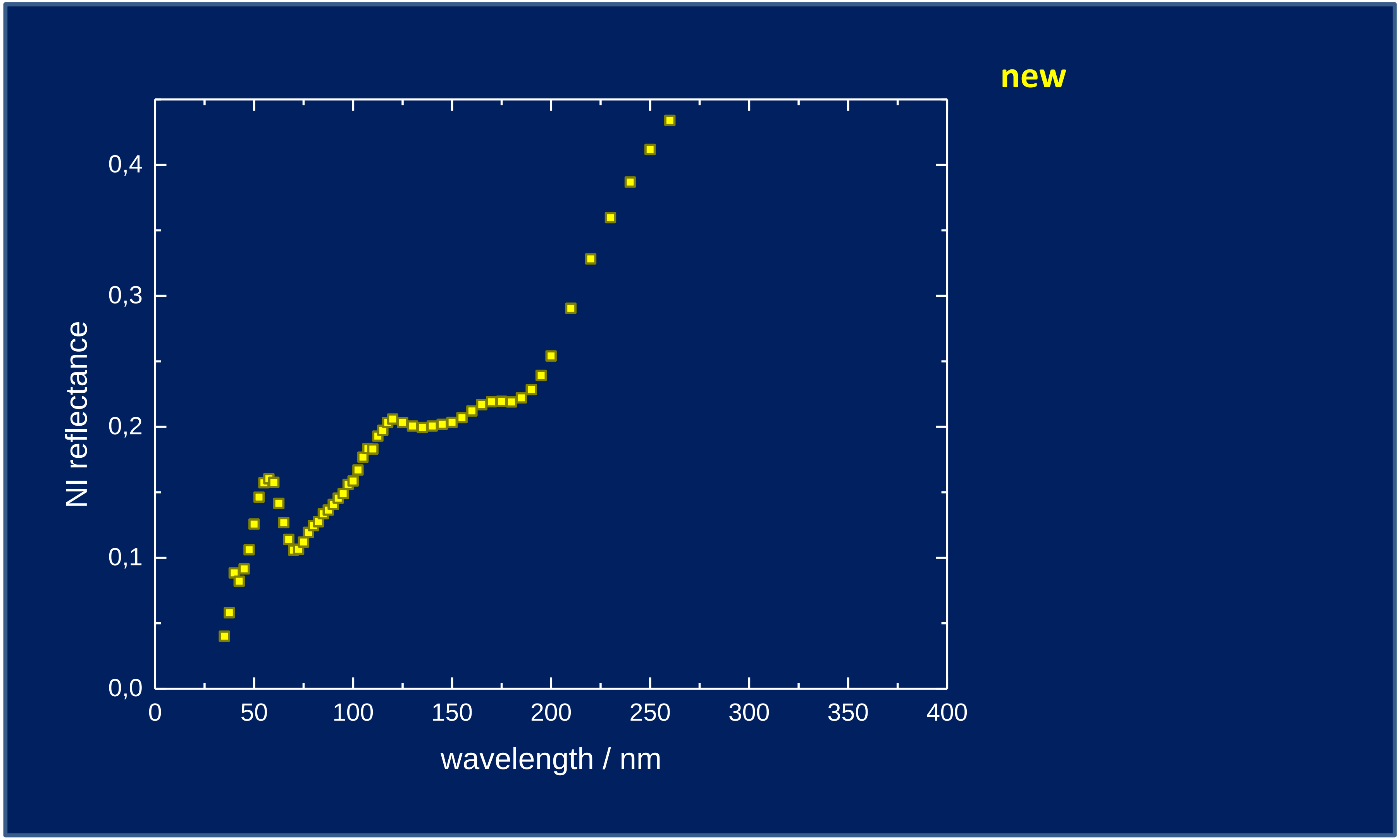
Why wavelengths matters…

- half-value layer thickness of representative materials



- **Very thin layers can significantly contribute**
- **absorbed energy is deposited in surface layer**

Example: Pt mirror



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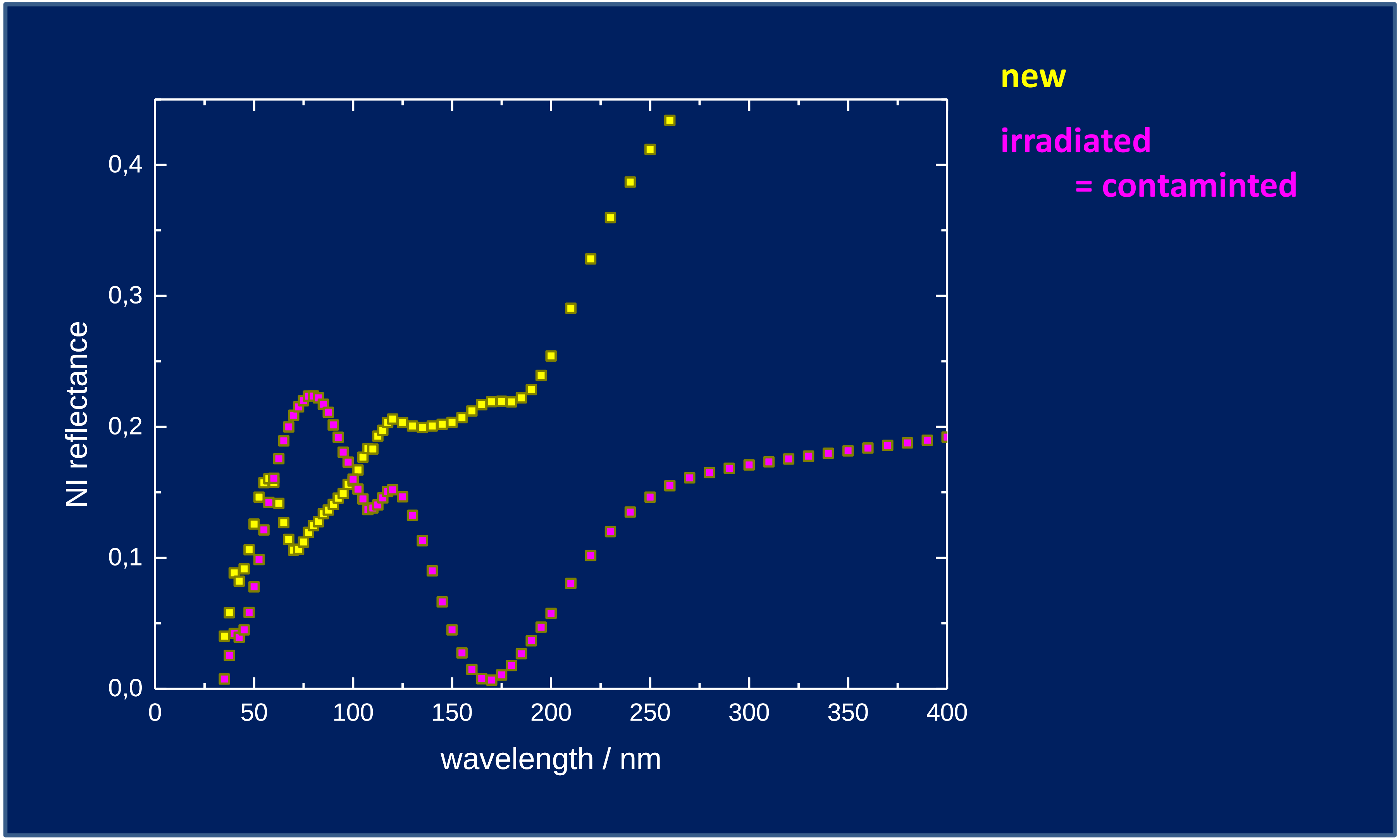

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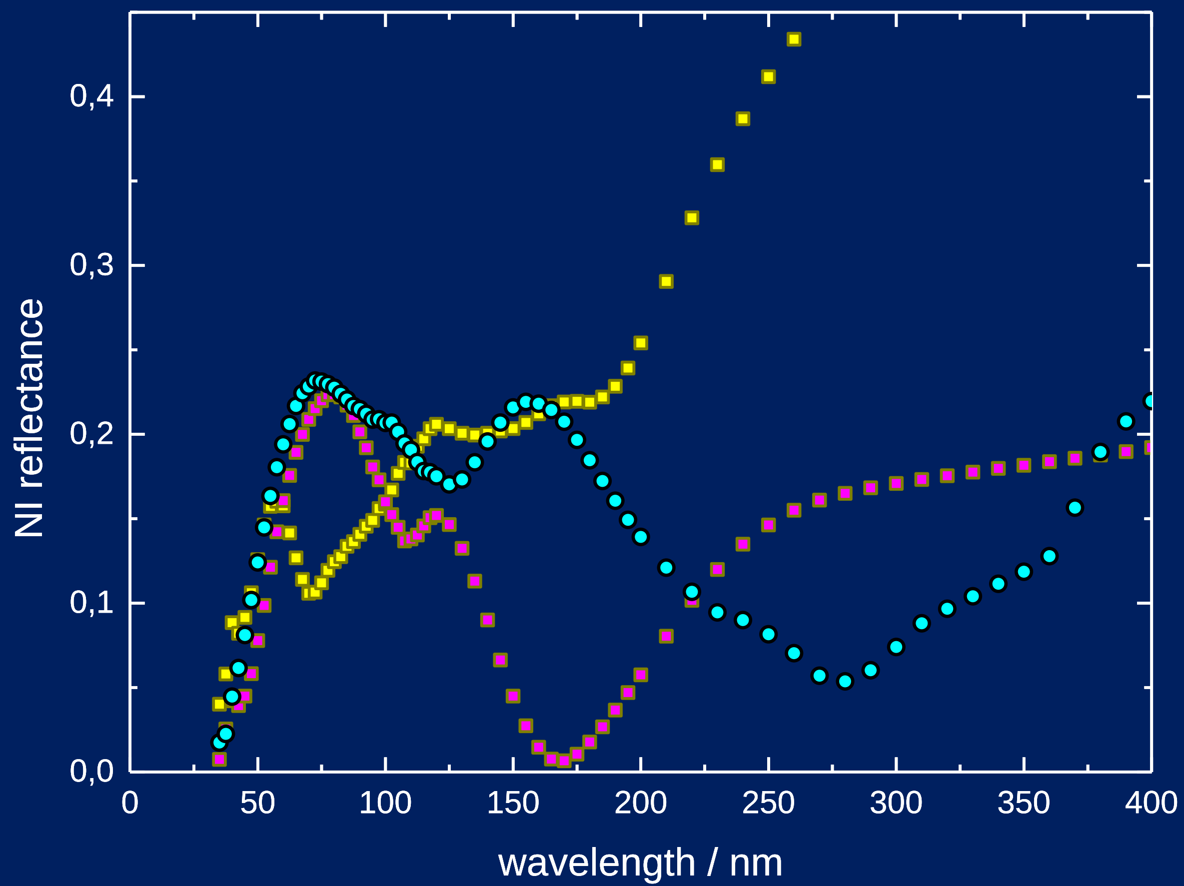


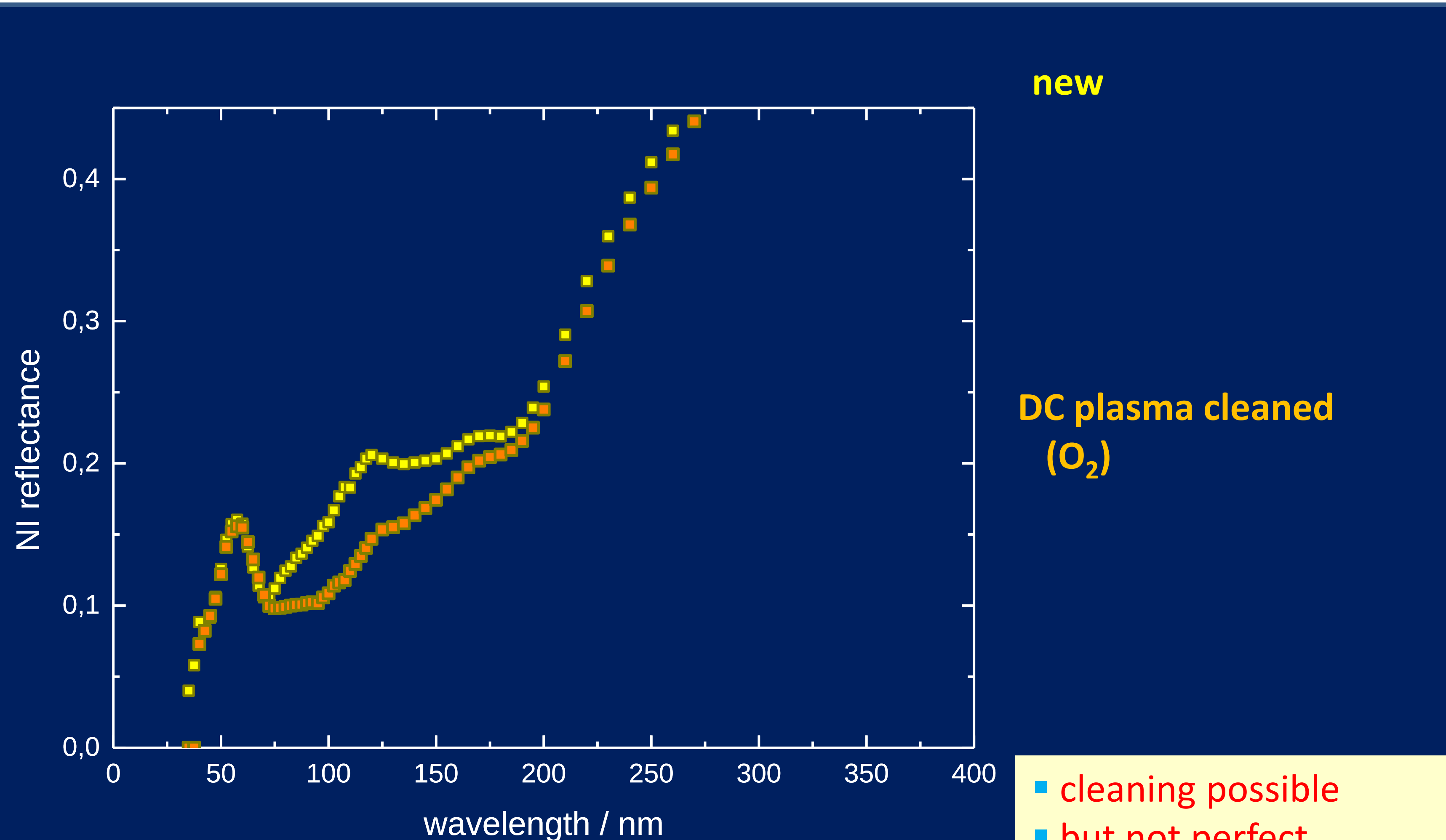
new

irradiated
= contaminted

thick carbon coating
(reference sample)

- thin carbon different than thick carbon
- depending of structure

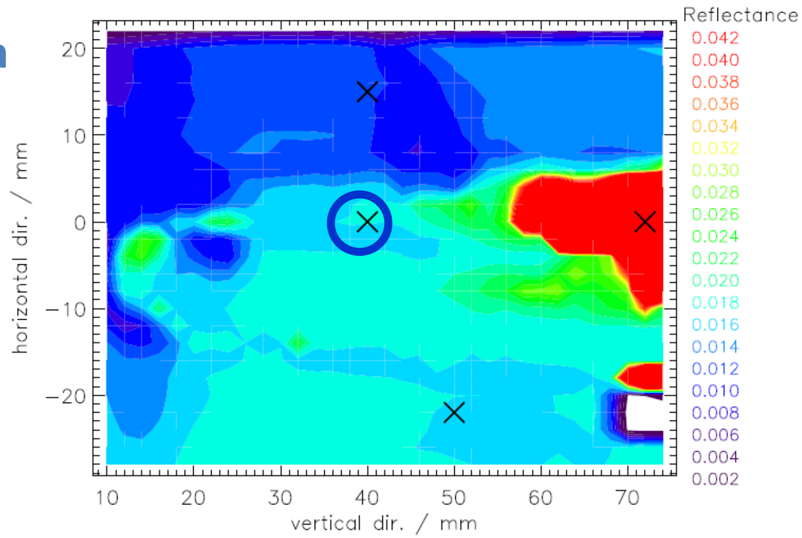
Example: Pt mirror



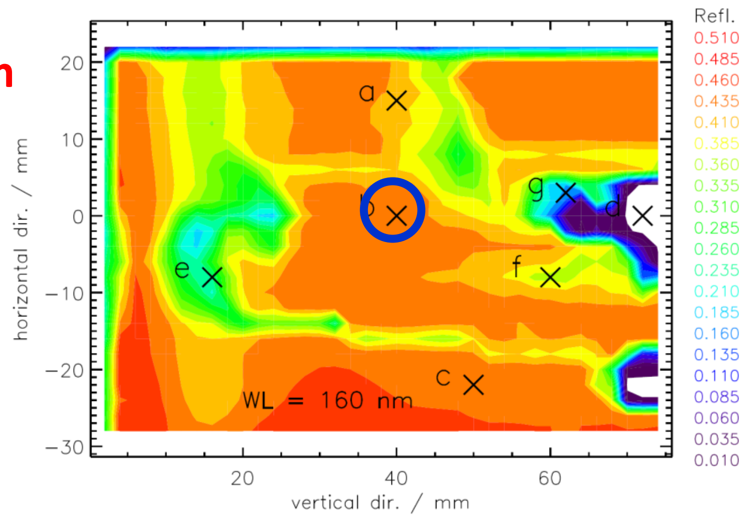
- cleaning possible
- but not perfect

Example: C-coated Si Mirror

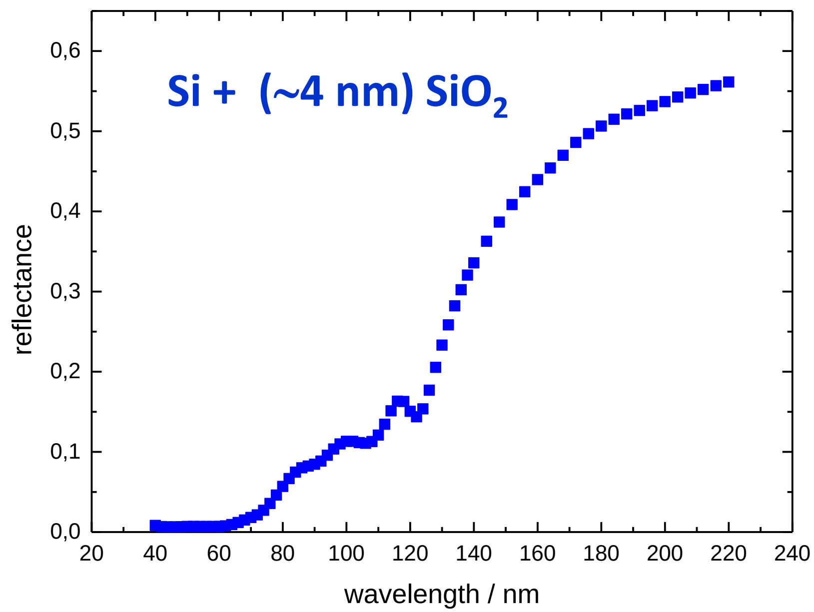
70 nm



160 nm



- 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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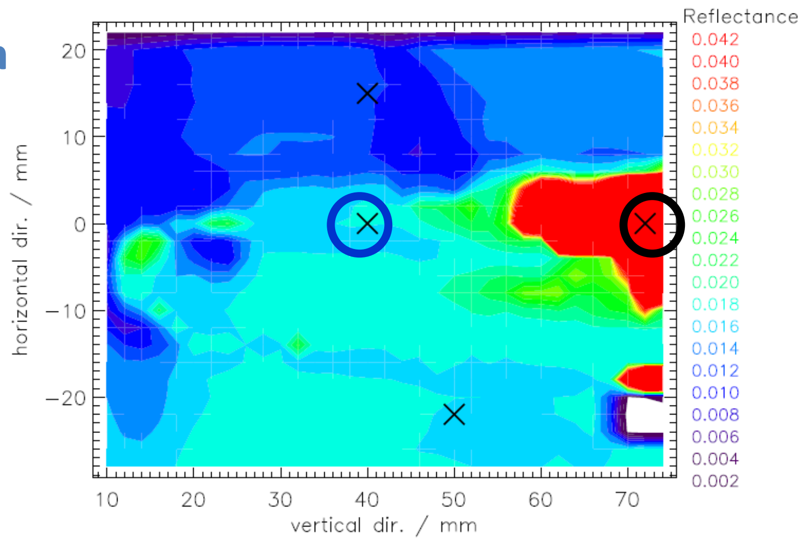

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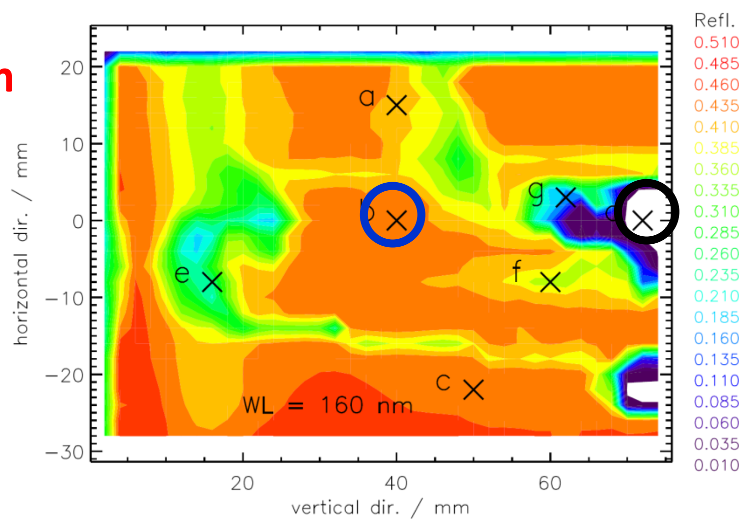

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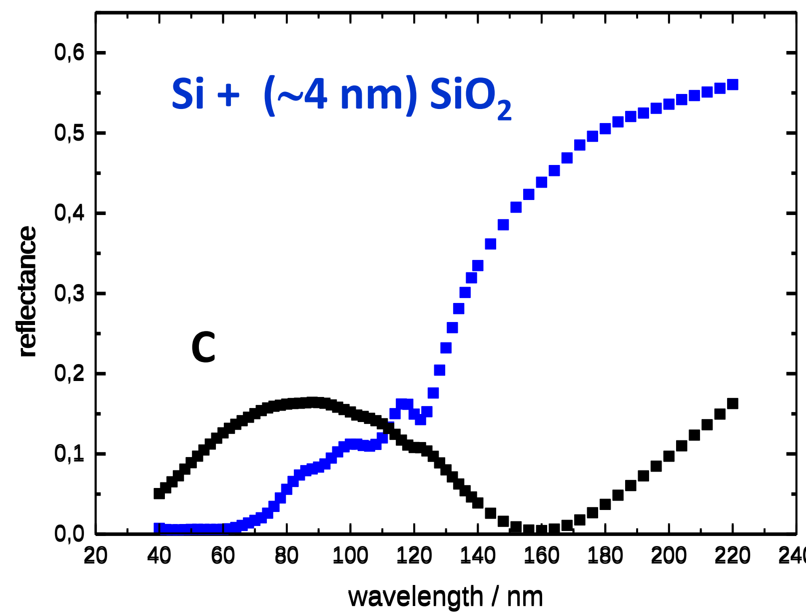

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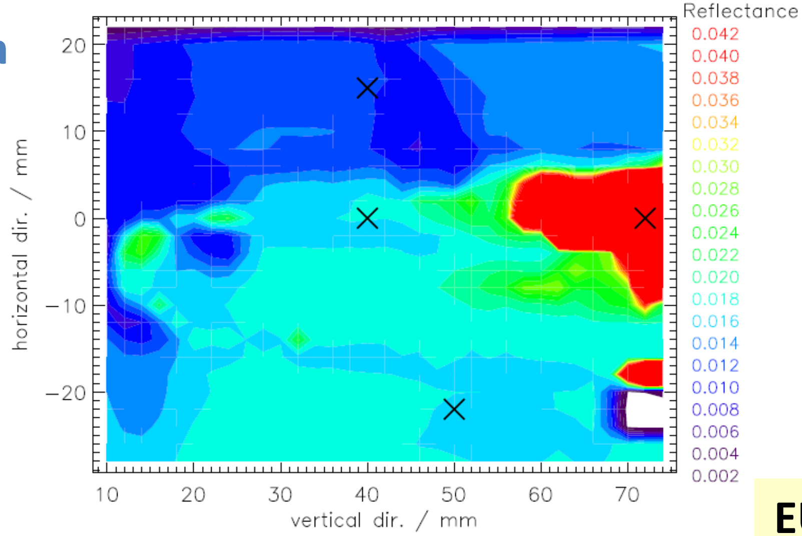

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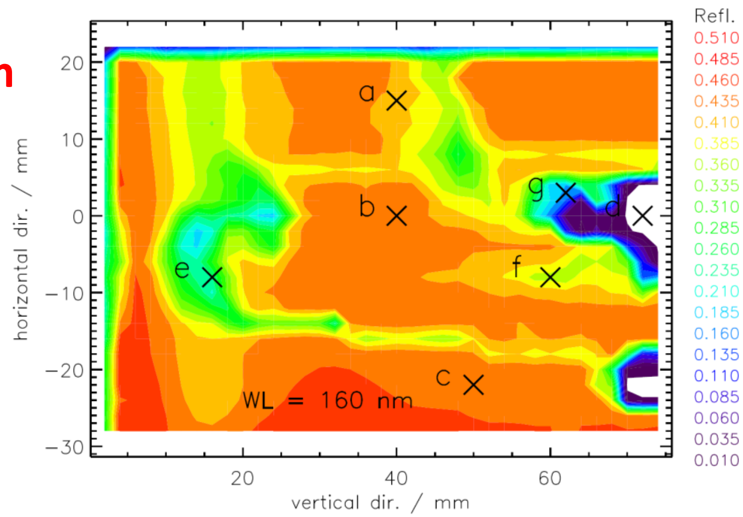

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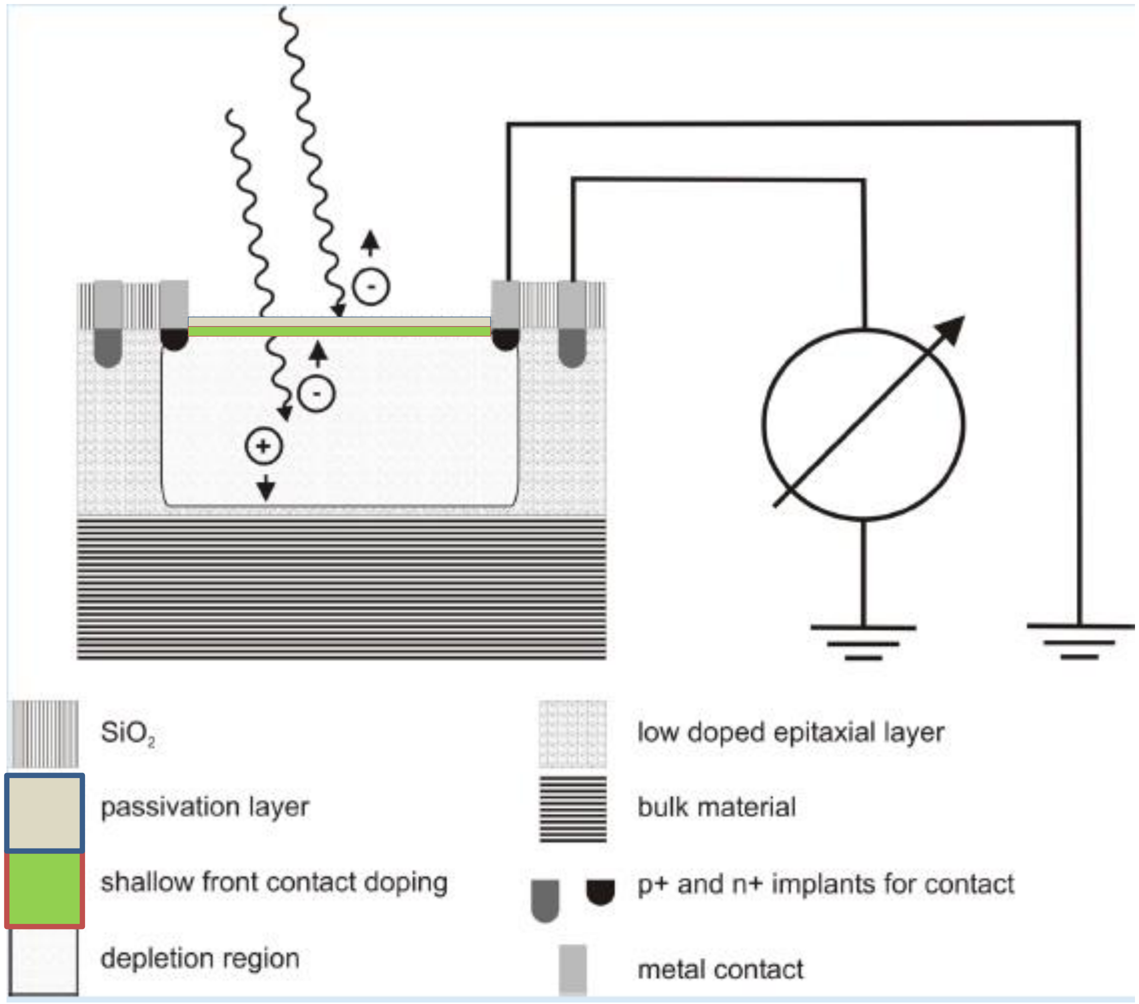
EUV/VUV reflectometry:

- 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

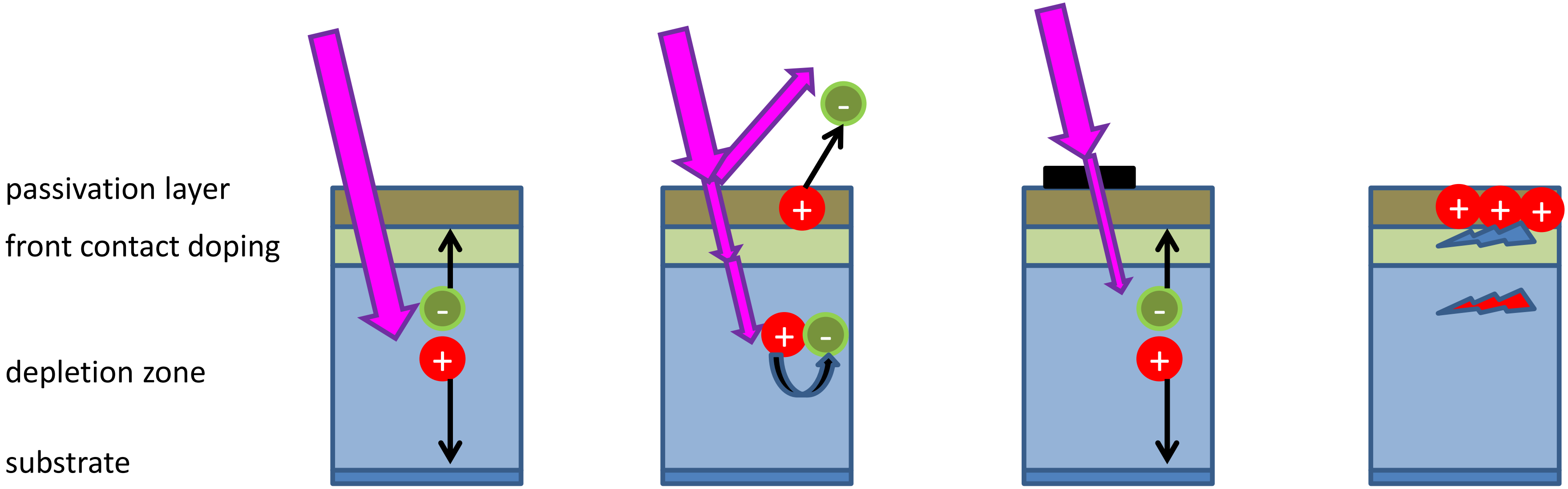


„typical“ Si photodiode

- surface contamination
- interface change/damage
- bulk damage

change of spectral responsivity

Primary effects in radiation detection



ideal case

loss mechanisms

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

unavoidable

contamination

- additional absorption
- local effects !
- photochemistry (carbon growth)

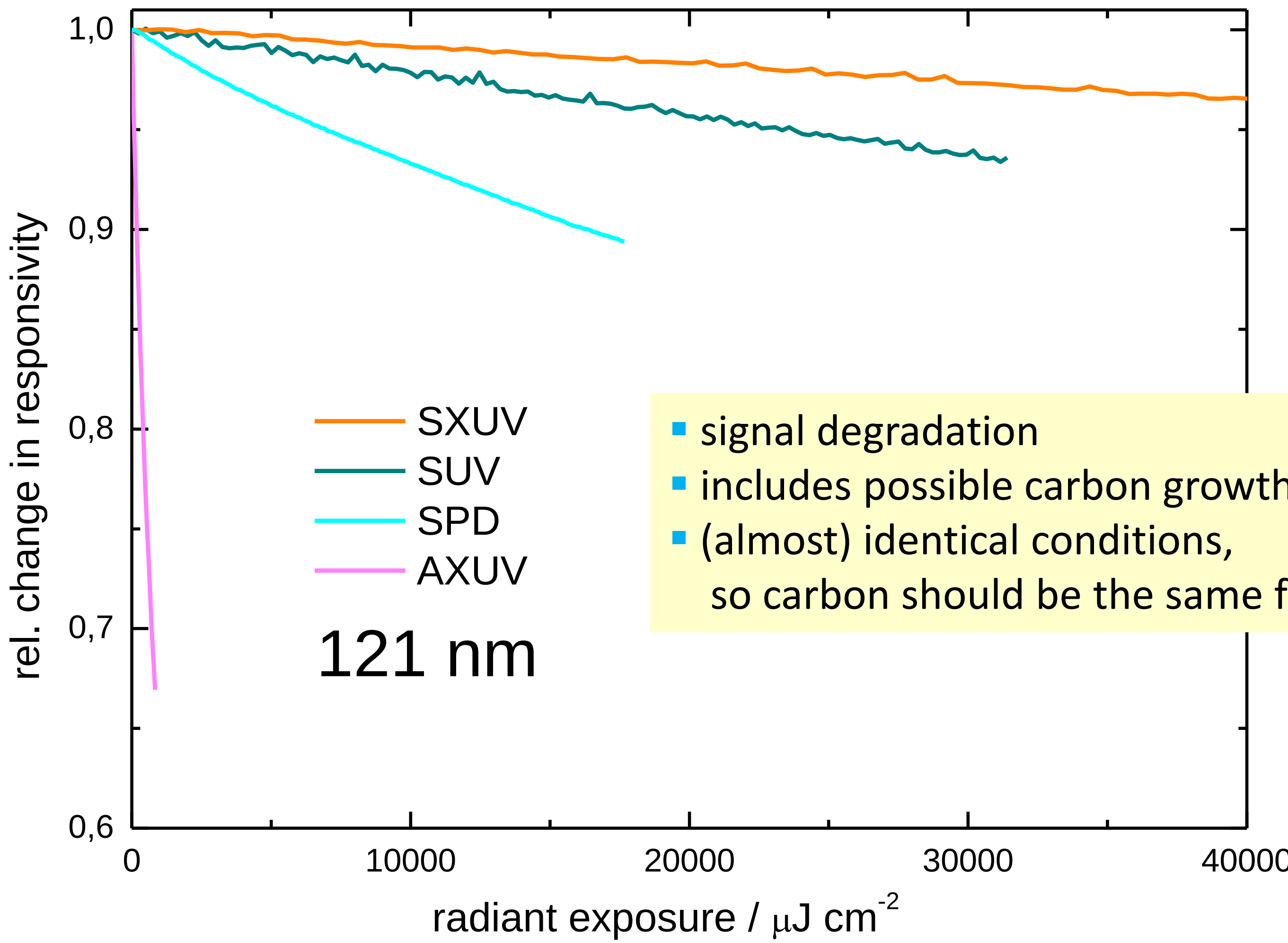
removable

ageing & damage

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

can be minimized by detector design

Irradiation Ageing

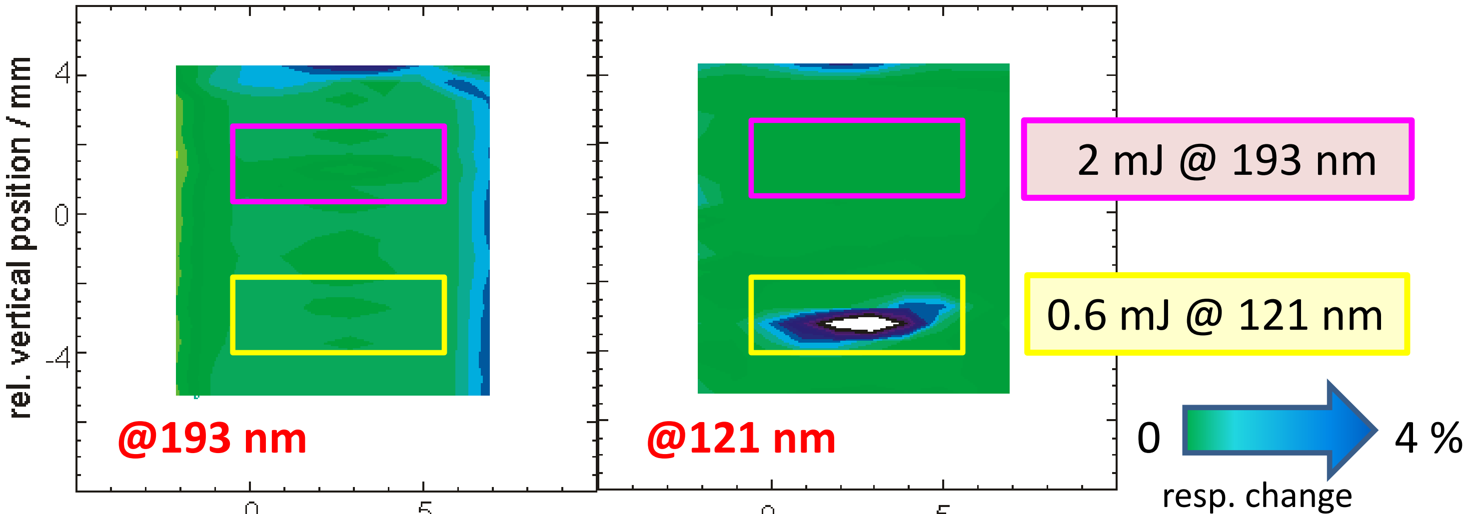


- signal degradation
- includes possible carbon growth
- (almost) identical conditions, so carbon should be the same for all.

Example for ageing: Si photodiode

AXUV-100G photodiode 10 mm x 10 mm

Relative spectral responsivity map:



SiO_2 transparent

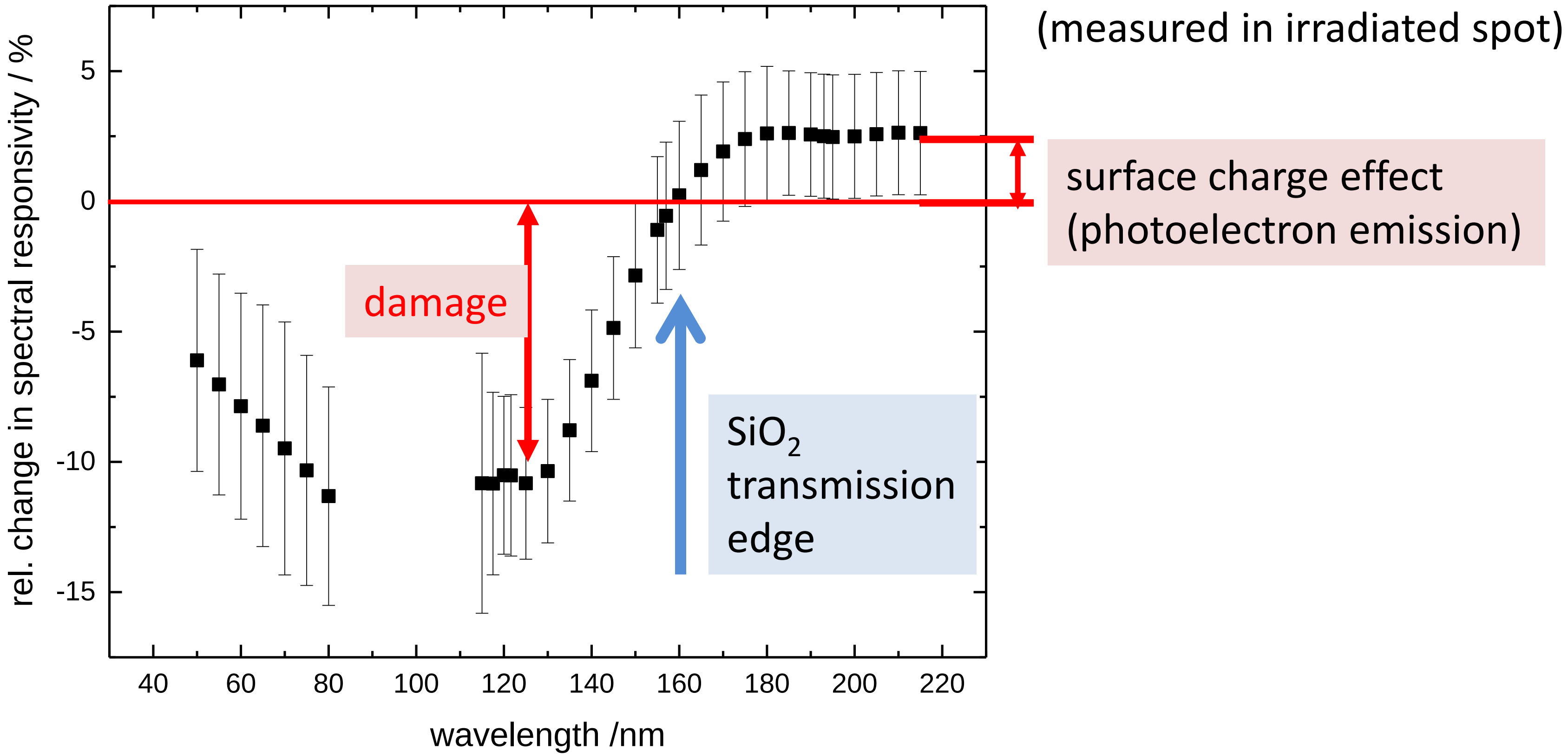
SiO_2 absorbing

- weak effect of 193 nm irrad.

- massive effect of 121 nm irradiation
- **irreversible damage of the SiO_2 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

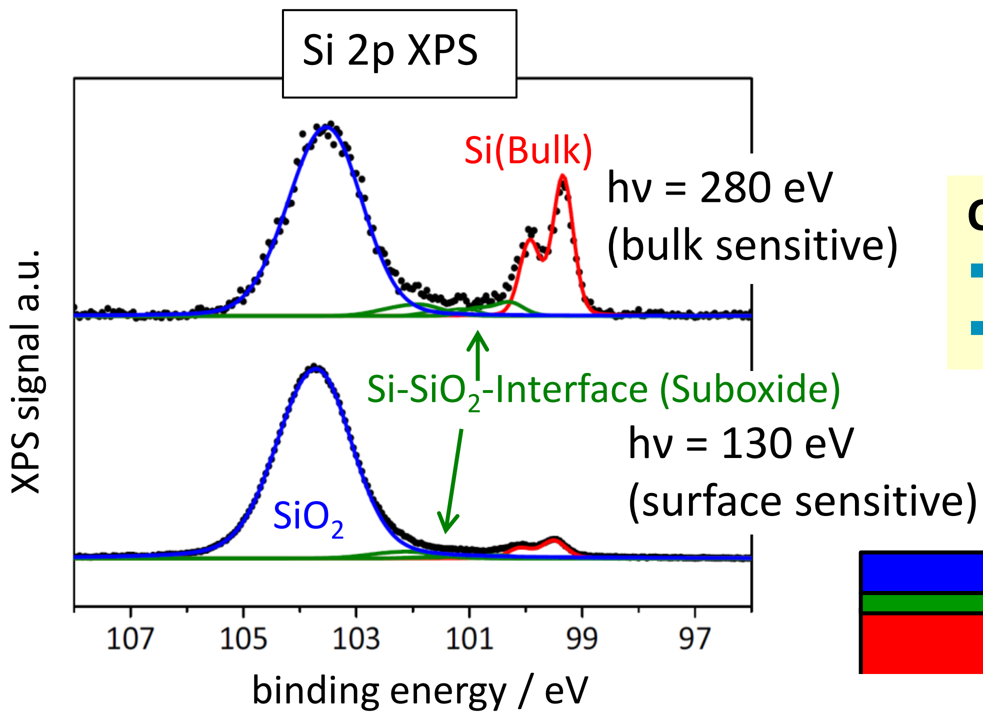


Outlook: Surface analysis to understand processes

Electron spectroscopy (XPS/UPS):
chemicial 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



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. Predediction 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

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