

F7030 Rentgenový rozptyl na tenkých vrstvách

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Prezentace k přednášce
Numerické simulace
Příklady experimentů
Vybrané vztahy

Syllabus

1. Experimentální technika: zdroje, vznik rtg záření, goniometry, optické prvky (monochromátory, kolimátory, zrcadla, fokusační optika), detektory. Základní experimenty: polykrystalové a monokrystalové metody, mapování reciprokého prostoru
2. Kinematická teorie rozptylu: úvod do teorie rozptylu, rozptyl na elektronu, izolovaném atomu, krystal, strukturní a geometrický faktor, omezená velikost krystalu
3. Difrakce na polykrystalech I: strukturní faktor, velikost krystalitu (Scherrerova formule), vliv deformace na polohy a šířky difrakčních maxim, zbytková napětí, kvantifikace fázového složení (vnitřní normál)
4. Polykrystaly II: Full profile fitting; Texture, ODF (orientation distribution function); Debyeův vztah, PDF (pair distribution function).
5. SAXS: teoretický popis, řídké roztoky – Guinierův a Porodův vztah, uspořádané částice – long range a short-range order
6. Dokonalé, téměř dokonalé krystaly, epitaxní vrstvy: Kinematická teorie na monokrystalu a epitaxní vrstvě – polohy difrakcí, truncation rod, deformace v epitaxní vrstvě, relaxace. Mozaikový krystal
7. Dynamická teorie rtg reflexe: Jednovlnná aproximace – hloubka vniku, reflexe na hladkém rozhraní, multivrstvy (formalismus přenosové matice), TRXRF
8. Dynamická teorie rtg difrakce: Dvojevlnná aproximace: případ Bragg a Laue, Borrmannův jev, stojatá vlna, GID, epitaxní vrstvy
9. Semikinematická teorie I: DWBA, Rozptyl na drsných rozhraních – popis drsného rozhraní, příklady: fraktálové rozhraní, dvouúrovňové, vicinální, spekulární odraz a nespekulární rozptyl, drsné multivrstvy
10. Semikinematická teorie II: GISAXS na částicích na povrchu a uvnitř vzorku, Difuzní rozptyl na defektech v krystalu v okolí difrakce
11. Experimentální rozlišení Experimentální rozlišení v reciprokém prostoru: analyzer streak, detector streak, monochromator streak, DuMondovy grafy, disperzní a nedisperzní uspořádání, koherenční šířka a délka
12. Další rentgenové metody: Fluorescenční spektroskopie, absorpční spektroskopie – XAFS, XMCD.

Dynamická teorie

$$(\Delta + K^2)E(\mathbf{r}) = \hat{V}(\mathbf{r})E(\mathbf{r})$$

$$\hat{V}(\mathbf{r}) = \text{graddiv} - K^2\chi(\mathbf{r})$$

Řešení v periodickém potenciálu
ve tvaru Blochovy vlny

$$D(\mathbf{r}) = \varepsilon_0(1 + \chi(\mathbf{r}))E(\mathbf{r})$$

$$E(\mathbf{r}) = \sum_{\mathbf{g}} E_{\mathbf{g}} e^{i\mathbf{k}_{\mathbf{g}} \cdot \mathbf{r}}$$

$$K^2 E_{\mathbf{g}} - \mathbf{k}_{\mathbf{g}} \times (E_{\mathbf{g}} \times \mathbf{k}_{\mathbf{g}}) = -K^2 \sum_{\mathbf{p}} \chi_{\mathbf{p}} E_{\mathbf{g}-\mathbf{p}}$$

$$(K^2 - k_{\mathbf{g}}^2) D_{\mathbf{g}} = \mathbf{k}_{\mathbf{g}} \times (\mathbf{k}_{\mathbf{g}} \times \sum_{\mathbf{p}} \chi_{\mathbf{p}} D_{\mathbf{g}-\mathbf{p}})$$

Dynamická teorie

Okrajové podmínky

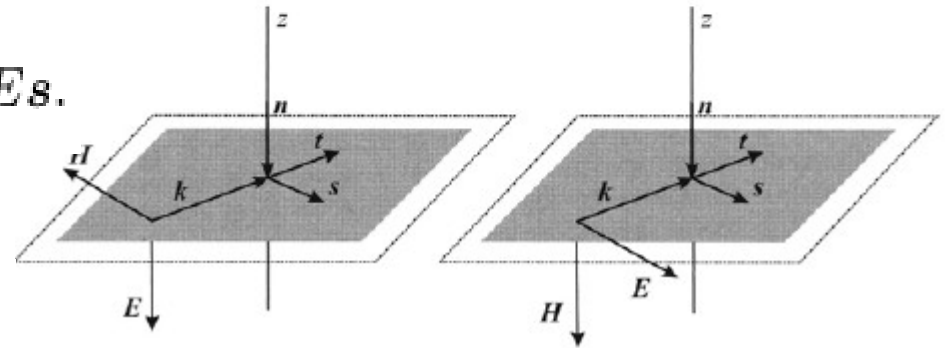
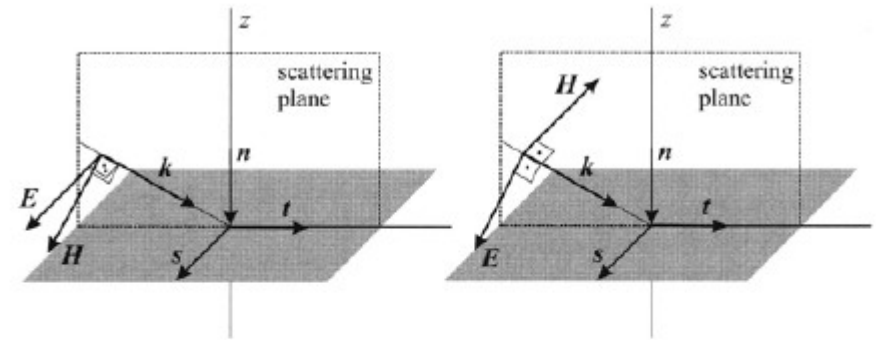
$$\mathbf{H} = \frac{c\epsilon_0}{K} \mathbf{k} \times \mathbf{E};$$

S-polarizace

$$\mathbf{E}_{\parallel} = E s, \quad \mathbf{H}_{\parallel} = \frac{k_z}{K} \epsilon_0 c E t.$$

P-polarizace

$$\mathbf{E}_{\parallel} \approx \frac{k_z}{K} E t, \quad \mathbf{H}_{\parallel} \approx -\epsilon_0 c E s.$$



$$\sum_n E_n^a s_n e^{i\mathbf{k}_n^a \cdot \mathbf{r}} \Big|_{\mathbf{r} \in S} = \sum_n E_n^b s_n e^{i\mathbf{k}_n^b \cdot \mathbf{r}} \Big|_{\mathbf{r} \in S},$$

$$\sum_n E_n^a k_{zn}^a t_n e^{i\mathbf{k}_n^a \cdot \mathbf{r}} \Big|_{\mathbf{r} \in S} = \sum_n E_n^b k_{zn}^b t_n e^{i\mathbf{k}_n^b \cdot \mathbf{r}} \Big|_{\mathbf{r} \in S},$$

Koplanární symetrická zjednodušení:

$$\sum_n E_n^a e^{i\mathbf{k}_n^a \cdot \mathbf{r}} \Big|_{\mathbf{r} \in S} = \sum_n E_n^b e^{i\mathbf{k}_n^b \cdot \mathbf{r}} \Big|_{\mathbf{r} \in S}$$

Dynamická teorie

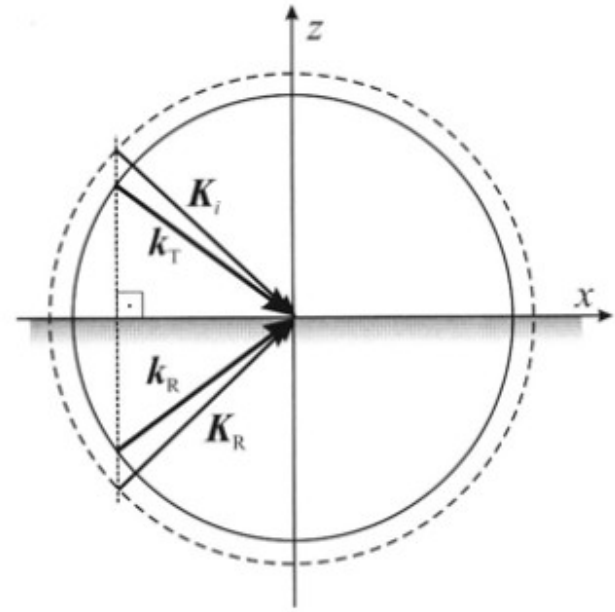
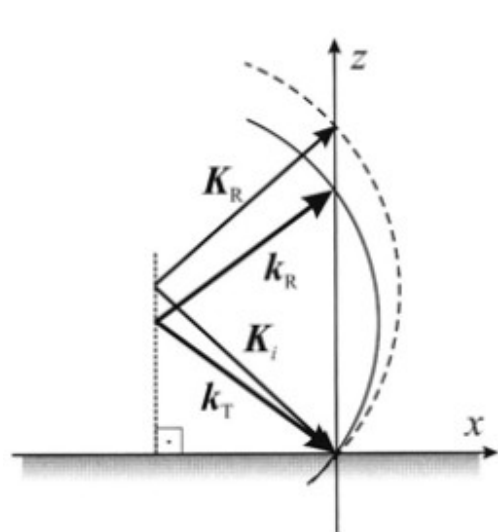
Jednovlnná aproximace

$$\mathbf{E}_0 e^{i\mathbf{k}_0 \cdot \mathbf{r}}$$

$$(k^2 - k_0^2) \mathbf{E}_0 = 0$$

$$k_0 = k \equiv nK = K \sqrt{1 + \chi_0} \approx K(1 + \chi_0/2)$$

$$\alpha_c \approx \sqrt{-\text{Re}(\chi_0)} = \lambda \sqrt{\frac{r_{\text{el}}}{\pi} \langle \rho(\mathbf{r}) \rangle}$$



Okrajové podmínky

$$E_T^a + E_R^a = E_T^b + E_R^b, \quad k_{Tz}^a (E_T^a - E_R^a) = k_{Tz}^b (E_T^b - E_R^b)$$

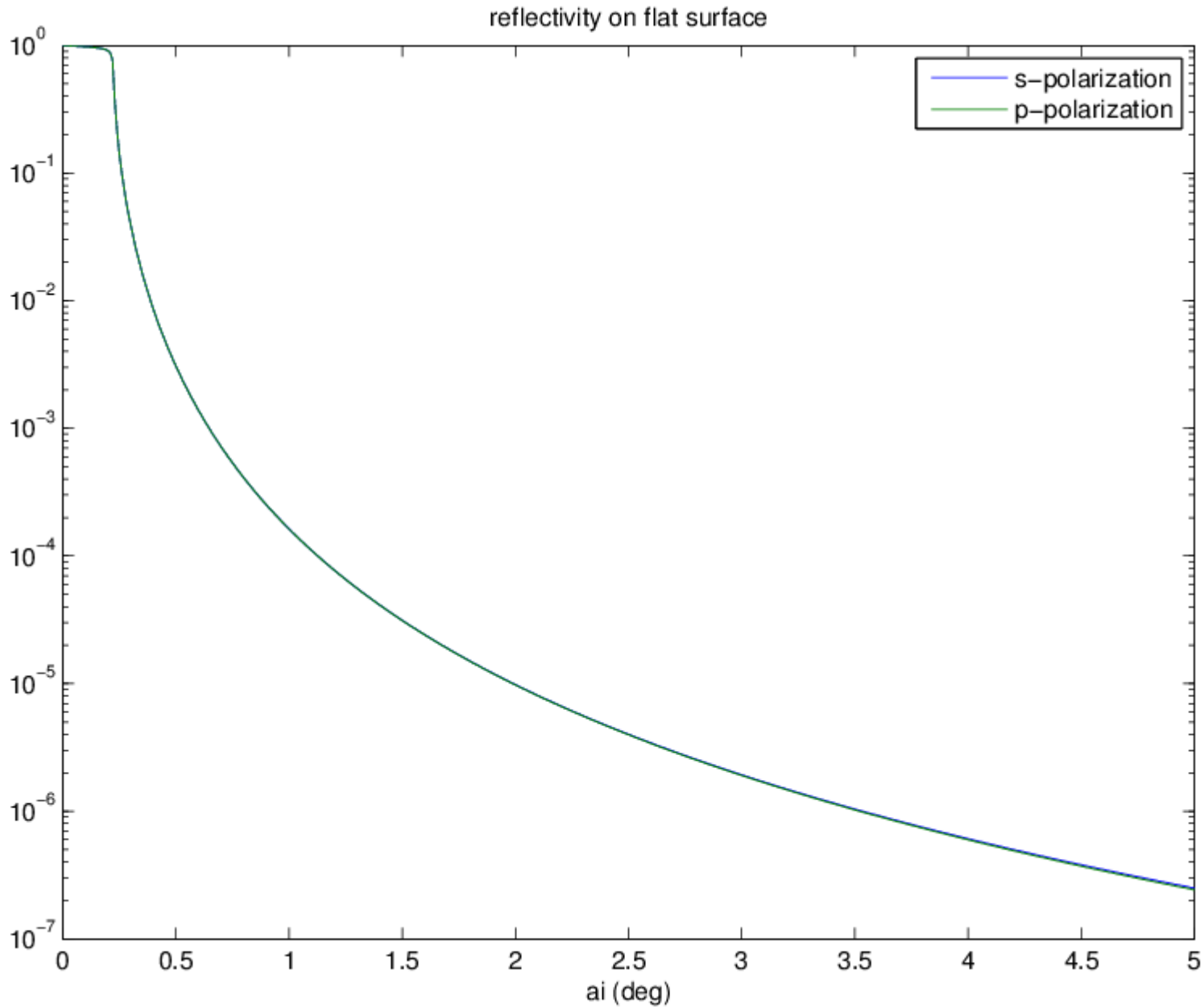
Polonekonečný substrát

$$E_i + E_R = E_T, \quad K_{iz} (E_i - E_R) = k_{Tz} E_T$$

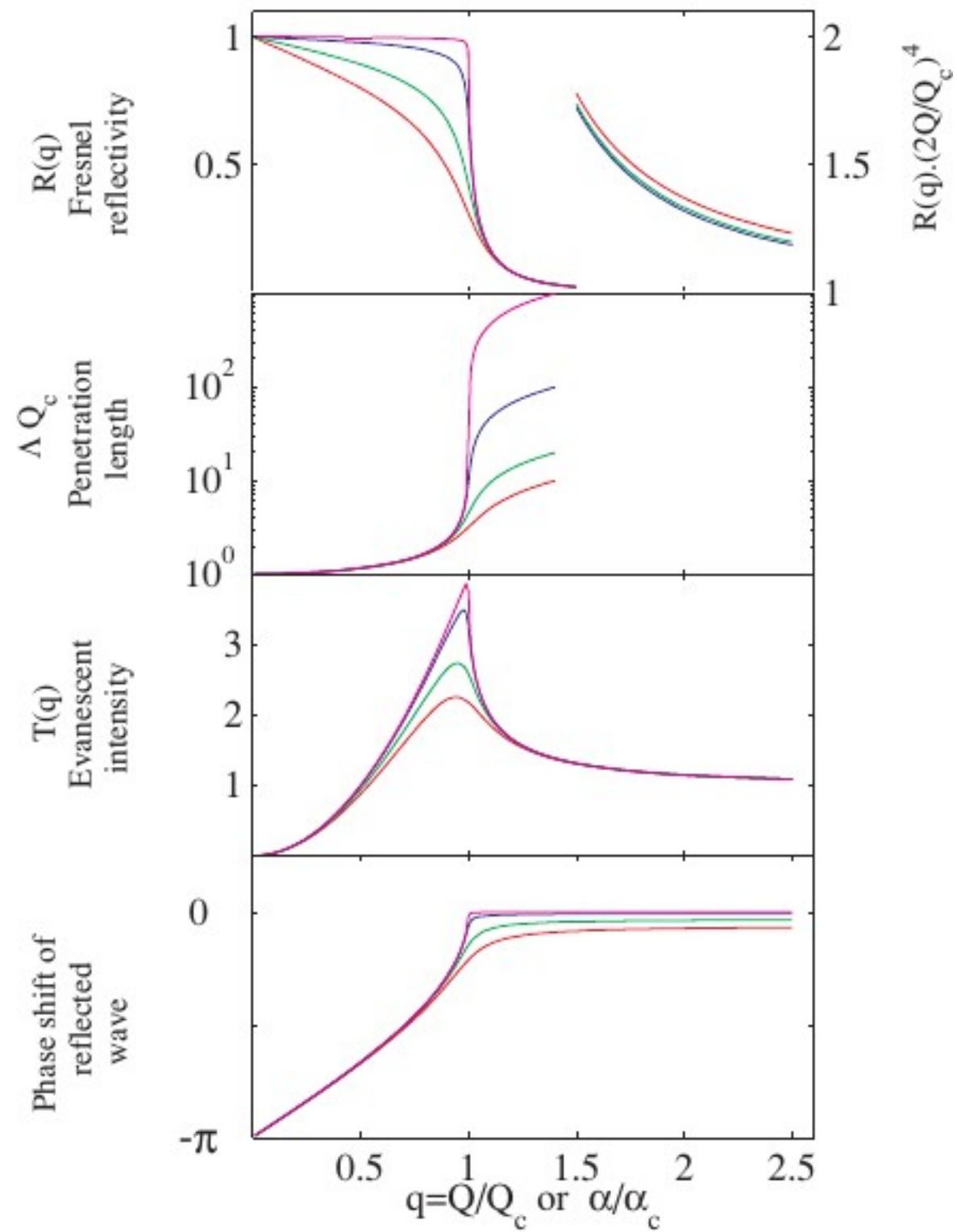
$$r = \frac{E_R}{E_i} = \frac{K_{iz} - k_{0z}}{K_{iz} + k_{0z}}, \quad t = \frac{E_T}{E_i} = \frac{2K_{iz}}{K_{iz} + k_{0z}}$$

Dynamická teorie

Odraz polonekonečném hladkém substrátu v jednovlnné aproximaci



The values of $b_\mu (= 2\mu k/Q_c^2)$ used were: 0.001, 0.01, 0.05, 0.1.



X-ray standing waves

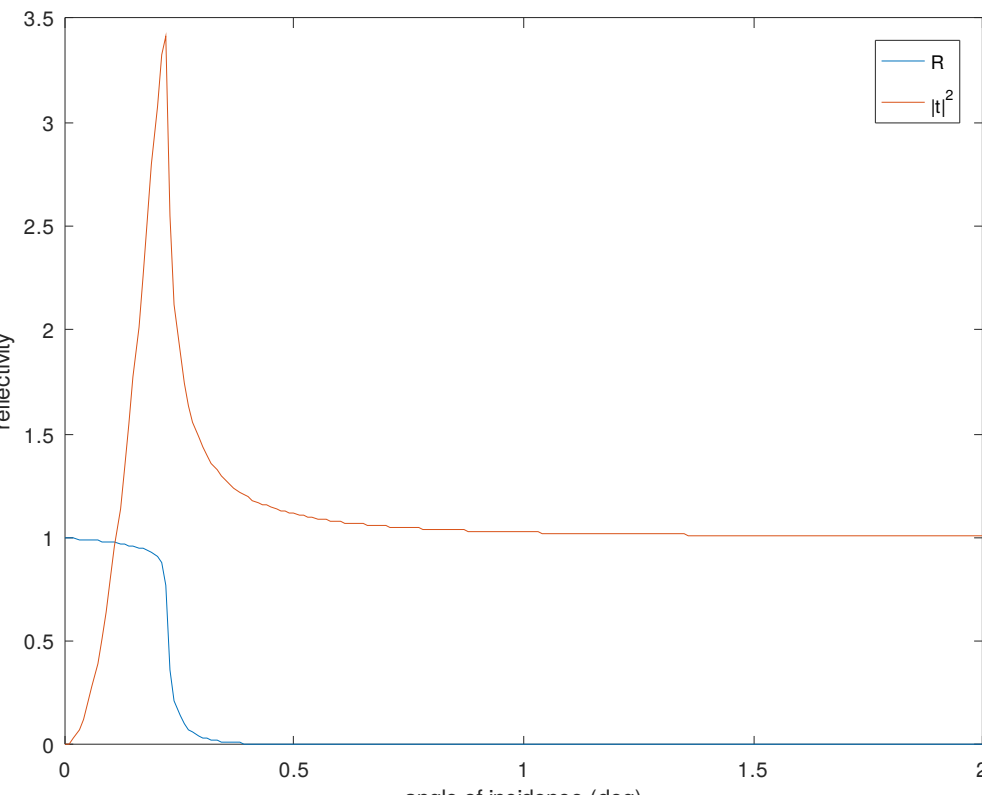
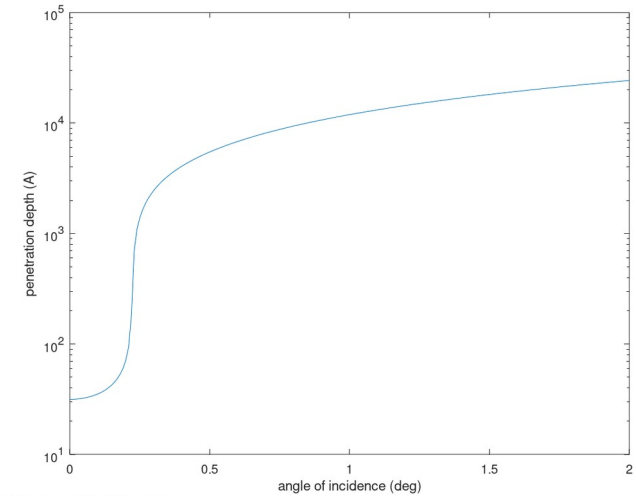
Total x-ray reflection

Above surface

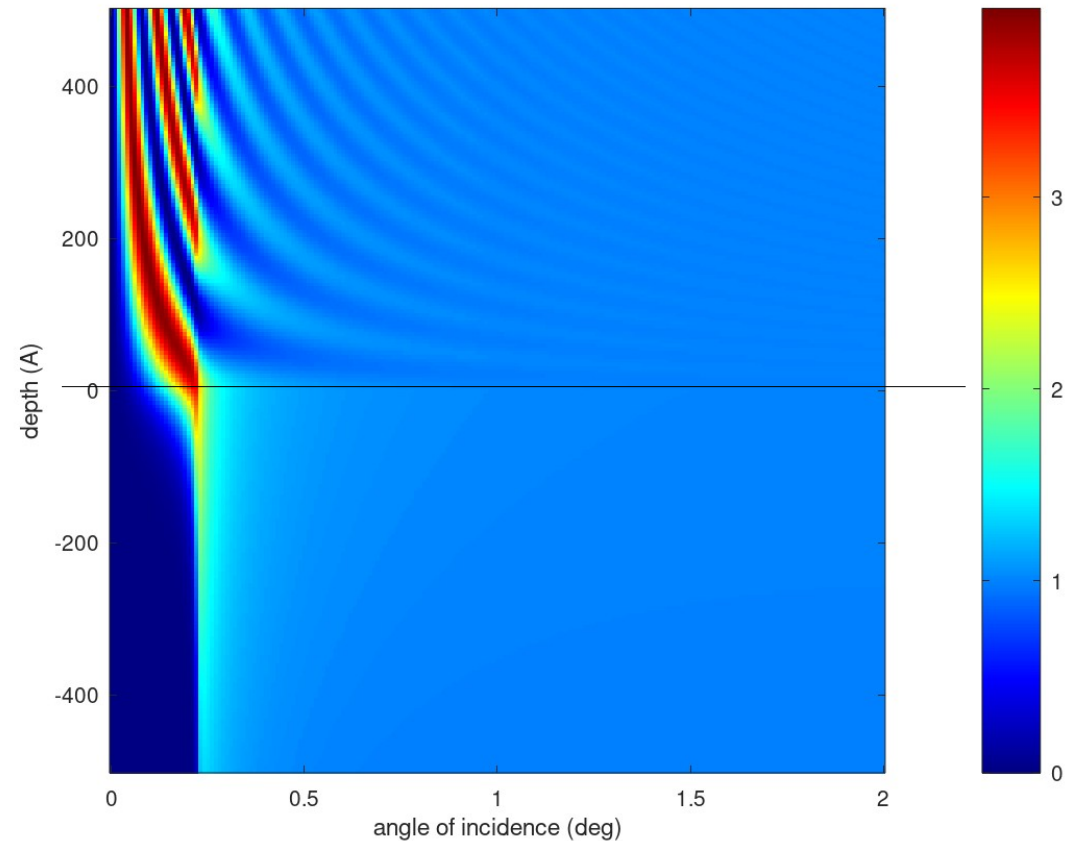
$$\exp(i K_z z) + r \exp(-i K_z z)$$

Below surface

$$t \exp(i k_z z)$$



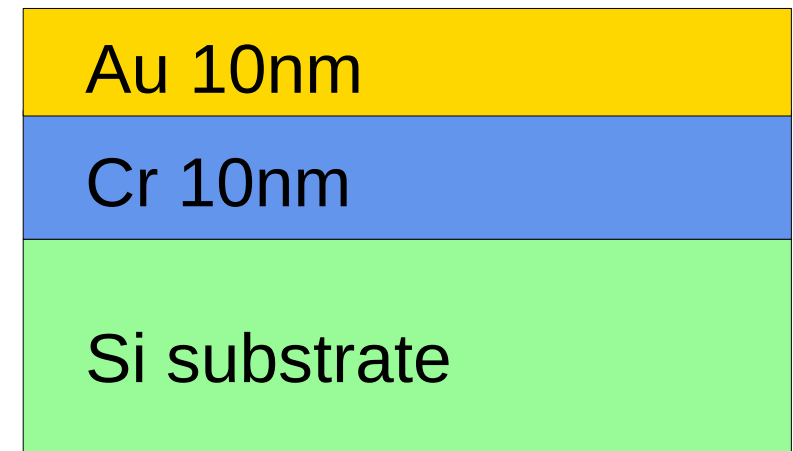
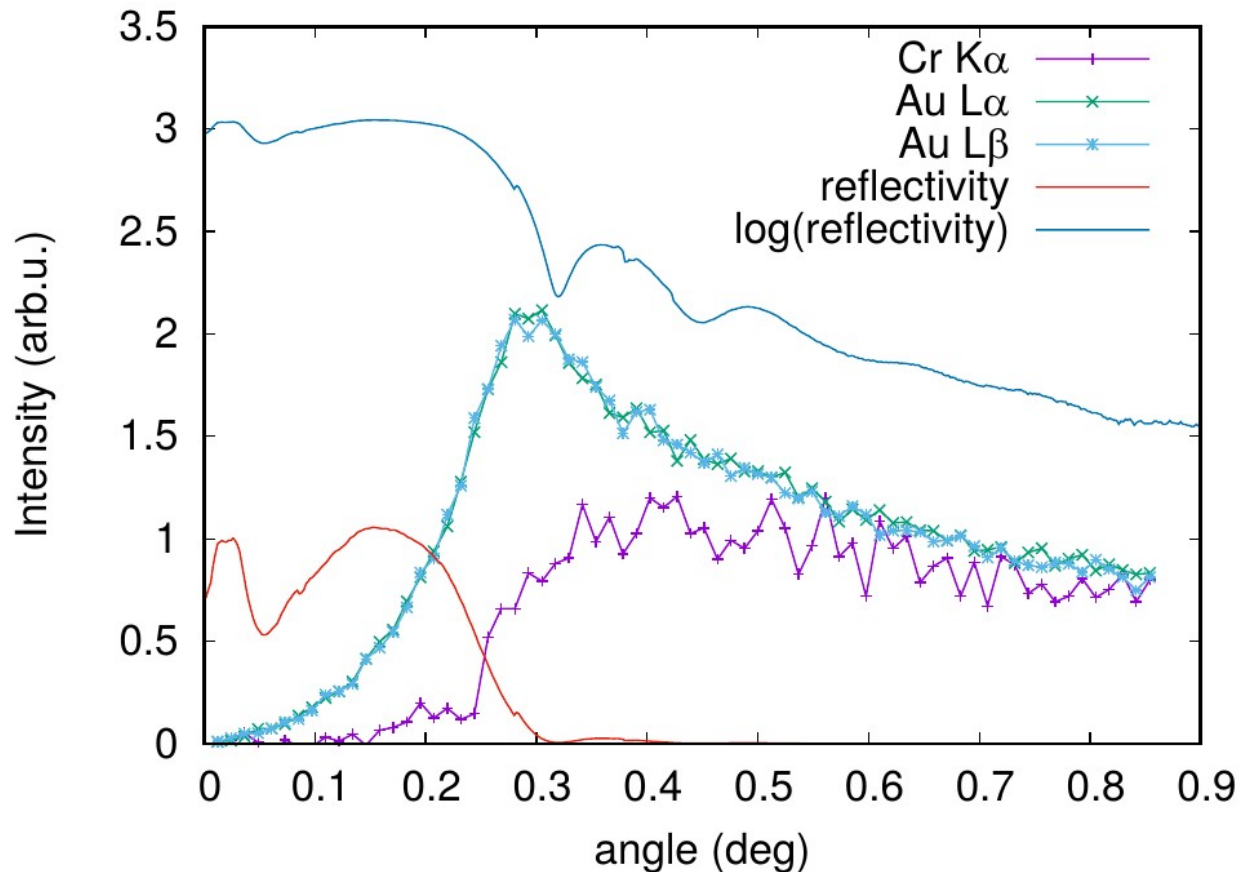
Intensity distribution



X-ray standing waves

Total reflection

Testing sample (Csaba Morvay)



Dynamická teorie

Odraz na vrstvě v jednovlnné aproximaci

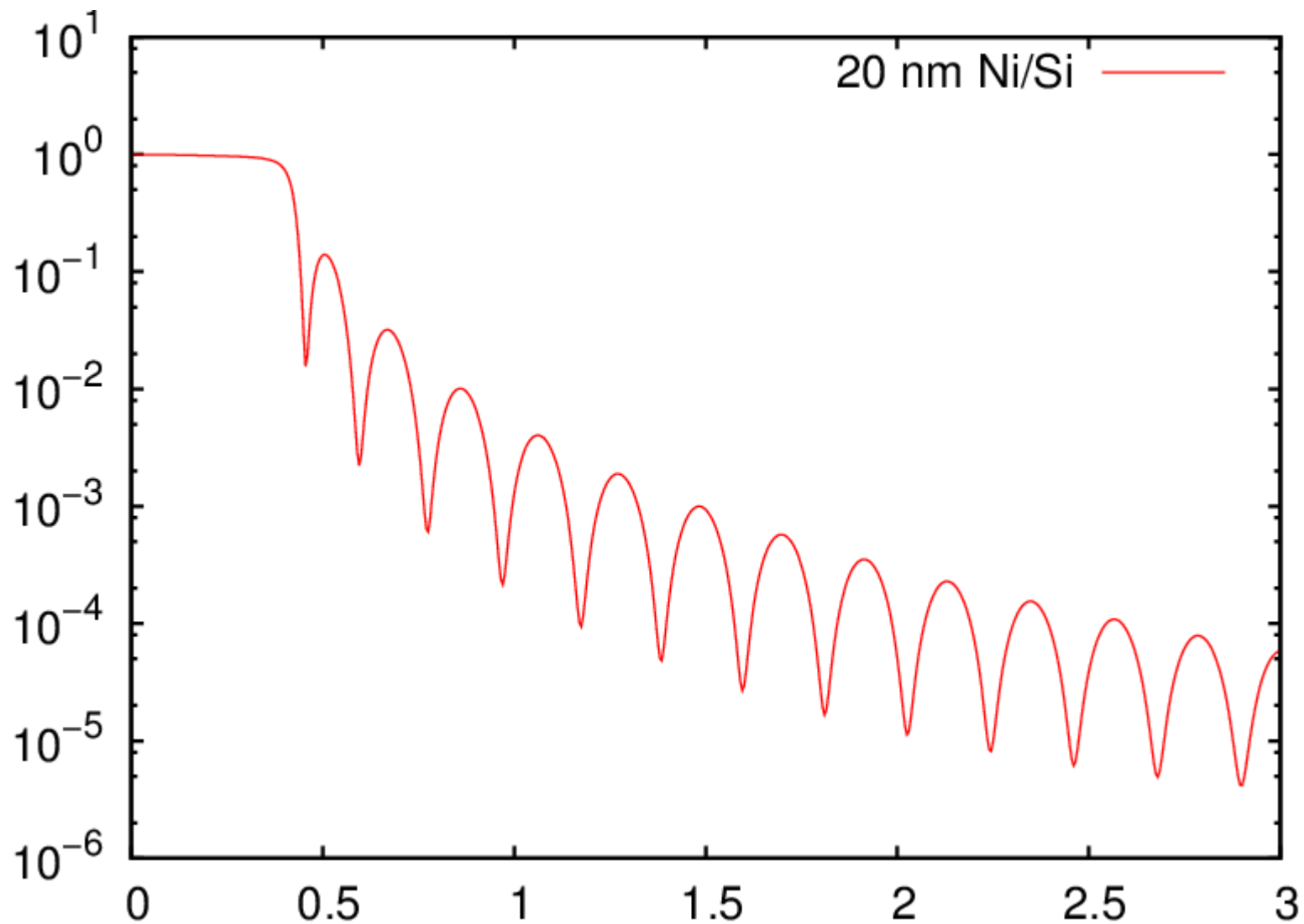
$$\mathcal{R} = \left| \frac{r_1 + r_2 e^{-2ik_0zT}}{1 + r_1 r_2 e^{-2ik_0zT}} \right|^2$$

$$2T \sqrt{\sin^2 \alpha_{im} - \sin^2 \alpha_c} = m\lambda$$

$$\sin \alpha_c = \sqrt{2(1-n)}$$

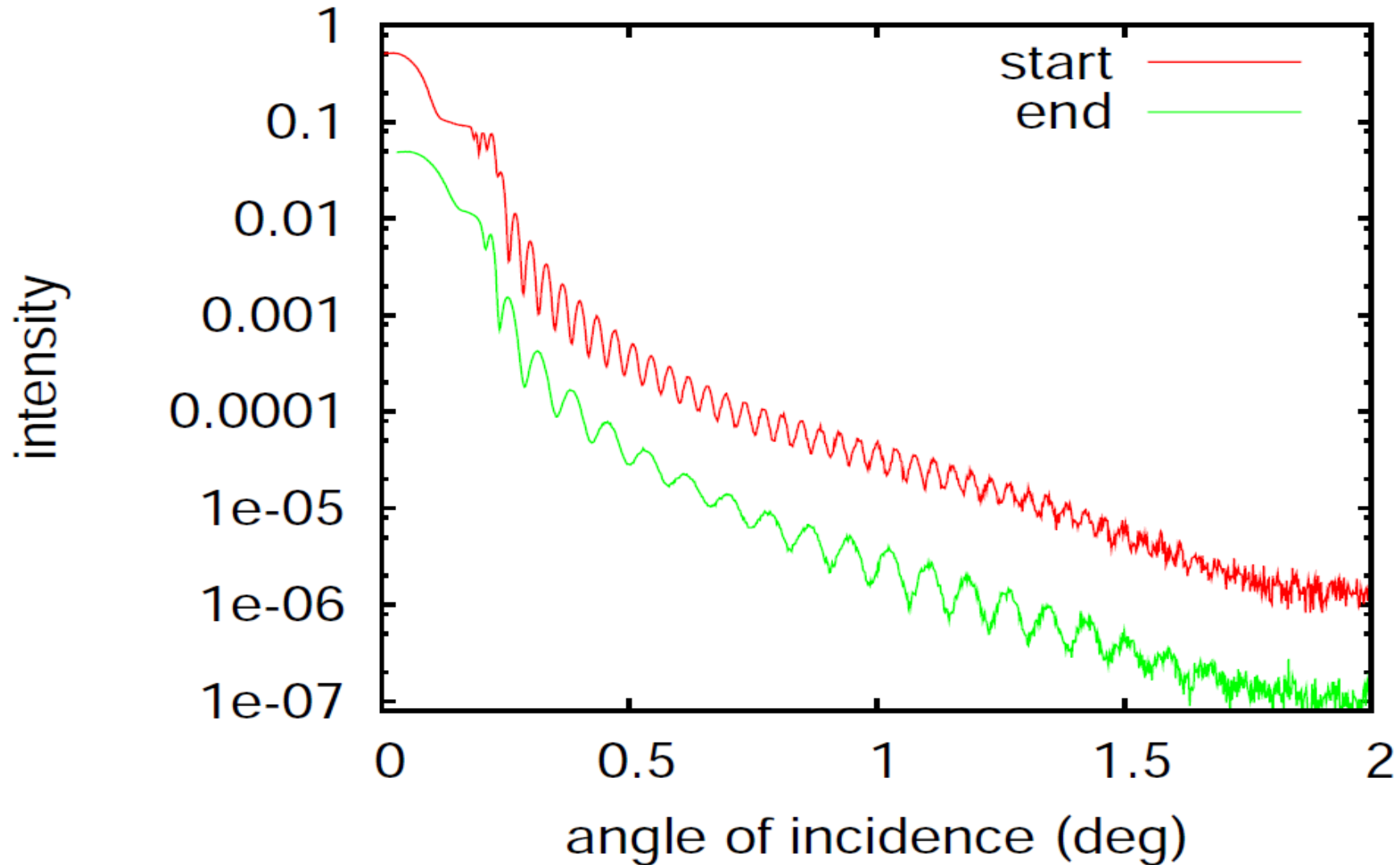
Dynamická teorie

Odraz na vrstvě v jednovlnné aproximaci



Dynamická teorie

Degradace polymerní vrstvy (tetravinylsilan/Si) vlivem UV

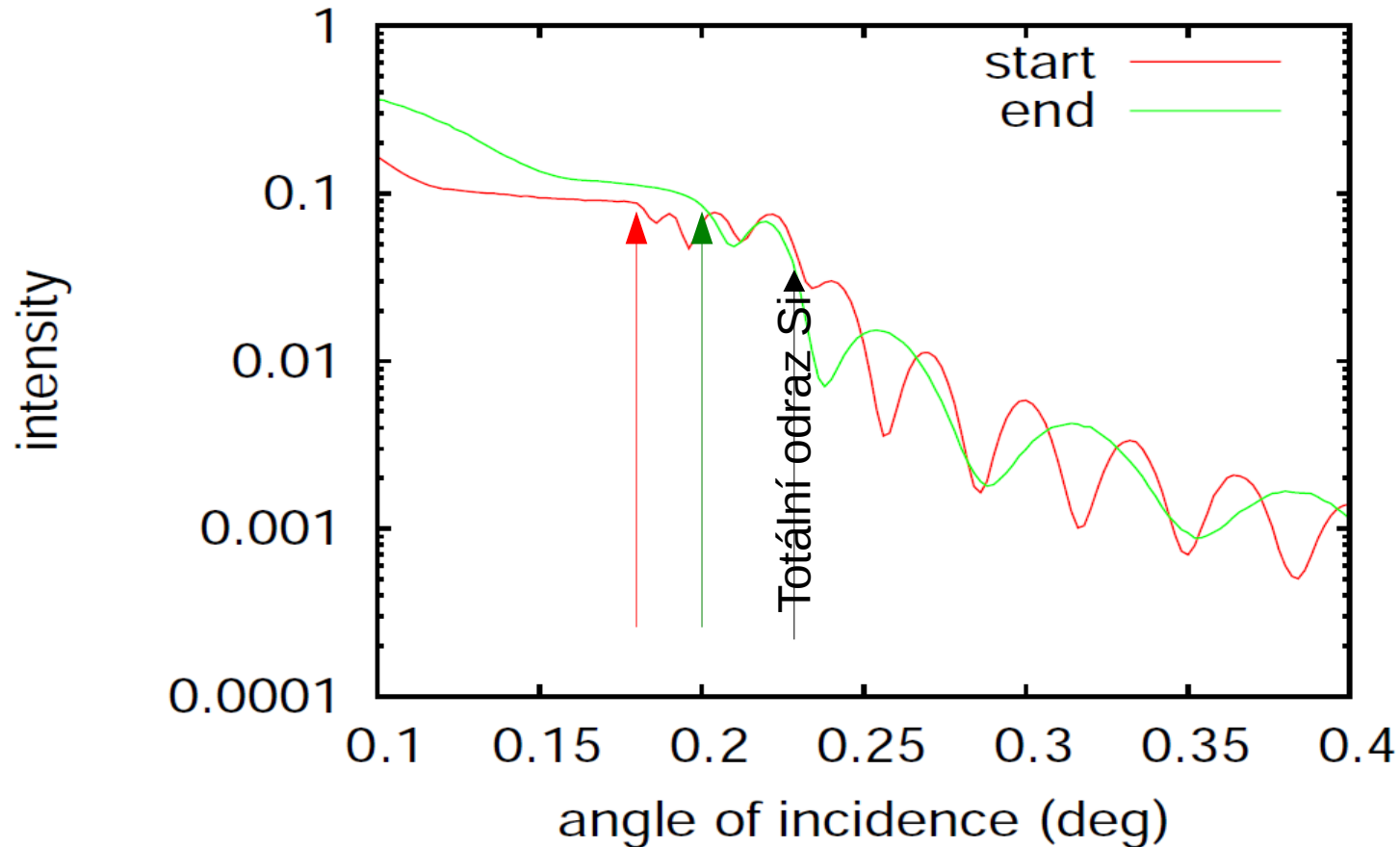


Tloušťka vlivem UV záření klesla ze 112 nm na 53 nm.

Dynamická teorie

Odraz na vrstvě v jednovlnné aproximaci

Degradace polymerní vrstvy (tetravinylsilan/Si) vlivem UV



Úhel totálního odrazu závisí na hodnotě indexu lomu.

Hustota stoupla ze 1.6 g/cm^3 na 2.0 g/cm^3 .

Rtg reflexe

Velikost vzorku

$$I_{\text{meas}} = I_{\text{calc}} A_{\text{sample}} / A_{\text{beam}}$$

$$A_{\text{sample}} < A_{\text{beam}}$$

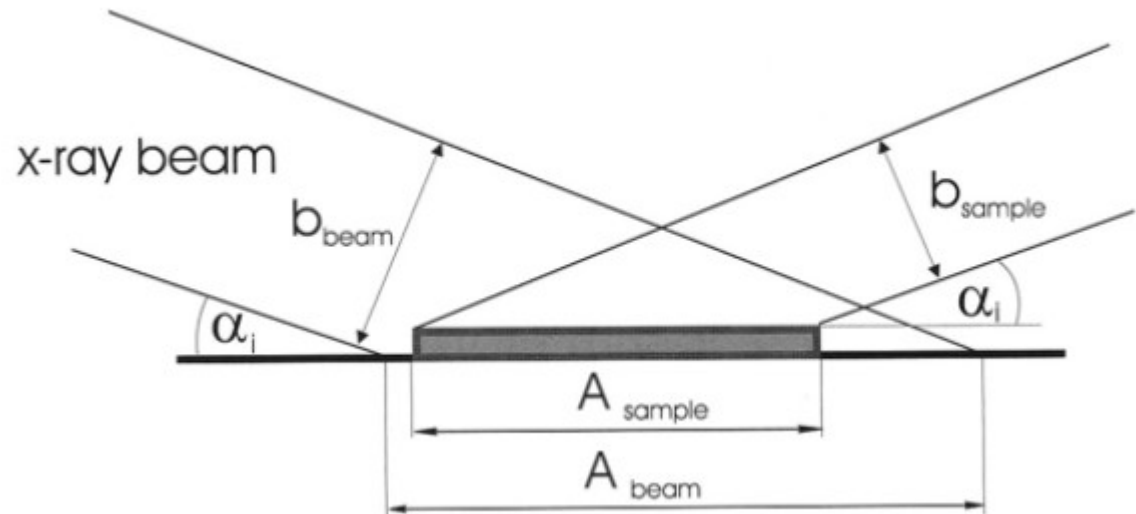
$$A_{\text{beam}} = b_{\text{beam}} \sin(\alpha_i)$$

$$I_{\text{meas}} = I_{\text{calc}} A_{\text{sample}} / b_{\text{beam}} \sin \alpha_i$$

$$I_{\text{meas}} = I_{\text{calc}}$$

$$A_{\text{sample}} < b_{\text{beam}} / \sin \alpha_i$$

$$A_{\text{sample}} > b_{\text{beam}} / \sin \alpha_i$$



Rtg reflexe

Multivrstvy formalismus přenosových matic

$$\mathbf{E}_j = \begin{pmatrix} E_T^{(j)} \\ E_R^{(j)} \end{pmatrix} \quad \begin{aligned} E_T^j e^{-ik_z^j d_j} + E_R^j e^{ik_z^j d_j} &= E_T^{j+1} + E_R^{j+1} \\ k_{zj} (E_T^j e^{-ik_z^j d_j} - E_R^j e^{ik_z^j d_j}) &= k_z^{j+1} (E_T^{j+1} - E_R^{j+1}) \end{aligned}$$

$$\hat{\mathbf{K}}_j \hat{\Phi}_j \vec{\mathbf{E}}_j = \hat{\mathbf{K}}_{j+1} \vec{\mathbf{E}}_{j+1} \quad \hat{\Phi}_j = \begin{pmatrix} e^{-ik_z^{(j)} T_j} & 0 \\ 0 & e^{ik_z^{(j)} T_j} \end{pmatrix}, \quad j = 0, \dots, N+1$$

$$\hat{\mathbf{K}}_j = \begin{pmatrix} 1 & 1 \\ k_z^j & -k_z^j \end{pmatrix}$$

$$\vec{\mathbf{E}}_0 = \hat{\mathbf{M}} \vec{\mathbf{E}}_{sub} = \hat{\Phi}_0^{-1} \hat{\mathbf{K}}_0^{-1} \hat{\mathbf{K}}_1 \hat{\Phi}_1^{-1} \hat{\mathbf{K}}_1^{-1} \hat{\mathbf{K}}_2 \hat{\Phi}_2^{-1} \dots \hat{\Phi}_N^{-1} \hat{\mathbf{K}}_N^{-1} \hat{\mathbf{K}}_{sub} \hat{\Phi}_{sub}^{-1} \vec{\mathbf{E}}_{sub}$$

$$\mathcal{R}_R = \left| \frac{M_{21}}{M_{11}} \right|^2$$

$$\hat{\mathbf{K}}_j^{-1} \hat{\mathbf{K}}_{j+1} = \frac{1}{t_{j,j+1}} \hat{\mathbf{R}}_j, \quad j = 0, 1, \dots, N \quad \hat{\mathbf{R}}_j = \begin{pmatrix} 1 & r_{j,j+1} \\ r_{j,j+1} & 1 \end{pmatrix}$$

$$r_{j,j+1} = \frac{k_z^j - k_z^{j+1}}{k_z^j + k_z^{j+1}}, \quad t_{j,j+1} = \frac{2k_z^j}{k_z^j + k_z^{j+1}}$$

Rtg reflexe

Rekurzivní formalismus

$$\mathfrak{R}_j = E_{\text{R}}^{(j)} / E_{\text{T}}^{(j)}$$

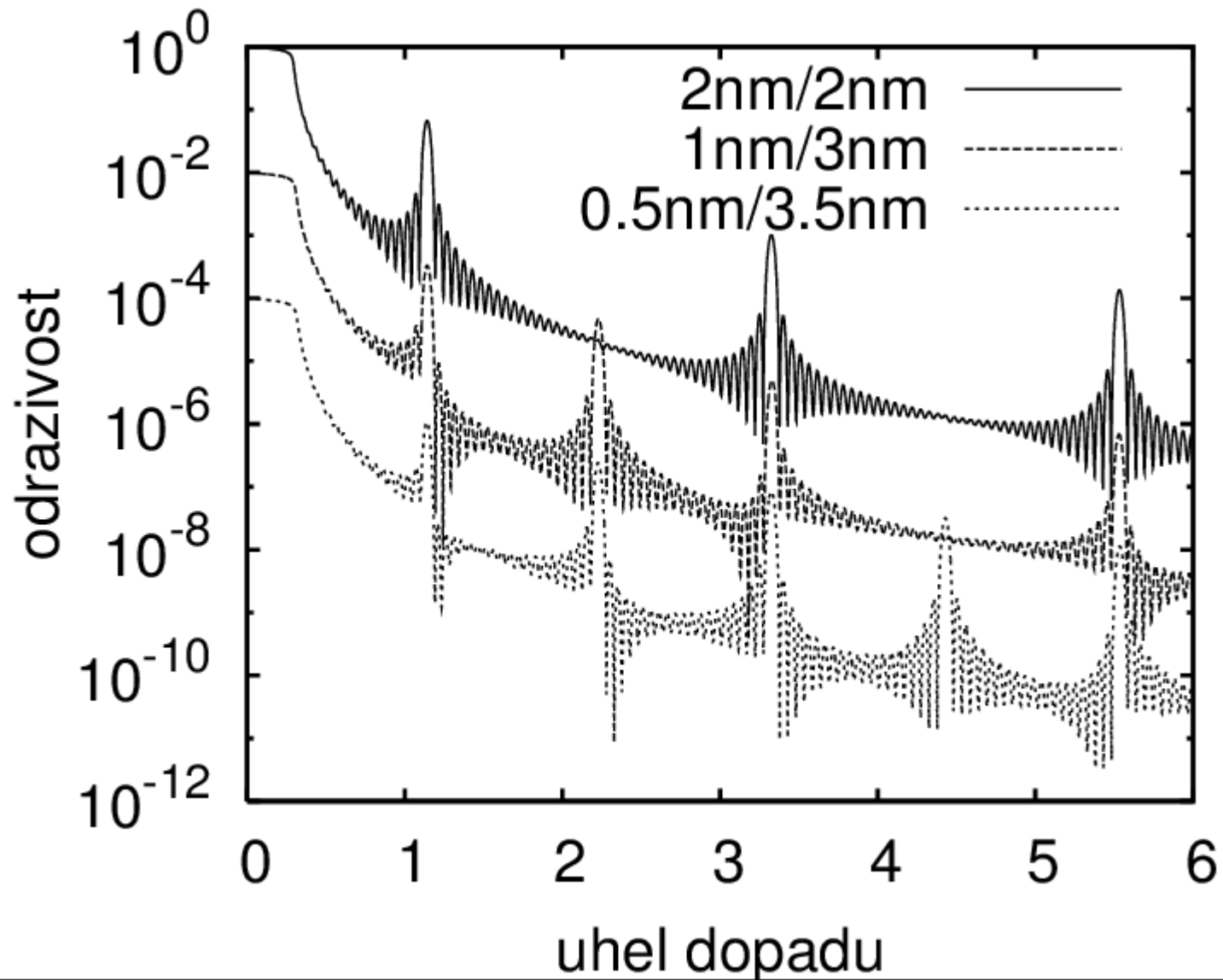
$$\mathfrak{R}_j = e^{-2ik_z^{(j)}T_j} \frac{r_{j+1} + \mathfrak{R}_{j+1}}{1 + r_{j+1}\mathfrak{R}_{j+1}}, j = 0, \dots, N$$

$$\mathcal{R}_{\text{sub}} \equiv \mathfrak{R}_{N+1} = 0$$

$$\mathcal{R}_{\text{R}} = |\mathfrak{R}_0|^2$$

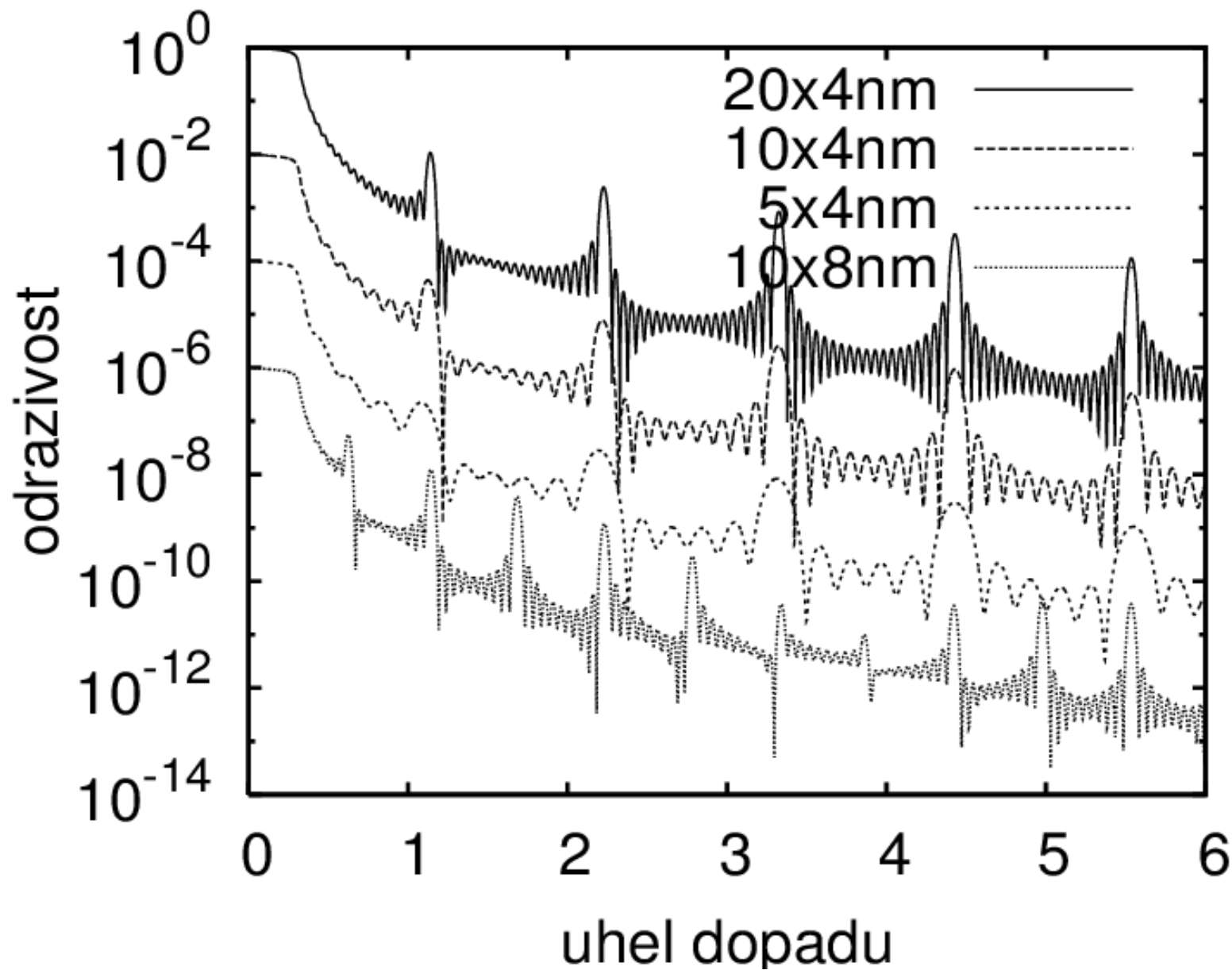
Dynamická teorie

Odraz na periodické multivrstvě v jednovlnné aproximaci



Dynamická teorie

Odraz na periodické multivrstvě v jednovlnné aproximaci

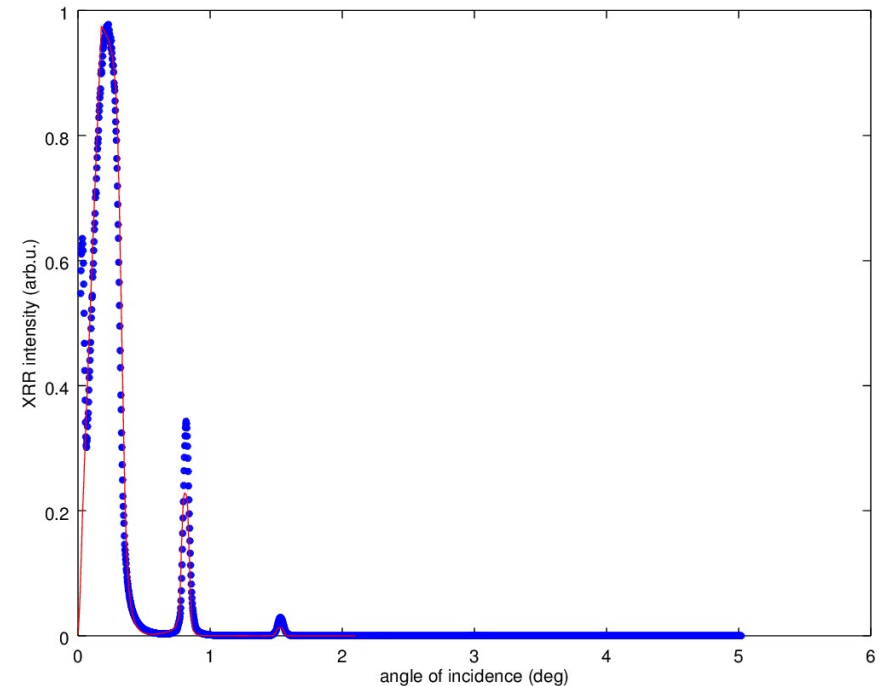
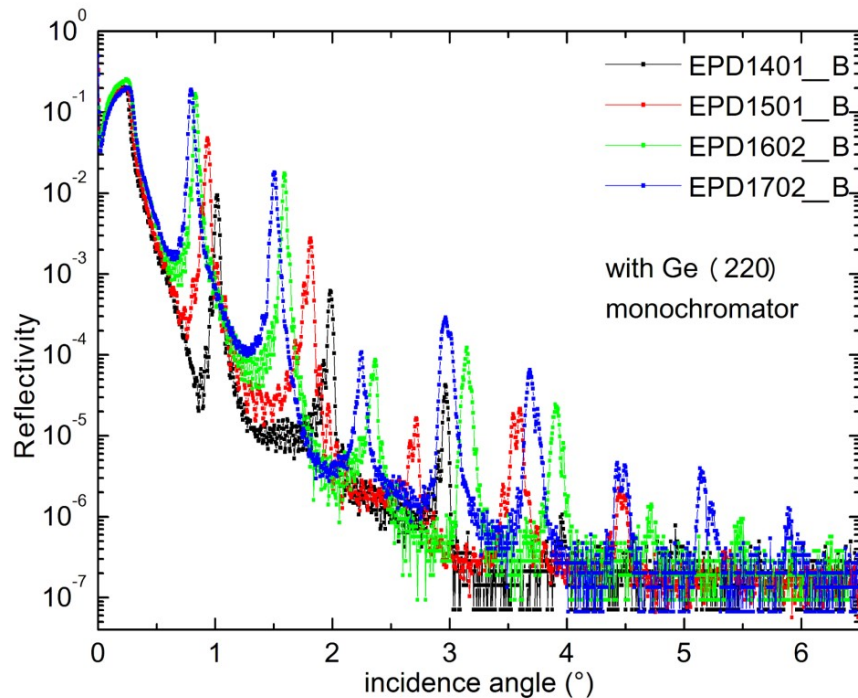


X-ray standing waves

Multilayers

X-ray reflectivity: superlattice peaks

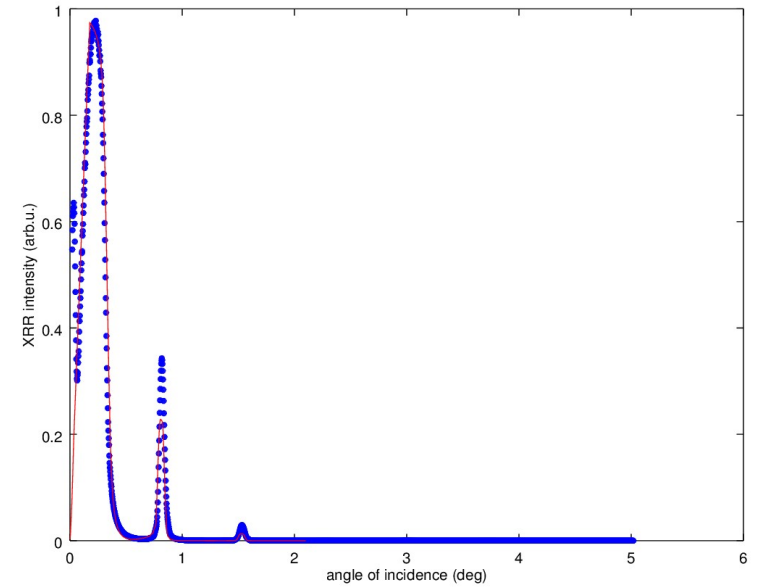
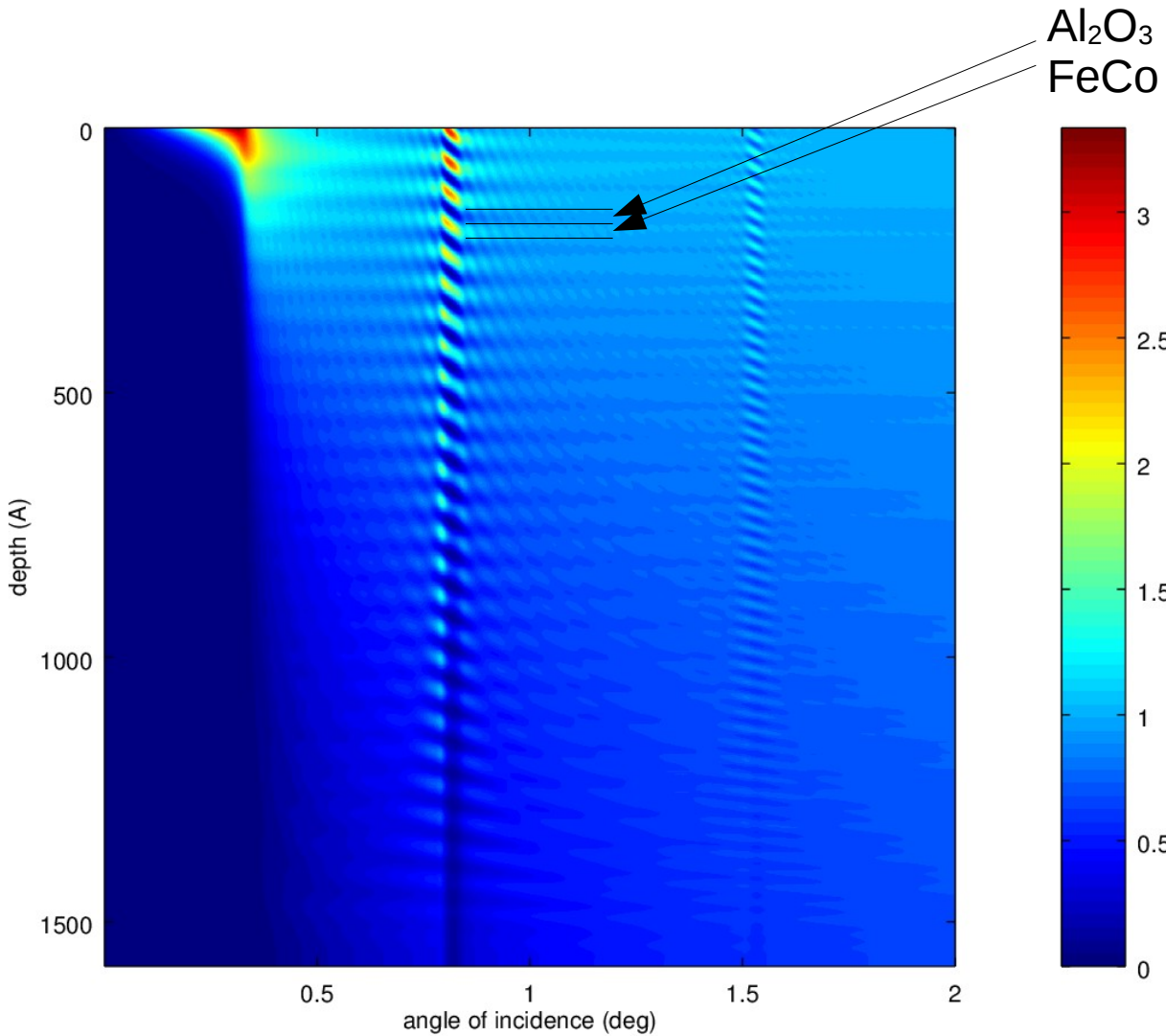
Bragg peaks corresponding superlattice period



X-ray standing waves

Multilayers

Depth Intensity distribution



X-ray standing waves

Samples: superparamagnetic multilayers

EPD layers in Load Lock Sputtering system

25x($\text{Fe}_{60}\text{Co}_{40}/\text{Al}_2\text{O}_3$) multilayers were prepared, with 3.5 nm nominal thick Al_2O_3 interlayers and the nominal layer thickness of the $\text{Fe}_{60}\text{Co}_{40}$ layers was changed from 0.5 nm to 2 nm + (5 nm Al_2O_3 buffer layer)

$\text{Fe}_{60}\text{Co}_{40}$ target -> DC sputtering (0.5 kW)

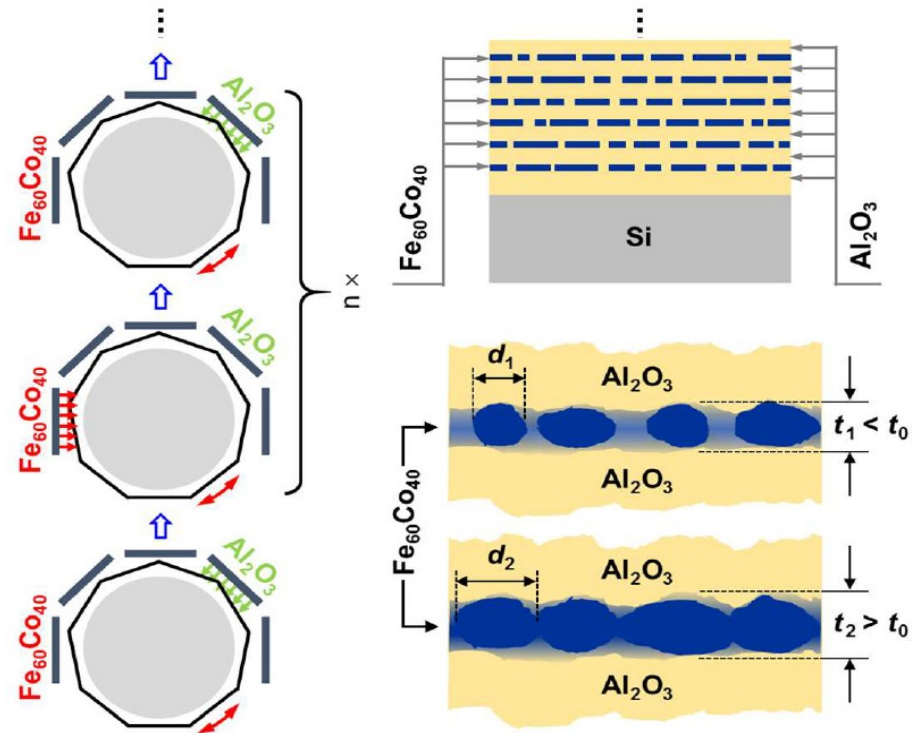
Al_2O_3 standard target -> RF sputtering (2.5 kW)

multilayers **EPD 14, 15, 16, 17**

$t_{\text{nominal}} = 0.5, 1.0, 1.5, 2.0$ nm

and another samples **r01-11** with

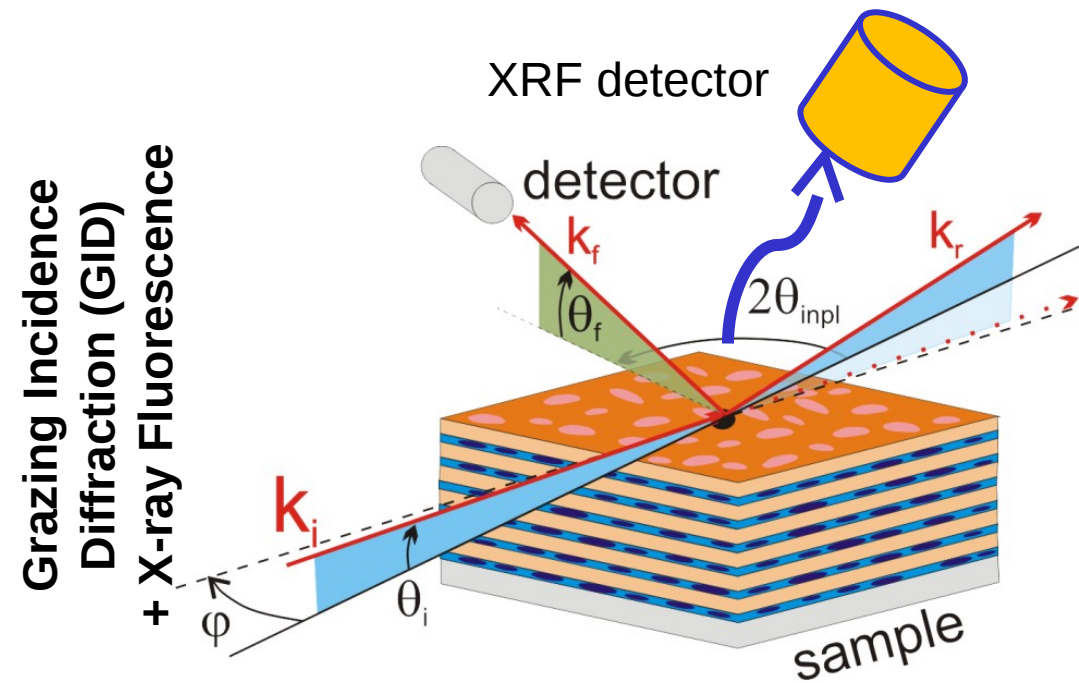
$t_{\text{nominal}} = 0.5 - 1.5$ nm (0.1 nm step)



X-ray standing waves

Experiment:

- scattering signal FeCo diffraction 002
 - Depth profile of crystalline phase
- Fluorescence signal
 - Chemical depth profile

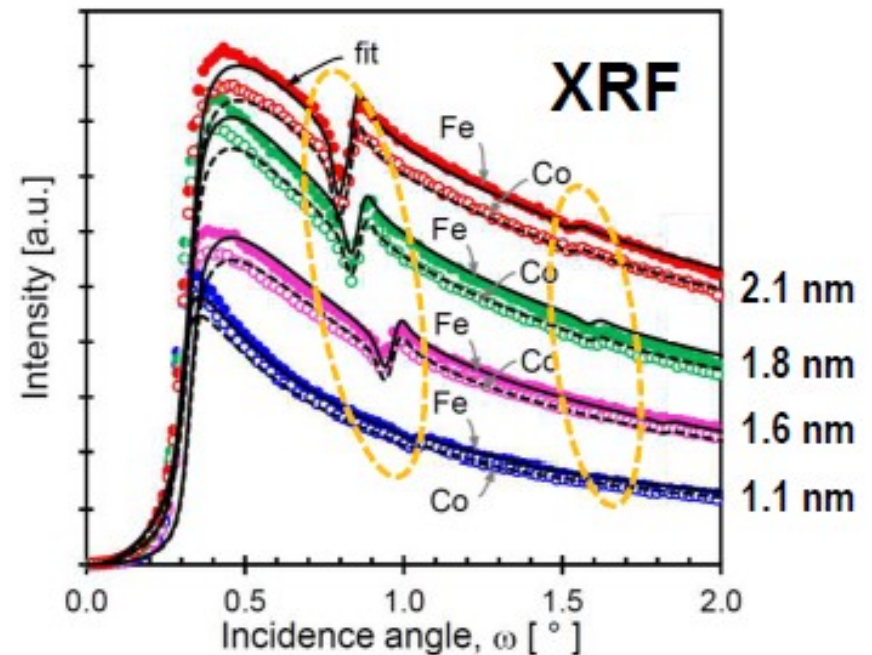
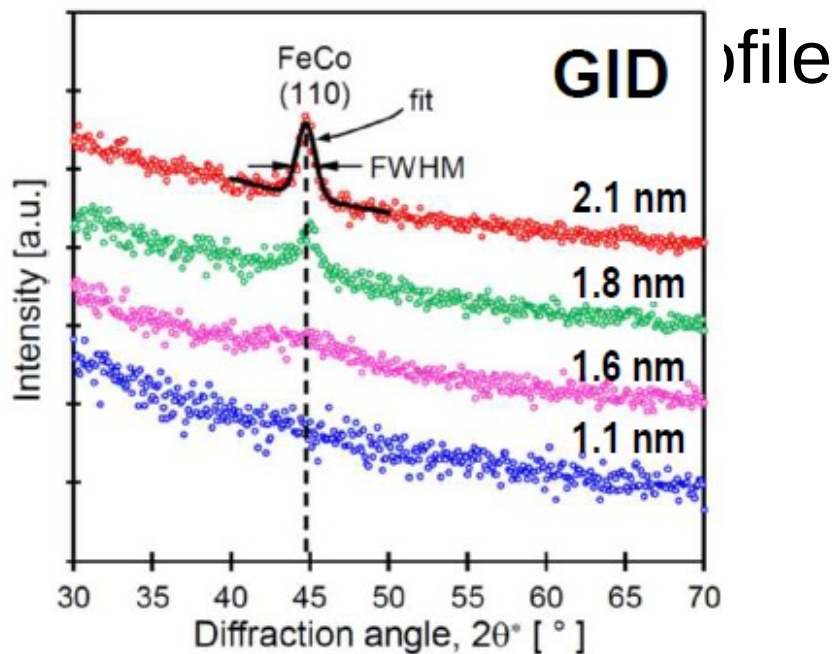
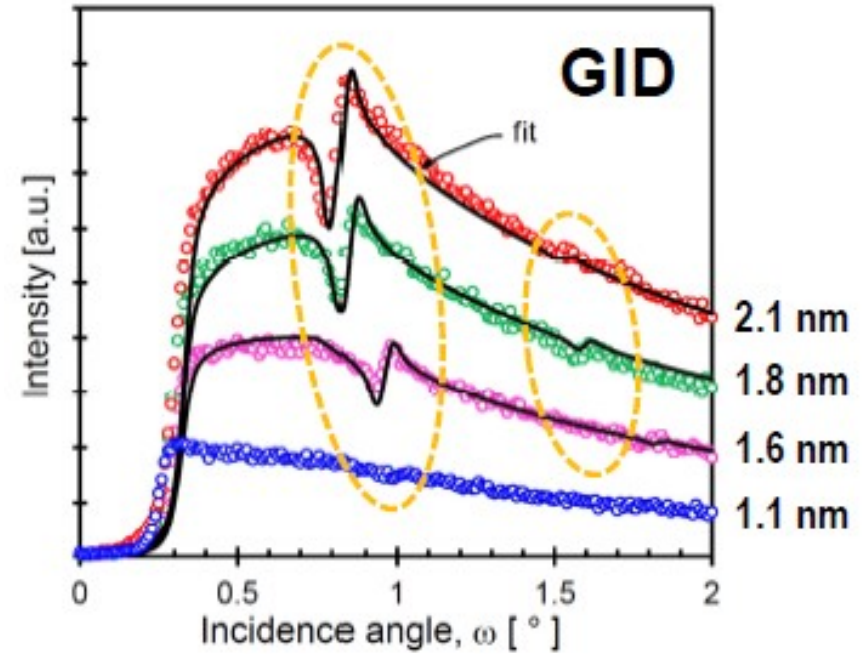


Standing wave x-ray diffraction was studied previously by J. Krčmář (PhD 2009)

X-ray standing waves

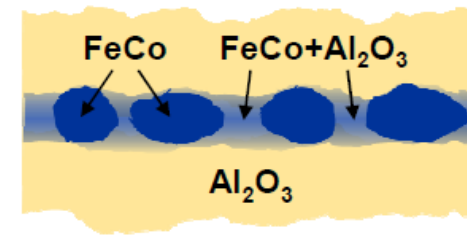
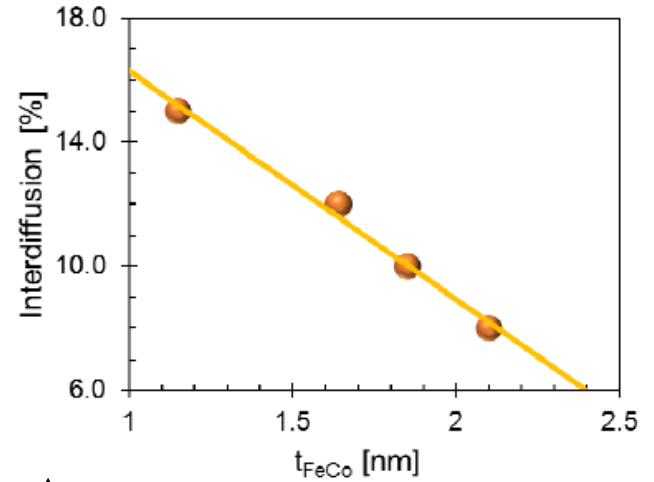
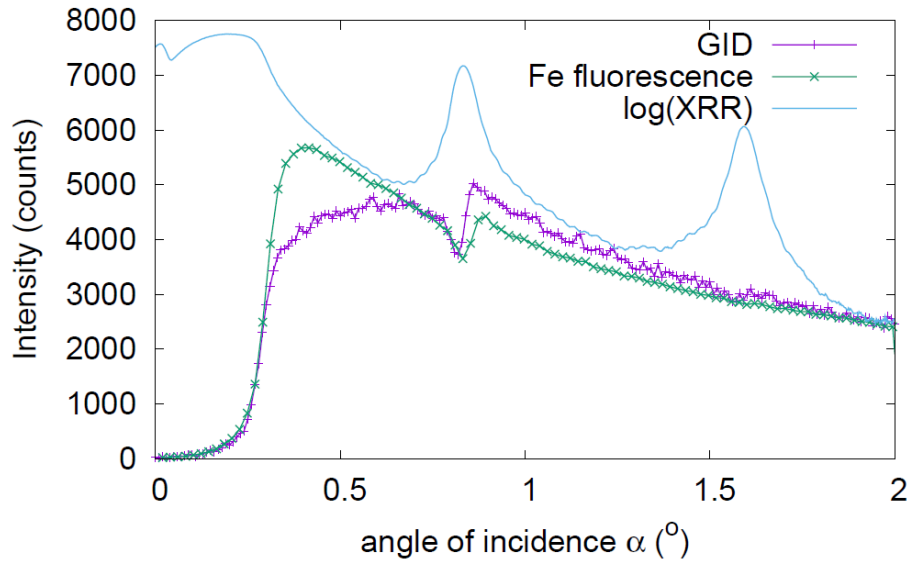
Experiment:

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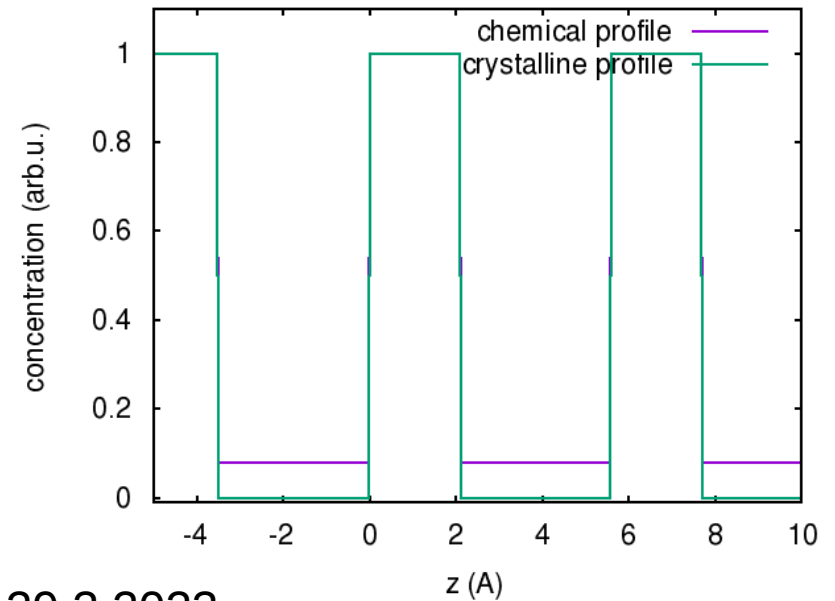
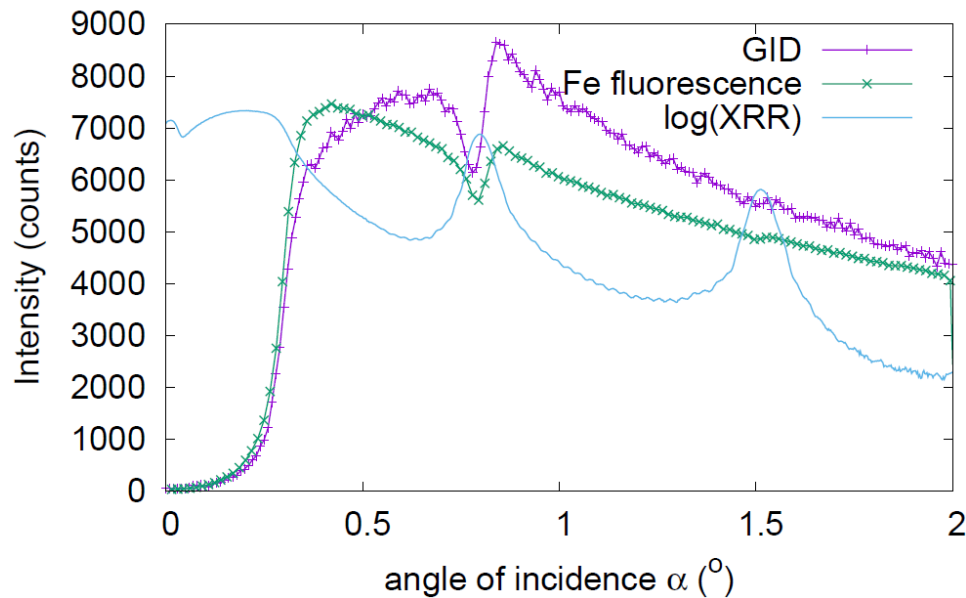


X-ray standing waves

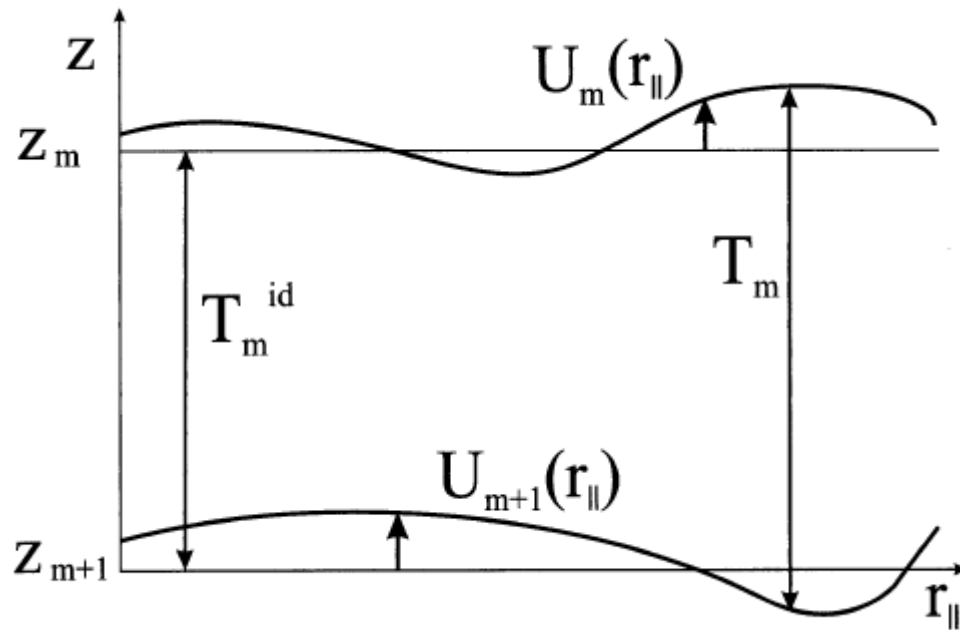
Comparison of XRR, GID, fluorescence, EPD 16



Comparison of XRR, GID, fluorescence, EPD 17



Rtg reflexe



$$w_m(U) = \frac{1}{S} \int_S d^2 \mathbf{r}_{\parallel} \delta(U_m(\mathbf{r}_{\parallel}) - U).$$

$$\chi_{U_m}(Q) = \langle e^{-iQU_m} \rangle = \int_{-\infty}^{\infty} dU w_m(U) e^{-iQU}$$

$$\langle U_m \rangle = 0.$$

$$\sigma_m^2 \equiv \langle U_m^2(\mathbf{r}_{\parallel}) \rangle = \int_{-\infty}^{\infty} dU w_m(U) U^2.$$

Rtg reflexe

$$\hat{U}_j = \begin{pmatrix} e^{ik_z^{(j)}U_j} & 0 \\ 0 & e^{-ik_z^{(j)}U_j} \end{pmatrix}, \quad \tilde{U}_{j+1} = \begin{pmatrix} e^{-ik_z^{(j)}U_{j+1}} & 0 \\ 0 & e^{ik_z^{(j)}U_{j+1}} \end{pmatrix}$$

$$\hat{\Phi}_j^{-1} = \hat{U}_j \hat{\Phi}_j^{\text{id}-1} \tilde{U}_{j+1}$$

$$\mathbf{E}_0 = \hat{\Phi}_0^{\text{id}-1} \tilde{U}_1 \hat{\mathbf{R}}_1 \hat{U}_1 \hat{\Phi}_1^{\text{id}-1} \tilde{U}_2 \hat{\mathbf{R}}_2 \hat{U}_2 \hat{\Phi}_2^{\text{id}-1} \dots \tilde{U}_{N+1} \hat{\mathbf{R}}_{N+1} \hat{U}_{N+1} \hat{\Phi}_{N+1}^{\text{id}-1} \mathbf{E}_{\text{sub}} \equiv \hat{M} \mathbf{E}_{\text{sub}},$$

$$\langle \mathbf{E}_0 \rangle \approx \langle \hat{M} \rangle \mathbf{E}_{\text{sub}}$$

$$\mathcal{R}_{\text{coh}} = \left| \frac{\langle M_{21} \rangle}{\langle M_{11} \rangle} \right|^2 \quad \left\langle \frac{M_{21}}{M_{11}} \right\rangle \approx \frac{\langle M_{12} \rangle}{\langle M_{11} \rangle}$$

$$\hat{\mathbf{R}}'_j = \langle \tilde{U}_j \hat{\mathbf{R}}_j \hat{U}_j \rangle = \frac{1}{t'_j} \begin{pmatrix} 1 & r'_j \\ r'_j & 1 \end{pmatrix}$$

$$t'_j = \frac{t_j}{\chi_{U_j}(k_z^{(j-1)} - k_z^{(j)})}, \quad r'_j = r_j \frac{\chi_{U_j}(k_z^{(j-1)} + k_z^{(j)})}{\chi_{U_j}(k_z^{(j-1)} - k_z^{(j)})}$$

Rtg reflexe

$$w(U) = \frac{1}{\sigma\sqrt{2\pi}} e^{-U^2/2\sigma^2}$$

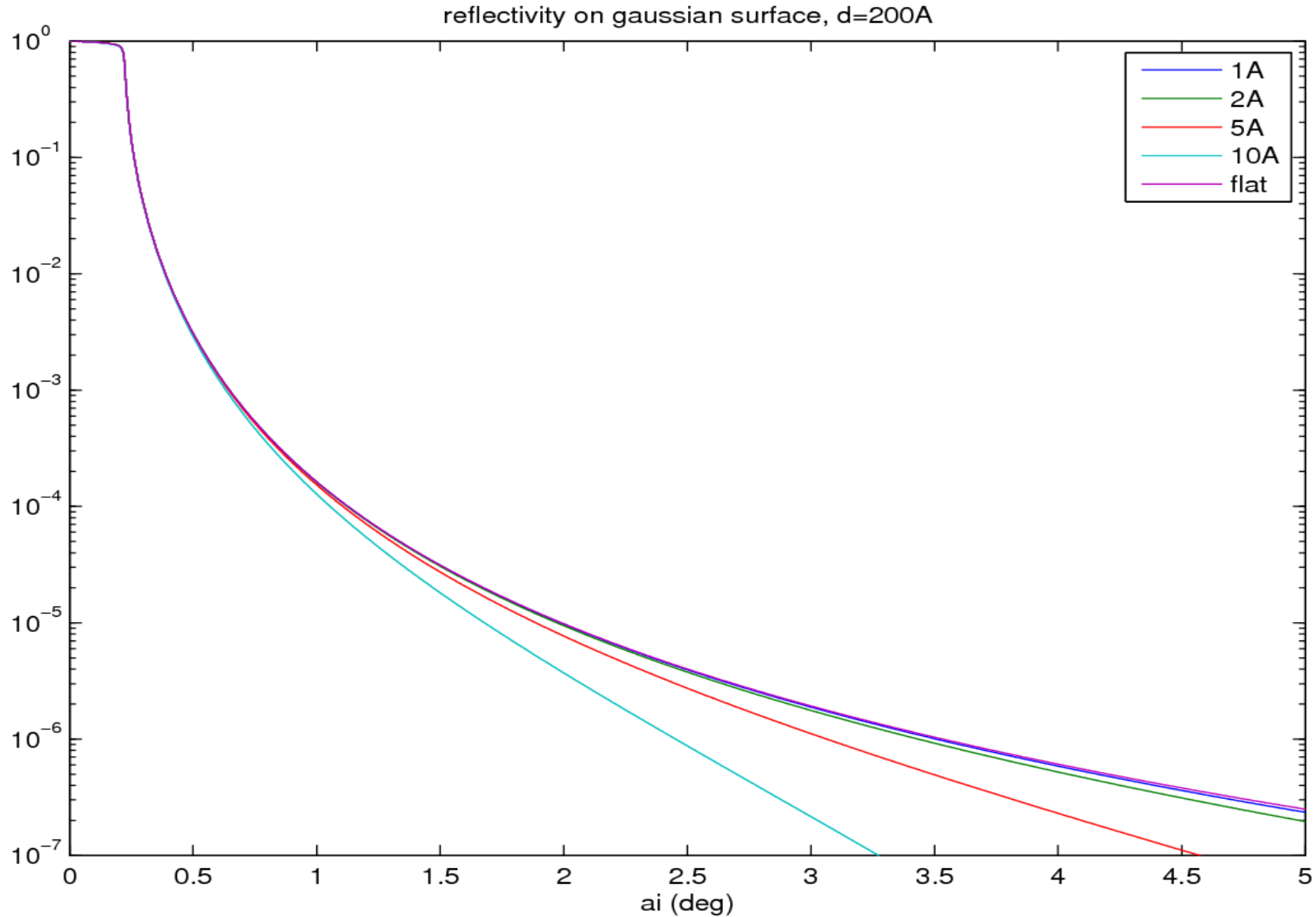
$$\chi_U(Q) = \int_{-\infty}^{\infty} dU w(U) e^{-iQU} = e^{-Q^2\sigma^2/2}$$

$$\mathcal{R}_{\text{coh}} = \mathcal{R}_{\text{flat}} \left| e^{-Q_z Q_z \tau \sigma^2 / 2} \right|^2 \quad \text{dynamical}$$

$$\mathcal{R}_{\text{coh}}^{\text{kin}} = \mathcal{R}_{\text{flat}}^{\text{kin}} e^{-Q_z^2 \sigma^2} \quad \text{kinematical}$$

Odraz na drsných rozhraních

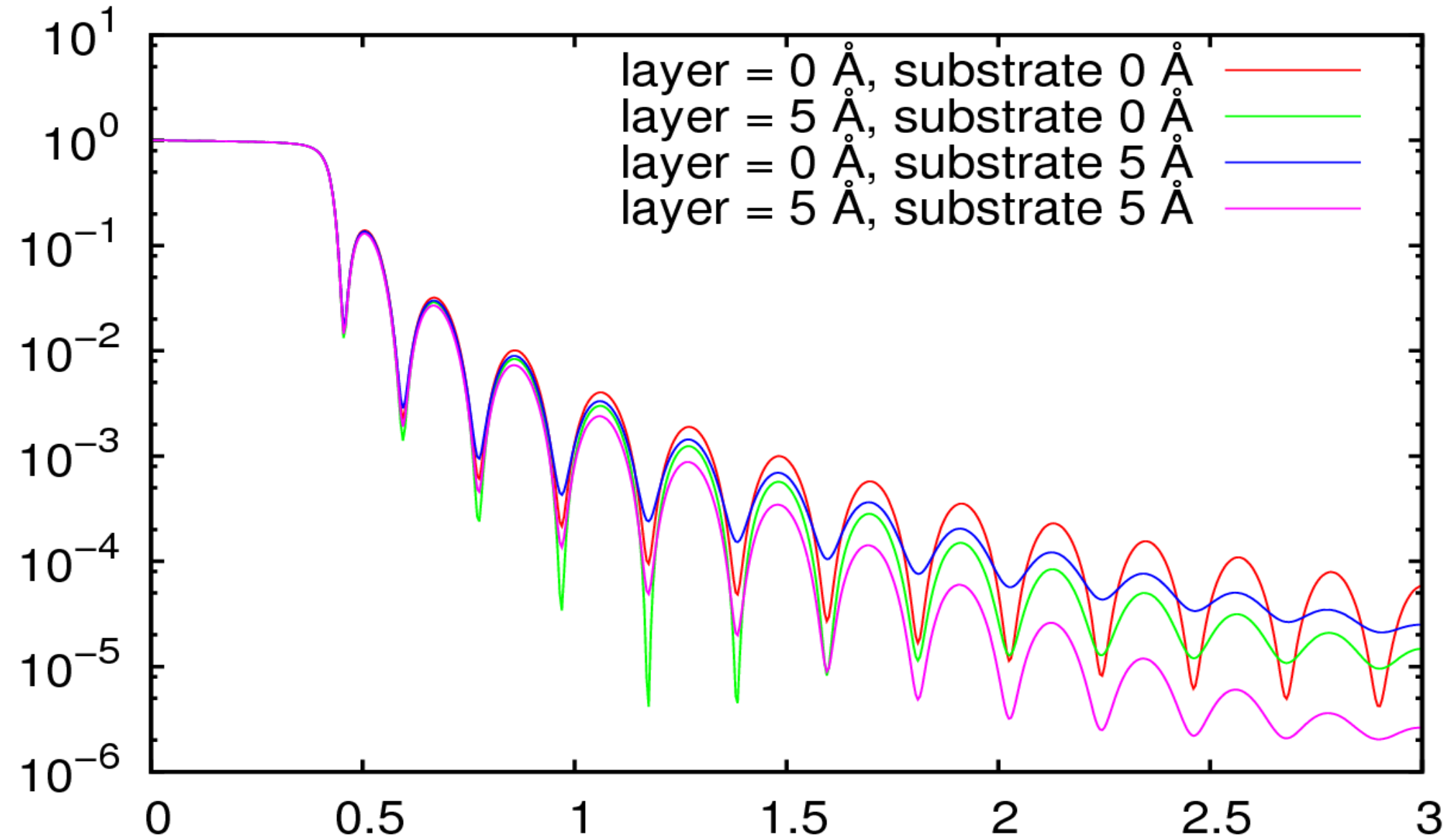
Jen substrát, různé drsnosti rozhraní, gaussovský model drsnosti, dvouúrovňové rozhraní



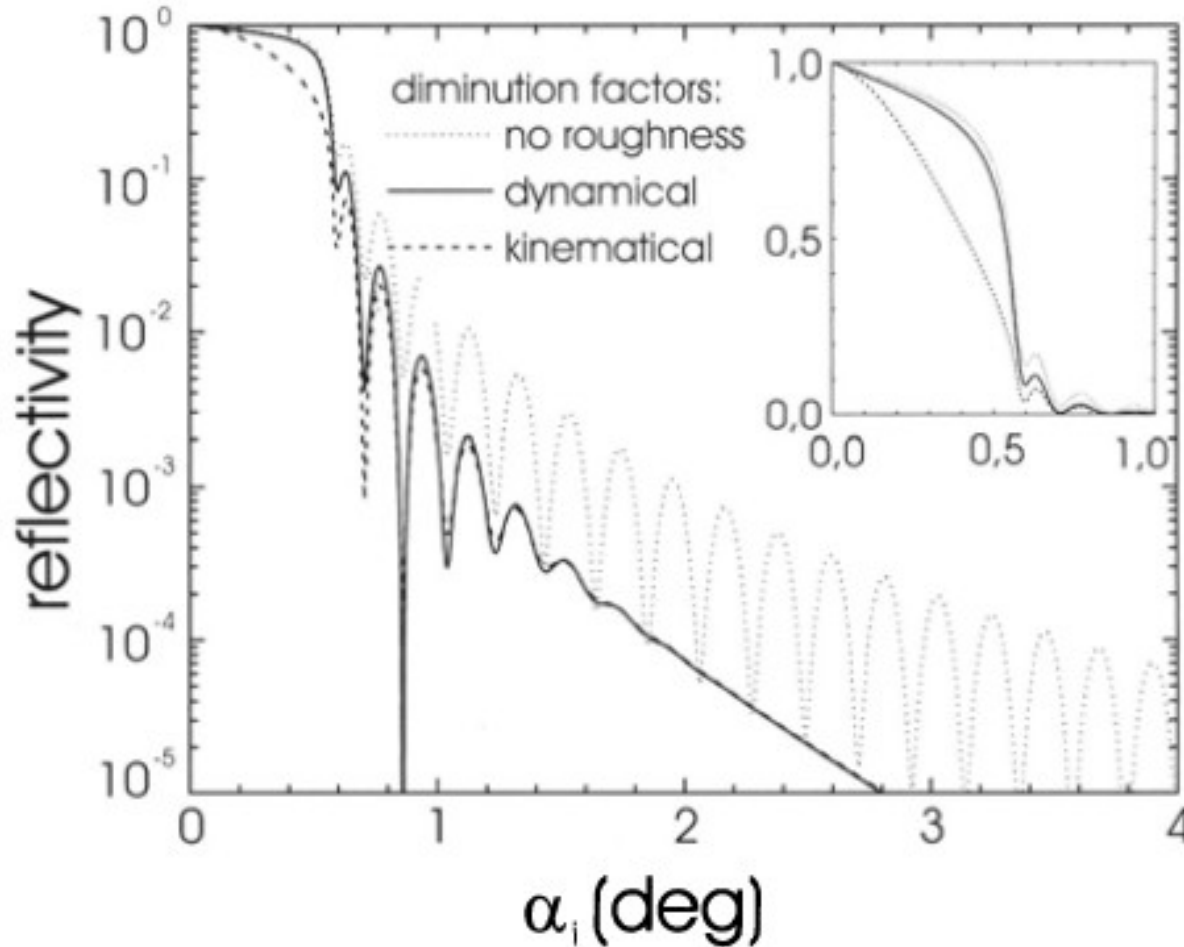
Odraz na drsných rozhraních

Vrstva na substrátu, různé drsnosti rozhraní, gaussovský model drsnosti

20 nm Ni/Si



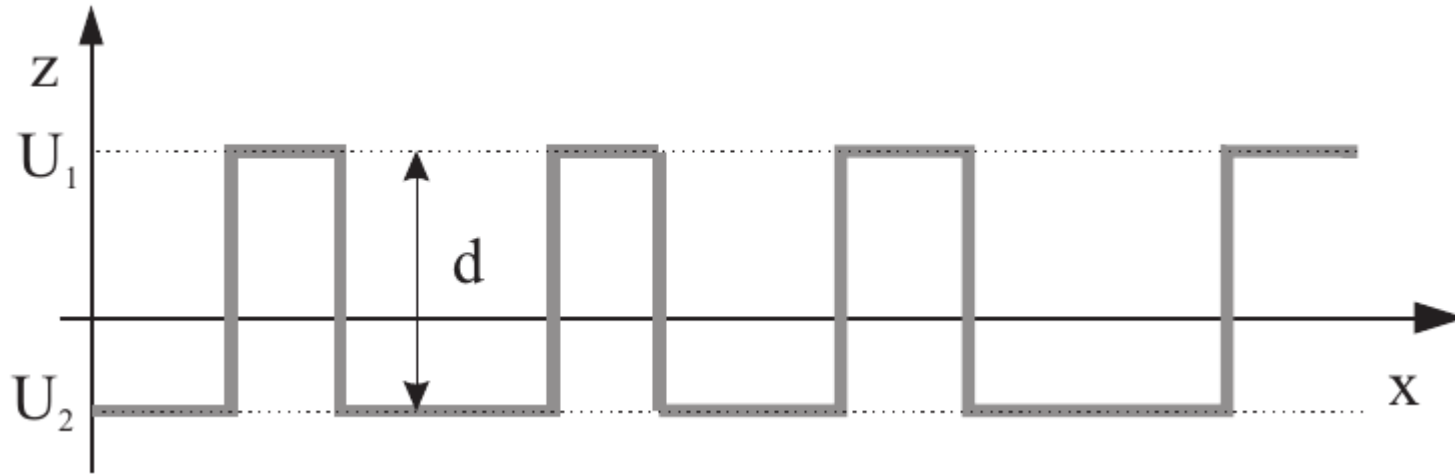
Rtg reflexe



Dynamical – fast roughness
Kinematical – slow roughness

D.K.G. deBoer: Phys. Rev. B **44**, 498 (1991)

Rtg reflexe



$$U_1 - U_2 = d.$$

$$p_2 = 1 - p_1$$

$$w(U) = p_1 \delta(U - U_1) + p_2 \delta(U - U_2)$$

$$\sigma^2 = U_1^2 p_1 + U_2^2 p_2 = p_1 p_2 d^2$$

$$\chi_U(Q) = e^{ip_2 Q d} (p_1 + p_2 e^{iQ d})$$

$$\mathcal{R}_{\text{coh}} = \left| e^{ip_2 Q d} (r^{(1)} + r^{(2)} e^{iQ d}) \right|^2, \quad r^{(i)} = r p_i$$

Odraz na drsných rozhraních

Jen substrát, různé drsnosti rozhraní, gaussovský model drsnosti, dvouúrovňové rozhraní

reflectivity on two step surface, $d=100\text{\AA}$

