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International Centre for Theoretical Physics

United Nations
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2015-21

Joint ICTP/IAEA Workshop on Advanced Simulation and Modelling for Ion Beam Analysis

23 - 27 February 2009

Pitfalls in ion beam analysis

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Pitfalls in ion beam analysis

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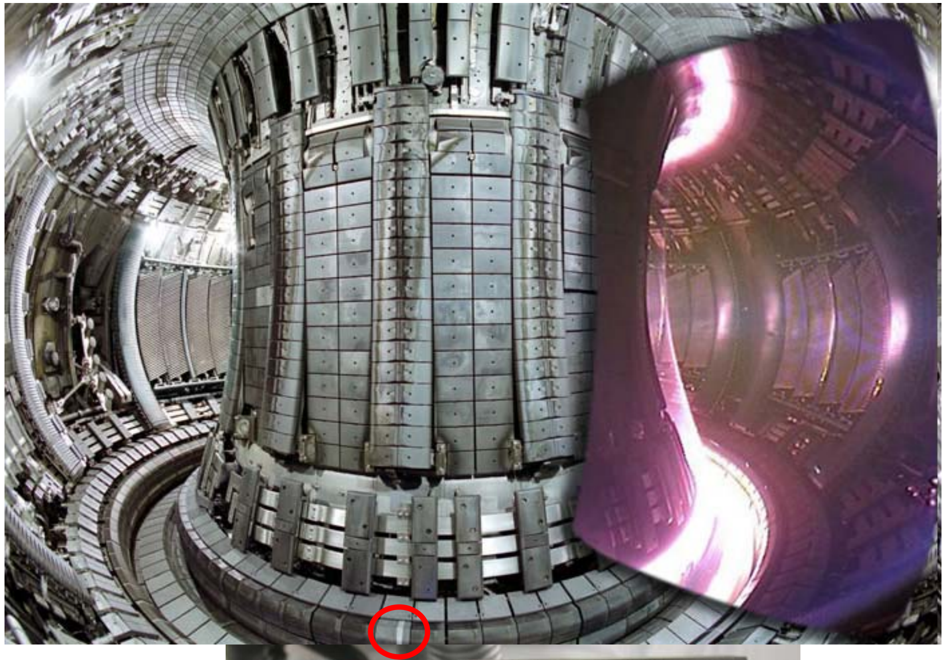
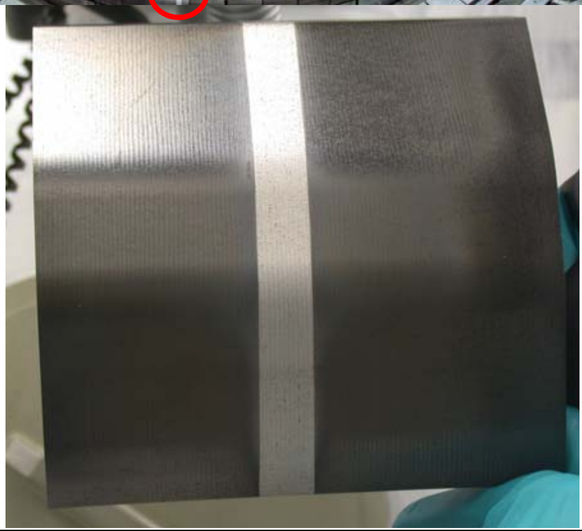
- **Ambiguity of Depth Profile and Roughness**
- **Over-interpretation of Data**
- **Deviations from Rutherford Cross-Section**
- **Electronic detection system effects:**
  Dead time and pulse pile-up

Ambiguity of Depth Profile and Roughness

Ambiguity of depth profile and roughness





Example: Tungsten marker in the divertor of the JET tokamak

- 3 µm W on carbon fibre composite (CFC)
- Exposed to plasmas for 3 years
- Aim:
  - Measurement of W erosion
    - $\Rightarrow$ W-thickness before and after exposure
  - Material mixing of C/W
  - Preparatory experiment for full-W divertor

Ambiguity of depth profile and roughness (2)

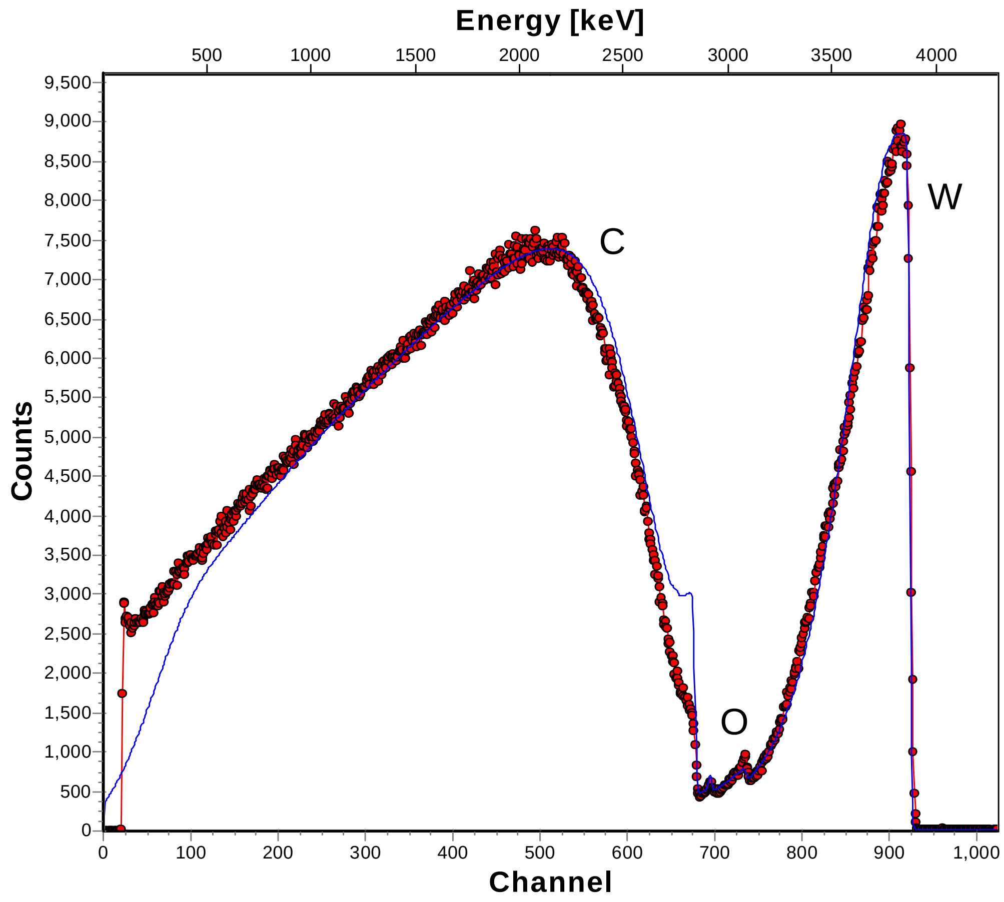
RBS spectrum can be fitted with
4 µm $W_{0.3}C_{0.7}$ layer on C + roughness

⇒ **Interpretation:** Carbide formation due to diffusion + high temperature

⇒ **Conclusion:** Diffusion barrier necessary between W and C

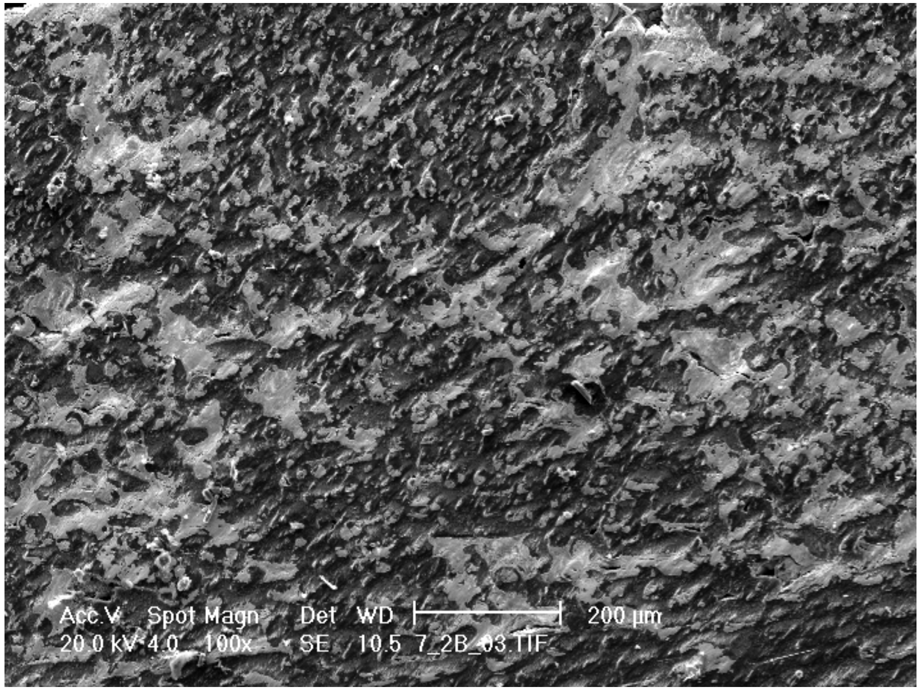
⇒ **Implication:** Additional costs of a few M€, technologically demanding

BUT: Is this interpretation correct?

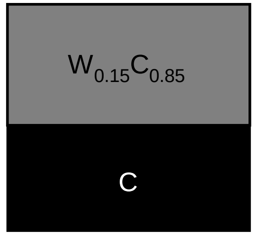


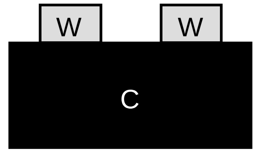
W-layer after exposure
RBS, 165°, 4 MeV protons

Ambiguity of depth profile and roughness (3)



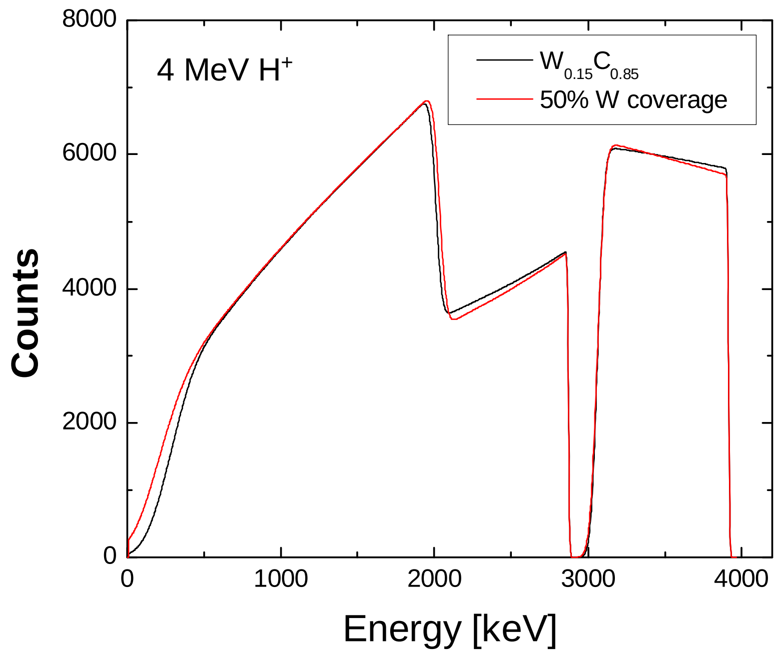
SEM of W-layer after exposure





100% coverage with $W_{0.15}C_{0.85}$,
thickness 13.1×10^{19} W-at./cm^2
$\Rightarrow$Total: 2×10^{19} W-at./cm^2

50% W-coverage,
thickness 4×10^{19} W-at./cm^2
$\Rightarrow$Total: 2×10^{19} W-at./cm^2



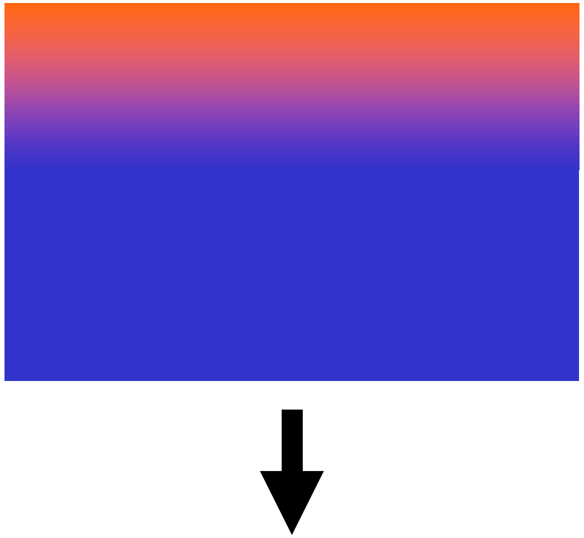
Laterally inhomogeneous samples
and depth distributions may give identical spectra

$\Rightarrow$ IBA methods alone may be not sufficient for determining sample structure

Ambiguity of depth profile and roughness (4)

Laterally homogeneous

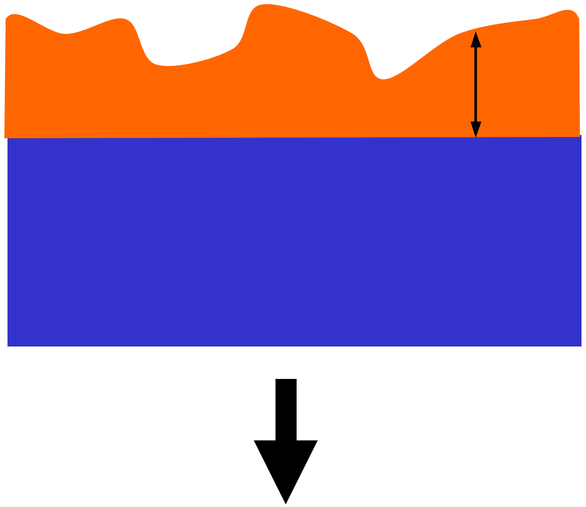
composition varying with depth



IBA methods provide
depth profile of elements

Laterally inhomogeneous,
roughness

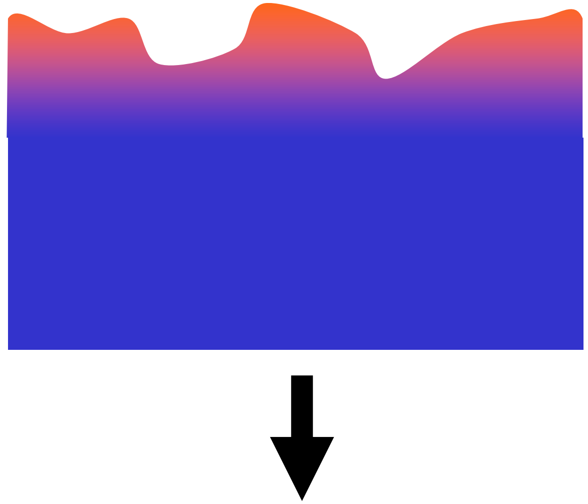
homogeneous composition



IBA methods provide
roughness distribution

Laterally inhomogeneous,
roughness

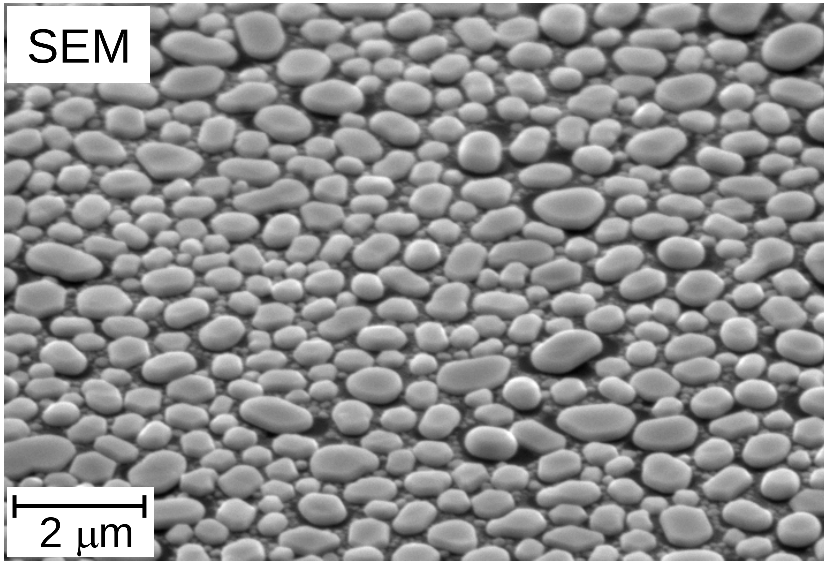
composition varying with depth

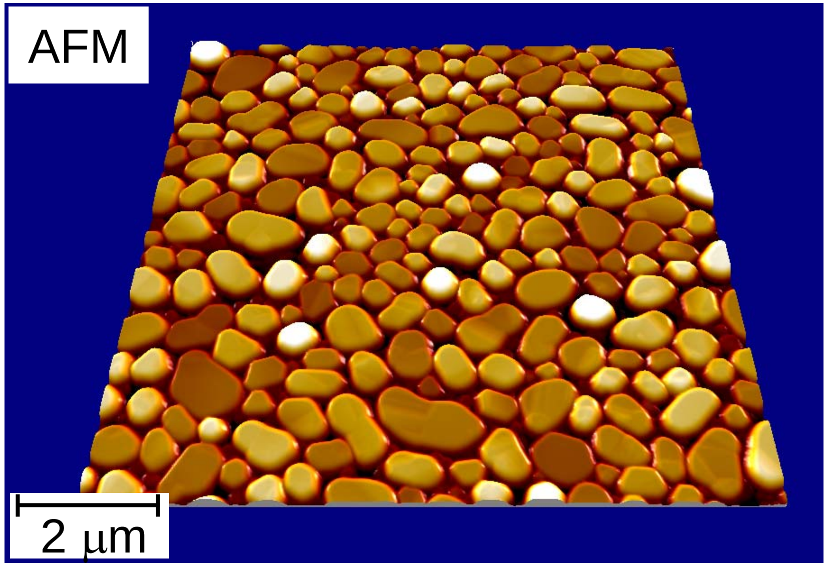


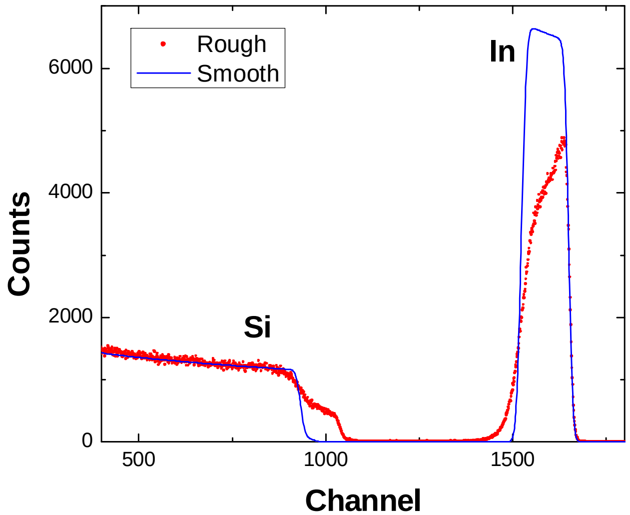
IBA methods provide
total amounts of elements
(within some error bar)

Depth profiling is demanding,
if possible at all

Measuring surface roughness with IBA





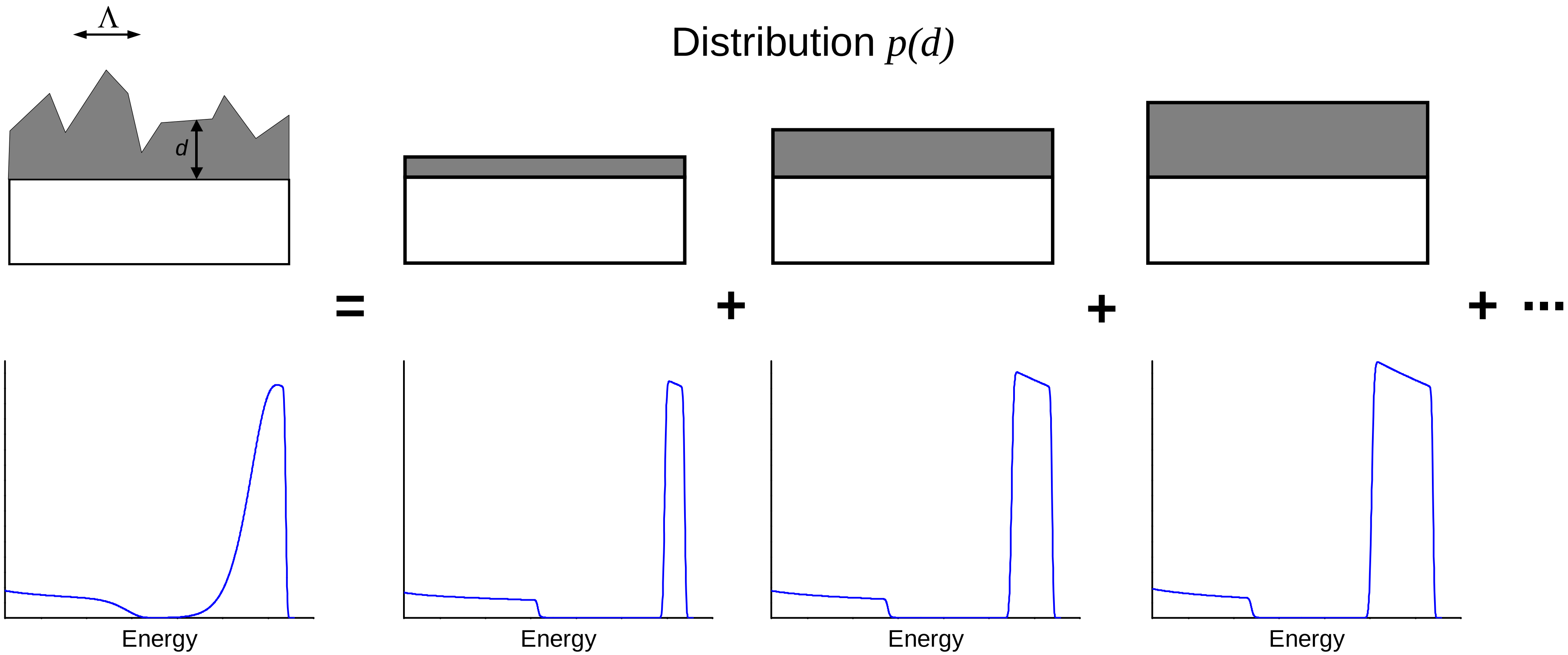


RBS

2 MeV ^{4}He, 165°

200 nm In on Si

Measuring surface roughness with IBA (2)



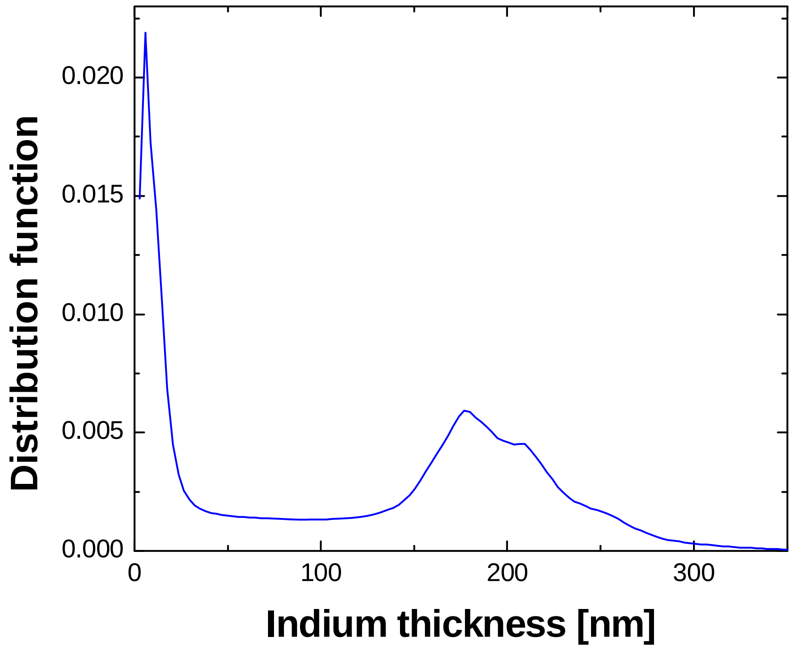
M. Mayer, NIM **B194** (2002) 177

Correlation effects are neglected

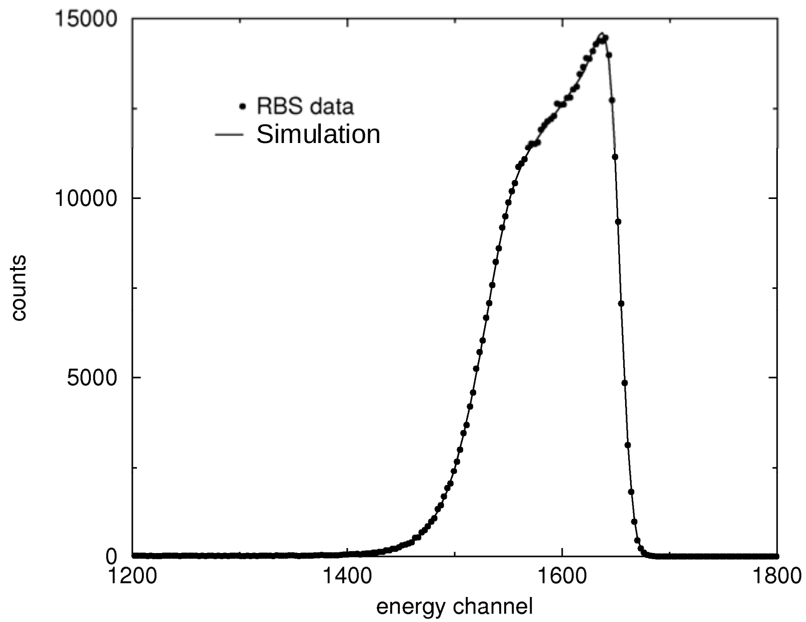
⇒ valid, if lateral variation Λ > d for typical RBS angles of 160°-170°

Measuring surface roughness with IBA (3)

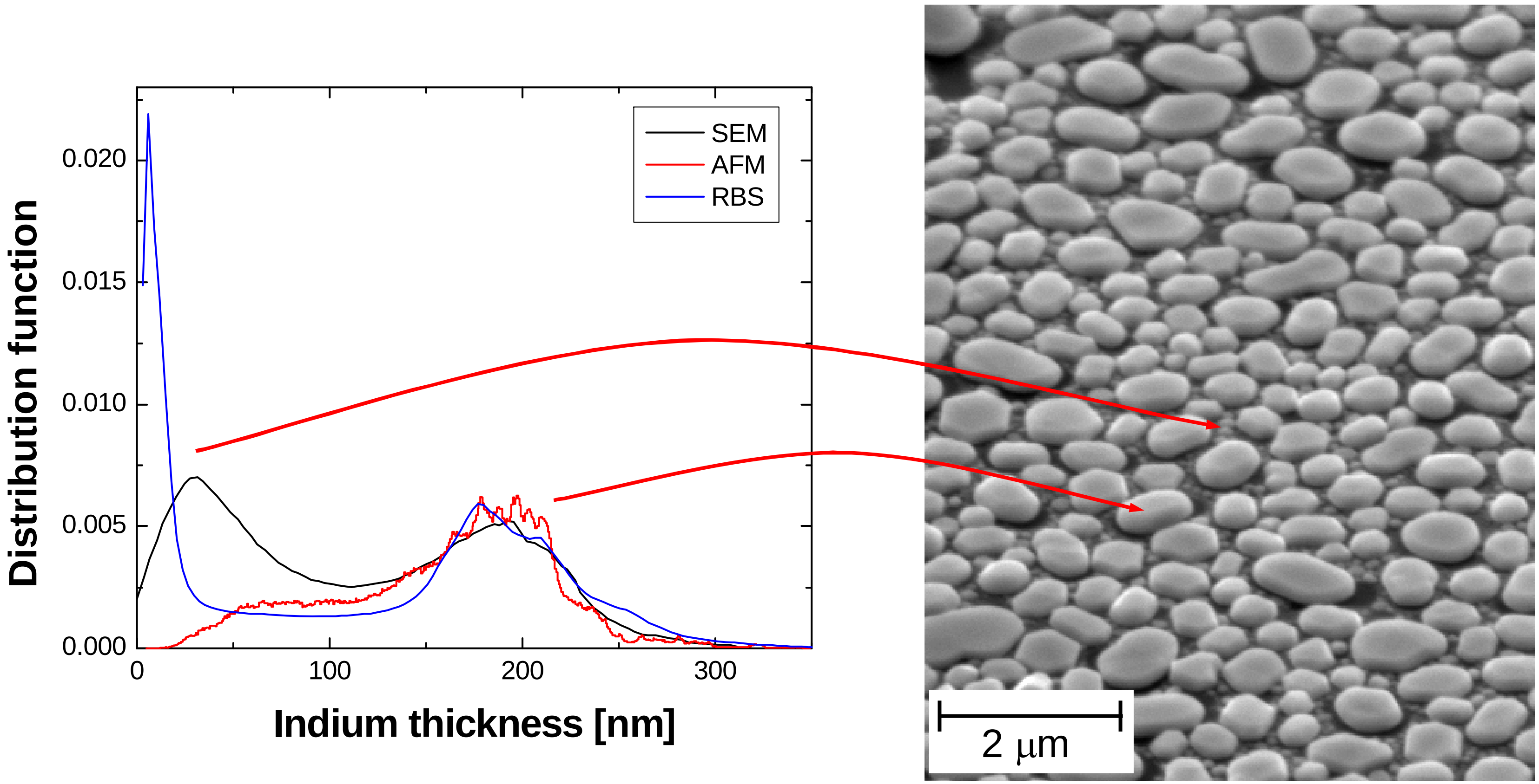
Film thickness distribution



RBS spectrum



Measuring surface roughness with IBA (4)



- Good agreement for large blobs (around 200 nm)
- Small blobs are only visible with RBS and SEM, but not AFM

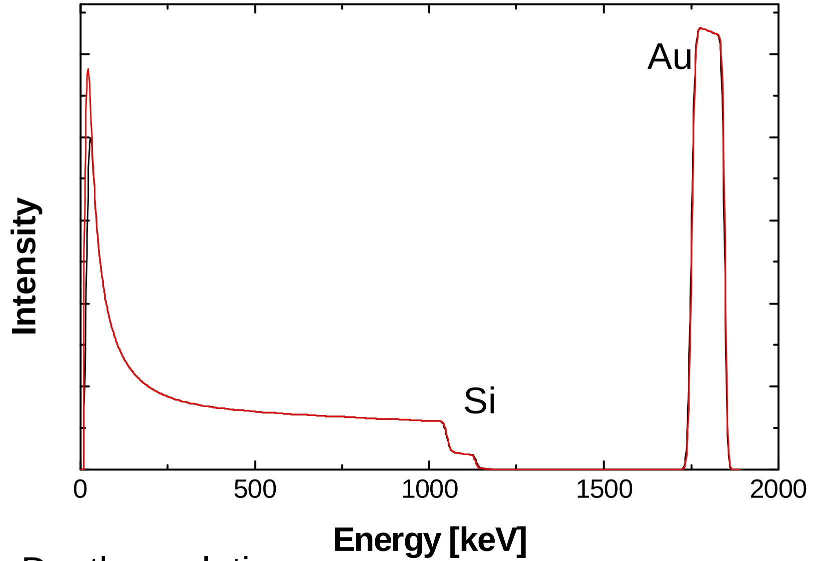
M. Mayer et al., NIM B 228 (2005) 349

Over-interpretation of Data

Ambiguity of too small structures

Example:

RBS from (imaginery) AuSi on Si
2 MeV ^{4}He

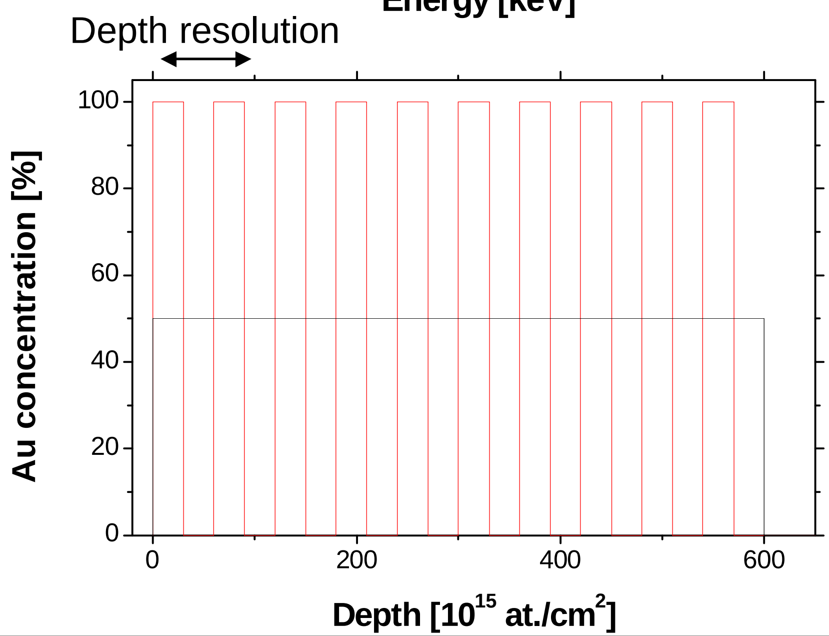


- Only structures larger than depth resolution are meaningful
- Depth profile is ambiguous for structures smaller than depth resolution

⇒ **Never use layers with thicknesses smaller than the depth resolution**

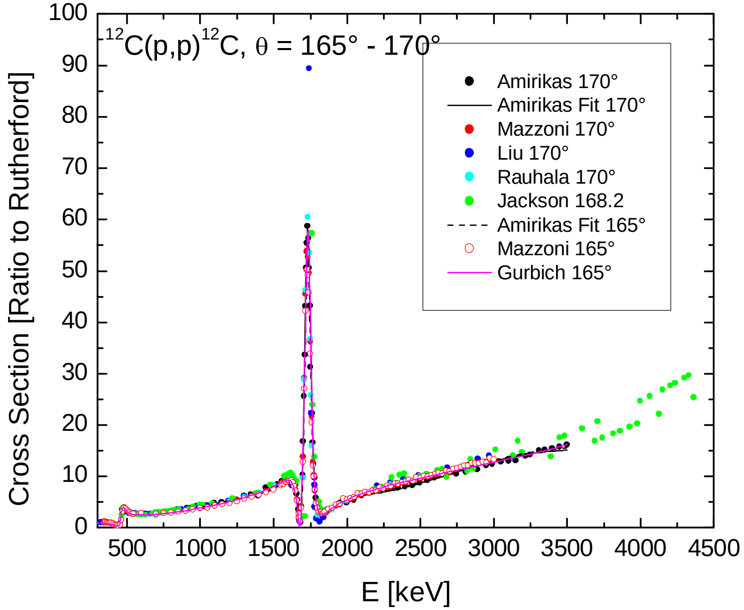
⇒ **Never interpret structures which are smaller than the depth resolution**

⇒ **Occam's razor: The depth profile with the smallest number of assumptions should be used**



Deviations from Rutherford Cross-Section

Deviations from Rutherford Cross Section



Deviations from Rutherford may be huge

⇒ Carefully check for
possible deviations from Rutherford

Energy at which the cross section deviates by > 4% from Rutherford at $160° \leq \theta \leq 180°$
Bozoian (1991)

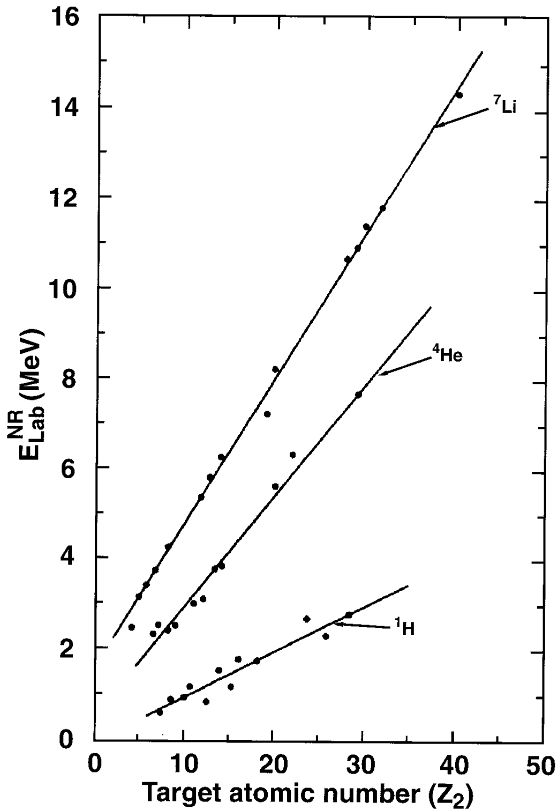
^{1}H: $E^{NR}_{Lab} = 0.12\ Z_2 - 0.5$ [MeV]

^{4}He: $E^{NR}_{Lab} = 0.25\ Z_2 + 0.4$ [MeV]

Linear Fit to experimental values (^{1}H, ^{4}He) or optical model calculations (^{7}Li)

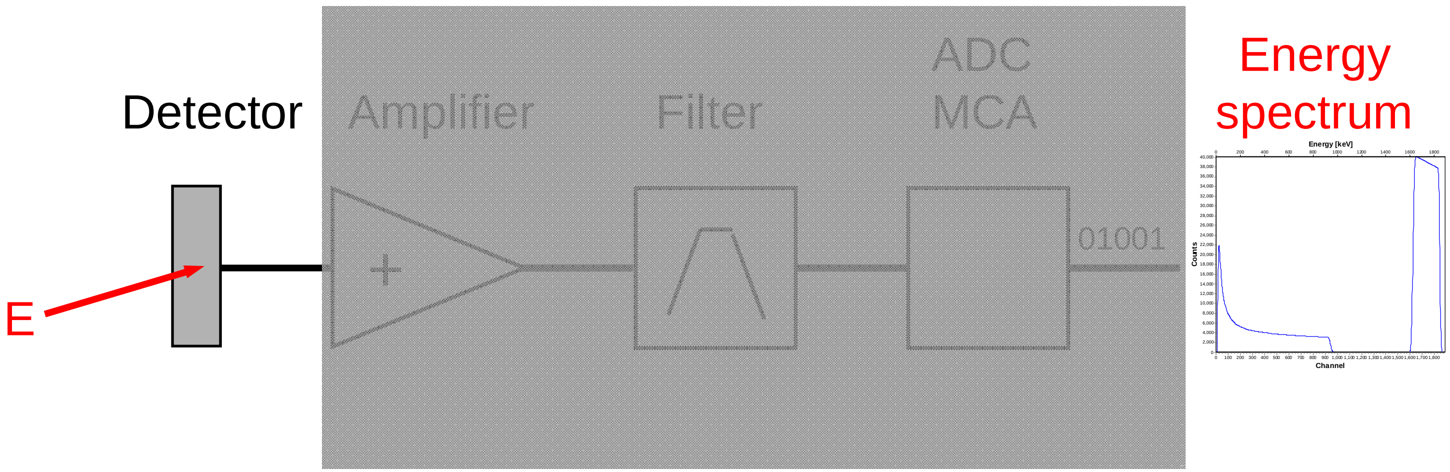
Accurate within ± 0.5 MeV

More accurate: Measured cross-section data

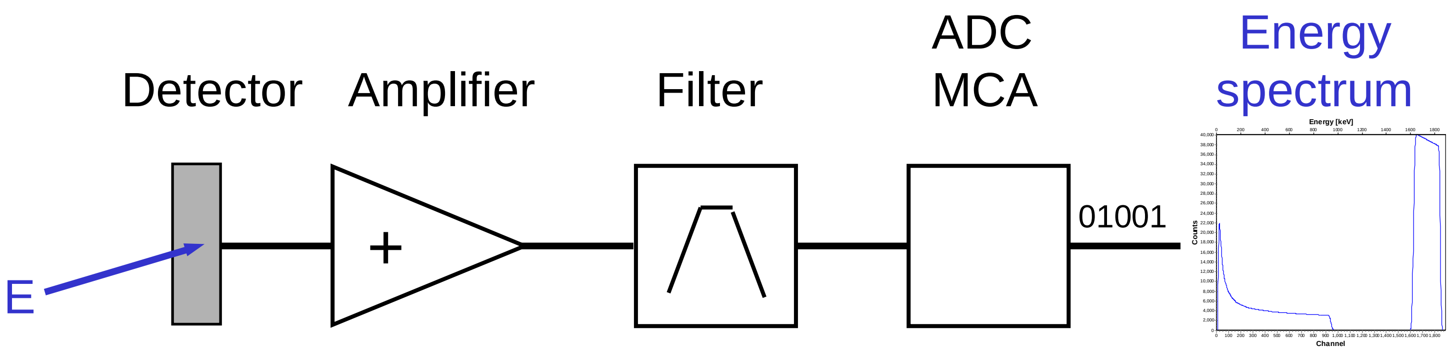


Electronic detection system effects: Dead time and pulse pile-up

Electronic detection system effects



Electronic detection system effects (2)



- Most people assume, that measured spectrum = incident spectrum

  ⇒ **This is incorrect!**

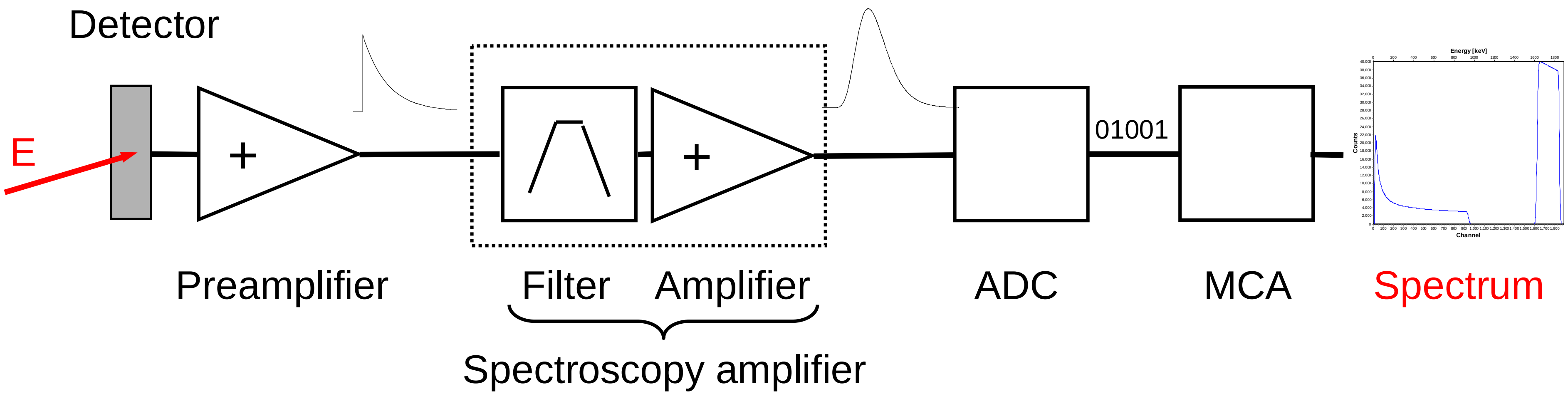
- Electronic detection system adds unavoidable distortions

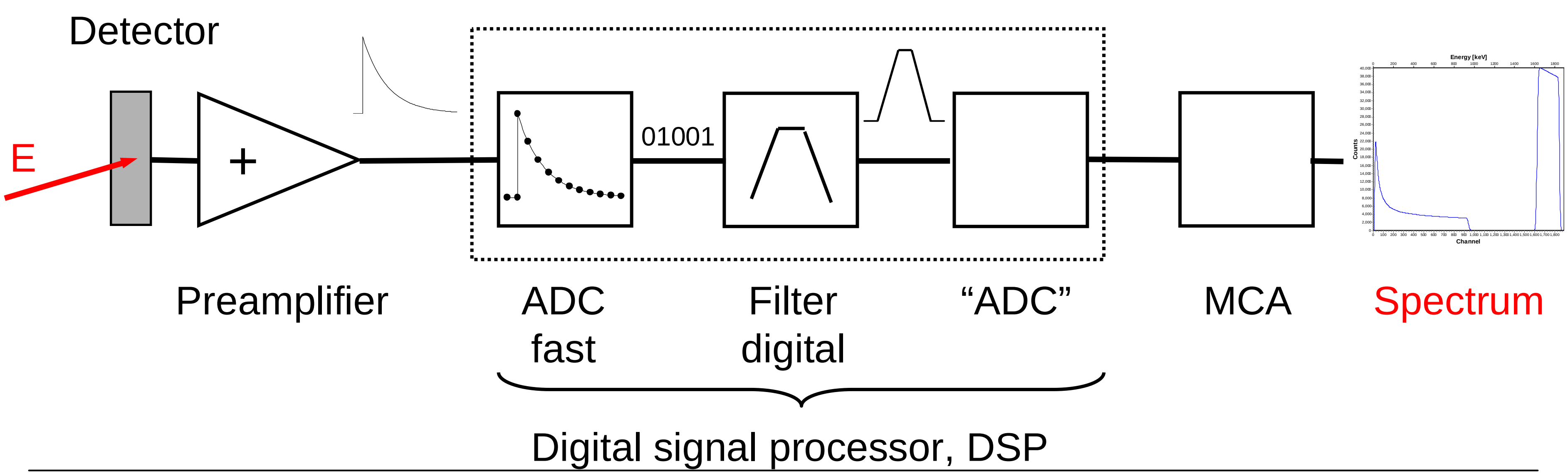
  – finite ADC conversion time
  ⇒ **Dead time losses** of incident pulses

  – finite electronic pulse widths
  ⇒ **Pulse pile-up**

- Distortions can be minimised at **low incident pulse rates**, but never completely avoided

Spectroscopy systems





Dead time losses

- ADC needs a certain time to digitise an analogue pulse

  ⇒ Paralysation of the system

- Additional pulses cannot be accepted during this time

  ⇒ Rejection of pulses

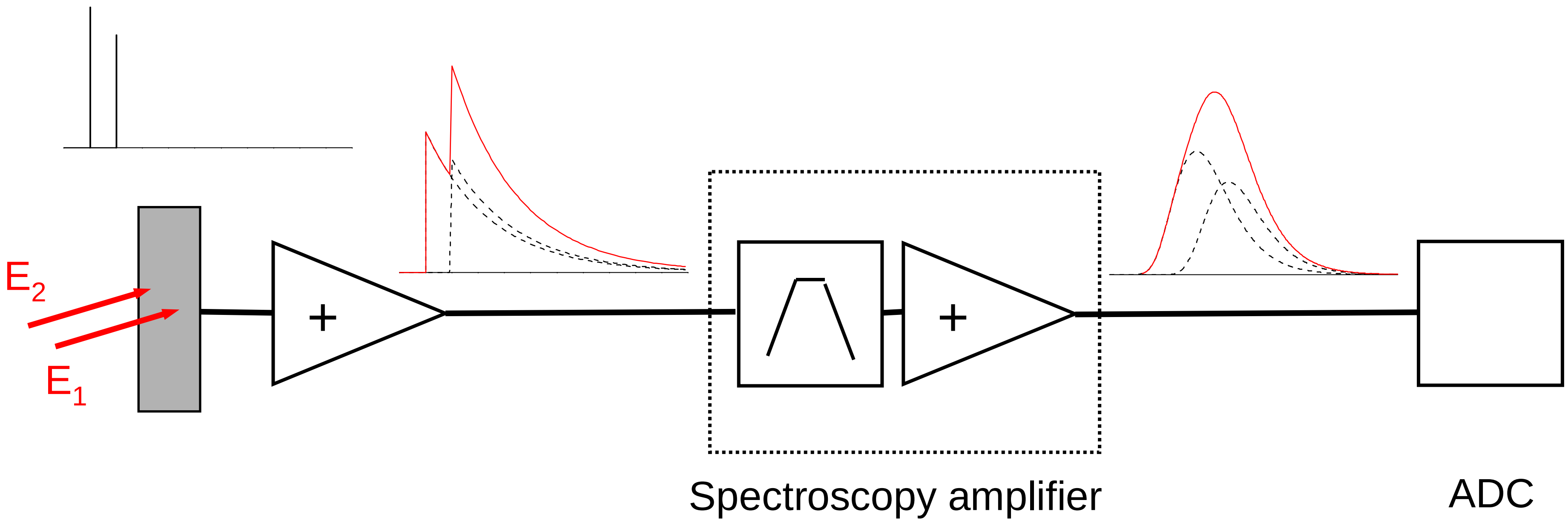
  ⇒ **Dead time losses**

- Pile-up rejector is additional source of dead time due to rejection of pulses

- Dead time losses are taken into account through

$$P_{Live} = \frac{T_{Live}}{T_{\mathrm{Re}al}}$$

Pulse pile-up

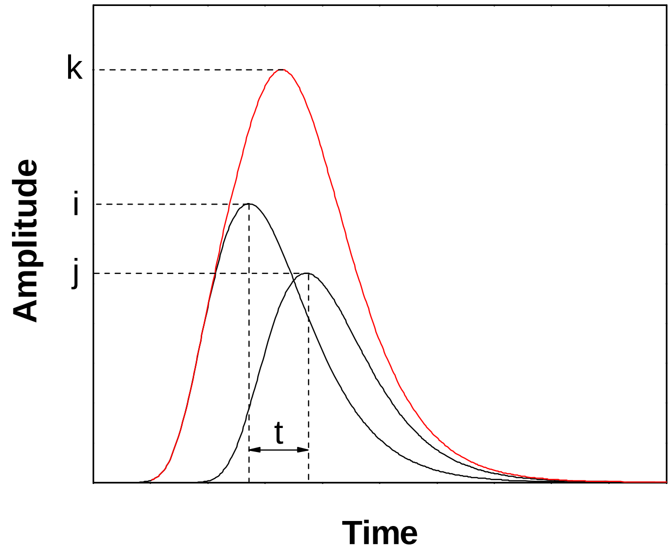


Arrival of 2 pulses within amplifier pulse width

$\Rightarrow$ Overlap of pulses

$\Rightarrow$ **Pulse pile-up**

Pulse pile-up (2)



- Loss of 2 pulses i, j
- gain of 1 erroneous pulse k
- P_{ijk}: Probability of pulses i, j to pile up to k

$$\begin{aligned} n_k^{PU} &= n_k - L_k + G_k \\ &= n_k - N P_k \sum_{j=1}^{m} P_j \sum_{i=a}^{j+k} (P_{kji} + P_{jki}) + N \sum_{i=1}^{k-1} \sum_{j=p}^{k-1} P_i \, P_j \, P_{ijk}, \end{aligned}$$

$$P_i = \frac{n_i}{N}$$

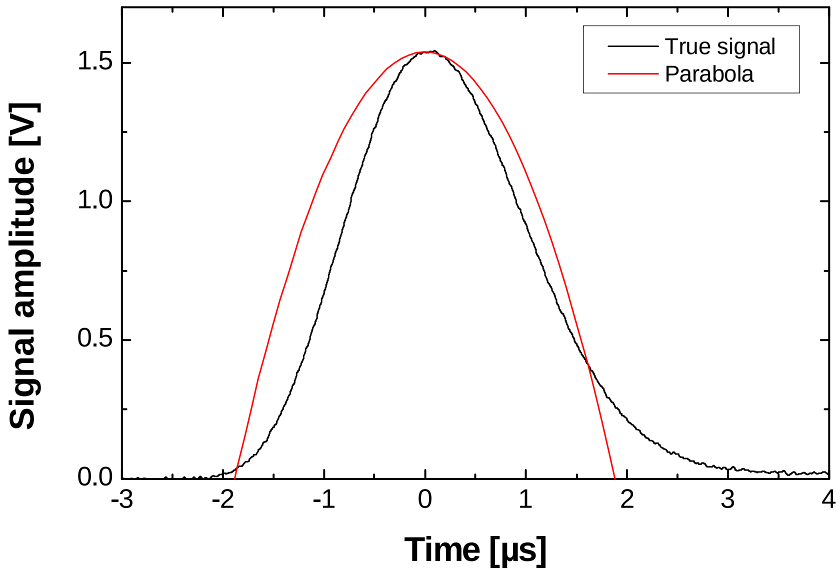
Probability of pulse i

Wielopolski and Gardner, 1976

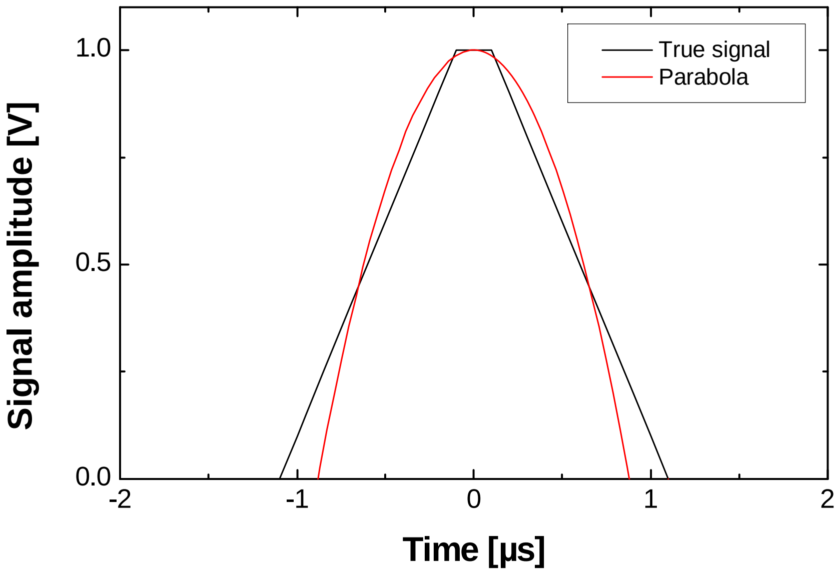
P_{ijk} can be obtained from

- Poisson arrival time distribution
- Approximation of pulse shape with parabola Wielopolski and Gardner, 1976 or numerical use of precise pulse shapes
- Double pulse pile-up (pile-up of >2 pulses neglected)

Spectroscopy amplifier (Ortec 672)

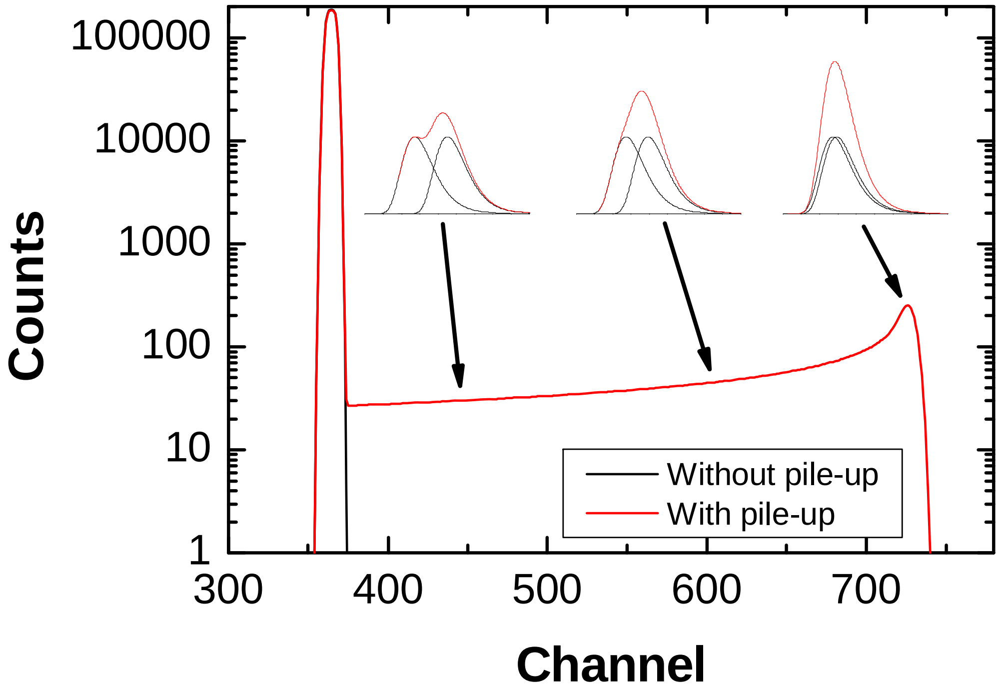


Digital signal processor



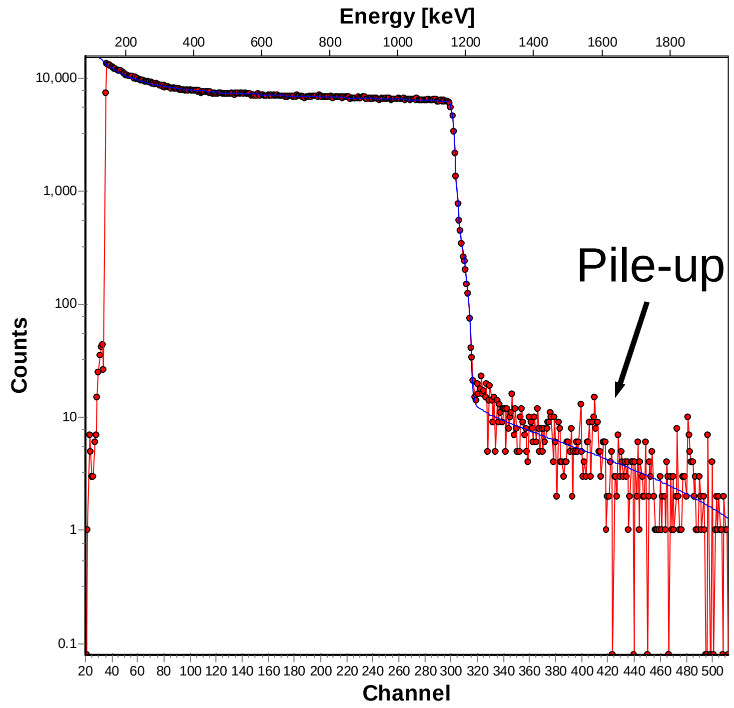
Pulse pile-up (4)

Calculated pile-up from a single peak



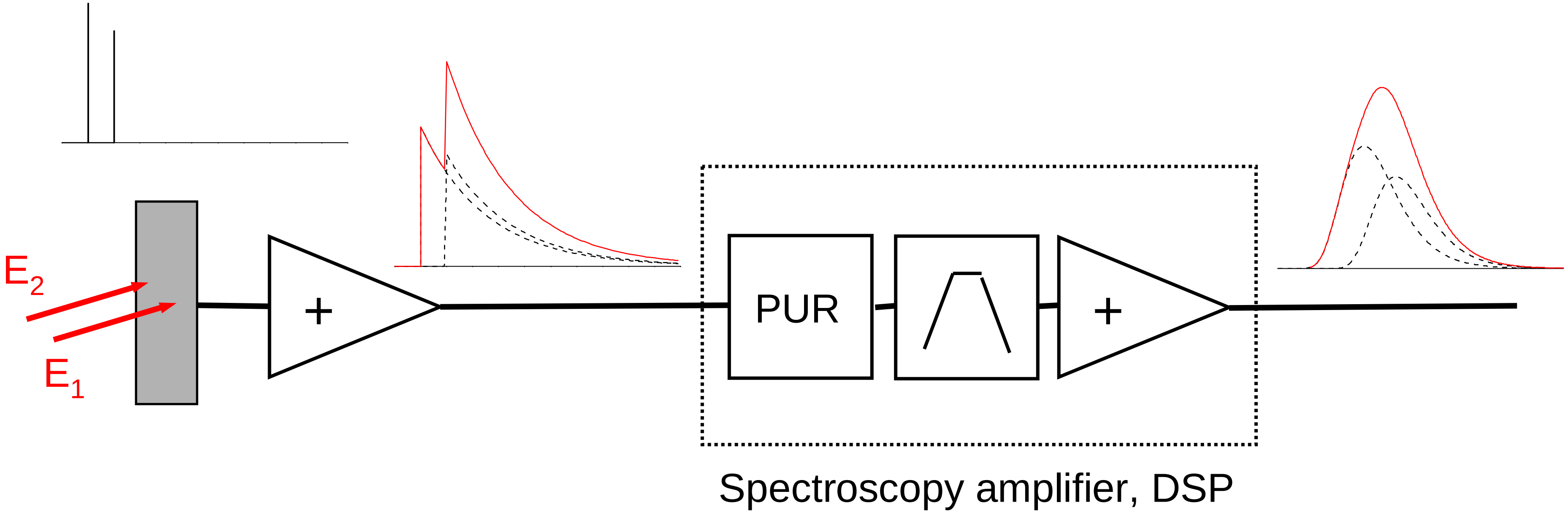
RBS, 2 MeV ^{4}He → Si

Amplifier, 0.5 µs shaping (M. Bianconi)



- Additional peaks at combined energy (PIXE, …)
- Additional intensity at higher energies

Pile-up rejector



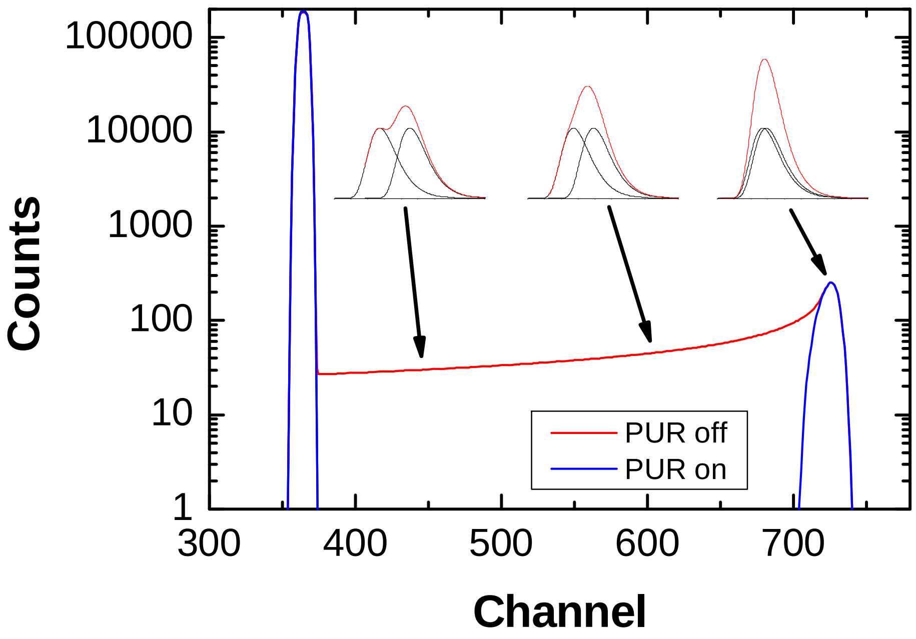
A pile-up rejector (PUR) rejects pulses only if t > pair resolution time

- pair resolution time 300 - 500 ns

  (specification of Canberra DSP 9660, Ortec Amplifier 672: < 500 ns)

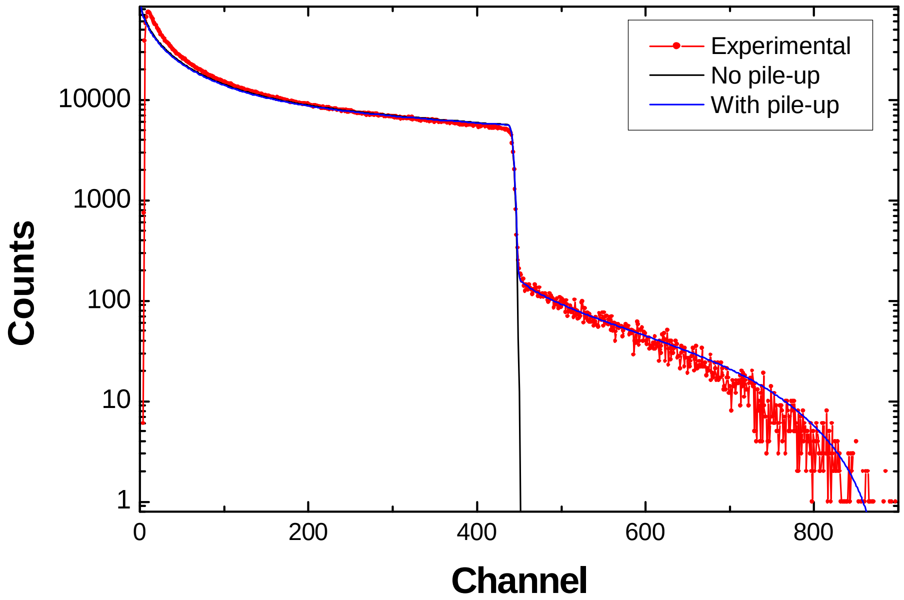
Pile-up rejector (2)

Calculated pile-up from a single peak



RBS, 2 MeV ^{4}He → Au

Canberra DSP 9660, PUR on



A pile-up rejector alleviates the problem of pile-up, but does not fully eliminate it

Summary

- **Ambiguity between depth profile and roughness**
  ⇒ Depth profiling only possible for laterally homogeneous samples
  ⇒ IBA methods measure roughness for laterally inhomogeneous samples
  ⇒ Lateral homogeneity has to be proven with other methods than IBA
- **Depth profile is ambiguous for structures smaller than depth resolution**
  ⇒ Only structures larger than depth resolution are meaningful
- **Deviations from Rutherford cross-section occur at higher energies**
  ⇒ Carefully check for possible deviations
- **Electronic data acquisition system adds dead-time and pulse pile-up**
  ⇒ Use low incident pulse rates + accurate simulation of pile-up