Surface Specific X-ray Scattering

X-ray Reflectivity:

Theory, application and sample preparation

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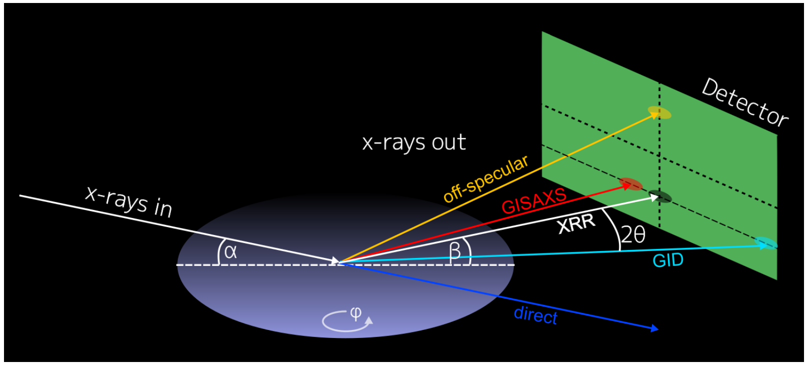
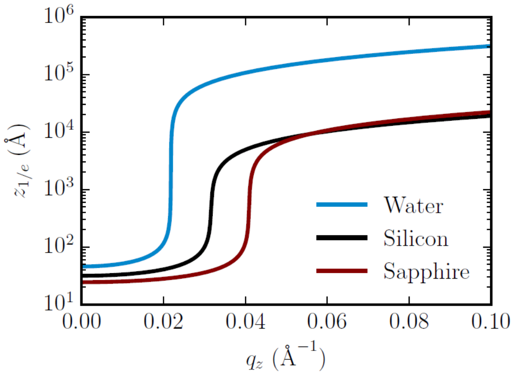
Office of Science

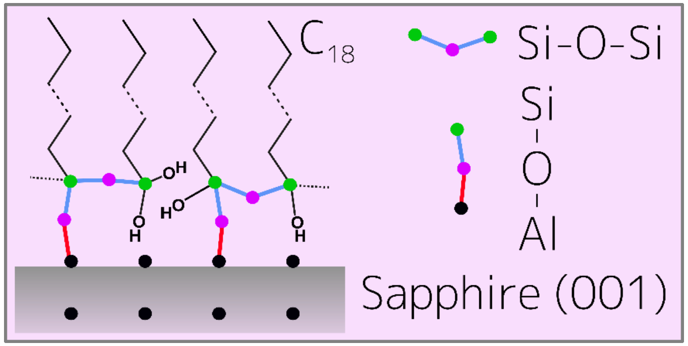
SLAC NATIONAL ACCELERATOR LABORATORY

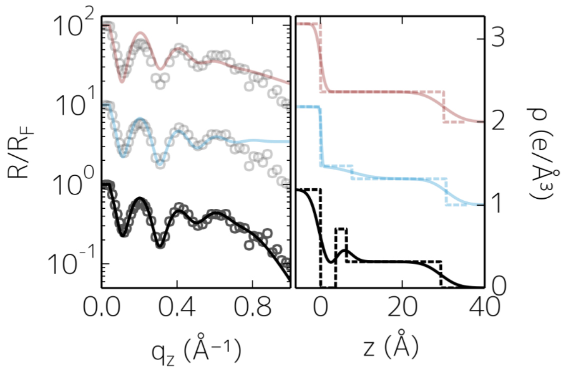
Outline

- **Introduction**
- **Surface x-ray diffraction**
  - **Surface sensitivity**
  - **Technique overview**
- **Focus on x-ray reflectivity**
  - **Theory**
  - **Application**
  - **Sample preparation**









Introduction

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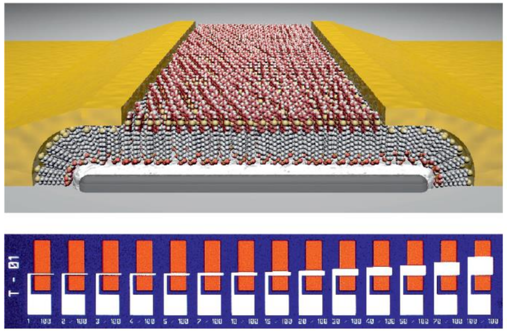
Surfaces

- Outer boundary of any material
- Dominate interaction with environment
- Decisive role in numerous ***natural*** and ***technological*** processes
  - Nanotechnology / Material science
  - Catalysis
  - Energy storage – e.g. batteries

X-rays: Structure-function relation

Molecular thick FETs – SAMFETs

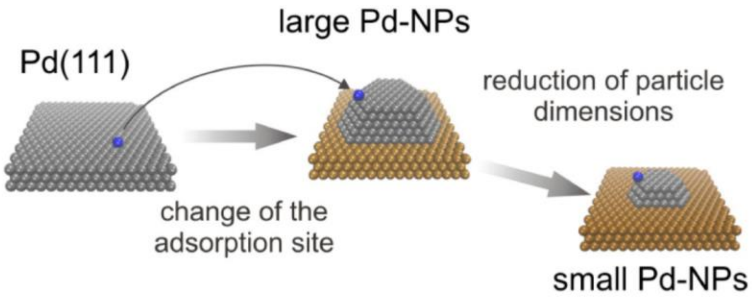
Transport affected by structure



Schmalz, Steinrück et al., Adv. Mat. 25, 4511–4514 (2013)

Adsorption sites for CO & O_2

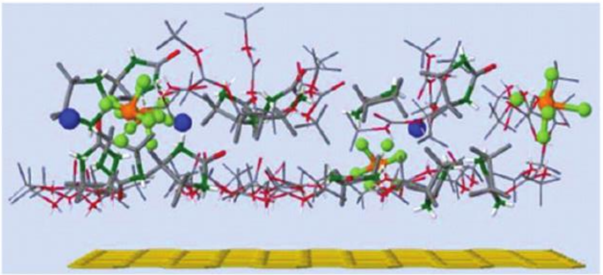
Local site configuration and particle size affect binding energy



Schauermann & Freund, Acc. Chem. Res. 48, 2775 (2015)

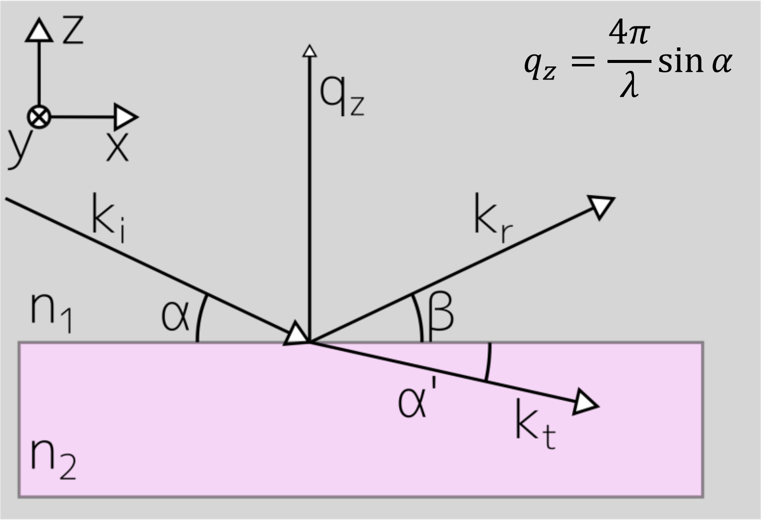
Ion transport into electrodes

Governed by molecular arrangement



Vatamanu et al., J. Phys. Chem. C 116, 1114 (2012)

Surface sensitivity



Refraction index:

$\boldsymbol{n = 1 - \delta - i\beta}$

$\delta = \frac{\lambda^2 r_e}{2\pi}\rho_e$: wavelength dependent scattering
$\sim 1e^{-6}$

β : wavelength dependent absorption
$\sim 1e^{-8}$ – *neglelible in most cases*

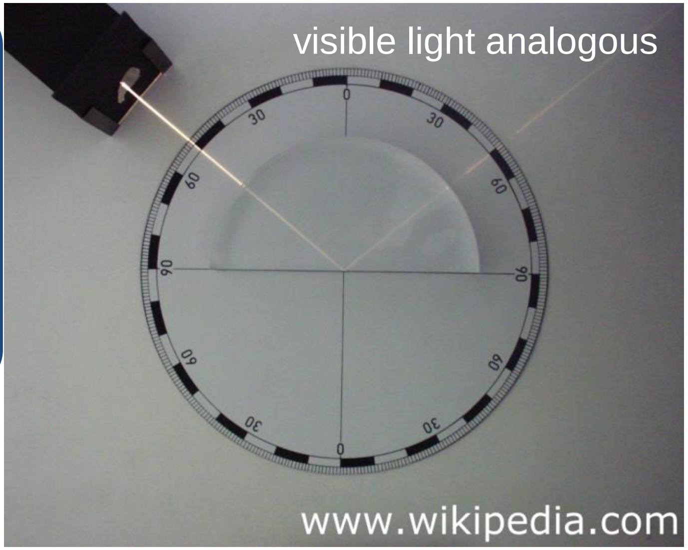
$\rightarrow \boldsymbol{n_1 > n_2}$: **Total external reflection**

Snell's law:

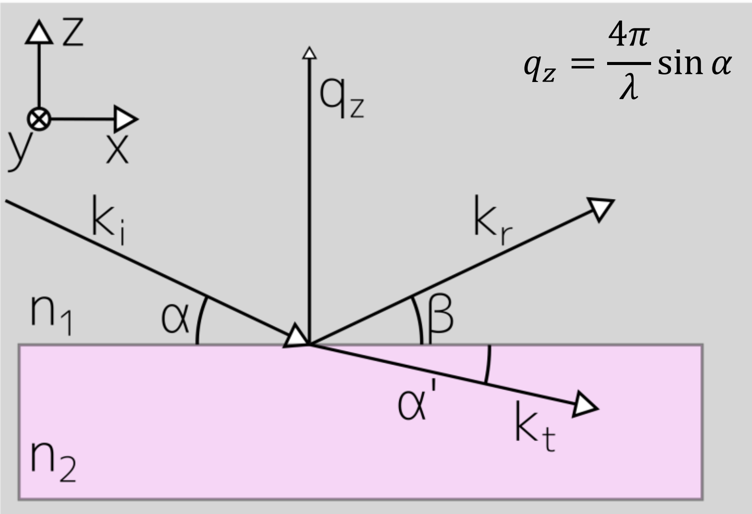
$n_1 \cos\alpha = n_2 \cos\alpha'$

in vacuum ($n_1 = 1$):

$\cos\alpha = n_2 \cos\alpha'$



Surface sensitivity



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Critical angle α_c

$$\alpha' = 90° \rightarrow \cos \alpha' = 1$$

$$\cos \alpha_c = n_2 = 1 - \delta_2$$

with $\cos \alpha_c \approx 1 - \alpha_c^2/2$

$$\alpha_c \approx \sqrt{2\delta_2} \approx 1°$$

Angle of refraction

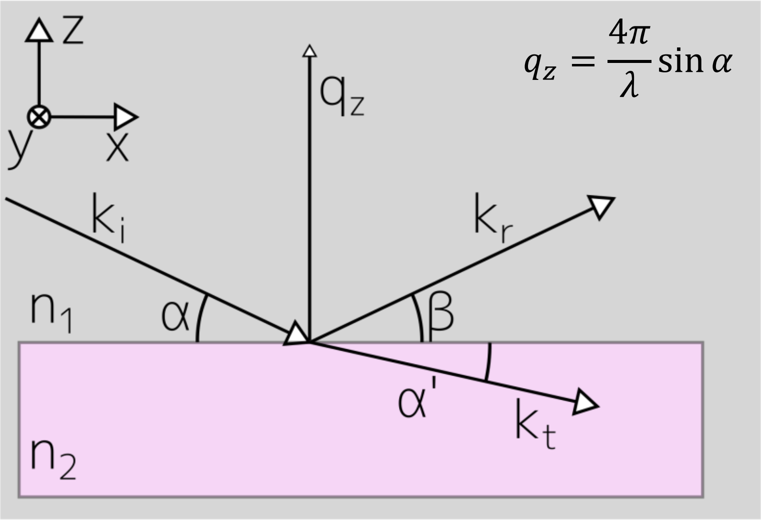
$$n = 1 - \delta = 1 - \alpha_c^2/2$$

in Snell's law

$$1 - \frac{\alpha^2}{2} = \left(1 - \frac{\alpha'^2}{2}\right)\left(1 - \frac{\alpha_c^2}{2}\right)$$

$$\boldsymbol{\alpha' = \sqrt{\alpha^2 - \alpha_c^2}}$$

Surface sensitivity



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$\cos\alpha_c = n_2 = 1 - \delta_2$

with $\cos\alpha_c \approx 1 - \alpha_c^2/2$

$\alpha_c \approx \sqrt{2\delta_2} \approx 1°$

Angle of refraction

What happens for $\alpha_c > \alpha$?

$\alpha' = \sqrt{\alpha^2 - \alpha_c^2}$

Evanescant wave – below the critical angle

How far do the x-rays penetrate into the material as a function of incoming angle / scattering vector?

z-component of amplitude of electromagnetic field inside material

$E_{\text{transmitted}}(z) = E_0 e^{i(wt - k'_z z)}$

what is k'_z ? *in terms of the incident angle ?*

$k'_z \approx n k_0 \sin\alpha'$

- *for small* $\alpha' \rightarrow \sin\alpha' = \alpha'$
- $n = 1$

$k'_z \approx k_0 \alpha'$

$\alpha' = \sqrt{\alpha^2 - \alpha_c^2}$

for $\alpha \ll \alpha_c \rightarrow$ *purely imaginary*

$\alpha' = i\alpha_c$

Evanescant wave – below the critical angle

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$\alpha' = i\alpha_c$

Using $k_0 = 2\pi/\lambda$ and $\alpha_c = q_c \lambda / 4\pi$

$k'_z \approx k_0 \alpha' = k_0 \cdot i \cdot \alpha_c = i \cdot \frac{2\pi}{\lambda} \cdot \alpha_c$ expressed in *critical* angle α_c

$= i \cdot \frac{q_c}{2}$ expressed in *critical* scattering vector q_c

Evanescant wave – below the critical angle

How far do the x-rays penetrate into the material as a function of incoming angle / scattering vector?

z-component of amplitude of electromagnetic field inside material

$$E_{\text{transmitted}}(z) = E_0 e^{i(wt - k'_z z)}$$

what is k'_z ? *in terms of the incident angle ?*

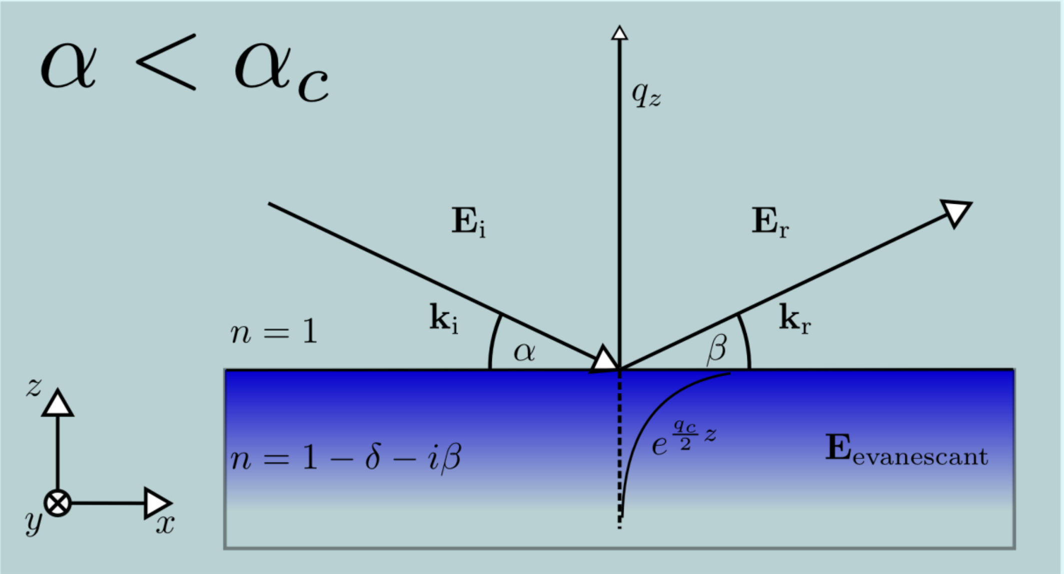
$$k'_z \approx i \cdot \frac{q_c}{2}$$

$$E_{\text{evanescant}} = E_0 e^{i(wt - k'_z z)} = E_0 e^{\frac{q_c}{2} z}$$

exponentially damped wave with

intensity decay length $z_{1/e}$:

$z_{1/e} \approx \frac{1}{q_c}$ **, typically 100 Å**



Evanescant wave – below the critical angle

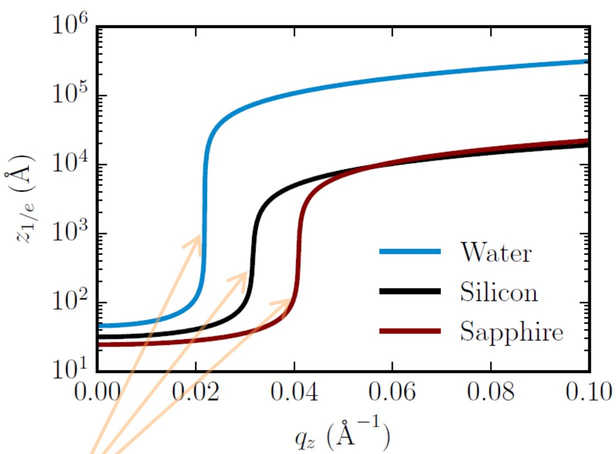
The general case for all α:

$$z_{1/e} = \frac{\lambda}{\frac{4\pi}{\sqrt{2}}\sqrt{\sqrt{(\alpha^2-\alpha_c^2)^2+4\beta^2}-(\alpha^2-\alpha_c^2)}}$$

$$\approx \frac{1}{\sqrt{q_c^2 - q_z^2}}$$

$$z_{1/e} \approx \frac{1}{q_c}$$

Some examples:

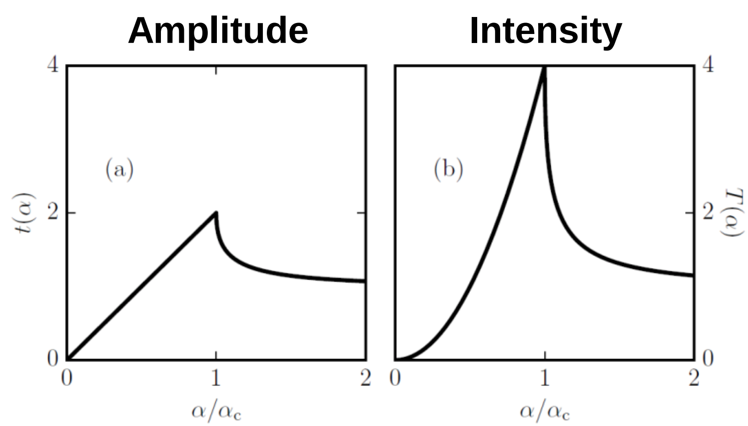


∝ critical angle

What is the intensity just below the interface?

Where does the observed scattering come from?

The surface enhancement factor



interference of incident and reflected wave:

→ **constructive** @ $\alpha = \alpha_c$

Up to 16x increased

Below critical angle:

- Significantly reduced penetration depth: ~ 100 Å
- Scattering enhanced

→ **X-rays are surface sensitive!**

Overview

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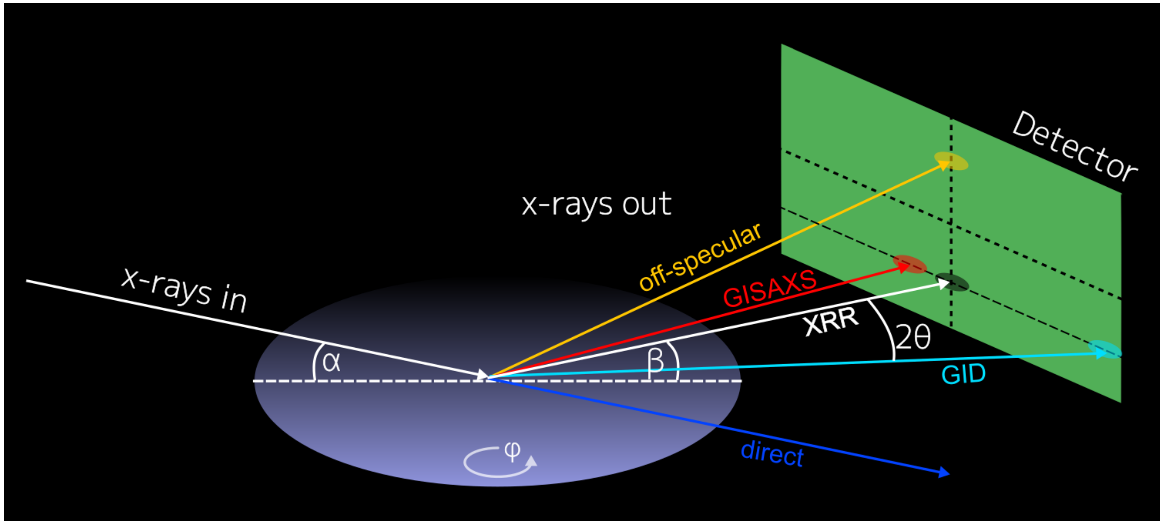
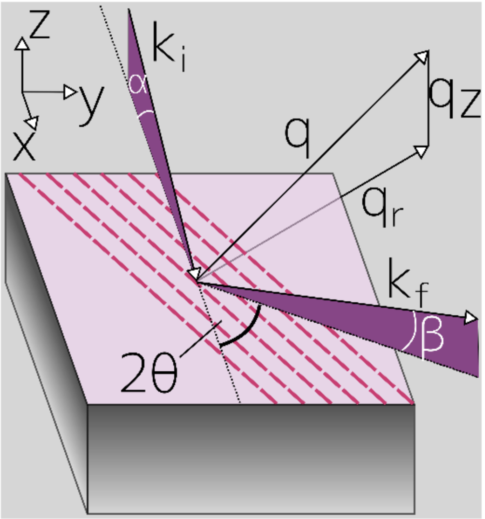
X-ray reflectivity XRR | **Off-specular diffuse scattering** | **Grazing incidence diffraction GISAXS & GID** | **Truncation rods**

The scattering geometry





1. X-rays impinge sample under *α*
2. Interact with the sample
3. Exit sample according to sample properties under *β* and *2θ*

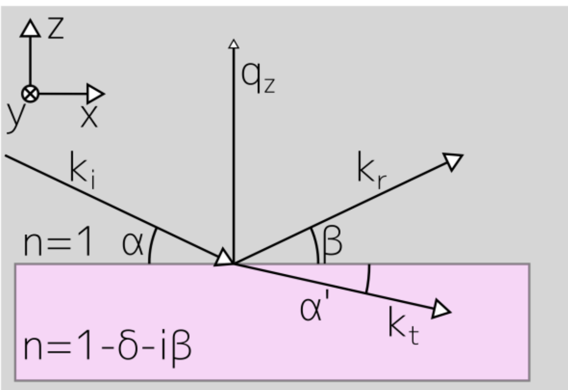
Direction of ***q*** important, not only magnitude

$$\boldsymbol{q} = \boldsymbol{k}_f - \boldsymbol{k}_i$$

$$q = \begin{pmatrix} q_x \\ q_y \\ q_z \end{pmatrix} = \frac{2\pi}{\lambda} \cdot \begin{pmatrix} \cos\beta \cdot \cos 2\theta - \cos\beta \cdot \cos 2\theta \\ \cos\beta \cdot \sin 2\theta \\ \sin\alpha + \sin\beta \end{pmatrix}$$

X-ray reflectivity - XRR

Geometry



α = β

α = varied

$\theta/2\theta$-scan / butterfly scan

$q_x = 0$, $q_y = 0$, $q_z \neq 0$

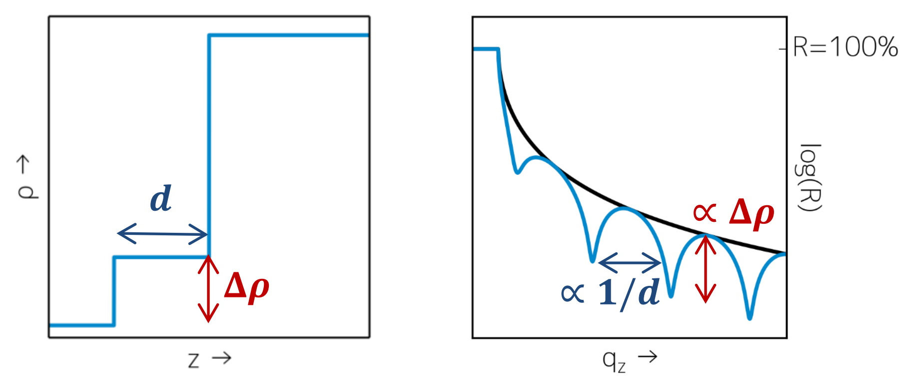
Information

Scattering vector solely perpendicular to surface

→ Surface normal information

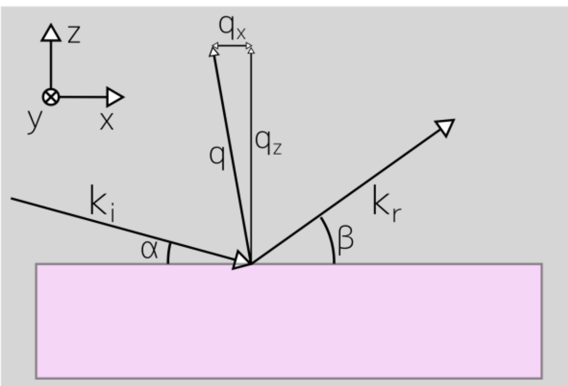
- Layer thickness
- Layer density
- Surface and interface roughness

Surface normal electron density profile



Off-specular diffuse scattering

Geometry



α = fixed, β = varied

Detector scan

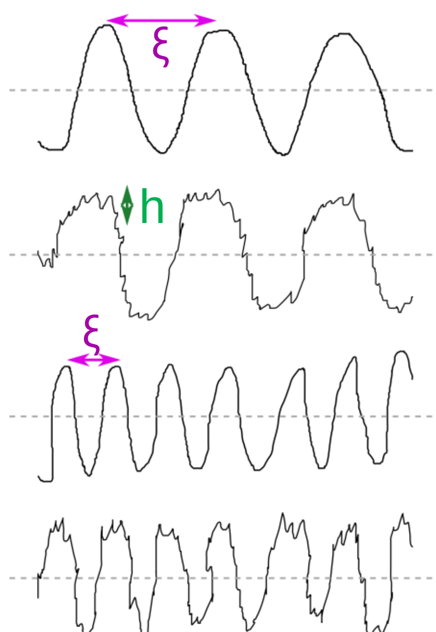
α + β = fixed, α = varied

Rocking scan

$q_x \neq 0$, $q_y = 0$, $q_z \neq 0$

Information

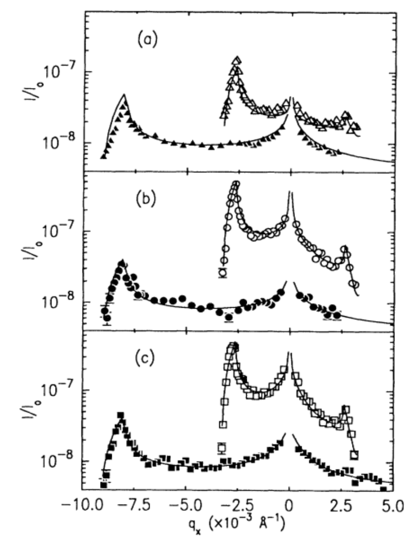
- Surface roughness lateral information!
- Correlation-length ξ
- Jaggedness h



Example

Wetting of a rough surface

Tidswell et al., Phys. Rev. Lett 66, 2108 (1991)



Thin film (< 60 Å):

Correlation to substrate roughness

Thick film:

Cappilary waves

GISAXS

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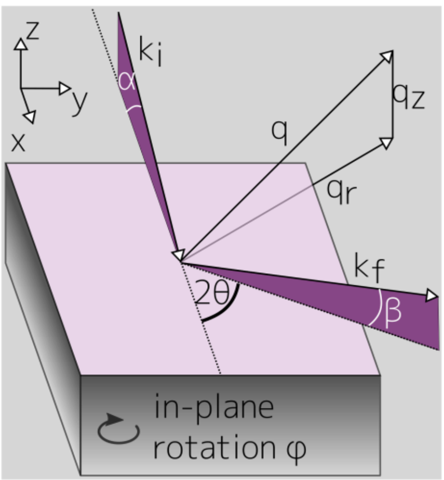
X-ray reflectivity XRR | Off-specular diffuse scattering | **Grazing incidence diffraction GISAXS & GID** | Truncation rods

Geometry



α = fixed
β = varied
2θ = varied
φ = 0 or varied
$q_x \neq 0,\ q_y \neq 0,\ q_z \neq 0$

$$q_r = \sqrt{q_x^2 + q_y^2}$$

Information

Typical $2\theta = 2°$

low q

→

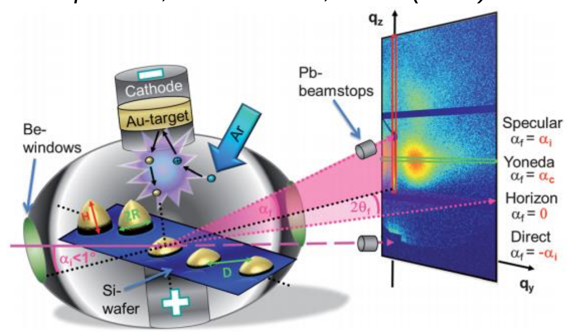
large in real space

- **Morphology**
  - **Surface**
  - **Particles**
- **Nano- macro-scale density correlations**

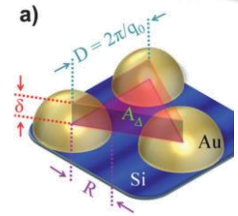
Example

In-situ gold cluster growth

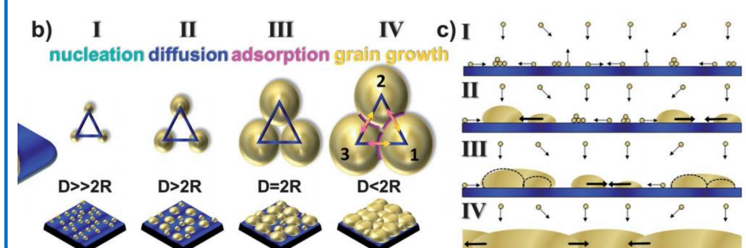
Schwartzkopf et al., Nanoscale 5, 5053 (2013)



a)



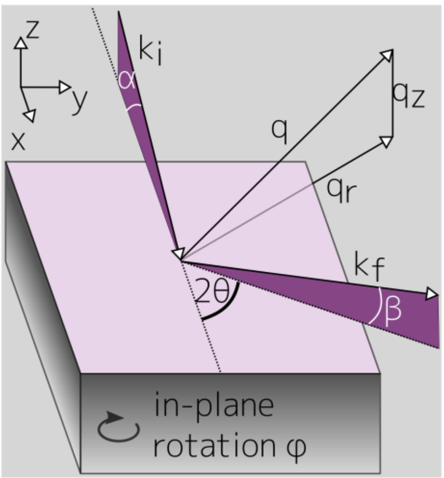
- **size R**
- **distance D**
- **shape, etc.**



GID - GIWAXS

X-ray reflectivity XRR | Off-specular diffuse scattering | **Grazing incidence diffraction GISAXS & GID** | Truncation rods

Geometry



α = fixed
β = varied
2θ = varied
φ = 0 or varied
$q_x \neq 0$, $q_y \neq 0$, $q_z \neq 0$

$$q_r = \sqrt{q_x^2 + q_y^2}$$

Information

Typical 2θ = 20°

large q

→

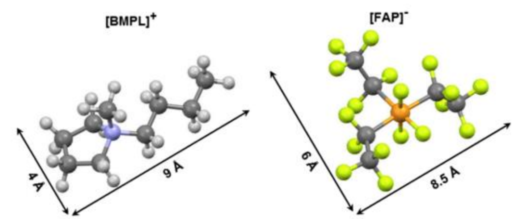
small in real space

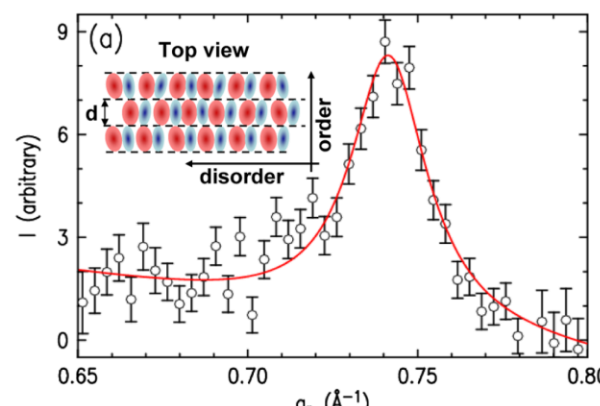
- **Atomic order**
- **Crystallinity**
  - *unit cell*
  - *crystal size*
- **Molecular orientation of adlayers**

Example

Checkerboard layering in ionic liquids (free surface)

Tamam et al., PRL 106, 197801 (2011)

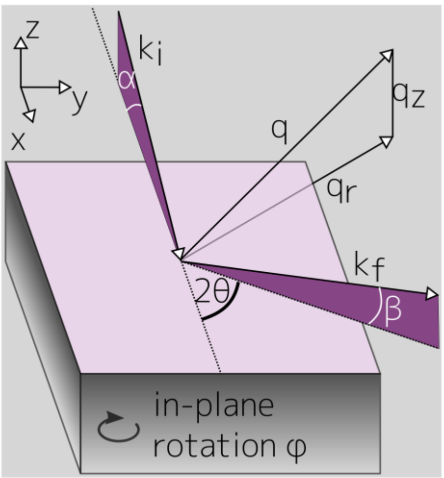




CTR

X-ray reflectivity XRR | Off-specular diffuse scattering | Grazing incidence diffraction GISAXS & GID | **Truncation rods**

Geometry



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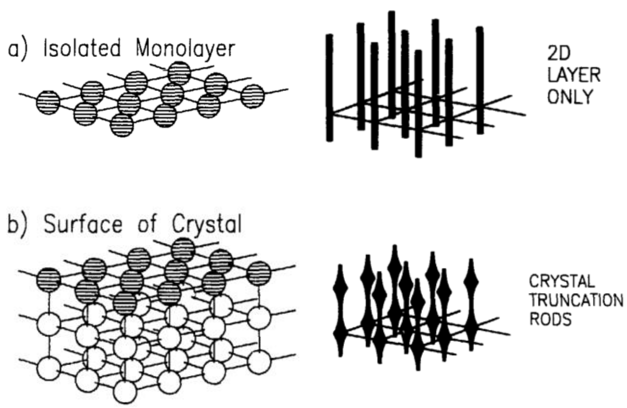
Information

Truncated surface

- Bragg peak shape change
- Crystallinity of surface

real space | reciprocal space

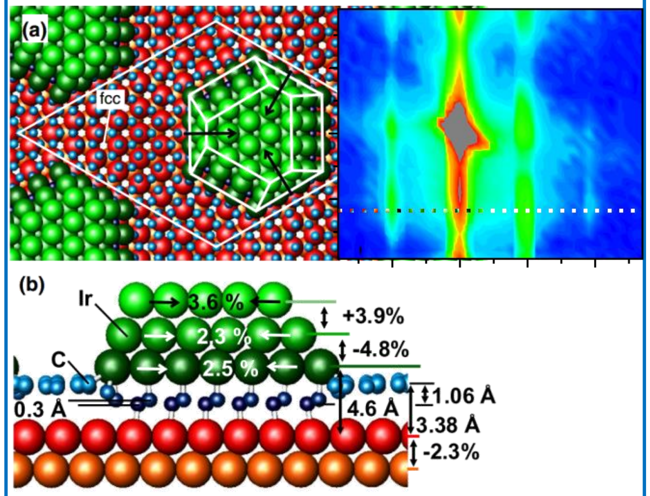


Robinson & Tweet, Rep. Prog. Phys. 55, 599 (1992)

Example

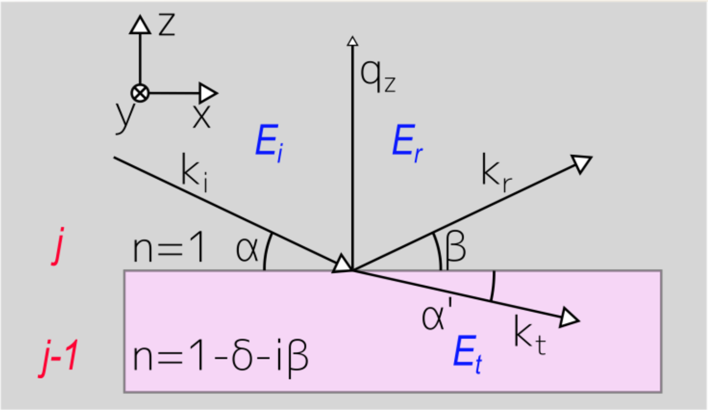
Structure of Graphen-supported Ir Nanoparticles

Franz et al., PRL 110, 065503 (2013)



- Crystallographic superlattice
- Epitaxy on graphene moire´ structure
- Compressive intraparticle strain

X-ray reflectivity



Specularly reflected intensity fraction

$$R(\alpha) = \frac{I(\alpha)}{I_0} \quad \text{at } \alpha = \beta$$

$$q_z = k_r - k_i = \frac{4\pi}{\lambda}\sin\alpha \qquad R(q_z) = \frac{I(q_z)}{I_0}$$

Fresnel reflectivity:

- Simples case of reflection of x-rays from a single interface
- Solve **Helmholtz equation**:

  propagation of light through medium characterized by refractive index

  Solution = plane wave: $E_j = A_j \cdot e^{-i(\omega t - \mathbf{k}_j \mathbf{r})}$

Electro-magnetic field must be continuous at the interface!

$$A_i + A_r = A_t \qquad (A_i + A_r)\sin\alpha = \frac{n_j}{n_{j-1}} A_t \sin\alpha'$$

Define reflection & transmission coefficient: $\boldsymbol{r_{j,j-1}} = \frac{A_r}{A_i}$ & $\boldsymbol{t_{j,j-1}} = \frac{A_t}{A_i}$

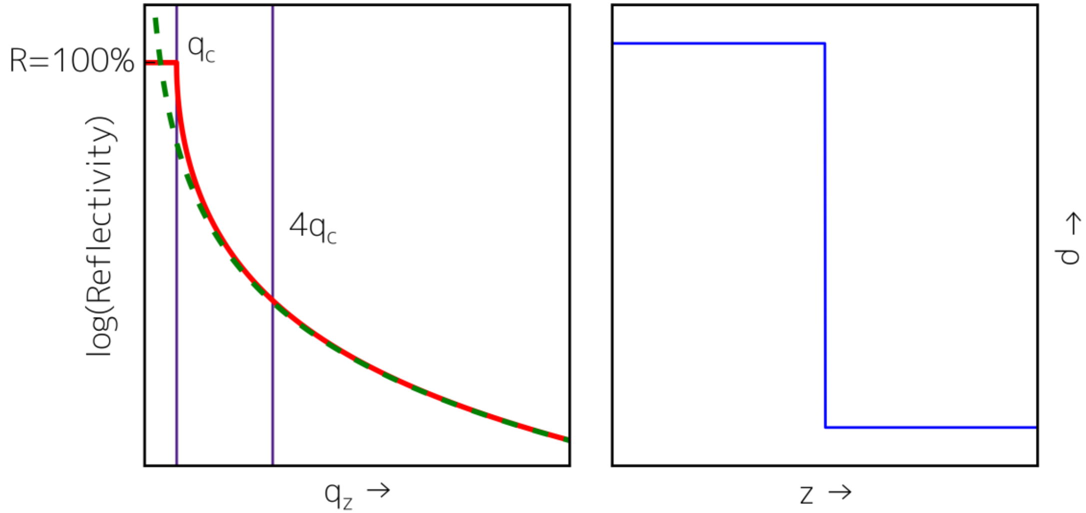
$$\xrightarrow{\text{with } k_j = n_j k_0, \text{ equate \& solve for } \boldsymbol{r}} \quad R(q_z) = |r_{j,j-1}|^2 = \left|\frac{k_{j,z} - k_{j-1,z}}{k_{j,z} + k_{j-1,z}}\right|^2 = \left|\frac{q_z - \sqrt{q_z^2 - q_c^2}}{q_z + \sqrt{q_z^2 - q_c^2}}\right|^2$$

X-ray reflectivity

$$R(q_z) = \left| \frac{q_z - \sqrt{q_z^2 - q_c^2}}{q_z + \sqrt{q_z^2 - q_c^2}} \right|^2 \equiv R_F$$

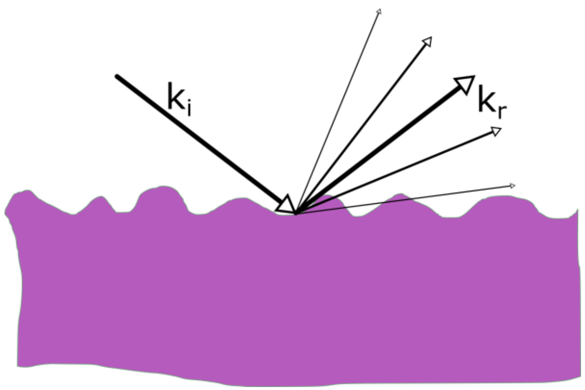
q_c is related to electron density ρ

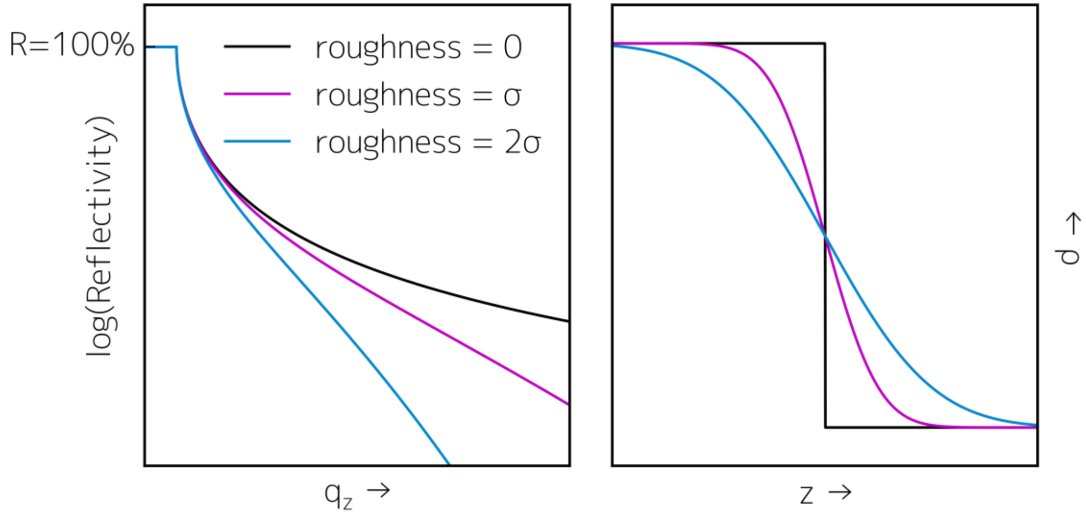
for $q_z > 4q_c$: $R(q_z) \approx \frac{q_c^4}{q_z^4}$



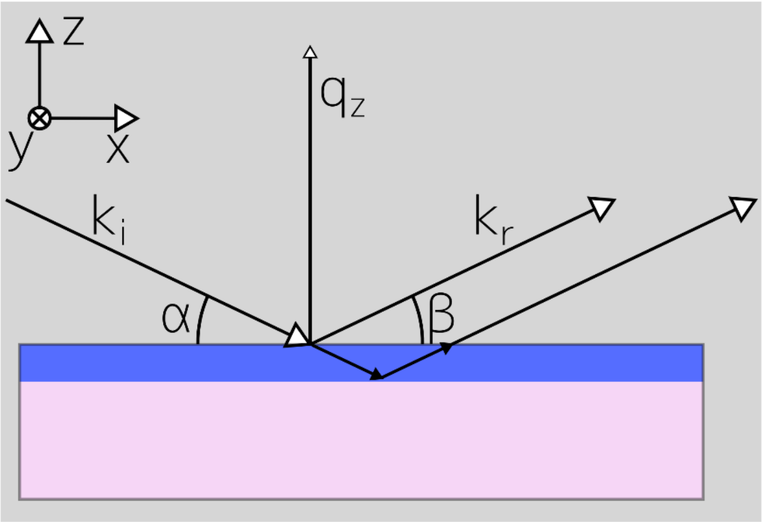
Roughness: Described by Debye-Waller-like factor: $r_{rough} = r \cdot e^{-\frac{q_z^2 \sigma^2}{2}}$

→ Damping of XRR





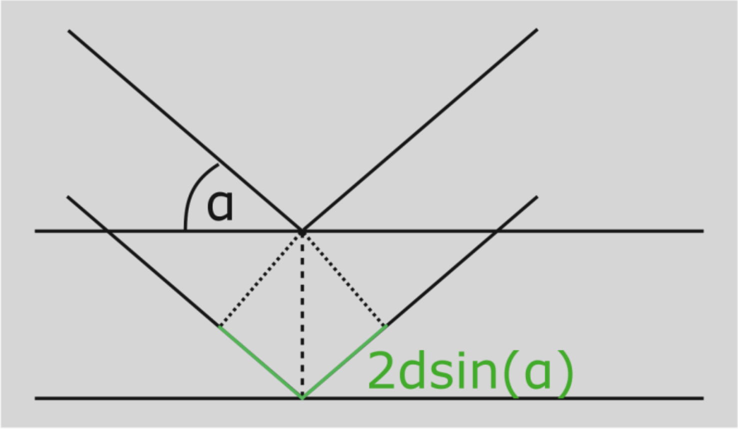
Layered systems



<u>Qualitativelly:</u>

X-ray reflected from different interfaces interfere constructivelly and destructivelly as a function of incoming angle: **Kiessig fringes**

Path length difference changes



Layered systems

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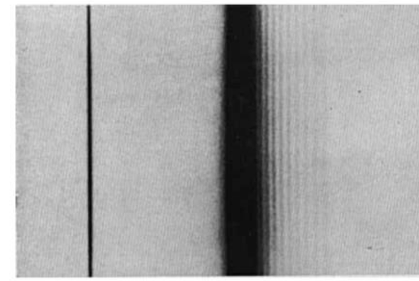
Some history - Kiessig fringes:

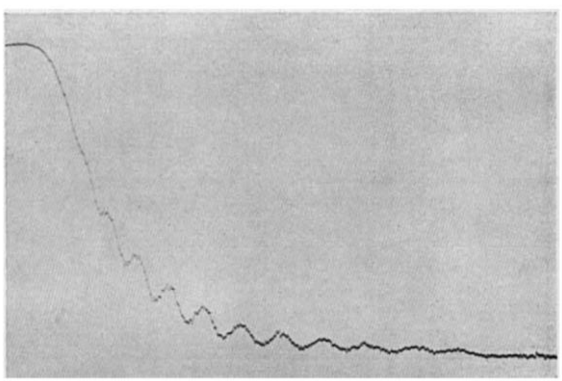
First observed by Heinz Kiessig in 1931 for Ni on glass

"Interferenz von Röntgenstrahlen an dünnen Schichten"

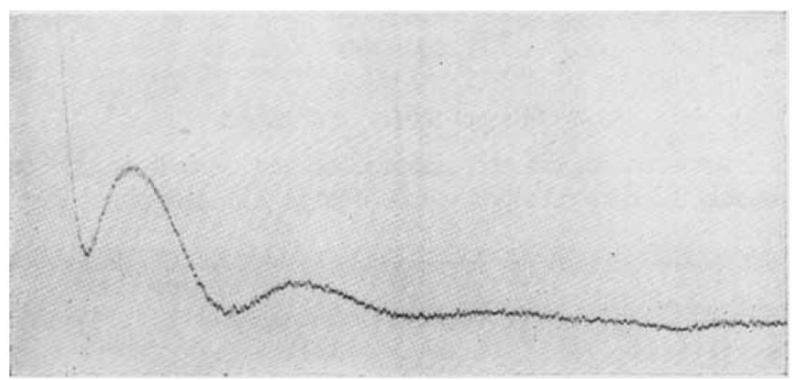
ANNALEN DER PHYSIK, 5. FOLGE, 1931 , BAND 10, HEFT 7

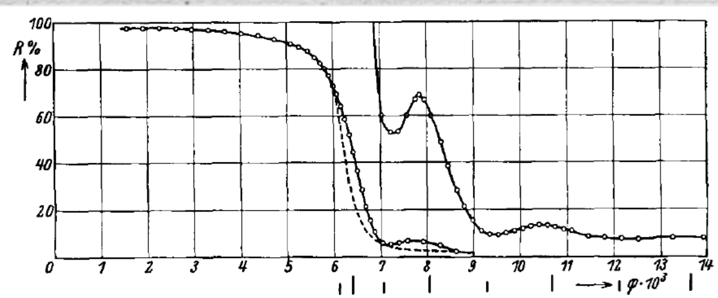


d = 1420 Å

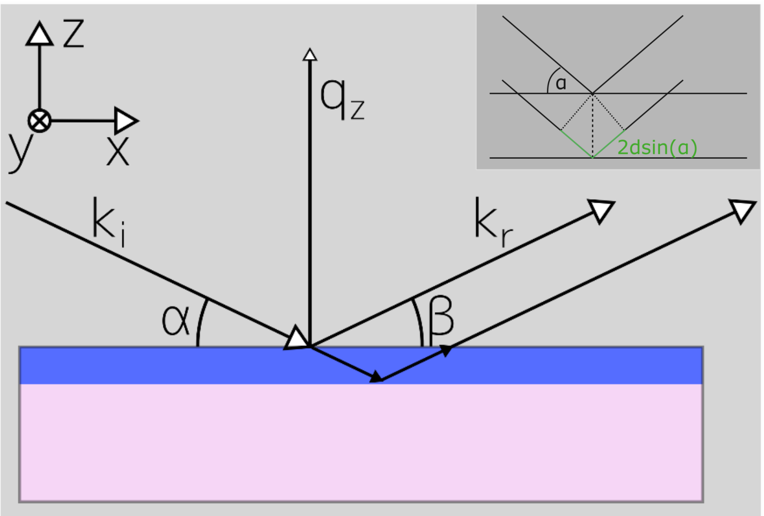


d = 220 Å





Layered systems



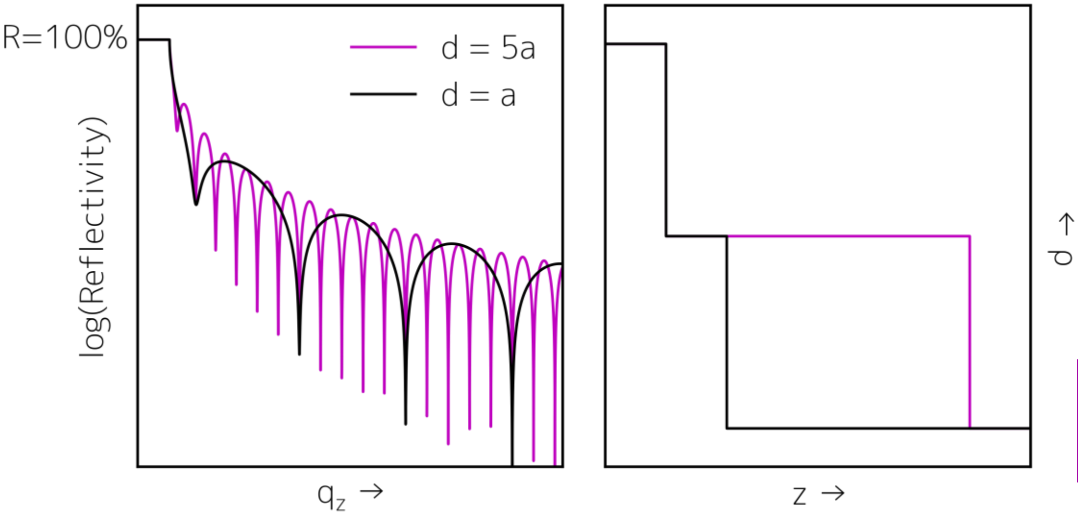
Qualitativelly:

X-ray reflected from different interfaces interfere constructivelly and destructivelly as a function of incoming angle: **Kiessig fringes**

Path length difference changes

Quantitativelly:

- Calculate reflection and transmission coefficient for each layer
- Add up iteratively (Parratt) or Matrix formalism



Single layer:

Phase shift

$$R = \frac{r_{0,1}^2 + r_{1,2}^2 + 2r_{0,1}r_{1,2}\cos 2k_{z1}h}{1 + r_{0,1}^2 r_{1,2}^2 + 2r_{0,1}r_{1,2}\cos 2k_{z1}h}$$

→ **Period** of fringes scales inversely with **thickness** of layers: ***2π/Δq***

Electron density & several layers

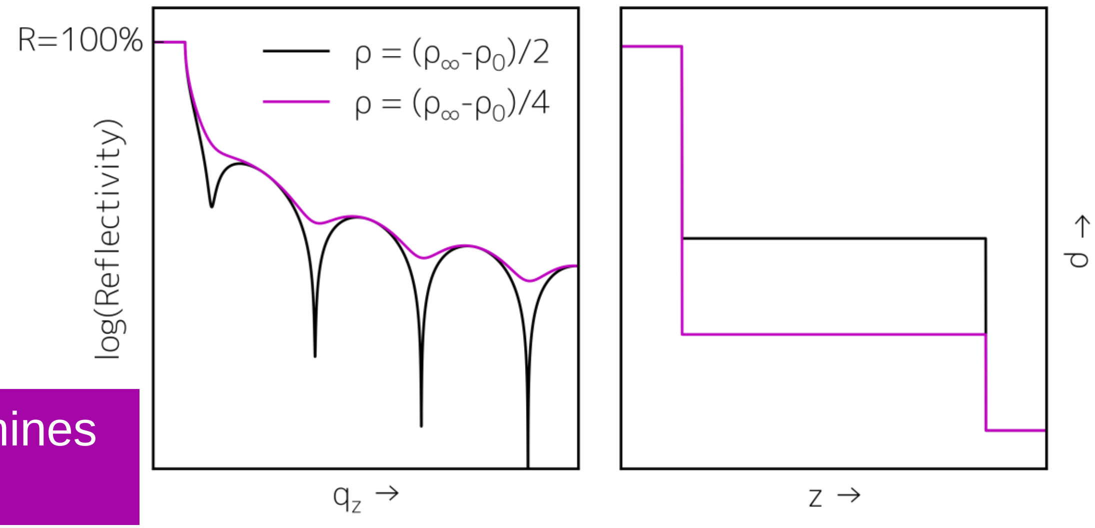
Electron density changes:

$$R = \frac{r_{0,1}^2 + r_{1,2}^2 + 2r_{0,1}r_{1,2}\,\mathbf{\cos 2k_{z1}h}}{1 + r_{0,1}^2 r_{1,2}^2 + 2r_{0,1}r_{1,2}\,\mathbf{\cos 2k_{z1}h}}$$

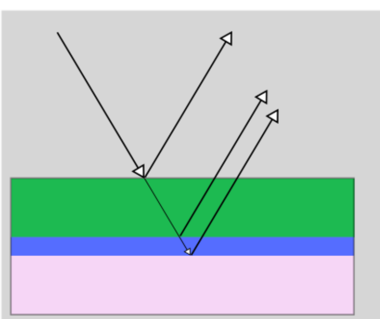
$$r_{j,j-1} = \frac{k_{j,z} - k_{j-1,z}}{k_{j,z} + k_{j-1,z}}, \quad k_j = n_j k_0$$

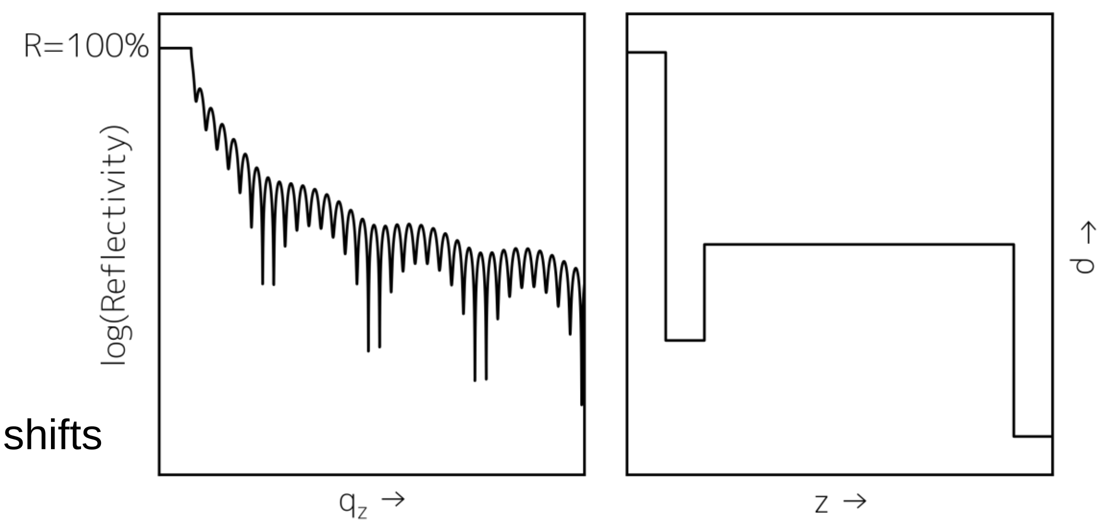
$$n = 1 - \delta - i\beta \qquad \delta = \frac{\lambda^2 r_e \rho_e}{2\pi}$$

→ **Electron density** contrast determines **amplitude** of fringes



Several layers:





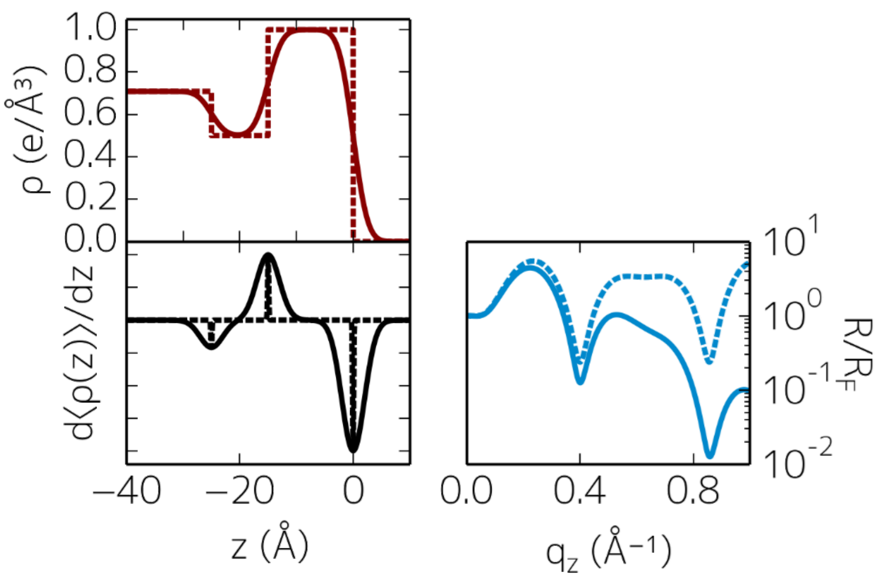
Interference of x-rays reflected from different interfaces → several phase shifts → **beating pattern**

Master formula

Arbitrary density profile:

- Slice into slabs
- Calculate via Master formula

$$\frac{R(q_z)}{R_F(q_z)} \approx \left| \frac{1}{\rho_\infty} \int_{-\infty}^{\infty} dz \frac{\partial \langle \rho(z) \rangle}{\partial z} e^{-iq_z z} \right|^2$$



Born approximation:

- *Easily derivable*
  - No multiple scattering
  - No refraction
  - No absorption
- *Approximated analytical expression*
- *More user friendly*

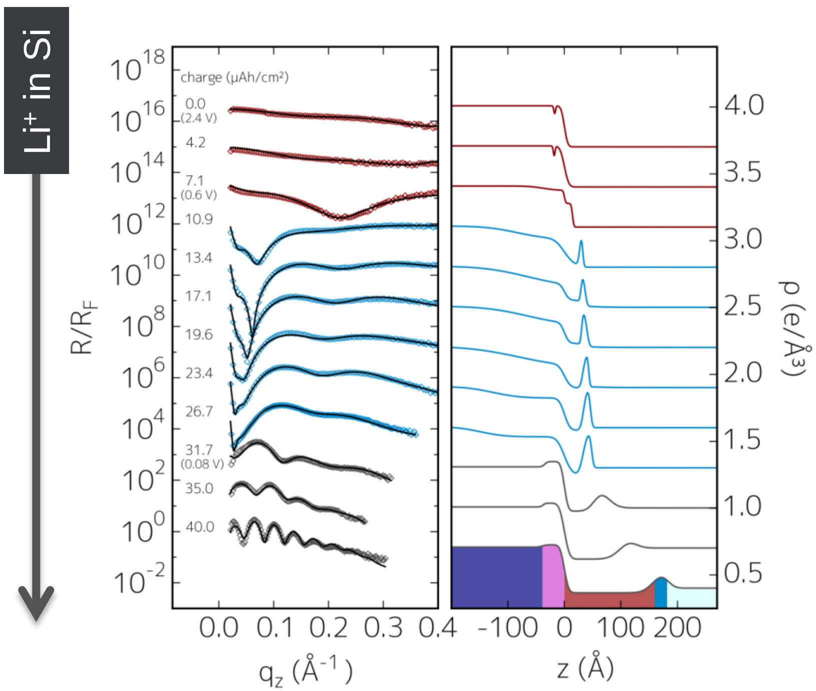
In-situ Study of Si Electrode Lithiation with X-ray Reflectivity

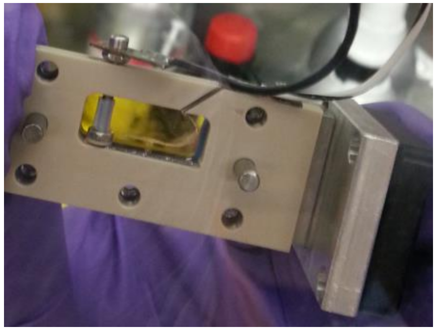
Cao, Steinrück et al., Nano Lett. 16, 7394-7401 (2016) & Adv. Mater. Interfaces 4, 1700771 (2017).

Silicon: A promising high capacity anode for Li-ion batteries

- Theoretical capacity 3580 mAh/g - 10 times higher than graphite
- **BUT**: Volume expansion & other issues limit commercialization

→ *Fundamental understanding of* ***Si lithiation process and SEI***





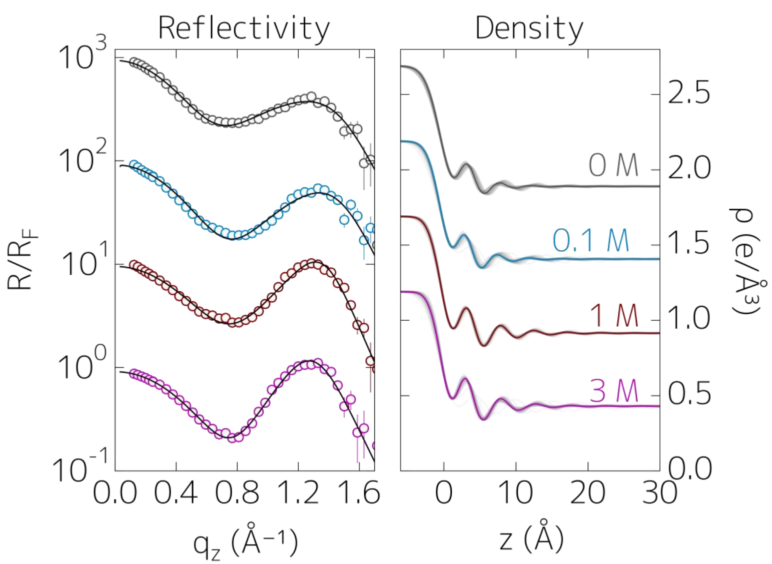
In-situ XRR measured at 12 keV, SSRL BL 2-1

reaction limited, layer-by-layer three-stage lithiation mechanism

Applications

The nanoscale structure of the electrolyte–metal oxide interface

Steinrück et al., Energy Environ. Sci., 11, 594-602 & Nano Lett. 18, 2105-2111 (2018).

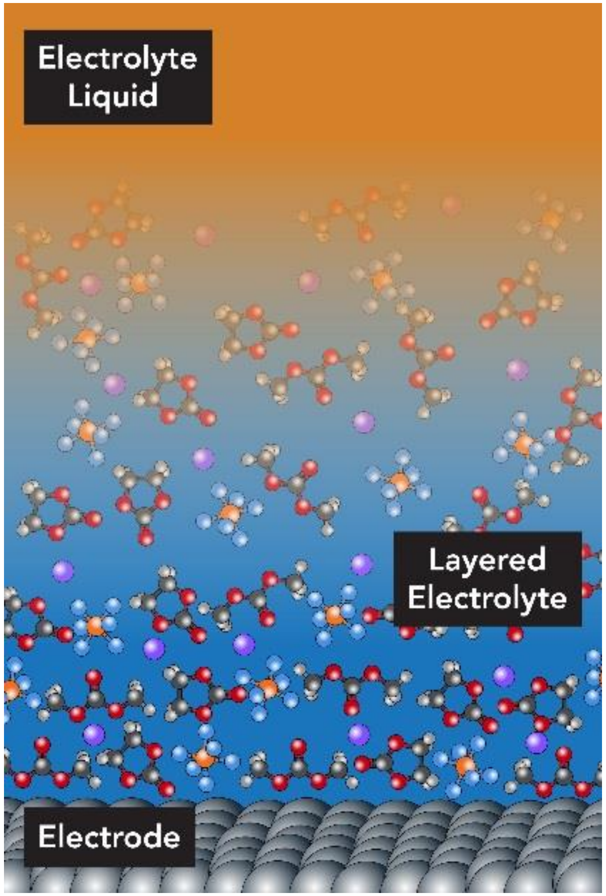


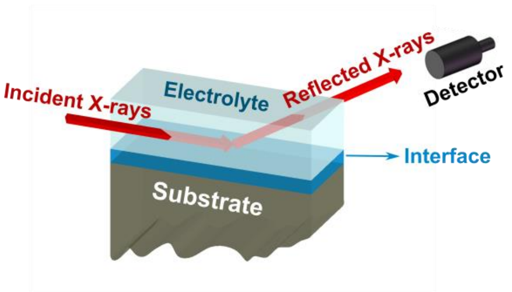
XRR measured at SSRL BL7-2

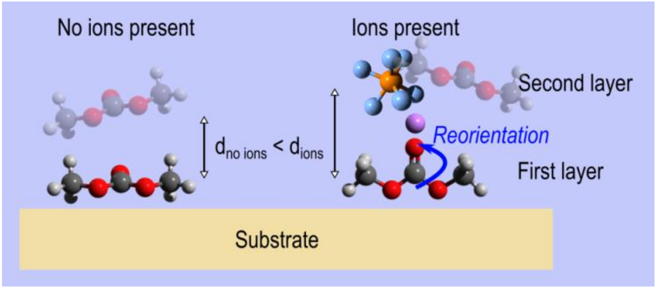
Analysis of XRR with distorted crystal model

→

Double layer formation







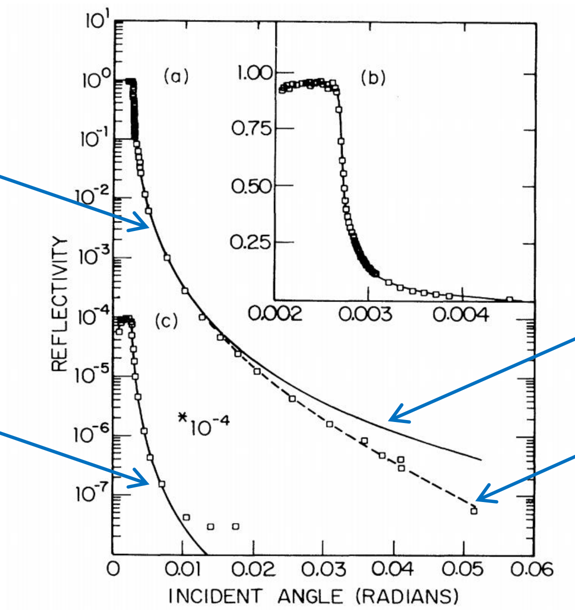
Applications

Surface Roughness of Water Measured by X-Ray Reflectivity

A. Braslau et al., Phys. Rev. Lett. 54, 114 (1985).

☺ Synchrotron experiment

☹ Lab source experiment



Fresnel-XRR

Capillary wave roughened XRR

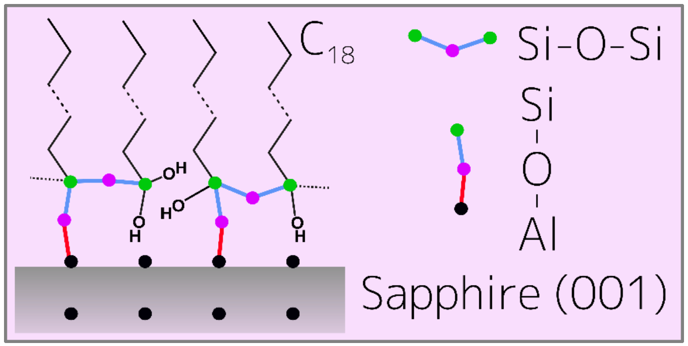
- Roughness of 3.24 Å
- Very close to what is expected from thermally excited capillary waves
- First such measurement of surface roughness of any liquid
- Synchrotron radiation necessary

Applications

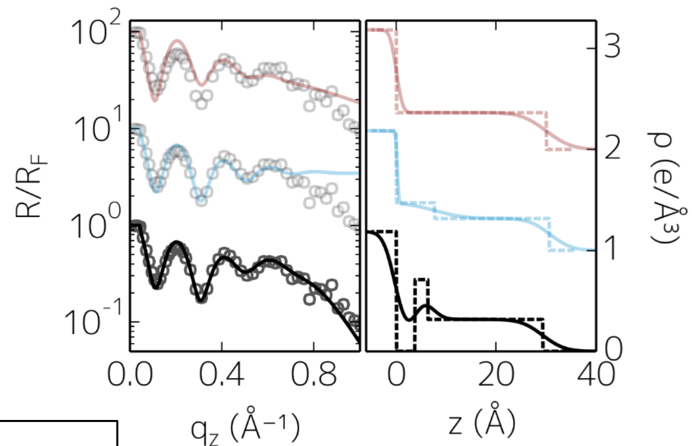
OTS on sapphire: Pseudorotational epitaxy

Steinrück et al., PRL 113, 156101 (2014)

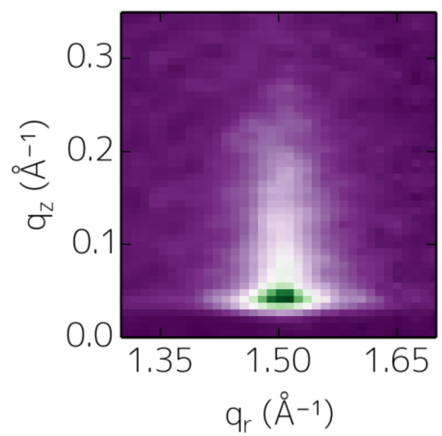
The system



XRR



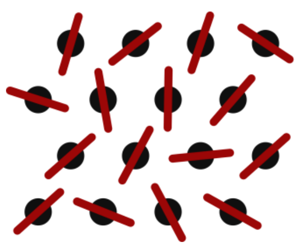
GID



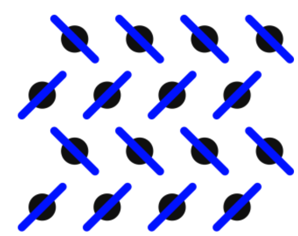
- 3 slab model!
- Molecules vertically aligned
- Packed in a rotator phase
- ***Lattice match with sapphire***

a_{OTS} = 4.82 Å, $a_{sapphire}$ = 4.76 Å

Rotator

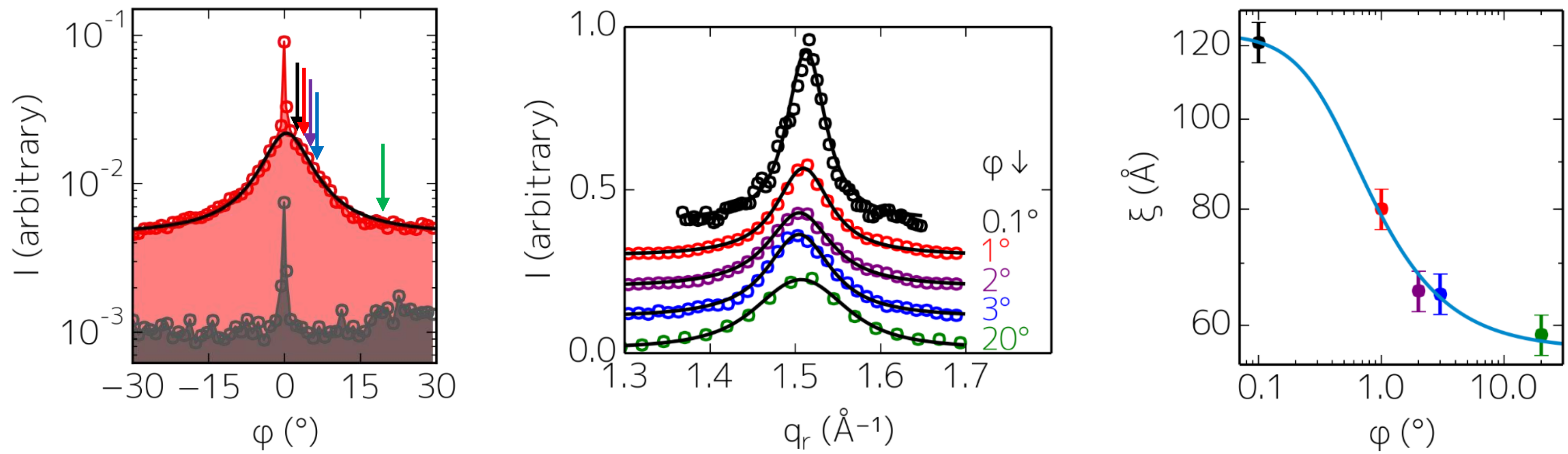


Crystalline



OTS on sapphire: Pseudorotational epitaxy

Steinrück et al., PRL 113, 156101 (2014)

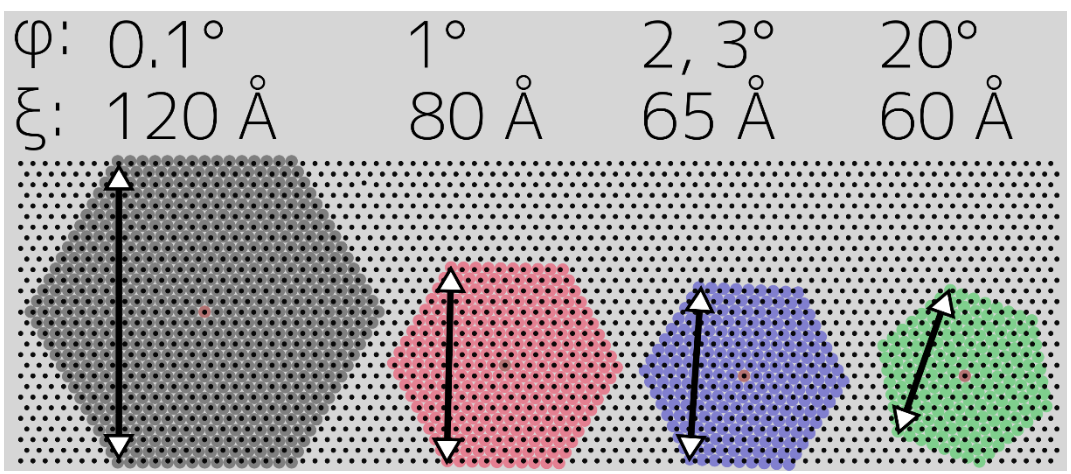


Geometrical model

Critical separation between

corresponding lattice sites

→ ***Loss of coherence length***

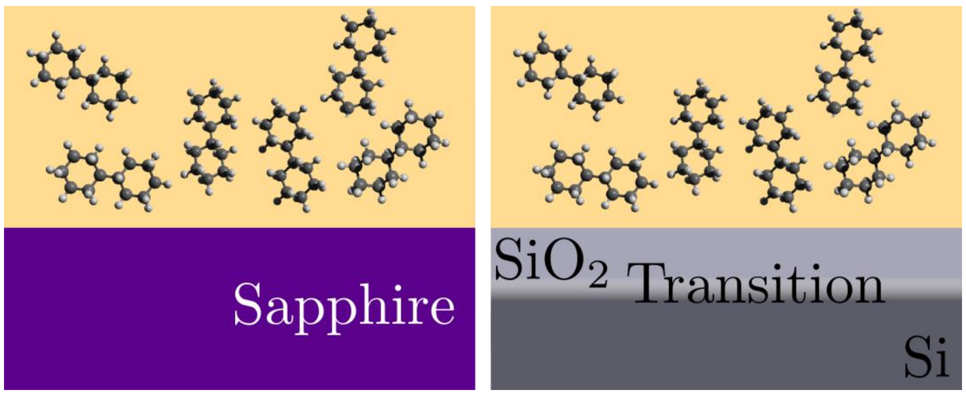


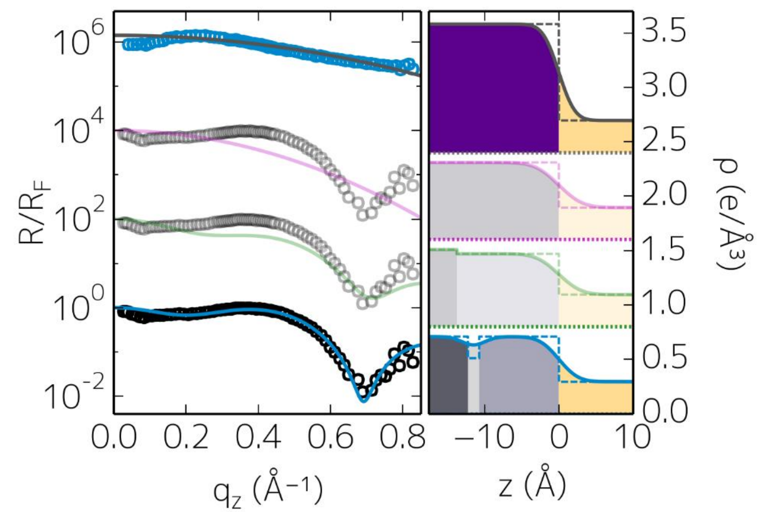
Applications

SLAC

Si/SiO_2 - nanoscale structure

Steinrück et al., ACS Nano 12, 12676 (2014)





<u>Sapphire:</u>

- Single interface

<u>Silicon:</u>

- ~~Single interface~~
- ~~1 slab model (Tidswell [1])~~
- **"Dip" model**

- Si and SiO_2: Bond density mismatch
- Various oxidation states [2]
  - → *Dangling bonds, hydrogen*
- Theory: Low density region [3]
- ~ 6 – 8 missing e^- per unit cell area

[1] Tidswell *et al.*, PRB **41**, 1111 (1990). [2] Braun *et al.*, Surf. Sci **180**, 279 (1987). [3] Tu *et al.*, PRL **84**, 4393 (2000).

Cleaning

- *Essential*: Any surface contamination will effect XRR
- *Very sample specific*: Cleaning methods may also effect sample

Examples:

- Ultrasonication in organic solvents:
  - EAT (Ethanol, Acetone, Toluene (polar and non-polar))
  - Removes organic contamination
- Piranha acid (H_2SO_4 and 30% hydrogen peroxide H_2O_2, *dangerous, use SOP*)
  - Strongly acidic and a strong oxidizer
  - Removes organic contamination
- UV-ozone (dry, simple to use)
  - UV irradiation, creates ozone
  - Decomposes organic matter
- Rinsing with ultra-pure water
- RCA clean (silicon wafer technology, *dangerous, use SOP*)
- Oxygen plasma cleaning

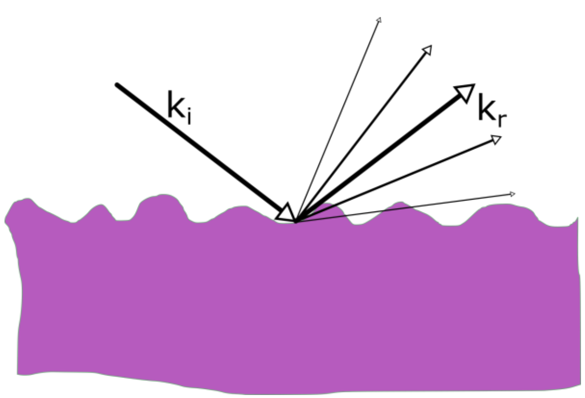
Sample preparation

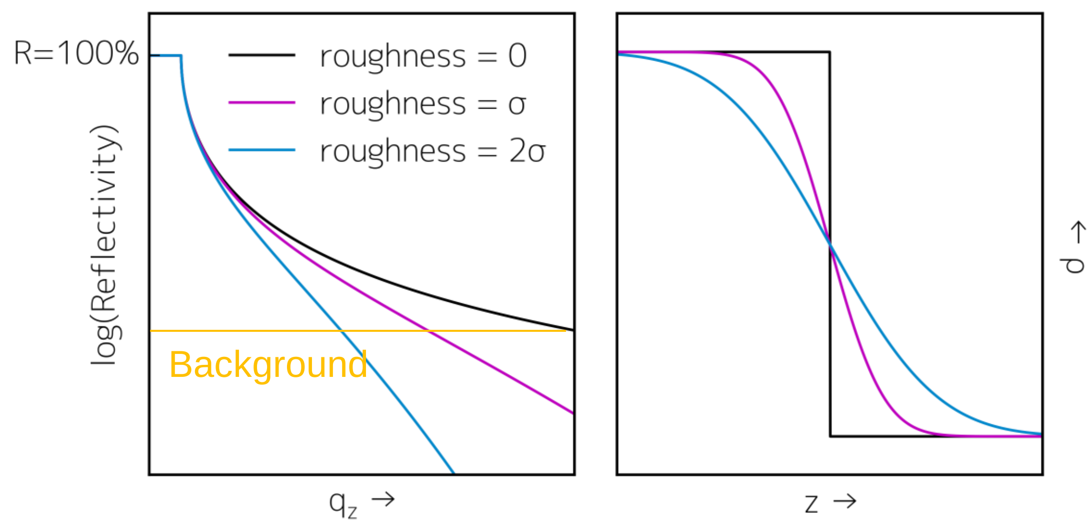
Surface roughness

- Check surface roughness via laboratory XRR, AFM, etc.
- Typically, root-mean-square roughness ≲ 10 Å required for XRR
- ≲ 3 Å required for molecular resolution
- Calculate expected XRR to test feasibility

Roughness: Described by Debye-Waller-like factor: $r_{\text{rough}} = r \cdot e^{-\frac{q_z^2 \sigma^2}{2}}$

→ Damping of XRR



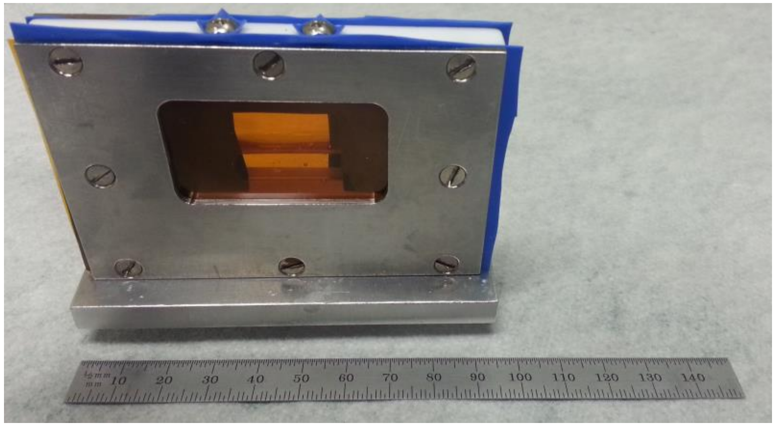


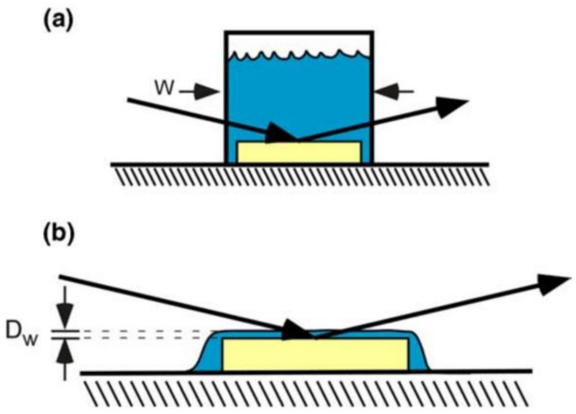
Experimental setup

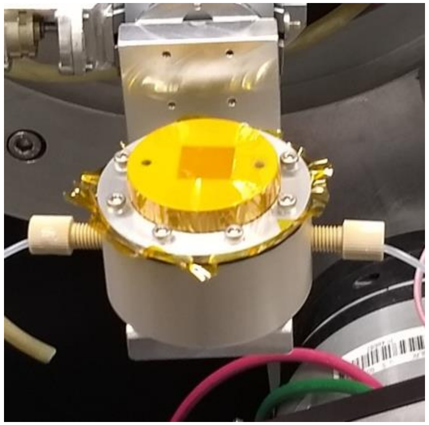
- **Sample size:**
  - Ideally larger than footprint at critical angle, otherwise corrections are necessary
- **Vertical beam size:**
  - Optimize with respect to sample size (footprint)
  - Optimize with respect to diffuse scattering (divergence)
- **Horizontal beam size:**
  - Optimize with respect to possible sample translation (beam damage)
- **X-ray energy:**
  - Higher energy: Less radiation damage (absorption cross-section $\propto 1/E^3$, valid up to ≈ 30 keV, where Compton dominates), spec. organics
  - Lower energy: Larger angles, mostly important for footprint effects
  - Consider absorption edges of compounds in sample
  - Consider transmission through e.g. liquid

Sample environment

- **Solid-vapor interfaces:**
  - Use Helium or Nitrogen environment
  - Reduces beam damage (oxidation)
  - Reduces background (air scattering)
- **Solid-liquid interfaces:**
  - Significant background/absorption from bulk liquid scattering (signal to noise)
  - Minimize transmission length (convoluted with footprint problem)
  - Use thin-film cells to minimize liquid scattering (Fenter et al., Prog. Surf. Sci. 77, 171–258 (2004))
  - Use film-stabilizing agent (e.g. polymer, Petach et al., ACS Nano 10, 4565−4569 (2016))

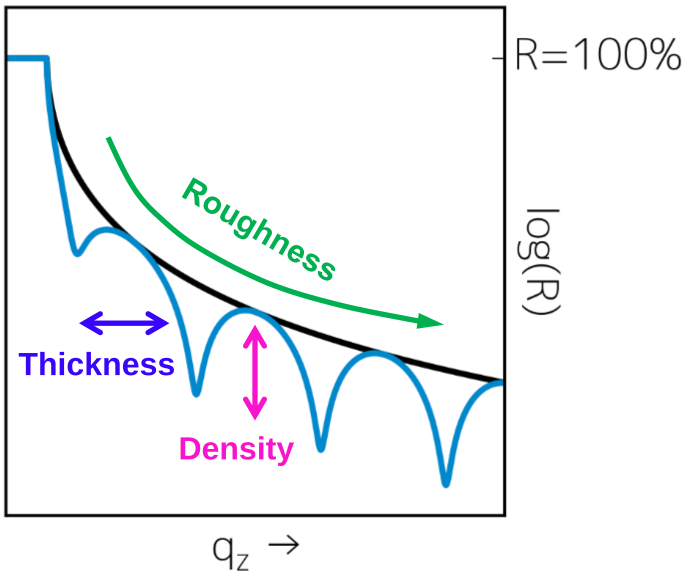






Take-away messages

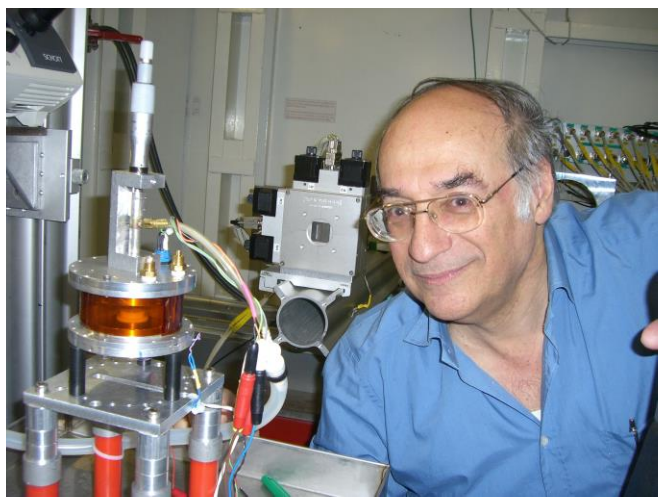
- Surfaces & interfaces are important & interesting
- X-ray scattering can be extremely surface sensitive (by choosing right geometry)
- Ideal for *in-situ* studies – buried interfaces
- Several techniques are sensitive to different information
  - Off-specular scattering: Lateral roughness
  - GID: Crystallinity of adlayers
  - GISAXS: Morphology of adlayers
  - CRTs: Crystalline surfaces
  - **XRR: Surface normal density profile**
    - ***Thickness***
    - ***Density***
    - ***Roughness***

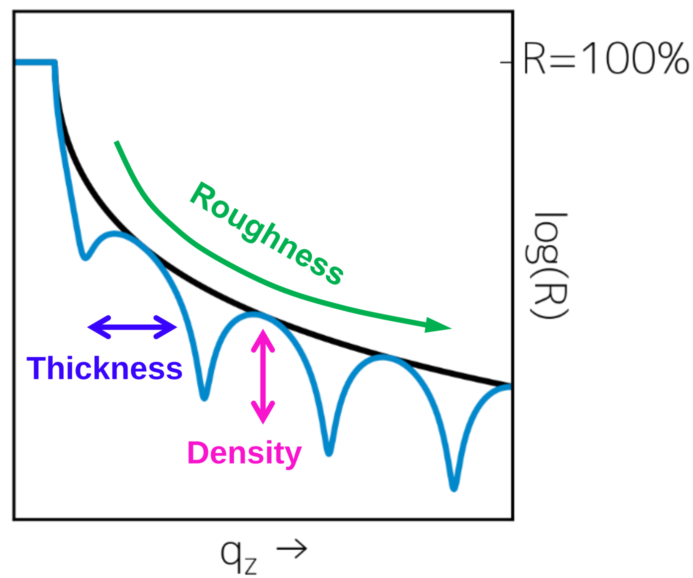


Take-away messages

Acknowledgements:

- XRS 2018 organizing chairs, support from the DOE-BES
- Toney group, Chuntian Cao, Mike Toney
- SSRL engineers, beamline engineers and scientists





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National Laboratory

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Bar-Ilan University,
Isreal

Literature

SLAC

General x-rays:

- J. Als-Nielsen and D. McMorrow, Elements of modern X-ray physics (John Wiley & Sons, New York, USA, 2011)
- D. S. Sivia, Elementary scattering theory: For X-ray and neutron users (Oxford University Press, New York, USA, 2011)

XRR & GID & off-specular diffuse scattering:

- M. Deutsch and B. M. Ocko, in Encyclopedia of Applied Physics, edited by G. L. Trigg (VCH, New York, USA, 1998), Vol. 23
- J. Daillant and A. Gibaud, X-ray and Neutron Reflectivity: Principles and Applications (Springer, Berlin, Germany, 2009)
- P. S. Pershan and M. Schlossman, Liquid Surfaces and Interfaces: Synchrotron X-ray Methods (Cambridge University Press, Cambridge, UK, 2012)
- M. Tolan, X-ray scattering from soft-matter thin films (Springer, Berlin, Germany, 1999)
- K. Kjaer, Physica B 198, 100 (1994)
- J. Als-Nielsen et al., Phys. Rep. 246, 251 (1994)
- S. K. Sinha et al., Physical Review B 38, 2297 (1988)

useful webpage: http://www.reflectometry.net/ (by Prof. Adrian R. Rennie, Uppsala, Sweden)

GISAXS:

- G. Renaud et al., *Surface Science Reports* 64, 255-380 (2009)
- P. Müller-Buschbaum, Anal Bioanal Chem. 376, 3-10, (2003)
- P. Müller-Buschbaum, Materials and Life Sciences Lecture Notes in Physics 776, 61-89 (2009)

useful webpage: http://gisaxs.com/ (by Dr. Kevin Yager, BNL)

Crystal truncation rods:

- R. Feidenhans'l, Surface Science Reports 10, 105-188 (1989)
- I. K. Robinson and D. J. Tweet, Rep. Prog. Phys. 55, 599 (1992)

Thank you for your attention !

Links

Links to X-ray analysis software:

Motofit:

https://sourceforge.net/projects/motofit/

by Dr. Andrew Nelson, ANSTO, Australia

reference: A. Nelson, J. Appl. Crystallogr. 39, 273 (2006)

GenX:

http://genx.sourceforge.net/

by Dr. Matts Björck, Swedish Nuclear and Fuel Management Company, Sweden

reference: M. Björck and G. Andersson, J. Appl. Crystallogr. 40, 1174 (2007)