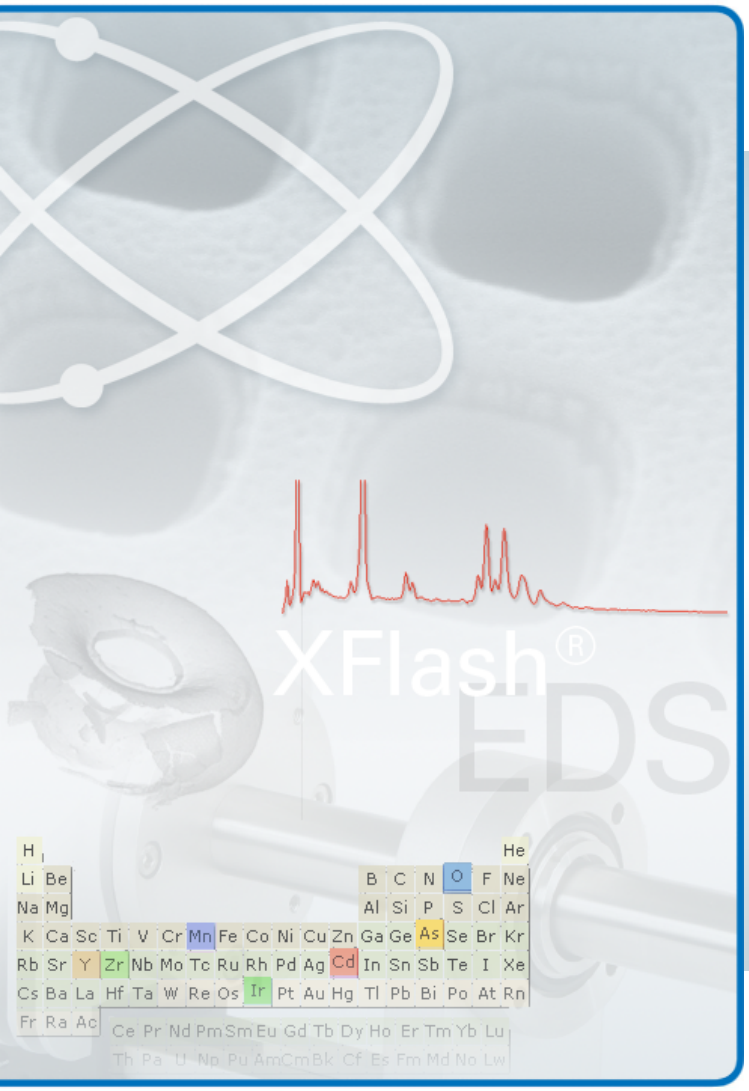



EDS User School

# Possible Application Mistakes with EDS analysis

Mats Eriksson
Spectral Solutions AB

# Errors sources in EDS analysis

**Adjustment of EDS and SEM**

- Working distance/ Tilt angle
- Accelerating voltage
- Pulse throughput / energy range
- Count rate

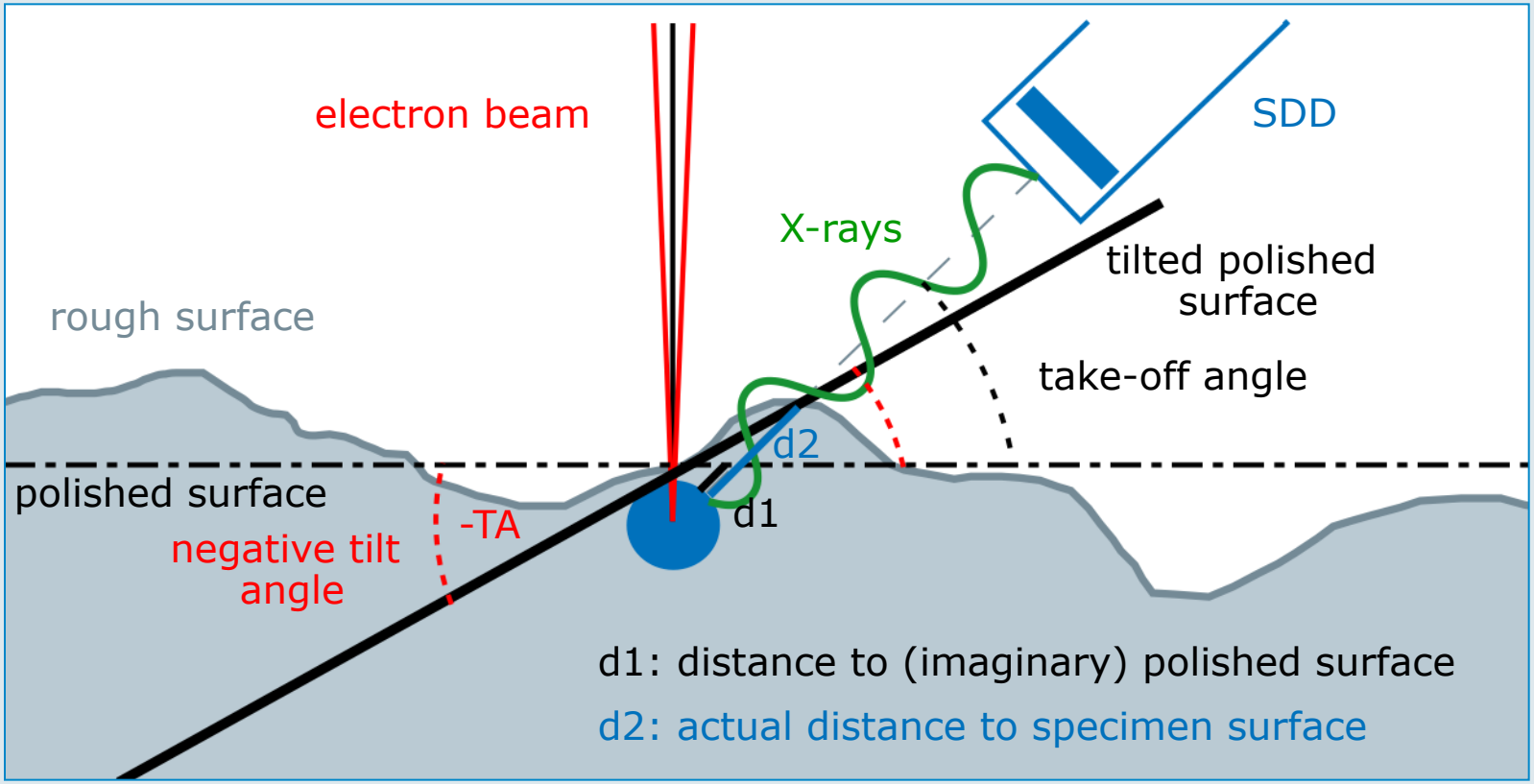
**Sample specificities**

- Sample homogeneity
- Surface structure
- Charging

**Miscellaneous**

- Energy / channel calibration
- Beam focusing

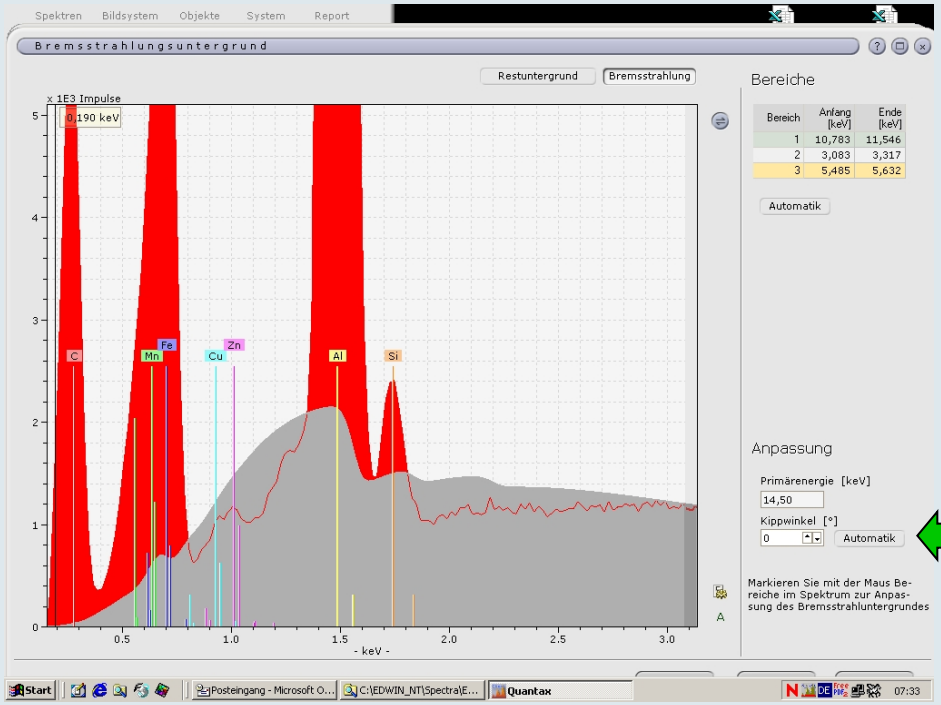
# Sample surface and absorption



# Sample surface of sample

## Undefined geometrical conditions:

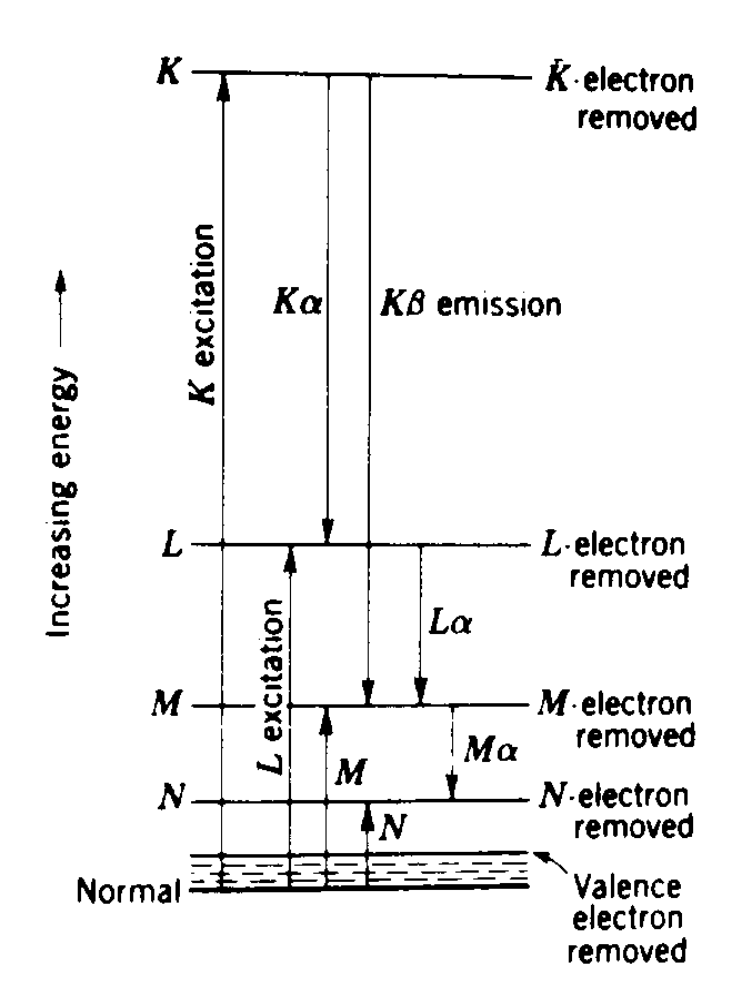
- **Samples without preparation**
- **Rough surface**
- **Particle**




**Correction of virtual tilt angle**

# Critical Ionization Energy

- Generation of X-ray photon requires ionization event
- Minimum energy to produce event has specific value: critical ionization energy ($E_c$)
- $E_c$ is different for each shell and subshell.
- Inner shell electrons (K) have highest $E_c$
- Incident beam energy high enough to excite K shell can excite X-rays from all other shells

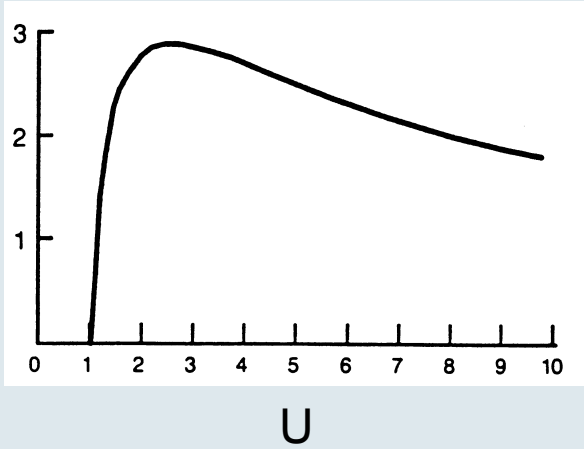


# Optimum overvoltage

The ionization cross-section describes the probability that a particular event will take place:

$$\text{For X-rays: } Q = 6.5\text{x}10^{-20} \frac{n_s b_s}{U E_c} \ln(c_s U)$$

$n_s$ = number of electrons in a shell
$E_c$ = critical excitation voltage
$b_s$ & $c_s$ = constants related to the electron shell
U = *overvoltage* ($E_o/E_c$)

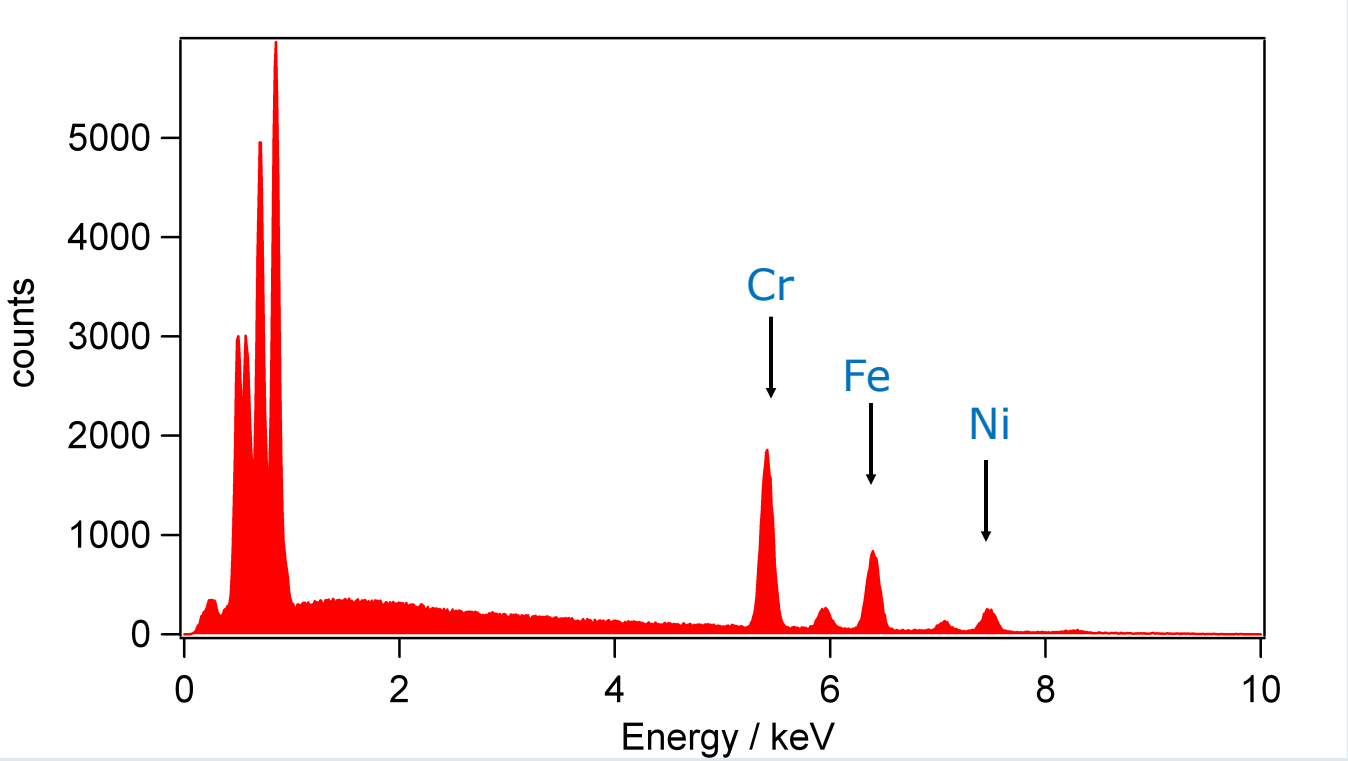



$$U = E_o/E_c$$

**Optimum overvoltage U is around 2-2.5**

Without sufficient overvoltage, x-ray production is dramatically lowered.

# Effects of low overvoltage




Cr: 33%

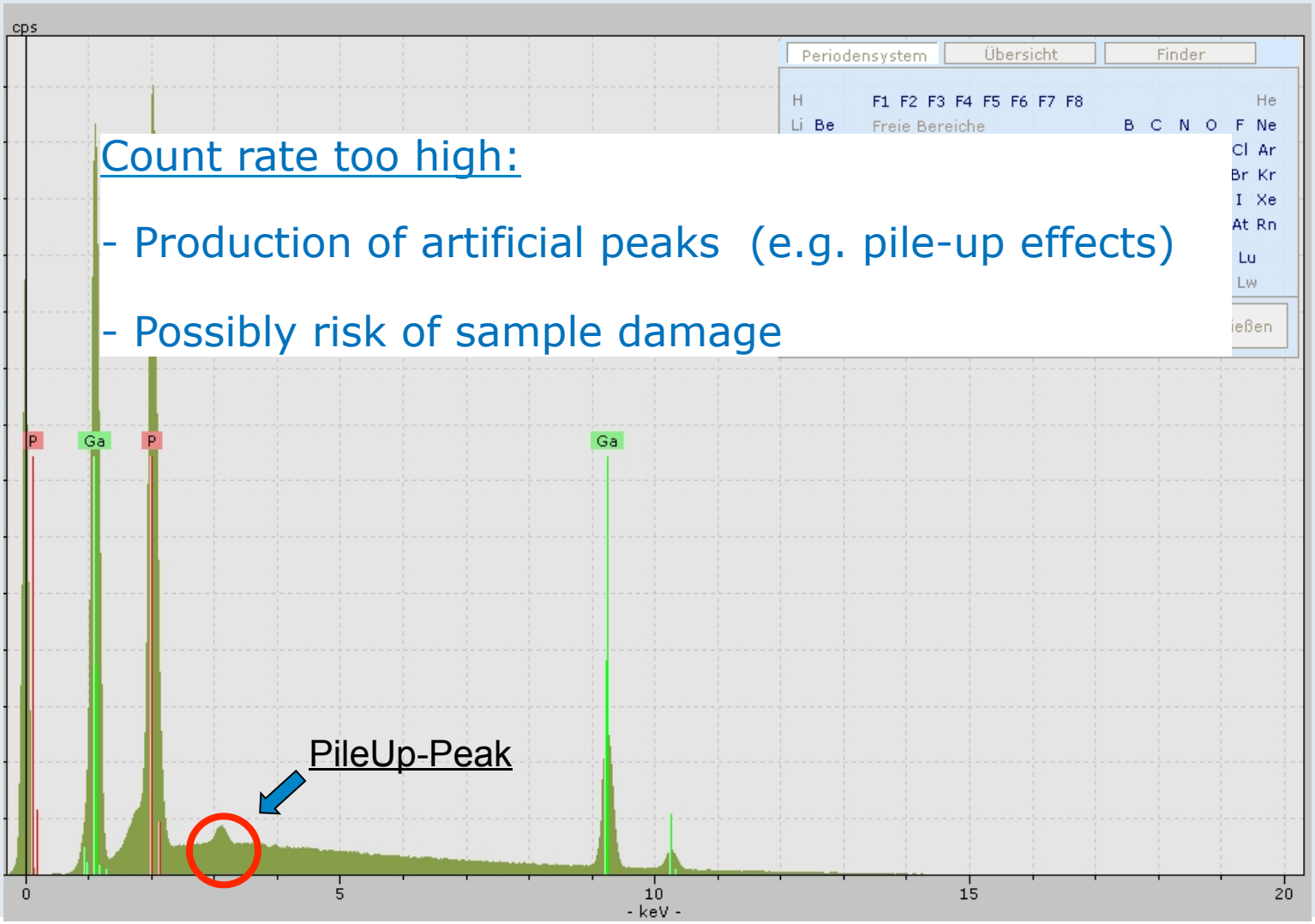
Fe: 33%

Ni: 33%

U = 10 keV

| $\frac{E_{exc}}{E_{bind}}$ : | Cr | Fe | Ni |
|---|---|---|---|
| | 1,847 | 1,561 | 1,337 |

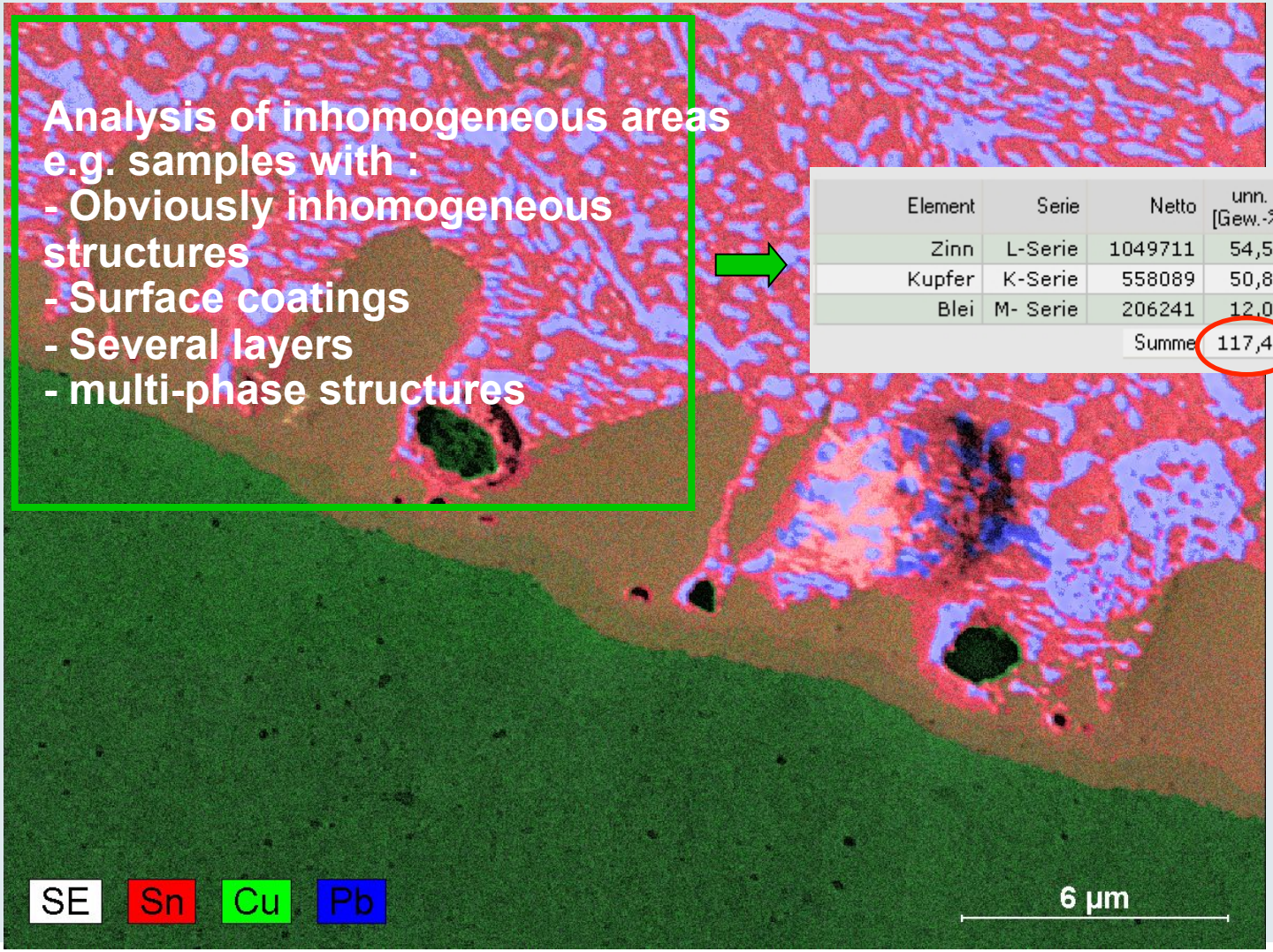
# Selection of the correct count rate for analysis




Count rate too high:

- Production of artificial peaks (e.g. pile-up effects)
- Possibly risk of sample damage

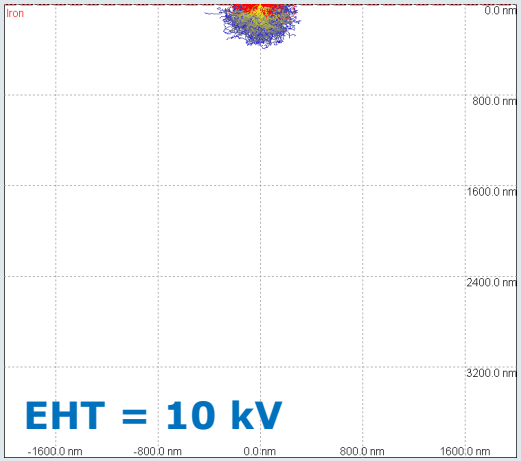
# Sample homogeneity – Quantitative analysis



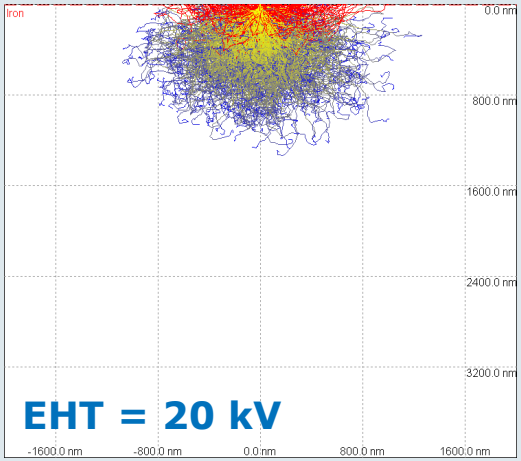

| Element | Serie | Netto | unn. C [Gew.-%] | nor. C [Gew.-%] | Atom-C [at.-%] |
|---|---|---|---|---|---|
| Zinn | L-Serie | 1049711 | 54,51 | 46,41 | 34,84 |
| Kupfer | K-Serie | 558089 | 50,88 | 43,31 | 60,74 |
| Blei | M- Serie | 206241 | 12,07 | 10,28 | 4,42 |
| | | Summe | 117,46 | 100,00 | 100,00 |

# Excitation volume

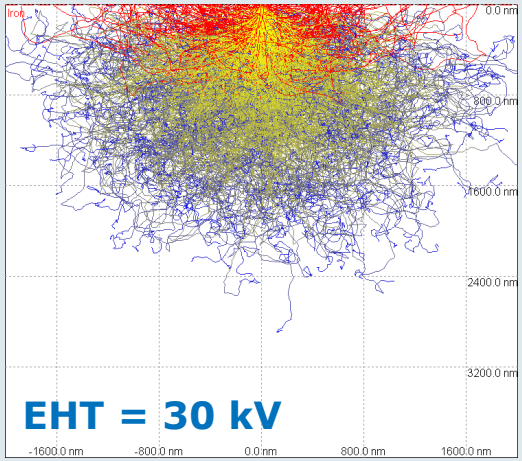
- Monte Carlo electron-trajectory simulations of interaction volume in iron as function of primary beam energy




$R_d \approx 0{,}4$ µm



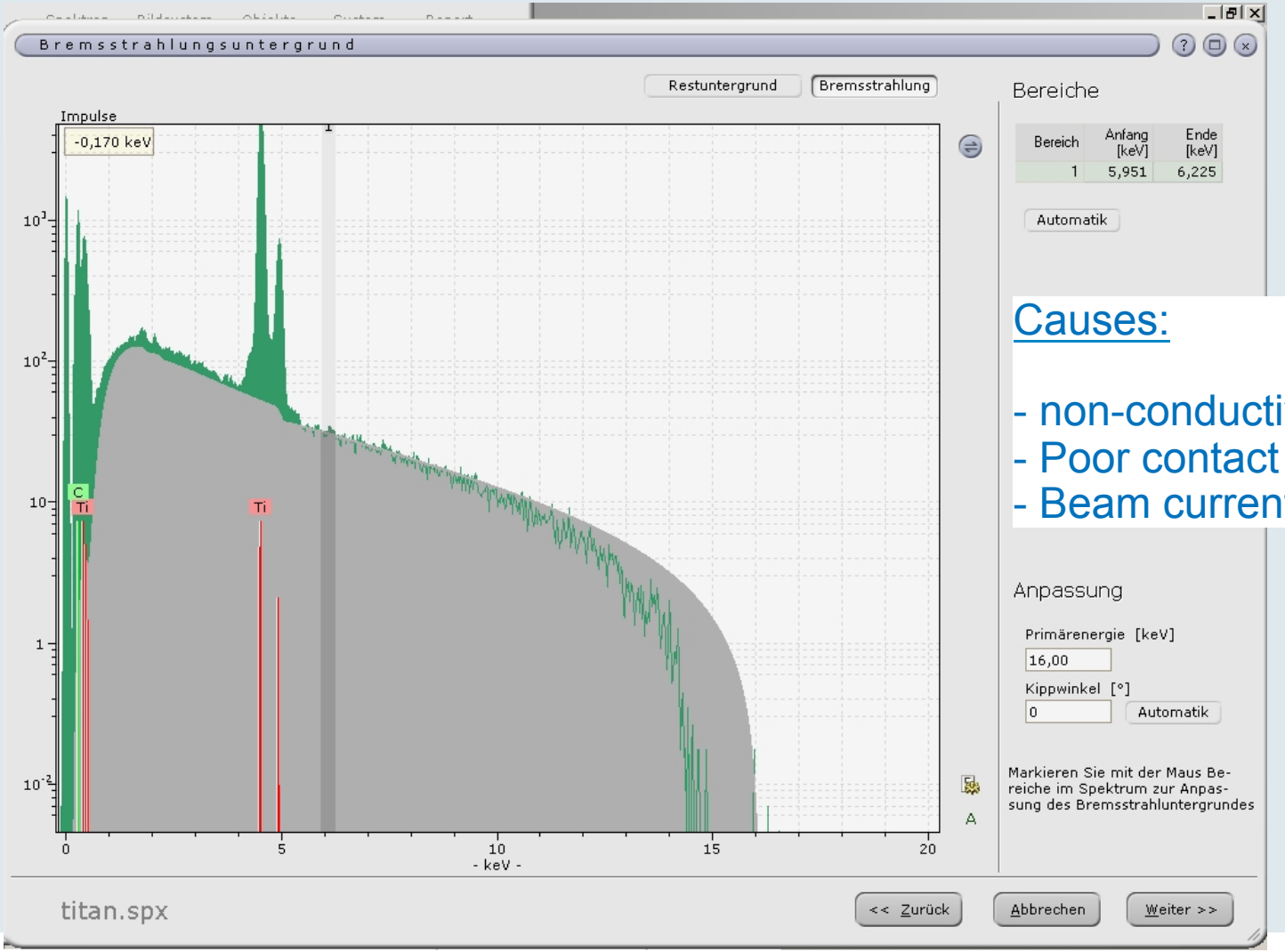

$R_d \approx 1{,}3$ µm




$R_d \approx 2{,}5$ µm

→ With higher primary electron energy penetration depth is increasing

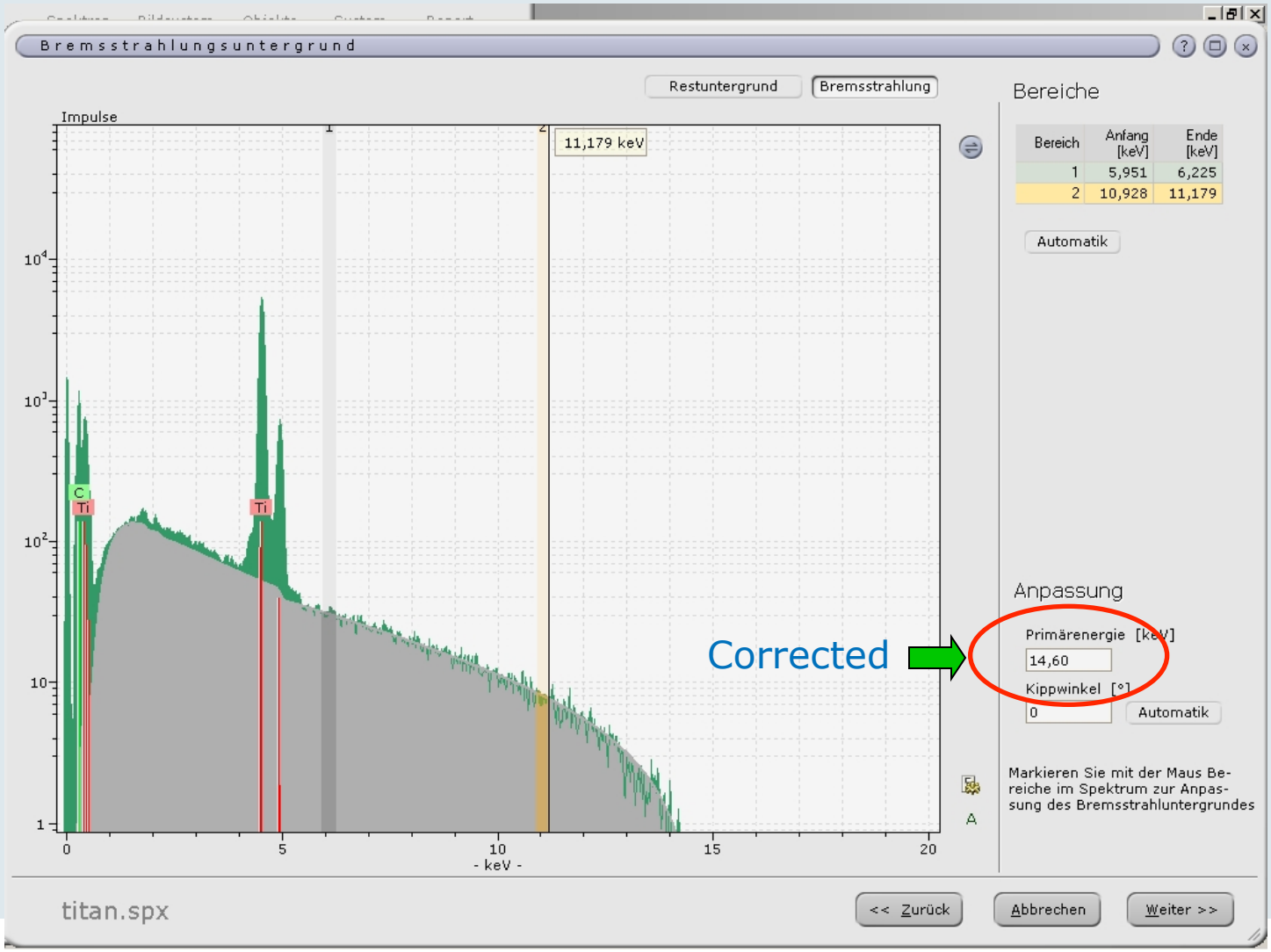
# Sample charging – charged sample



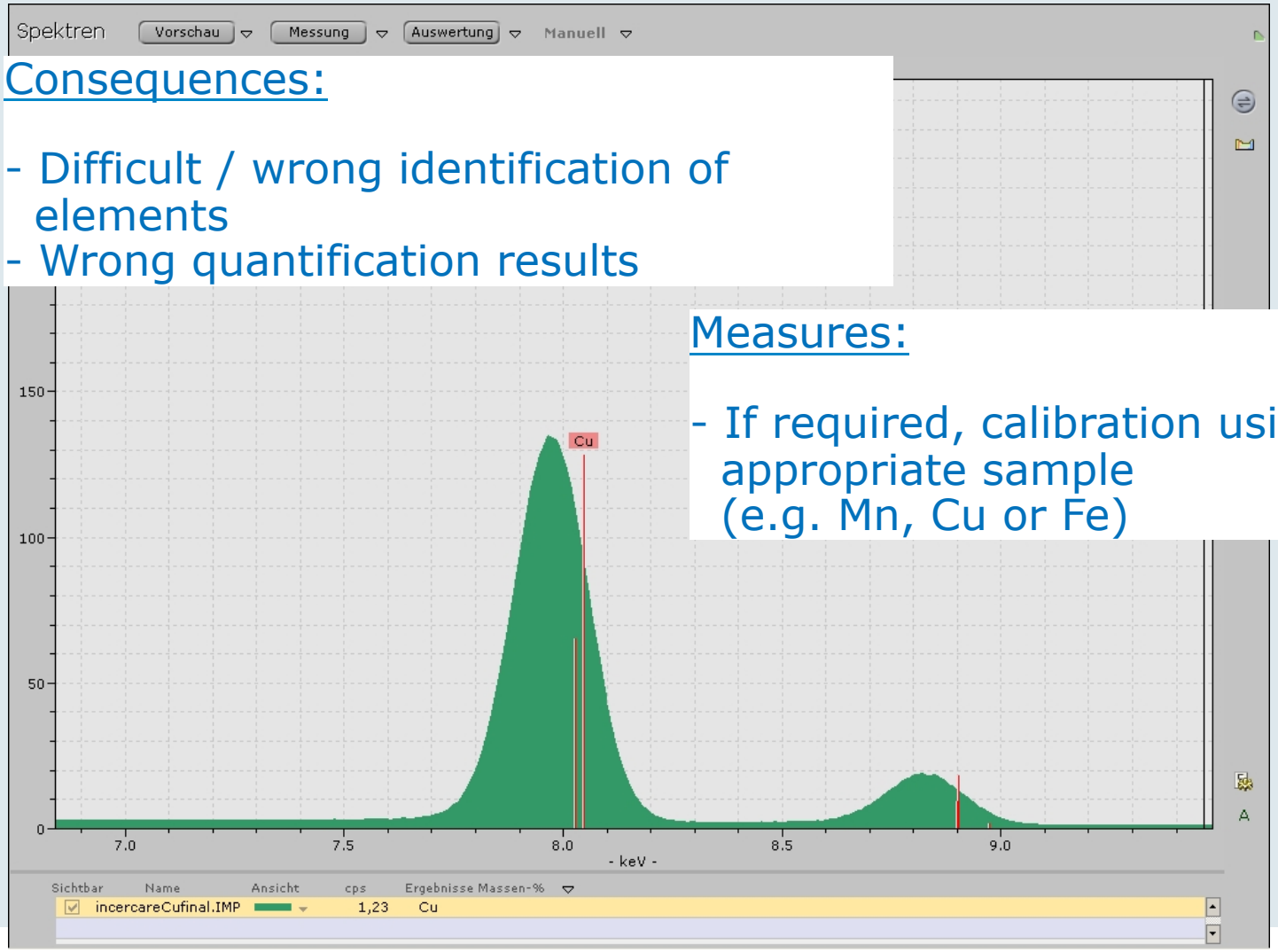

Causes:

- non-conductive sample
- Poor contact
- Beam current too high

# Sample charging – optimized primary energy setting



# Bad energy / channel calibration




Consequences:

- Difficult / wrong identification of elements
- Wrong quantification results

Measures:

- If required, calibration using an appropriate sample (e.g. Mn, Cu or Fe)

# Overview of the influence of different parameters on EDS analysis

| Parameter | Qualitative Analysis | Quantitative Analysis |
|---|---|---|
| wrong working distange / tilt angle | Count rate, absorption | bad background fit |
| inappropriate accelerating voltage | Elements (lines) not excited | too low overvoltage, validitiy of quantification strategies questionable |
| wrong puls throughput / energy range | Spectrum cut off, "C" invisible | degarded energy resolution, less accurate quantification results |
| too high count rate | Pile-up peaks in the spectrum | Pile-up peaks may cause difficulties in deconvolution |
| Sample inhomogeneous | - | Violation of physical conditions (commonly unnormalized concentrations >110%) |
| rough surface | Absorption | bad fit of low to intermediate energy background |
| charging effects | Primary energy | bad fit of the high energy background |
| faulty energy / channel calibration | difficult / wrong identification | bad results |

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