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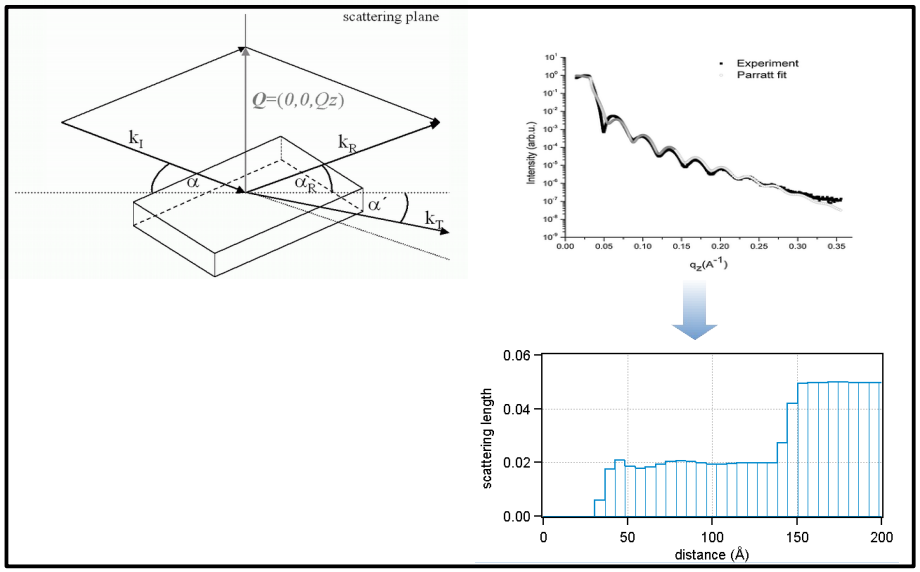
ep4

Resonance X-ray reflectivity — a tool to extract valence profiles and atomic scattering factors

Volodymyr Zabolotnyy

Dresden, December 2018

Experimental Setup



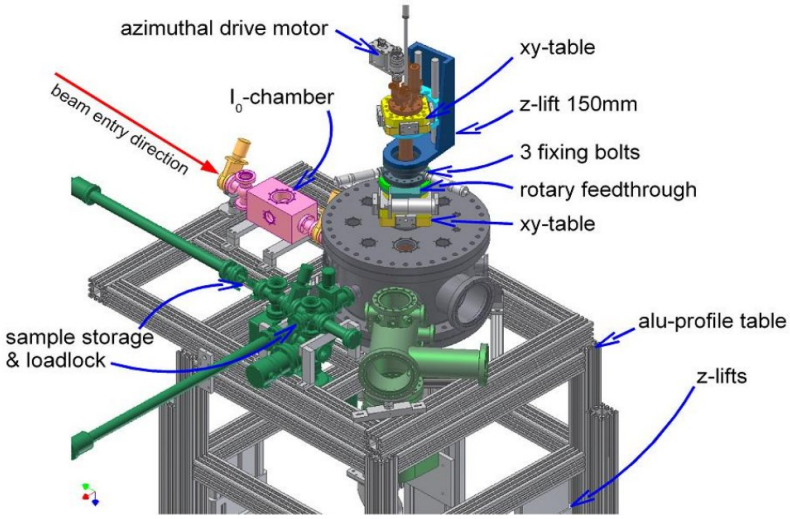


Image http://www.is.mpg.de

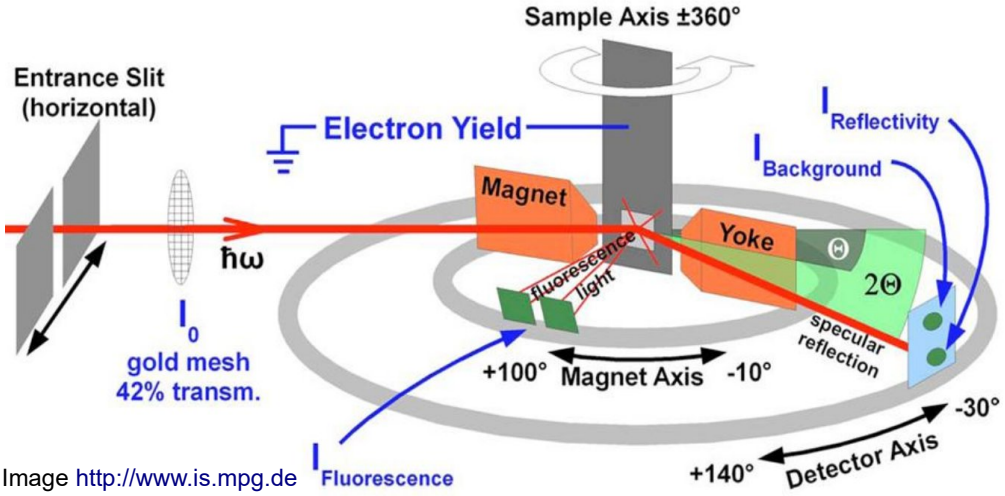


Image http://www.is.mpg.de

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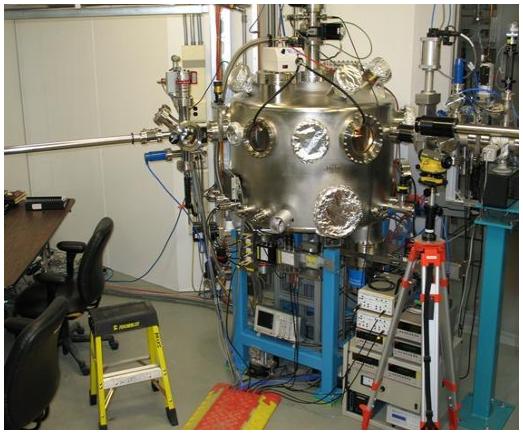
REIX beamline at CLS

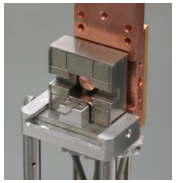
ep4



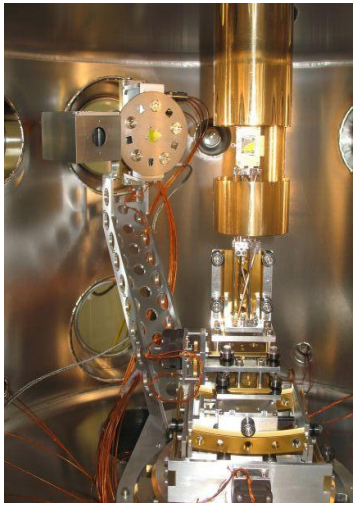
RSXS Scattering Chamber

RSXS Endstation

1. (Resonant) Soft X-ray Scattering (RSXS) / X-Ray reflectometry
   - 10-motion UHV diffractometer (4-circle diffractometer + x,y,z and detector motions)
   - Detectors: Channeltron, Photodiode, with multiple slits and filters, 2D Micro-Channelplate (MCP), Polarization Analyzer, Multi-channel Scaler, Electrometer.
2. X-ray Absorption Spectroscopy (XAS)
   - by total electron yield (TEY), total fluorescence yield (TFY)
3. Magnetic Circular Dichroism (MCD)
   - full polarization control of the incoming beam.



Permanent magnet

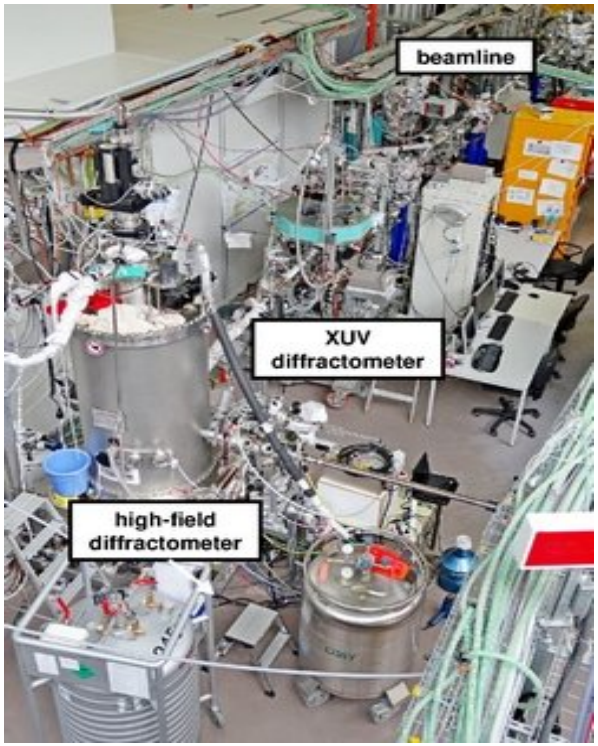


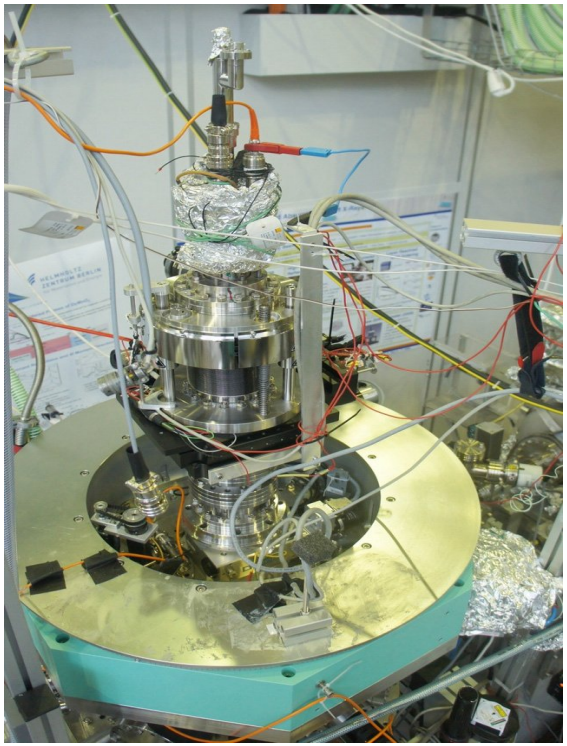
In-Vacuum Diffractometer

Status	**RSXS Endstation:** Operational, Accepting Proposals **XES Endstation:** Accepting Special Request
Source	Elliptically Polarizing Undulator (EPU)
Energy Range	80 – 2000 eV
Wavelength	155 – 6.2 Å
Resolution Δ E / E @ E	5×10^{-5} @100 eV 1.3×10^{-4} @ 1000 eV
Flux (y/s/0.1%BW) @ 100 mA	1×10^{12} @ 100 eV 5×10^{11}@ 1000 eV
Spot size (HxV)	RSXS: 250 x 150 μm XES: 60 x 10 μm

He4 closed cycle cryostat

UE46-PGM1 beamline at BESSY



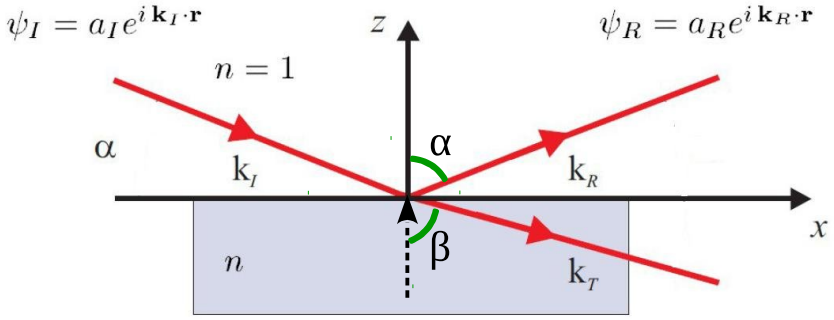


Selected Applications

- Resonant diffraction from magnetic, charge, and orbital order superstructures
- Spectroscopy of electronic ordering phenomena
- Magnetization states of single molecular magnets
- Element-specific magnetic hysteresis loops

Calculating reflectivity from a stack

A. Single interface ⇒ Fresnel equations



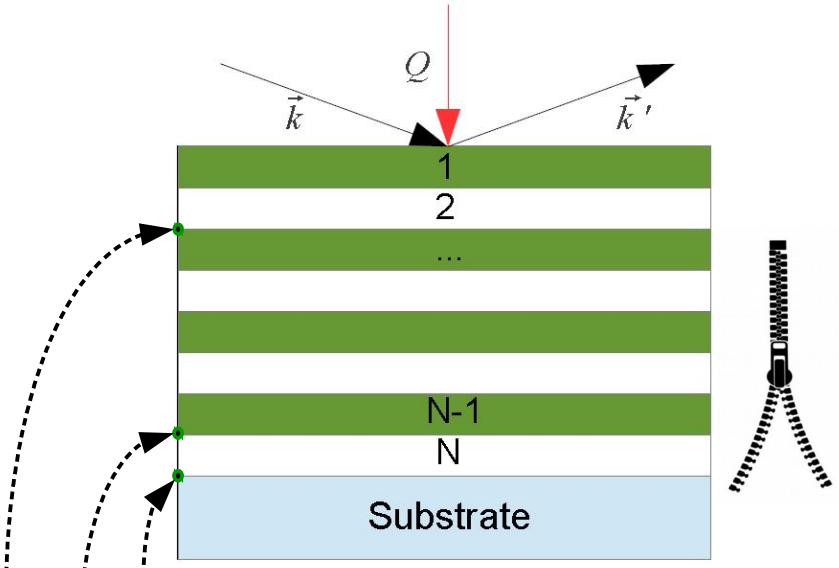
$$\left(\frac{E_{0t}}{E_{0e}}\right)_s = t_s = \frac{2n_1\cos\alpha}{n_1\cos\alpha + n_2\cos\beta}$$

$$\left(\frac{E_{0r}}{E_{0e}}\right)_s = r_s = \frac{n_1\cos\alpha - n_2\cos\beta}{n_1\cos\alpha + n_2\cos\beta}$$

$$\left(\frac{E_{0t}}{E_{0e}}\right)_p = t_p = \frac{2n_1\cos\alpha}{n_2\cos\alpha + n_1\cos\beta}$$

$$\left(\frac{E_{0r}}{E_{0e}}\right)_p = r_p = \frac{n_2\cos\alpha - n_1\cos\beta}{n_2\cos\alpha + n_1\cos\beta}$$

B. Thick sample ⇒ iterative Parrat approach



$$r'_{N,\infty} = \frac{Q_N - Q_\infty}{Q_N + Q_\infty}$$

$$r_{N-1,N} = \frac{r'_{N-1,N} + r'_{N,\infty}p_N^2}{1 + r'_{N-1,N}r'_{N,\infty}p_N^2}$$

$$r_{N-2,N-1} = \frac{r'_{N-2,N-1} + r_{N-1,N}p_{N-1}^2}{1 + r'_{N-2,N-1}r_{N-1,N}p_{N-1}^2}$$

Calculating reflectivity from a stack

C. Thick sample, arbitrary polarization+anisotropic ε*(z)* ⇒ matrix approach

Surface Science 96 (1980) 41–53
© North-Holland Publishing Company

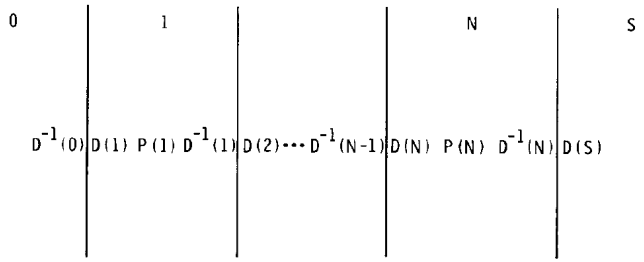
OPTICS OF ANISOTROPIC LAYERED MEDIA: A NEW 4 × 4 MATRIX ALGEBRA

Pochi YEH
Rockwell International Science Center, Thousand Oaks, California 91360, USA

Received 20 August 1979

Continuity condition:

$$\boldsymbol{D}(n-1)\begin{pmatrix}A_1(n-1)\\A_2(n-1)\\A_3(n-1)\\A_4(n-1)\end{pmatrix}=\boldsymbol{D}(n)\boldsymbol{P}(n)\begin{pmatrix}A_1(n)\\A_2(n)\\A_3(n)\\A_4(n)\end{pmatrix}$$



SURFACE

SUBSTRATE

$$\begin{pmatrix}A_1(n-1)\\A_2(n-1)\\A_3(n-1)\\A_4(n-1)\end{pmatrix}=\underbrace{\boldsymbol{T_{0,1}T_{1,2}T_{2,3}\ldots T_{N-1,N}T_{N,s}}}_{\boldsymbol{M}}\begin{pmatrix}A_1(s)\\A_2(s)\\A_3(s)\\A_4(s)\end{pmatrix}$$

$$\begin{matrix}\text{incident }\sigma\\\text{reflected }\sigma\\\text{incident }\pi\\\text{reflected }\pi\end{matrix}\begin{pmatrix}A_s\\B_s\\A_p\\B_p\end{pmatrix}=\begin{pmatrix}M_{11}&M_{12}&M_{13}&M_{14}\\M_{21}&M_{22}&M_{23}&M_{24}\\M_{31}&M_{32}&M_{33}&M_{34}\\M_{41}&M_{42}&M_{43}&M_{44}\end{pmatrix}\begin{pmatrix}C_s\\0\\C_p\\0\end{pmatrix}\begin{matrix}\rightarrow\text{ transmitted }\sigma\\\\\rightarrow\text{ transmitted }\pi\\\end{matrix}$$

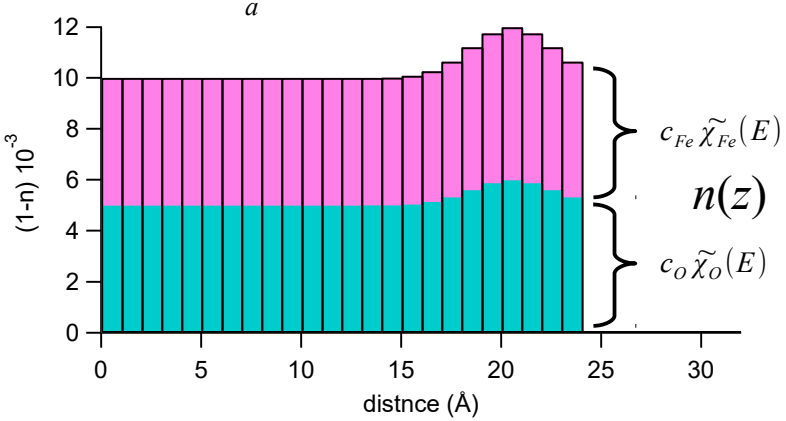
Where does element sensitivity comes from?

sum over chemical elements

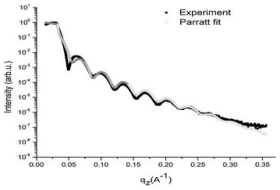
atomic concentration in mol/cm³

$$n-1=\sum_a \frac{2\pi c_a r_0}{k^2}\underbrace{\left(f'(E)+if''(E)\right)}_{\text{atomic scattering length}}$$

$$n-1=\sum_a c_a \tilde{\chi}_a(E)$$

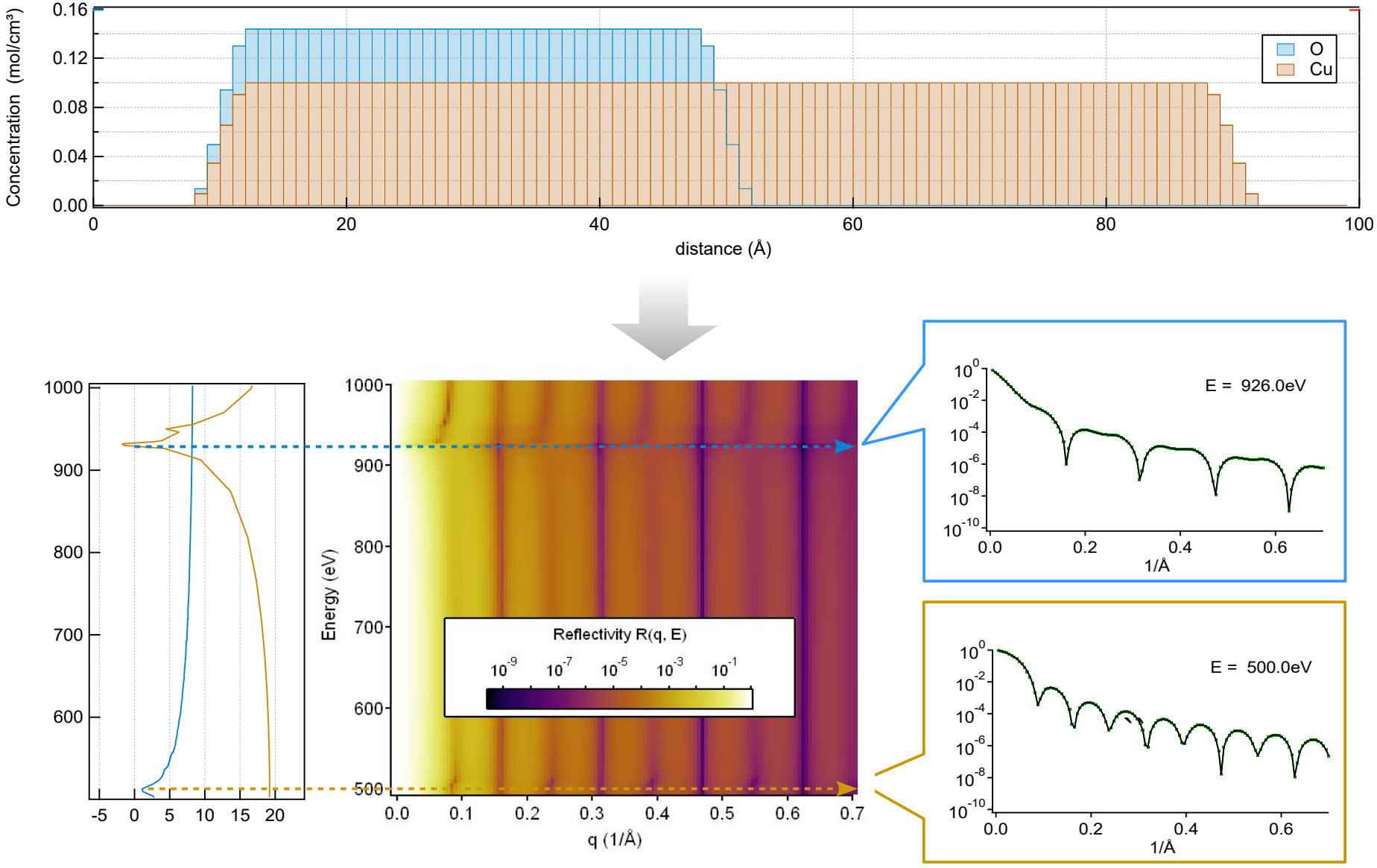


$R(q_z)$

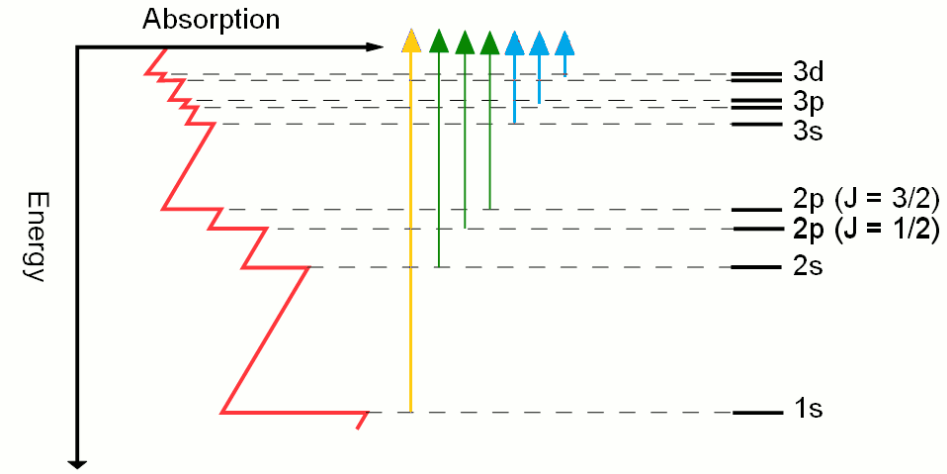


Lab-based of single-energy reflectivity measurements cannot be element sensitive.

Where does element sensitivity arise from?



“Habitable zone„ for soft X-ray reflectivity



K- edges
L-edges
M-edges

1 H																2 He
3 Li	4 Be										5 B	6 C	7 N	8 O	9 F	10 Ne
11 Na	12 Mg										13 Al	14 Si	15 P	16 S	17 Cl	18 Ar
19 K	20 Ca	21 Sc	22 Ti	23 V	24 Cr	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn	31 Ga	32 Ge	33 As	34 Se	35 Br	36 Kr
37 Rb	38 Sr	39 Y	40 Zr	41 Nb	42 Mo	43 Tc	44 Ru	45 Rh	46 Pd	47 Ag	48 Cd	49 In	50 Sn	51 Sb	52 Te	53 I	54 Xe
55 Cs	56 Ba		72 Hf	73 Ta	74 W	75 Re	76 Os	77 Ir	78 Pt	79 Au	80 Hg	81 Tl	82 Pb	83 Bi	84 Po	85 At	86 Rn
87 Fr	88 Ra		104 Rf	105 Db	106 Sg	107 Bh	108 Hs	109 Mt	110 Ds	111 Rg	112 Cn	113 Uut	114 Uuq	115 Uup	116 Uuh	117 Uus	118 Uuo
Lanthanides			57 La	58 Ce	59 Pr	60 Nd	61 Pm	62 Sm	63 Eu	64 Gd	65 Tb	66 Dy	67 Ho	68 Er	69 Tm	70 Yb	71 Lu
Actinides			89 Ac	90 Th	91 Pa	92 U	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No	103 Lr

Optical constants

1. Theoretical calculation away from resonances

- *Chantler et al. AIP **652**, 370 (2003)*
- *Chantler et al. J Phys Chem Ref Data **24**, 71 (1995)*
- *Chantler et al. J Phys Chem Ref Data **29**, 597 (2000)*

Theoretical Form Factor, Attenuation and Scattering Tabulation for $Z = 1–92$ from $E = 1–10$ eV to $E = 0.4–1.0$ MeV

C. T. Chantler
School of Physics, University of Melbourne, Parkville, Victoria 3052, Australia

Received September 27. 1994: revised manuscript received October 5. 1994

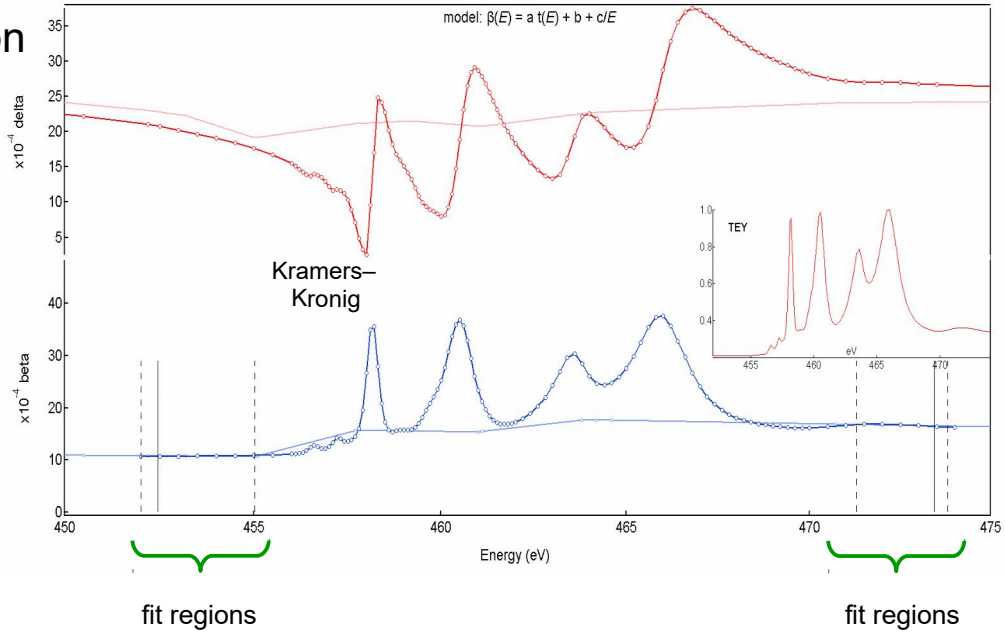
Tables for form factors and anomalous dispersion are widely used in the UV, x-ray and γ-ray communities, and have existed for a considerable period of time. Much of the recent

next most red 563 pages:

TABLE 1: Form factors, attenuation and scattering cross-sections, $Z = 1–92$, from $E = 0.001–0.010$ keV to $E = 400–1000$ keV — Continued.

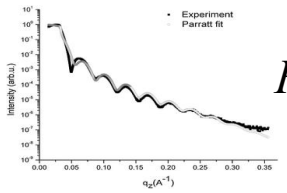
E keV	f_1 e/atom	f_2 e/atom	μ cm²/g	σ(coh+inc) cm²/g	μ_K cm²/g	λ nm
4.118862E+00	1.64400E+01	3.8997E+00	9.9405E+02	2.1733E+00	8.922E+02	3.010E−01
4.334821E+00	1.80390E+01	3.5640E+00	8.6321E+02	2.0998E+00	7.731E+02	2.860E−01
4.633924E+00	1.88984E+01	3.1991E+00	7.2483E+02	2.0037E+00	6.518E+02	2.676E−01
4.953664E+00	1.93754E+01	2.8820E+00	6.1083E+02	1.9080E+00	5.503E+02	2.503E−01
5.295467E+00	1.96982E+01	2.5972E+00	5.1493E+02	1.8133E+00	4.647E+02	2.341E−01

2. Total Electron Yield used as absorption



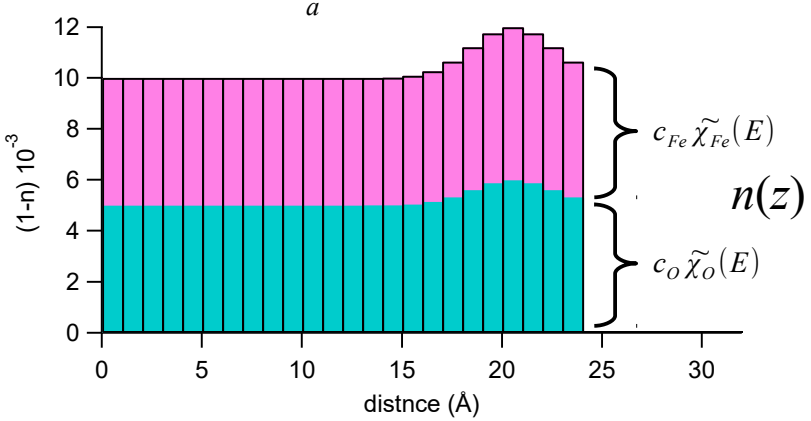
Where the vicious circle begin...

wanted, but close to impossible



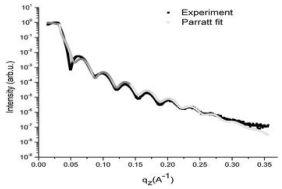
$R(q_z)$ $n(z)$

$$n-1=\sum_a c_a\,\tilde{\chi}_a(E)$$



trivial and rarely needed

$R(q_z)$ $n(z)$



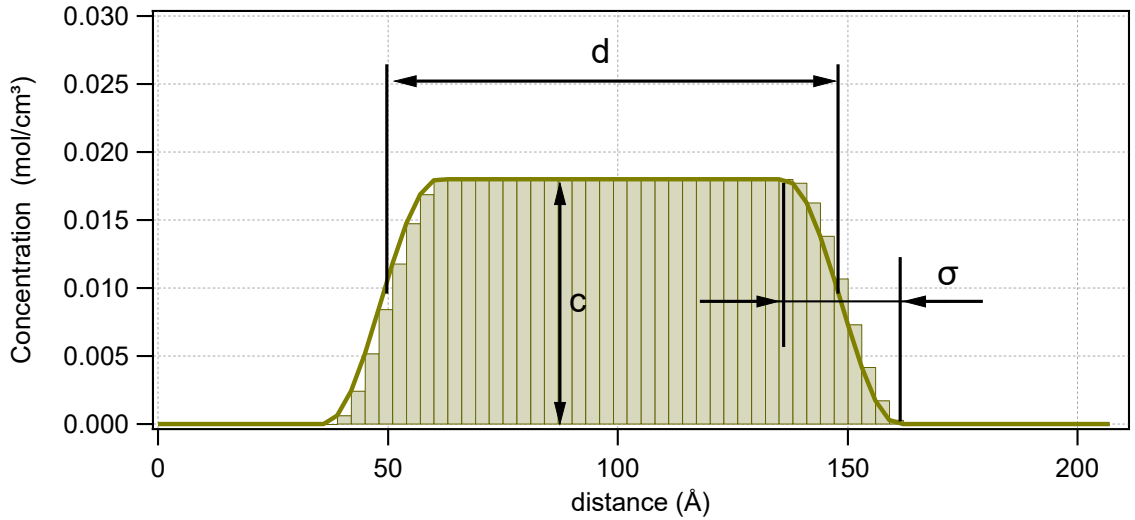
optimize & try again

Fitting R(q) to chemical profiles

Totally unrestricted fit:

1) 4 elements
2) 300Å thik sample (3Å x100 slices)
3) Elemental concentration in the range 0–0.100cm³ at Δc = 0.005cm³ resolution (5%)

Size of the "phase space" 400 fit parameters, each with 20 levels = $20^{400} \sim 2 \times 10^{520}$

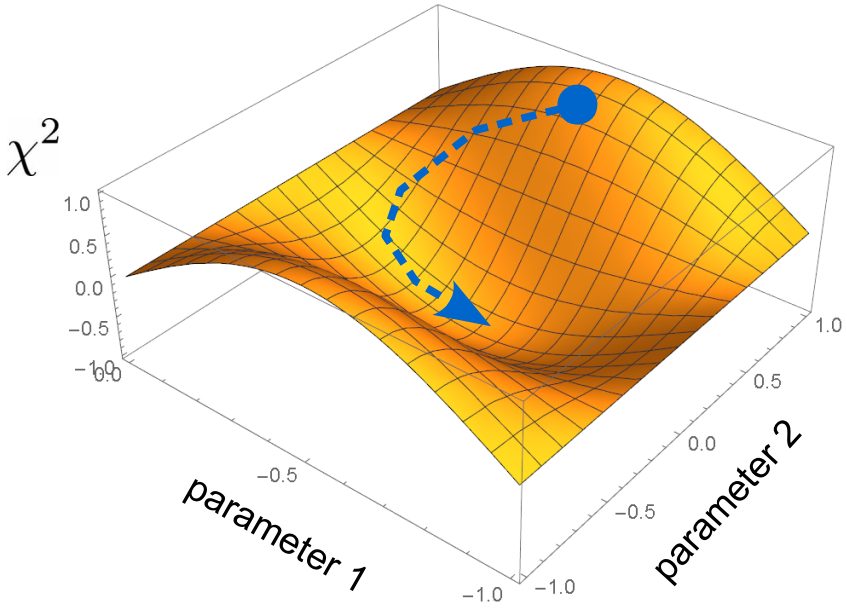


Parametrization in terms of Roughness σ, thickness d, concentration c, brings the number of "free" fit parameters typically below 3 x 3 layers x 4 elements = 36
⇒ $20^{36} \sim 7 \times 10^{46}$ distinct points in "phase space" with **plenty of local minima.**

⇒ **Necessity of sophisticated fit methods.**

Fitness profile

$$\chi^2(p_1, ..., p_N) = \sum_{\{q,\omega\}} [R_{\mathrm{exp}}(q,\omega) - R_{\mathrm{model}}(q,\omega,p_1,..,p_N)]^2 \rightarrow \min$$



Commonly used gradient descent methods are not suitable to fit reflectivity data, due to large number of local minima and strong non-linearity

Fitting approaches:

THE JOURNAL OF CHEMICAL PHYSICS **125**, 244702 (2006)

Fitting a free-form scattering length density profile to reflectivity data using temperature-proportional quenching

Charles F. Laub and Tonya L. Kuhl
Department of Chemical Engineering and Materials Science, University of California, Davis, California 95616

(Received 31 May 2006; accepted 7 November 2006; published online 27 December 2006)

IOP PUBLISHING JOURNAL OF PHYSICS D: APPLIED PHYSICS
J. Phys. D: Appl. Phys. **40** (2007) 6000–6004 doi:10.1088/0022-3727/40/19/033

Genetic algorithm using independent component analysis in x-ray reflectivity curve fitting of periodic layer structures

IOP PUBLISHING JOURNAL OF PHYSICS D: APPLIED PHYSICS
J. Phys. D: Appl. Phys. **40** (2007) 6000–6004 doi:10.1088/0022-3727/40/19/033

Genetic algorithm using independent component analysis in x-ray reflectivity curve fitting of periodic layer structures

IOP PUBLISHING JOURNAL OF PHYSICS D: APPLIED PHYSICS
J. Phys. D: Appl. Phys. **40** (2007) 4259–4263 doi:10.1088/0022-3727/40/14/023

Fitness function and nonunique solutions in x-ray reflectivity curve fitting: crosserror between surface roughness and mass density

J. Phys. D: Appl. Phys. **33** (2000) 1757–1763. Printed in the UK PII: S0022-3727(00)11144-1

Characterization of a layer stack by wavelet analysis on x-ray reflectivity data

E Smigiel and A Cornet

computer programs

Journal of Applied Crystallography
ISSN 0021-8898

Received 23 May 2007
Accepted 14 September 2007

***GenX*: an extensible X-ray reflectivity refinement program utilizing differential evolution**

Matts Björck*‡ and Gabriella Andersson

Department of Physics, Uppsala Universitet, Box 530, SE-751 21, Uppsala, Sweden. Correspondence e-mail: matts.bjorck@psi.ch

Some modification of an Evolution algorithms appear to be the most suited to fit reflectivity data.

Fitting R(q) to chemical profiles

RemagX (S. Macke)

Simulation:
- Parrat formalism
- Matrix formalism

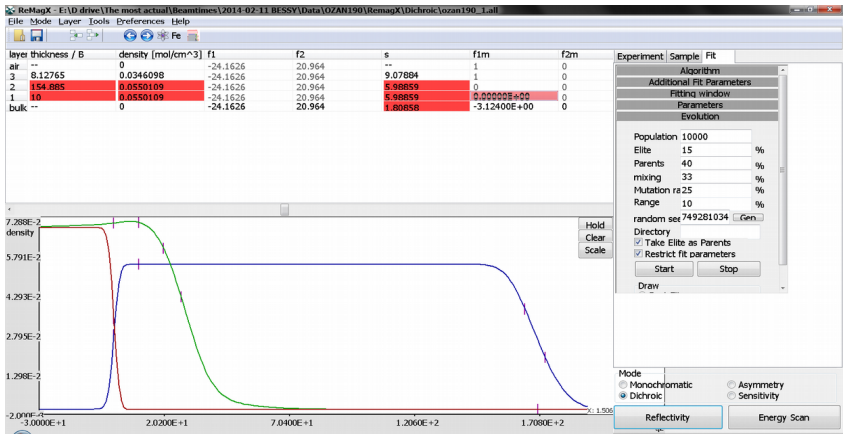
Fit:
- Evolution
- Simplex
- Levenberg&Marquardt

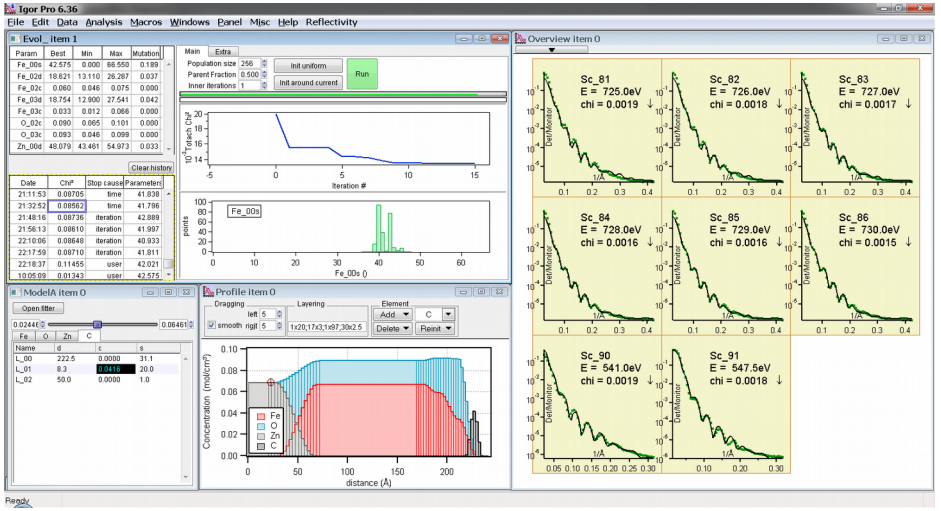
Own code under IgorPro

Simulation:
- Parrat formalism
- Coupling with theory (Quanty)

Fit:
- Evolution+dynamic mutation

- direct import of SPECS data
- preprocessing (cutting, resampling, backround)

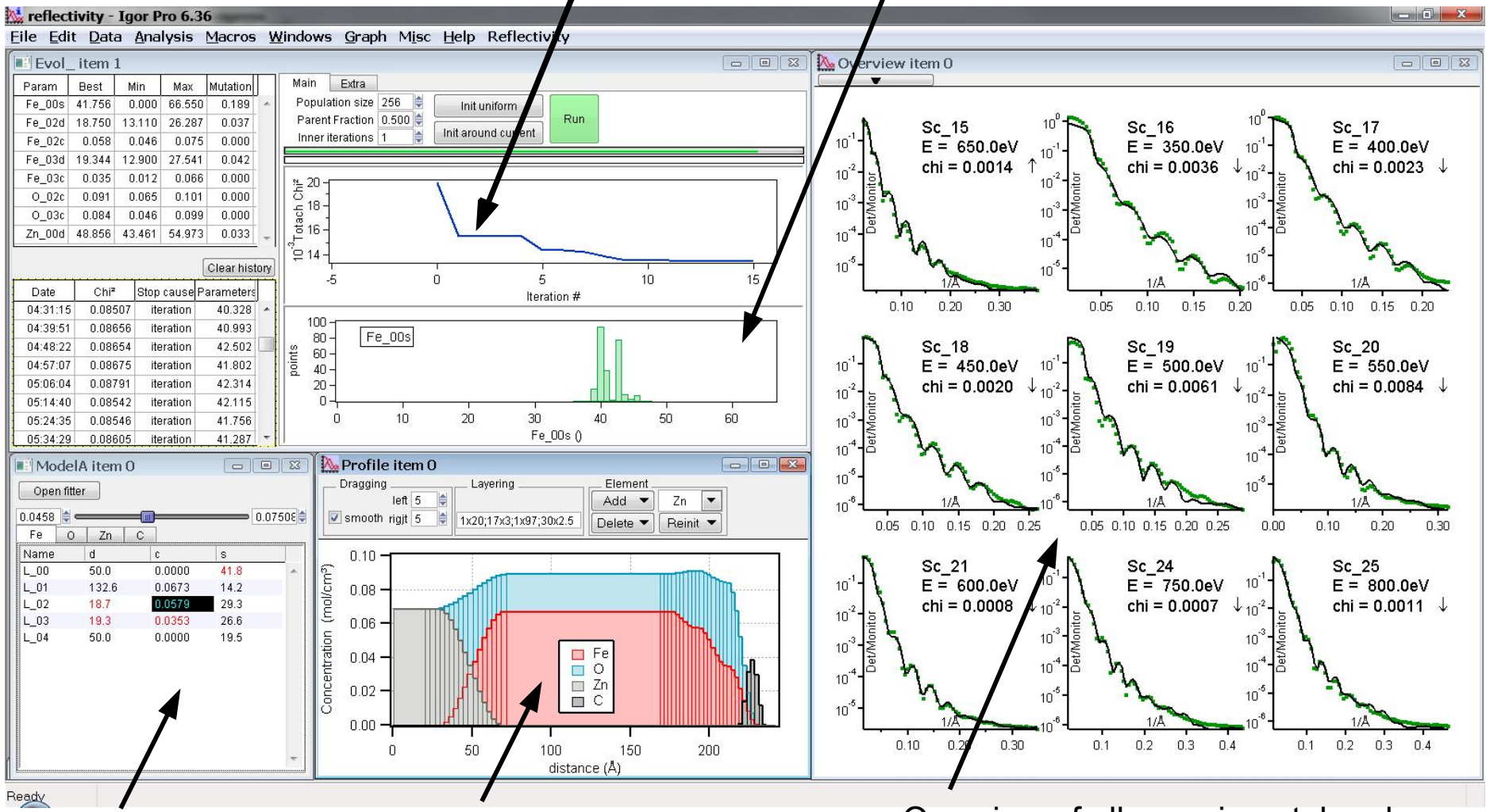




Fitting R(q) to chemical profiles

Time convergence of χ^2(p1, ...pN)

Param. distribution in the population



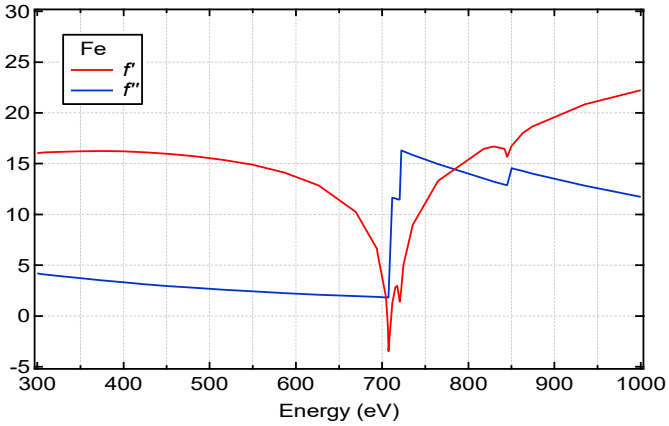
dcσ-model

Atomic conc. profiles

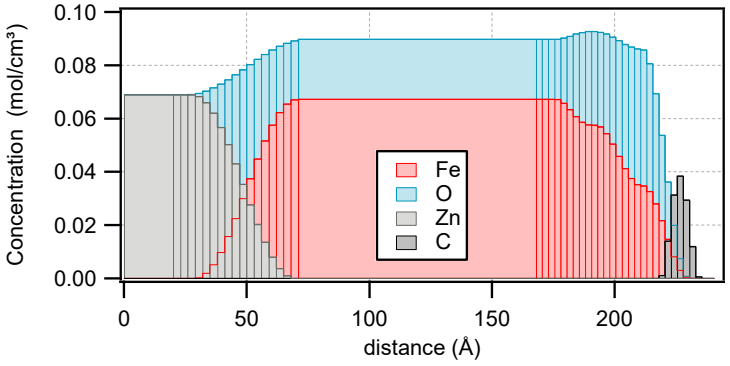
Overview of all experimental and simulated spectra

"Real life" example: ZnO/Fe_3O_4

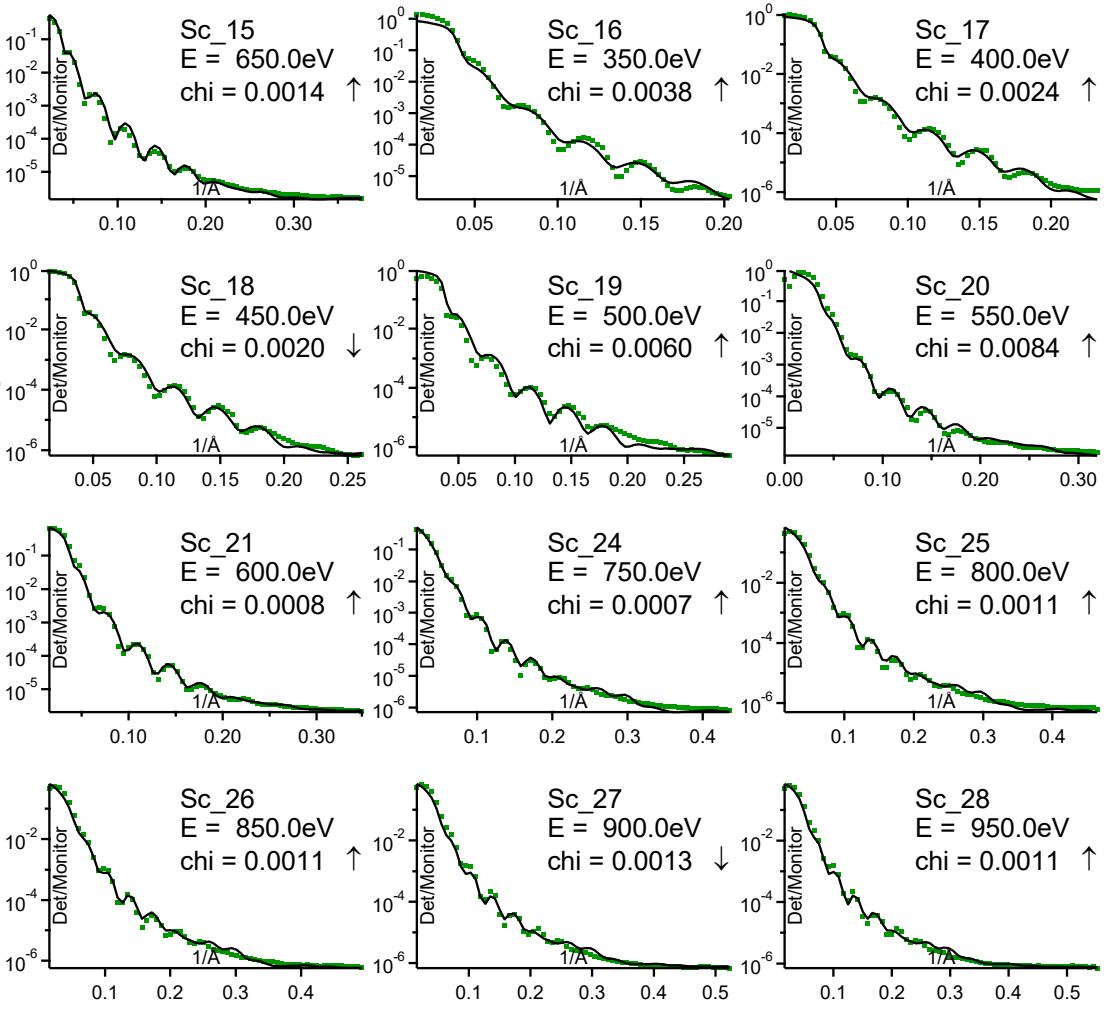
Fe optical constants, Chantler



Chemical profile

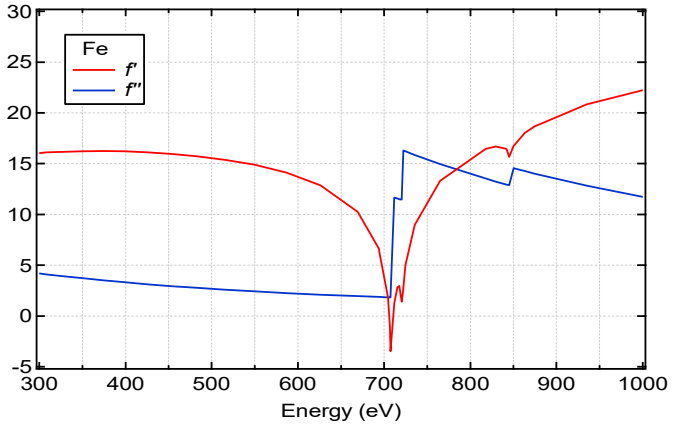


Off-resonance spectra, E = 350–1000eV

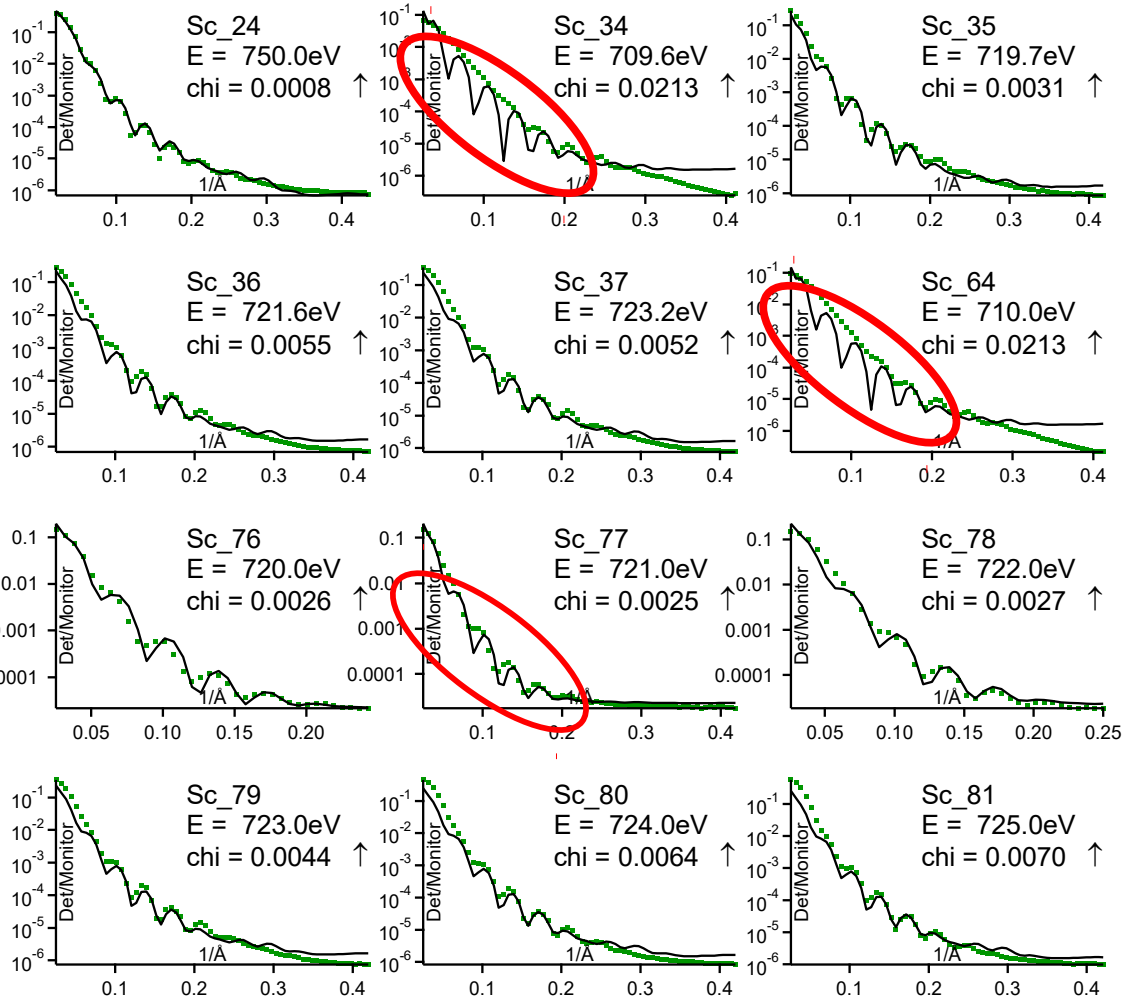


"Real life" example: ZnO/Fe_3O_4

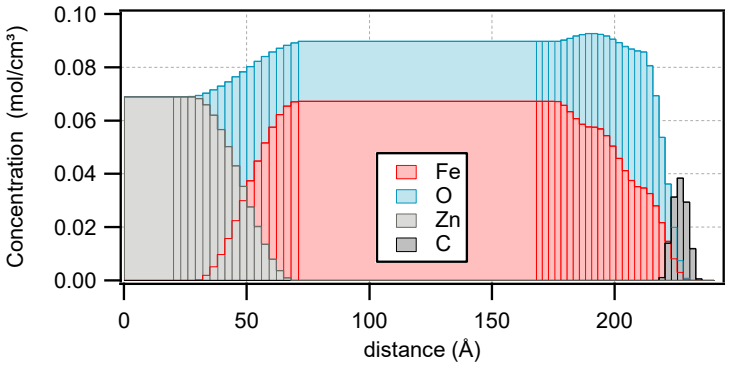
Fe optical constants, Chantler



on-resonance spectra, E = 700–730eV

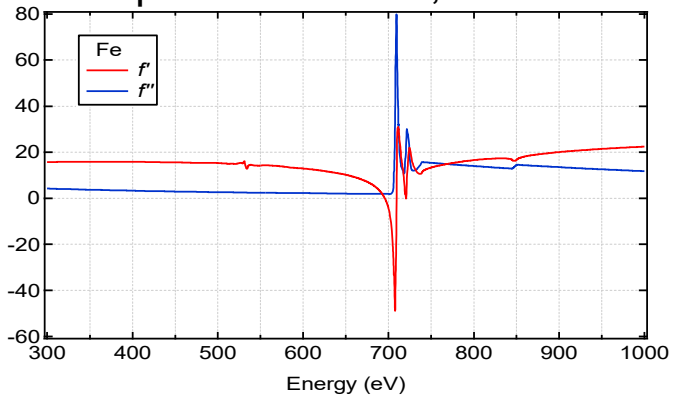


Chemical profile

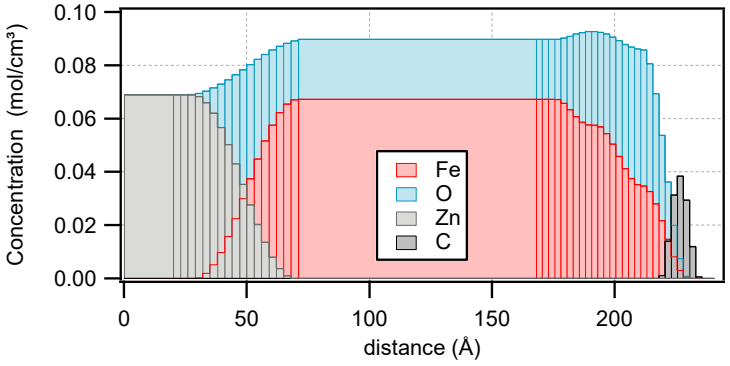


"Real life" example: ZnO/Fe_3O_4

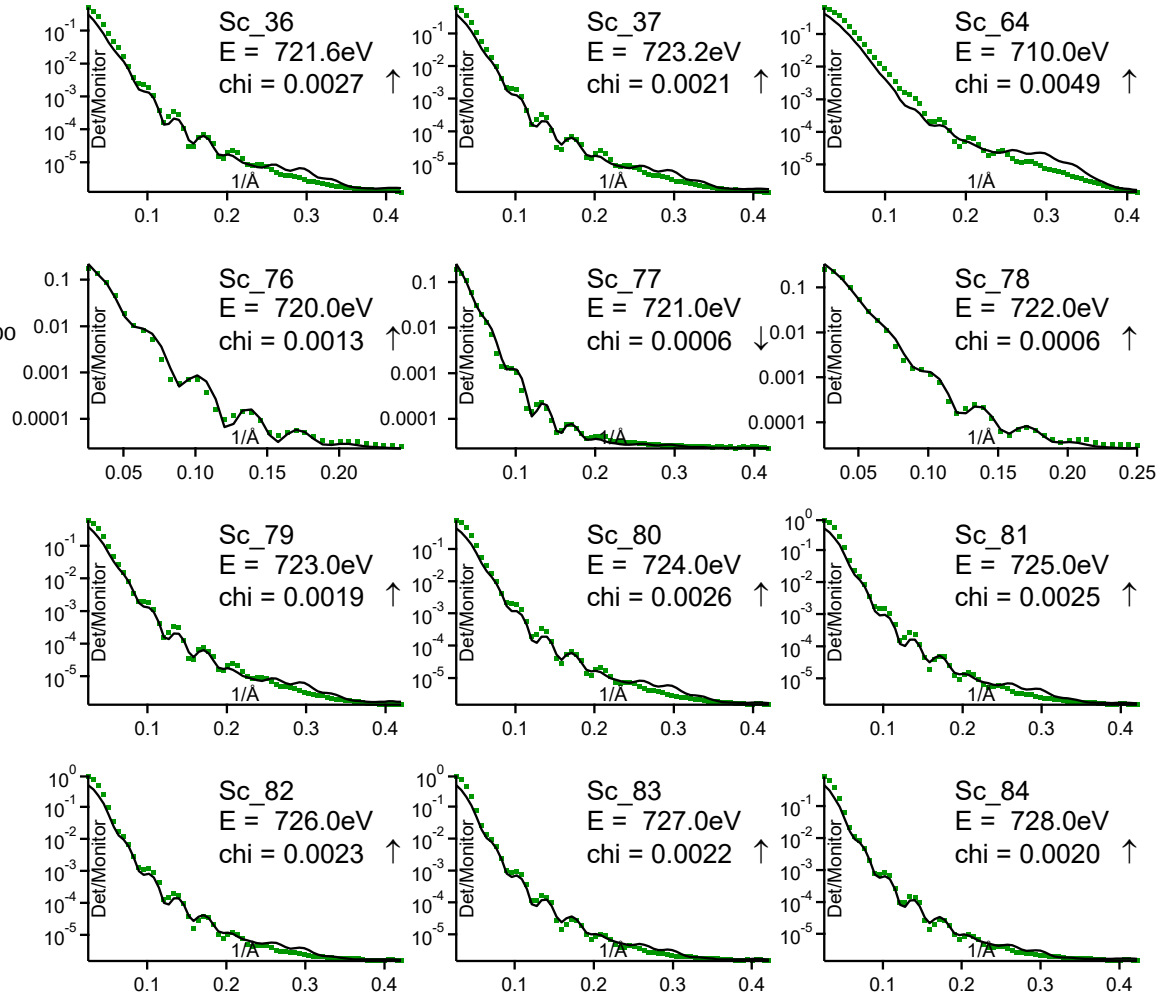
Fe optical constants, corrected



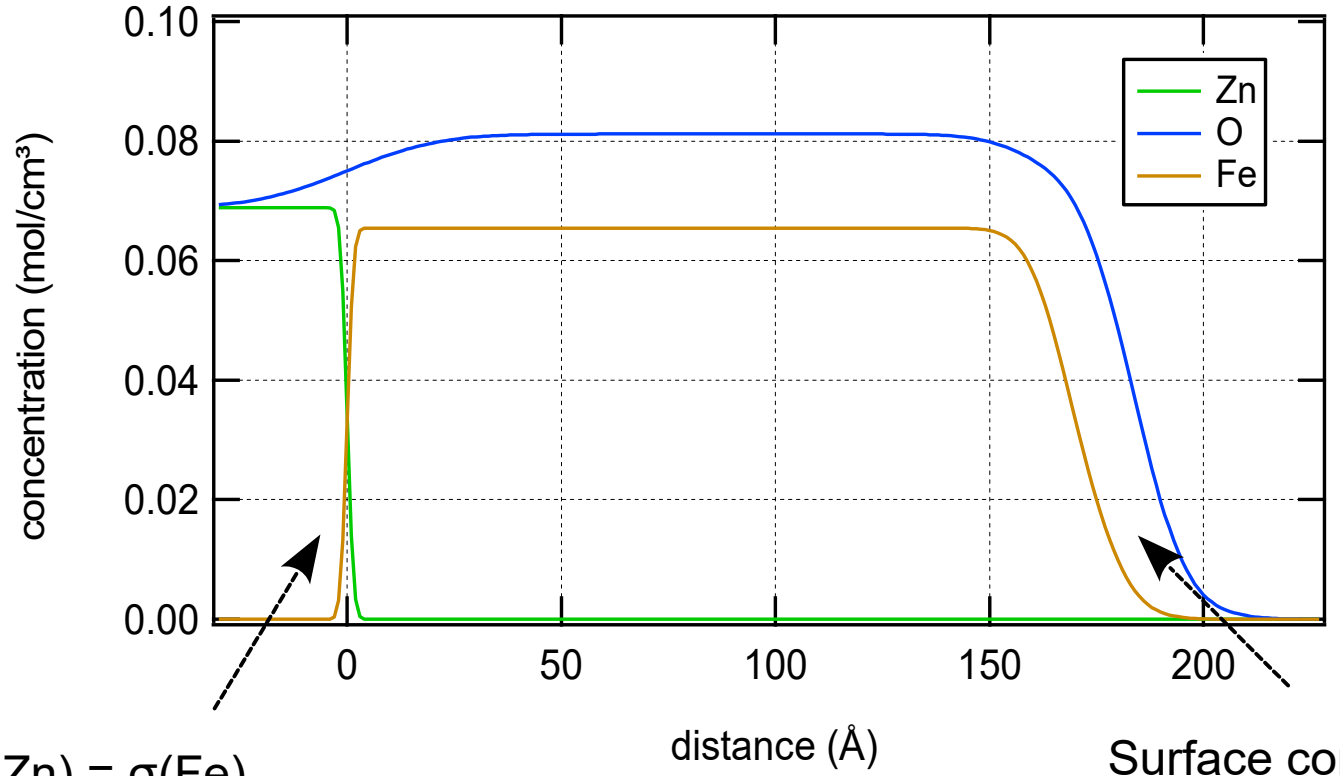
Chemical profile



on-resonance spectra, E = 700–730eV



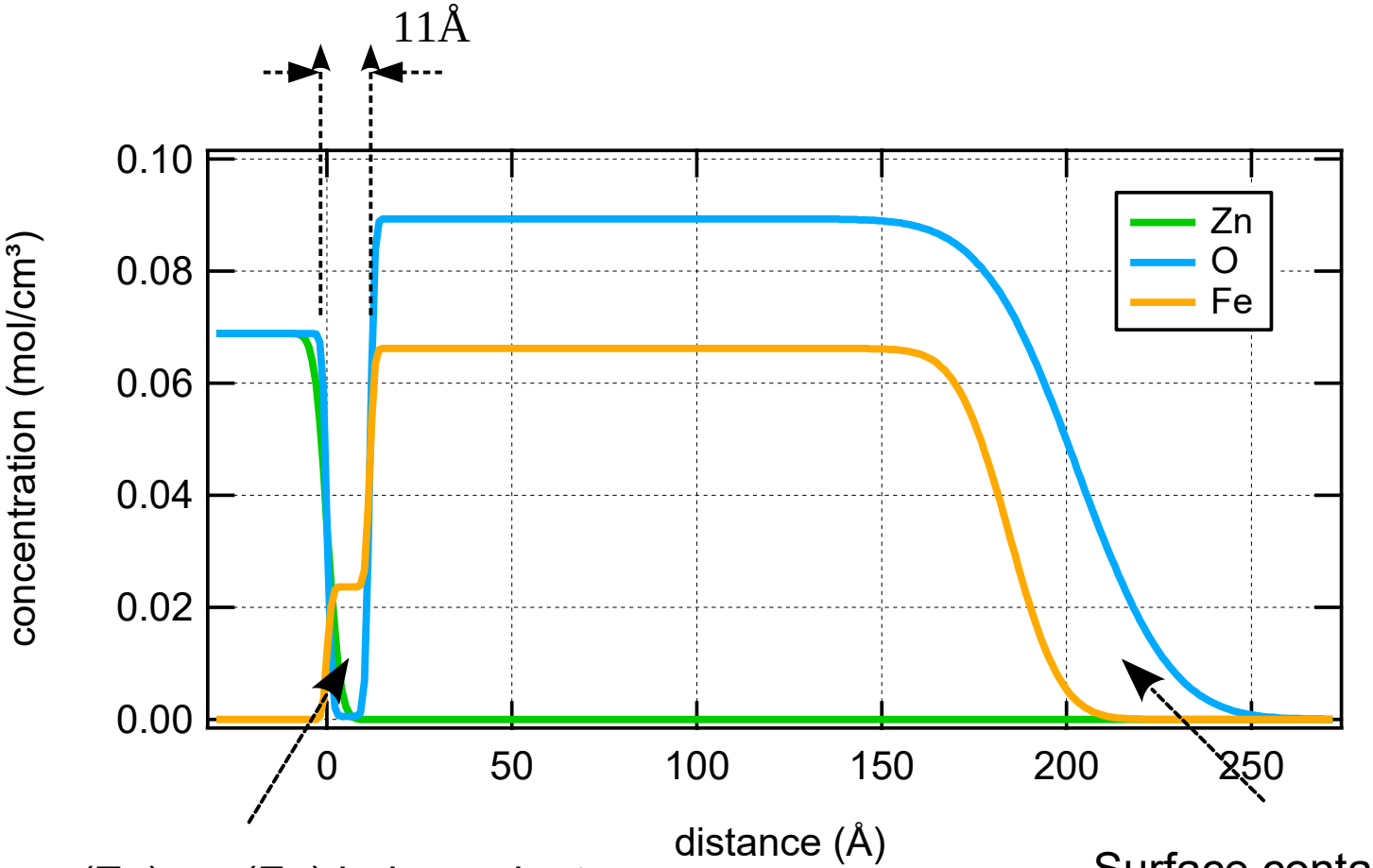
Ozan190 – O terminated



σ(Zn) = σ(Fe)
independent from σ(O)

Surface contaminaton
effectively accounted
by oxygen

Ozan194 – Zn terminated



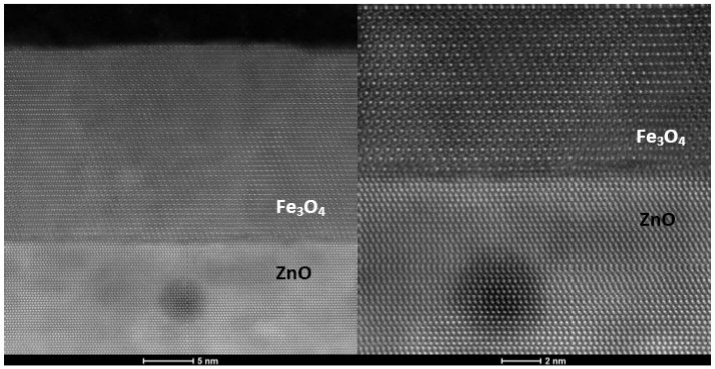
σ(Zn) = σ(Fe) independent from σ(O)

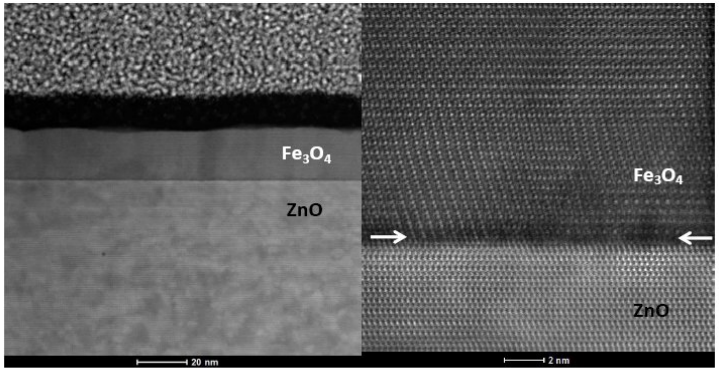
Surface contaminaton effectively accounted by oxygen

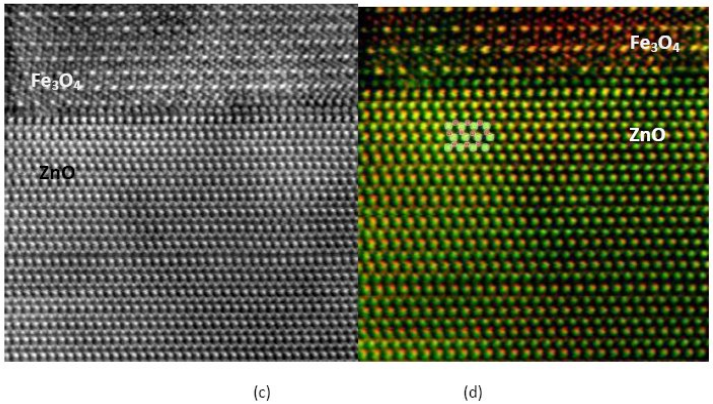
Comparison to STEM

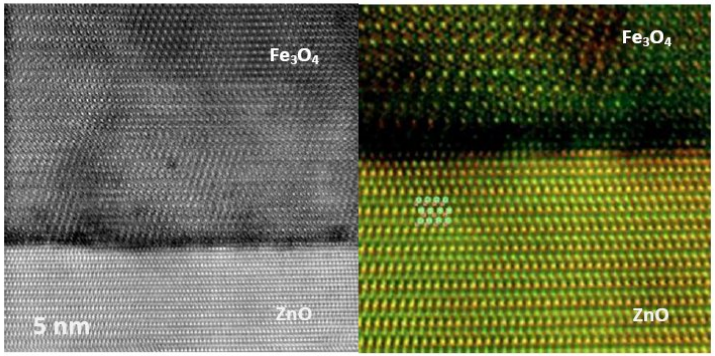
O terminated

Zn terminated









exfoliation?

O. Kirilmaz et al. in preparation

Optical constants

1. Theoretical calculation away from resonances

- *Chantler et al. AIP **652**, 370 (2003)*
- *Chantler et al. J Phys Chem Ref Data **24**, 71 (1995)*
- *Chantler et al. J Phys Chem Ref Data **29**, 597 (2000)*

Theoretical Form Factor, Attenuation and Scattering Tabulation for $Z = 1–92$ from $E = 1–10$ eV to $E = 0.4–1.0$ MeV

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School of Physics, University of Melbourne, Parkville, Victoria 3052, Australia[a]

Received September 27. 1994; revised manuscript received October 5. 1994

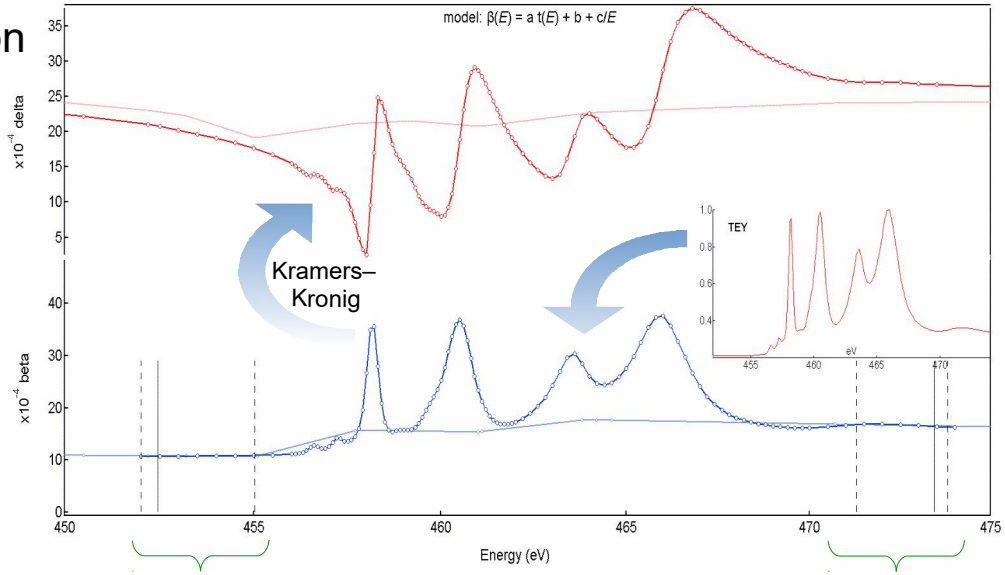
Tables for form factors and anomalous dispersion are widely used in the UV, x-ray and γ-ray communities, and have existed for a considerable period of time. Much of the recent

next most red 563 pages:

TABLE 1: Form factors, attenuation and scattering cross-sections, $Z = 1–92$, from $E = 0.001–0.010$ keV to $E = 400–1000$ keV — Continued.

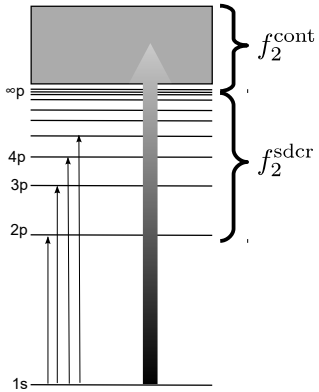
E keV	f_1 e/atom	f_2 e/atom	μ cm²/g	σ(coh+inc) cm²/g	μ_K cm²/g	λ nm
4.118862E+00	1.64400E+01	3.8997E+00	9.9405E+02	2.1733E+00	8.922E+02	3.010E−01
4.334821E+00	1.80390E+01	3.5640E+00	8.6321E+02	2.0998E+00	7.731E+02	2.860E−01
4.633924E+00	1.88984E+01	3.1991E+00	7.2483E+02	2.0037E+00	6.518E+02	2.676E−01
4.953664E+00	1.93754E+01	2.8820E+00	6.1083E+02	1.9080E+00	5.503E+02	2.503E−01
5.295467E+00	1.96982E+01	2.5972E+00	5.1493E+02	1.8133E+00	4.647E+02	2.341E−01

2. Total Electron Yield used as absorption

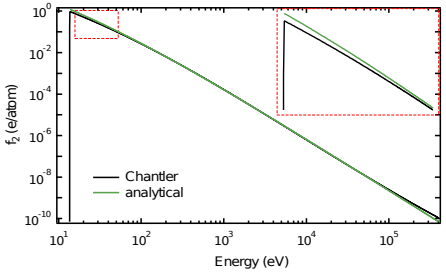


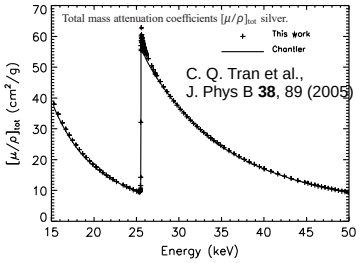
Hydrogen atom

H atom is a suitable test system, since its discrete and continuous spectrum of is known analytically



Comparison to C.T. Chanter data:





$f_2 \rightarrow \sigma_{abs} \rightarrow$ transition rate $\rightarrow$ matrix element

$$f_2(\hbar\omega) = \frac{\omega\sigma_{\text{abs}}}{4\pi c r_e} = \frac{\omega\sigma_{\text{abs}}}{4\pi c}\frac{4\pi\epsilon_0 mc^2}{e^2} = \frac{\omega\sigma_{\text{abs}}\epsilon_0 mc}{e^2} = \frac{2m\hbar}{A_z^2 e^2}\frac{dw}{dt}$$

$$\frac{dw}{dt} = \frac{2\pi}{\hbar}\sum_{\mathbf{n}}|F_{\mathbf{n}_0,\mathbf{n}}|^2 \times \frac{1}{\pi}\text{Im}\left[\frac{1}{(E_{\mathbf{n}} - E_{\mathbf{n}_0} - \hbar\omega) - i\lambda}\right], \lambda \to 0$$

Comparison to experimental polarizability:

$$\alpha' = \lim_{\omega\to 0}\alpha'(\omega) = r_e c^2 \lim_{\omega\to 0}\frac{f_1(\omega)}{\omega^2}$$

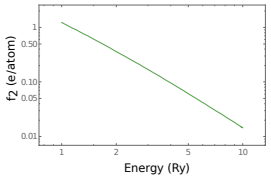
$$\alpha'_{\text{tot}} = \alpha'_{\text{dscr}} + \alpha'_{\text{cont}} = (5.424 + 1.239)\cdot 10^{-31} \approx$$

$$\approx 6.66 \cdot 10^{-31}\text{m}^3$$

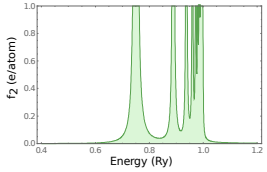
$$\alpha'_{\text{exp}} = 6.67 \cdot 10^{-31}\text{m}^3$$

Analytical expressions for $f_2(E)$ can be derived:

$$f_2^{\text{cont}}(\hbar\omega) = \frac{2m\hbar}{A_z^2 e^2}\frac{dw}{dt} = \frac{64\pi}{3}\left(\frac{Ry}{\hbar\omega}\right)^3 \frac{1}{(e^{2\pi/\kappa_0} - 1)}\exp\left[\frac{4\arctan(1/\kappa_0)}{\kappa_0}\right]$$

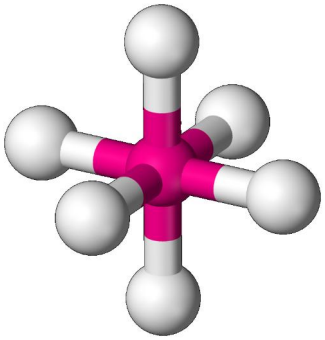


$$f_2^{\text{dscr}}(\hbar\omega) = \frac{128\pi}{3}\left(\frac{\hbar\omega}{Ry}\right)^2 \sum_{n=2}^{+\infty}\frac{1}{n^3}\left(\frac{n-1}{n+1}\right)^{2n}\frac{1}{\left(1 - \frac{1}{n^2}\right)^5} \times \frac{1}{\pi}\text{Im}\left[\frac{1}{(1 - \frac{1}{n^2} - \frac{\hbar\omega}{Ry}) + i\frac{\lambda}{Ry}}\right]$$

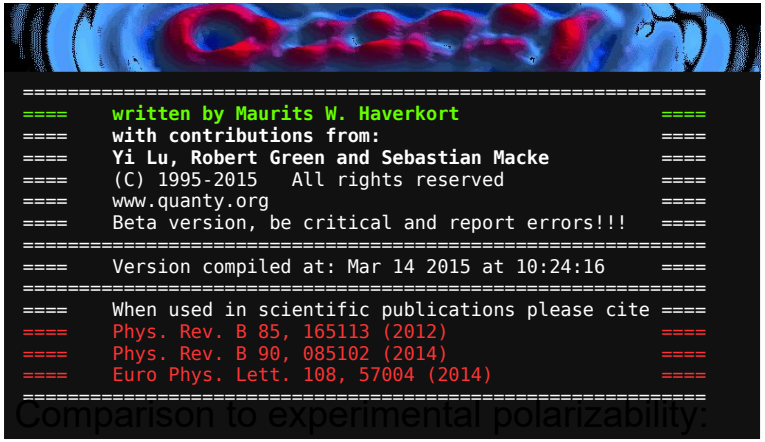


Theoretical optical constants

Crystal/Ligand field cluster calculation

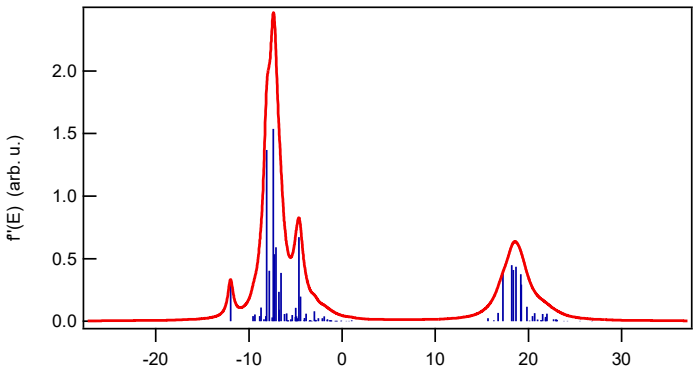


$$\hat{H}_{\mathrm{CM}} = \sum_{\mu\sigma} \varepsilon_{\mu\sigma} \hat{l}^{\dagger}_{\mu\sigma} \hat{l}_{\mu\sigma} + \sum_{m\sigma} \varepsilon_{m\sigma} \hat{c}^{\dagger}_{m\sigma} \hat{c}_{m\sigma} + \sum_{n\sigma} \varepsilon^{2p}_{n\sigma} \hat{c}^{\dagger}_{n\sigma} \hat{c}_{n\sigma}$$
$$+ \sum_{\mu m\sigma} \left(t_{m\mu} \hat{c}^{\dagger}_{m\sigma} \hat{l}_{\mu\sigma} + t_{\mu m} \hat{l}^{\dagger}_{\mu\sigma} \hat{c}_{m\sigma} \right)$$
$$+ \frac{1}{2} \sum_{mm'm''m'''} \sum_{\sigma,\sigma'} U^{3d-3d}_{mm'm''m'''} \hat{c}^{\dagger}_{m\sigma} \hat{c}^{\dagger}_{m'\sigma'} \hat{c}_{m'''\sigma} \hat{c}_{m''\sigma'}$$
$$+ \sum_{mm'nn'} \sum_{\sigma,\sigma'} U^{3d-2p}_{mnm'n'} \hat{c}^{\dagger}_{m\sigma} \hat{c}^{\dagger}_{n\sigma'} \hat{c}_{n'\sigma} \hat{c}_{m'\sigma'}$$
$$+ \frac{\lambda^{3d}_{\mathrm{SO}}}{2} \sum_{mm',\sigma\sigma'} \left[\underline{\hat{L}} \cdot \underline{\hat{S}} \right]_{m\sigma,m'\sigma'} \hat{c}^{\dagger}_{m\sigma} \hat{c}_{m'\sigma'}$$
$$+ \frac{\lambda^{2p}_{\mathrm{SO}}}{2} \sum_{nn',\sigma\sigma'} \left[\underline{\hat{L}} \cdot \underline{\hat{S}} \right]_{n\sigma,n'\sigma'} \hat{c}^{\dagger}_{n\sigma} \hat{c}_{n'\sigma'}$$



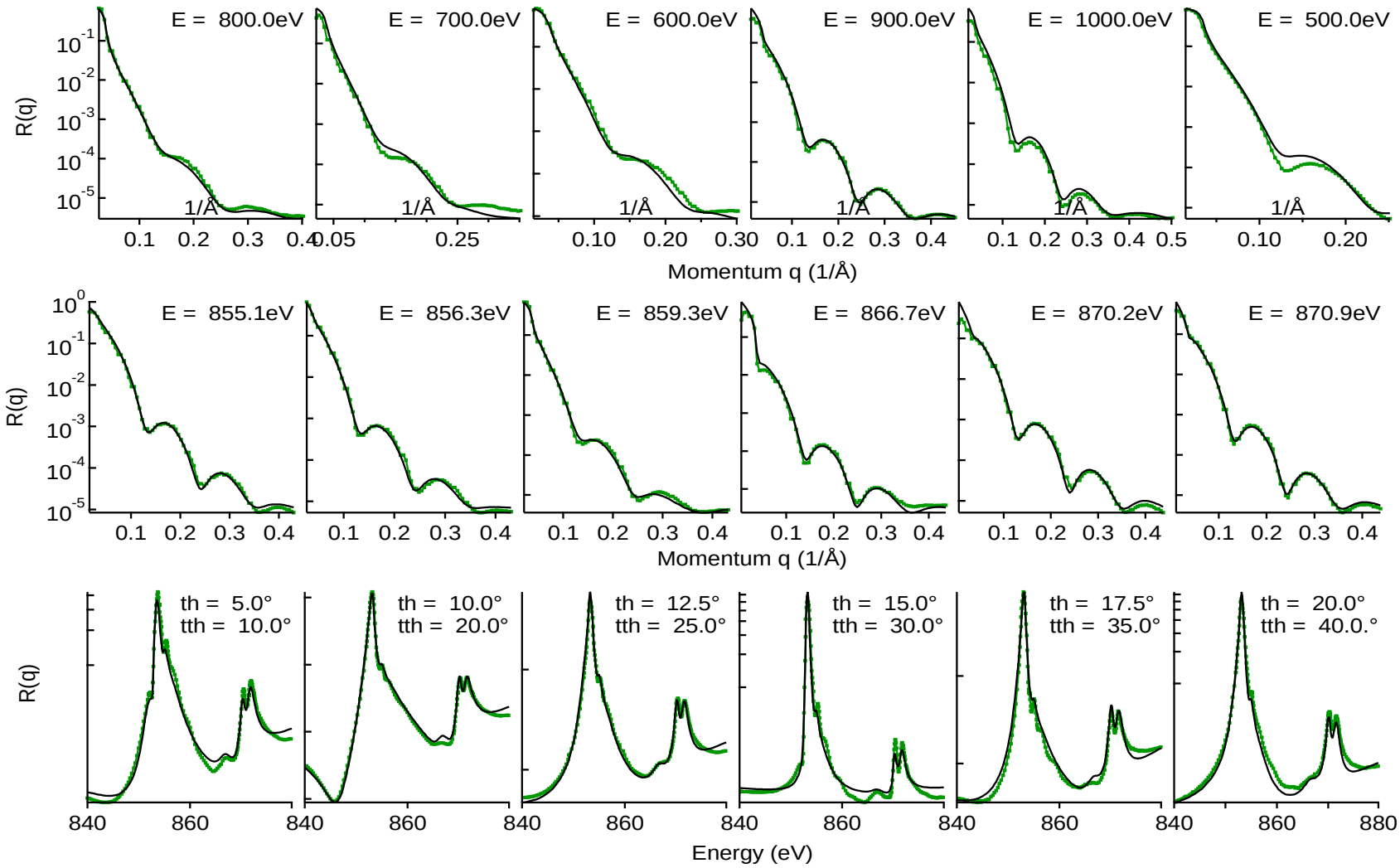
Comparison to experimental polarizability:

$$f_2^{\mathrm{res}}(E) \sim \sum_f \left| \langle \Psi_i | \cos\theta | \Psi_f \rangle \right|^2 \mathrm{Im} \left[\frac{1}{(E_f - E_i - E) - i\Gamma} \right]$$



*M. W. Haverkort et al. PRB **85**, 165113 (2012)*

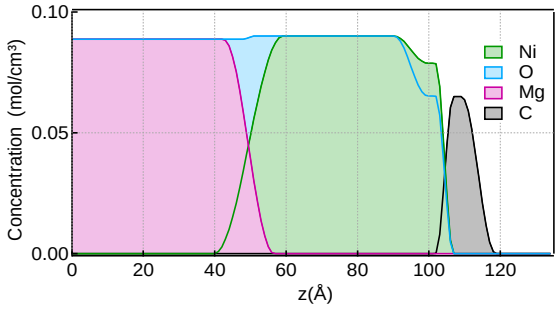
NiO – Theory restricted fit

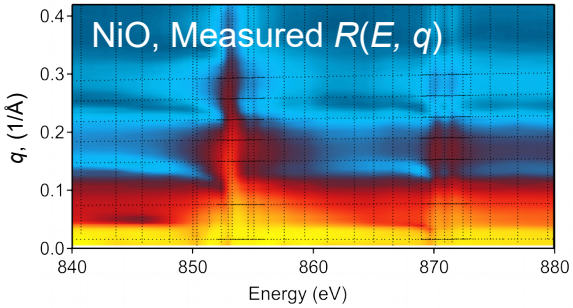


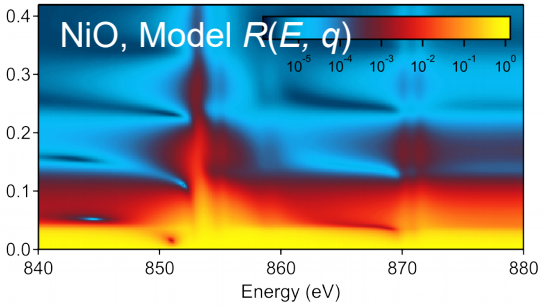
NiO – Theory restricted fit

$$f_2^{\mathrm{res}}(E) = \boxed{\frac{mE^2}{\hbar^2}\,|\langle R_{2p}|r|R_{3d}\rangle|^2}\sum_f |\langle \Psi_i|\cos\theta|\Psi_f\rangle|^2\,\mathrm{Im}\left[\frac{1}{(E_f - E_i - E) - i\Gamma}\right].$$

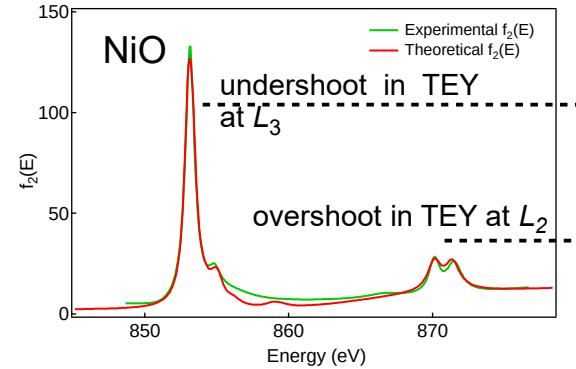
off-resonant

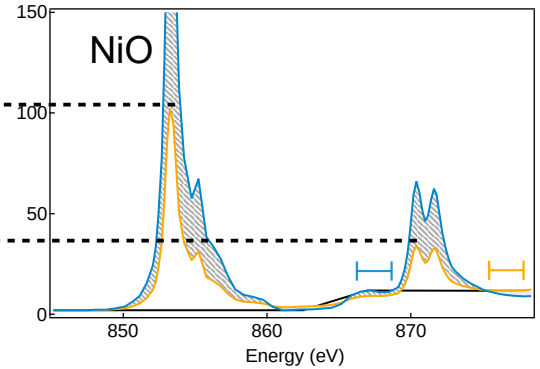






on-resonant

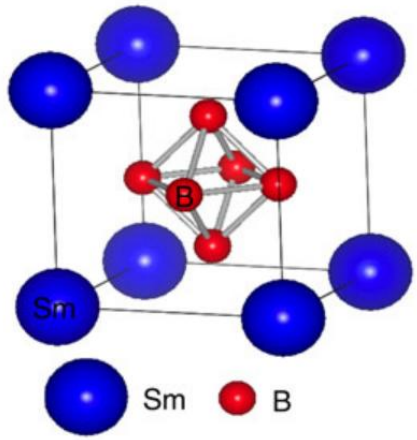




	10Dq	T_{pp}	ζ_{3d}	$F^{(2)}_{dd}$	$F^{(4)}_{dd}$	ζ_{2p}	$F^{(2)}_{2p3d}$	$G^{(1)}_{2p3d}$	$G^{(3)}_{2p3d}$	V_{eg}	V_{t2g}
NiO, starting values	0.56	0.72	0.08	11.14	6.87	11.51	6.67	4.92	2.80	2.06	1.21
fitted values	0.50	0.74	0.10	12.51	7.72	11.32	7.49	5.53	3.15	1.88	1.26

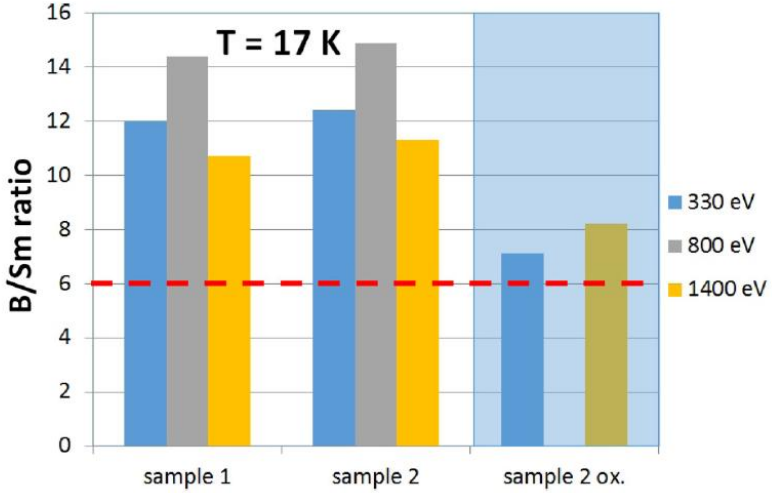
*K. Fürsich, V. Zabolotnyy, E Schierle et al. PRB **97**, 165126 (2018)*

SmB_6 — Intro



- Mixed valent system
- Kondo insulator
- Topologically protected ingap surface state

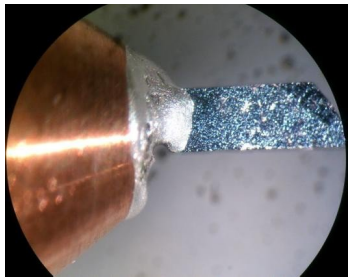
*M. Neupane et al. Nat Comm **4**, 2991 (2013)*



Disrupted stoichiometry at the cleaved surface?

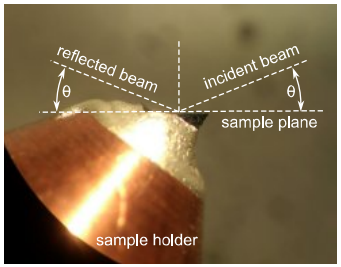
*Heming et al. B **90**, 195128 (2014)*

SmB_6 – samples

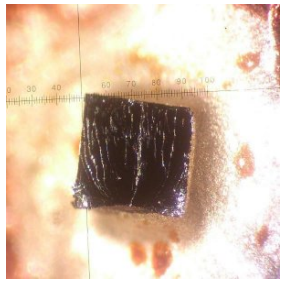


Often samples with polished surfaces are used for resonant x-ray experiments

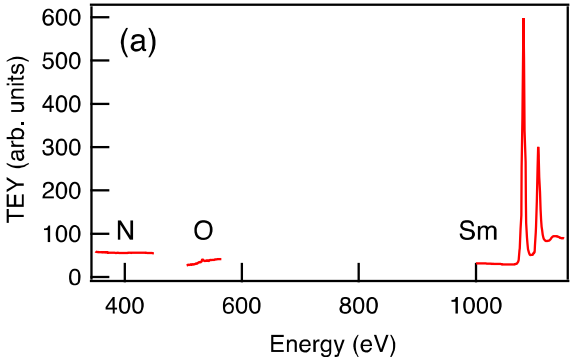
- Surface contaminated and unstable
- It is too rough for reflectivity



We use floating-zone samples, cut in rods, pre-oriented



- Cleavage in fast entry at 10^{-8} mBar, transfer to $2*10^{-10}$ mBar in reflectivity chamber
- Cleaves along (100) even if cut along (111)
- Surface size ~ 1mm x 1mm
- Sufficiently large terraces (beam ~ 100 μm)



No indications of oxygen in TEY spectrum

SmB_6 – a multivalent system

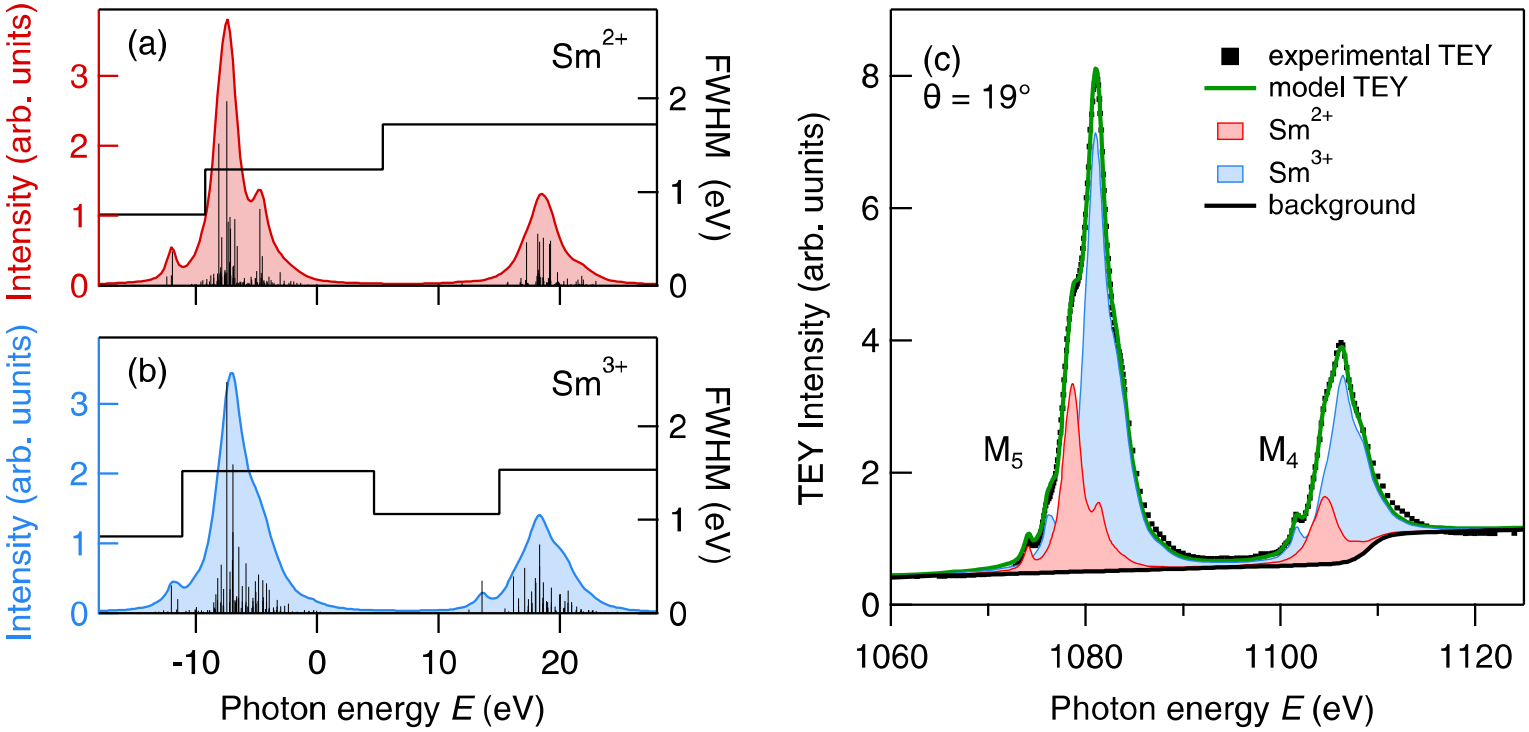
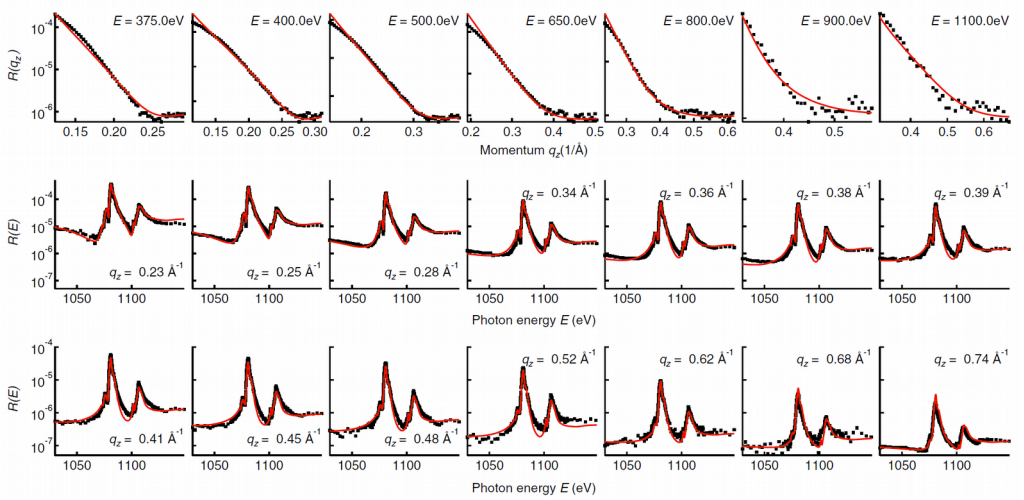
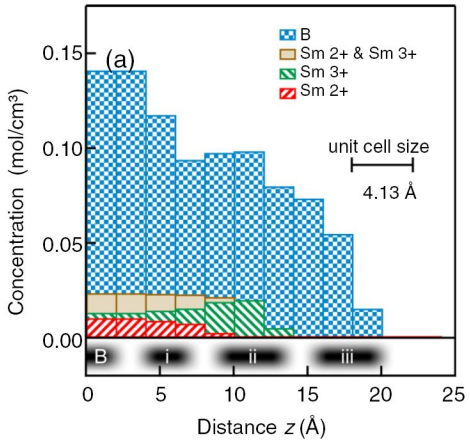


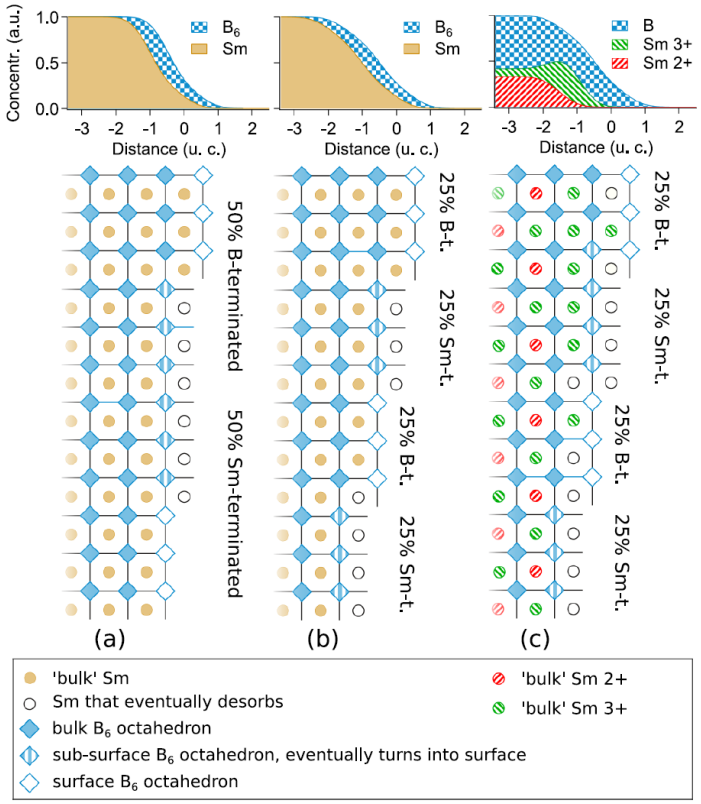
TABLE I. Optimized CFT parameters for Sm^{2+} and Sm^{3+} ions.

Ion	State	Configuration	$F^{(2)}_{ff}$	$F^{(4)}_{ff}$	$F^{(6)}_{ff}$	ζ_{4f}	$F^{(2)}_{df}$	$F^{(4)}_{df}$	$G^{(1)}_{df}$	$G^{(3)}_{df}$	$G^{(5)}_{df}$	ζ_{3d}
Sm^{2+}	Initial	$3d^{10}4f^6$	10.828	6.751	4.845	0.136						
	Final	$3d^9\ 4f^7$	11.548	7.218	5.185	0.165	6.701	3.075	4.670	2.734	1.888	10.514
Sm^{3+}	Initial	$3d^{10}4f^5$	10.950	6.873	4.945	0.152						
	Final	$3d^9\ 4f^6$	11.548	7.260	5.227	0.180	7.211	3.337	5.086	2.979	2.058	10.510

SmB_6 – a multivalent system







V. Zabolotnyy, K. Fürsich, R. J. Green et al. PRB ***97****, 205416 (2018)*

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ep4

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