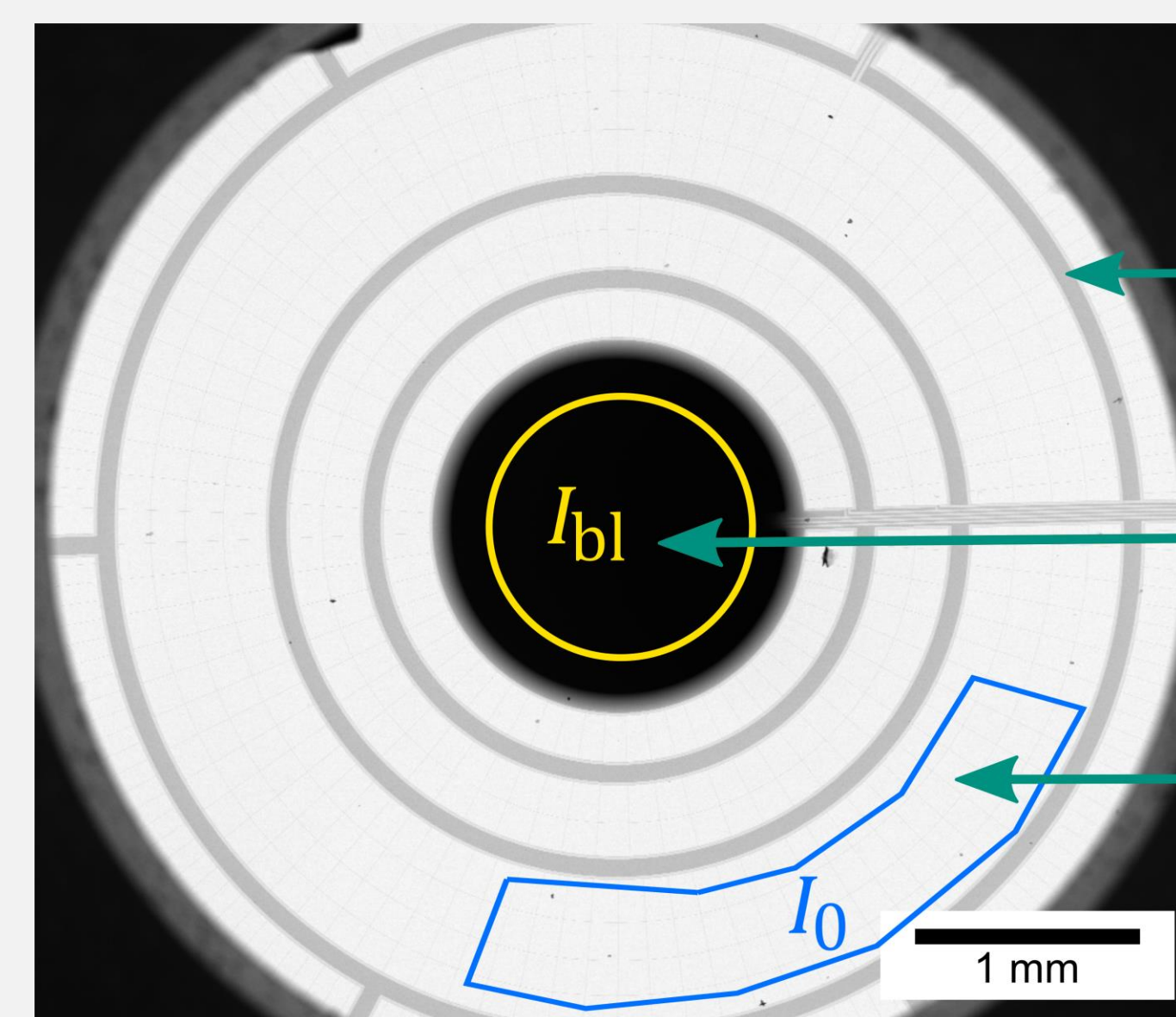


## Thickness Determination: Overview

- Available techniques: e.g. EELS/EFTEM [Zha], CBED [Wil], thickness contours [Wil]
- Drawbacks: limited accuracy, time consumption, limited to small sample regions, energy filter or crystalline sample needed
- Alternative solution: thickness determination using STEM-in-SEM combined with Monte-Carlo simulations [Vol, Čal]
- Advantages: high precision (uncertainty of thicknesses ~7%), no further costly equipment is needed, fast, covers large regions
- Requirements: STEM detector in scanning electron microscope, knowledge of composition and material density of the specimen

## Step 1: Determination of Detector-Specific Properties

- Inner and outer radii of the active segments → investigated scattering-angle range
- Images taken with HAADF STEM; BF STEM yields less reliable results
- Contrast and brightness settings to avoid under- and oversaturation
- Measurement of the intensity in active and inactive detector areas ( $I_0$ ,  $I_{bl}$ )



less sensitive area (e.g. contact lines) = 3% of the total area → geometrical correction factor  $c=0.97$

inactive area ~ black level intensity ( $I_{bl}$ )

active area ~ intensity of the primary electron beam ( $I_0$ )

Image of the STEM detector obtained by scanning the detector, adapted from [Hug]

## Step 2: Calculation of the Normalized HAADF-STEM Intensity

- $I_{exp}$  = measured HAADF-STEM intensity of a sample region
- Normalization of  $I_{exp}$  with respect to  $I_0$  and  $I_{bl}$

$$I_{nor} = \frac{I_{exp} - I_{bl}}{c \cdot I_0 - I_{bl}} \quad (0 \leq I_{nor} \leq 1)$$

- Next step: comparison of normalized image intensities with data obtained by Monte-Carlo simulations

## Summary

- ✓ Determination of local sample thickness by STEM-in-SEM
- ✓ 3-step workflow:
  - Determination of detector-specific properties
  - Normalization of measured HAADF-STEM intensities
  - Comparison with detector-specific Monte-Carlo simulations

[Čal] Čalkovský et al., Ultramicroscopy 207, 112843 (2019)

[Hug] Hugenschmidt et al., J. Microsc. 274, 150 (2019)

[Rit] Ritchie, Surf. Interface Anal., 37, 1006 (2005)

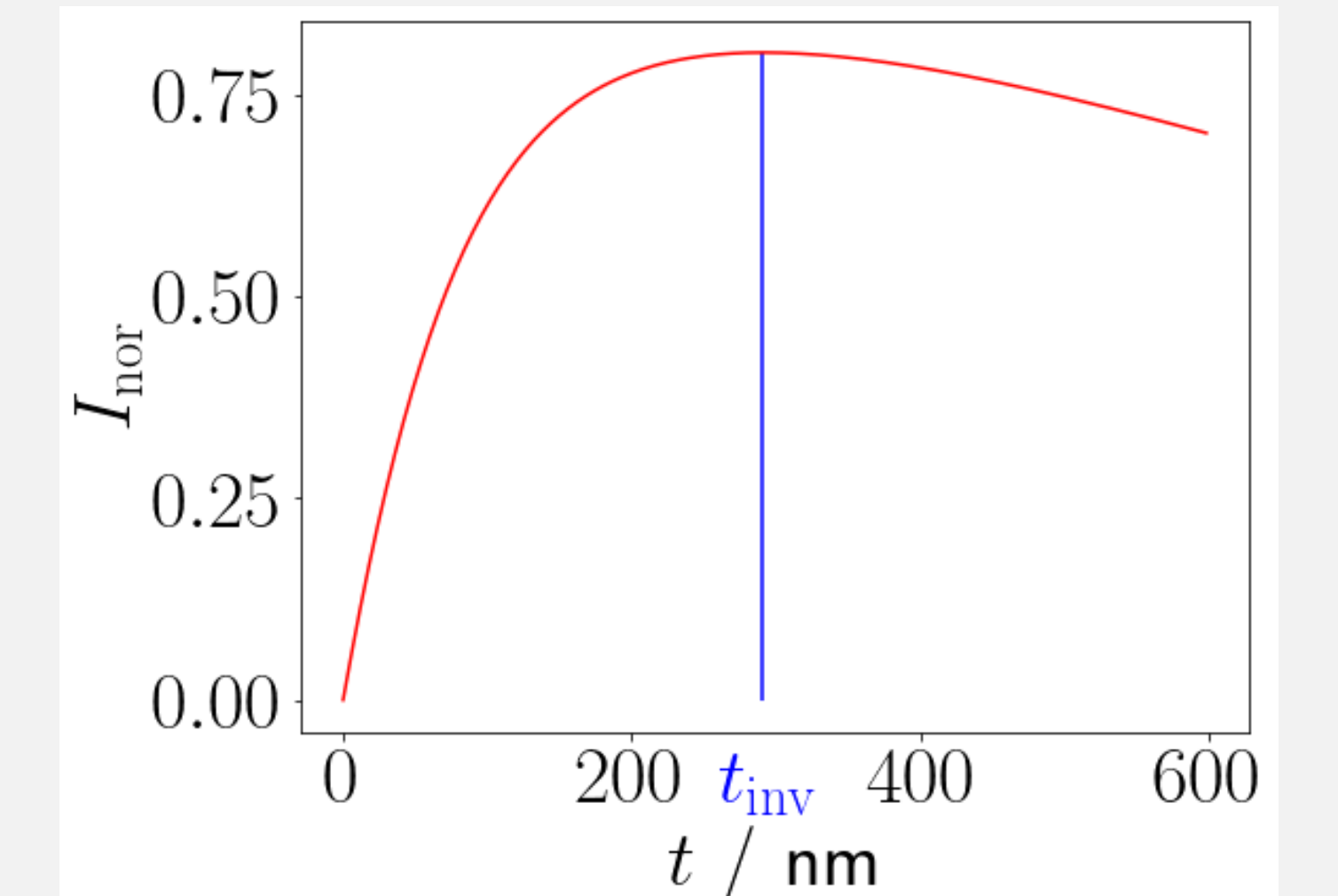
[Vol] Volkenandt et al., Microsc. Microanal. 16, 604 (2010)

[Wil] Williams, Carter, Transmission electron microscopy: A textbook for materials science. 2nd ed. New York: Springer, 2008

[Zha] Zhang et al., Micron 43, 8 (2012)

## Step 3: Comparison with Monte-Carlo Simulations

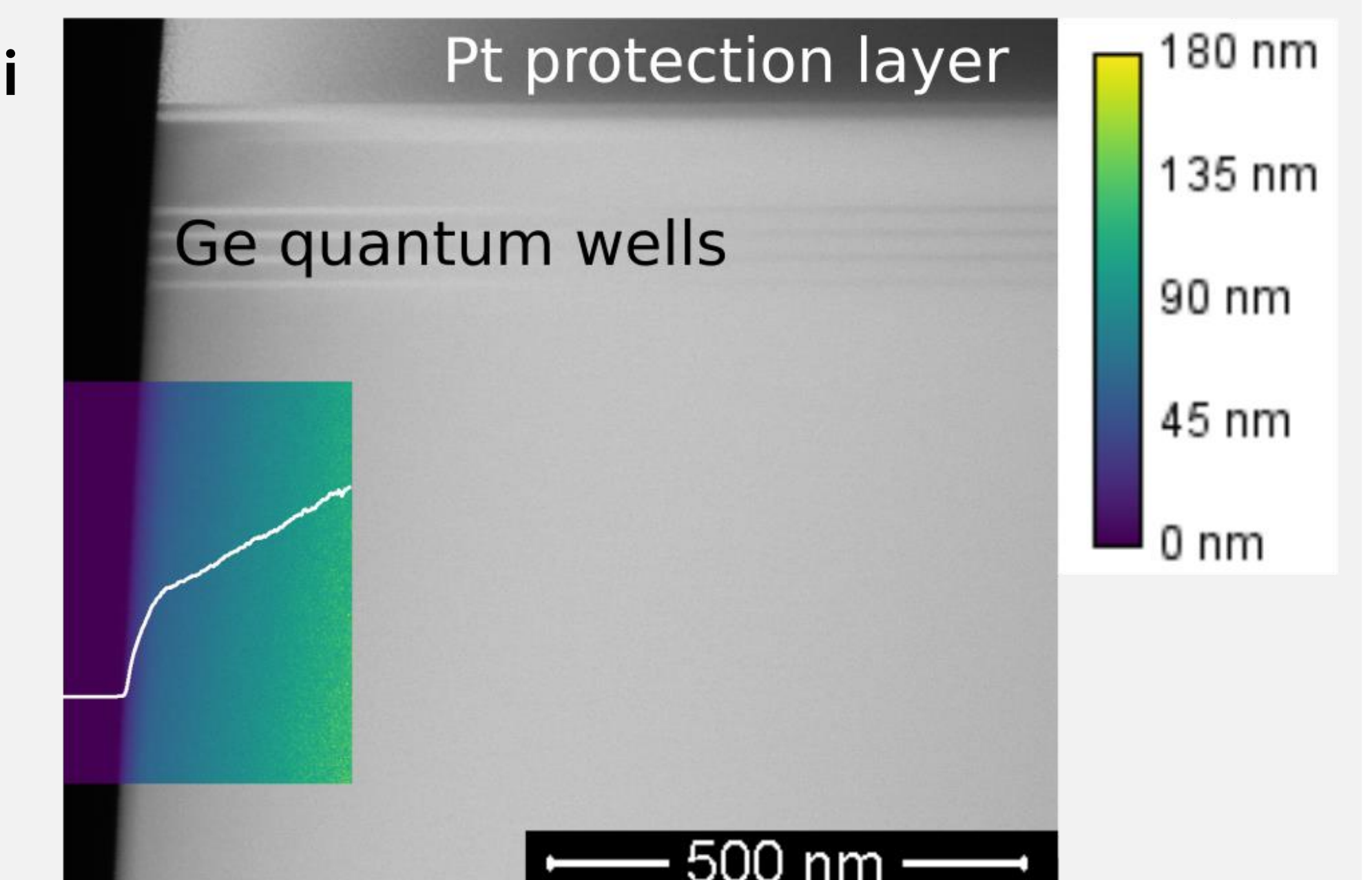
- Calculate  $I_{nor}$  for scattering-angle range of detector segment as a function of sample thickness  $t$  (modified NISTMonte 1.2 [Rit])
- Input parameters: geometrical set-up of the detector, composition and density of the material, electron energy
- Normalize calculated count number with total count number
- Compare measured and simulated  $I_{nor}$
- Accuracy of the input parameters determines simulation accuracy
- Limitation:  $t < t_{inv}$  (inversion point)



Monte-Carlo simulations of  $I_{nor}$  at 30 keV as a function of thickness for Si

## Application Examples

- Wedge-shaped TEM sample of Si
- Color-coded inset is a thickness map



- Determination of contamination thickness (square patches) on an amorphous carbon film

