LAboratorio di Spettroscopie Elettroniche e di Correlazione

GRaphene to Electrons:
Energy and Angular resolved Transmission

# ELECTRON TRANSMISSION THROUGH MONOLAYER GRAPHENE BETWEEN 200 AND 5000 eV

PTOLEMY COLLABORATION MEETING, 01/07/2025

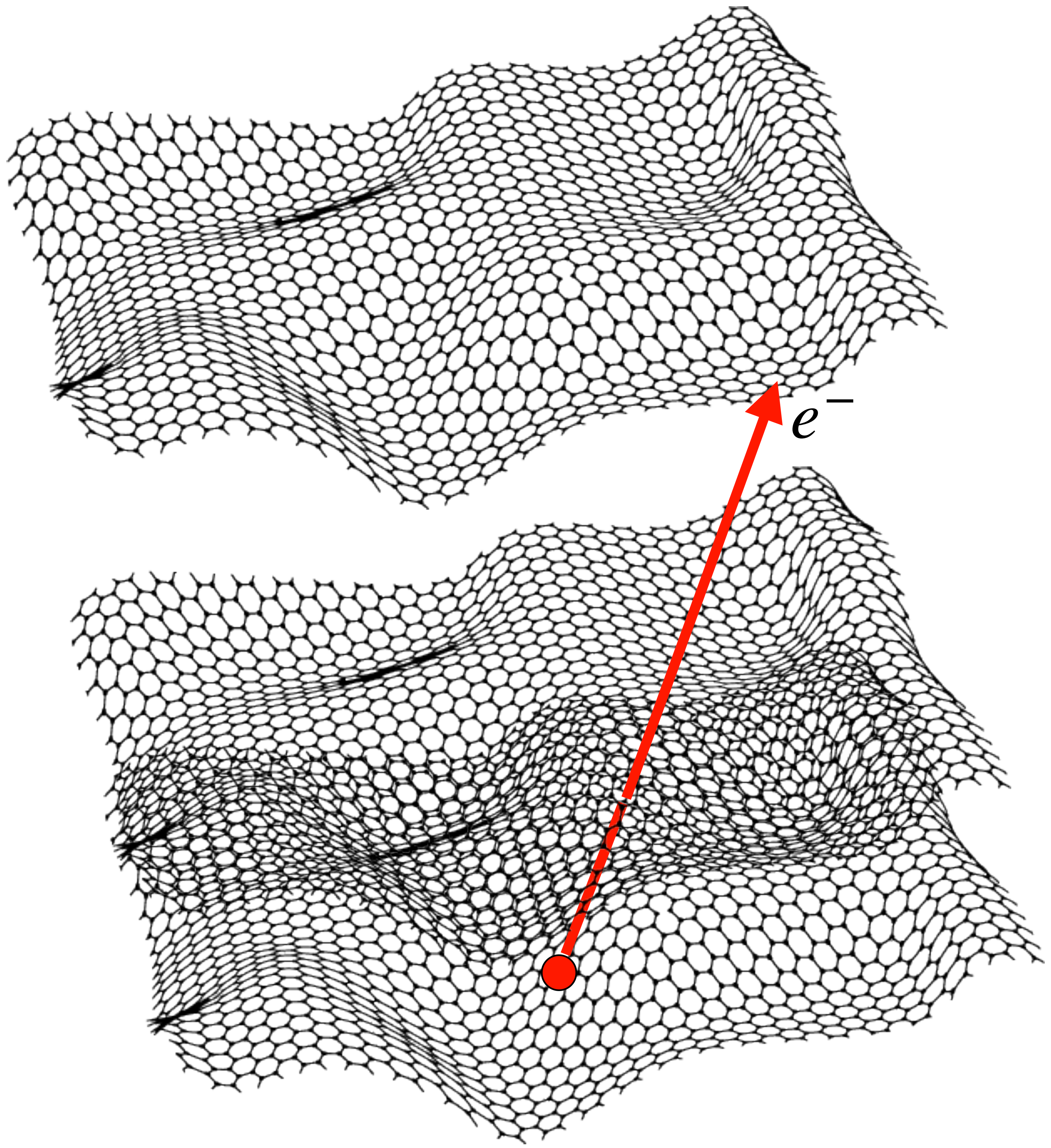
ALICE APPONI, ORLANDO CASTELLANO, MARTINA CHIRICO , FRANCESCO PANDOLFI, ALESSANDRO RUOCCO

# WE NEED TO OPTIMIZE THE TRITIUM SOURCE

- 1 $\mu$g of T correspond to $\mathcal{O}(\mathbf{cm}^2)$ of graphene

1) Which is the best way to arrange all this graphene?
2) Can we stack graphene monolayers one on top of the other?

Check whether $\beta$ electrons of T are affected by the passage through graphene



# THE GREEAT PROJECT

Research article

## Transmission through graphene of electrons in the 30 – 900 eV range

Alice Apponi [a,b], Domenica Convertino [c], Neeraj Mishra [c,d], Camilla Coletti [c,d], Mauro Iodice [b], Franco Frasconi [e], Federico Pilo [e], Narcis Silviu Blaj [f], Daniele Paoloni [a,b], Ilaria Rago [g], Giovanni De Bellis [h,i], Gianluca Cavoto [g], Alessandro Ruocco [a,b]

**Transmission up to 5 keV**

**Total cross section**

**Differential cross section**

**Extend the study to other nanostructures**

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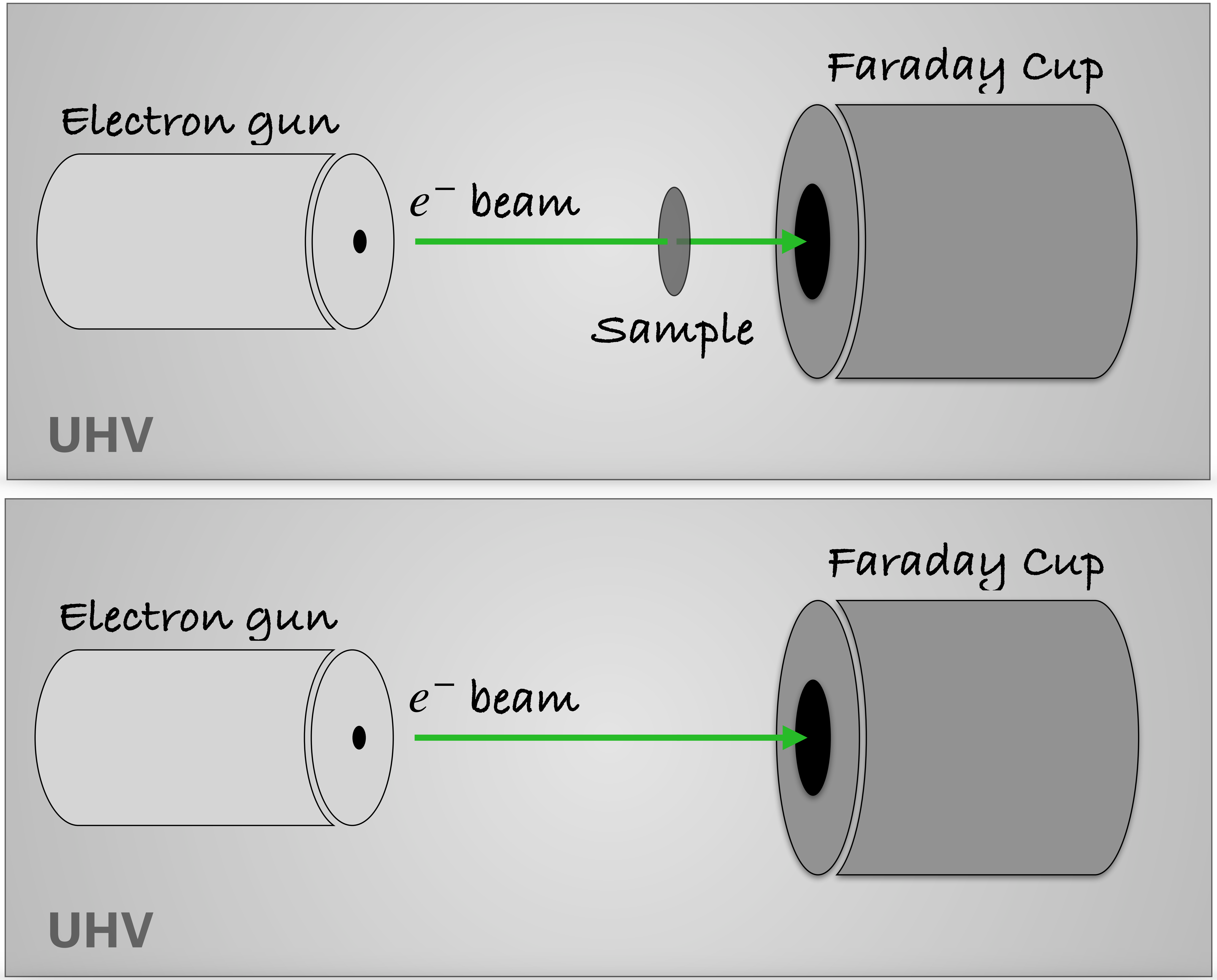
This talk!

Total cross section

Differential cross section

Extend the study to other nanostructures

# ELECTRON TRANSMISSION THROUGH GRAPHENE

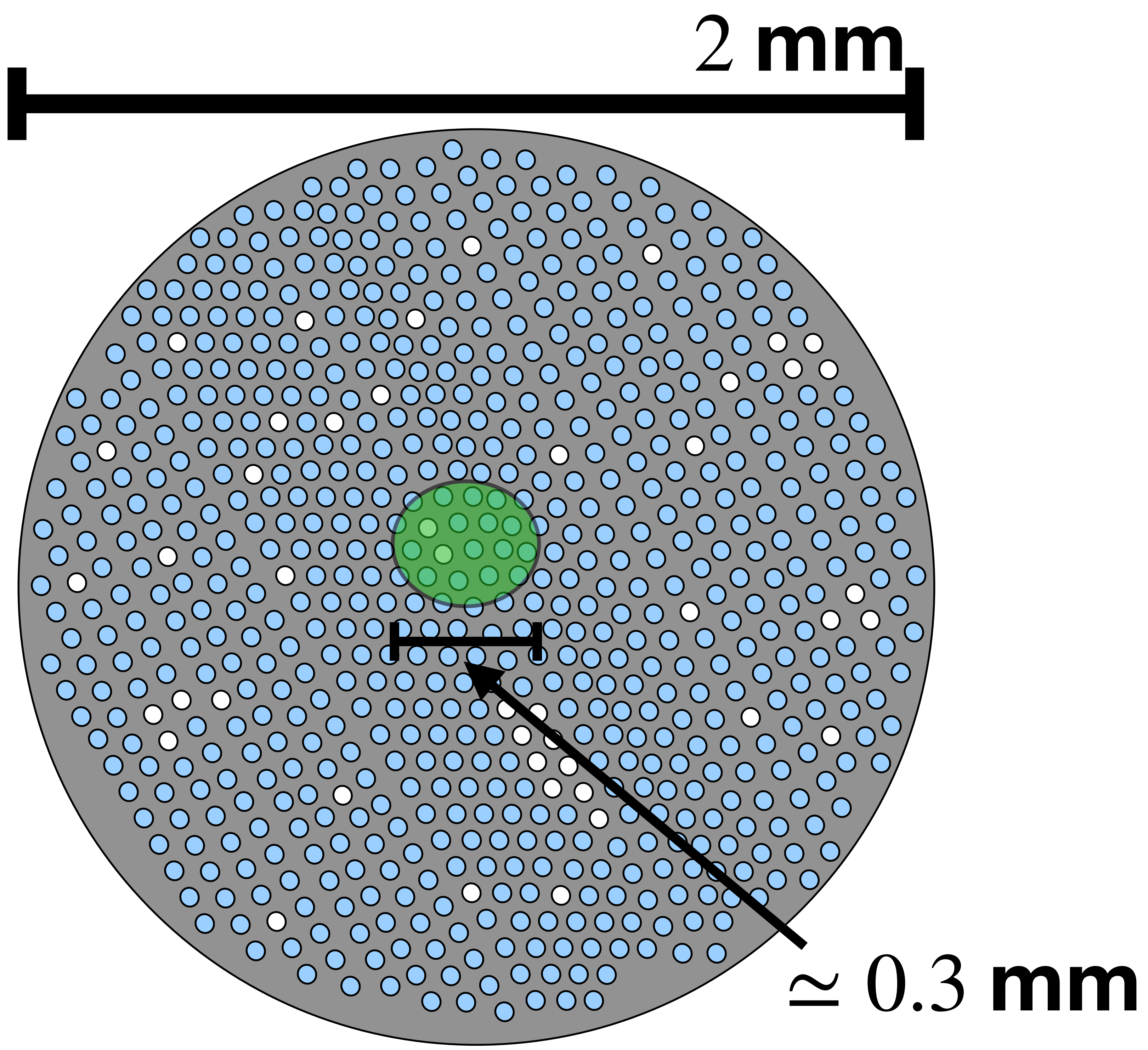



- **Measure** $I_{sample}$

**Reconstruct** $T_G$

- **Measure** $I_0$

# $I_{MEAS}$ IS A SUM OF TWO CONTRIBUTIONS




= covered hole

= electron spot

= uncovered hole

= grid

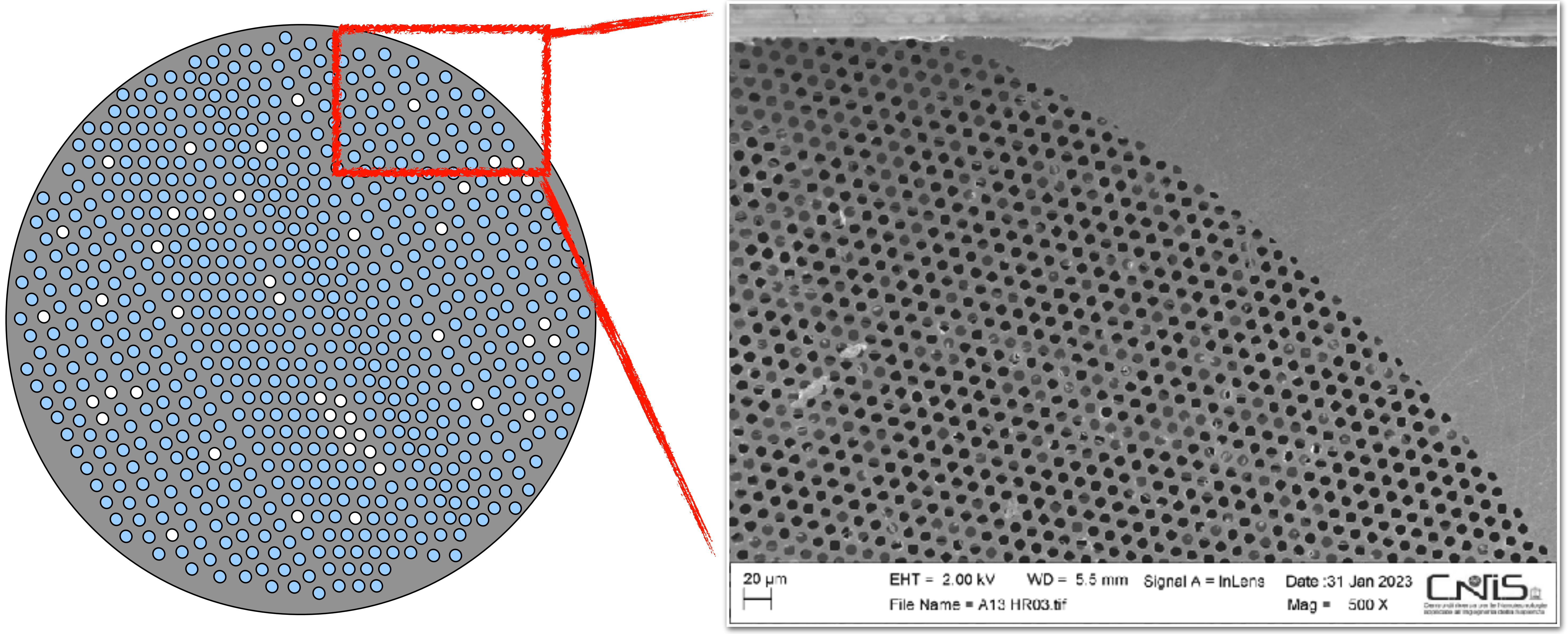
$$I_{meas} = I_0 T_{geom}[a T_G + (1 - a)]$$

**Where:**

- $T_{geom}$ = $\dfrac{\text{Area ( + )}}{\text{Area ( + + )}}$

- $a$ = $\dfrac{\text{Area ( )}}{\text{Area ( + )}}$

# SAMPLE CHARACTERIZATION @ SEM



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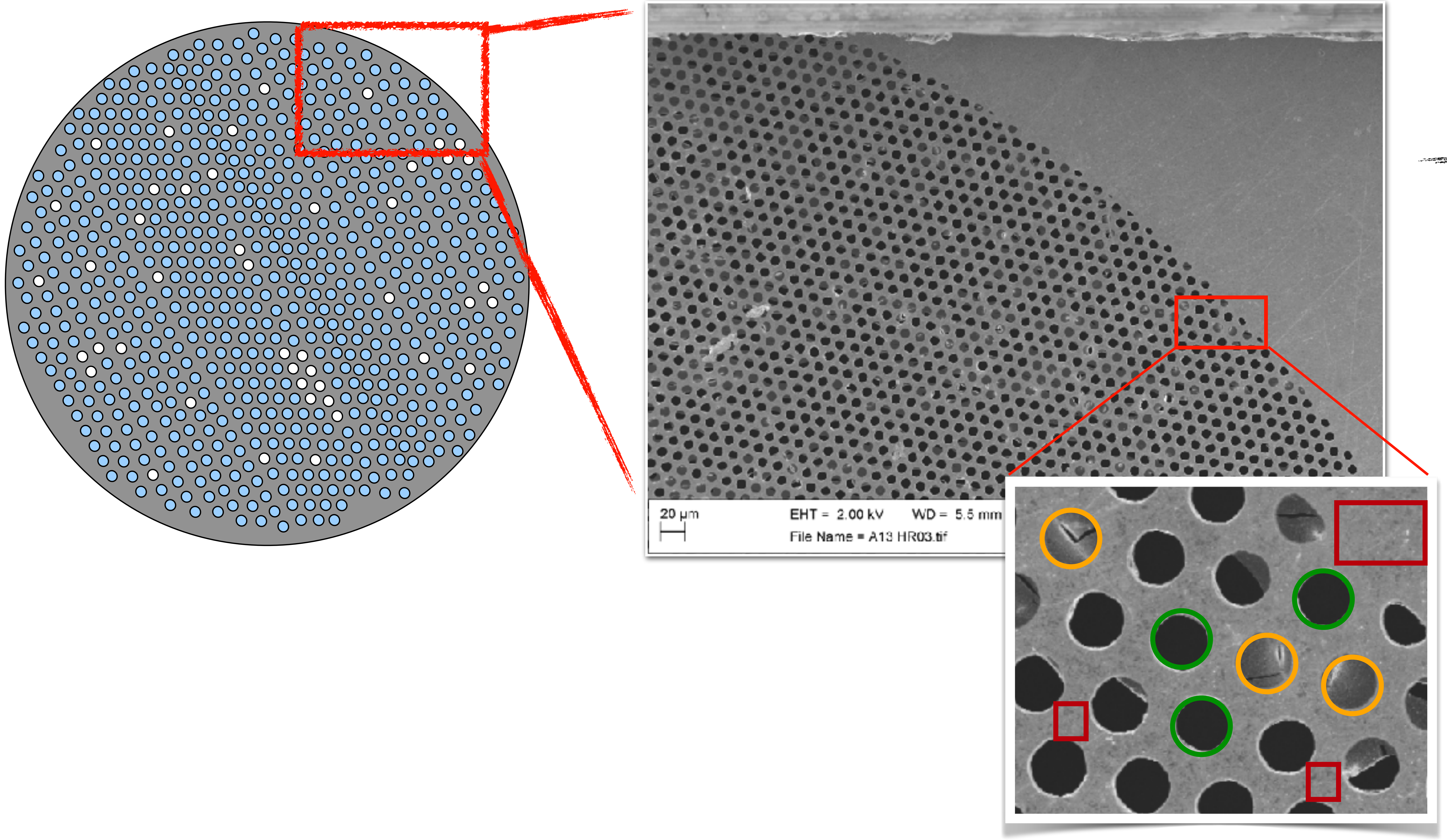
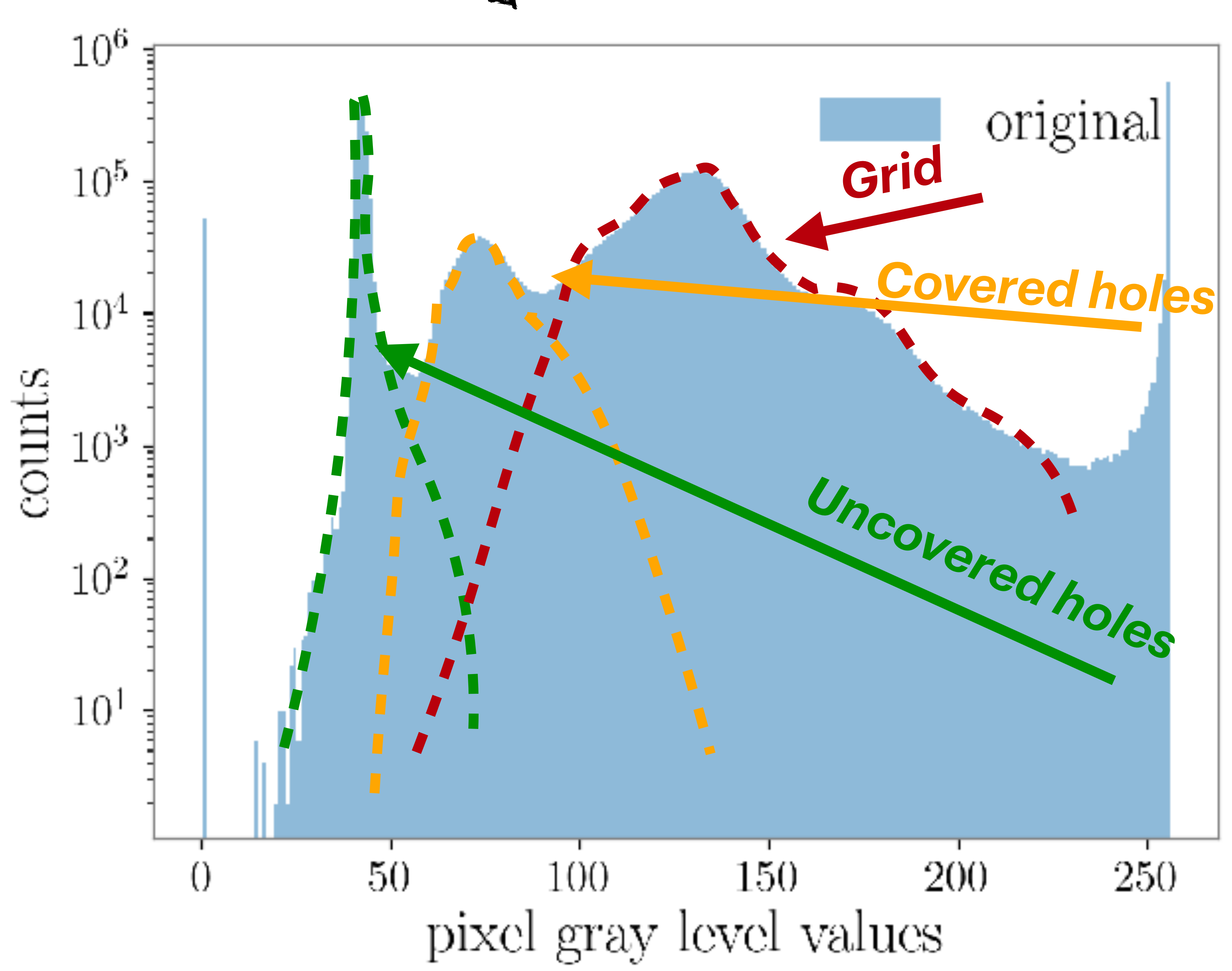



**Pixel gray level histogram**



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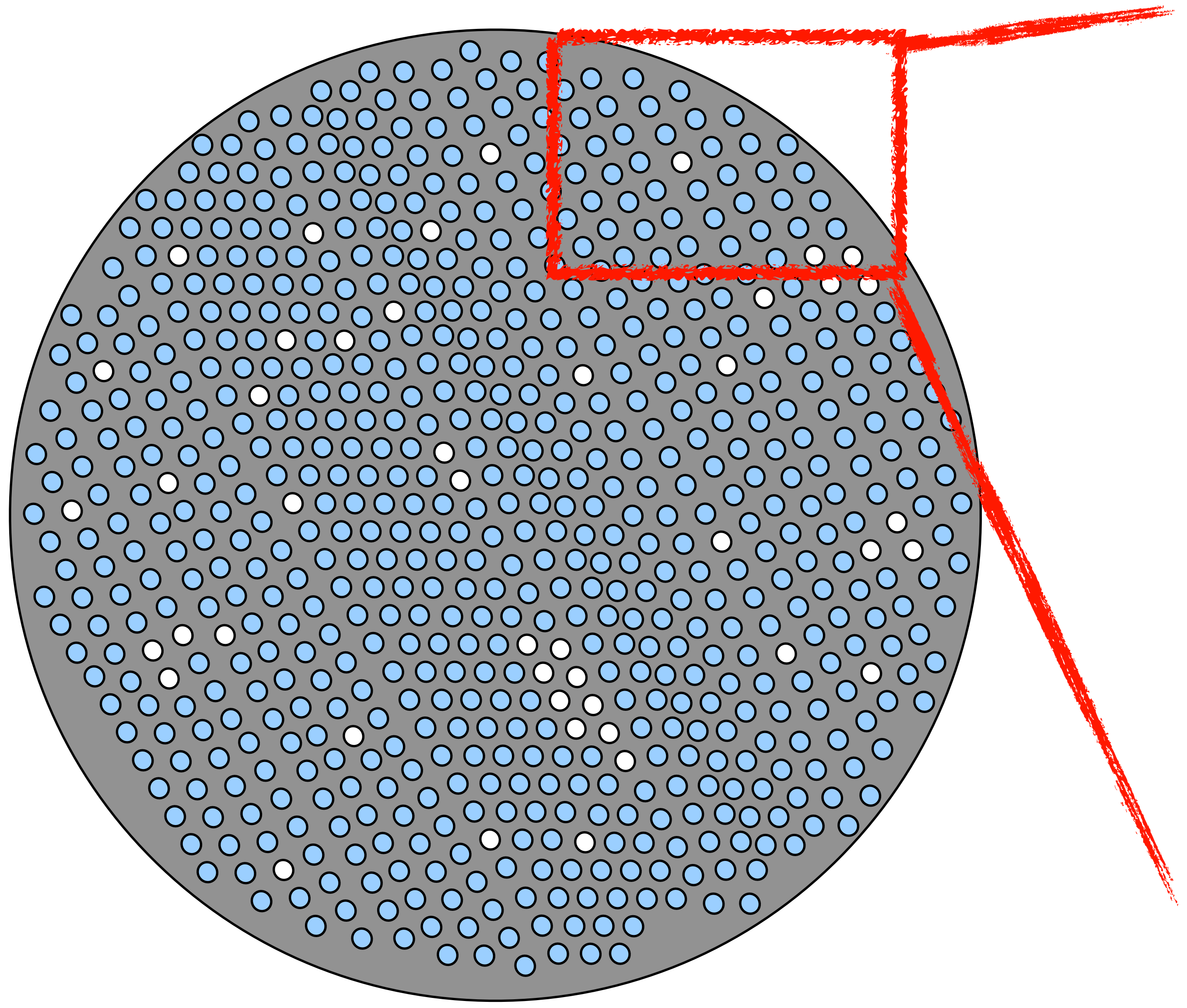

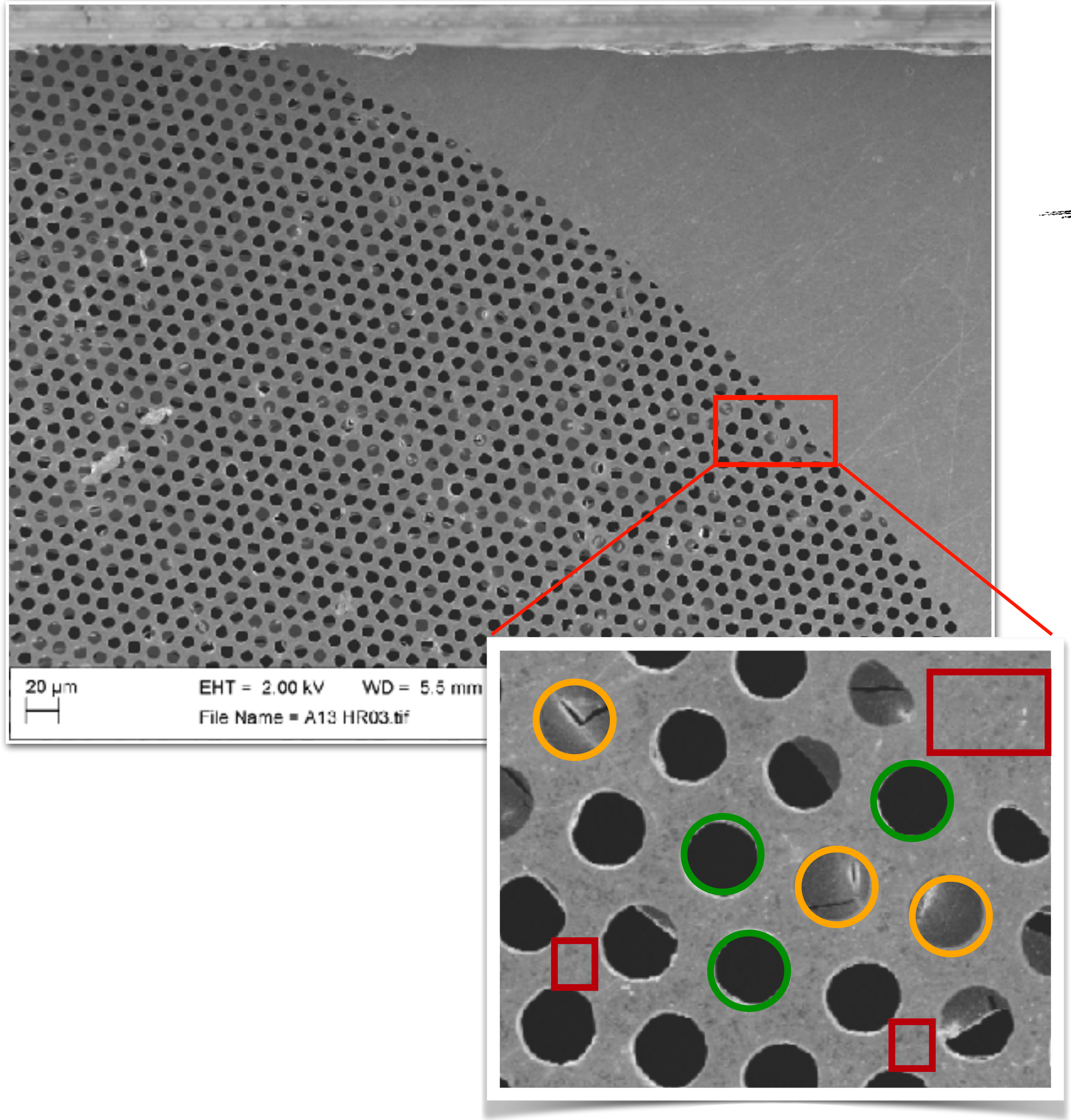


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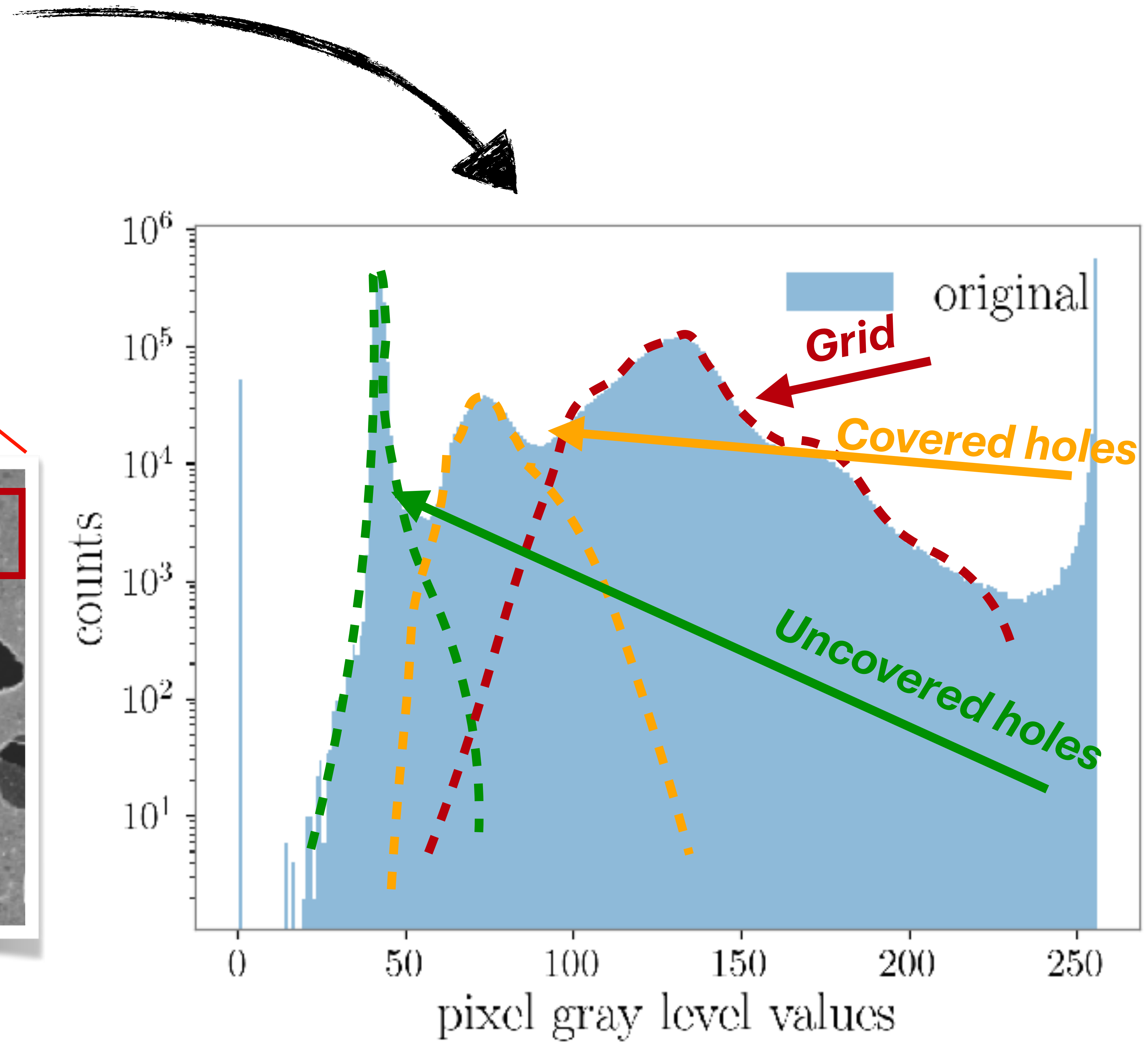
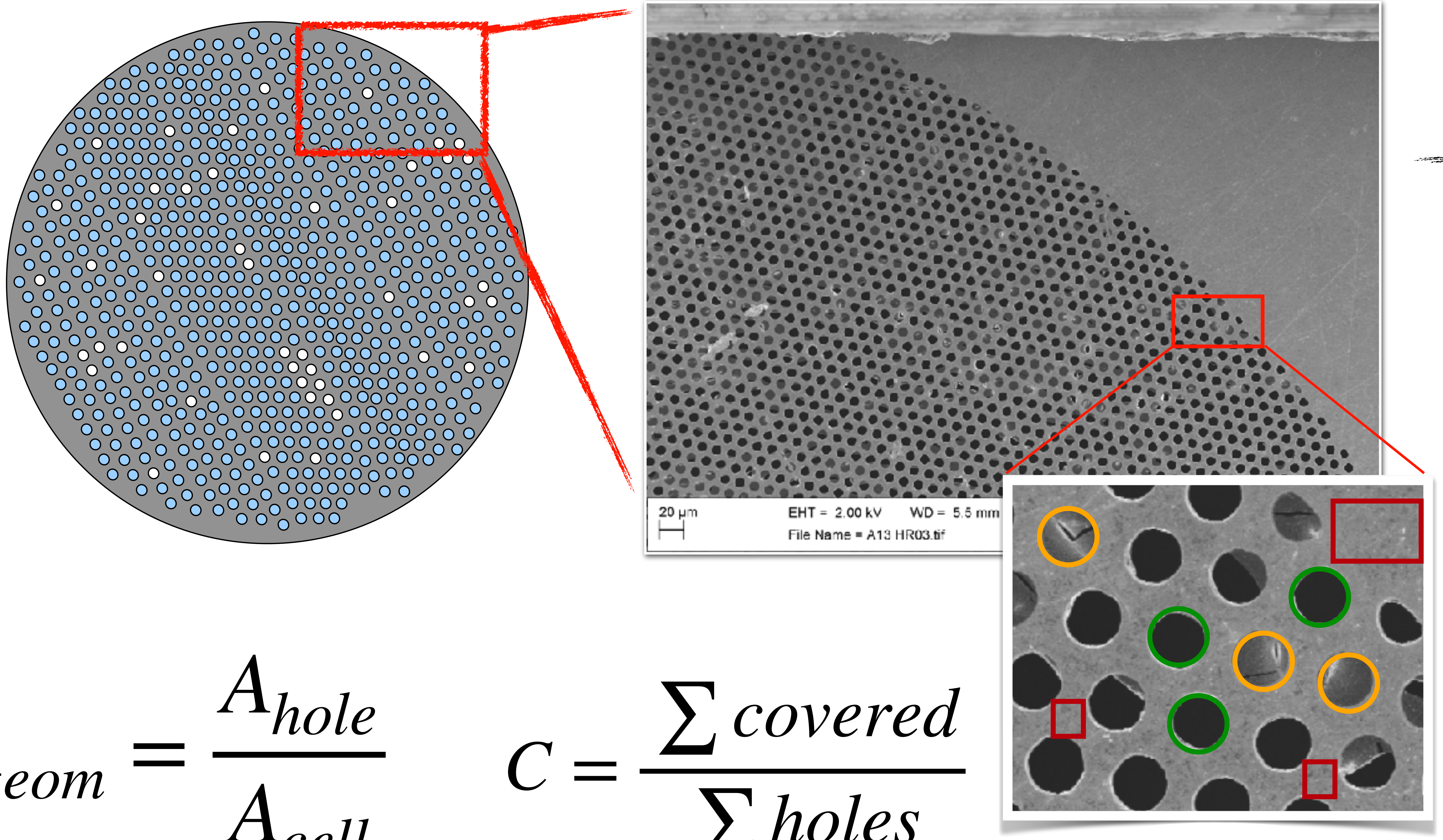
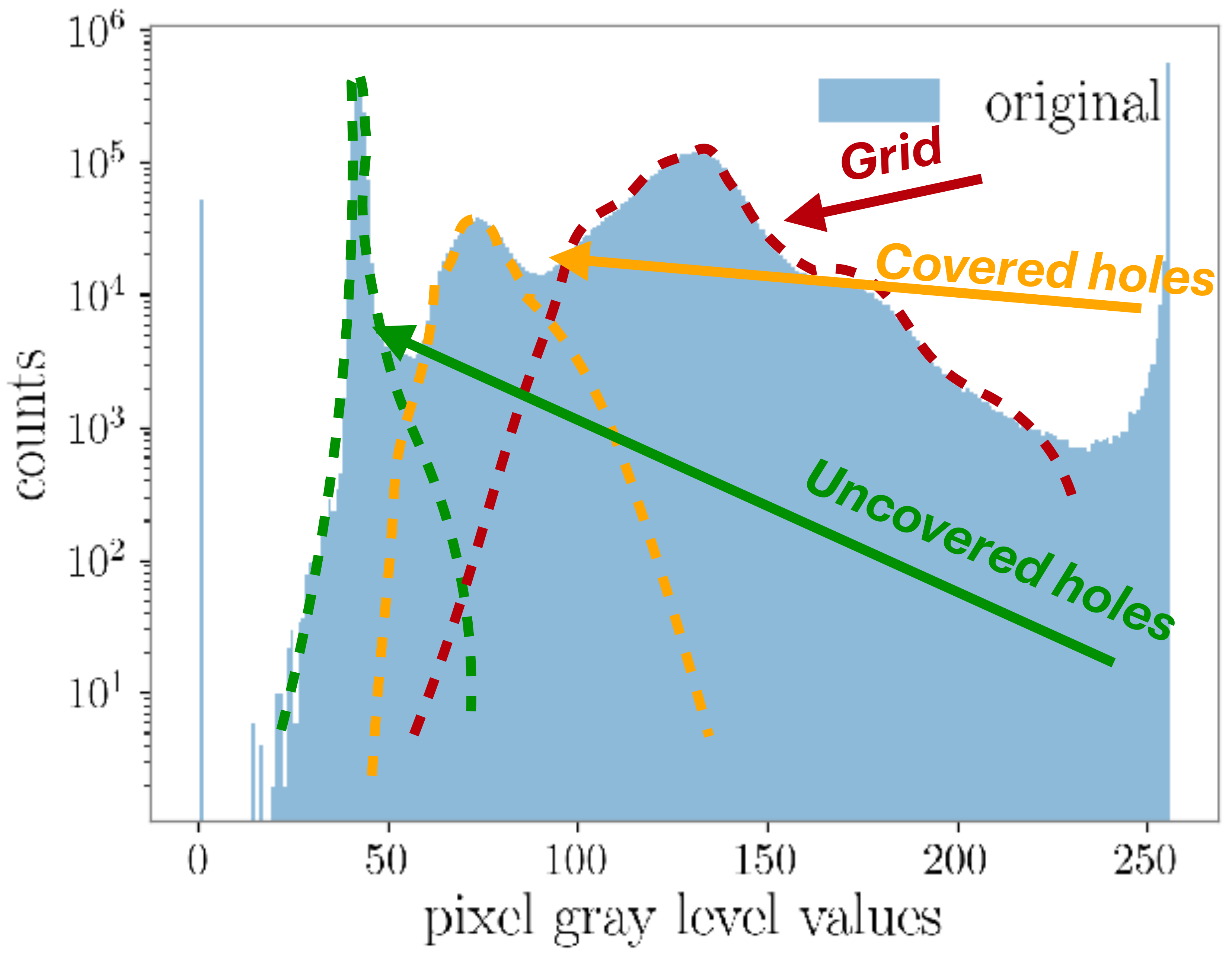



$$T_{geom} = \frac{A_{hole}}{A_{cell}}$$

# SAMPLE CHARACTERIZATION @ SEM




Pixel gray level histogram

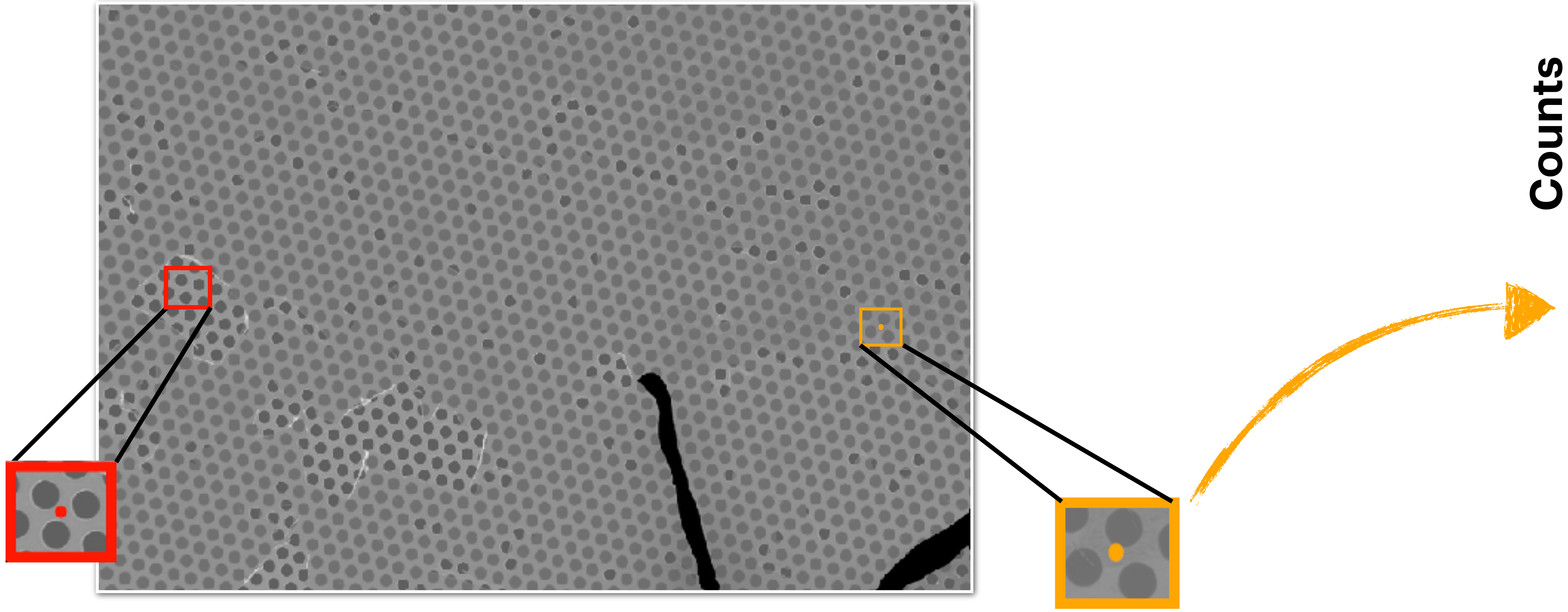



$$T_{geom} = \frac{A_{hole}}{A_{cell}}$$

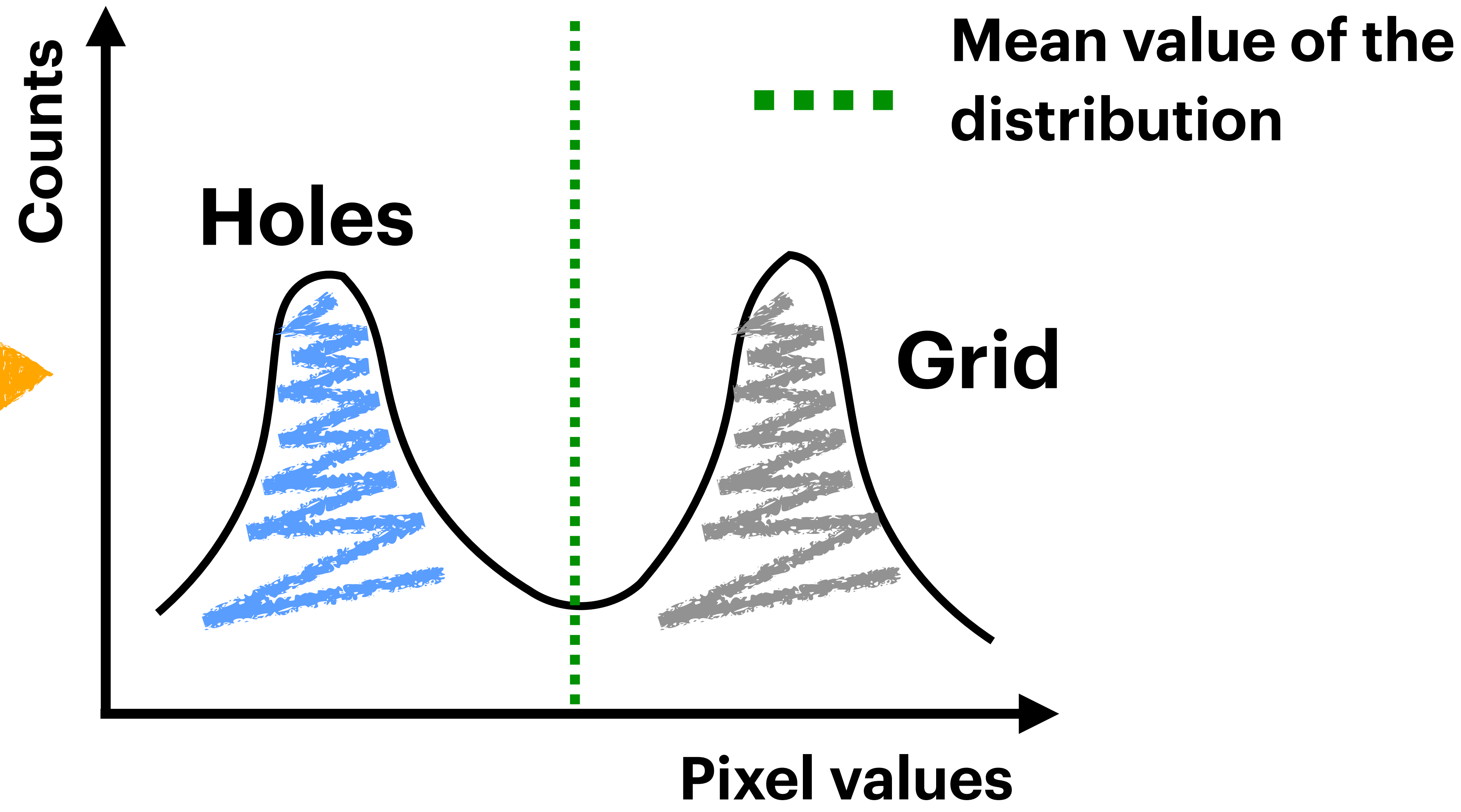
$$C = \frac{\sum covered}{\sum holes}$$

# CREATE A MASK TO EXTRACT HOLES

- Cut labels and corrupted parts



Pixel gray level histogram inside the box

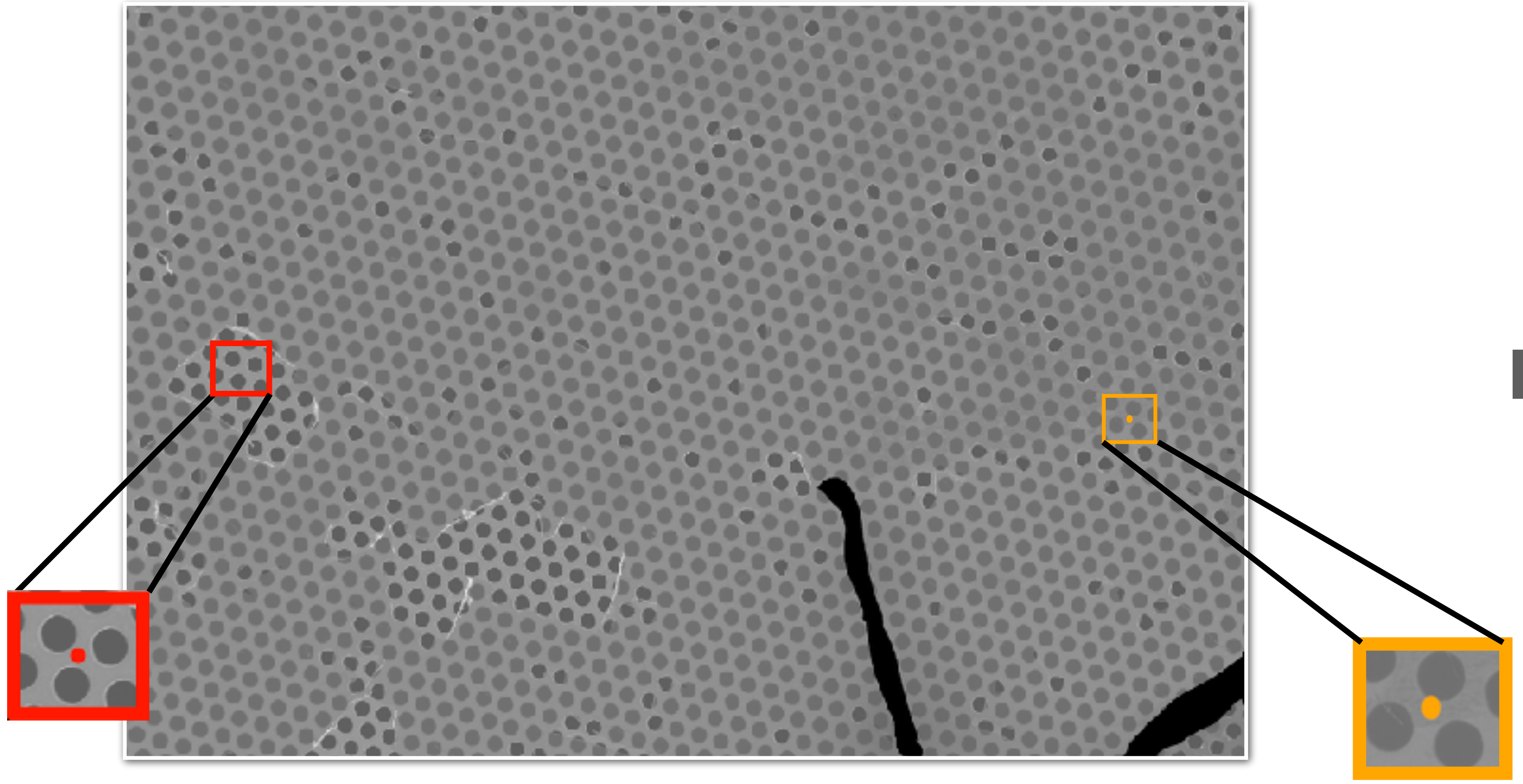


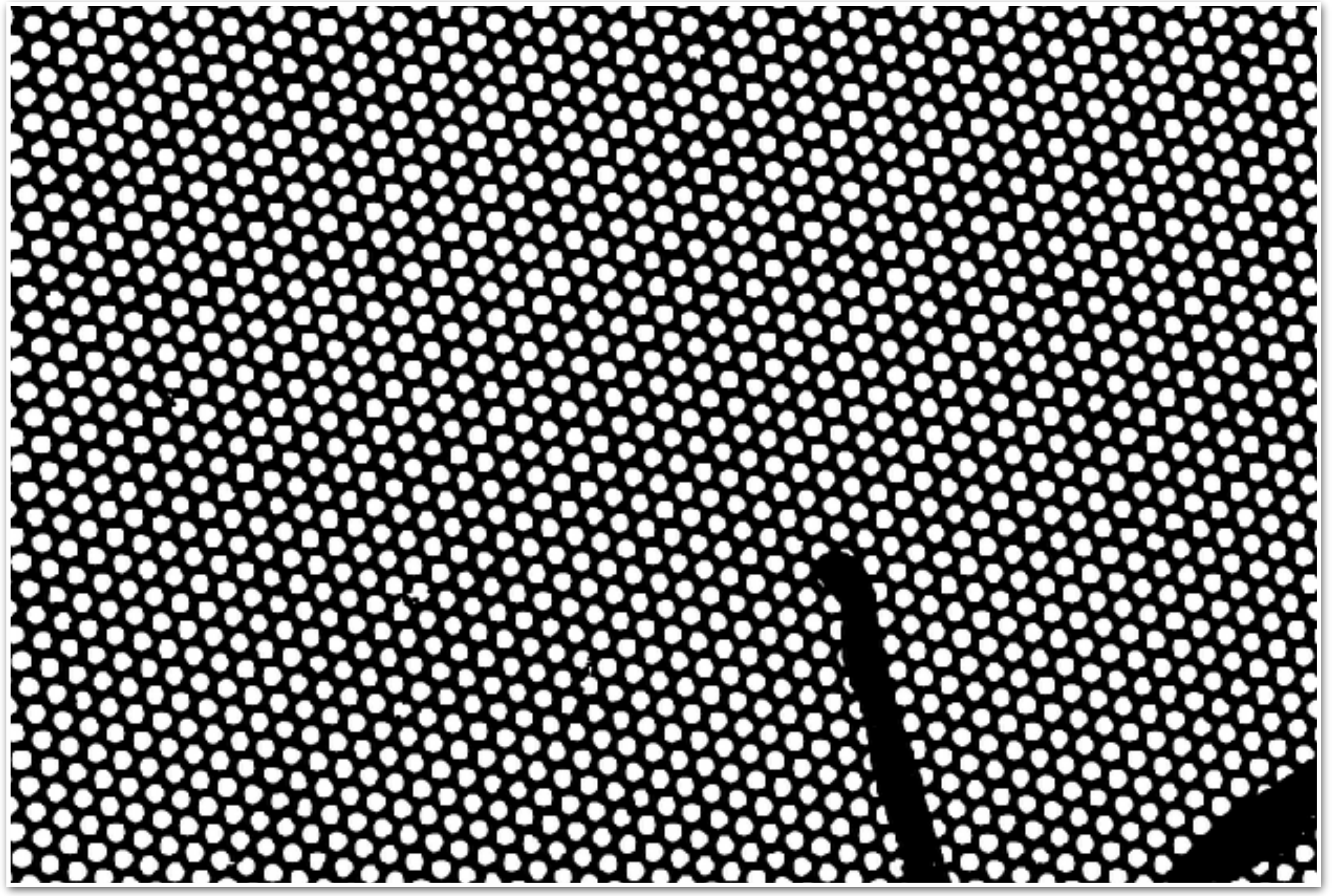

- Choose threshold pixel by pixel (*Adaptive threshold*): mean inside the box + offset

Actually the best choice depends on whether you are considering covered or uncovered places of the grid

# CREATE A MASK TO EXTRACT HOLES

- **Cut labels and corrupted parts**





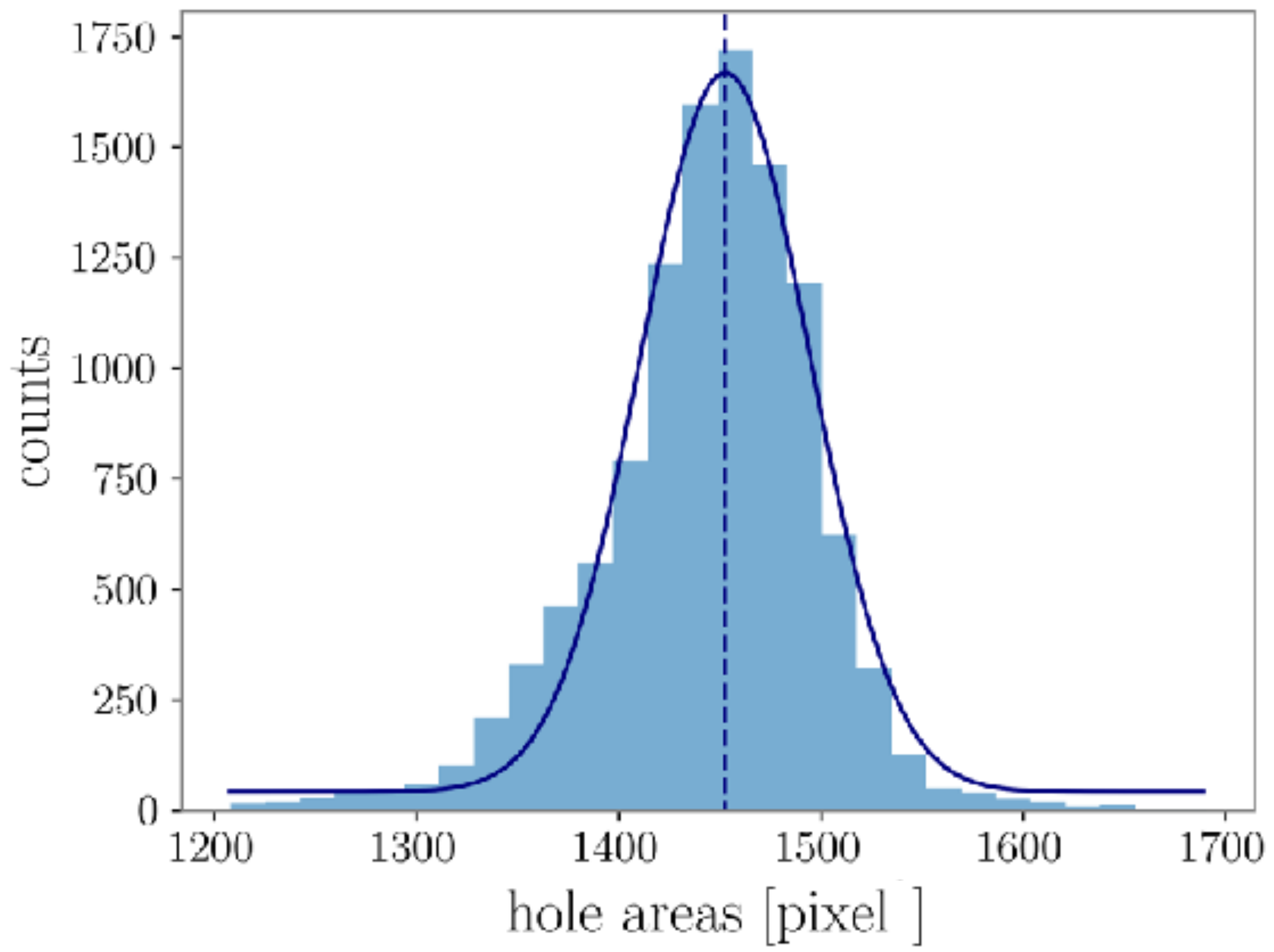
- **Select *good* holes:**

  **- size** $\in [1100,1700]$ **pixel**$^2$

  **- circularity** $\in [0.92,1.00]$

- **Choose threshold pixel by pixel (*Adaptive threshold*): mean inside the box + offset**

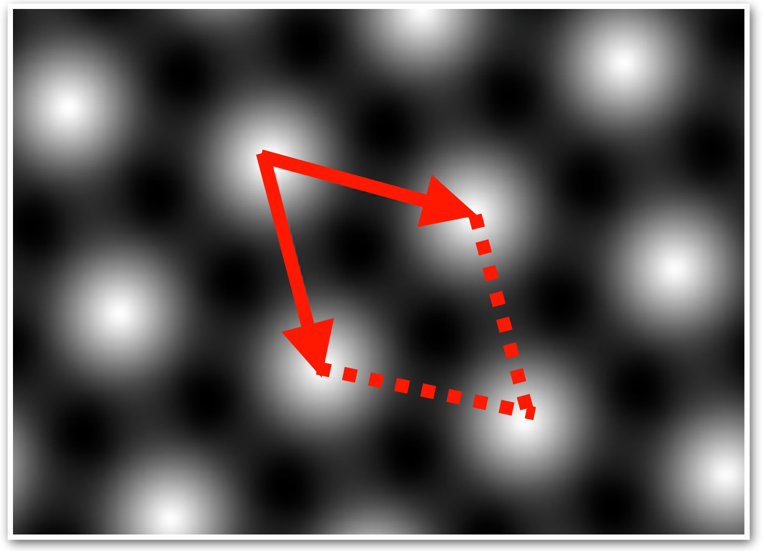
# HOLE AREAS FOR GEOMETRIC TRANSMISSION




$\bar{A} = (1452 \pm 7)$ **pixel**

$A_{cell} = (3473 \pm 3)$ **pixel**

**Autocorrelation spectrum:**



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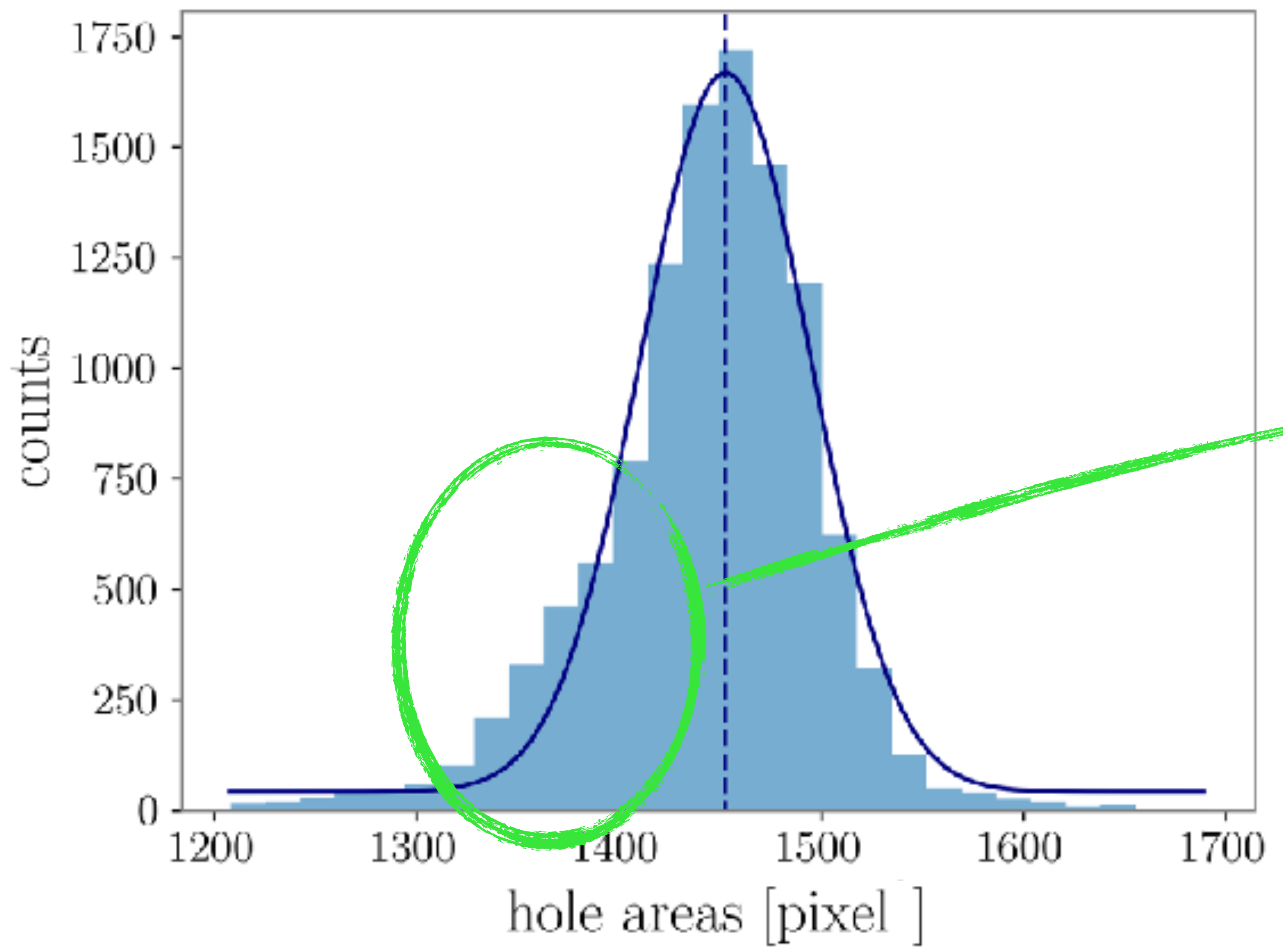
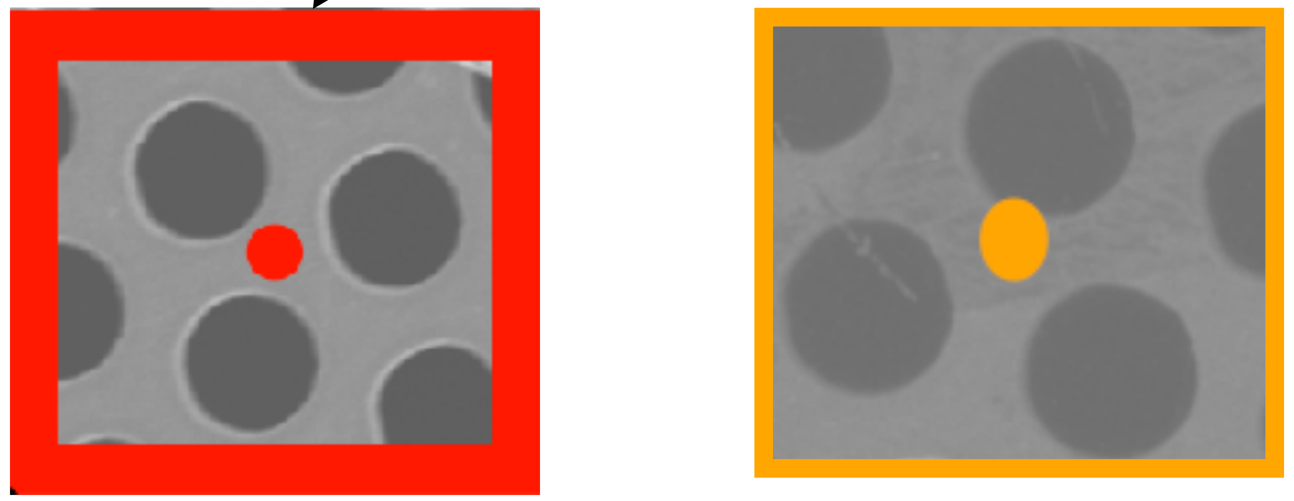



This histogram contains **two populations** of areas, one for the covered holes and one for the uncovered. It is an artifact of the thresholding prescription we are using

Different gradients at the borders



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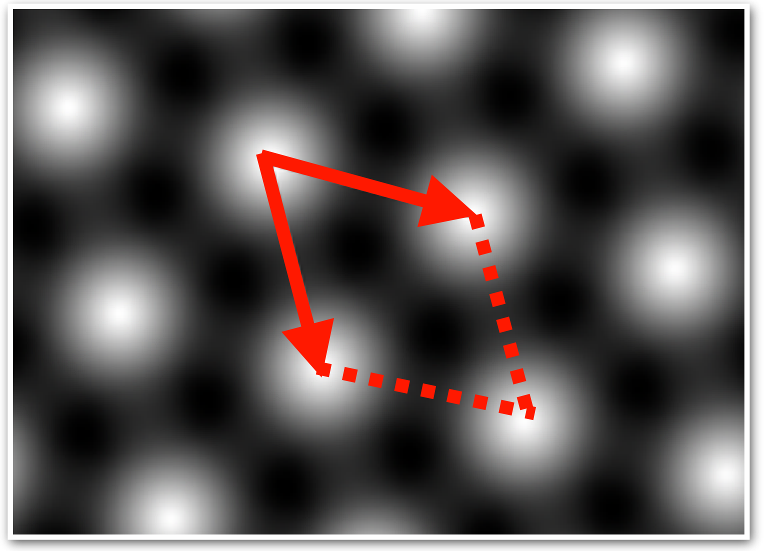
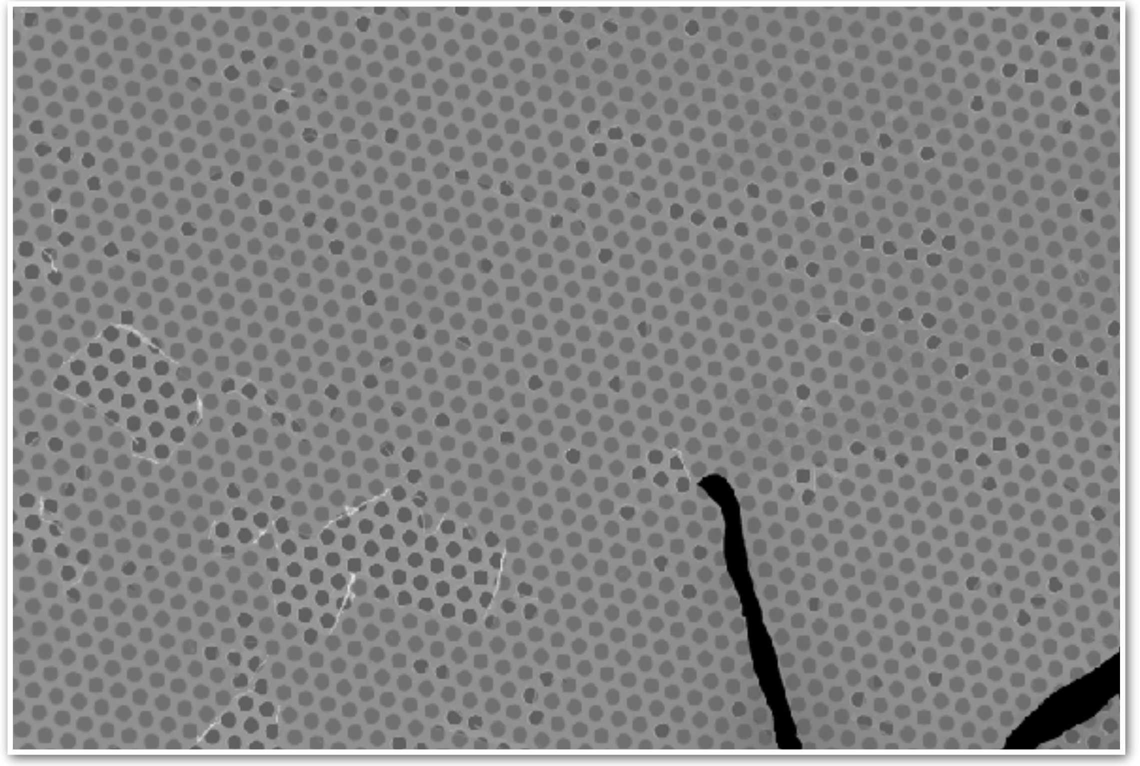
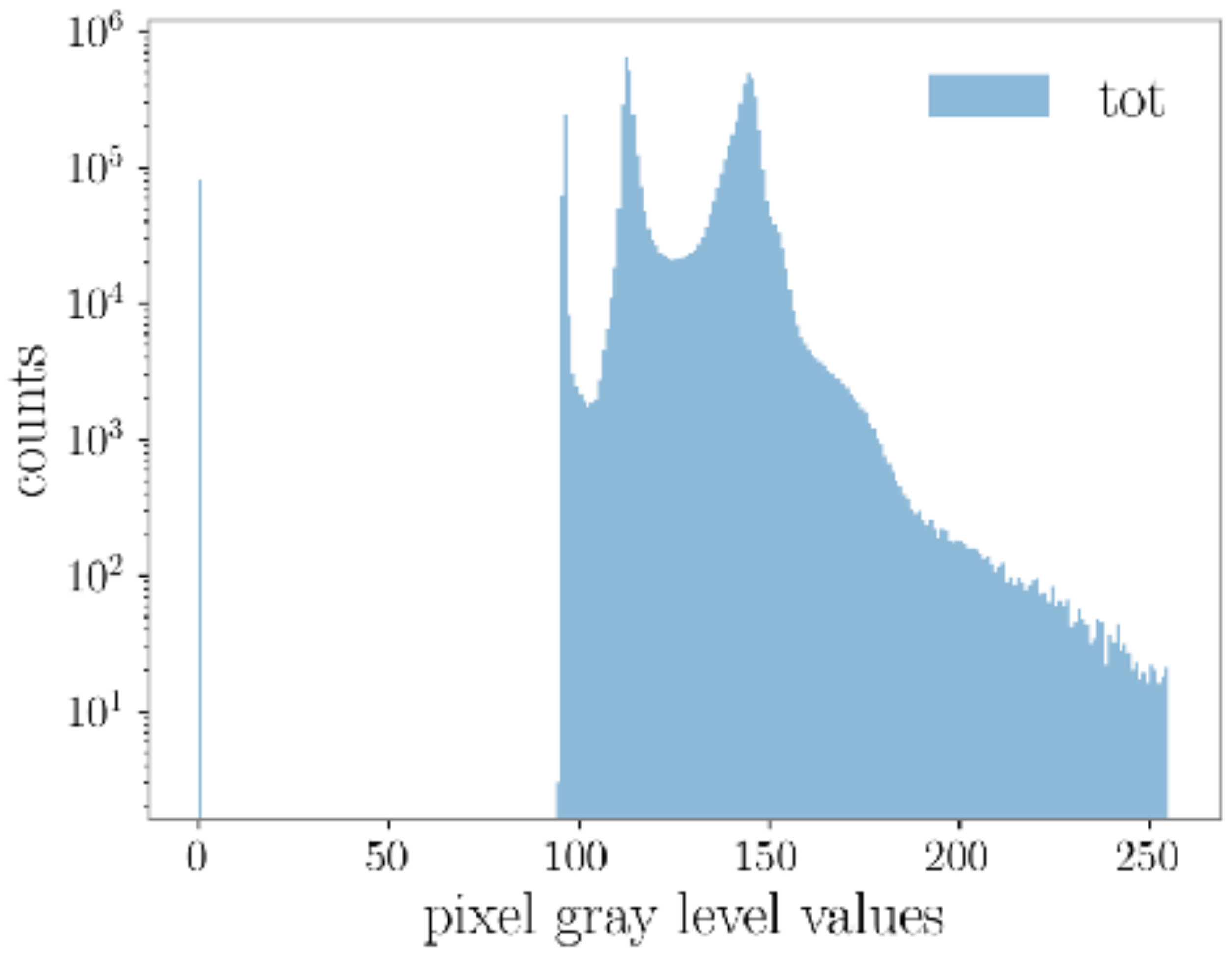


$$T_{geom} = (42.0 \pm 1.6)\ \%$$

# GRAPHENE IDENTIFICATION





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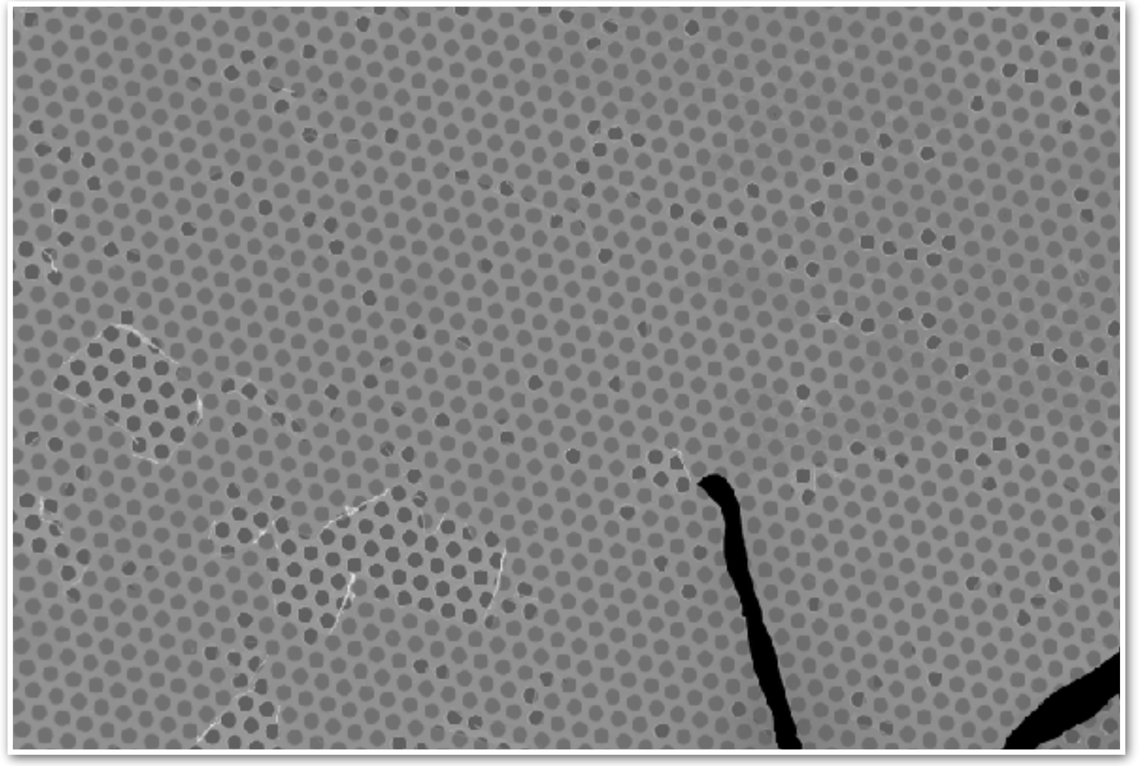

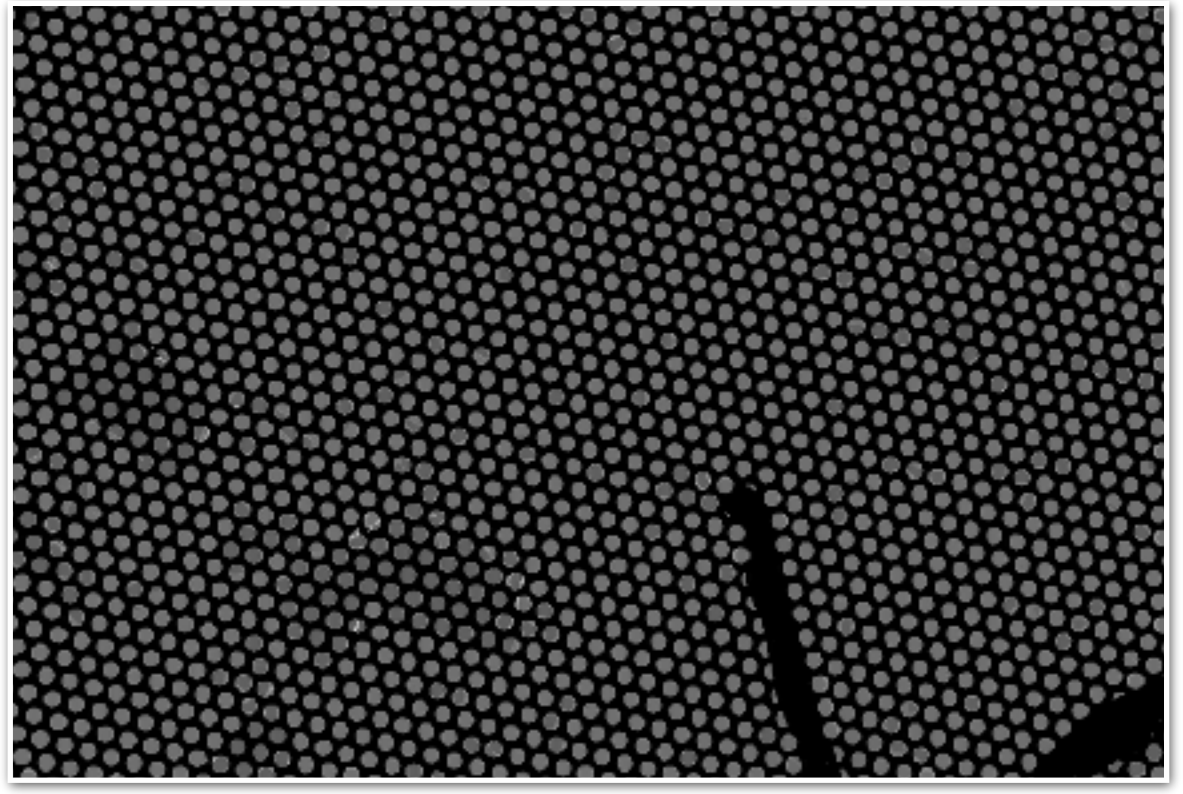

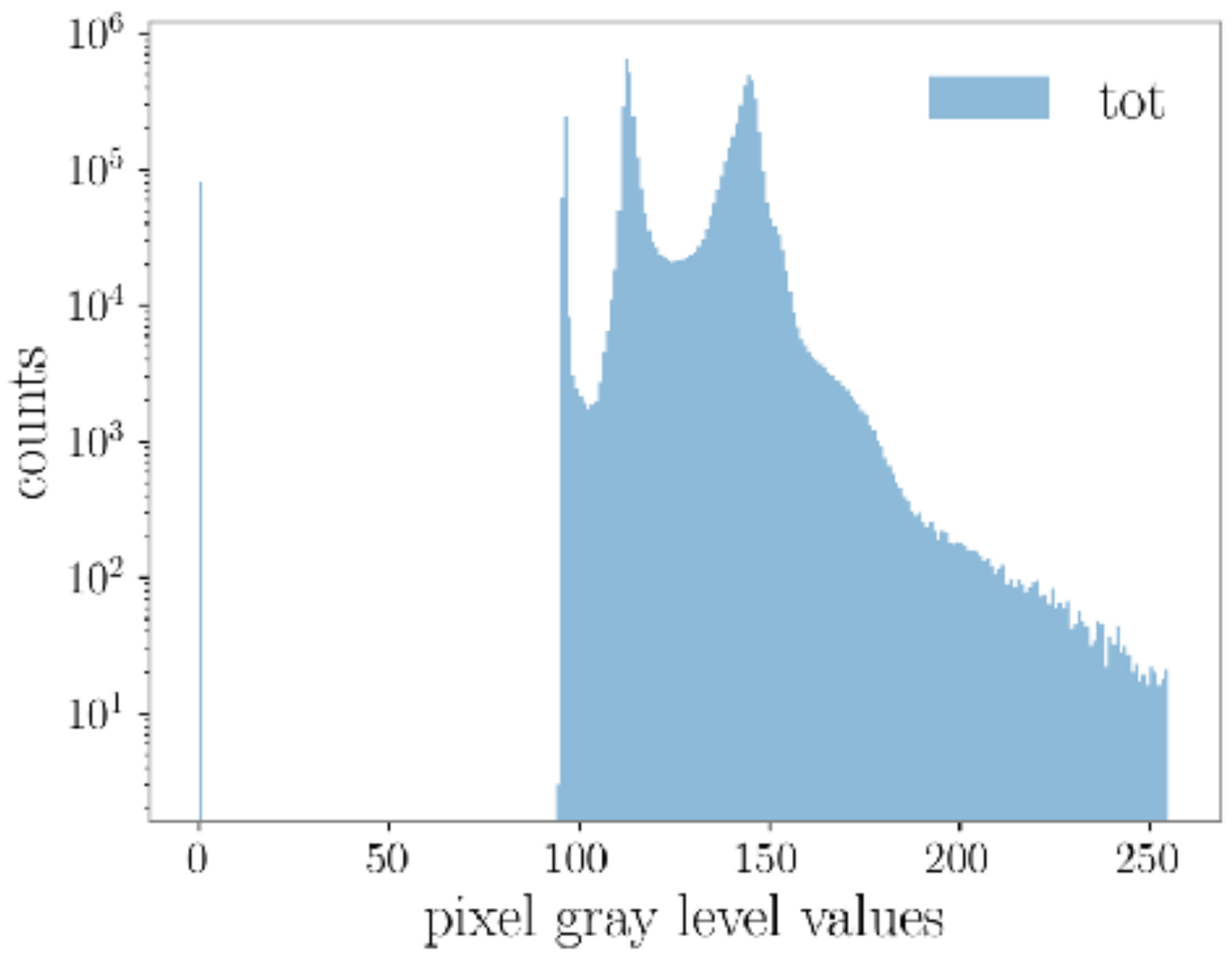


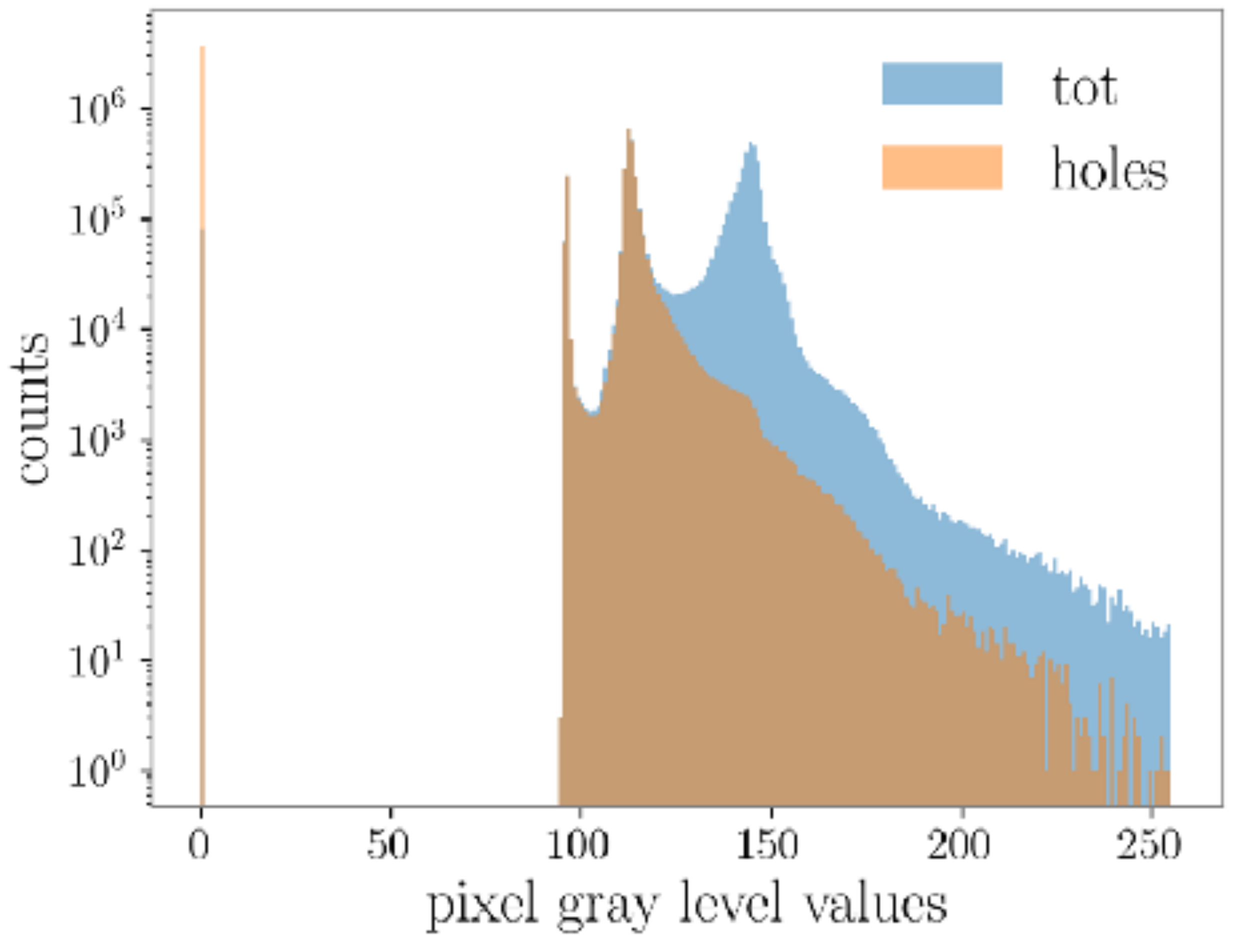

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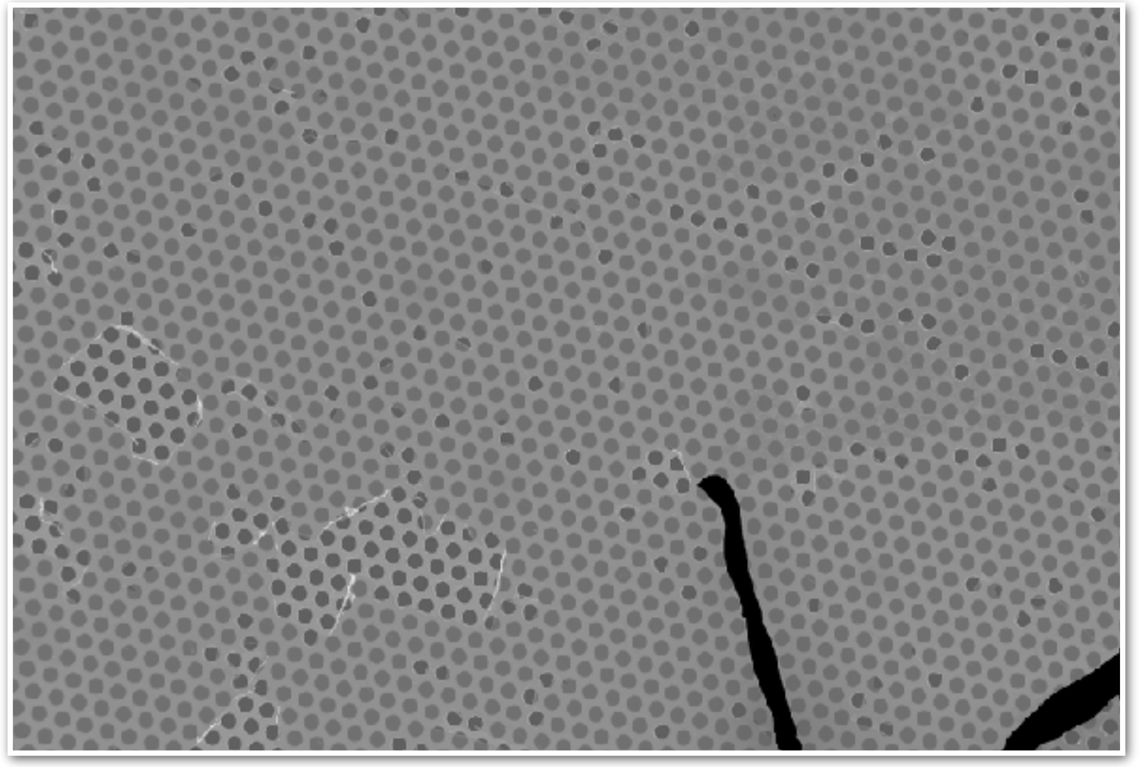

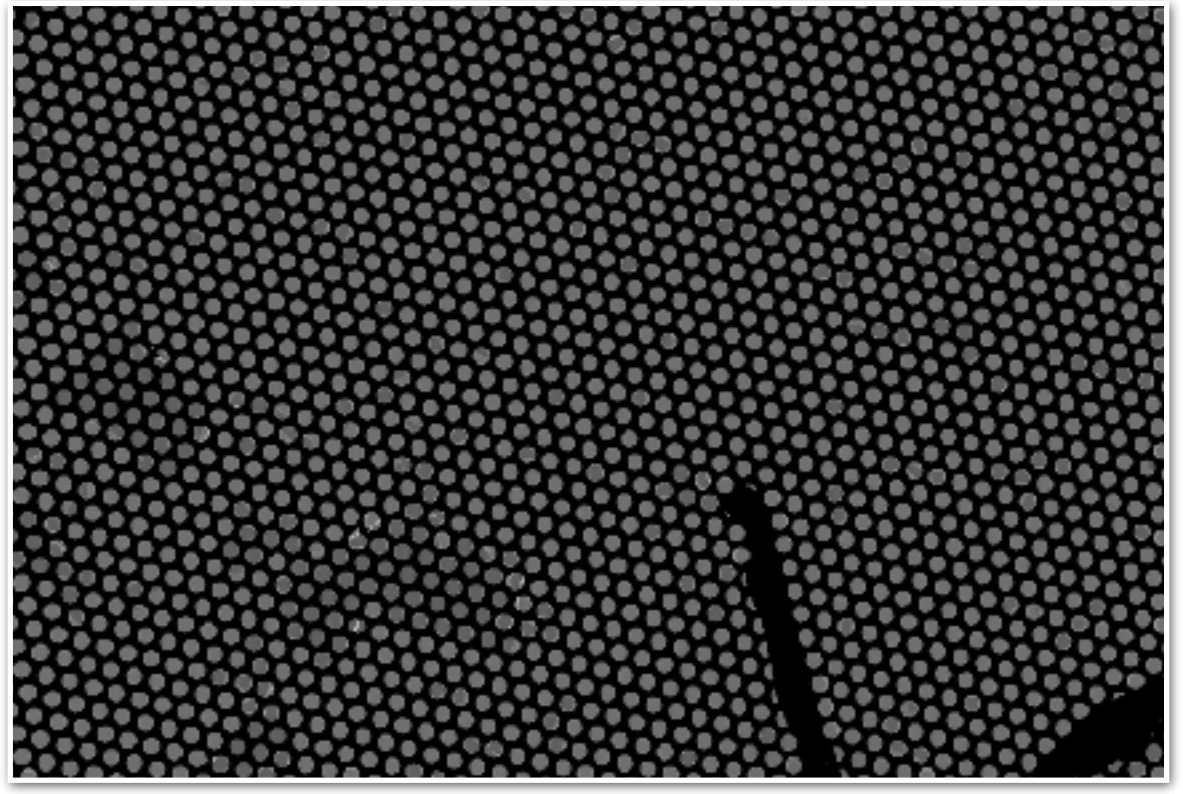

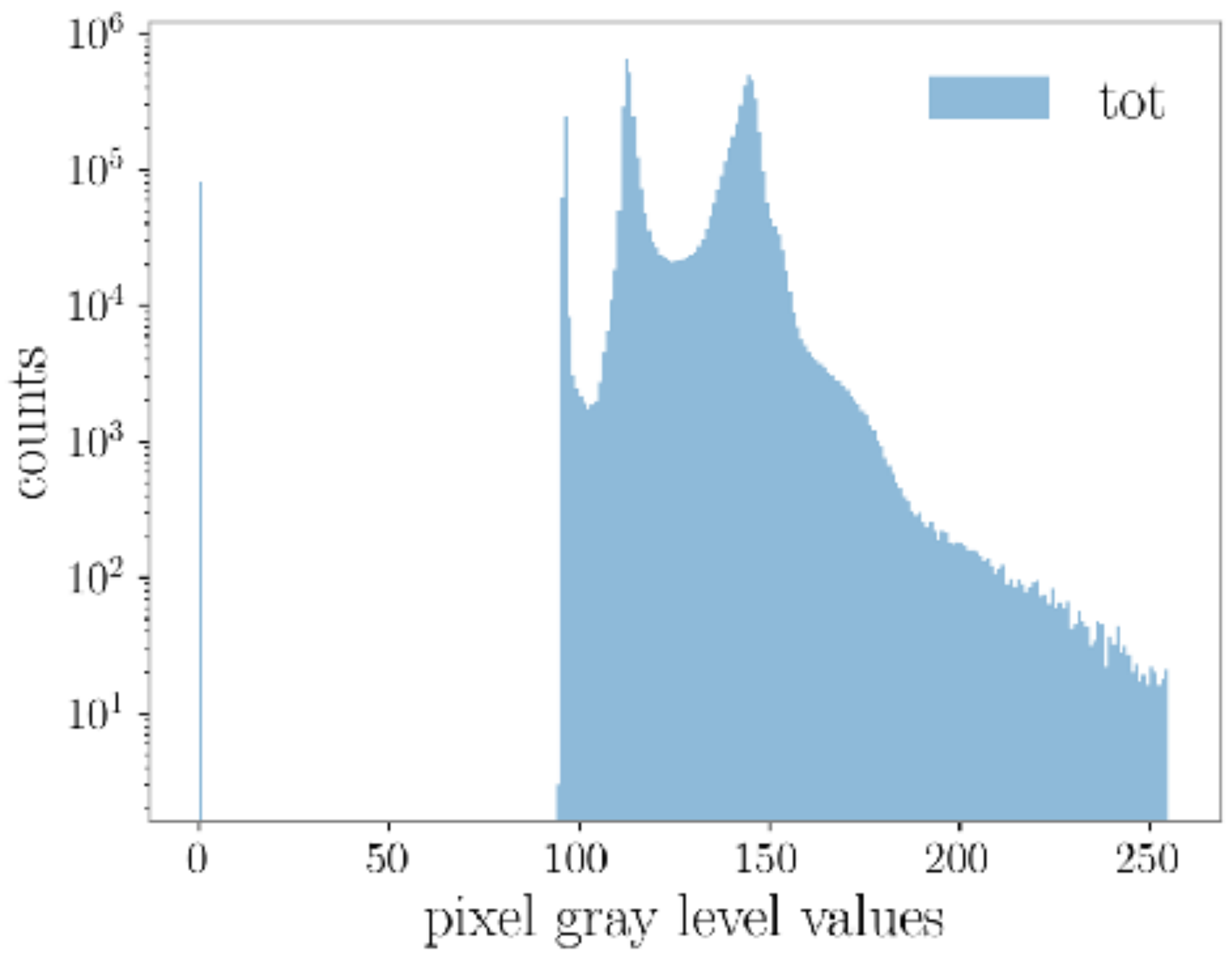


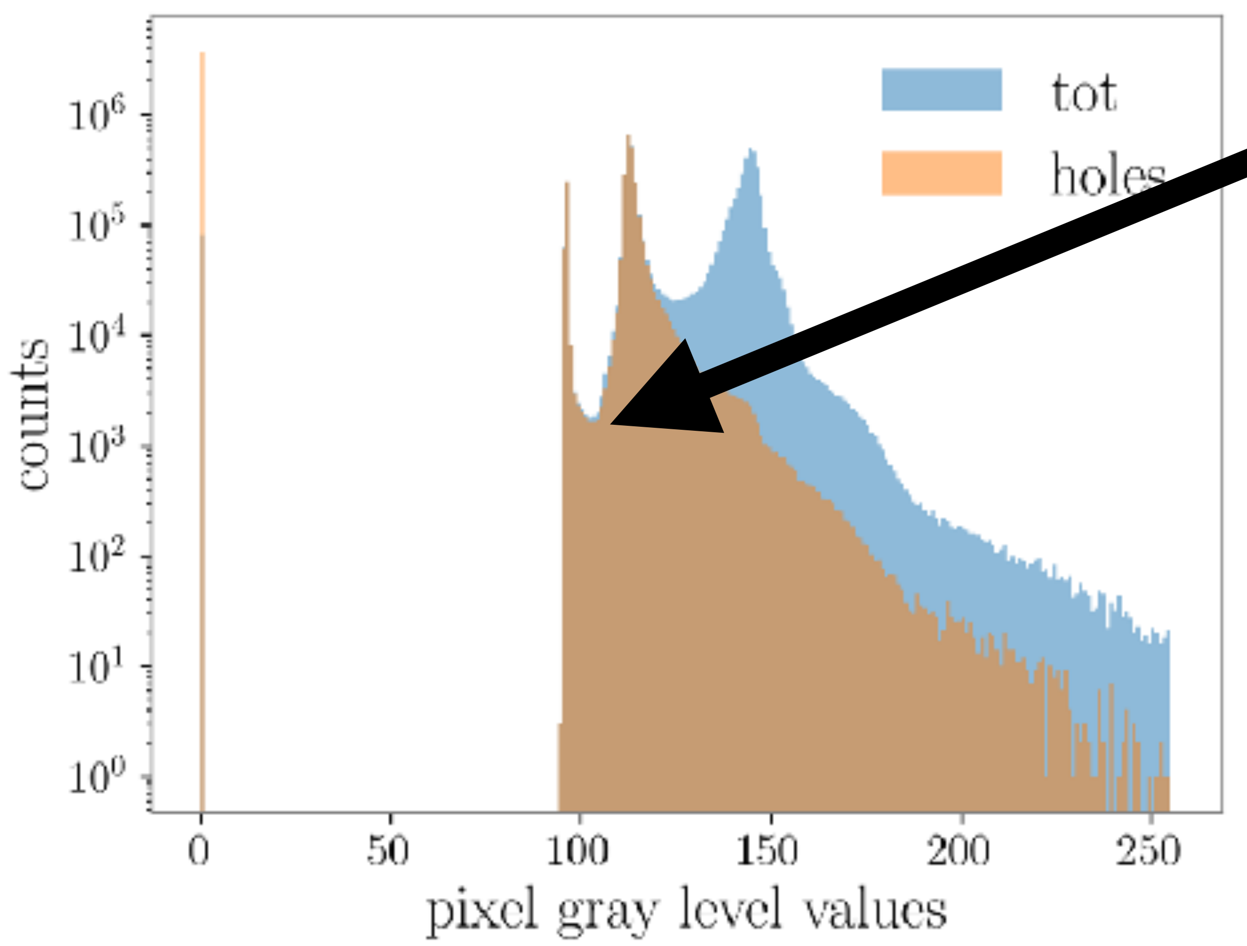


**Compute minimum to separate covered and uncovered holes**

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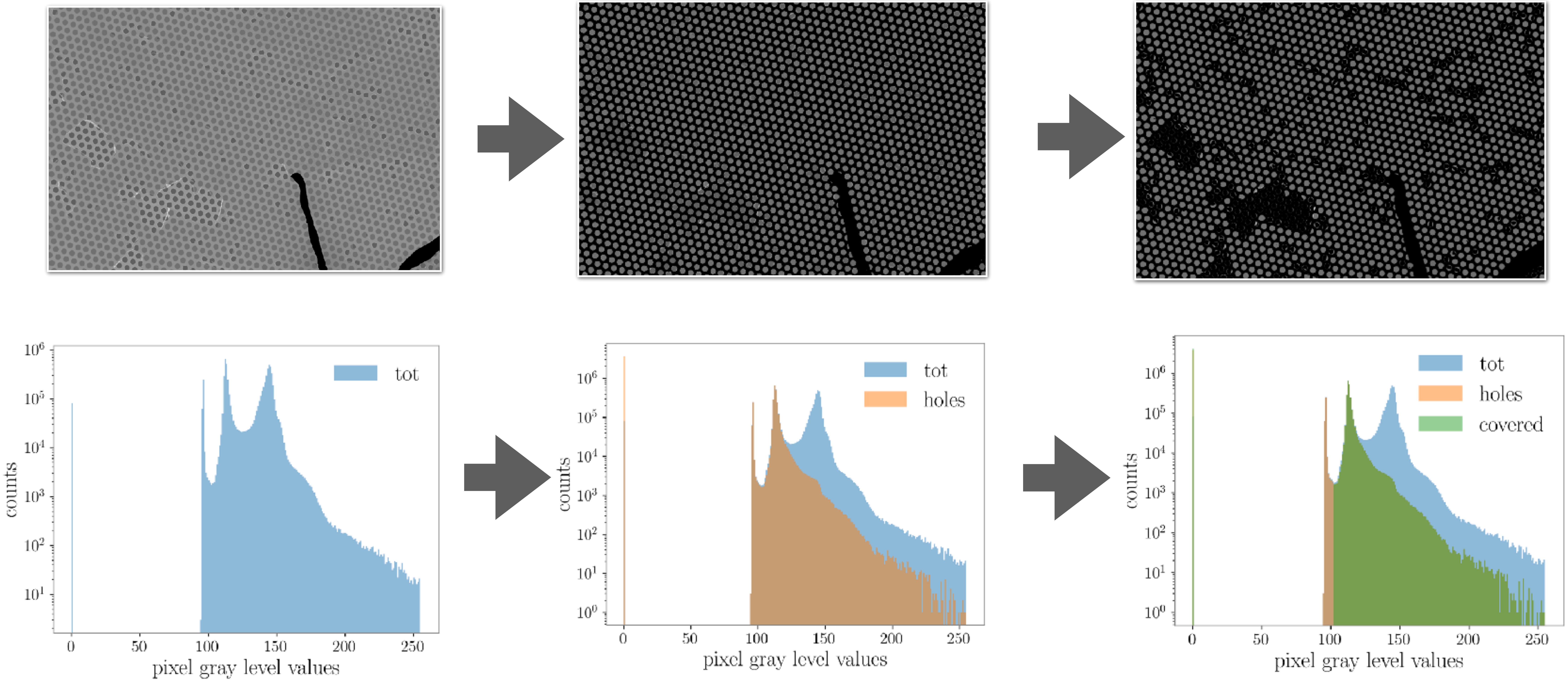
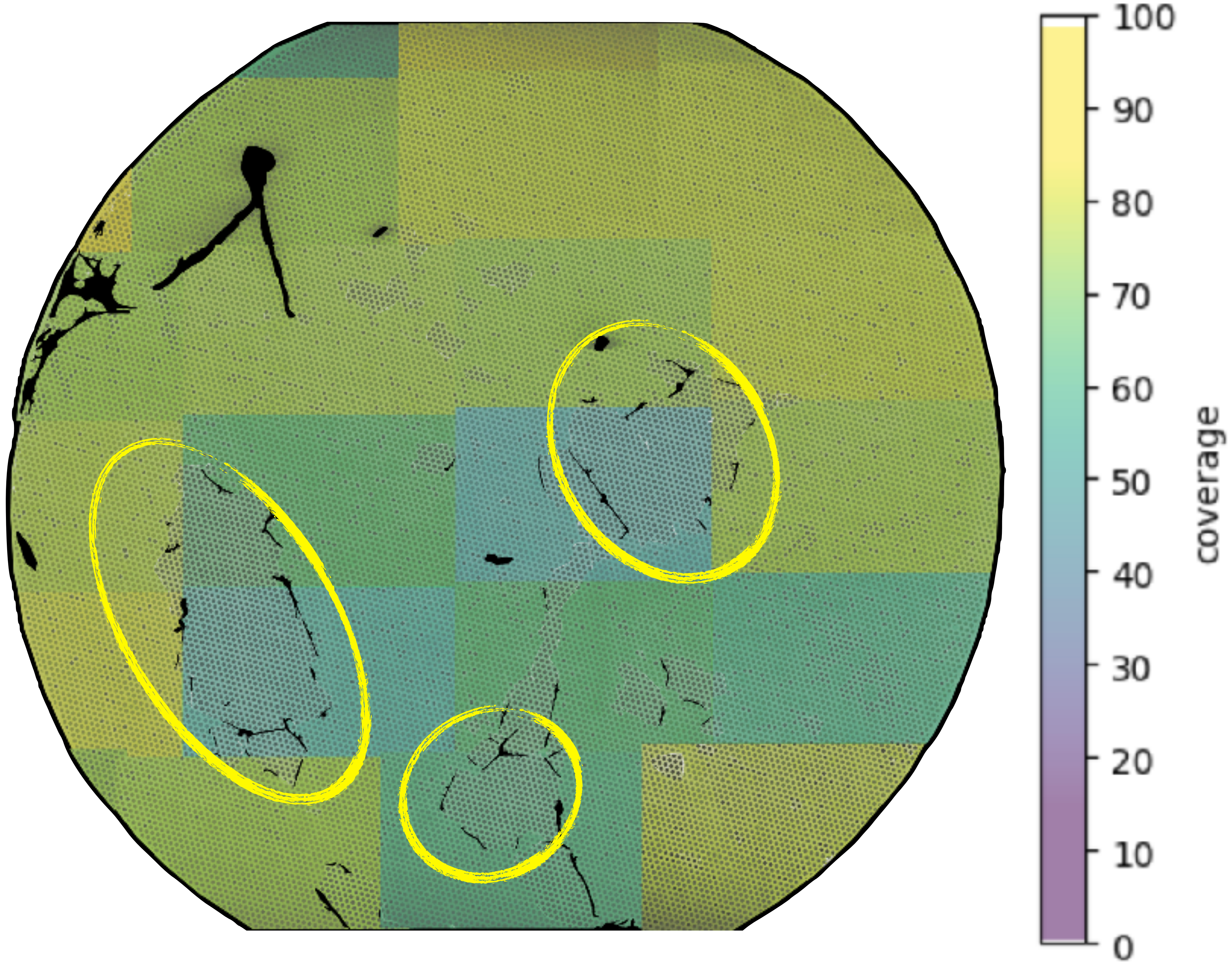
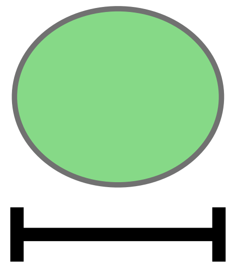


# SAMPLE VISUALIZATIONS

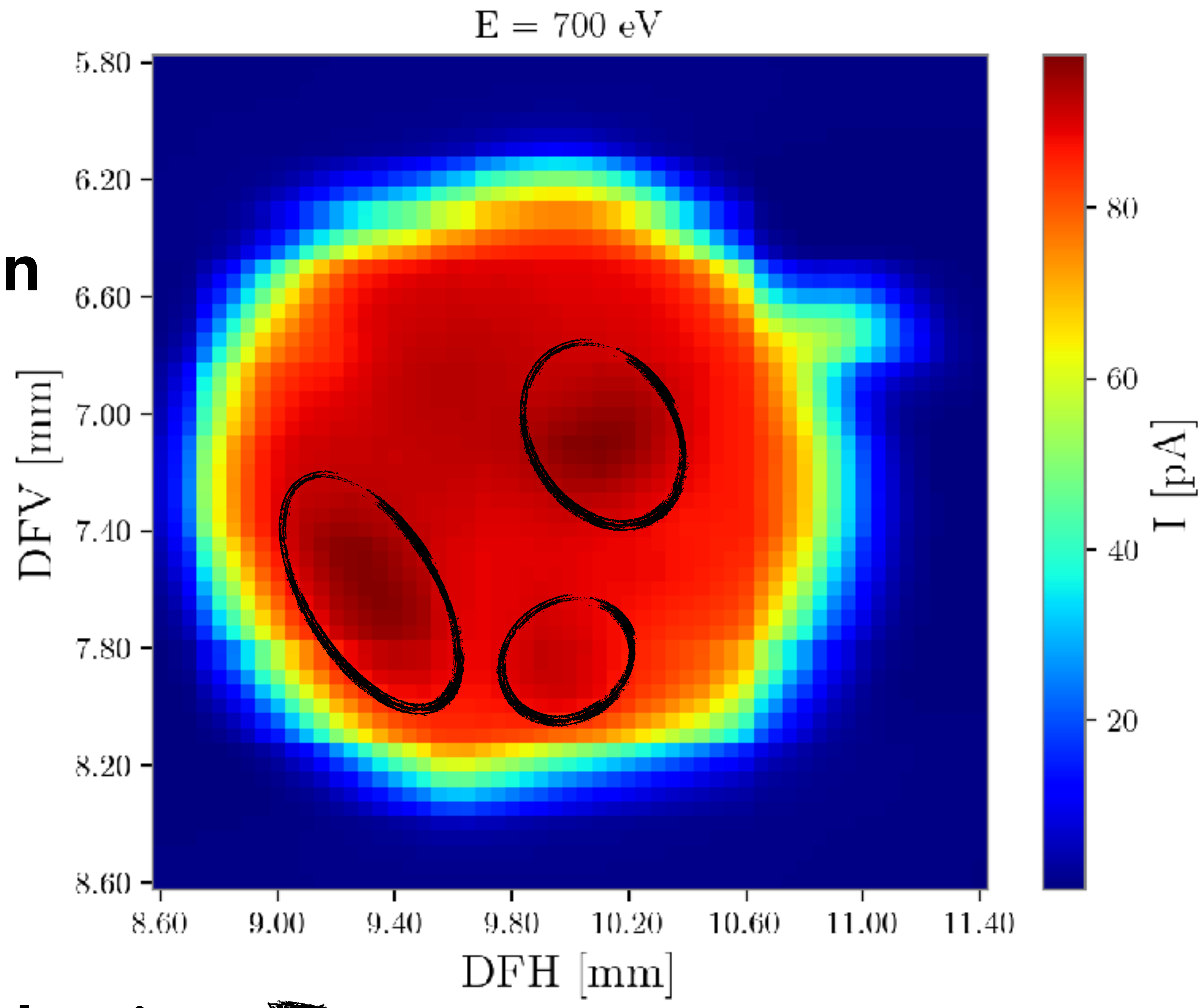
- **SEM:**




- **Transmission map with our apparatus:**

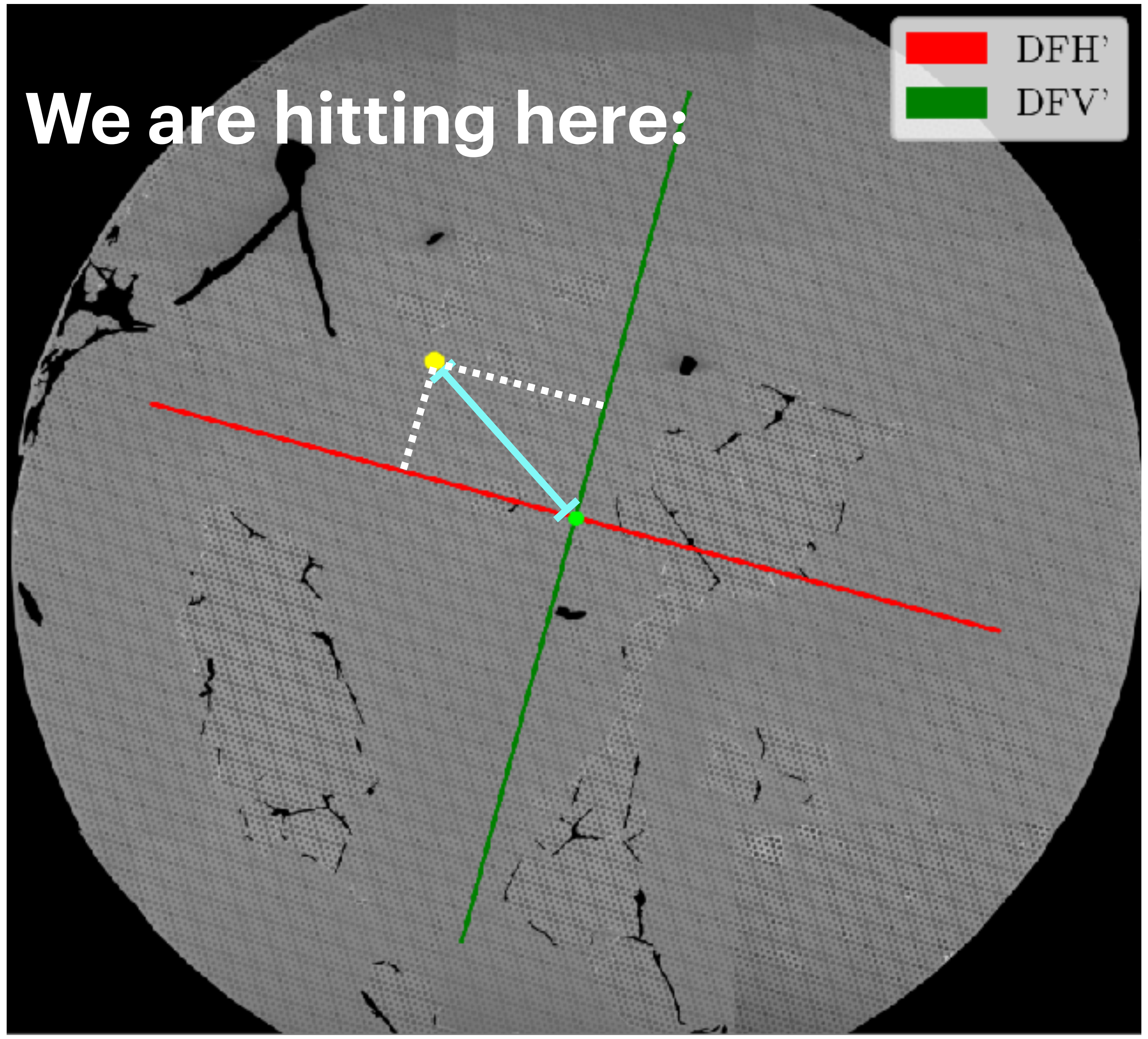


**= electron spot**




**Gun DeFlections**

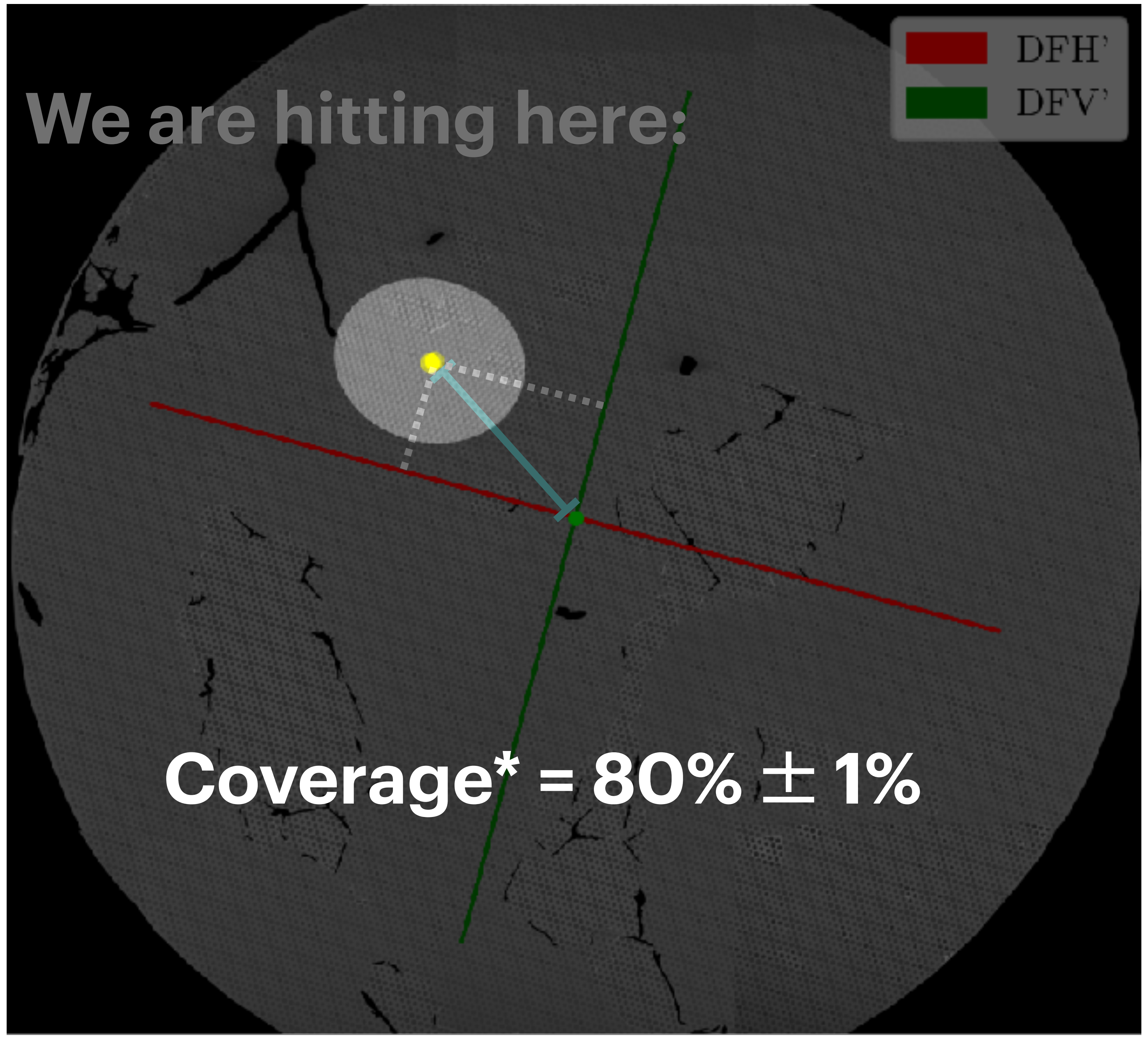
# TRANSMISSION "*AS PREPARED*"




$$T_G = \frac{1}{a}\left(\frac{I_{meas}}{I_0 T_{grid}} - (1 - a)\right)$$

***Sample as prepared* = graphene + PMMA**

# TRANSMISSION "*AS PREPARED*"

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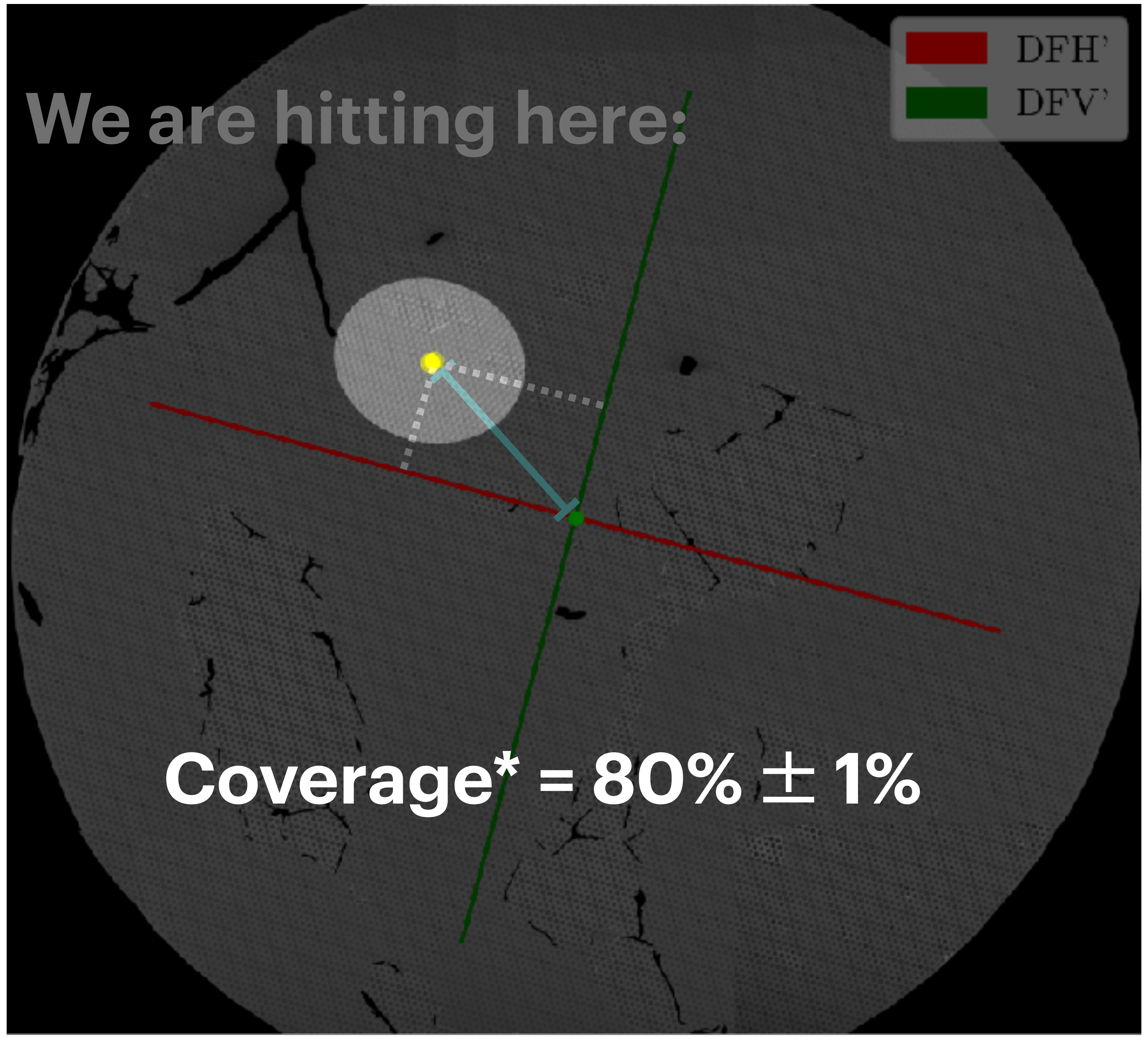
# TRANSMISSION "AS PREPARED"

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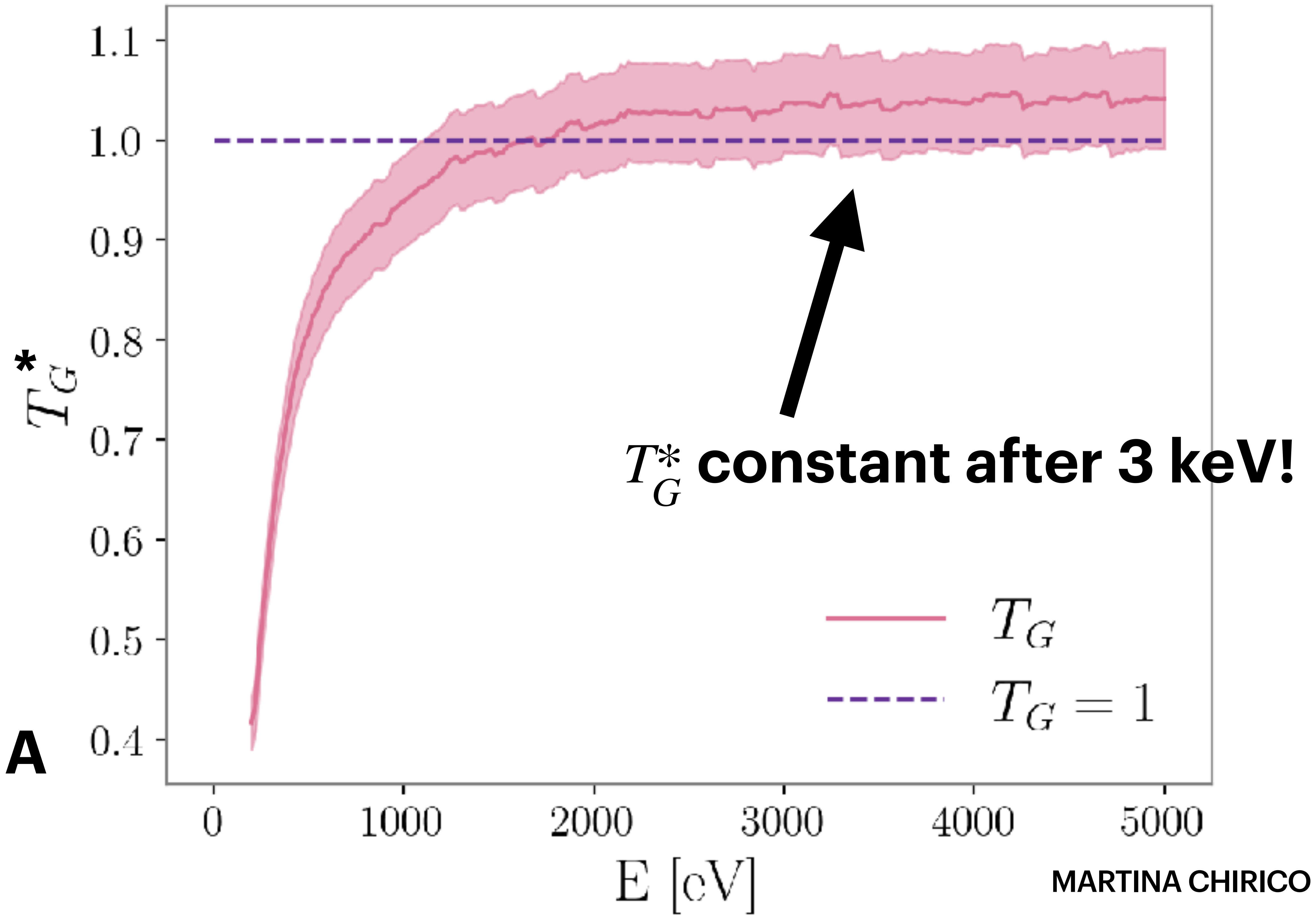


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# SAMPLE IS CLEANED THROUGH ANNEALING

- We want to preserve as much graphene as possible

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**1 hour @ 350°C**

↓ **XPS and EELS**

**1 hour @ 350°C**

↓

**XPS and EELS**

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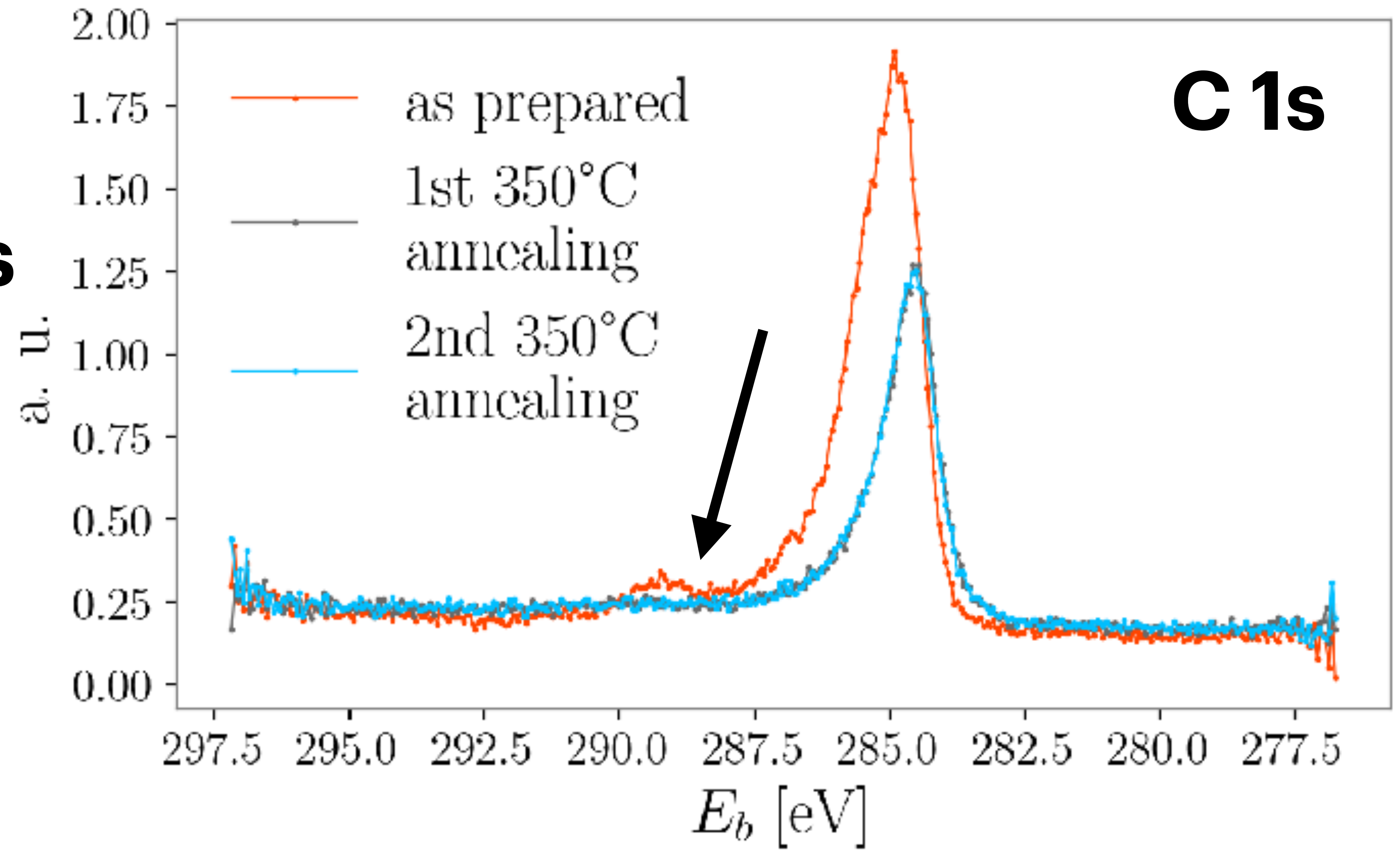

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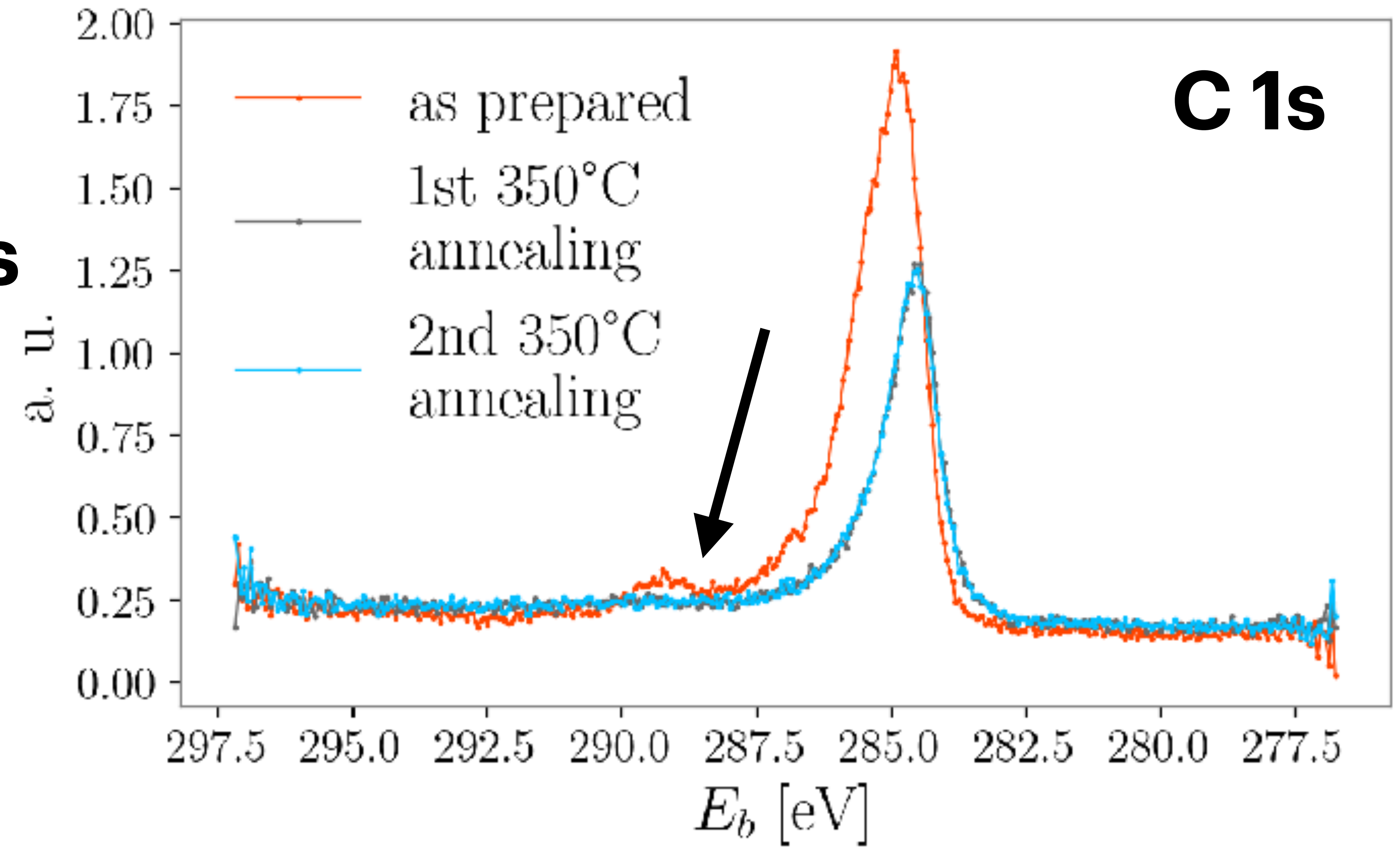


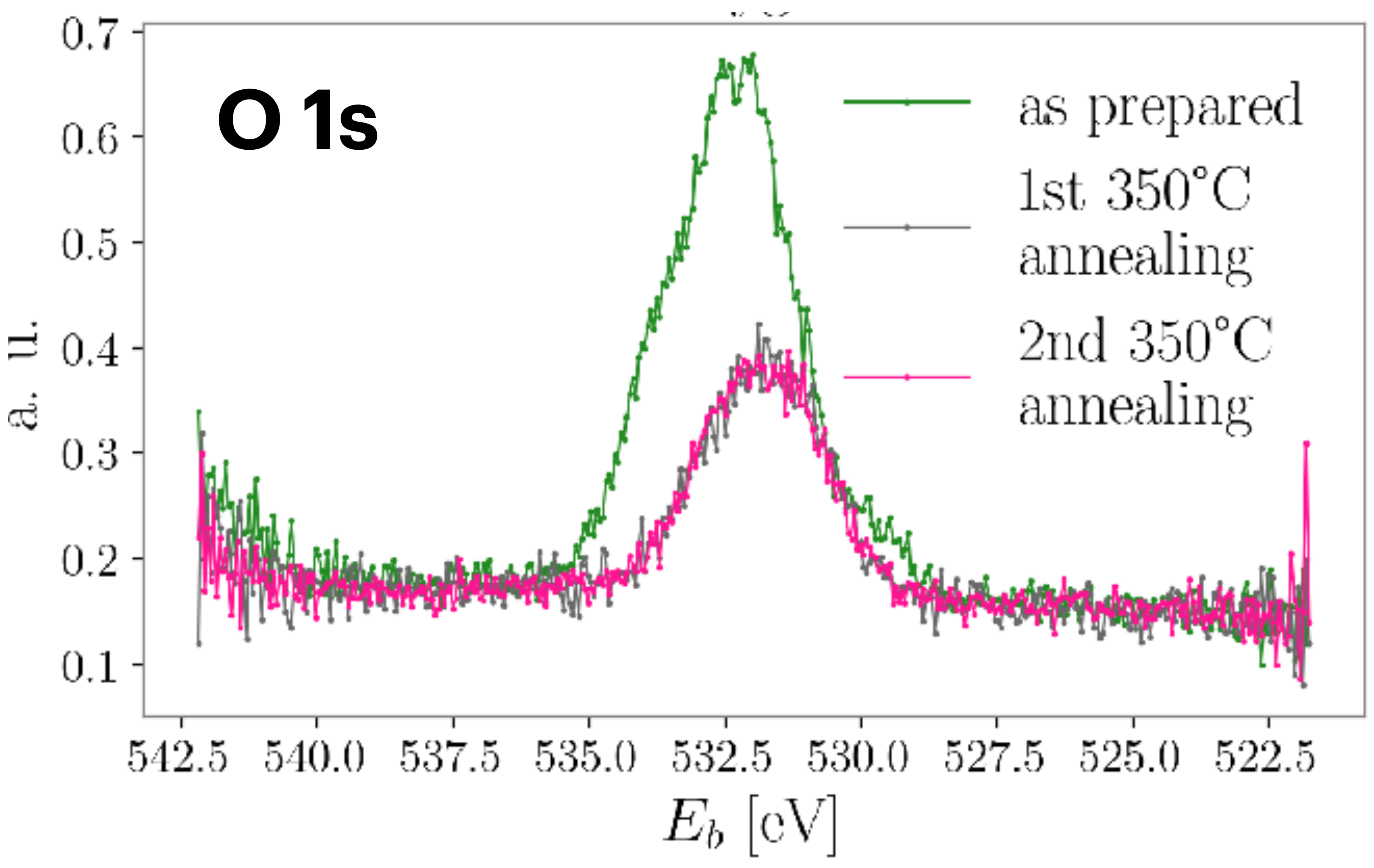

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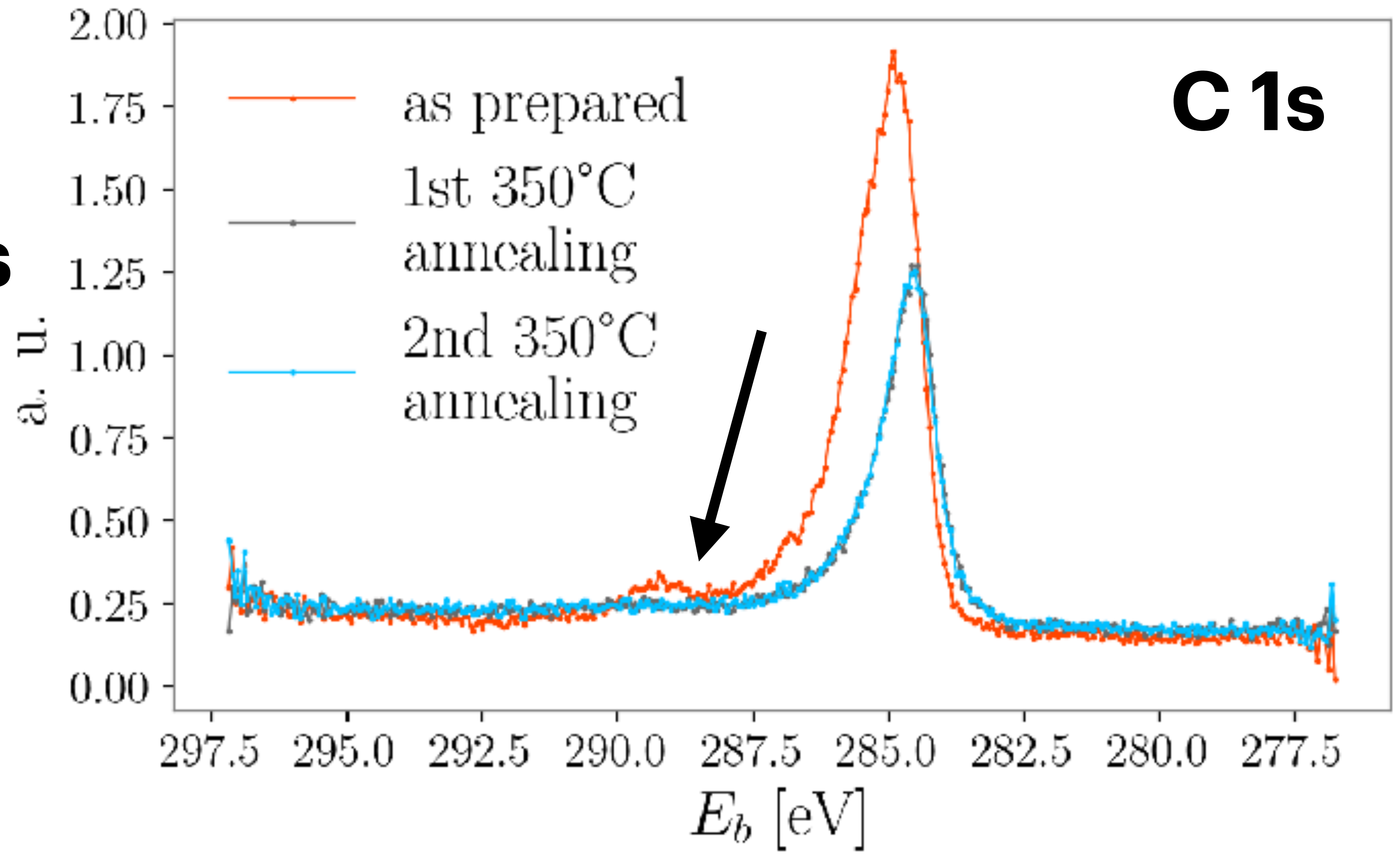
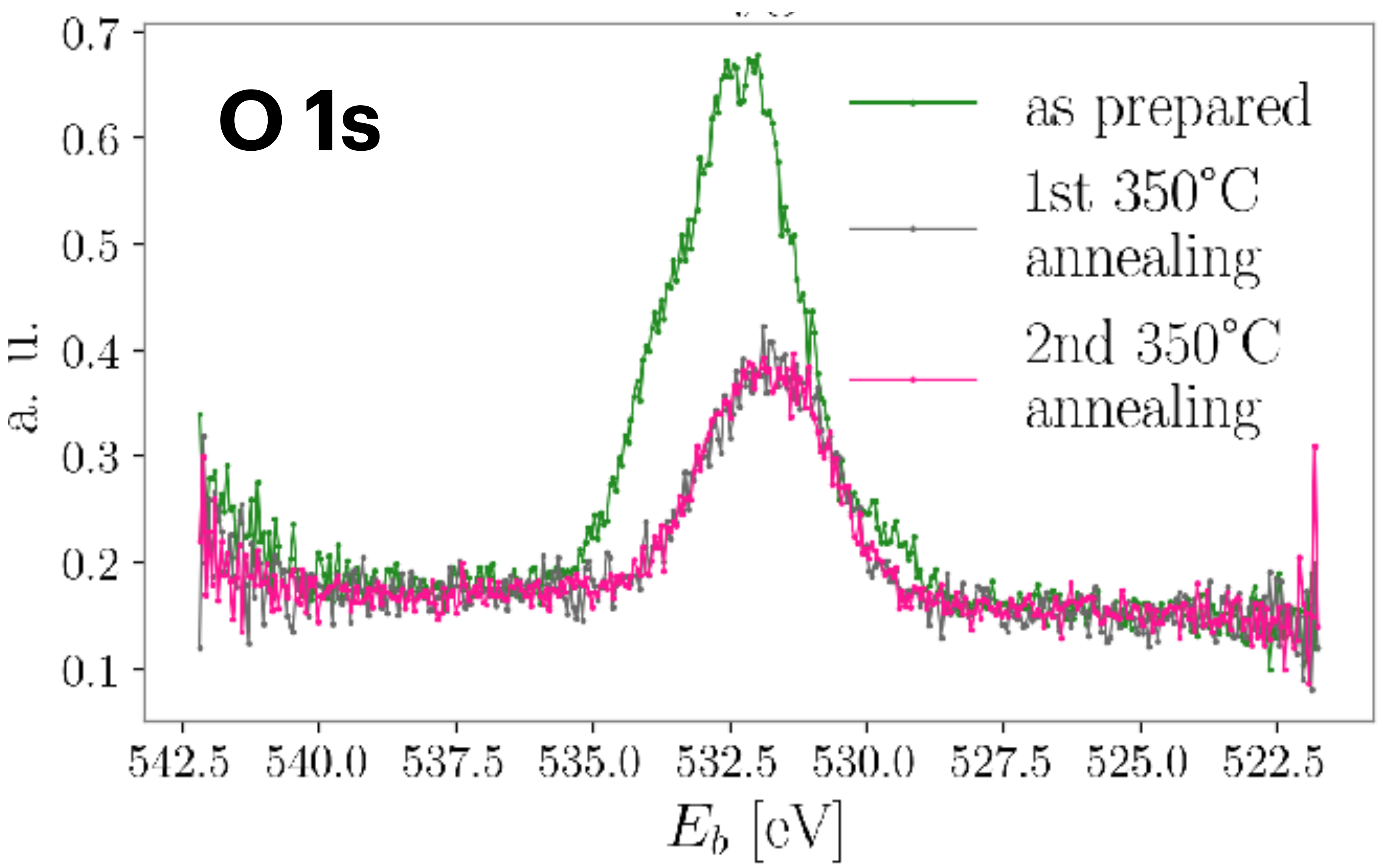
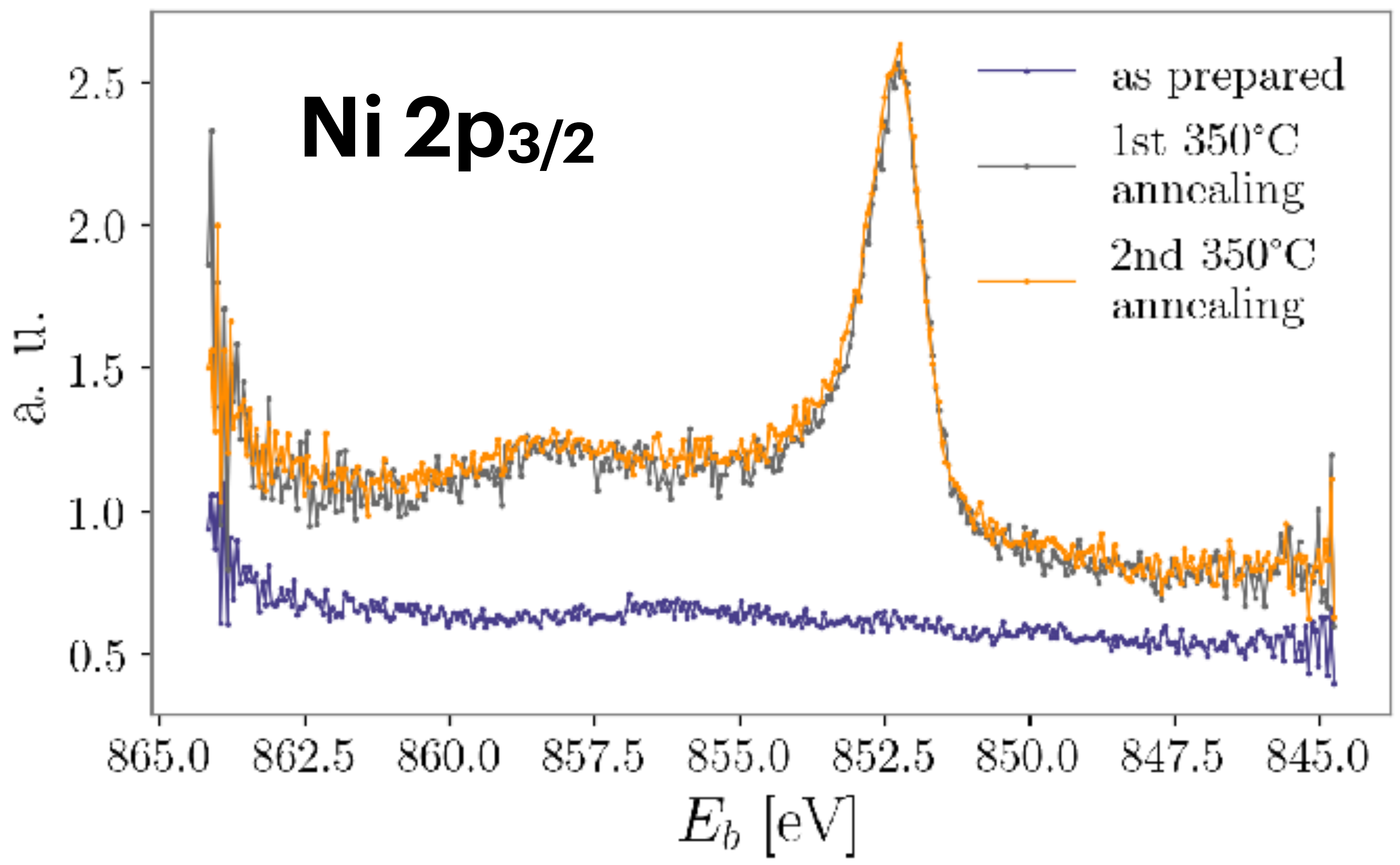
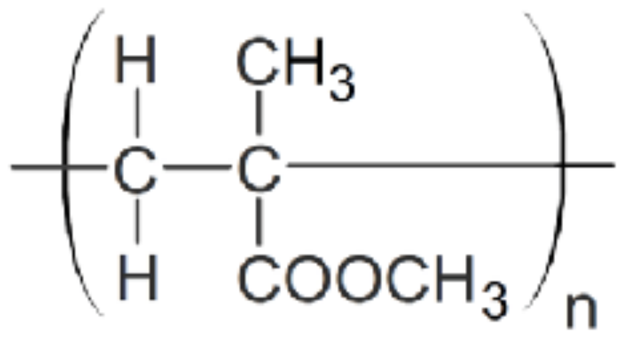


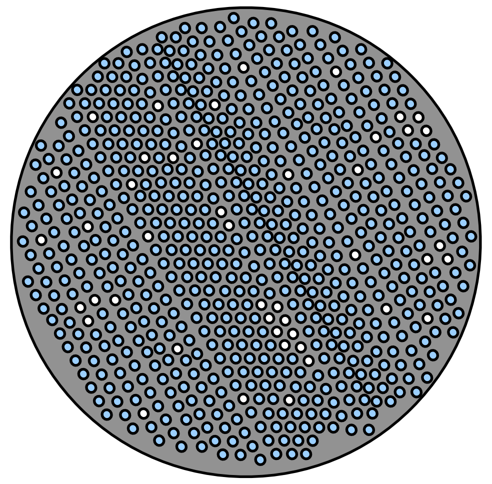




**PMMA:**



**Nickel grid:**



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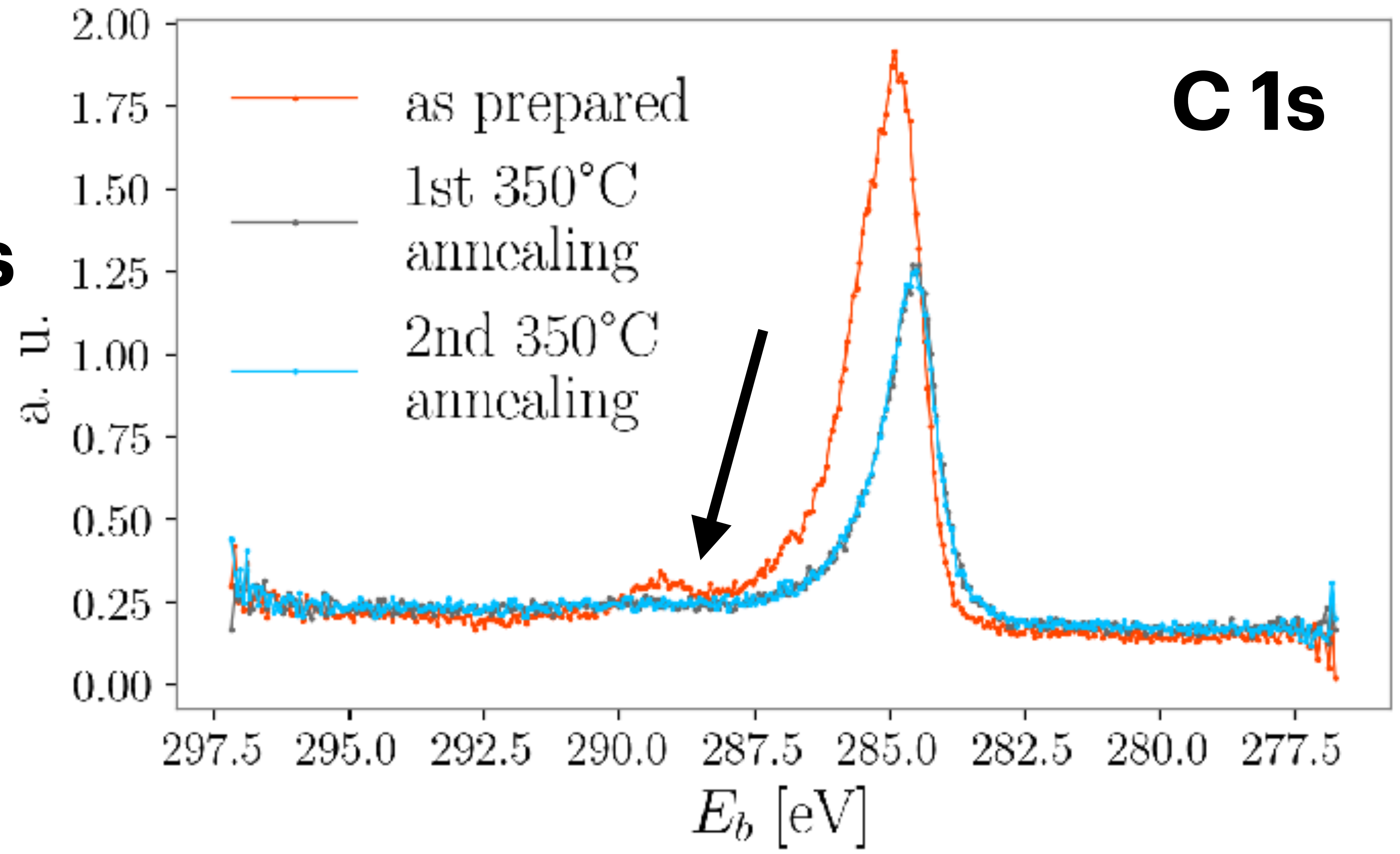
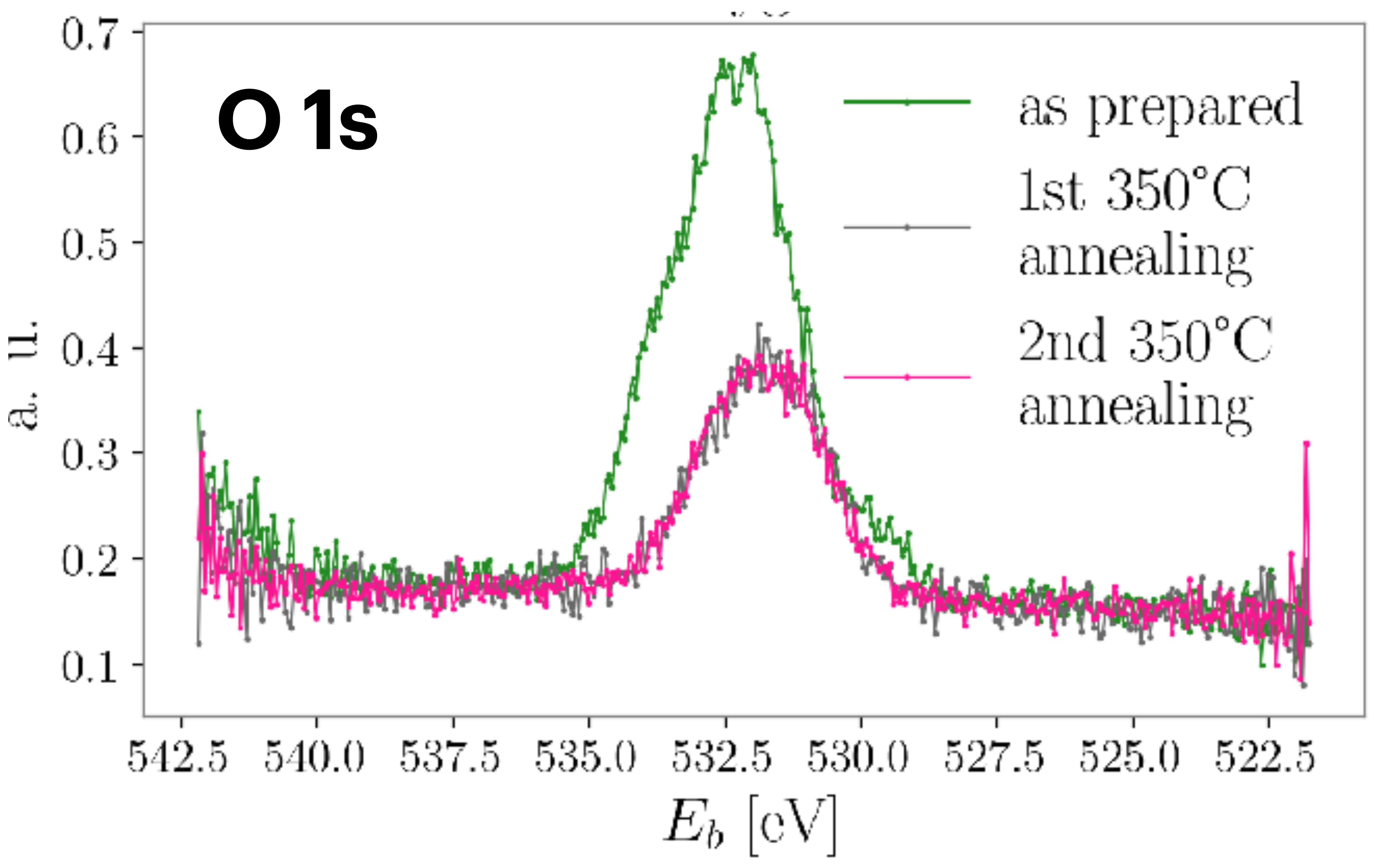
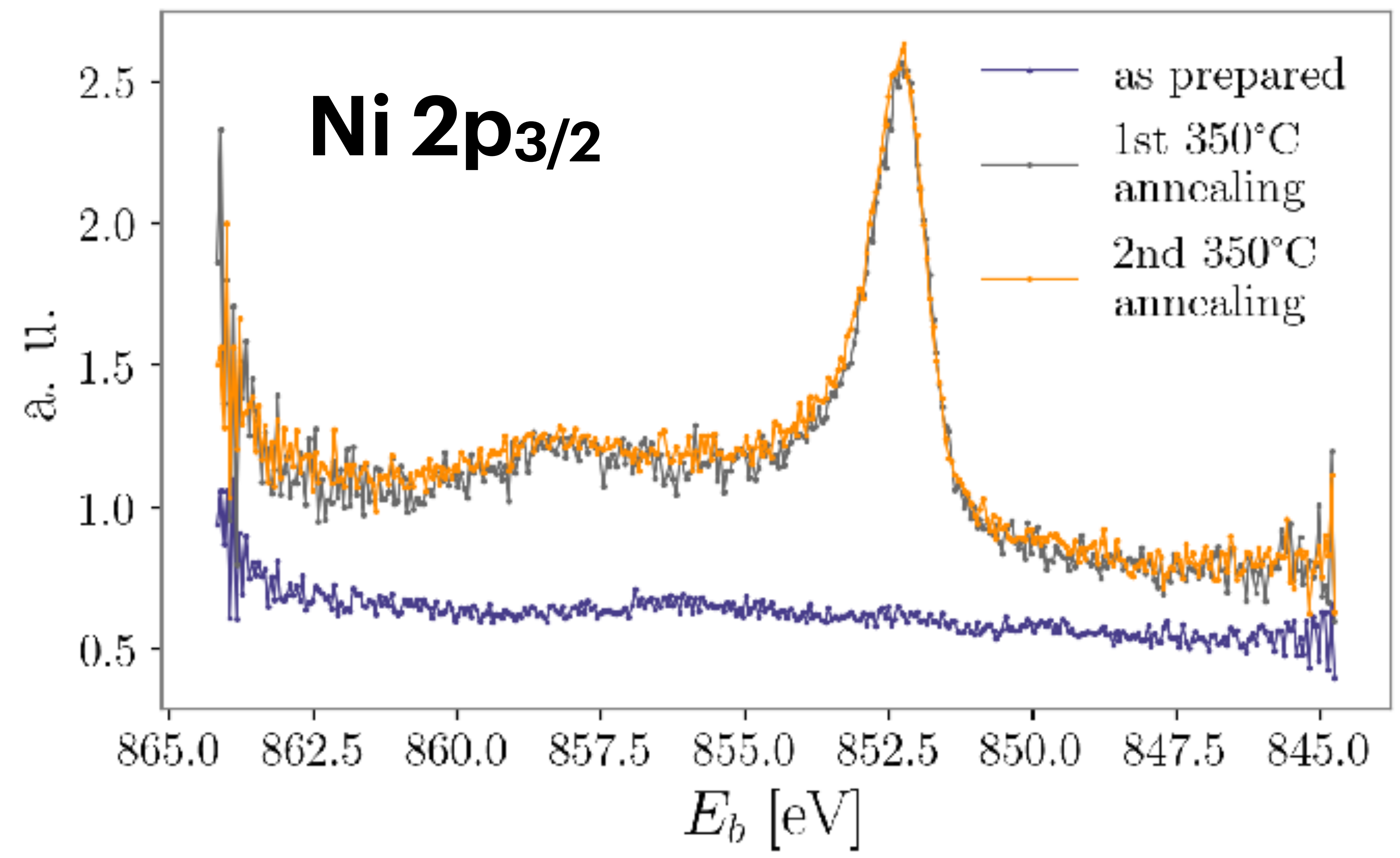
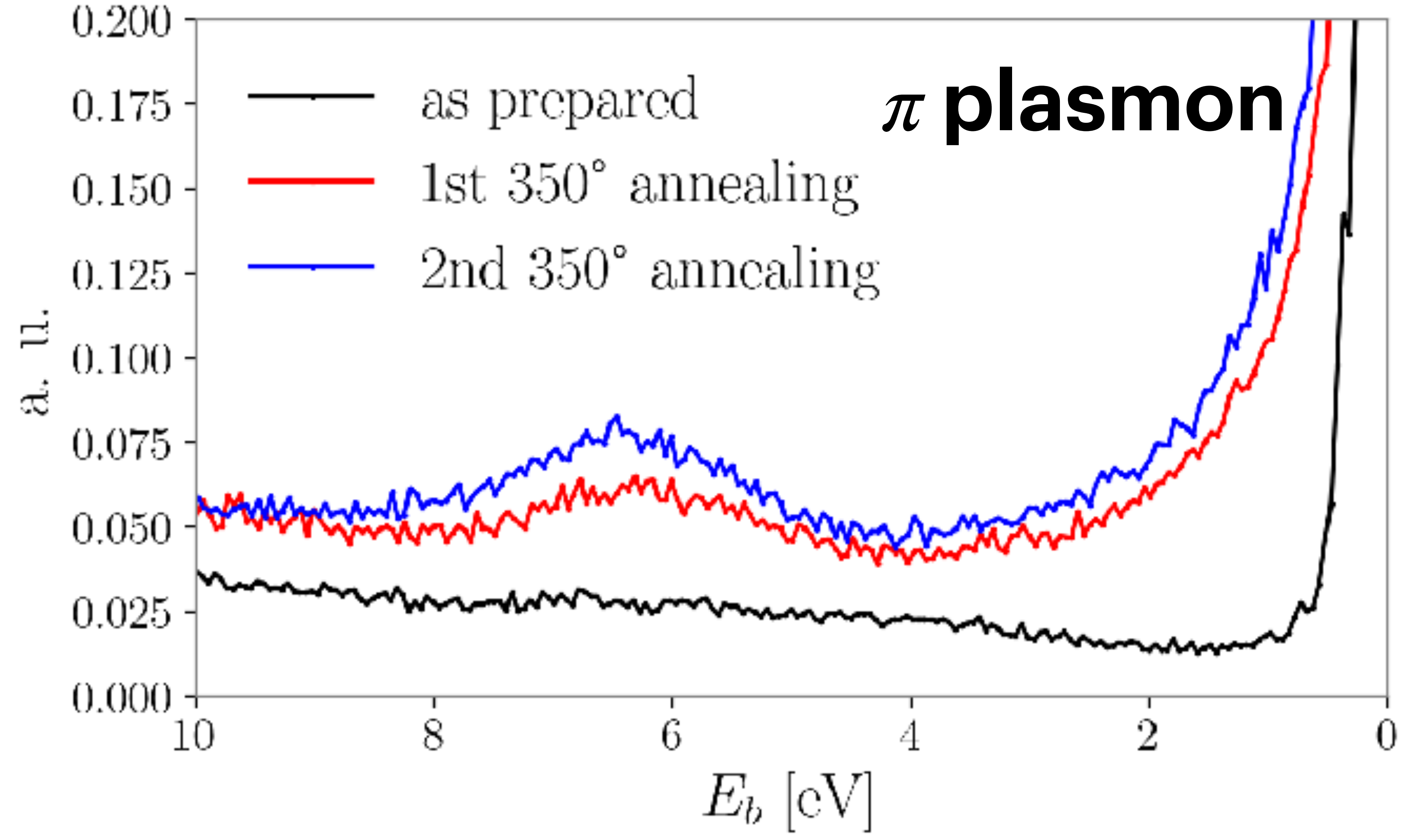


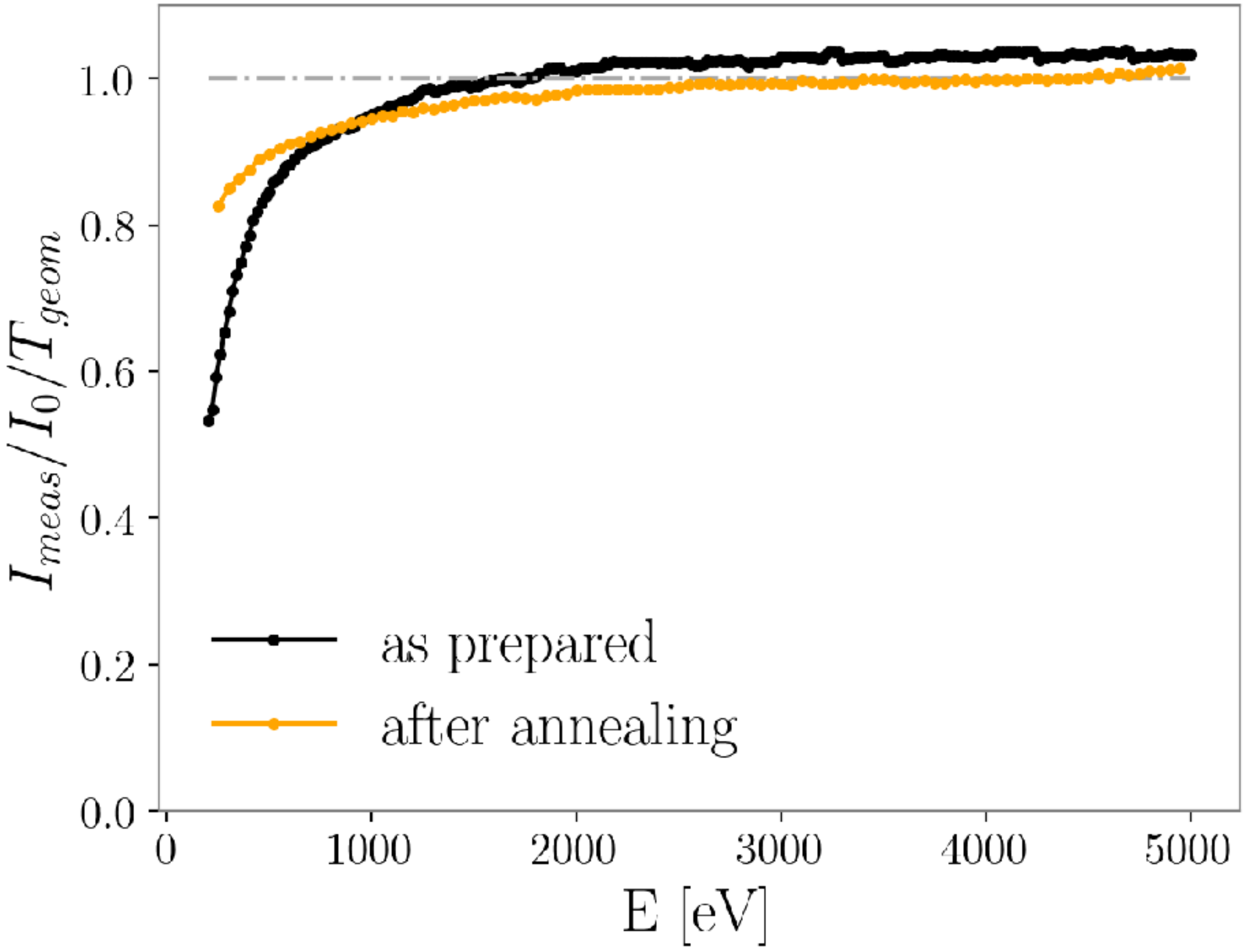



# TRANSMISSION AFTER ANNEALING

**WORK IN PROGRESS!**

**First considerations:**

- **Energy dependency: we still have graphene on the sample!**
- **Transmission improves after annealing**
- **Transparency after 3 keV!**



# TO CONCLUDE:

- The measurement of the transmission of electrons through graphene between 200 eV and 5000 eV has been performed on the as prepared sample
- Graphene monolayer as prepared is practically transparent to electrons above 3 keV
- The sample has been cleaned with double annealing at 350° C
- Transmission in the same range through the clean sample is reveals higher transmission at lower energies and is still transparent to electrons
- SEM investigation scheduled for next week

*Thank you for your attention!*

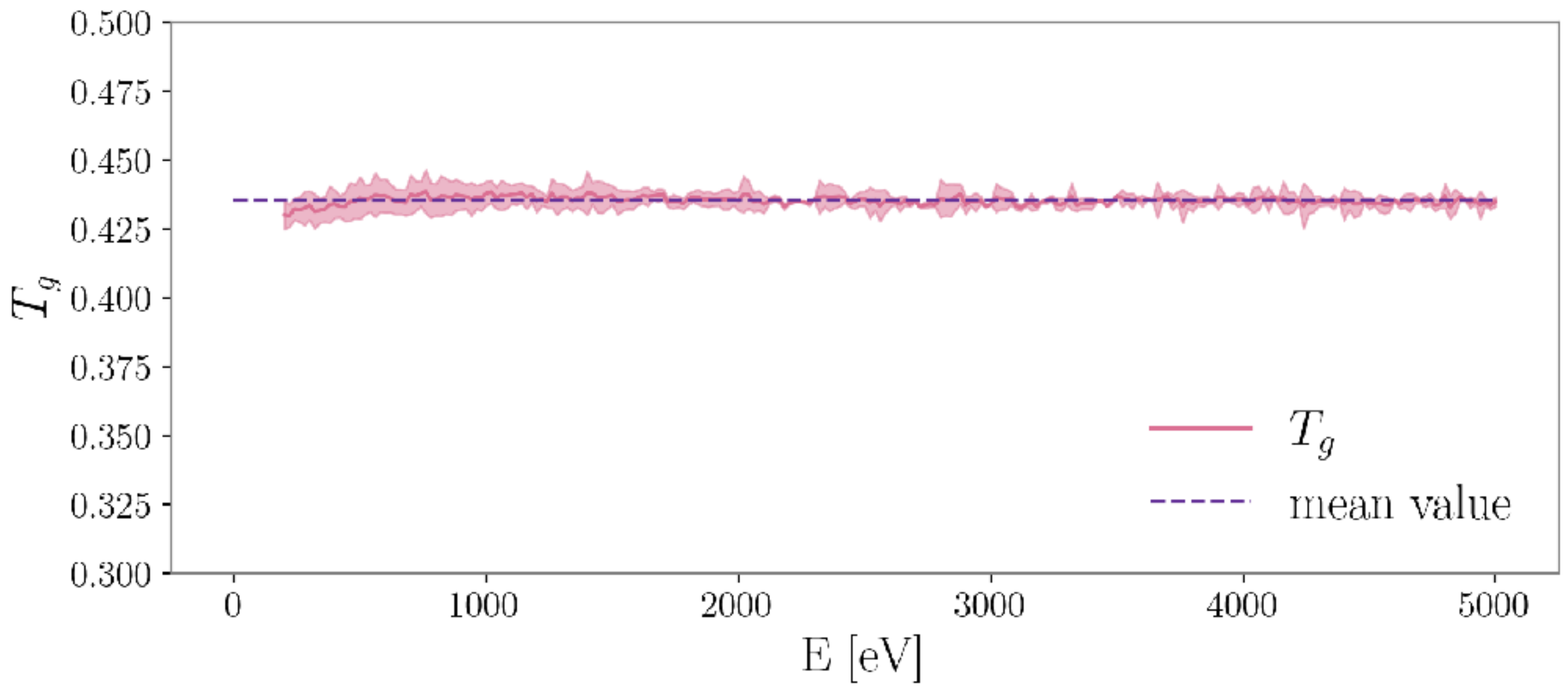
# BACKUP

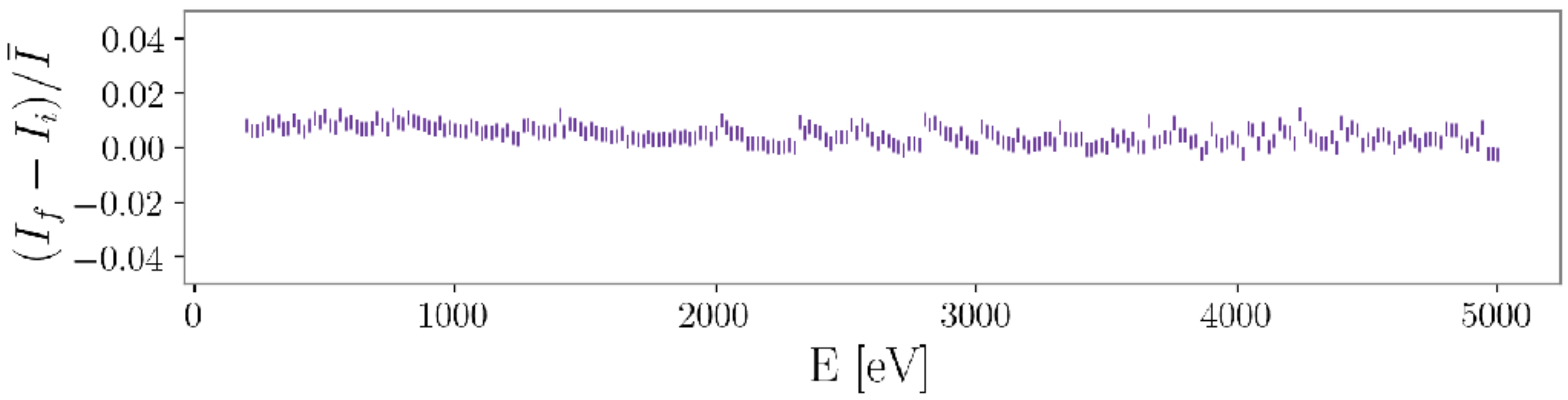
# CHECKS WITH AN EMPTY GRID

- Geometric transmission of an empty grid does not depend on in energy

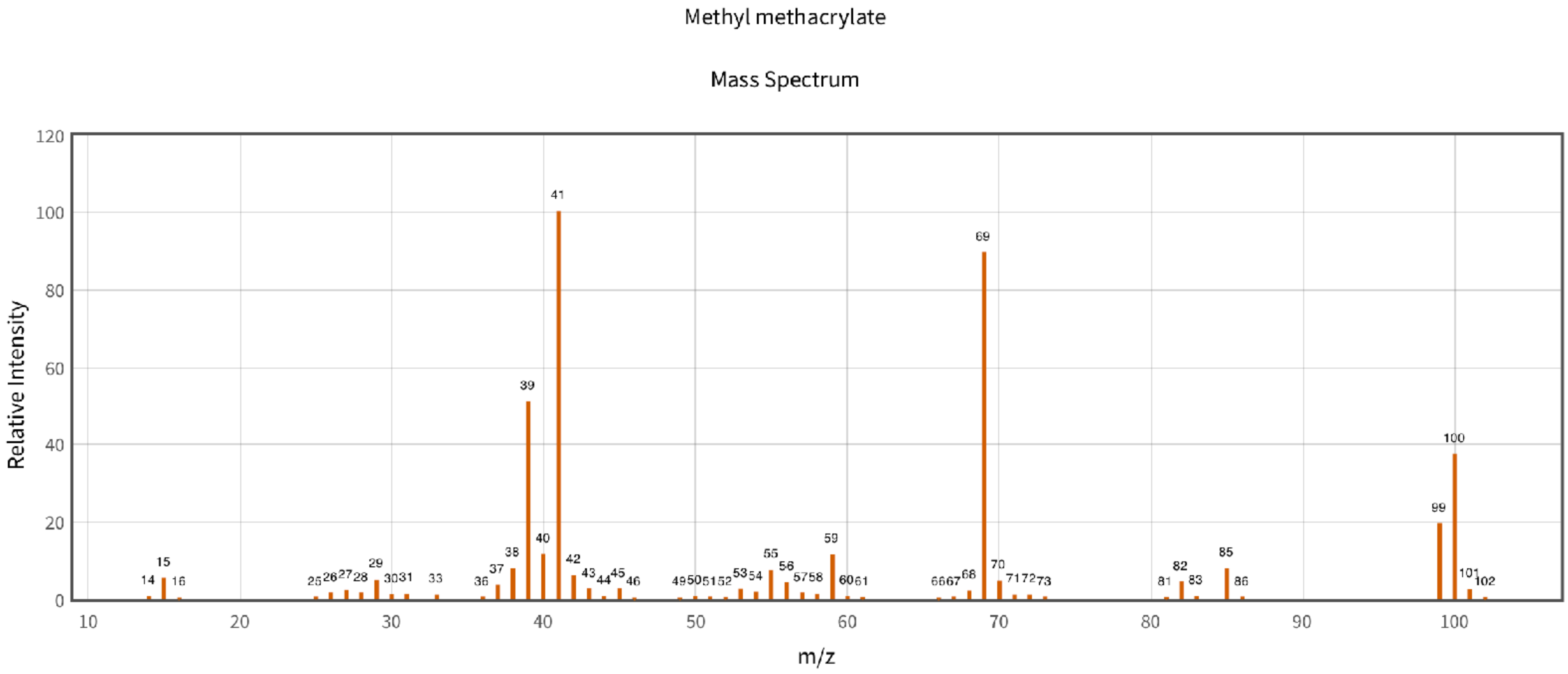
We can use SEM images of the grid to estimate it

- Current fluctuations during a single measurement below 1%

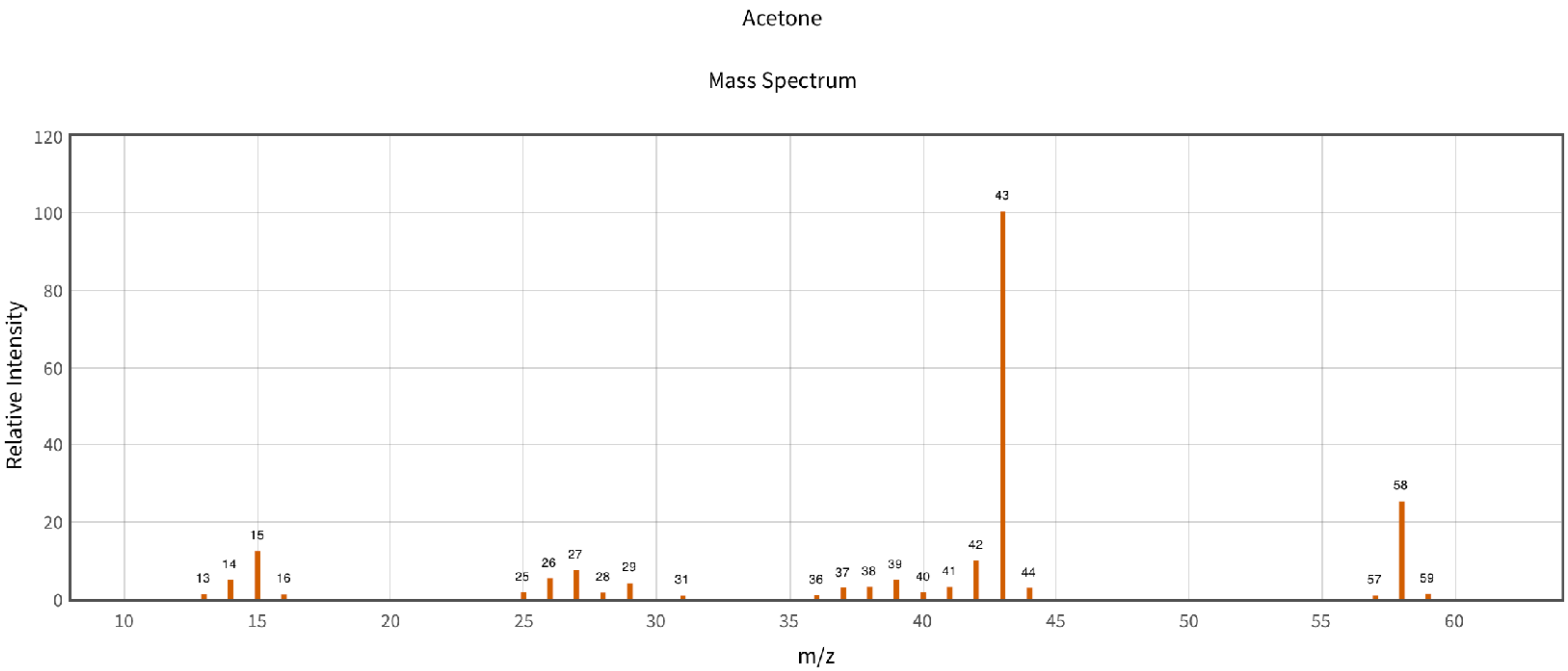


# PMMA FRAGMENTATION PATTERN

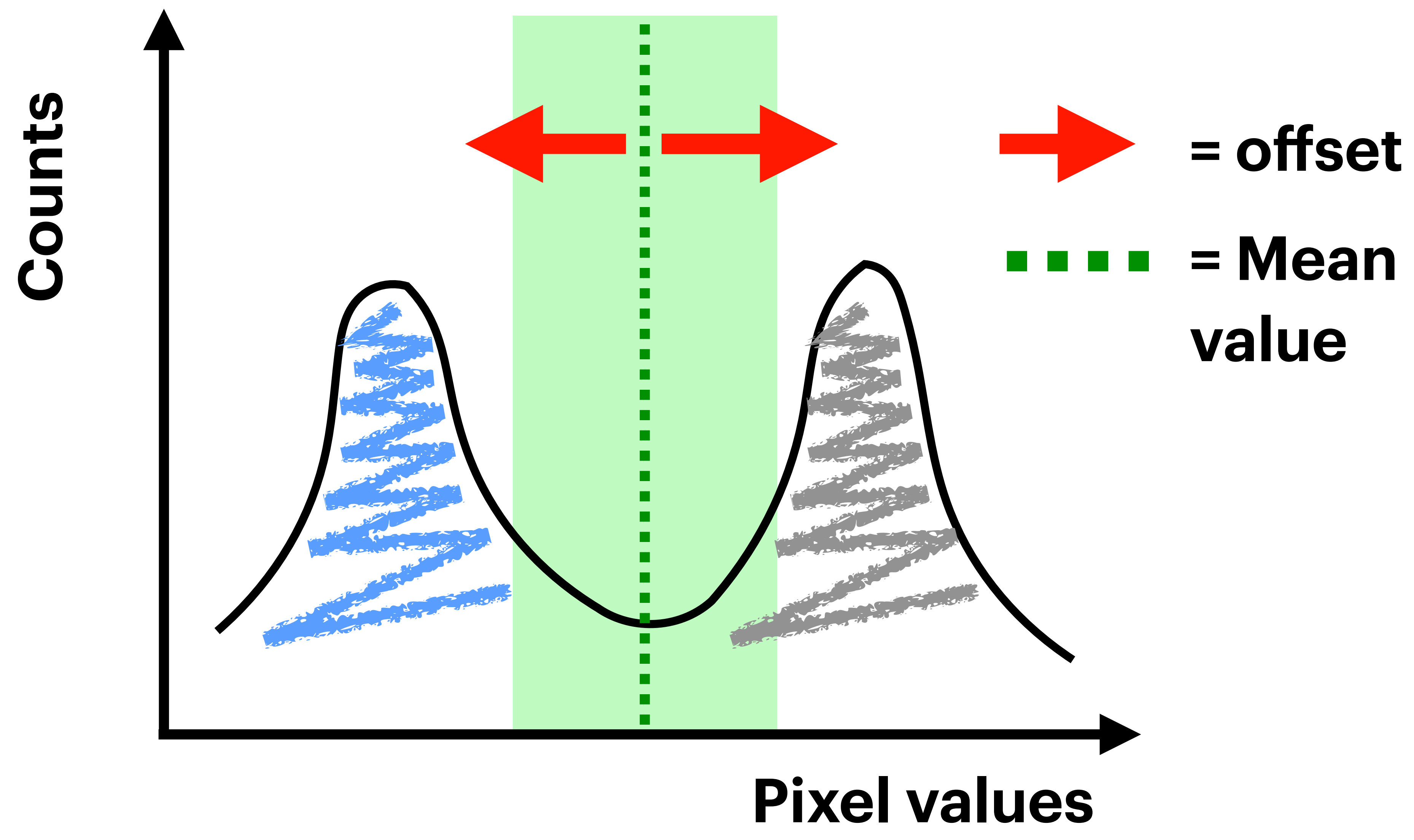


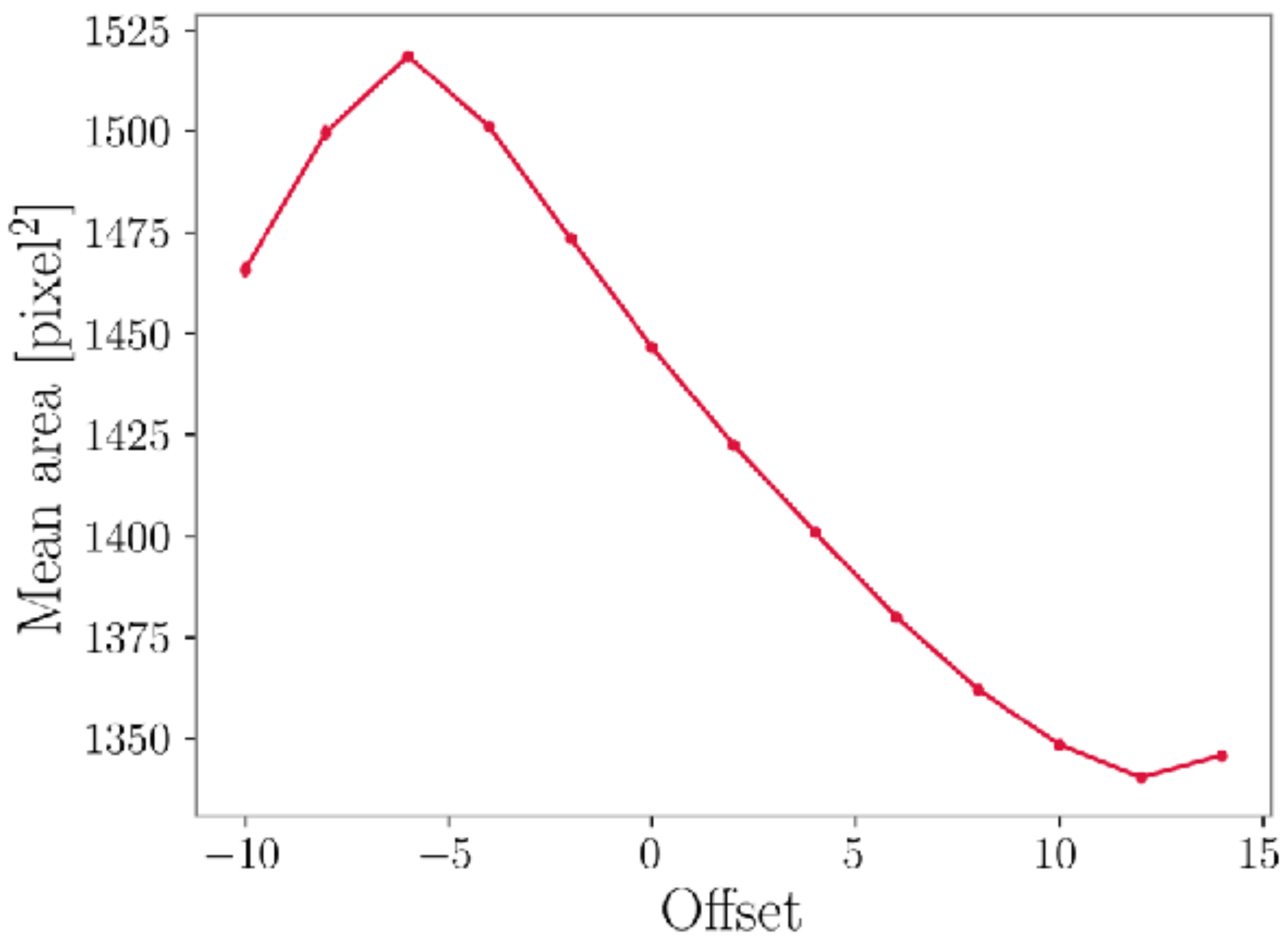
# ACETONE FRAGMENTATION PATTERN



# MEASURE HOLE AREAS

- Uncertainty in the definition of the border of a hole. With this method covered and uncovered holes appear to have different areas
- Change threshold by adding an offset to the mean value and see how the mean area changes

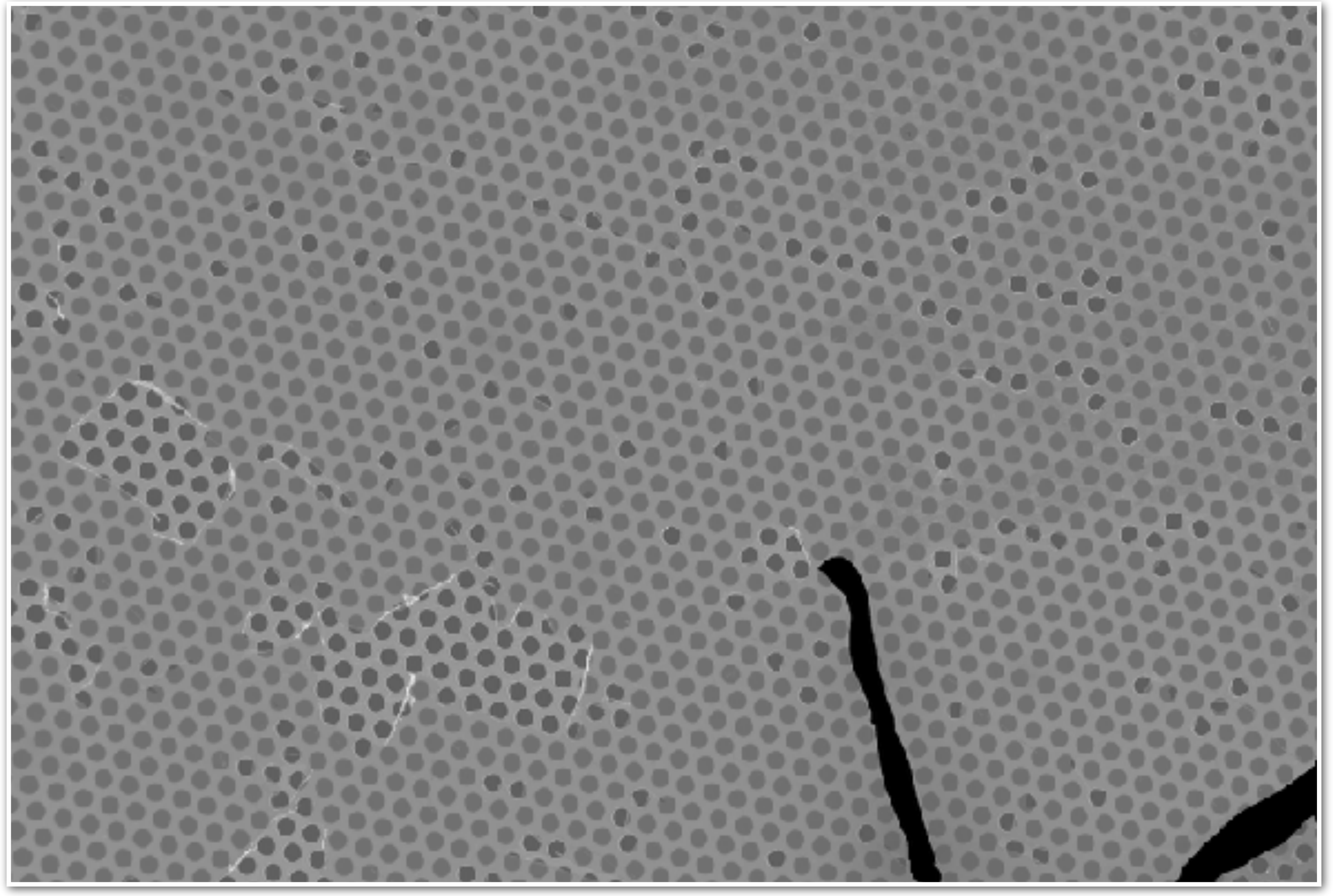



- New SEM analysis will help us to investigate this aspect

# CREATE A MASK TO EXTRACT HOLES

- **Cut labels and corrupted parts**



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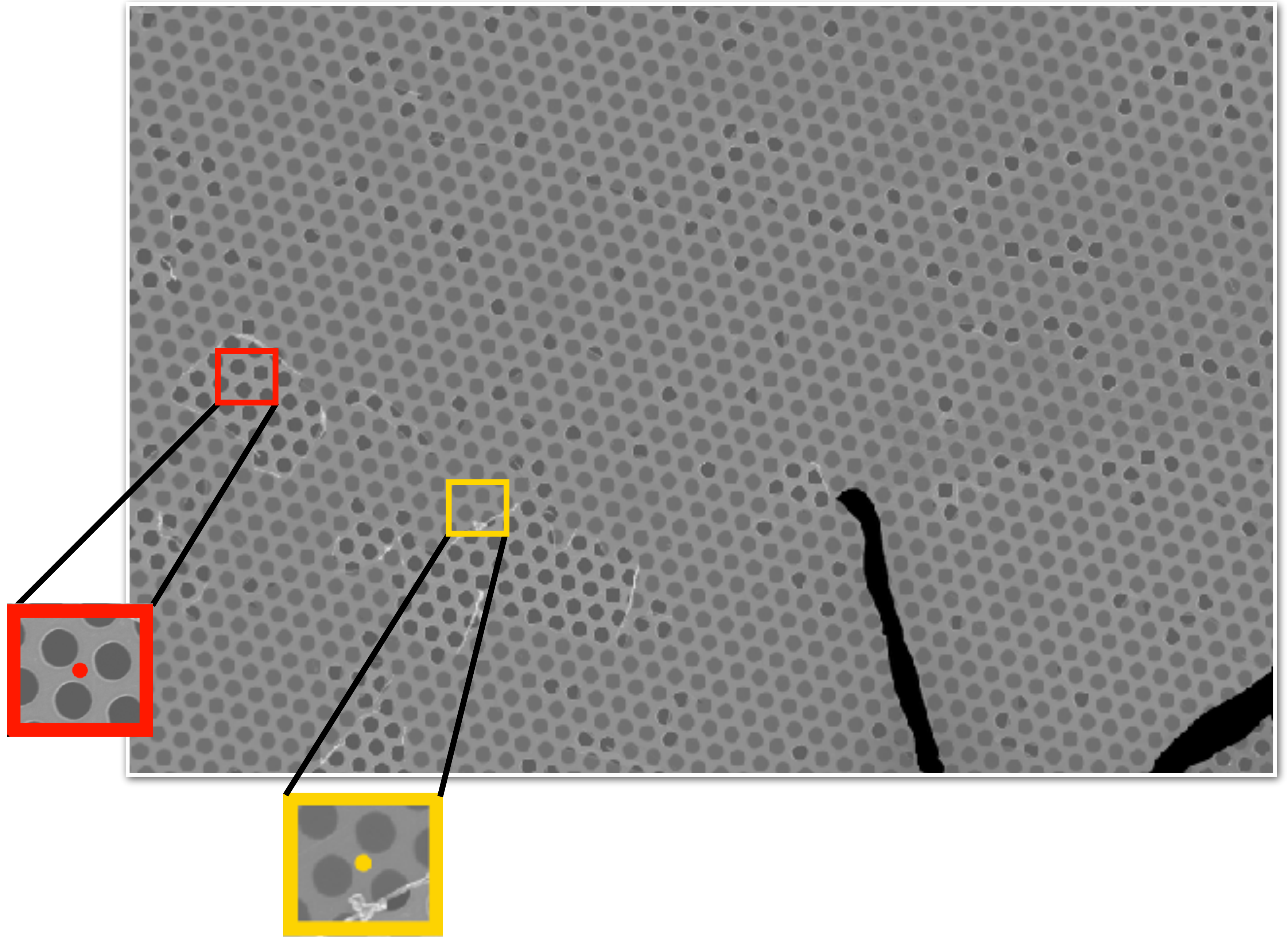

- **Choose threshold pixel by pixel (*Adaptive threshold*): mean inside the box + offset**

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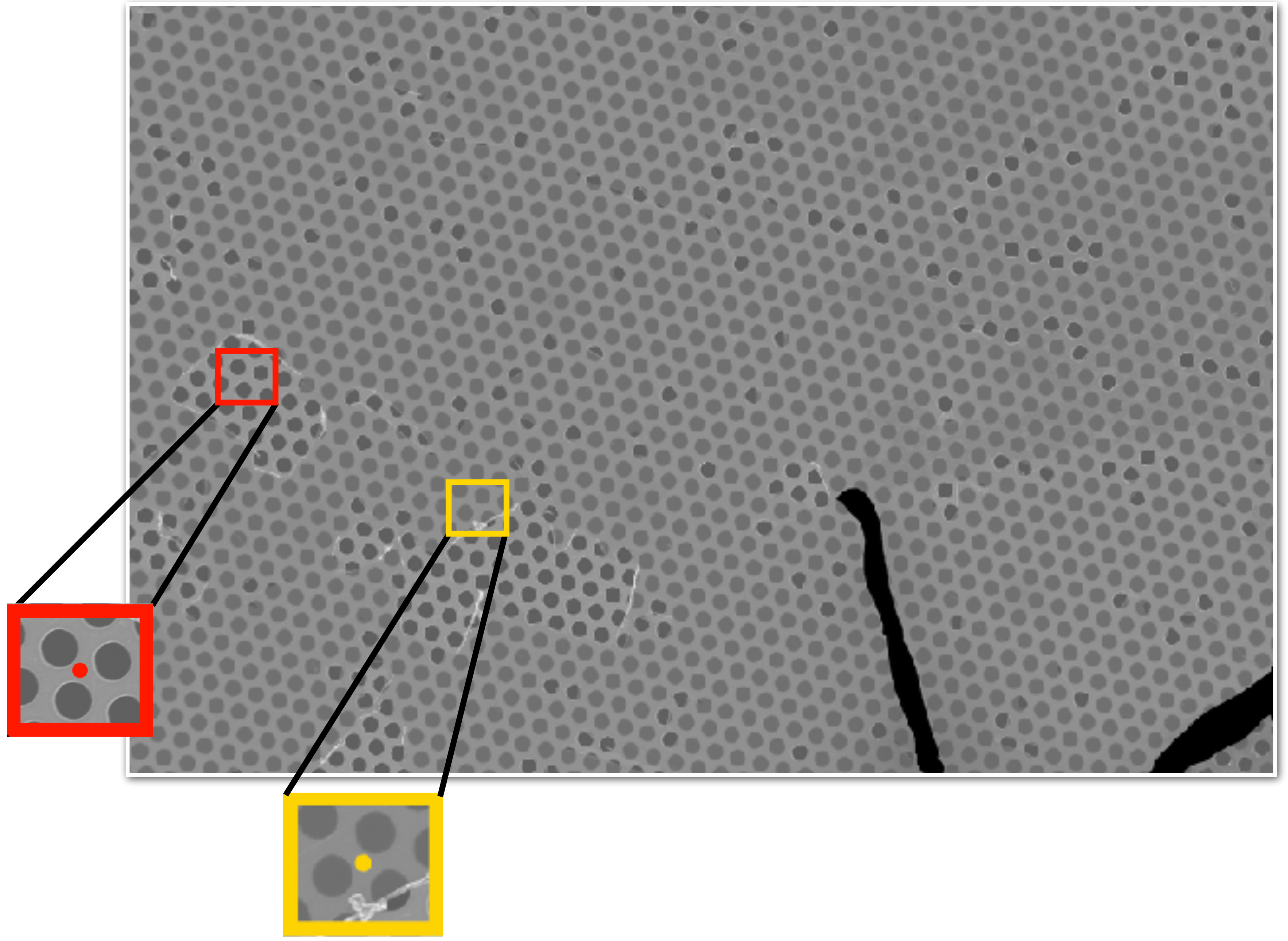

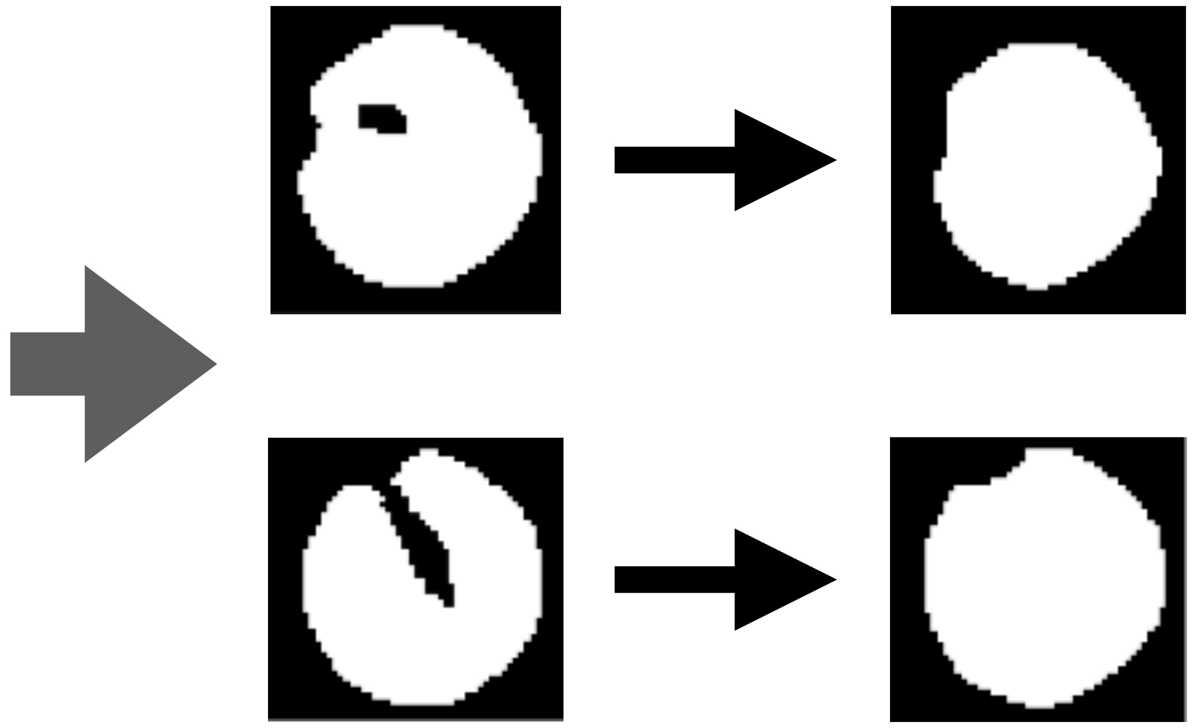

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- **"Compactify" objects (*Morphological closure)***

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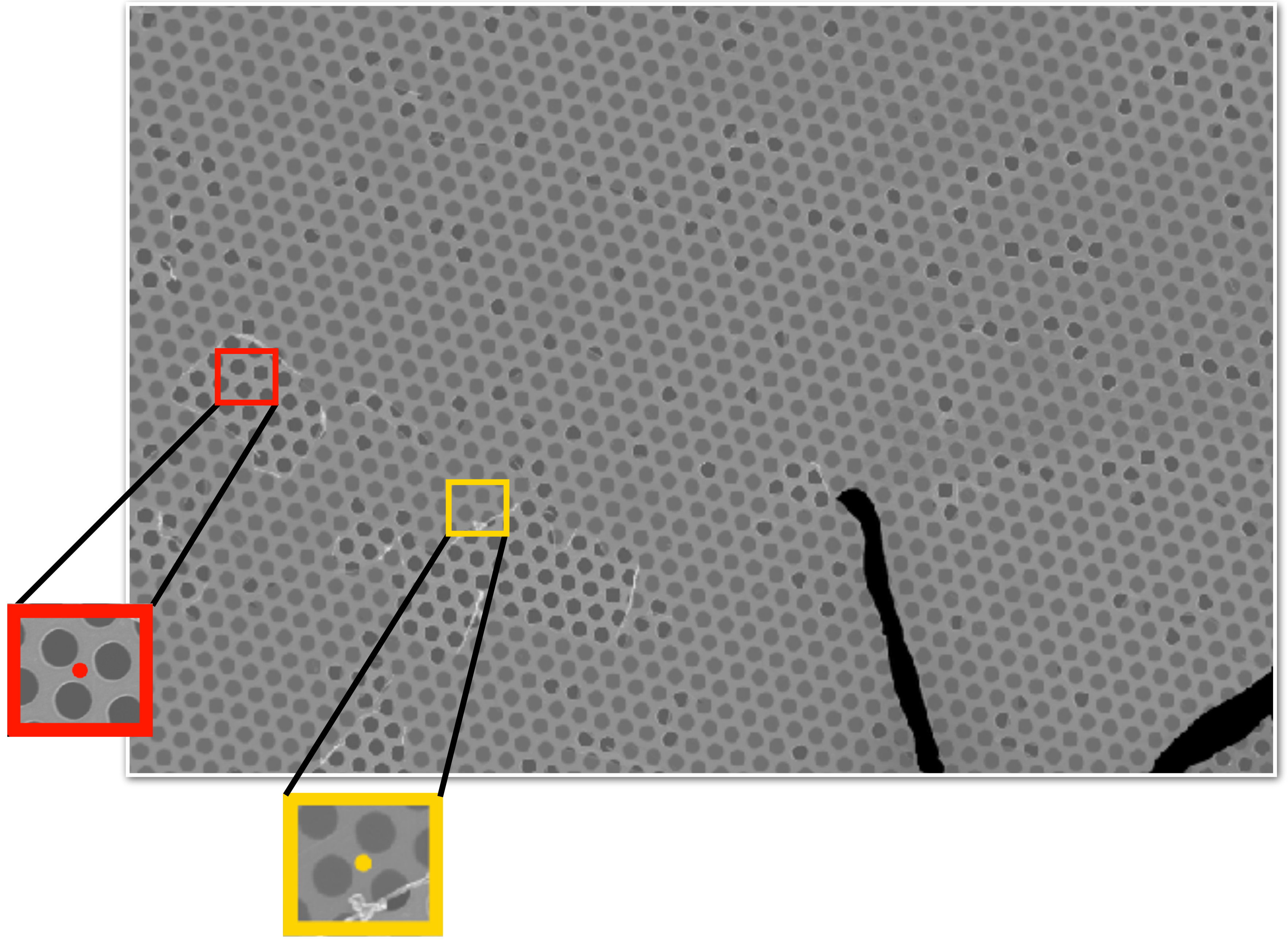

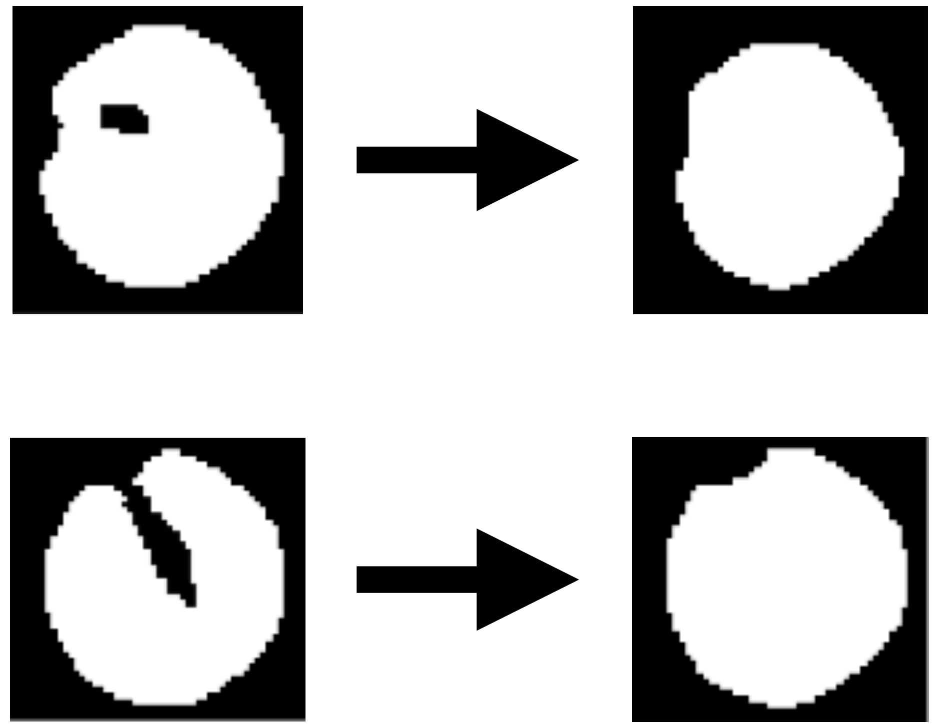

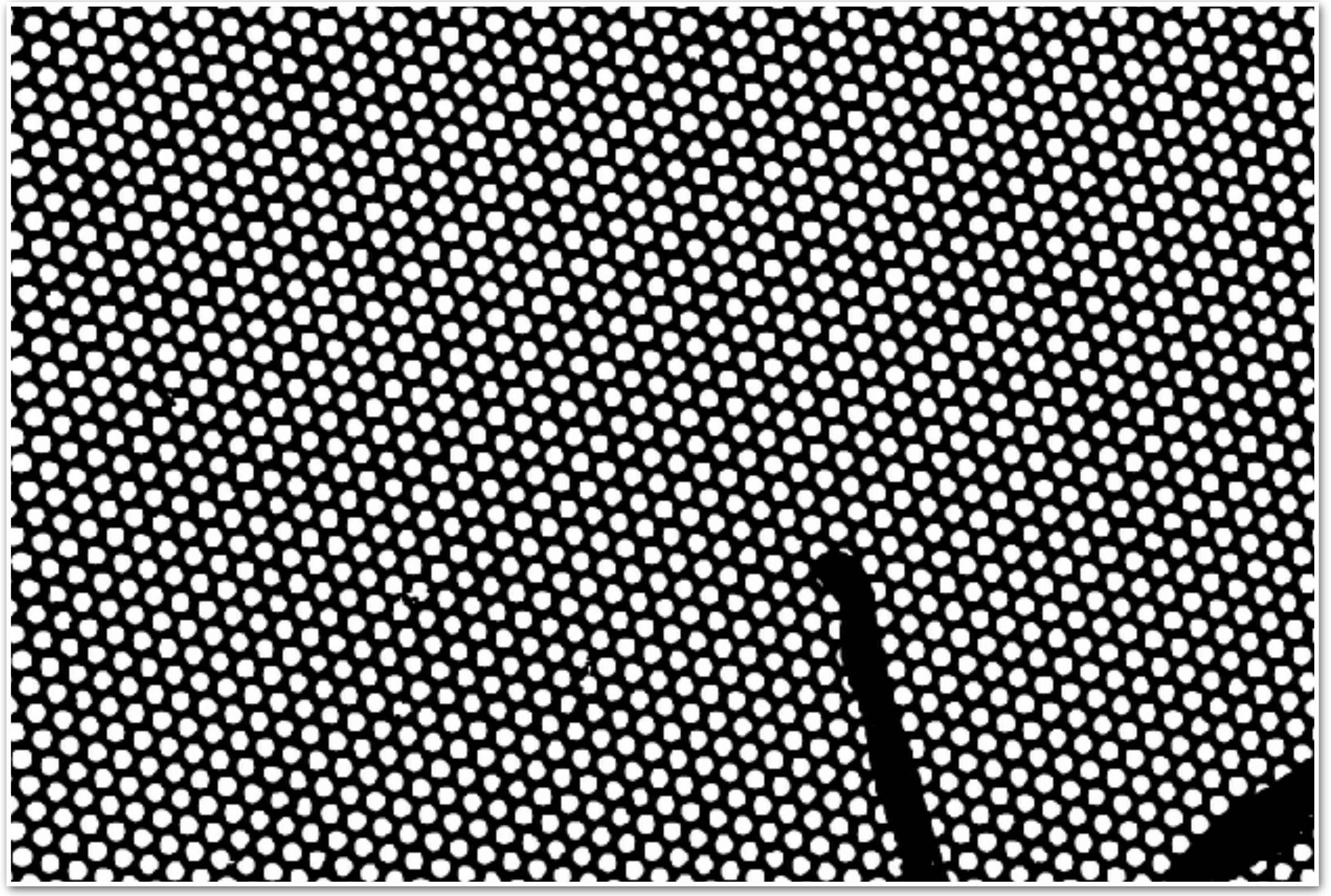

- **Choose threshold pixel by pixel (*Adaptive threshold*): mean inside the box + offset**
- **"Compactify" objects (*Morphological closure)***
- **Select *good* holes:**
  - **size** $\in [1100,1700]$ **pixel**$^2$
  - **circularity** $\in [0.92,1.00]$