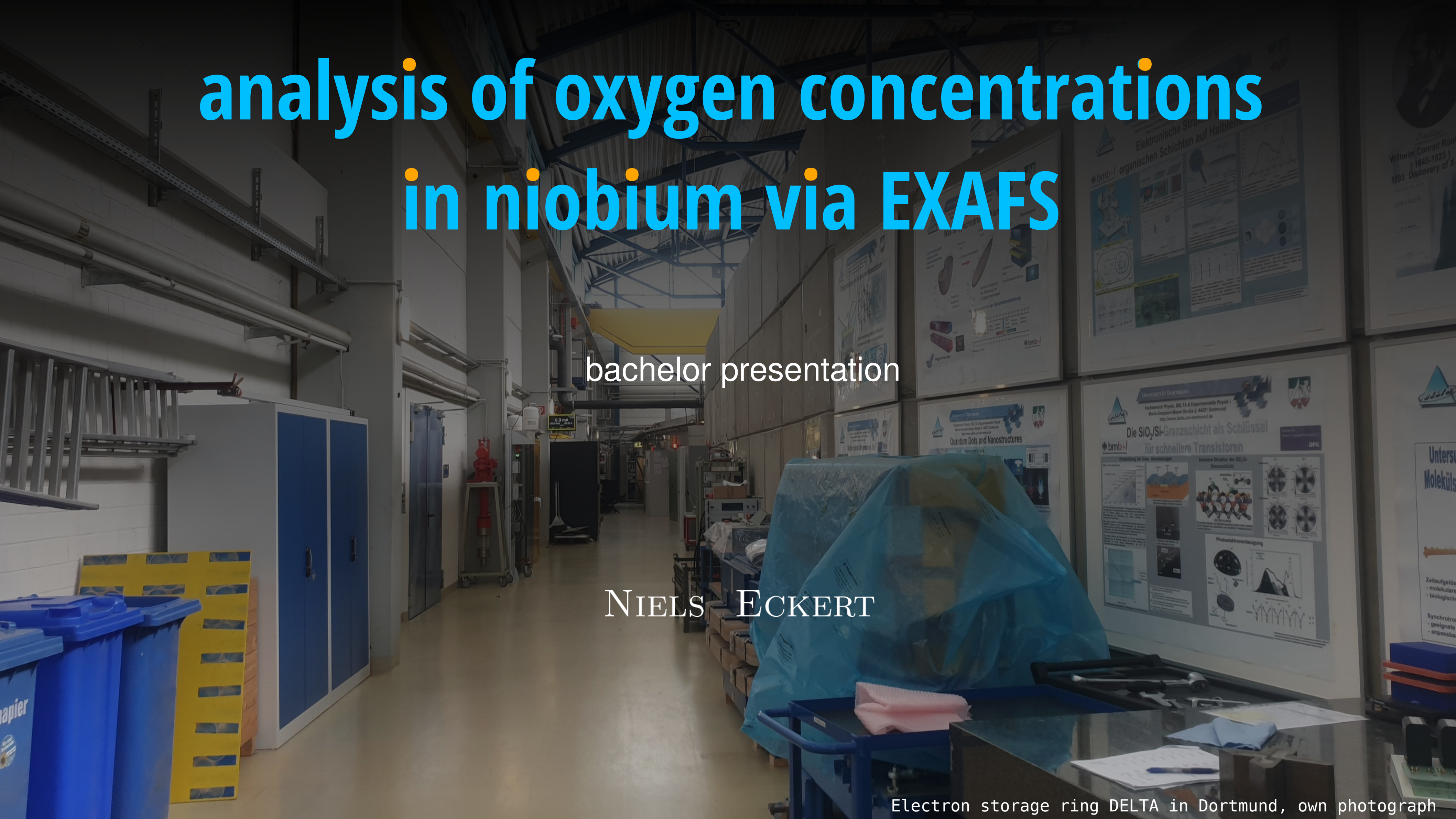
analysis of oxygen concentrations in niobium via EXAFS

bachelor presentation

NIELS ECKERT



Electron storage ring DELTA in Dortmund, own photograph

Table of general content

1. What **MSL-related** problem do I connect to?

2. Which **specific field** do I contribute to?

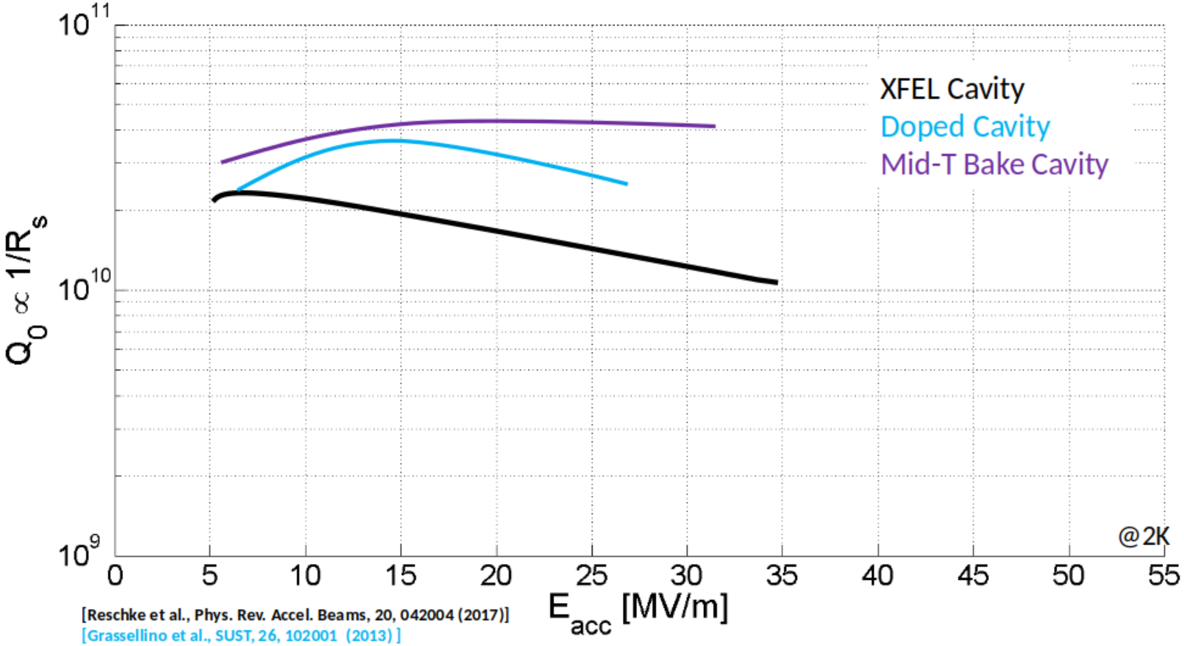
3. What’s the **greater field** this is leading to?

4. What are my **thesis plans?**

Link to the MSL group

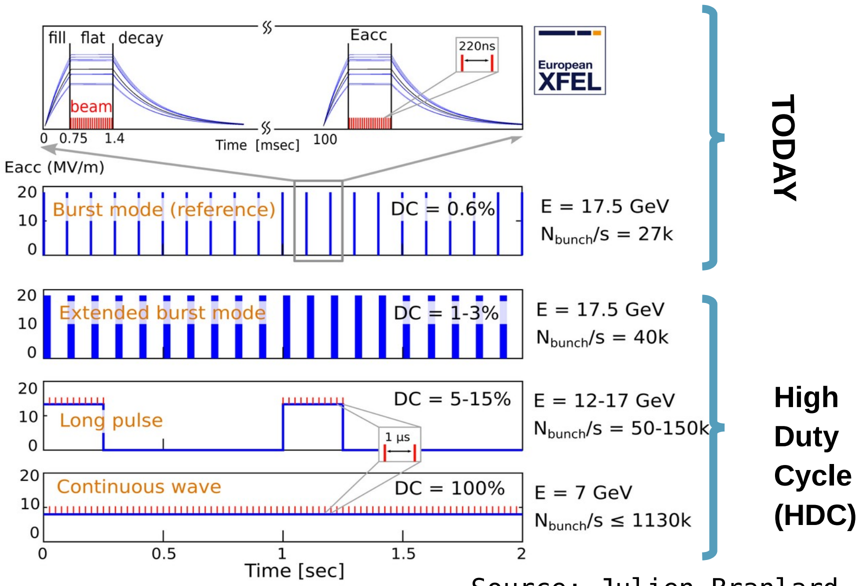
In acceleration physics, we'd like to take our SRF cavities to the extreme

Acceleration gradient performance



[Reschke et al., Phys. Rev. Accel. Beams, 20, 042004 (2017)]
[Grassellino et al., SUST, 26, 102001 (2013)]
[Posen et al., Phys. Rev. Applied 13, 014024 (2020)]

Duty cycle of accelerator



Source: Julien Branlard

<u>But:</u> thermal or magnetic quenches eventually occur

Link to the MSL group

known empirical result: **impurities of niob <u>can</u> improve performance**

observed for:

Nitrogen impurity (Grassellino, 2013)

Oxygen impurity (Posen, 2019)

my interest

<u>Problem of interest:</u> **Which impurities lead to improvements and which make it worse?**

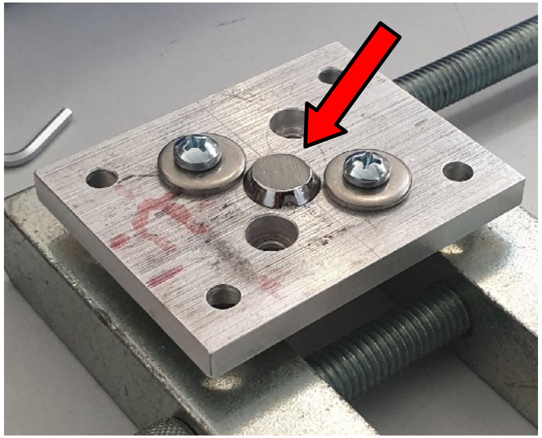
Link to the MSL group

Idea:

1. Take a piece of cavity material and analyze its crystallographic structure ← my contribution

2. Check for cavity performance of that material

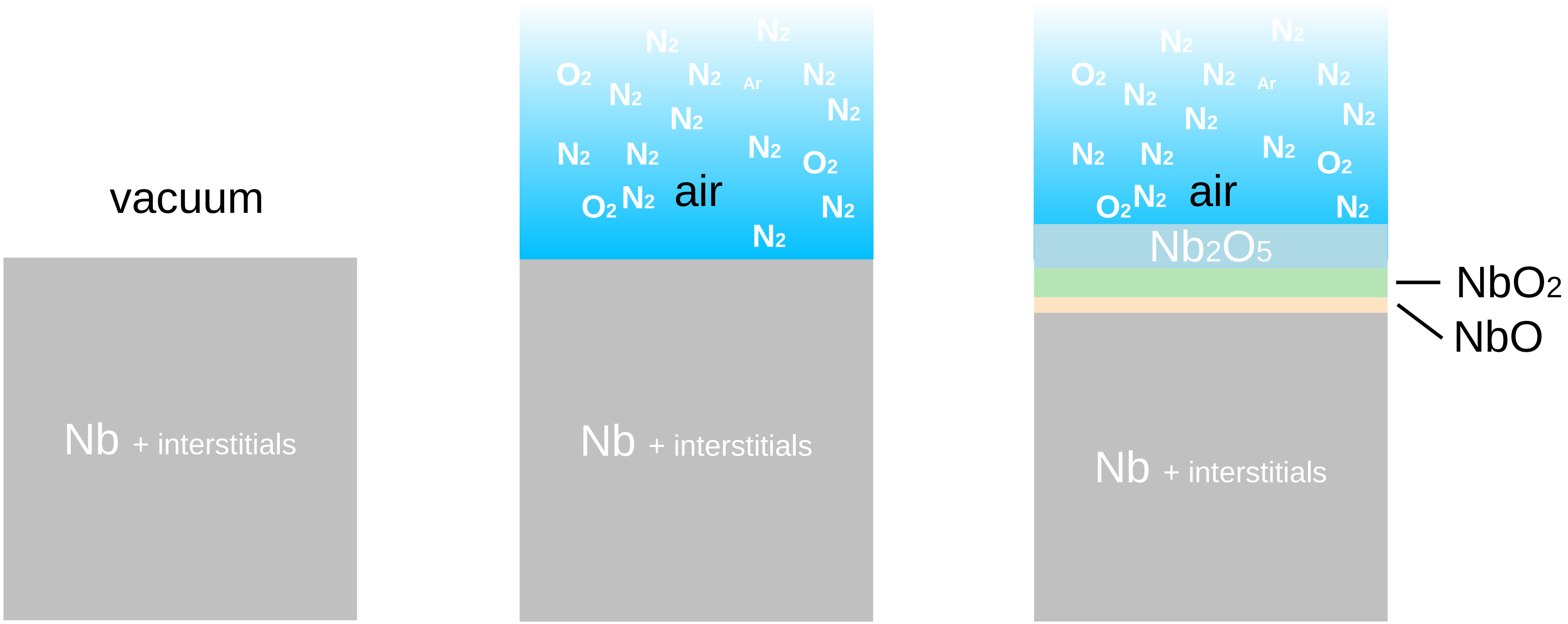
3. Infer physical correlations



Truncated cone as a cavity sample on a mount, own photograph

Oxidation of Nb crystals

natural impurity



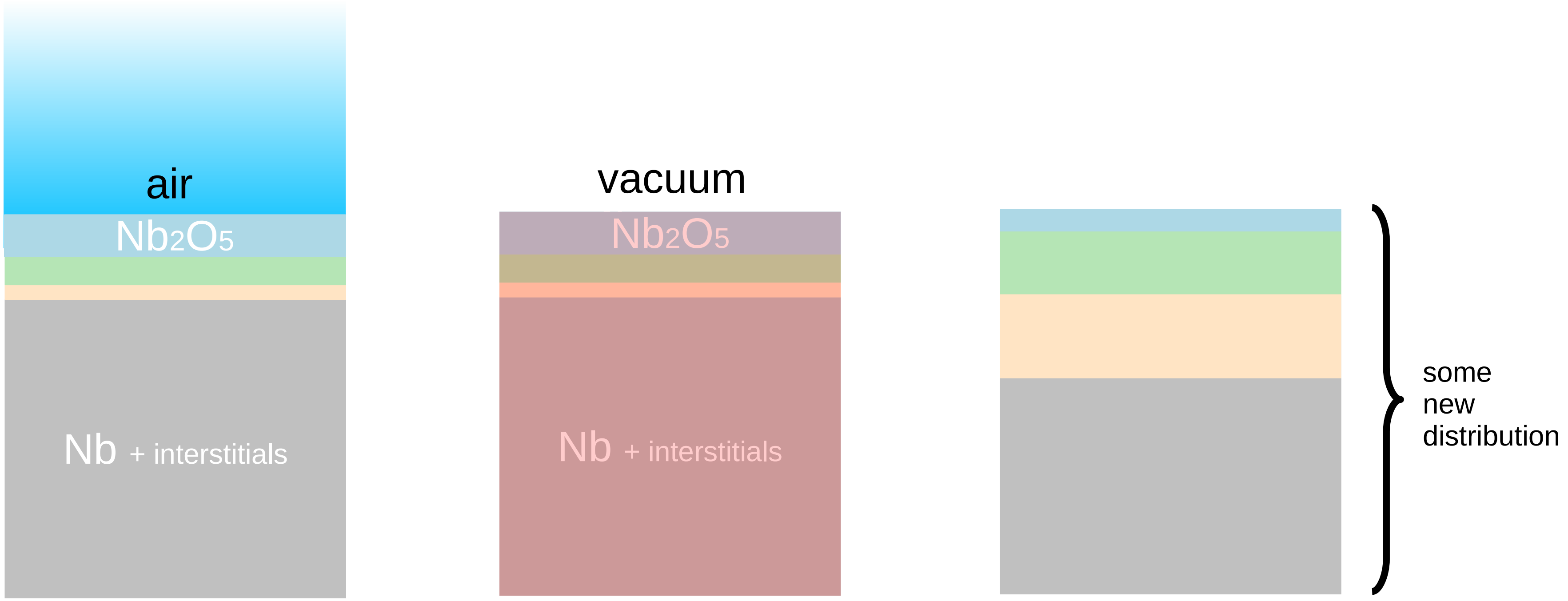
<u>before oxidation:</u>
pure Nb crystal

<u>while oxidation:</u>
Oxygen diffuses
into Nb crystal at 20°C

<u>after oxidation:</u>
interstitials + 3 layers of oxides

Heat treatments of cavities

intentional impurity



<u>before baking</u>

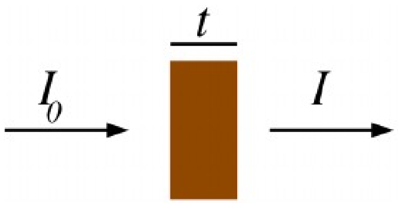
<u>in the oven:</u>
kinetic energy
activates diffusion processes

<u>after baking:</u>
O atoms diffuse deep
into the sample, feeded
through the oxide layer

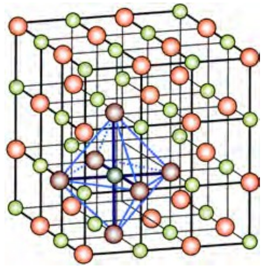
Analyzation of crystallographic structures via EXAFS

The general topic I am contributing to

Investigate absorption behaviour at certain energies → Information about coordination numbers and lattice constants



Newville, 2004



Lecture Schroer, 2024

Described by the quantum mechanical EXAFS - **e**xtended **x**-Ray **a**bsorption **f**ine **s**tructure

Recap: Essence of quantum mechanics

In quantum mechanics some principles are replaced by others

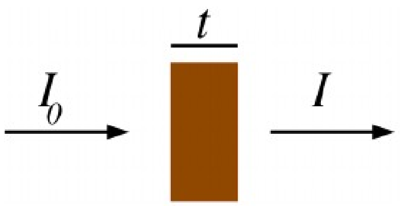
 locality ➡ states as wave functions $|\psi\rangle$

 causality ➡ interaction by probability $|\langle f|V|i\rangle|^2$

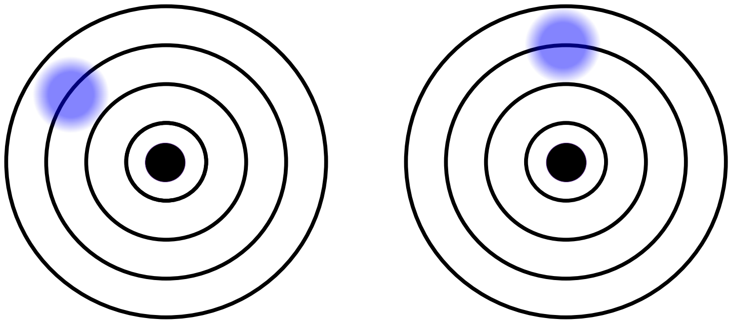
properties ➡ operators and eigenvalues $\hat{H}|\psi\rangle = E|\psi\rangle$

It remains essential to **be clear about the model used**.
What objects exist and which interactions can occur?

Basic model of EXAFS



Interaction between an γ of a radiation source and two atoms (representing an solid):
An absorbing atom and an reflecting atom,
each having at least one **tightly bound** e^-

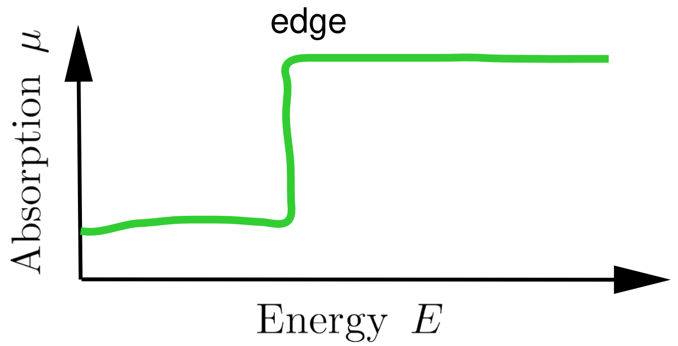


$:=$ Solid

<u>Objective:</u> What does the **absorption curve** look like?

What does the absorption curve look like?

Photoelectric effect:



What does the absorption curve look like?

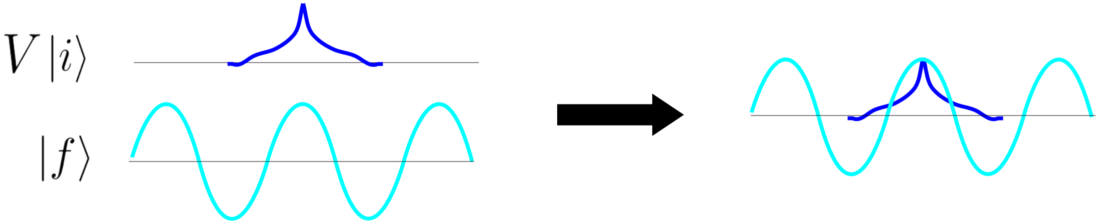
<u>Fermis golden rule:</u> Transition rate between an **initial** $|i\rangle$ and a **final** $|f\rangle$ eigenstate of a system under a **perturbation** V is given by

incoming photon

$$\Gamma_{f\leftarrow i} = \frac{2\pi}{\hbar}\rho(E_f)\boxed{|\langle f|\,V\,|i\rangle|^2}$$

measure for the overlap
of the states wave functions

more overlap



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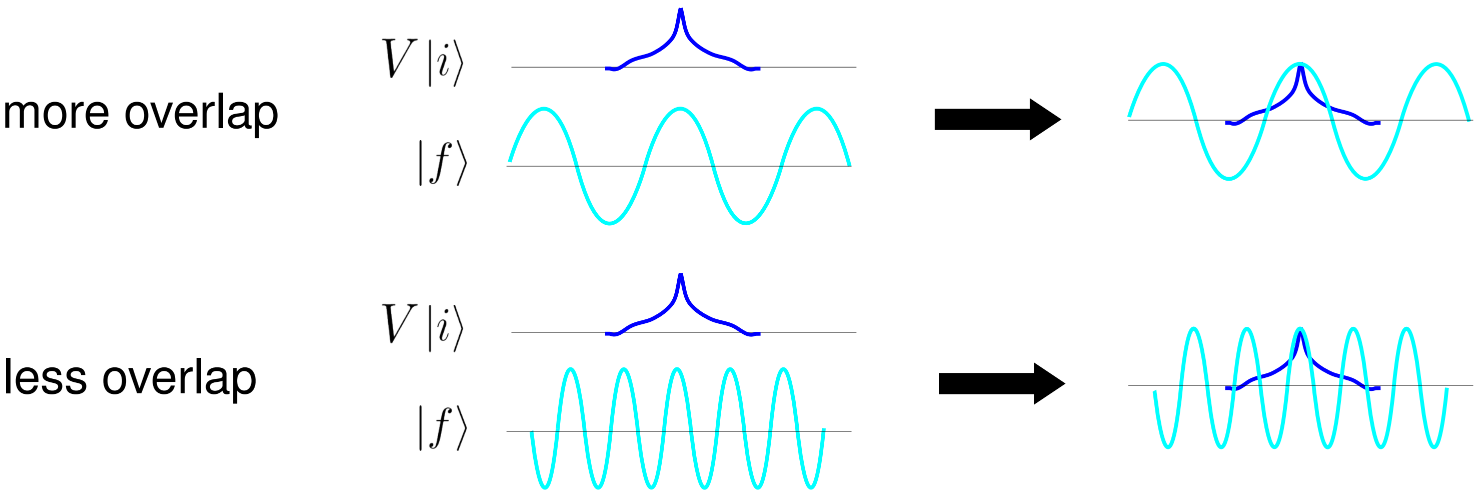
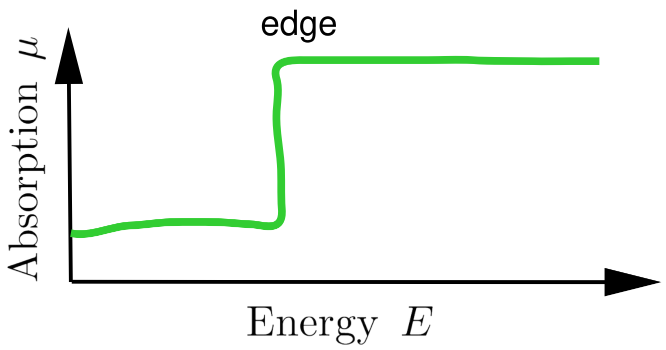


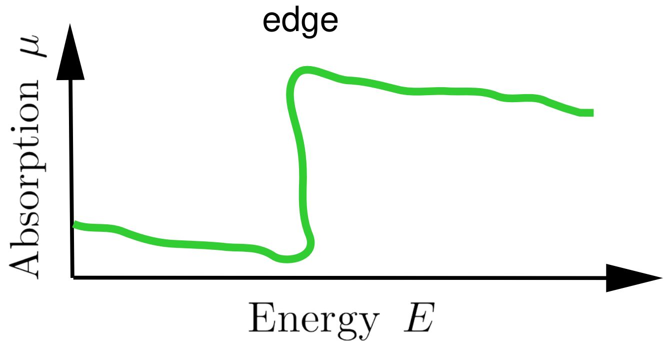
More energetic photons ionize electrons less often

What does the absorption curve look like?

Photoelectric effect:



State overlap:



What does the absorption curve look like?

Now EXAFS comes into play:

The ionized electron $|f\rangle$ can get reflected at the neighboring atom and superpose with itself into $|f'\rangle = |f\rangle + |r\rangle$

$$\mu(E) \;=\; \mu_0(E)(1+\chi(E)) \overset{\mu \propto \Gamma_{f' \leftarrow i}}{\propto} |\langle f'| V |i\rangle|^2$$

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$$= |\langle f| V |i\rangle|^2 + 2\mathrm{Re}(\langle f| V |i\rangle \overline{\langle r| V |i\rangle}) + |\langle r| V |i\rangle|^2$$

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$$= \underbrace{|\langle f| V |i\rangle|^2}_{=:\, \mu_0} \left(1 + \underbrace{\frac{2\mathrm{Re}(\langle f| V |i\rangle \overline{\langle r| V |i\rangle})}{|\langle f| V |i\rangle|^2}}_{=:\, \chi \propto \langle r| V |i\rangle} + \underbrace{\frac{|\langle r| V |i\rangle|^2}{|\langle f| V |i\rangle|^2}}_{\approx 0}\right)$$

What does the absorption curve look like?

Investigate $\chi(E)$ *further*

So we've got:

$$\boxed{\begin{aligned}&\mu_0(E) \propto |\langle f| V |i\rangle|^2 \\ &\chi(E) \propto \langle r| V |i\rangle\end{aligned}}$$

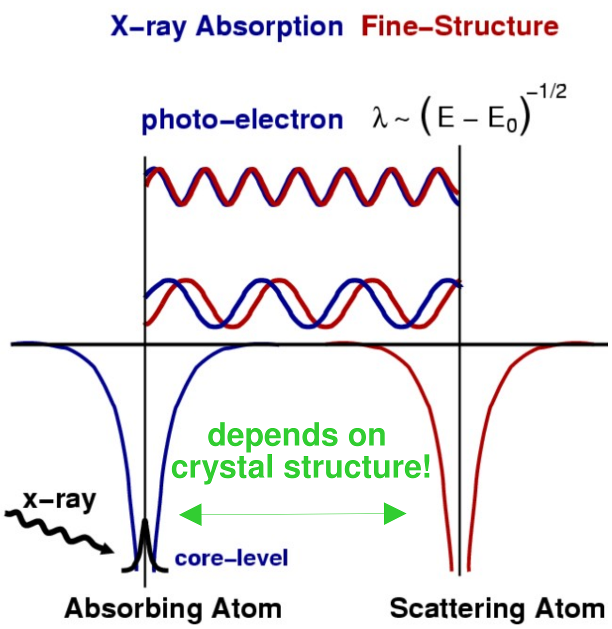
describes the downwards trend

V is approx. proportional to a spherical wave e^{ikr}

Because of tight bond

$$\Rightarrow \chi(E) \propto \langle i| V |r\rangle \approx \int dr\delta(r)e^{ikr}\psi_{\text{refl}}(r) = \psi_{\text{refl}}(0)$$

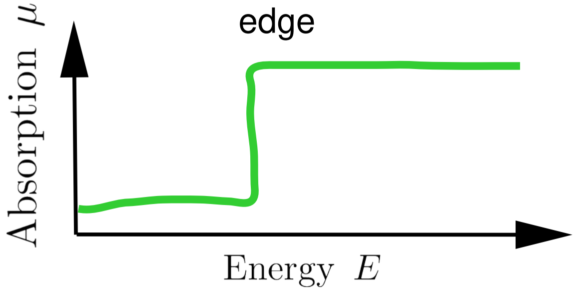
$\longrightarrow$ $\chi(E)$ describes an <u>oscillation</u>



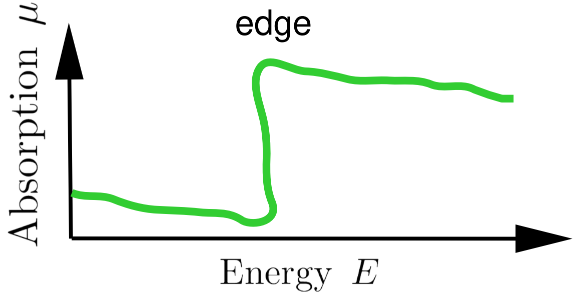
Source: Newville, 2004

What does the absorption curve look like?

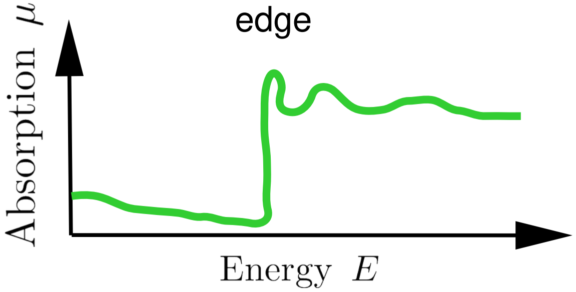
Photoelectric effect:



State overlap:



Reflection on neighboring atom:

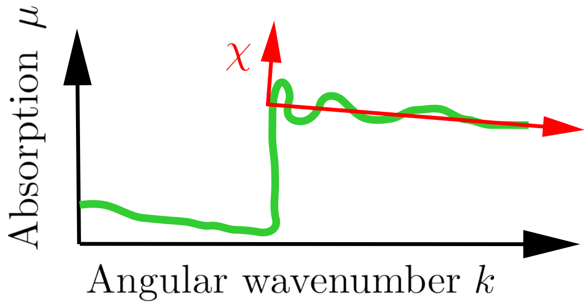


This is EXAFS

What does the absorption curve look like?

Deduction of the EXAFS equation

Taking **additional aspects** into account leads to a proper description of EXAFS oscillation



The EXAFS equation

$$\chi(k) = \sum_j \frac{N_j e^{-2k^2\sigma_j^2} e^{-2R_j/\lambda(k)} f_j(k)}{kR_j^2} \sin(2kR_j + \delta_j(k))$$

k	angular wavenumber	λ	mean-free-path of photo-electron				
j	index of coordination sphere	N	no. of neighbors in that sphere	R	radius of that sphere	σ	std. dev. of thermal displacement

$f_j(k)$ $\delta_j(k)$

scattering properties of reflecting atom

My thesis plans

deductive

Simulate EXAFS curves
(varying concentration distributions)

compare with actual measurements
for qualitative similarities

inductive

Analyze Nb samples
(multiple heat treatments)

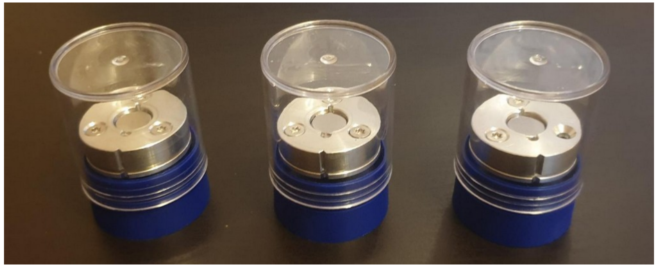
calculate
concentration distributions

check for similarities between
own theory and own measurements

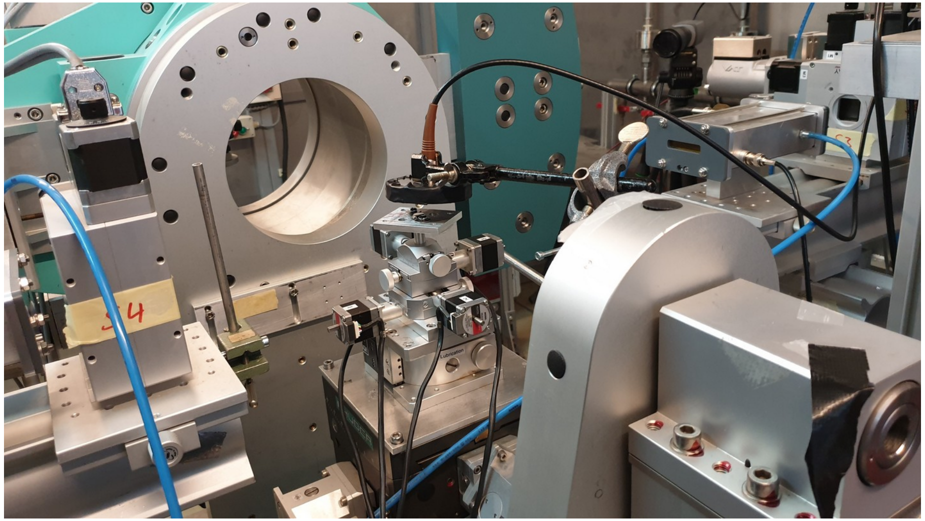
State of affairs

Measurements of 3 samples at DELTA last week:

- Sample 243: 3h@300°C
- Sample 245: 20h@250°C
- Sample 247: 48h@120°C



Used samples, own photograph



experimental setup, own photograph

Work in progress...

Overview of the taken measurements at DELTA. Each successfull scan took 23 min

apparently bad measurement	anscheinend ggf. schlechte Messung	unknown content	not useable but measured

Sample 243	ϑ = 0.1°		ϑ = 0.15°		ϑ = 0.17°		ϑ = 0.2°		ϑ = 0.3°		ϑ = 0.5°		ϑ = 0.7°		ϑ = 1°	
	Day	Night	Day	Night	Day	Night	Day	Night	Day	Night	Day	Night	Day	Night	Day	Night
# spectra:	6		8		7		3		5		4		3		4	
300°C	34	62	38	65	43	68	47	71	50	76	53	86	56	88		59
	35	63	39	66	44	69	48	72	51	85	54		57			60
3h	36	64	40	67	45	70	49	73	52		55		58			61
	37		41		46			74								89
mid-T			42					75								

Sample 245	ϑ = 0.1°		ϑ = 0.15°		ϑ = 0.17°		ϑ = 0.2°		ϑ = 0.3°		ϑ = 0.5°		ϑ = 0.7°		ϑ = 1°	
	Day	Night	Day	Night	Day	Night	Day	Night	Day	Night	Day	Night	Day	Night	Day	Night
# spectra:	5		5		5		5		5		5		5		4	
250°C	159	196	162	178	170		208	181	210	184	213	187	216	190	219	193
	160	197	163	179	171		209	182	211	185	214	188	217	191		194
20h	161		164	180	172			183	212	186	215	189	218	192		195
	175			198	174											
	176			199	200											
	177				201											
					203											
					204											

Sample 247	ϑ = 0.1°		ϑ = 0.15°		ϑ = 0.17°		ϑ = 0.2°		ϑ = 0.3°		ϑ = 0.5°		ϑ = 0.7°		ϑ = 1°	
	Day	Night	Day	Night	Day	Night	Day	Night	Day	Night	Day	Night	Day	Night	Day	Night
# spectra:	6		7		7		6		6		6		6		6	
120°C	99	126	102	128	108	131	111	134	114	136	117	138	120	140	155	123
	100	127	103	129	109	132	112	135	115	137	118	139	121	141		124
48h	101	144	104	130	110	133	113	147	116		119		122			125
				145		146			152		153		154			142
EXFEL Recipe																143

Scan history	
1	~~aborted exafsscan~~
2-16	*ascan*
17	~~aborted exafsscan~~
18-33	*ascan*
34 - 76	**exafsscan**
77 - 81	~~invalid exafsscan~~
82 - 84	*ascan*
85 - 86	**exafsscan**
87	~~aborted exafsscan~~
88 - 89	**exafsscan**
90 - 98	*ascan*
99 - 104	**exafsscan**
105 - 107	*ascan*
108 - 147	**exafsscan**
148	~~aborted exafsscan~~
149 - 151	*ascan*
152 - 155	**exafsscan**
156 - 158	*ascan*
159 - 164	**exafsscan**
165 - 166	~~invalid exafsscan~~
167 - 169	*ascan*
170 - 172	**exafsscan**
173	~~invalid exafsscan~~
174 - 201	**exafsscan**
202	~~aborted exafsscan~~
203 - 204	**exafsscan**
205 - 207	*ascan*
208 - 219	**exafsscan**
220	~~aborted exafsscan~~

Beam history

Beam	Beamshut afterwards:
1	calibration
	measurements
2	recalibration
	measurements
	new sample calibration
	measurements
3	recalibration
	measurements
4	recalibration
	measurements
	new sample calibration
	measurements
5	recalibration
	measurements
6	recalibration
	measurements

Sample
243
247
245

Thank you.

Setup of the beamline experiment

For EXAFS analysis at DELTA, Dortmund