# E-beam lithography and cryo-etching for the definition of sharp edges in 2D-materials

Vito Clericò

*Clean Room Technical Manager Universidad de Salamanca*

**vito_clerico@usal.es**

**NANOLITO 2021:**
SUMMER SCHOOL IN BASICS AND APPLICATIONS OF NANOLITHOGRAPHY
June 29-30 & July 1st 2021
**UNIVERSITY OF SALAMANCA**

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# Outline

USAL Laboratory of Nanoelectronics and Nanomaterials

- **PART I: SEM (Scanning Electron Microscope):**

- **PART II: EBL (Electon Beam lithography):**

- **PART III: Temperature dependence (and cryoetching) for edge definition (in 2D materials)**

# PART I: SEM
# (Scanning Electron Microscope)

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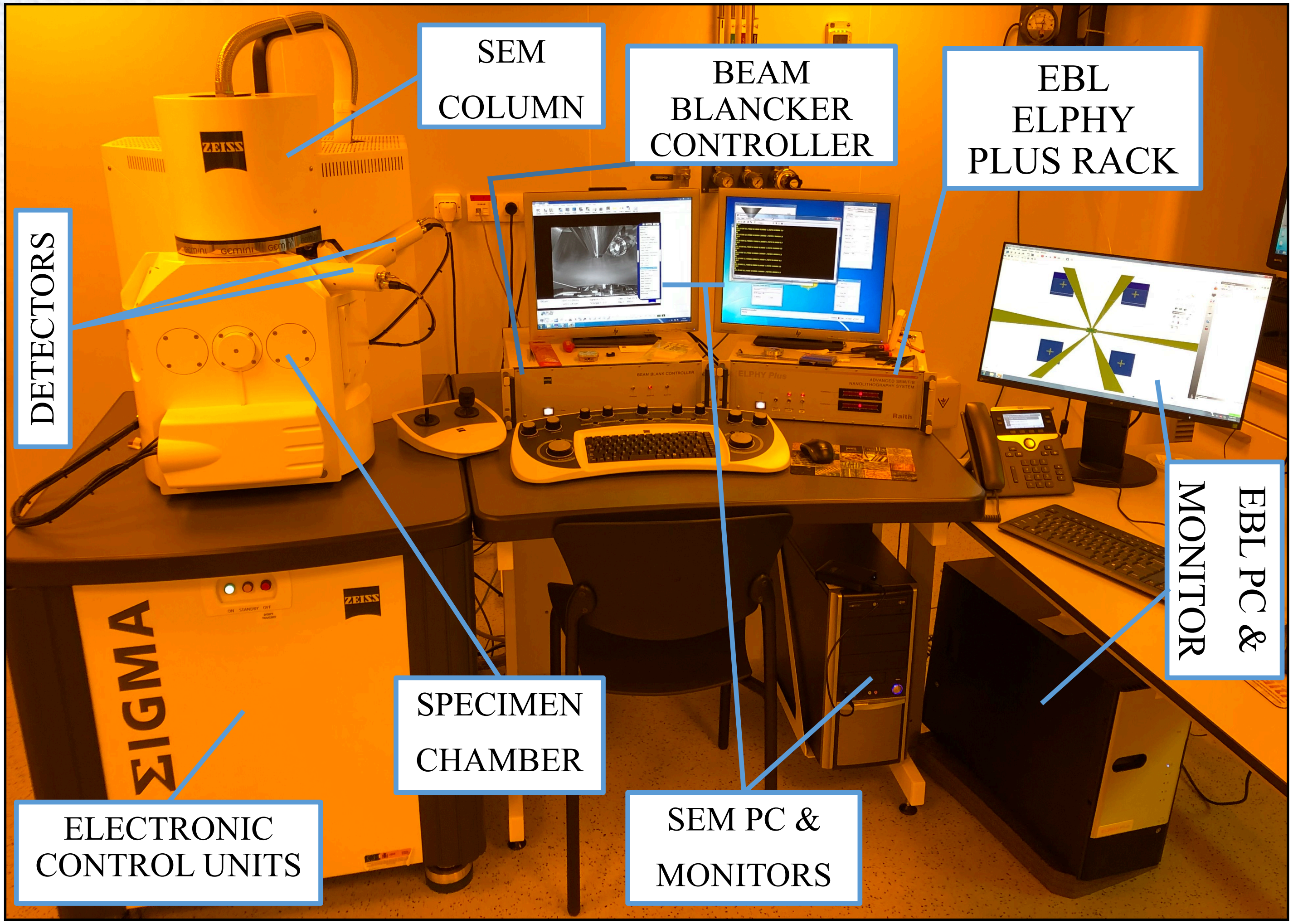
Excellent SEM USER

EBL USER

# EBL system


SEM
COLUMN
BEAM
BLANCKER
CONTROLLER
EBL
ELPHY
PLUS RACK
DETECTORS
EBL PC &
MONITOR
SPECIMEN
CHAMBER
ELECTRONIC
CONTROL UNITS
SEM PC &
MONITORS
ZEISS
SIGMA

# Nominal SEM Resolution (Analogy with Optical Microscope)

The diffraction limit depends on: $\lambda$

It is related to the momentum according: $\lambda = \frac{h}{p}$

the momentum depends on the accelarationg voltage: $p = \sqrt{2meV}$

So $\lambda = \frac{h}{\sqrt{2meV}} = \sqrt{\frac{1.5}{[V]}}$ nm

For an acceleration voltage of 5 kV $\lambda \cong 0.017$ nm

# Real SEM resolution: electron spot size

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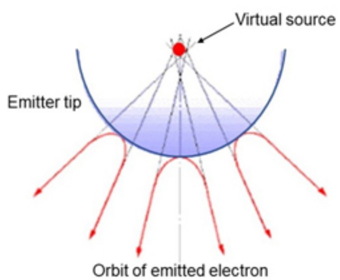
A part of the diffraction limit, the theoretical electron spot is given by:

$$d = (d_g^2 + d_s^2 + d_c^2 + d_d^2)^{\frac{1}{2}}$$

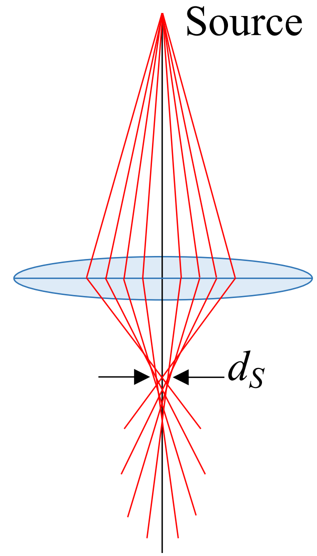
Source size: - 5-10 nm FEG
-15-20 nm SEG

$$d_g = \frac{d_f}{M}$$

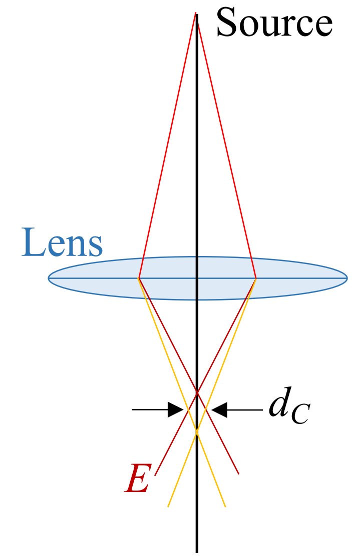
Magnification




Virtual source contribution




Spherical aberration




Chromatic aberration

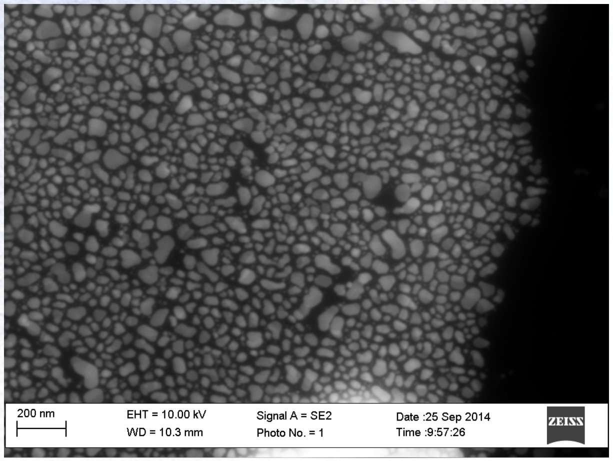
Analogy with optical microscope

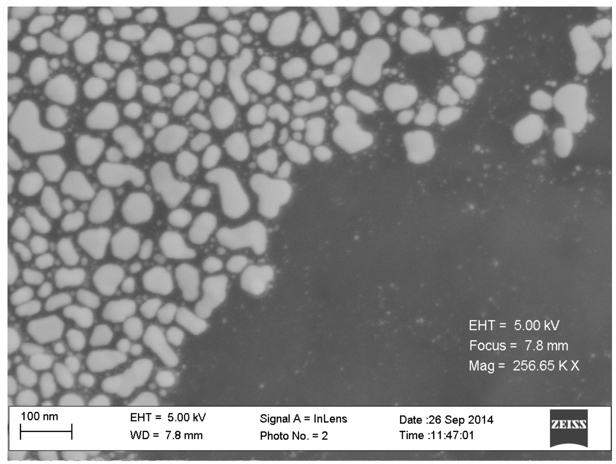
-*Electron beam lithography and its use on 2D materials* (Chapter 3), Book Nanofabrication: nanolithography techniques and their applications Institute of Physics, UK, 2020

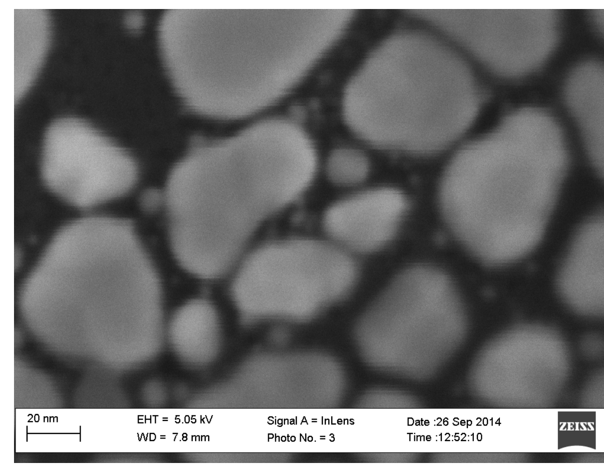
-https://www.jeol.co.jp/en/words/semterms/search_result.html?keyword=virtual%20source

# Final resolution depends on the sample

**Resolution**: Mimimum distance that can be separated as two distinguishable distance between two object in SEM image



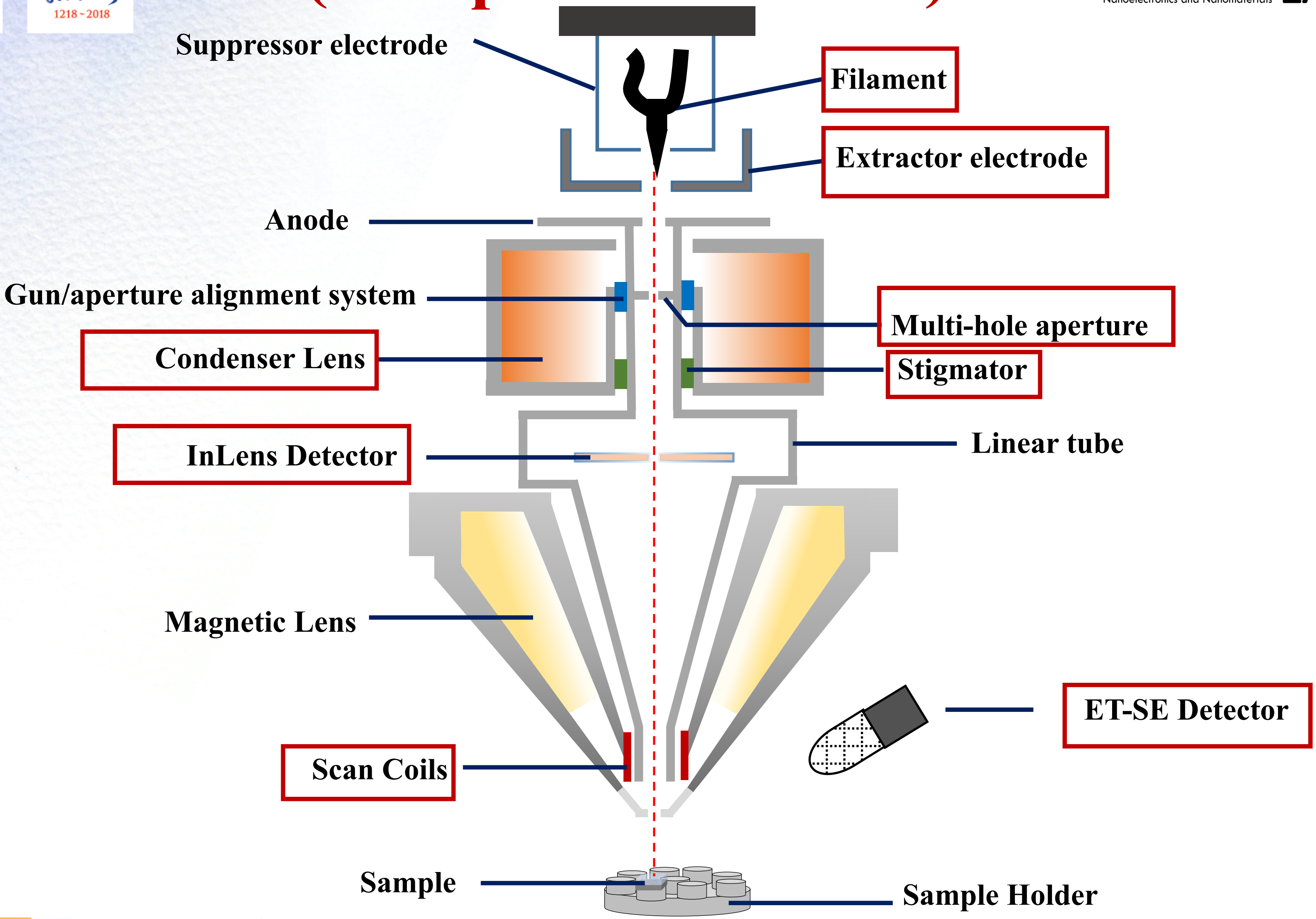




**Gold sample**

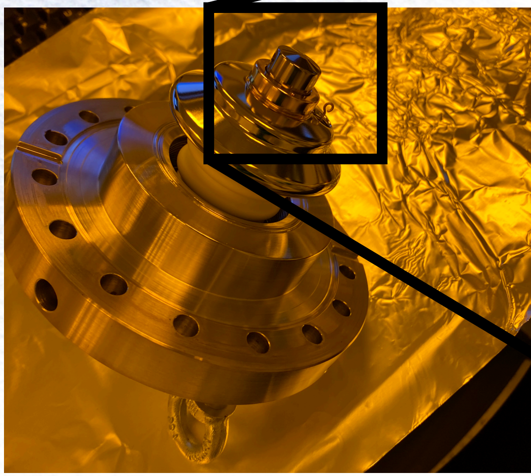
≦1 nm and 20 nm

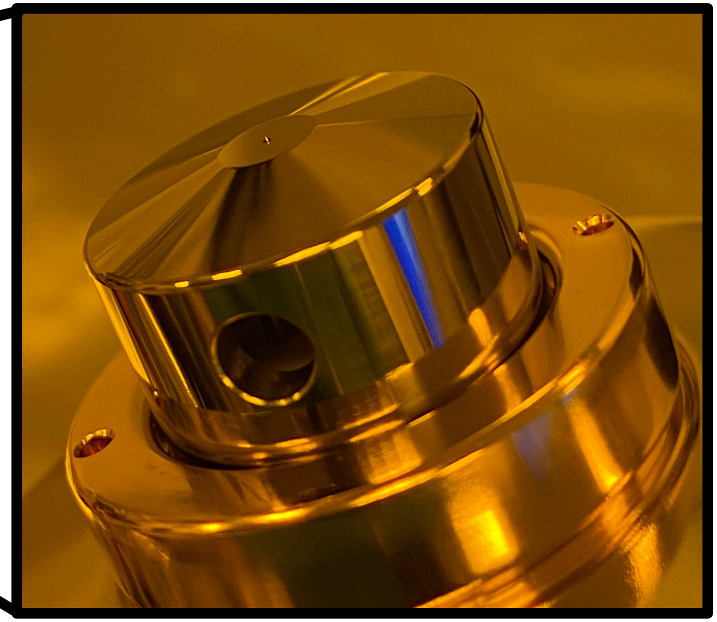
# SEM (Components Details)




*Electron beam lithography and its use on 2D materials* (Chapter 3), Book Nanofabrication: nanolithography techniques and their applications Institute of Physics, UK, 2020.

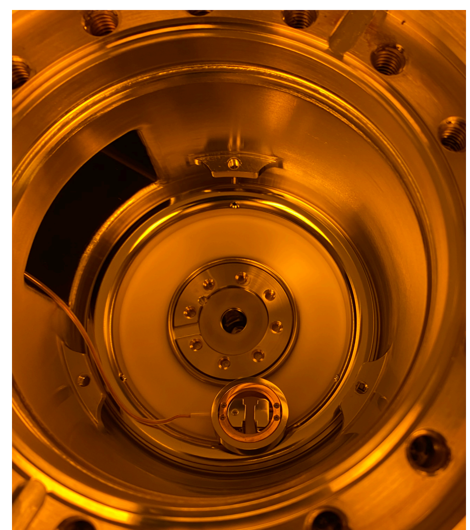
# Filament/Aperture/ beam blanker





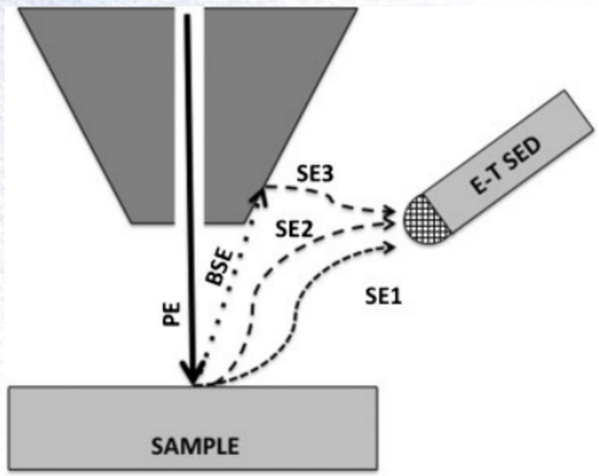
Filament

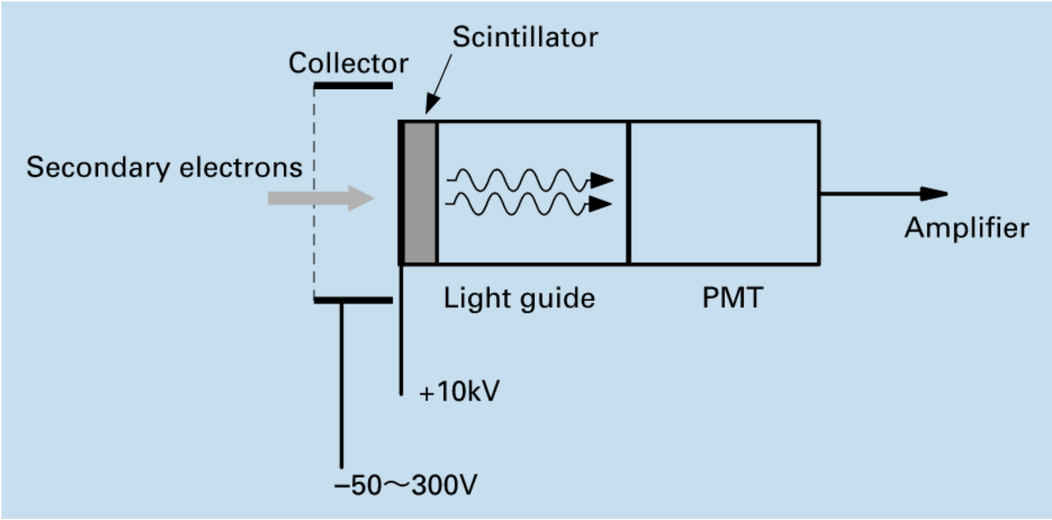
Multi-holes Aperture



Beam Blancker

# SE electrons and SE Detector (Basic Principle)




SE electrons low energy (<50eV)

-“**SE1**” are generated by the primary electron beam interaction with the sample

-“**SE2**” are generated by emergent high-energy BSE

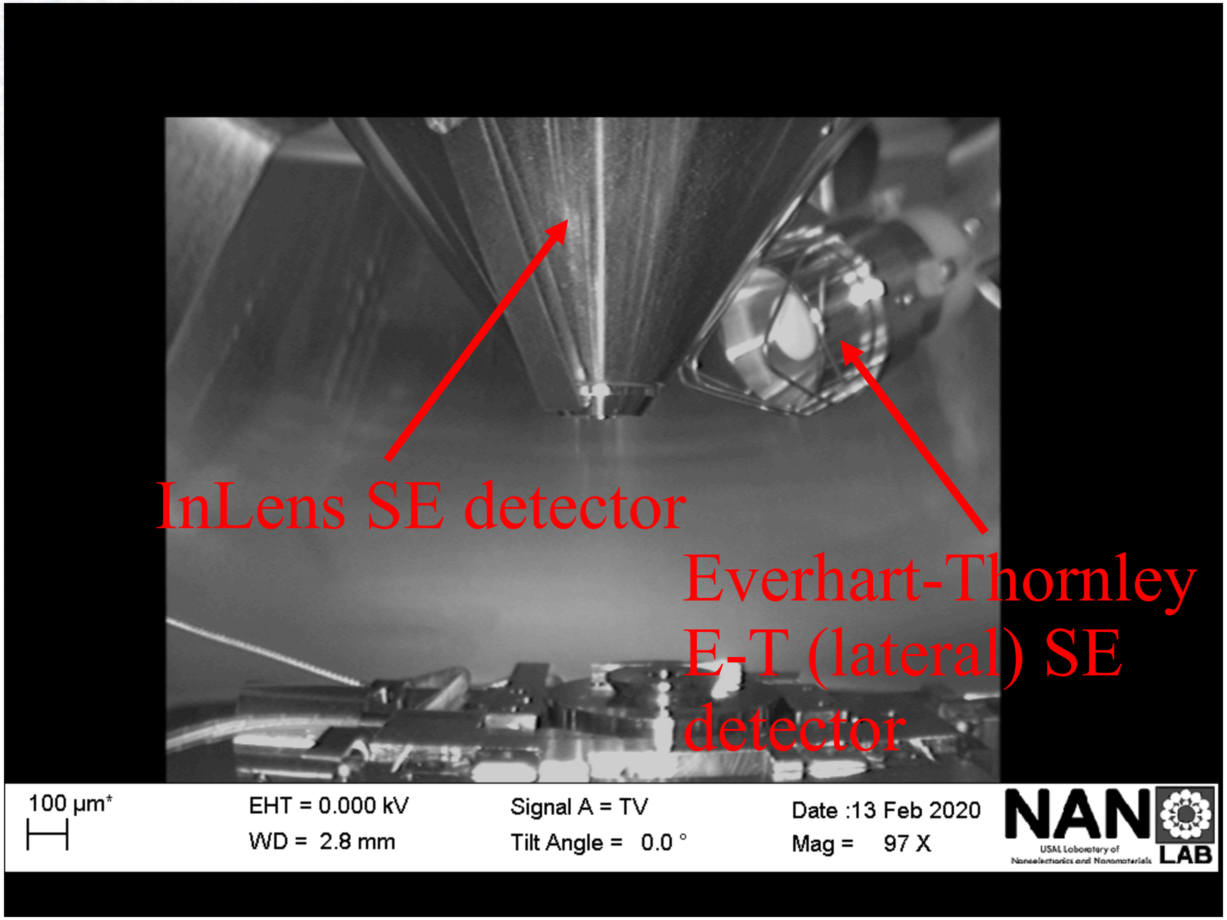
-“**SE3**” are generated by the BSE colliding with the chamber and column components of the SEM



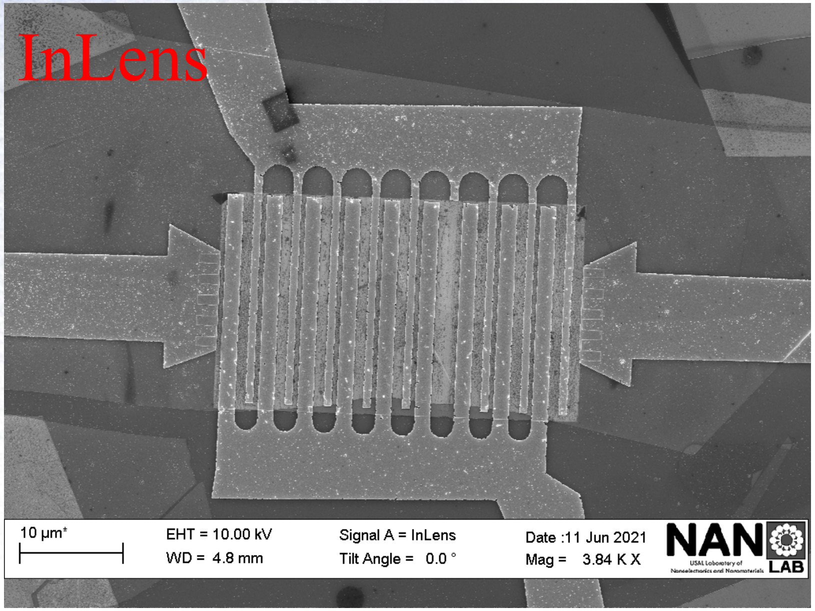

A Comparison of Conventional Everhart-Thornley Style and In-LensSecondary Electron Detectors—A Further Variable in Scanning Electron Microscopy, B.J. Griffin SCANNING VOL. 33, 162–173 (2011)

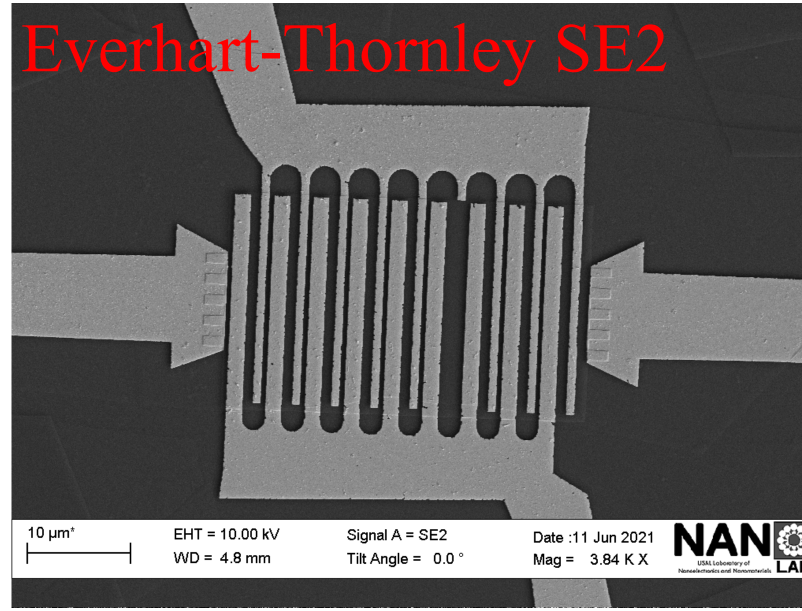
-SEM Scanning electron microscope A to Z (Jeol) https://www.jeol.co.jp/en/applications/pdf/sm/sem_atoz_all.pdf

# Inlens SE and E-T (lateral) detector



# InLens and E-T detectors (Images)




- low energy secondary electrons and provides images with a higher resolution
- sensitive to the surface characteristics of the sample
- Works at low acceleration potentials (<5 kV) (minimising the charging effect on non-conductive samples).

- topography image of the sample surface with a large depth of field
- medium and low resolutions with high acceleration potentials
- Tilted image (cross-section)

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Dr. Juan Antonio Delgado Notario device

# Control of a SEM: SEM joystick and keyboard

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- Z stage movement

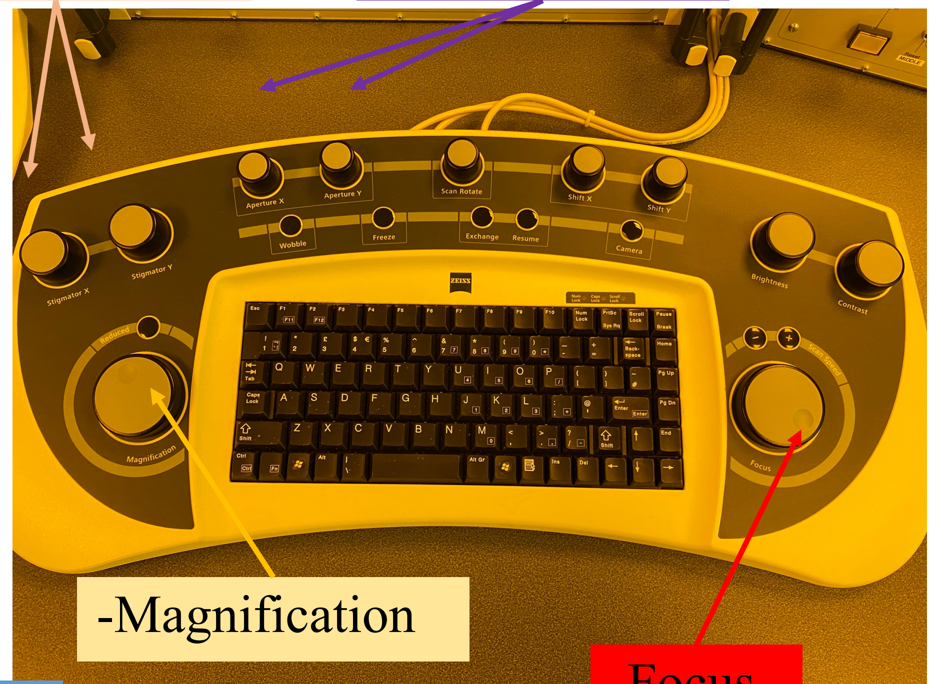
-Tilt


-X-Y stage movements

-Rotation

-X-Y Stigmator

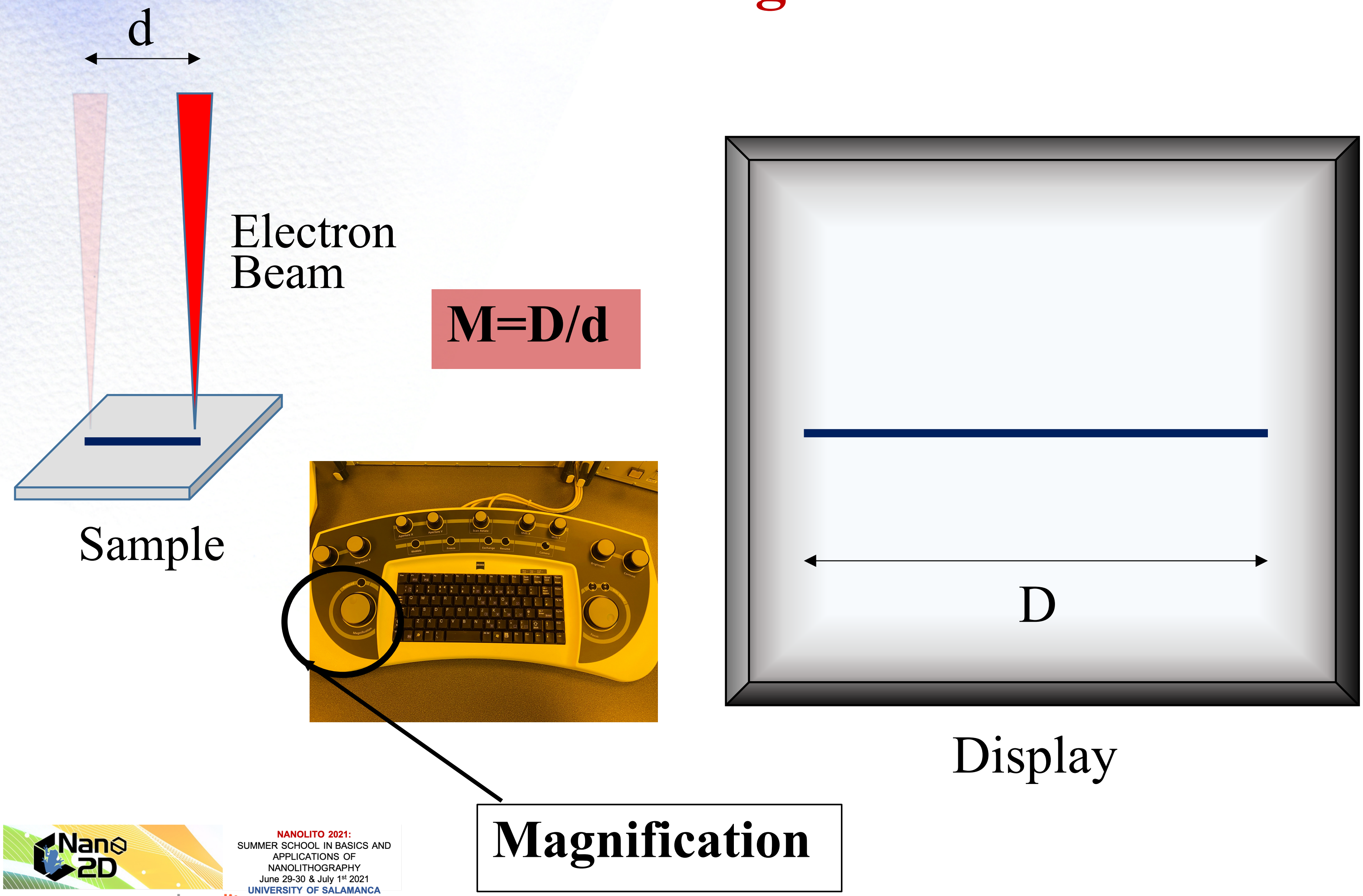
-X-Y Aperture



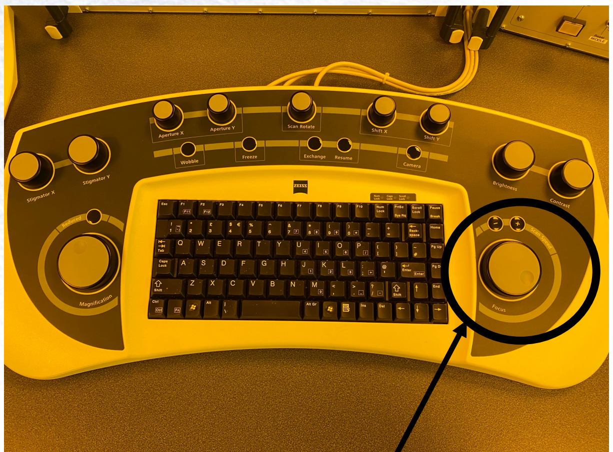

-Magnification

-Focus

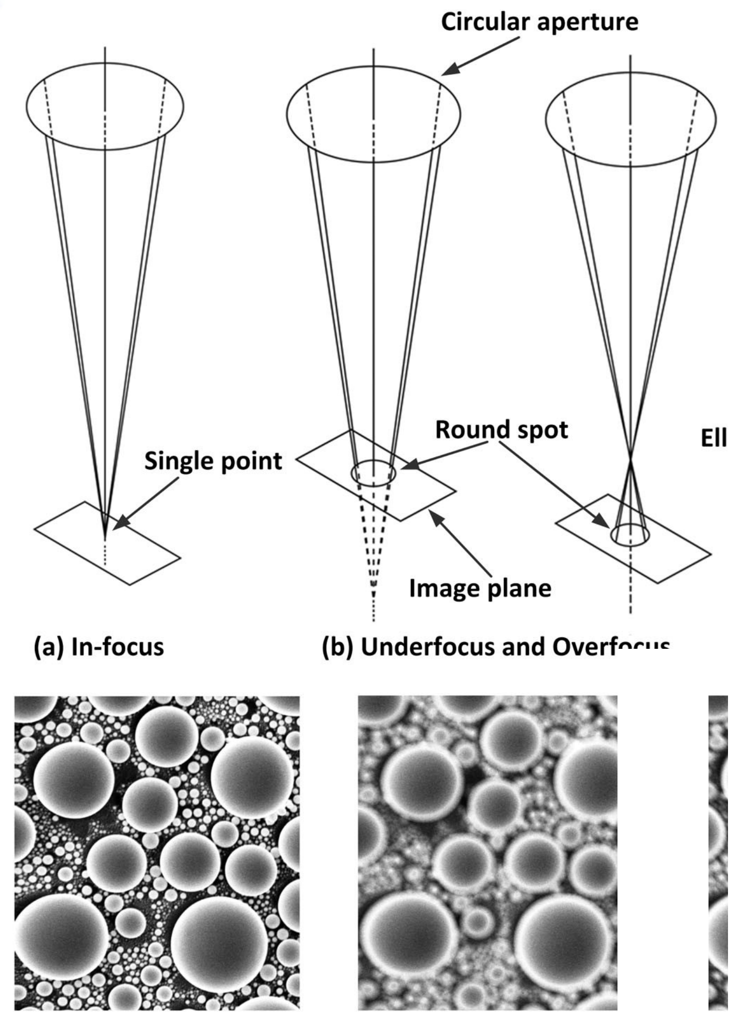
# Magnification



# Correction of SEM parameters: Focus



Focus

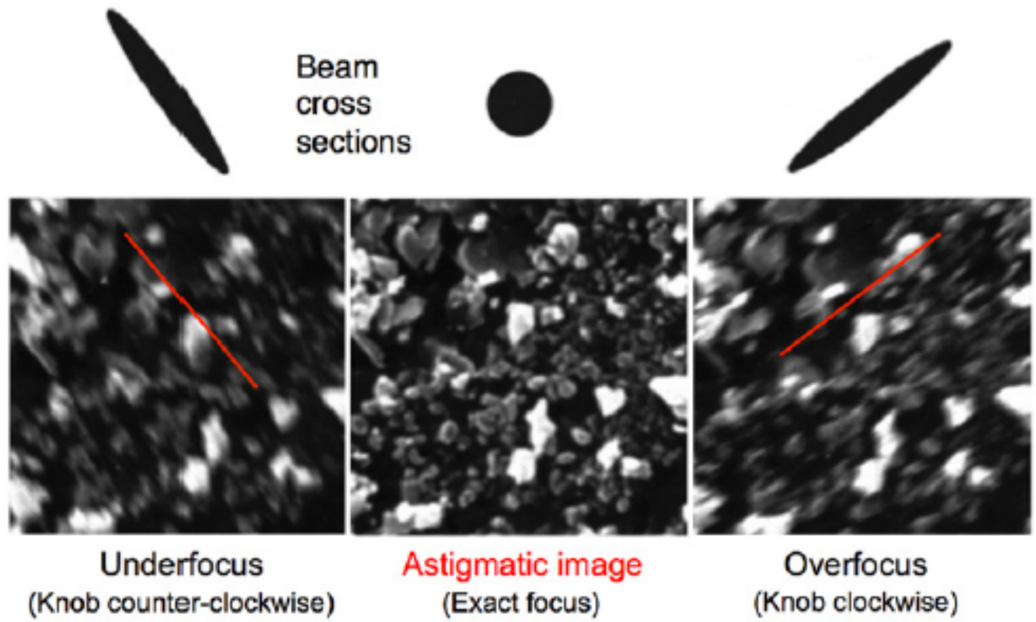



Yihua Lu; Xianmin Zhang; Hai Li; *AIP Advances* **8,** 015124 (2018)

# Correction of SEM parameters: Astigmatism

- Due to the imperfection of the lens system
- Round objects might appear elliptical or out of focus

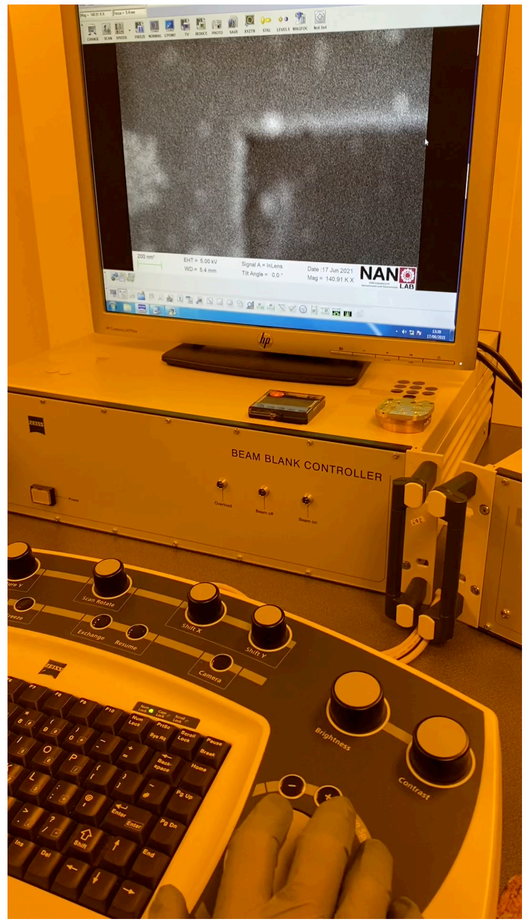
How do you detect an astigmatic image?




Microscopy Today , Volume 27 , Issue 3 , May 2019 , pp. 32 – 35

https://www.chems.msu.edu/resources/tutorials/SEM/generic-operation

# Correction of SEM parameters: Astigmatic image




How do you detect an astigmatic image?

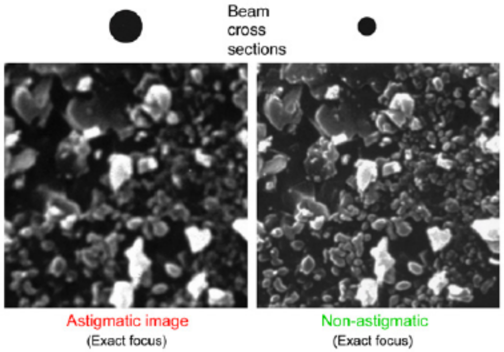
# Correction of SEM parameters: Astigmatic image

How do you correct the astigmatism?

We use a **stigmator:** special device (coils) used to compensate for imperfections in the construction and alignment of the **SEM** column

- sharpen the image with the x-stigmator control
- re-focus the image with the objective lens control
- sharpen the image with the y-stigmator
- re-focus the image
- - - repeat steps 4 through 7 until the image cannot be further improved

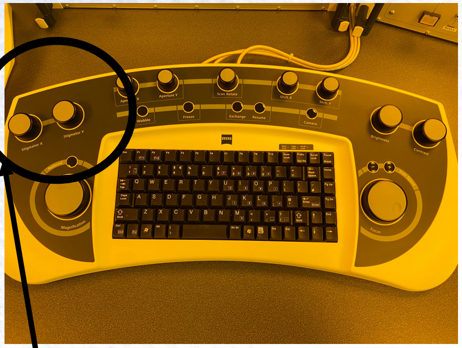
Without correcting for astigmatism, the smallest electron probe for that particular condenser lens setting will not have been achieved.





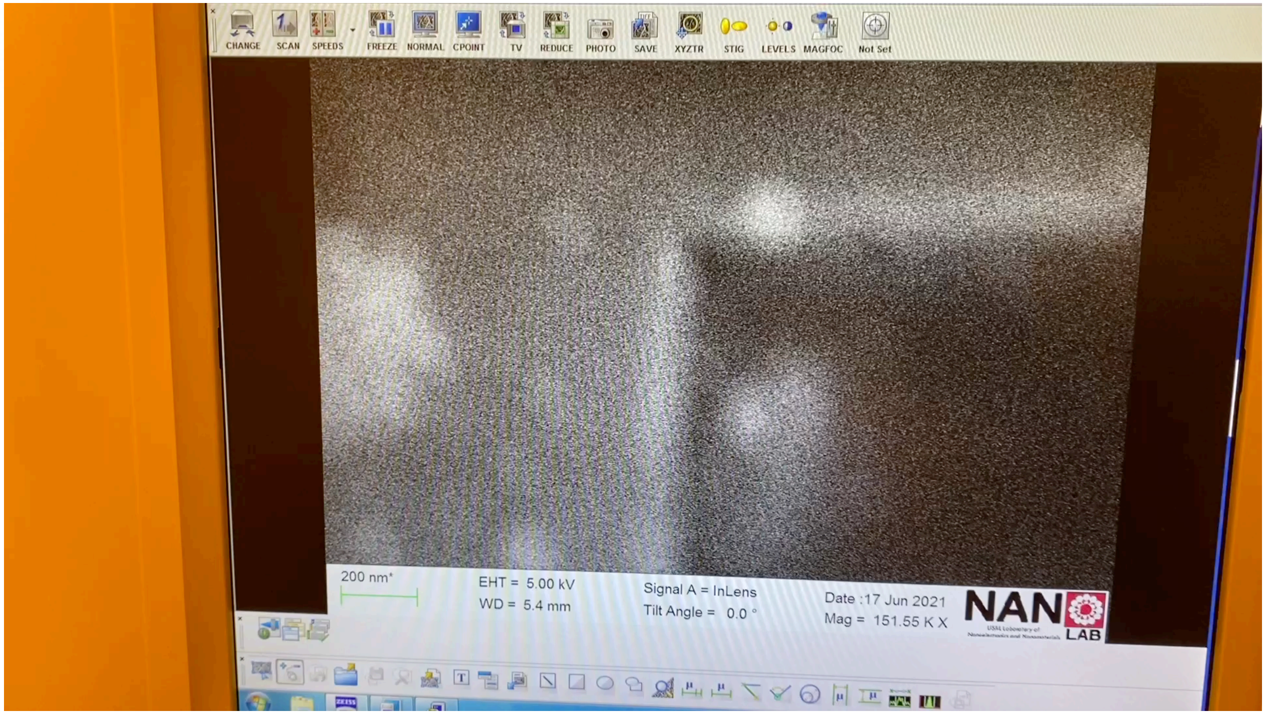

Microscopy Today , Volume 27 , Issue 3 , May 2019 , pp. 32 - 35

# Correction of SEM parameters: Astigmatic image



Sx y Sy

Correction of Astigmatic image on gold film



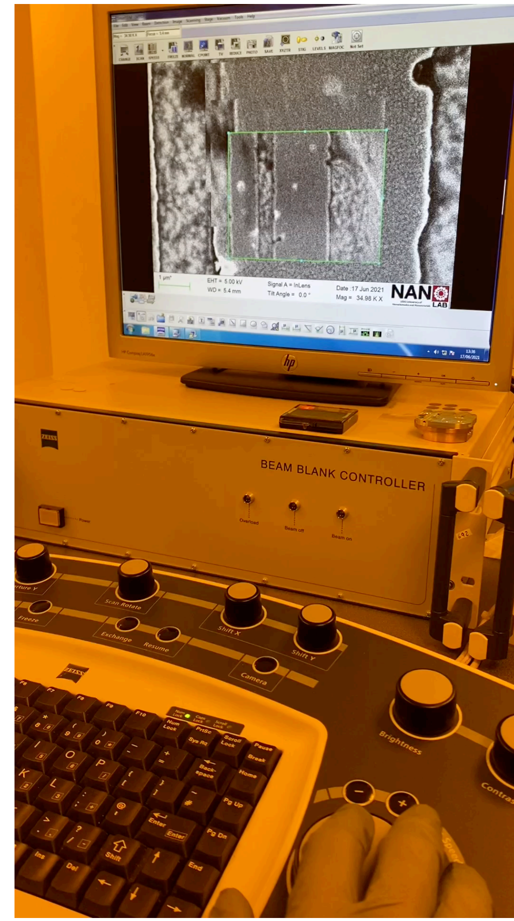
# Correction of SEM parameters: Alignment of aperture

The aperture is off-axis from the condenser lens

(the beam entrance angle will determine it's behavior, and if misaligned the final image quality will deteriorate)

How do you detect the misalignment of aperture?

Feature will appear to move across the screen as it goes in and out of focus



# Correction of SEM parameters: Alignment of aperture

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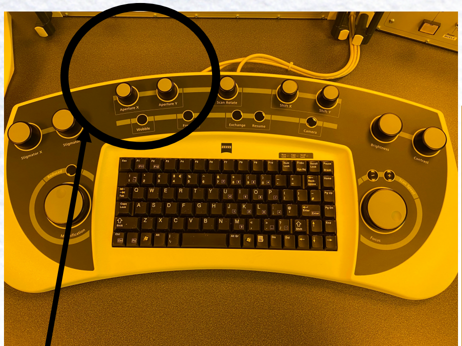
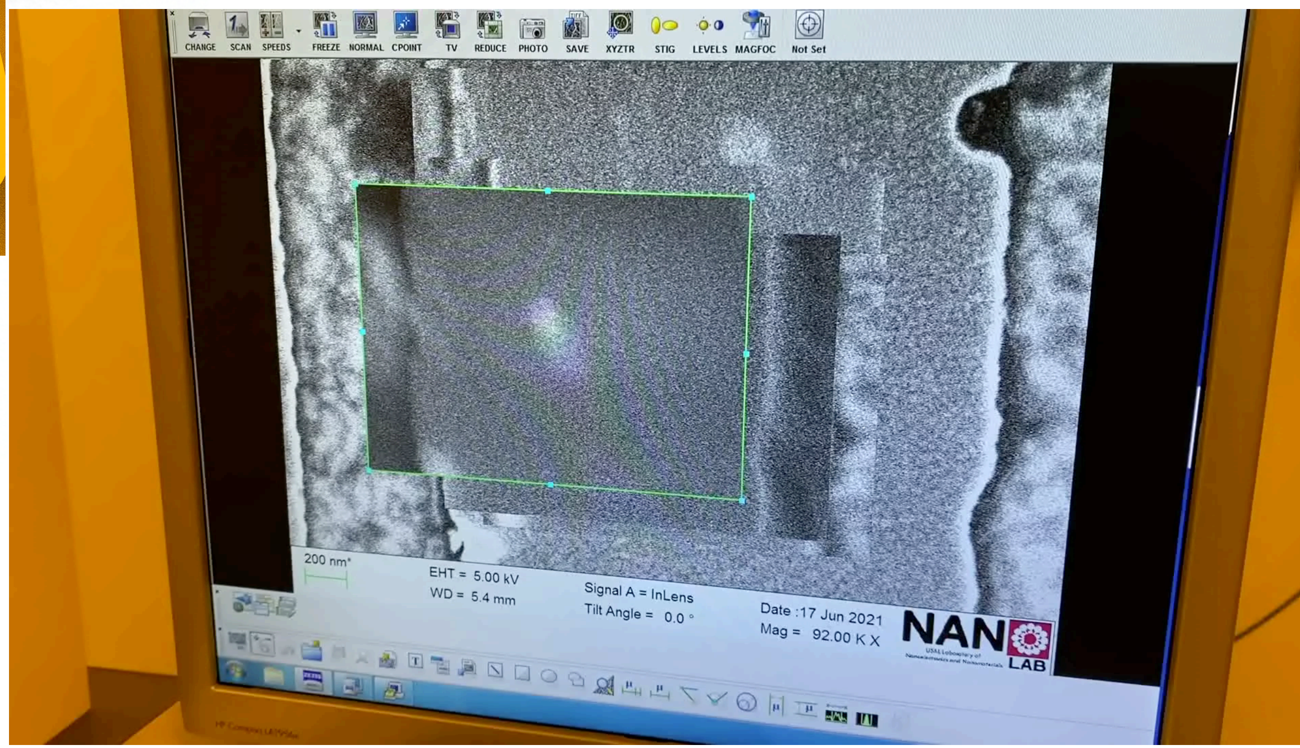
**'Wobble' on**: the feature image goes in and out of focus around the same point on the screen



Ax y Ay



# PART II: EBL
# (Electron Beam lithography)

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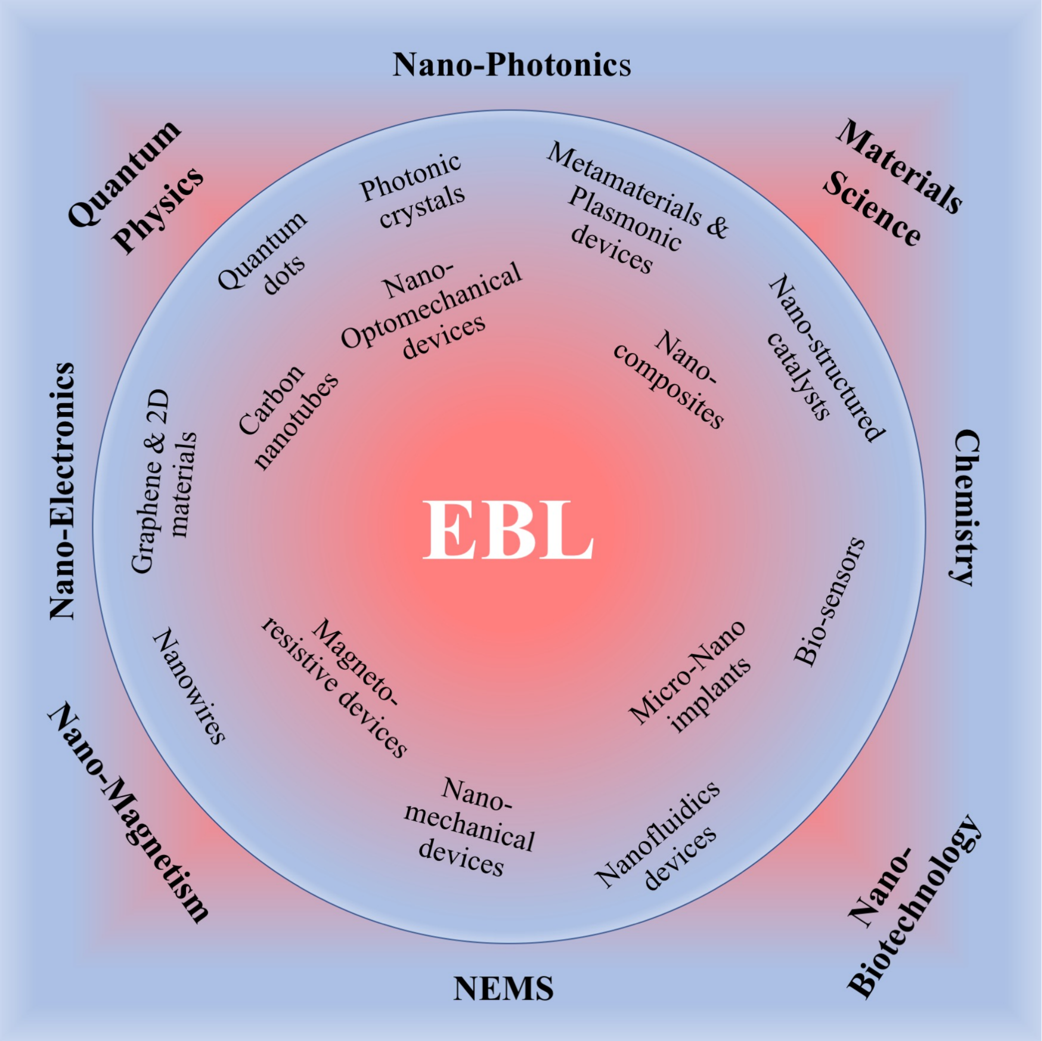
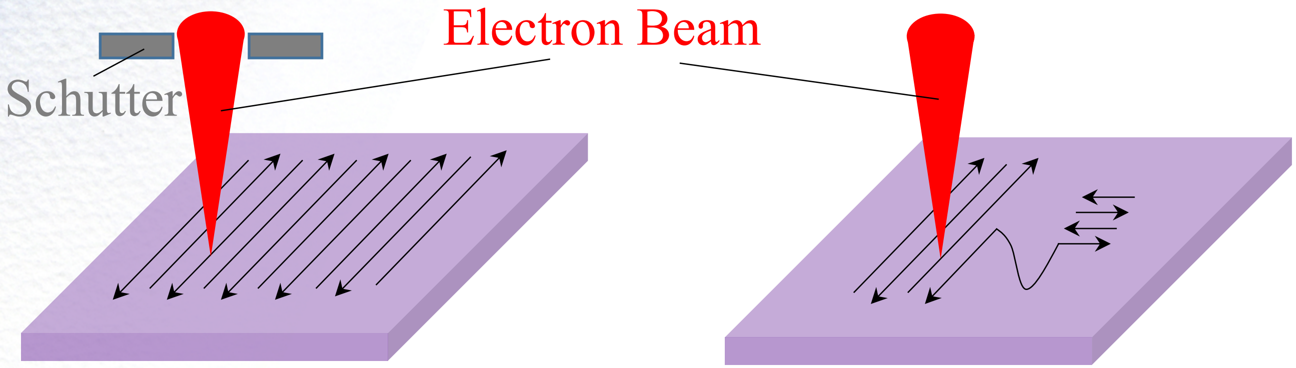
# EBL
# (General Application)



# Raster scan vs vector scan



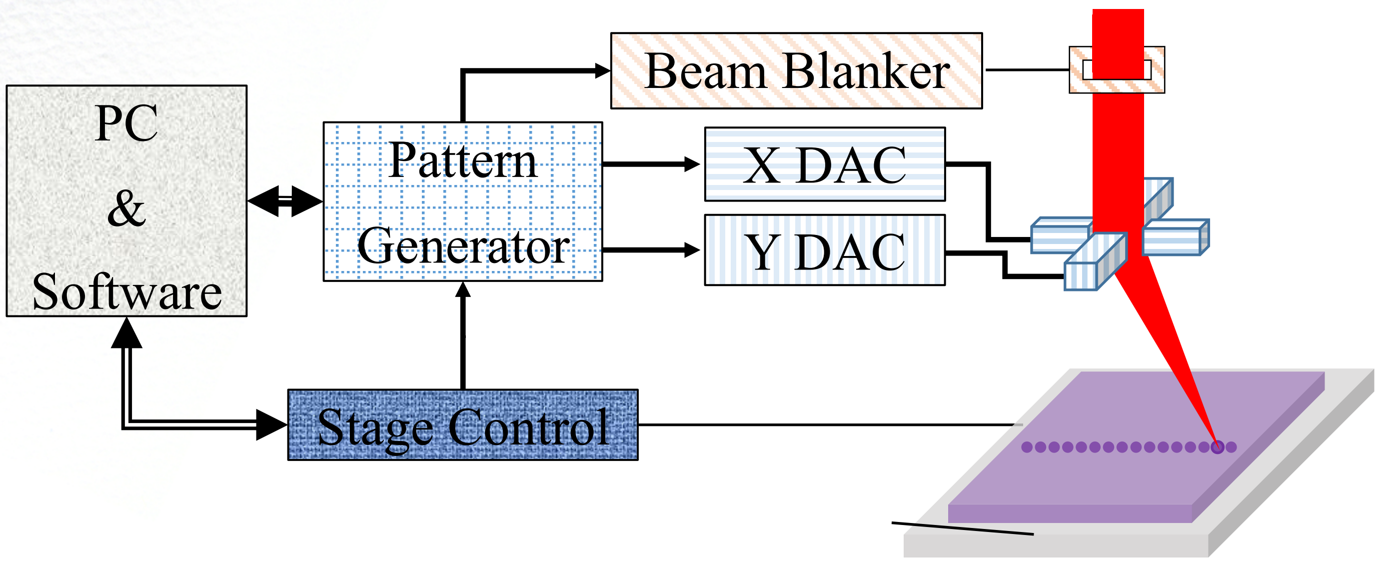

**Raster Scan** | **Vector Scan**

| Raster Scan | Vector Scan |
|---|---|
| The entire field is scanned with the raster scan method and the beam is blanked where exposure is not required | System scans only over the required positions and the beam is blanked as it moves between different scanning places |
| - Slow in sparse pattern<br>- Beam is moved only in one direction (stage for the other direction)<br>- Very reproducible | - Fast in sparse pattern<br>- Beam moved in both direction (No stage movement in the same WF)<br>- Need to wait at each pattern |



*Electron beam lithography and its use on 2D materials* (Chapter 3), Book Nanofabrication: nanolithography techniques and their applications Institute of Physics, UK, 2020.

# Schematic EBL set-up

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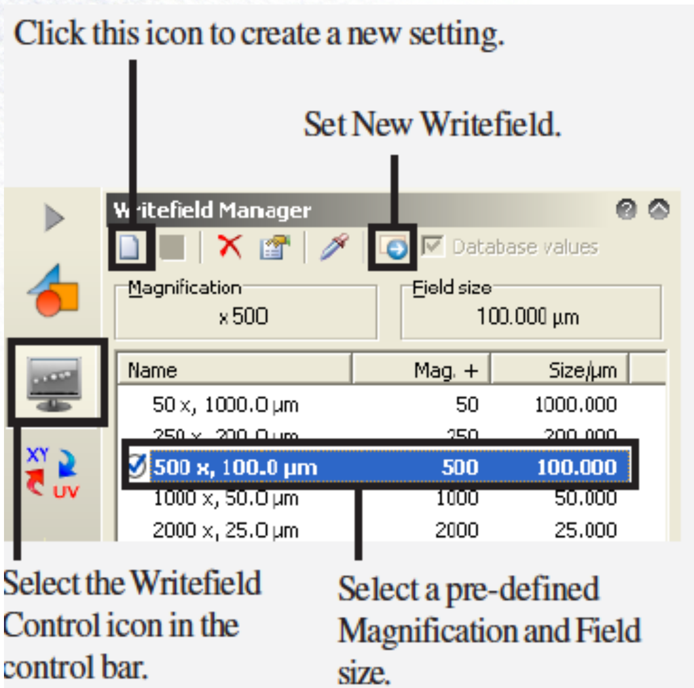
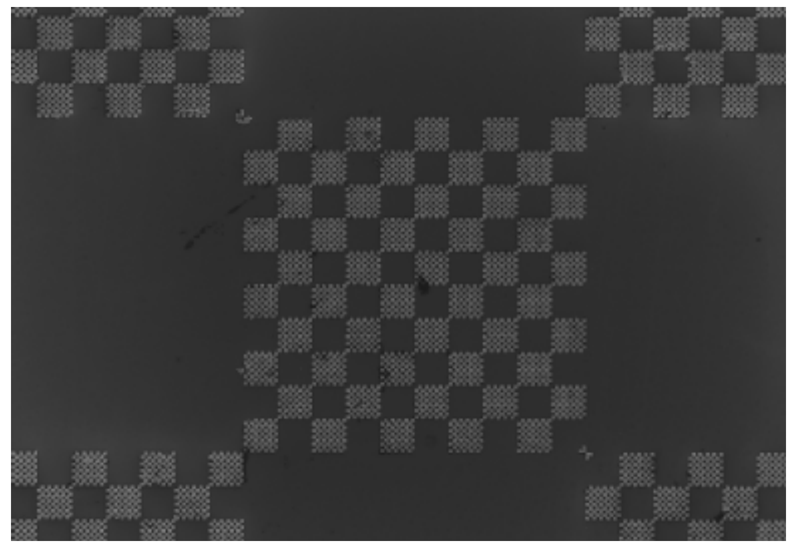
*Electron beam lithography and its use on 2D materials* (Chapter 3), Book Nanofabrication: nanolithography techniques and their applications Institute of Physics, UK, 2020.

# Writing Field (WF)

-The largest area exposed without the stage movement
-WF depends onthe SEM magnification




Calibration of WF: Chessy sample

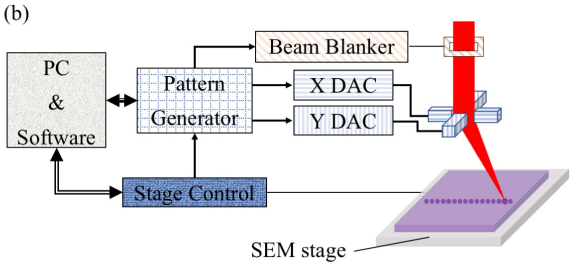




*Elphy Plus Operation Manual*

# Writing Field (WF) & DAC

- Each WF is composed by a finite number of steps in the exposure (exposure elements (EXELs)), determined by the deflection of the beam according to a certain value of an applied potential.




- This potential is controlled by a PC through a card for digital to analogic conversion (DAC), connected with the deflection system of the SEM

- For real systems, for a DAC of 16 bits for each axis for example, the WF is subdivided in a writing grid of $2^{16}$x$2^{16}$= 65 536 x 65 536 EXELs

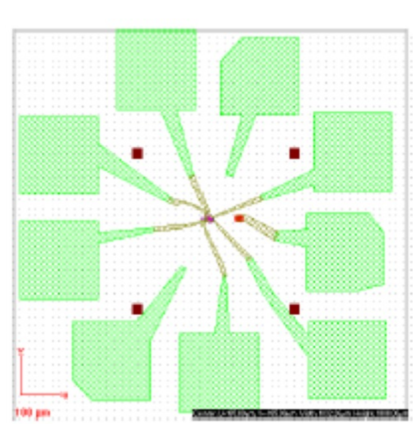


-*Electron beam lithography and its use on 2D materials* (Chapter 3), Book Nanofabrication: nanolithography techniques and their applications Institute of Physics, UK, 2020.
-Rius Suñé G 2008 Electron beam lithography for nanofabrication PhD Thesis Universitat Autònoma de Barcelona

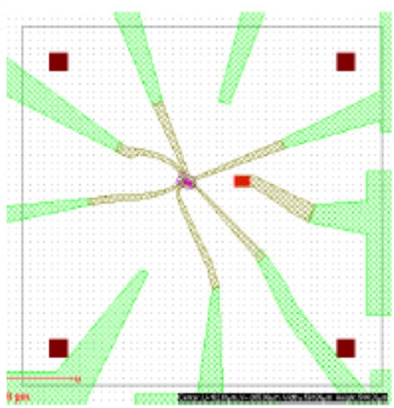
# Working Area (WA)

WA= Pattern size

WA=WF

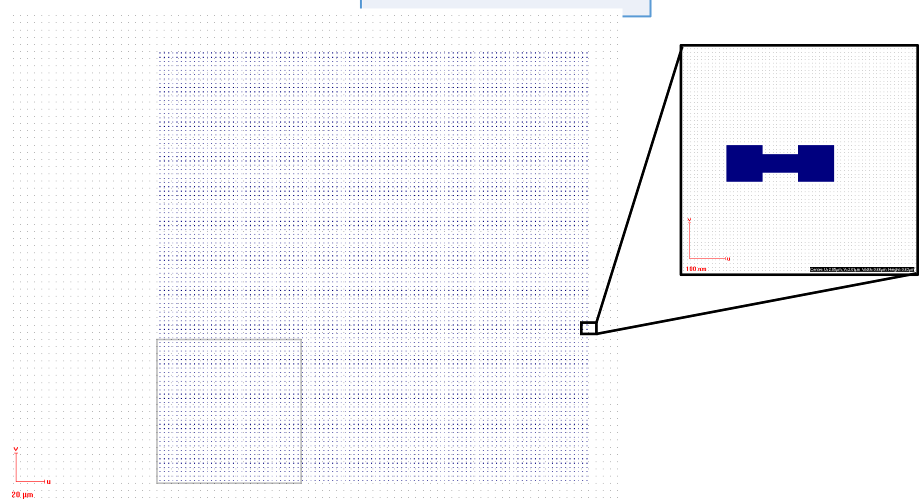


WF 1000 µm



WF 500 µm

WA>WF



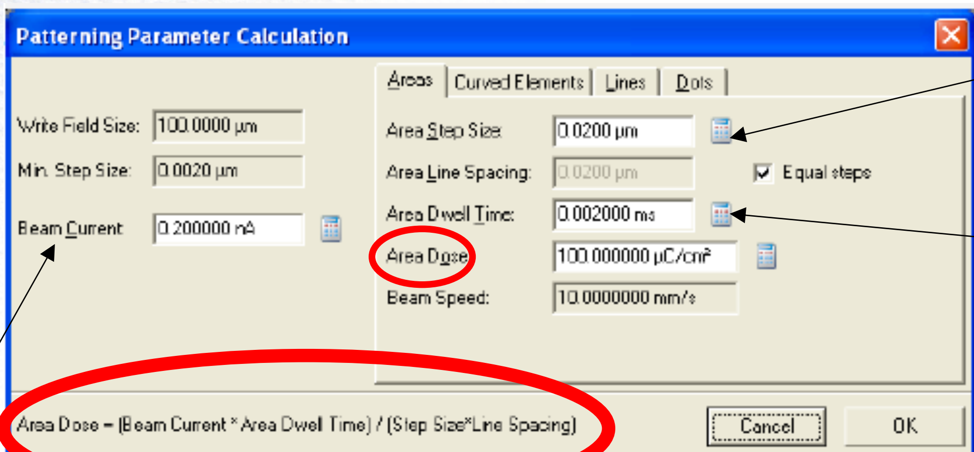
Stitching problem

Conventional Stage ('bad' stitching)

Interferometric Stage (very good stitching)

# Dose

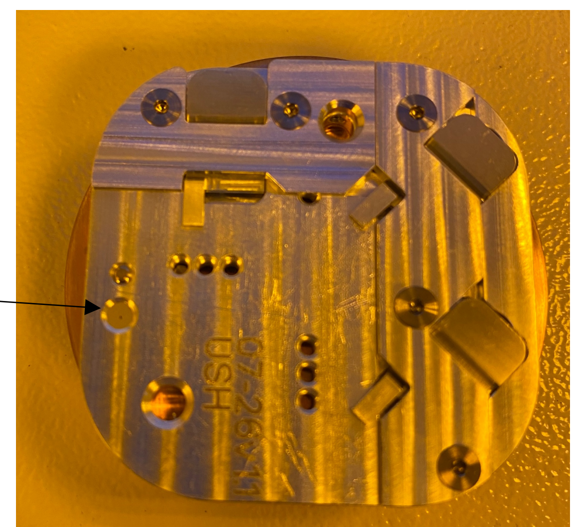
The real dose is measured in terms of current per unit area




Distance between exposure area

Time the beam remains in each pixel

Measured in the Faday cup

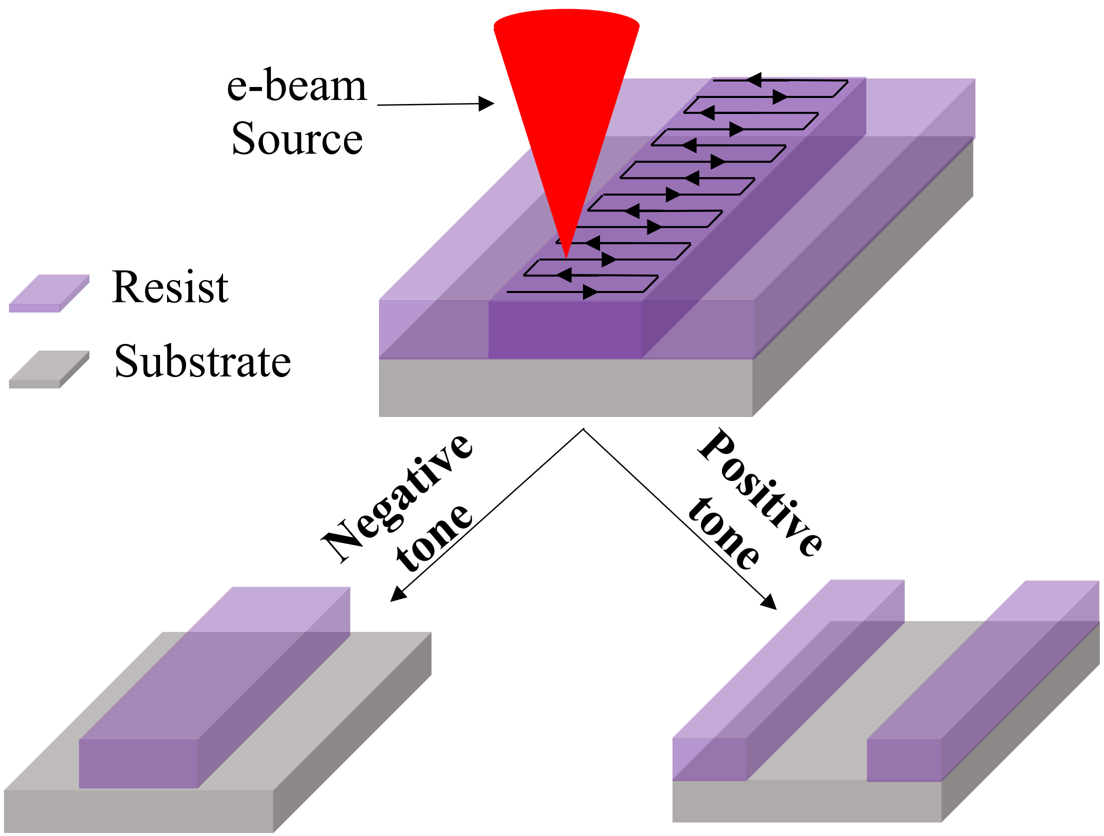


# Resist

Resists are either organic or inorganic polymeric materials dissolved in solvents and they can be classified in two different classes according their tone: positive or negative tone

The choice of a resist is typically taken according to three principal characteristics:

**resolution**, **contrast** and **sensitivity**

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# Resist

**Resolution** : minimum feature size (or minimum feature distance) that can be resolved

**Contrast:** Capability in differencing between exposed and unexposed areas

**Sensitivity** : alteration of the resist with respect to the electron beam doses

-it is desirable to have high contrast and high sensitivity resist,

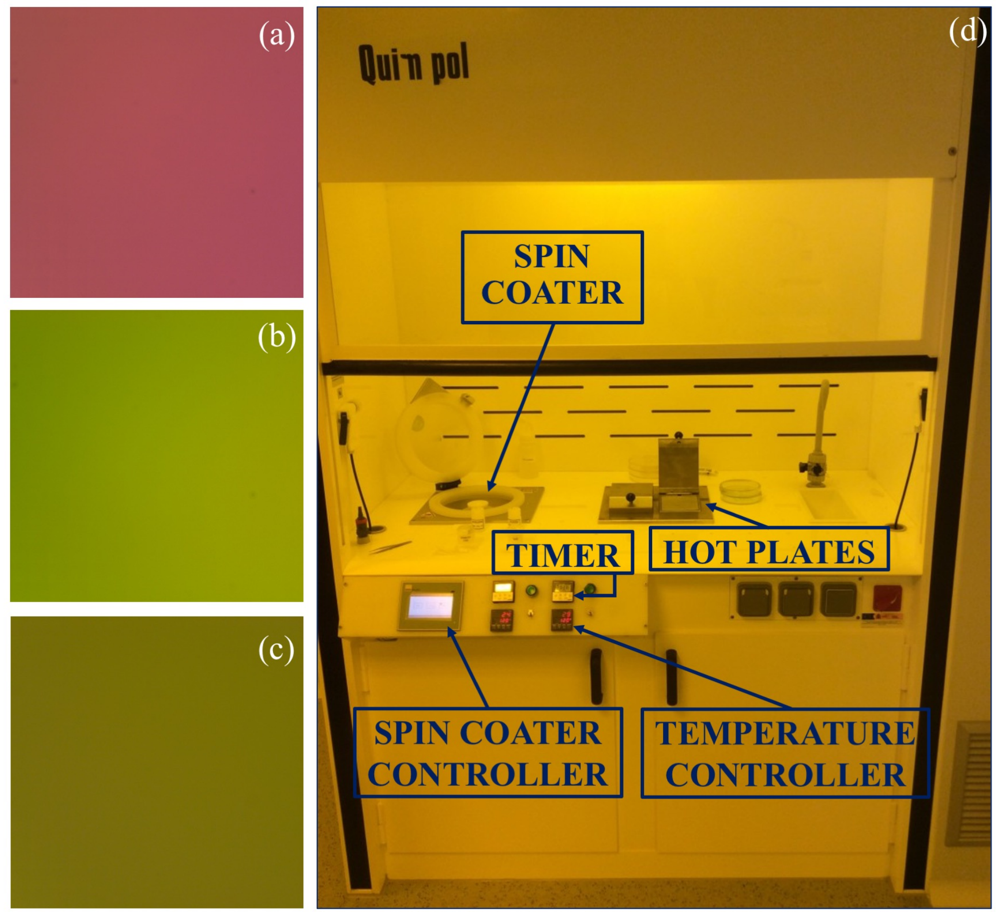
-an increase of contrast results in the worsening of the sensitivity and vice versa.



Gangnaik A S, Georgiev Y M and Holmes J D 2017 New generation electron beam resists: a review Chem. Mater. 5 1898

# Resist coating

Colour depends on the thikness of the resist



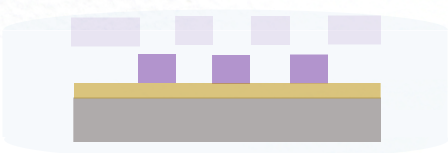
# Resists:



Apart from the electron energy, the **type and concentration of the developer**, the **developing time** and **temperature** affect the contrast and sensitivity

DEVELOPMENT

(Immersion in chemical solution)



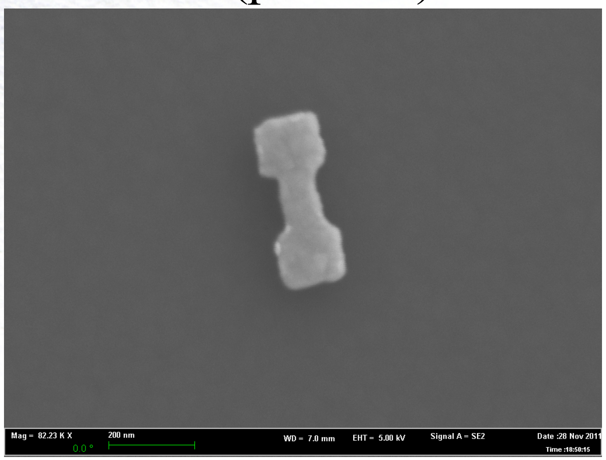
| Risist | Tone | Resolution(nm) | Sensitivity($\mu C\ cm^{-2}$) | Developer |
|---|---|---|---|---|
| PMMA | Positive | 10 | 100.0 | MIBK:IPA |
| EBR-9 | Positive | 200 | 10.0 | MIBK:IPA |
| PBS | Positive | 250 | 1.0 | MIAK:2-pentanone 3:1 |
| ZEP | Positive | 10 | 30.0 | Xylene: p-dioxane |
| AZ5206 | Positive | 250 | 6.0 | KLK PPD 401 |
| SML | Positive | 15 | 60 | 7:3 IPA:DI Water |
| SU-8 | Negative | 25 | 5.0 | EC solvent |
| COP | Negative | 1000 | 0.3 | MEK: ethanol 7:3 |
| SAL-606 | Negative | 100 | 8.4 | MF312:DI water |
| HSQ | Negative | <5 nm | 500.0 | TMAH:DI-water |
| NEB | Negative | <10 nm | 12.0 | CD26 |
| Calix[n]arene | Negative | 15 | 115.0 | MIBK |

- Gangnaik A S, Georgiev Y M and Holmes J D 2017 New generation electron beam resists: a review Chem. Mater. 5 1898

-- Chen Y 2015 Nanofabrication by electron beam lithography and its applications: a reviewMicroelectron. Eng. 135 57

# Example: PMMA vs HSQ

PMMA (ARP679.02 from Allresist©) (positive)



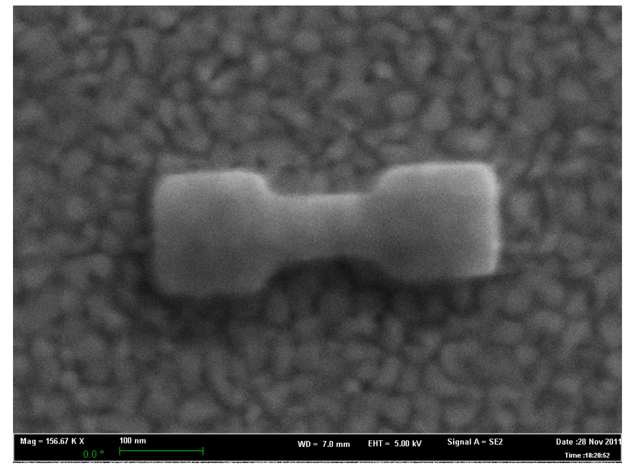

Dose 200 μC/cm2

Versatile

Lower Resolution

Lower Contrast

HSQ resist (XR-1541-004 Dow Corning) (negative)



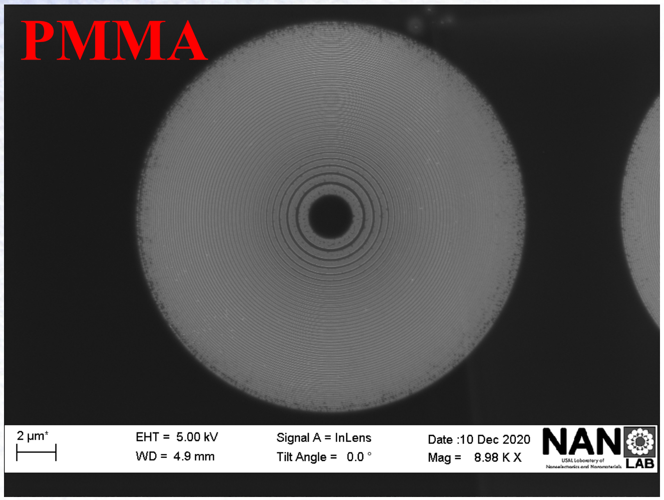

Dose 2000 μC/cm2

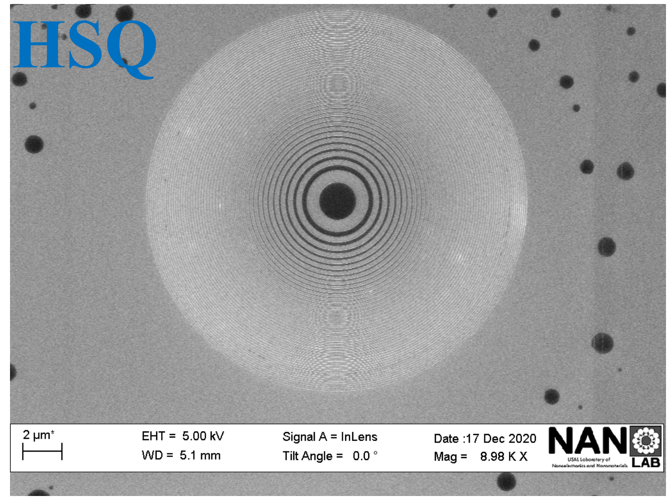
No Versatile

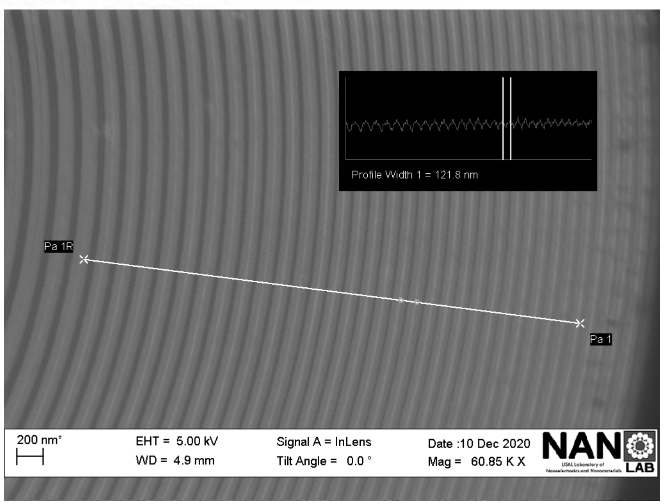
Higher Resolution

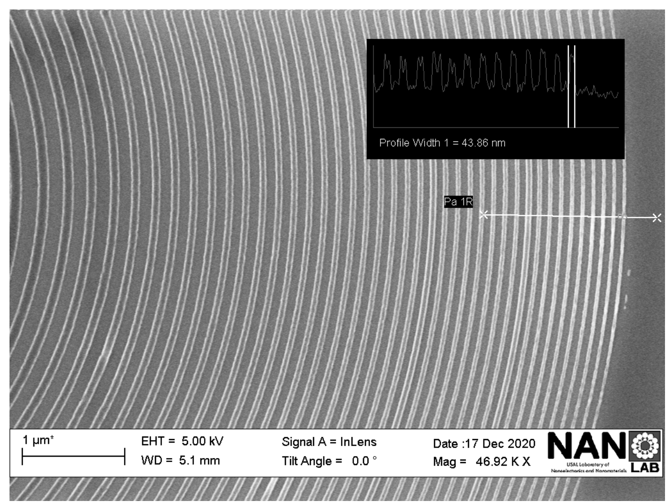
Higher Contrast

# PMMA vs HSQ: Fresnel Lens



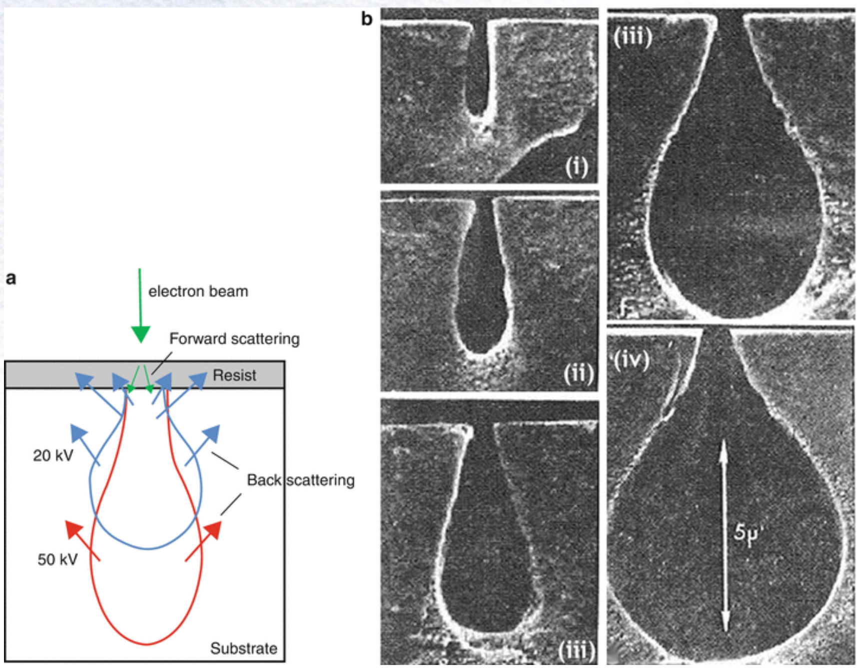




TFG
Javier Gil
Duran

# EBL Main Limitation: Proximity Effect

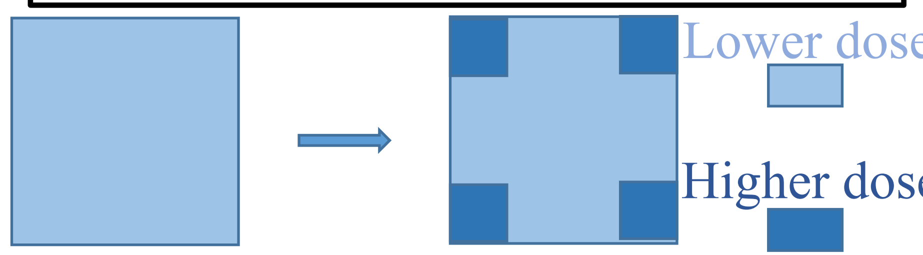



PE relates to the resist outside the targeted area of exposure that unavoidably receives a non-zero electron dose.

**Different solution:**

-thinner resist

- Multilayer resists
- High-contrast resist
- Fragmentation (CAD)




Pala N., Karabiyik M. (2012) Electron Beam Lithography (EBL). In: Bhushan B. (eds) Encyclopedia of Nanotechnology. Springer, Dordrecht. https://doi.org/10.1007/978-90-481-9751-4_344

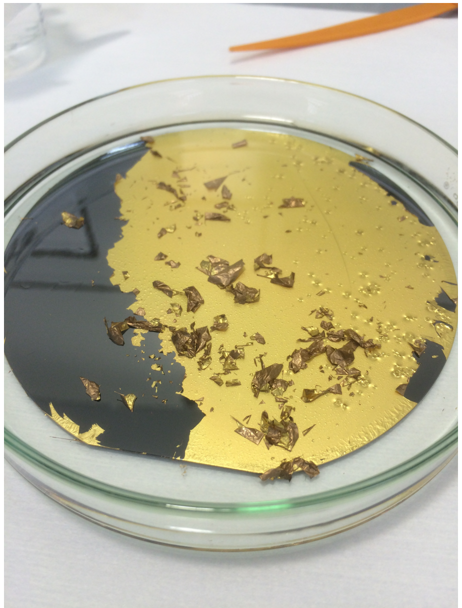
# EBL problem: Crosslinking

Cross-linking might appear when the energy of the electron beam is much higher than the resist ionization energy

The crosslinked resist cannot be easily removed from the sample surface

it is a ‘post-exposure’ problem related to a second fabrication step, typically the dry-etching process or during metal evaporation

The final result is a permanent resist mask after a dry etching process, or in case of evaporation problems during the lift-off process



# EBL on bare wafer: Global coordinates

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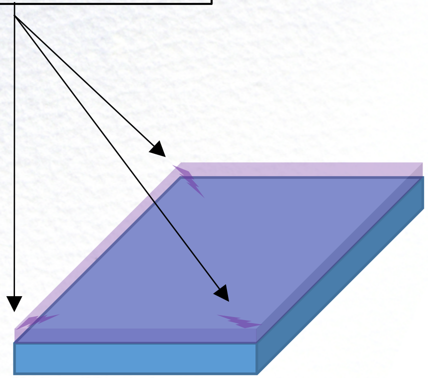
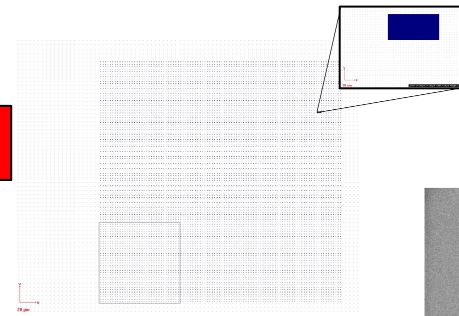
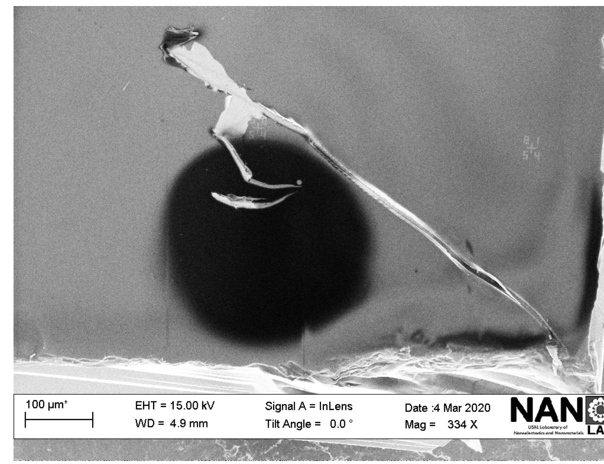
3 scratches



**WA>WF**



They help to determine the plane and correct the sem parameters (aperture alignment/astigmatism correction)




First of all we correct the global coordinates (correspondence between stage coordinates (X,Y) and the (U,V) coordinates of the sample):

- Take a *global* origin (normally bottom left corner of the sample)
- Define the *global* angle the X(U)-axis (we need to define 2 points on the axis)

From this moment it is possible to move the SEM stage using the (U,V) coordinates

# EBL on bare wafer: Three points alignment

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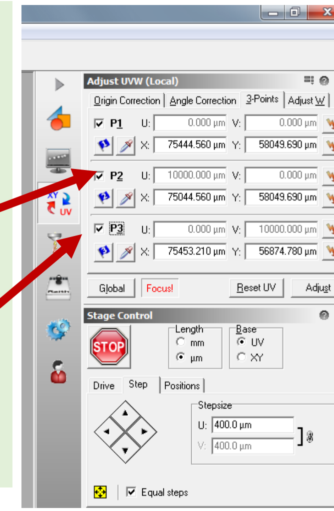
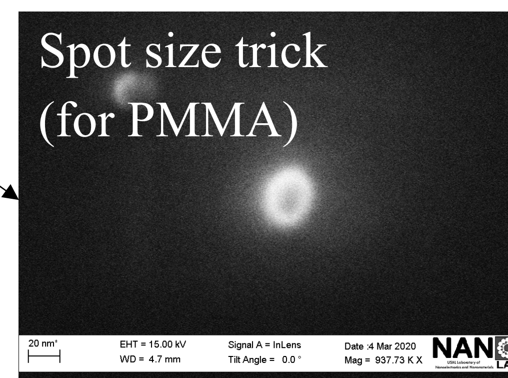
Define the plane (three points alignment):

- The bottomleft corner scratch as first (U,V) point (0,0) mm
- (after moving the sem) The bottomright corner scratch is the second (U,V) point (example (10,0) mm)
- (after moving the sem) The third (U,V) point at (0, 10) mm is ideally chosen at the top-left corner scratch of the sample.




In all the three points we have to correct perfectly the sem parameters (Aperture alignment, Astigmatism correction, **Focus**)

The procedure have to be repeated at least twice

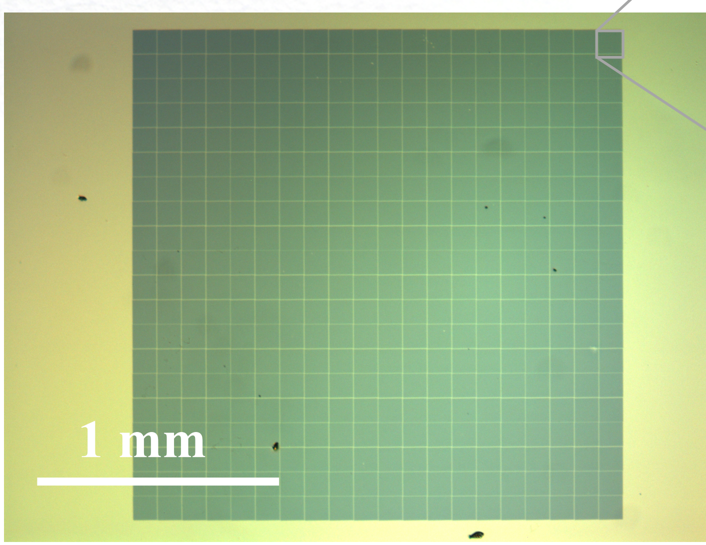


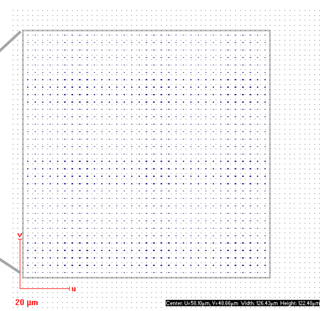
# EBL on bare wafer: Positioning list (exposition)

Move to the Faray Cup to meaure current (exact dose)

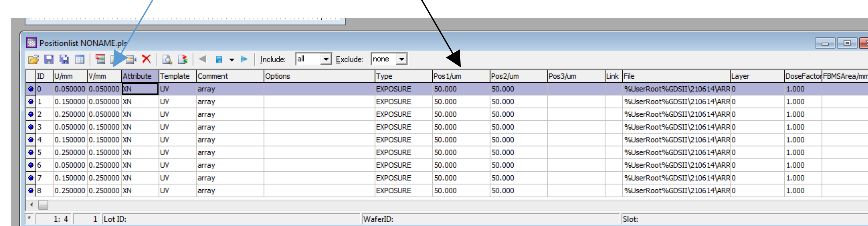
Determination between the coordinates in the sample (U,V) and coordinates in the CAD file (u,v) must be found

Final Exposure




WA=WF and matrix of WA



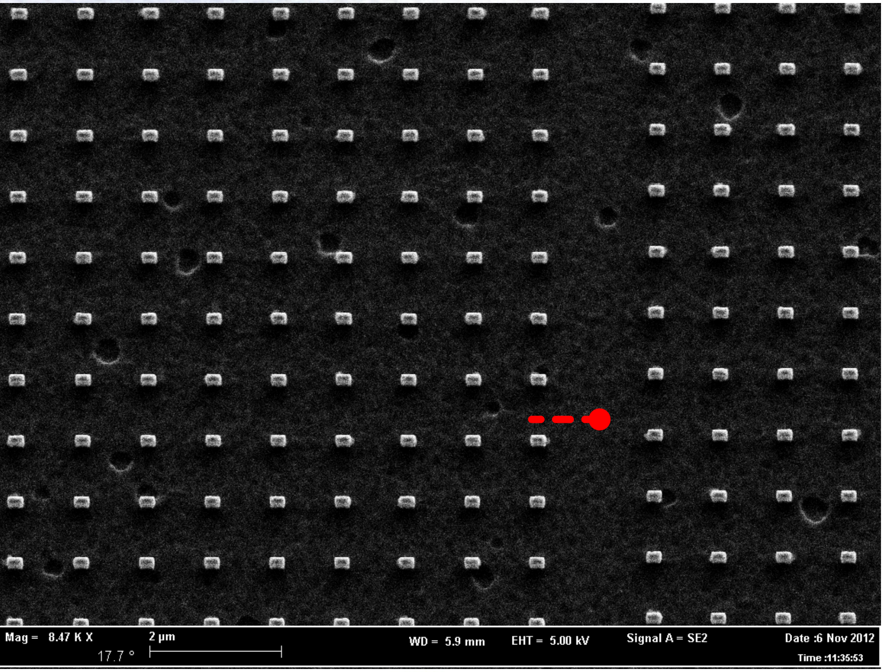
Positionlist NONAME.pls

Include: all Exclude: none

| ID | U/mm | V/mm | Attribute | Template | Comment | Options | Type | Pos1/um | Pos2/um | Pos3/um | Link | File | Layer | DoseFactor | FBMSArea/mm/s |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 0 | 0.050000 | 0.050000 | XN | UV | array | | EXPOSURE | 50.000 | 50.000 | | | %UserRoot%GDSII\210614\ARR | 0 | 1.000 | |
| 1 | 0.150000 | 0.050000 | XN | UV | array | | EXPOSURE | 50.000 | 50.000 | | | %UserRoot%GDSII\210614\ARR | 0 | 1.000 | |
| 2 | 0.250000 | 0.050000 | XN | UV | array | | EXPOSURE | 50.000 | 50.000 | | | %UserRoot%GDSII\210614\ARR | 0 | 1.000 | |
| 3 | 0.050000 | 0.150000 | XN | UV | array | | EXPOSURE | 50.000 | 50.000 | | | %UserRoot%GDSII\210614\ARR | 0 | 1.000 | |
| 4 | 0.150000 | 0.150000 | XN | UV | array | | EXPOSURE | 50.000 | 50.000 | | | %UserRoot%GDSII\210614\ARR | 0 | 1.000 | |
| 5 | 0.250000 | 0.150000 | XN | UV | array | | EXPOSURE | 50.000 | 50.000 | | | %UserRoot%GDSII\210614\ARR | 0 | 1.000 | |
| 6 | 0.050000 | 0.250000 | XN | UV | array | | EXPOSURE | 50.000 | 50.000 | | | %UserRoot%GDSII\210614\ARR | 0 | 1.000 | |
| 7 | 0.150000 | 0.250000 | XN | UV | array | | EXPOSURE | 50.000 | 50.000 | | | %UserRoot%GDSII\210614\ARR | 0 | 1.000 | |
| 8 | 0.250000 | 0.250000 | XN | UV | array | | EXPOSURE | 50.000 | 50.000 | | | %UserRoot%GDSII\210614\ARR | 0 | 1.000 | |

* 1: 4 1 Lot ID: WaferID: Slot:

# EBL on bare wafer: Final Array (SEM limitations)



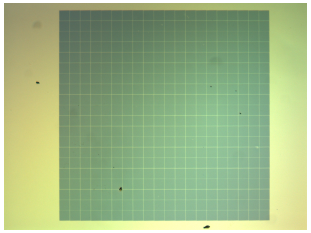

**Stitching Problem:**

Standard SEM stages have micrometric precision

Different WFs result overlapped or shifted

Solutions:

- Use of markers (error due to the user)
- Interferometric stage (error 10 nm)



Exposition time: >24h (array of 2*2 mm)



Clericò V, Masini L, Boni A, Meucci S, Cecchini M, Recchia F A, Tredicucci A and Bifone A 2014 Water-dispersible three-dimensional LC-nanoresonators PLoS One 9 e105

# EBL on 2D materials (EBL with markers)

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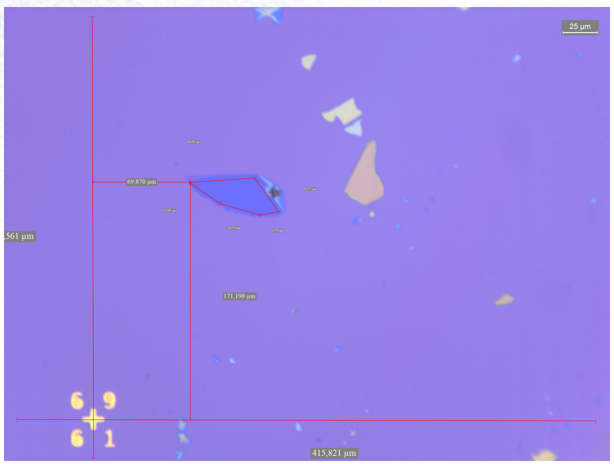
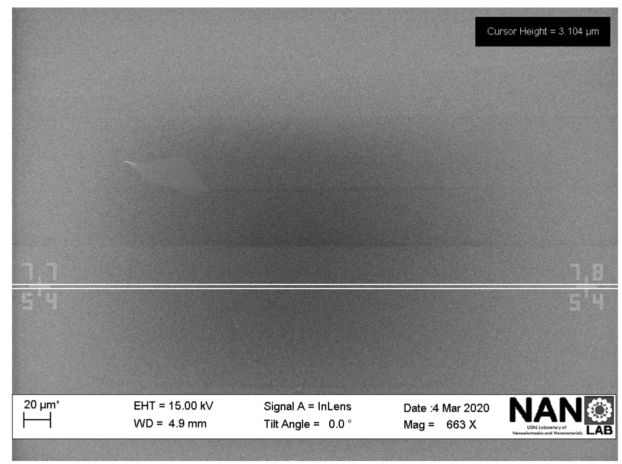
**WA=WF**

Determination of coordinates of flake (heterostucture) from the close marker

Optical microscope

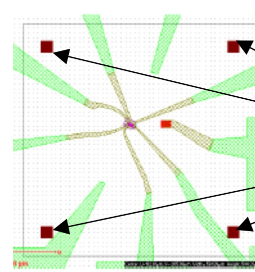



SEM




exposition

Preparation of the CAD and choose different WFs



Layer 63 is the manual marker layer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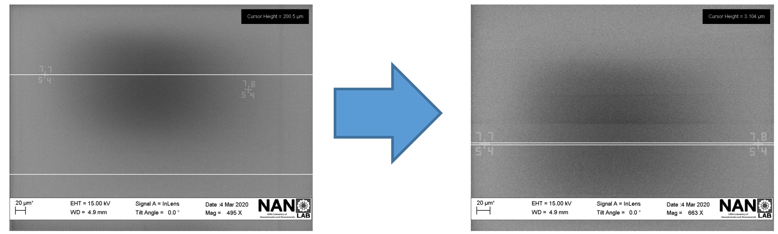
rednanolito

# EBL on 2D materials (EBL with markers)

Physical correction of angle:



Correction the global coordinates (correspondence between stage coordinates (X,Y) and the (U,V) coordinates of the EBL system):

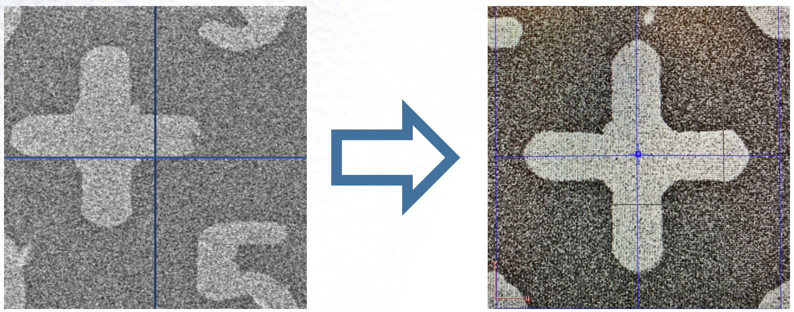
- Take a marker as a *global* origin
- Define the *global* angle the X(U)-axis (we need to define 2 points (here 2 markers) on the axis)

Define the plane (three points alignment) ‘close’ to the flake:

- The bottomleft marker (U,V) point (0,-400) m
- The bottomright corner marker is the second (U,V) point (example (400,-400) mm)
- The third (U,V) point at (0, 800) mm is chosen

# EBL on 2D materials (EBL with markers)

Before the exposition we select the layer 63 to correct the stage movement:



We manually correct the stage movement error

'Window' dimensions are chosen in the CAD

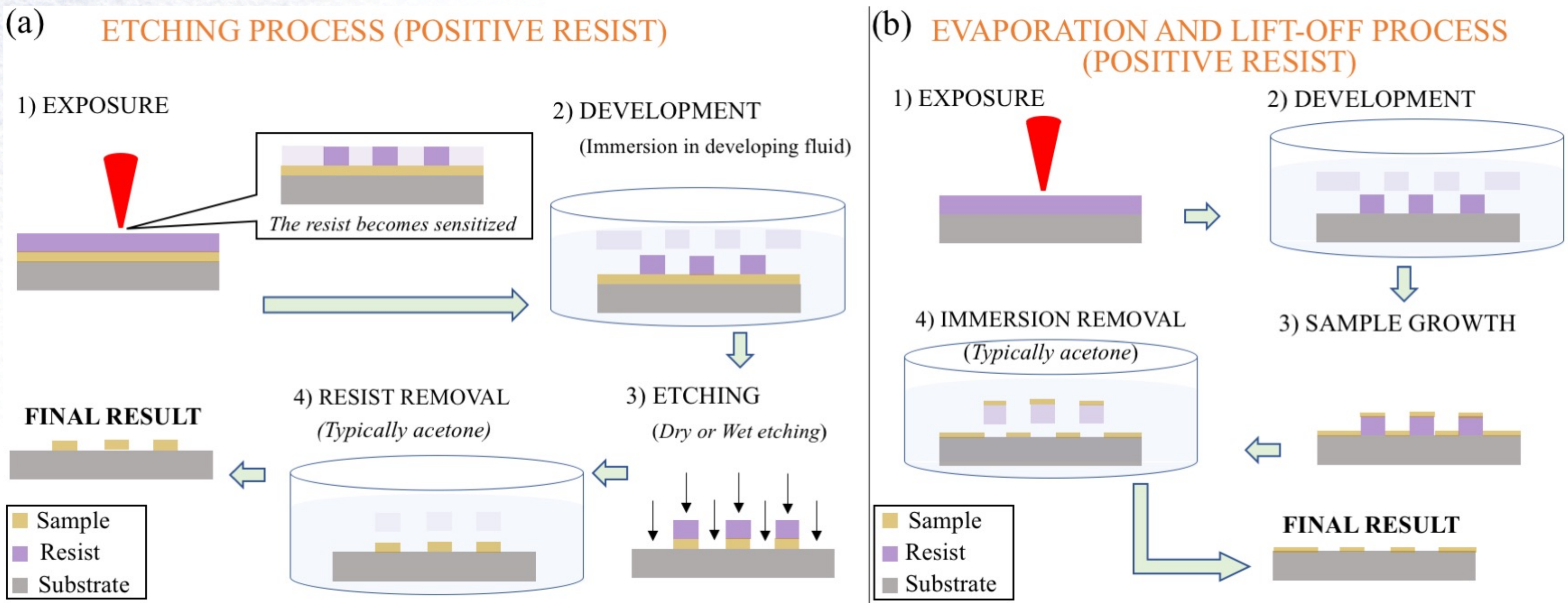
Exposition time: <1h

EBL is largely used for 2D materials devices

PD: For the whole device normally we have to use different aperture and differentent WFs (and some times different EBL processes)

If we cannot see the center of the marker but only a 'part' of it…we can repeat the alignment markers.

# Use of EBL processes






*Electron beam lithography and its use on 2D materials* (Chapter 3), Book Nanofabrication: nanolithography techniques and their applications Institute of Physics, UK, 2020.

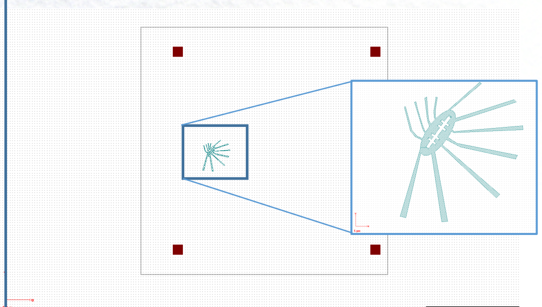
# EBL on 2D materials (EBL with markers)

Ex. Nanoconstriction in encaspulated graphene:

EBL for bar definition

(WF 500 um)

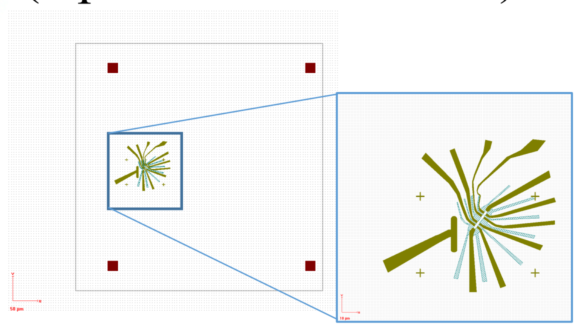
(Aperture 10 um)

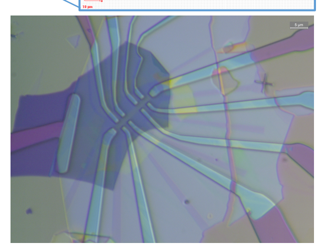


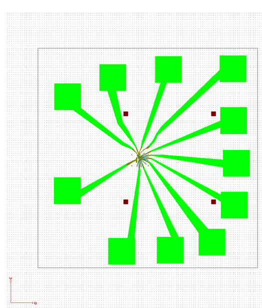
EBL for bar contacts and Pads definition

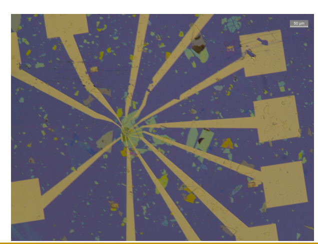
(WF 500 um/1000 um)

(Aperture 10um/60um)





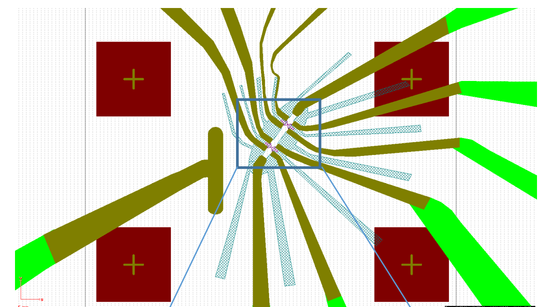


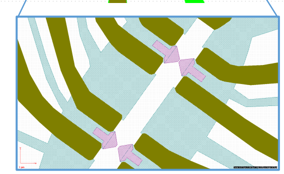


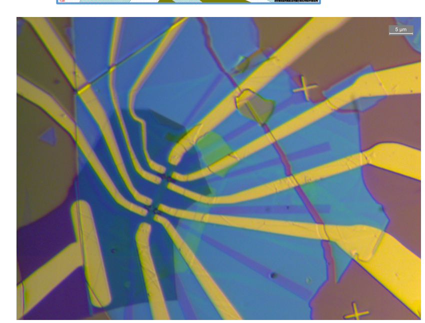
EBL for NCs definition

(WF 100 um)

(Aperture 7.5 um)







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# Some results
# (use of EBL in 2D materials)

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www.nature.com/scientificreports

SCIENTIFIC REPORTS
natureresearch

## Quantum nanoconstrictions fabricated by cryo-etching in encapsulated graphene

V. Clericò[1], J. A. Delgado-Notario[1], M. Saiz-Bretín[2], A. V. Malyshev[2,3], Y. M. Meziani[1], P. Hidalgo[2], B. Méndez[2], M. Amado[1], F. Domínguez-Adame[2] & E. Diez[1]

ORIGINAL PAPER

physica status solidi pss a

Graphene Nanoconstrictions

www.pss-a.com

## Quantized Electron Transport Through Graphene Nanoconstrictions

*Vito Clericò, Juan A. Delgado-Notario, Marta Saiz-Bretín, Cristina Hernández Fuentevilla, Andrey V. Malyshev, Juan D. Lejarreta, Enrique Diez, and Francisco Domínguez-Adame**

COMMUNICATIONS PHYSICS

ARTICLE

https://doi.org/10.1038/s42005-020-00460-9 OPEN

Check for updates

## Excitons, trions and Rydberg states in monolayer $MoS_2$ revealed by low-temperature photocurrent spectroscopy

Daniel Vaquero[1], Vito Clericò[1], Juan Salvador-Sánchez[1], Adrián Martín-Ramos[1], Elena Díaz[2], Francisco Domínguez-Adame[2], Yahya M. Meziani[1], Enrique Diez[1] & Jorge Quereda[1]

## Asymmetric dual-grating gates graphene FET for detection of terahertz radiations

Cite as: APL Photonics **5**, 066102 (2020); https://doi.org/10.1063/5.0007249
Submitted: 10 March 2020 . Accepted: 26 May 2020 . Published Online: 09 June 2020

J. A. Delgado-Notario, V. Clericò, E. Diez, J. E. Velázquez-Pérez, T. Taniguchi, K. Watanabe, T. Otsuji, and Y. M. Meziani

# PART III:

# Temperature dependence (and cryoetching) for edge definition (in 2D materials)

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# Edges contacts in 2D materials

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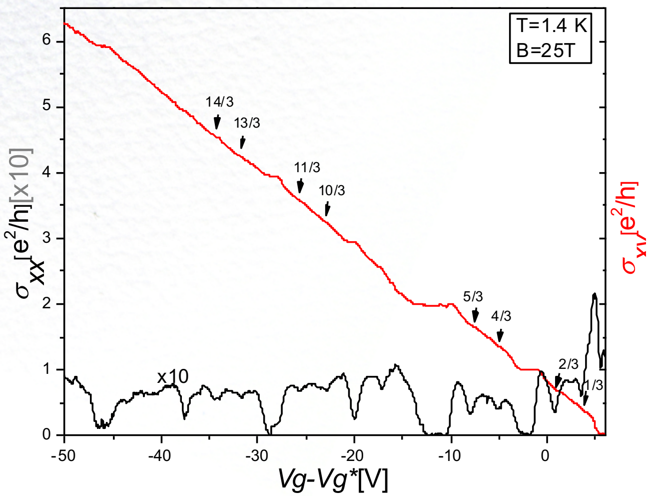
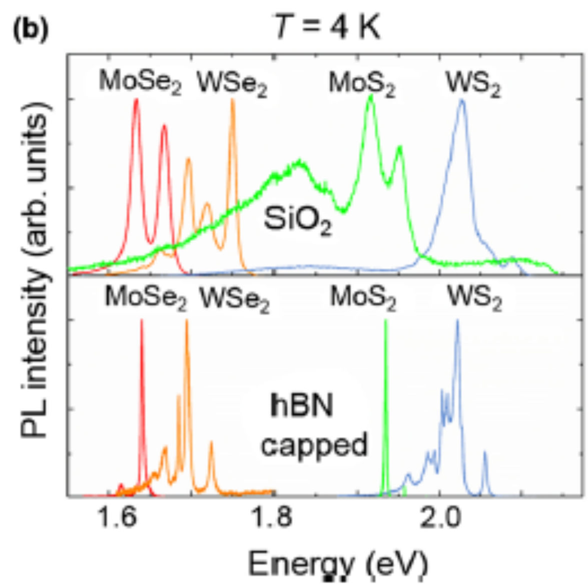
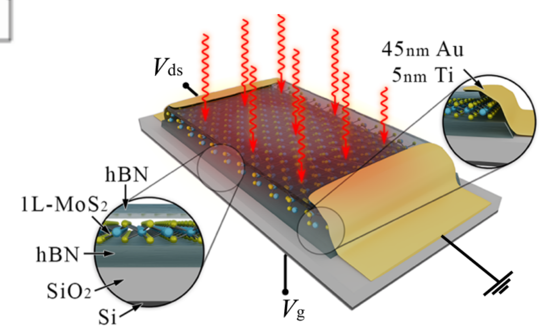
Encapsulated 2D materials devices have much higher quality and/or different phenemena:

**FQHE**



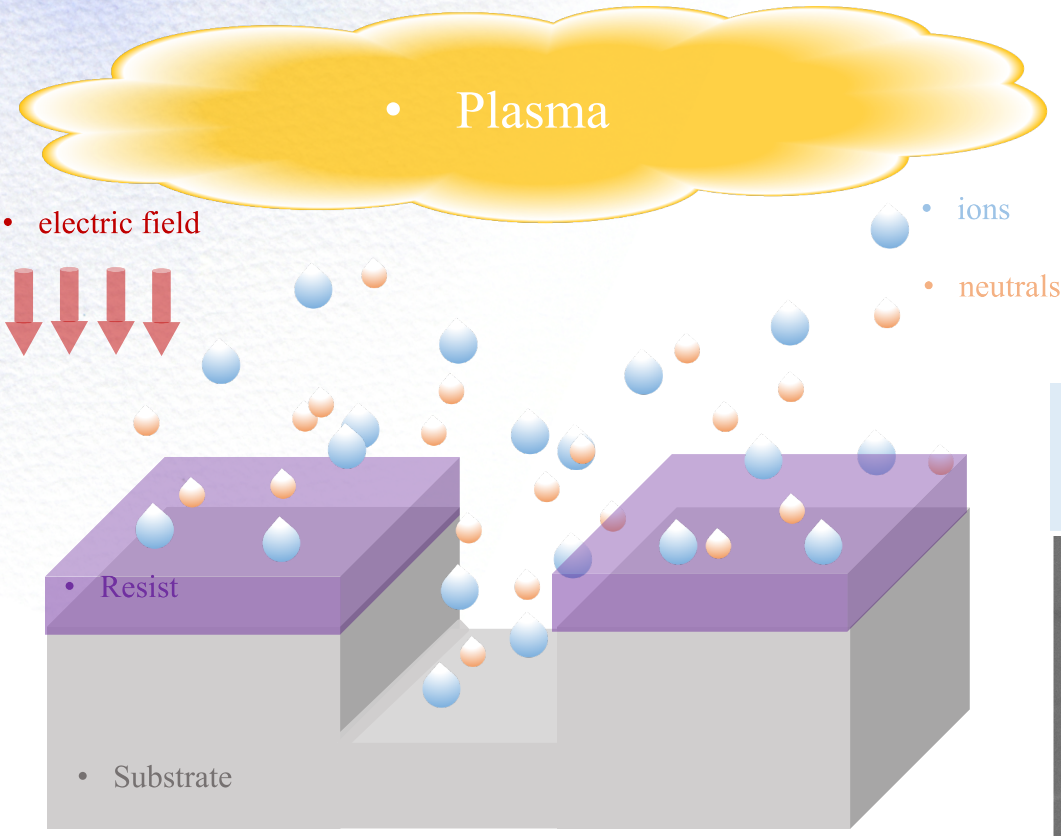

Etching processes in 2D materials have a fundamental role (edge contacts):



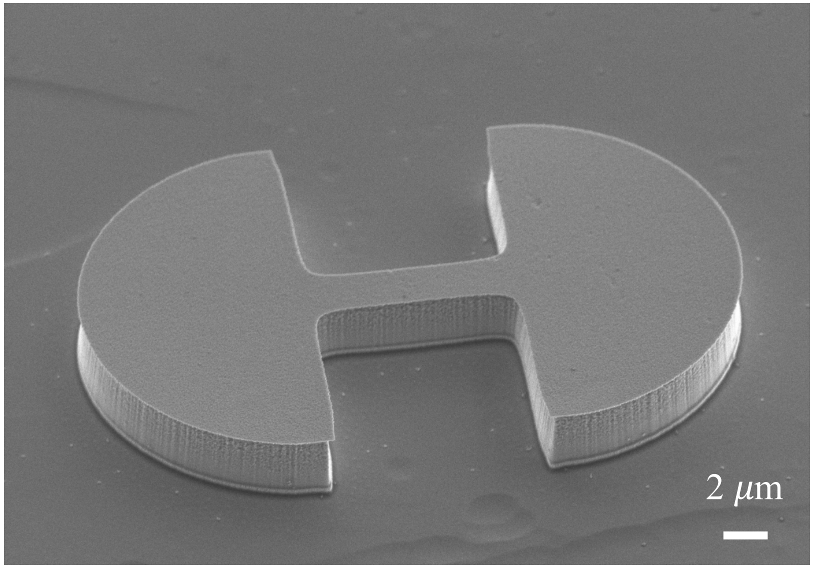



-Excitonic Linewidth Approaching the Homogeneous Limit in -Based van der Waals HeterostructuresF Cadiz, E Courtade, C Robert, G Wang, Y Shen, H Cai, T Taniguchi, ... Physical Review X 7 (2), 021026
-Excitons, trions and Rydberg states in monolayer MoS 2 revealed by low-temperature photocurrent spectroscopyD Vaquero, V Clericò, J Salvador-Sánchez, A Martín-Ramos, E Díaz, Communications Physics 3 (1), 1-8

# Dry etching: ICP-RIE




Normally a vertical etching is desiderable:



# Our ICP-RIE

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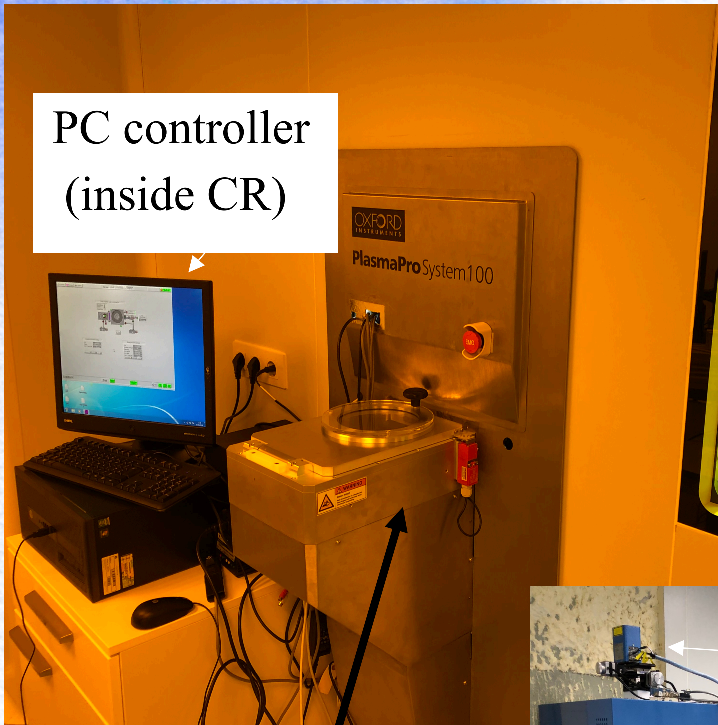
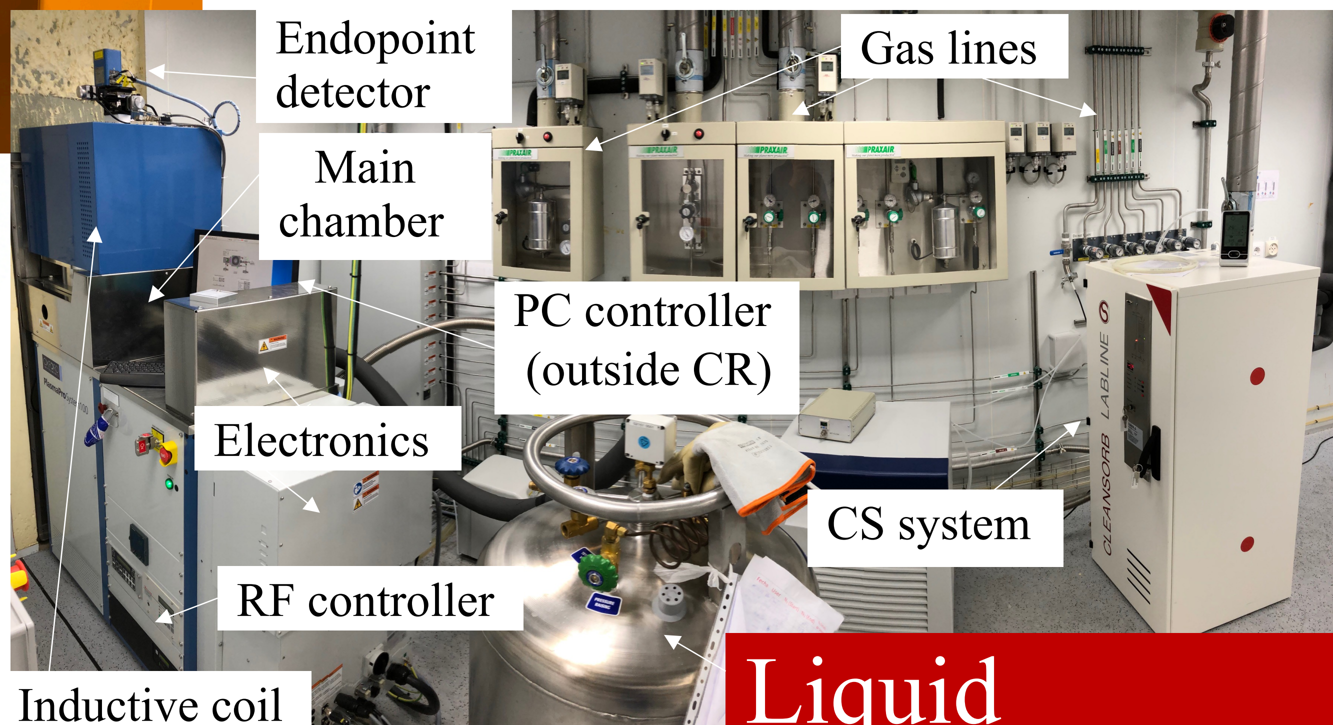


# Control the etching profile on Silicon (Similar recipe)

$SF_6$ and $O_2$ gases

[redacted]

**Pressure**

Adjust pressure to control profile, rate & undercut

Increase pressure $\rightarrow$ increased etch rate / selectivity

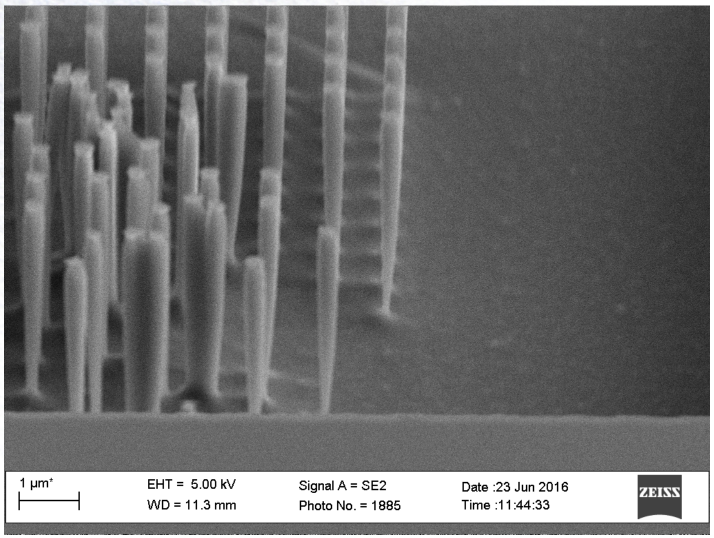
profile harder to control difficult to control

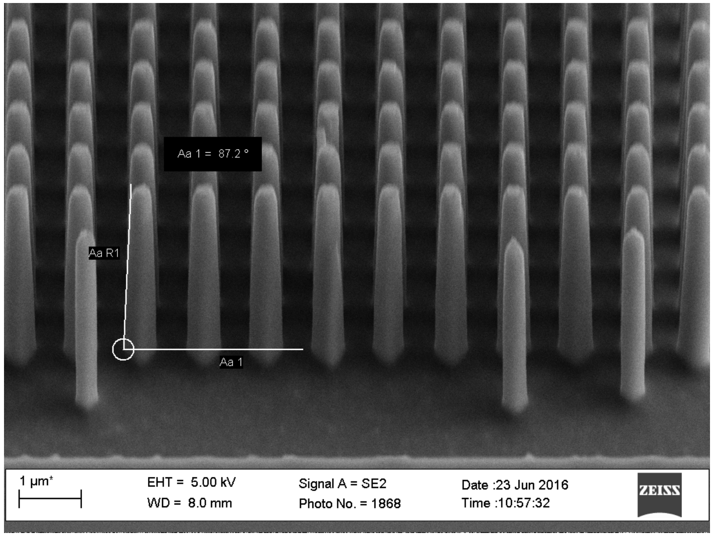
**Temperature**

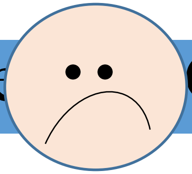
Higher temperature $\rightarrow$ more undercut

Lower temperature $\rightarrow$ positive profiles

# Oxigen on Si nanowires Profile




Incre  O2

Control the profile with T

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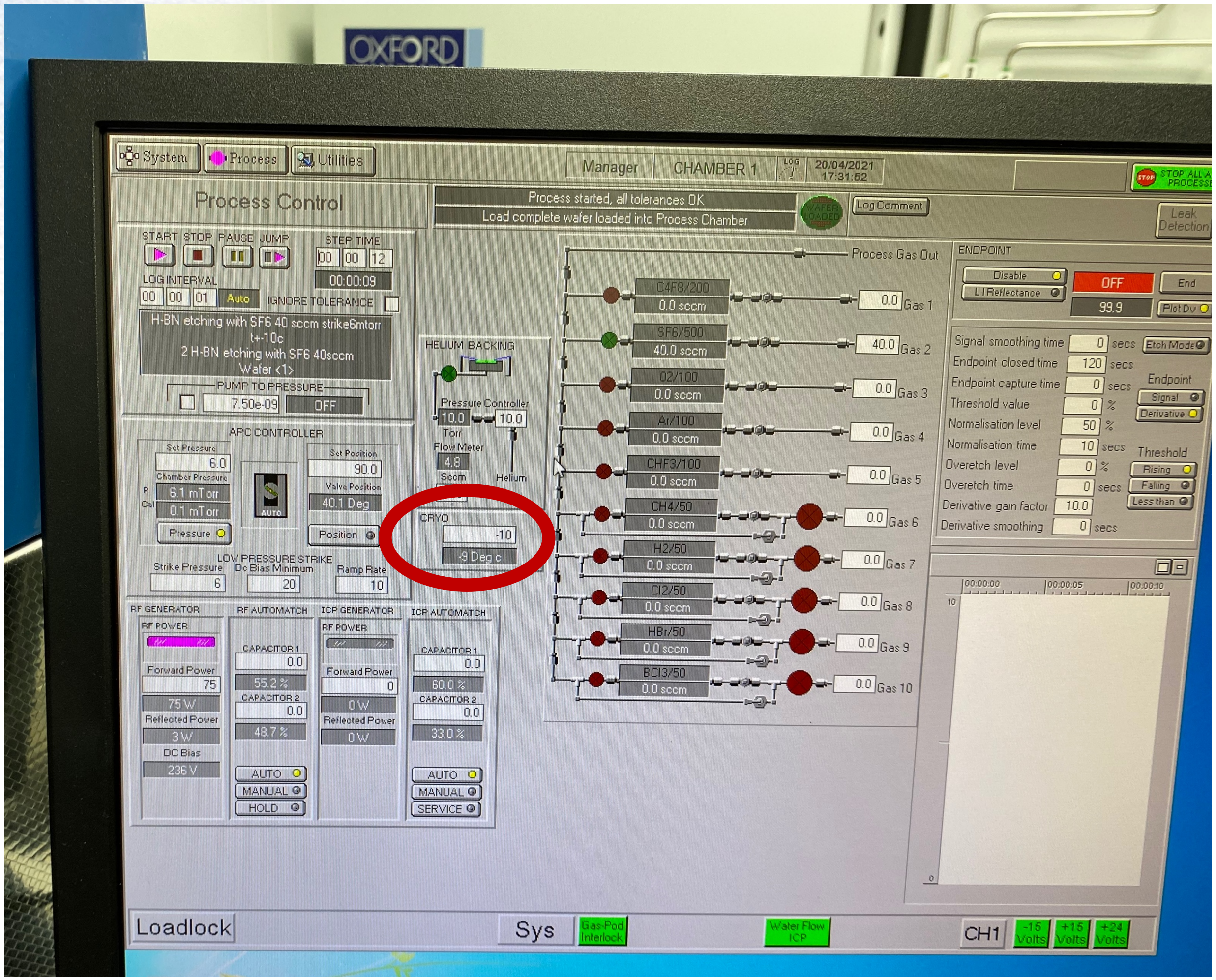
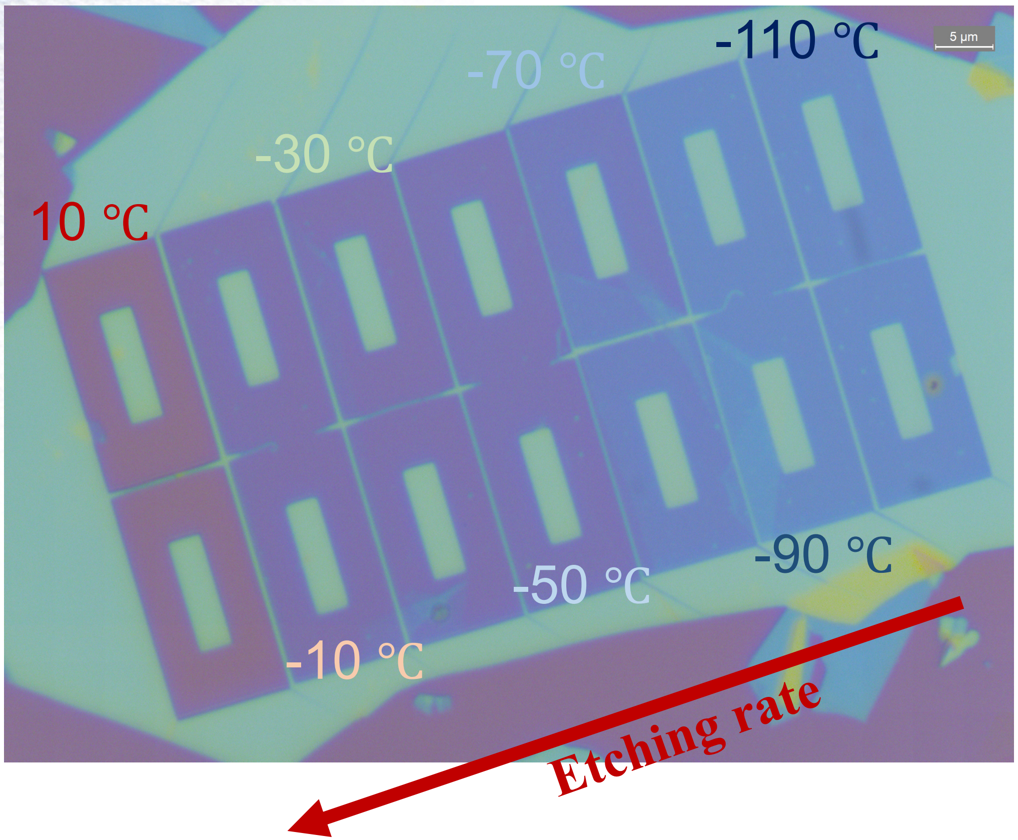
# Recipe for edge contacts



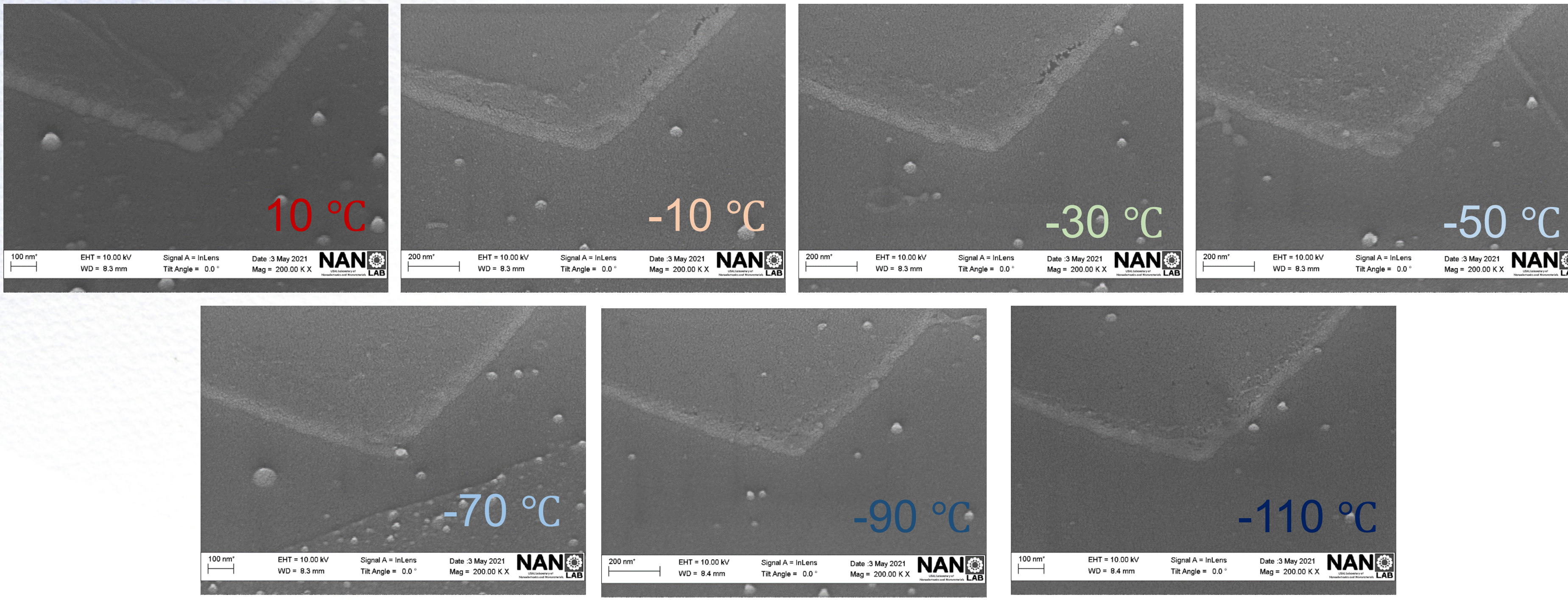
# Etching on hBN flake (Different T)




Very thin hBN flake (30 nm) (Exfoliation by Juan Salvador Sánchez)

Etching rate change (from 2 nm/s to 3.6nm/s)

# SEM image
# (Tilted image)




**InLens Detector**
**(tilt 45)**

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# SEM image (Tilted image)

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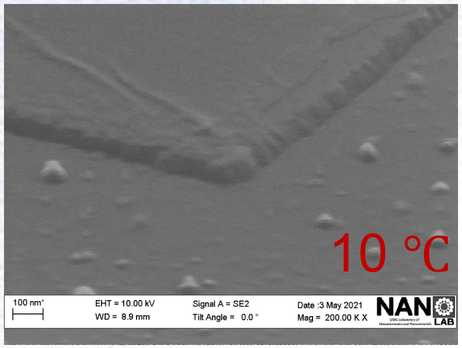
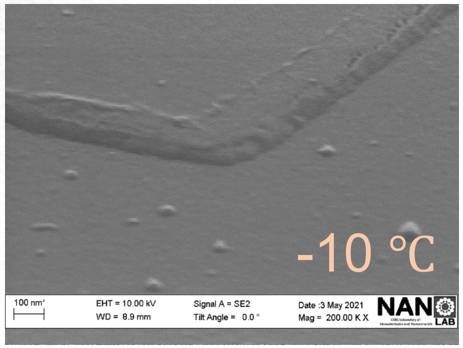
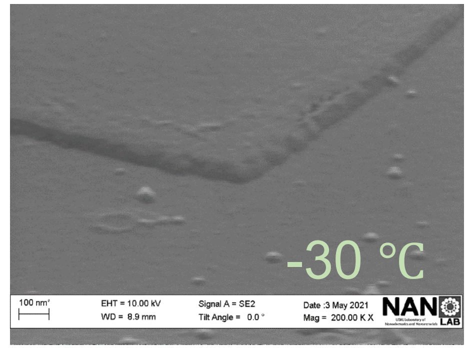
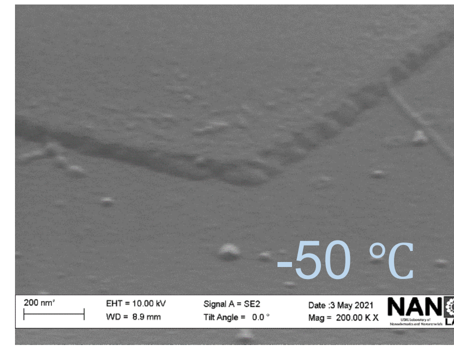
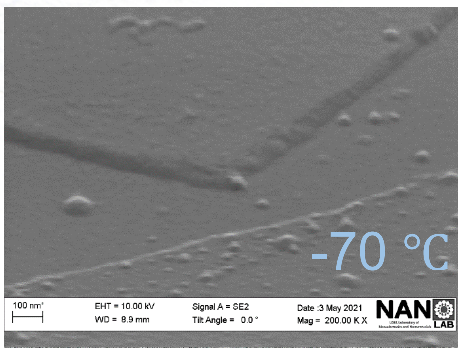
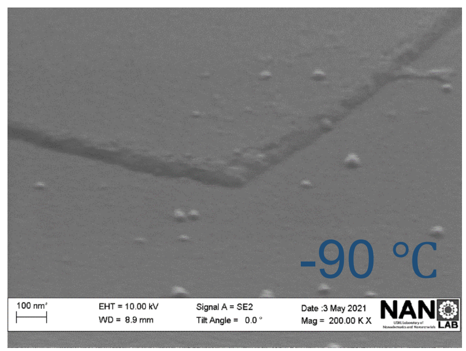


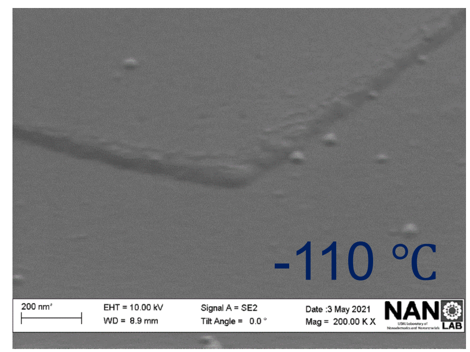



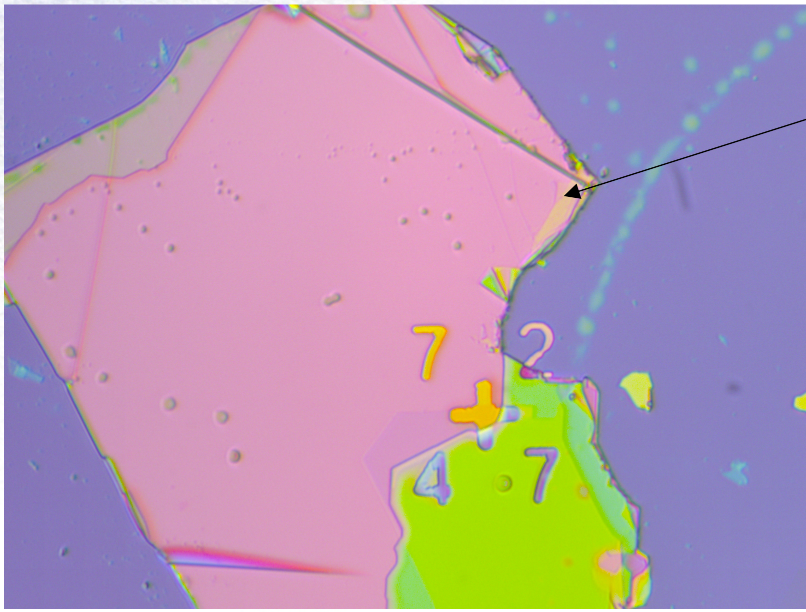




**E-T SE2 Detector (tilt 60)**

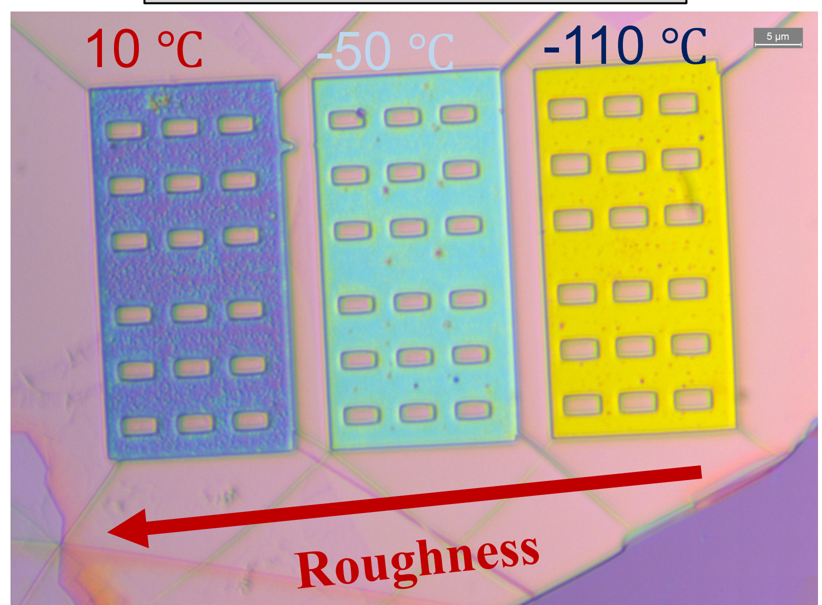
# Etching on hBN flake (Different T)




Very thick hBN flake (230 nm)

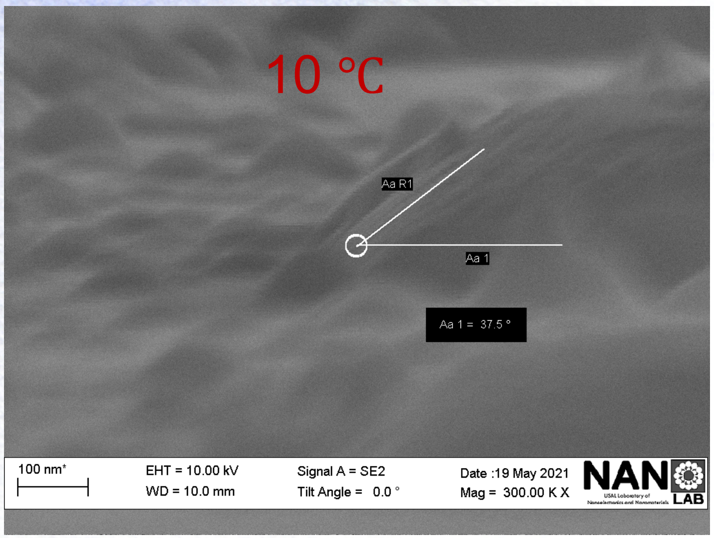
(Exfoliation by Dr. Juan Antonio Delgado)

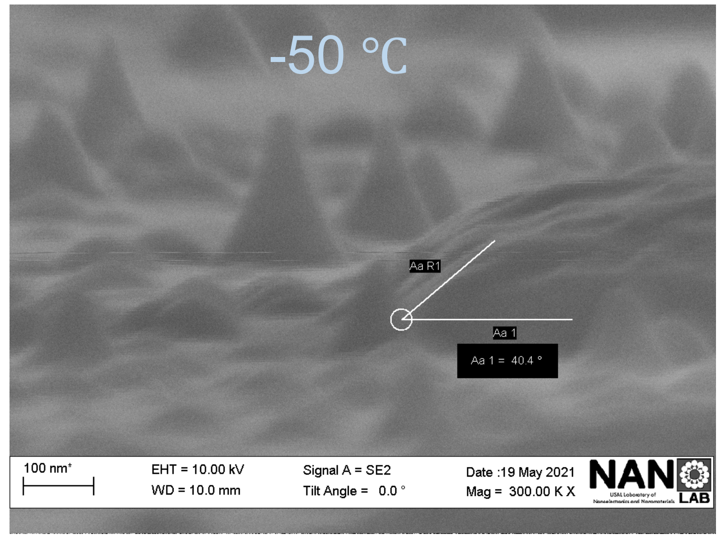
After etching for t=60s




Roughness reduced at low T

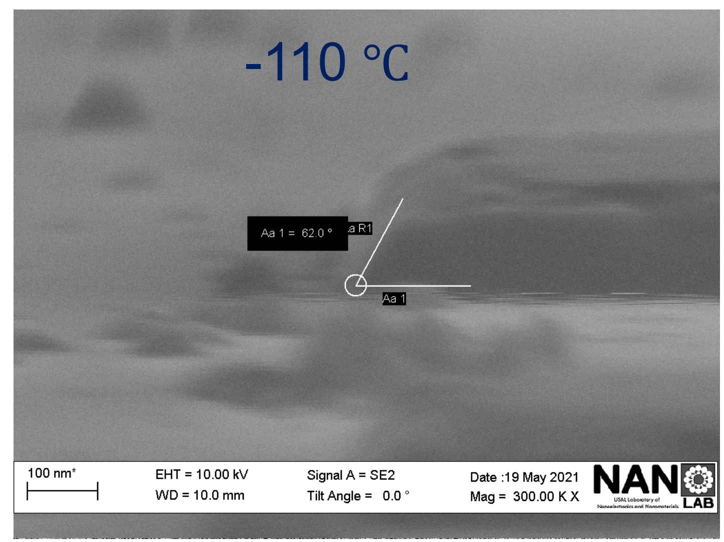
# SEM image (Cross Section 85)



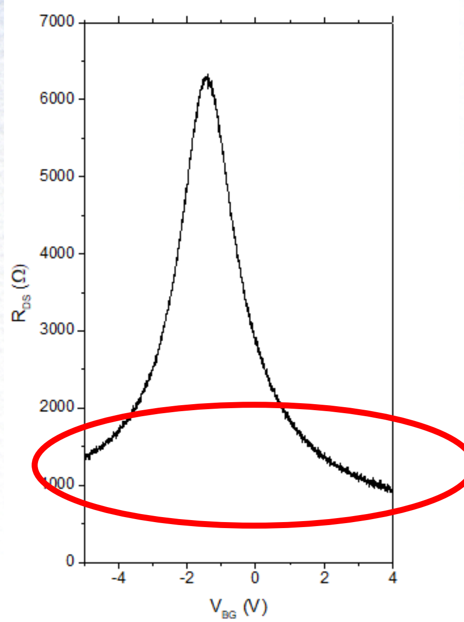




Profile is more vertical decreasing the temperature

Roughness is reduced decreasing the temperature

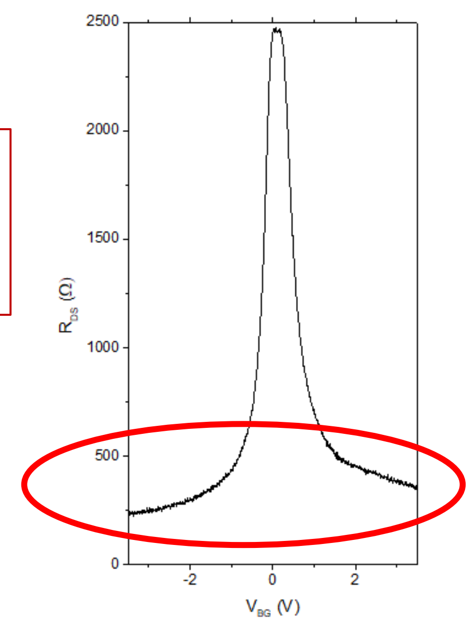


# Contact Resistance (Recipe 10 °C)




T-room

2-probes measurement




Encapsulated monolayer graphene

(on silicon oxide)

Encapsulated bilayer graphene

(on graphite backgate)

**Next Step:**

**Study of the T-dependence etching process on the contact resistance of 2D materials**

# Etching for smooth definition of Nanoconstrictions in graphene

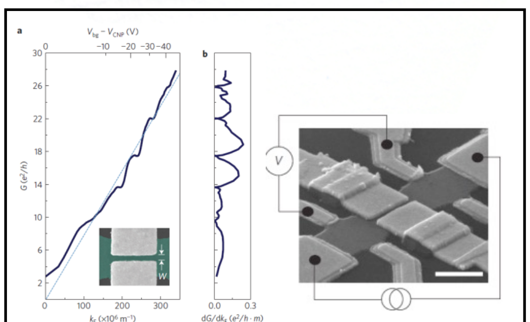
USAL Laboratory of Nanoelectronics and Nanomaterials

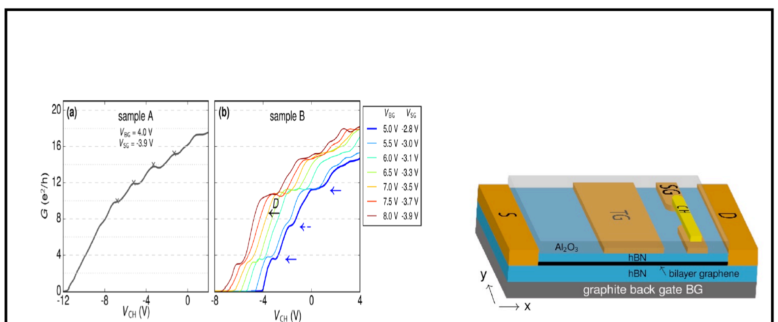
**Controlled etching can be used also to define nanostructures in 2D materials**

Conductances quantized in multiples of the fundamental quantum 2e2/h are the hallmark of ballistic quantum transport in nanostructures such as semiconductor quantum point contacts

**Two ways of achieving quantized conductance in graphene (natural 2D material and high mobility) without B**

**Electrostatic potencial gates**





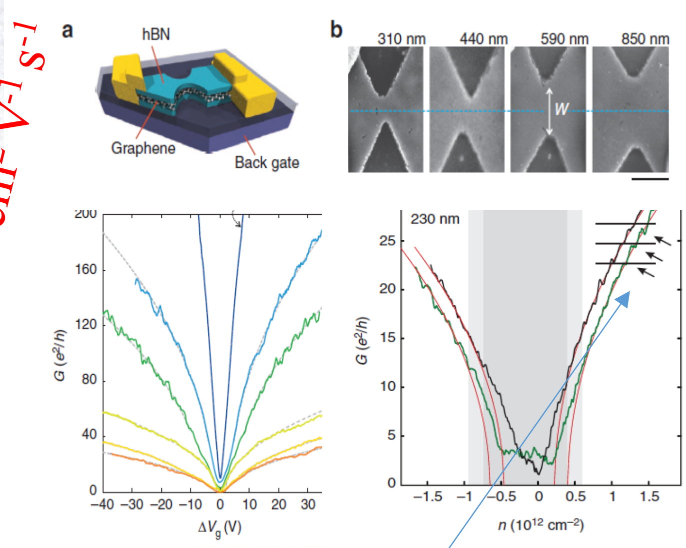
**Physically etched Nanoconstrictions (NCs)**


-Kim, M., Choi, JH., Lee, SH. *et al.* Valley-symmetry-preserved transport in ballistic graphene with gate-defined carrier guiding. *Nature Phys* **12,** 1022–1026 (2016)

-Overweg, H., Eggimann, H., Chen, X., Slizovskiy, S., Eich, M., Pisoni, R., Lee, Y., Rickhaus, P., Watanabe, K., Tanaguchi, T., Fal'ko, V., Ihn, T., & Ensslin, K. (2017). Electrostatically Induced Quantum Point Contacts in Bilayer Graphene. *Nano Letters.*

# Physically etched NCs

$\mu > 100.000\ cm^2 V^{-1} s^{-1}$




Quantization!
(spikes)

Semiclassical formula:

$$G = \left(\frac{2e^2}{h}\right)\frac{2Wk_F}{\pi}c_0$$

$c_0 = 0.56$

Low transmission coefficent due to roughness!

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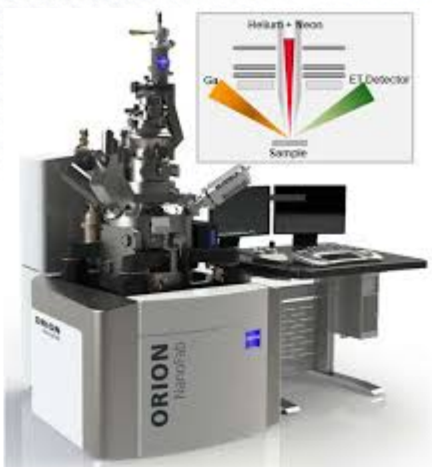
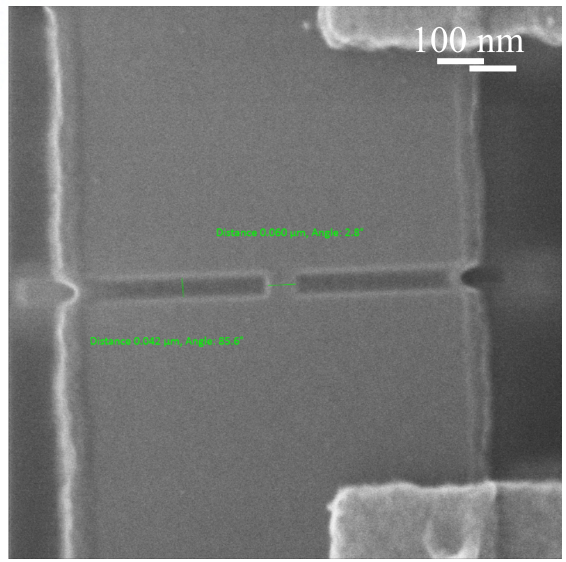
June 29-30 & July 1st 2021
UNIVERSITY OF SALAMANCA

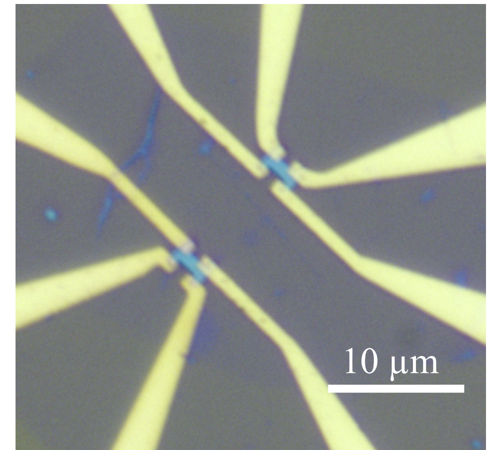
Terrés, B., Chizhova, L., Libisch, F. *et al.* Size quantization of Dirac fermions in graphene constrictions. *Nat Commun* **7,** 11528 (2016). https://doi.org/10.1038/ncomms11528

# How to define the NC with low roughness?

Carl Zeiss Center Peabody (USA)



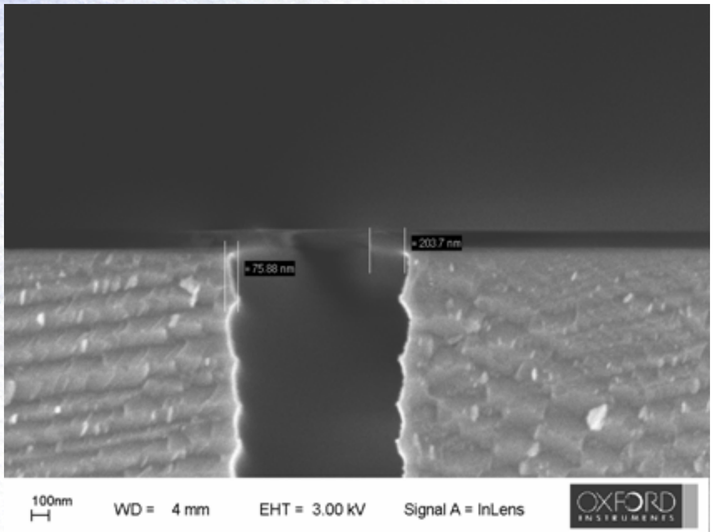




After the etching process with Orion Nanofab Helium Microscope…

The devices died ☹

**New way: Cryoetching**

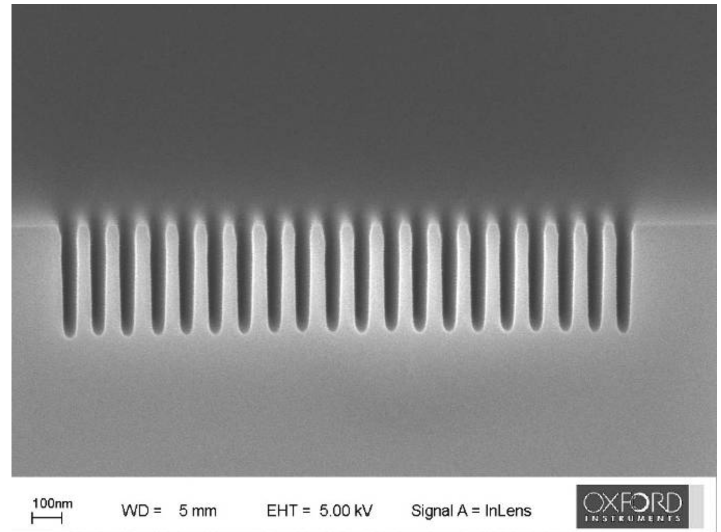
# Comparison with Si




Bosch 500nm feature

Roughness is ~100nm but nanoscale is 100nm and below!

Therefore insufficient for most nanoscale applications




Cryo 50nm feature

Roughness <5nm

Shallow etches controllable

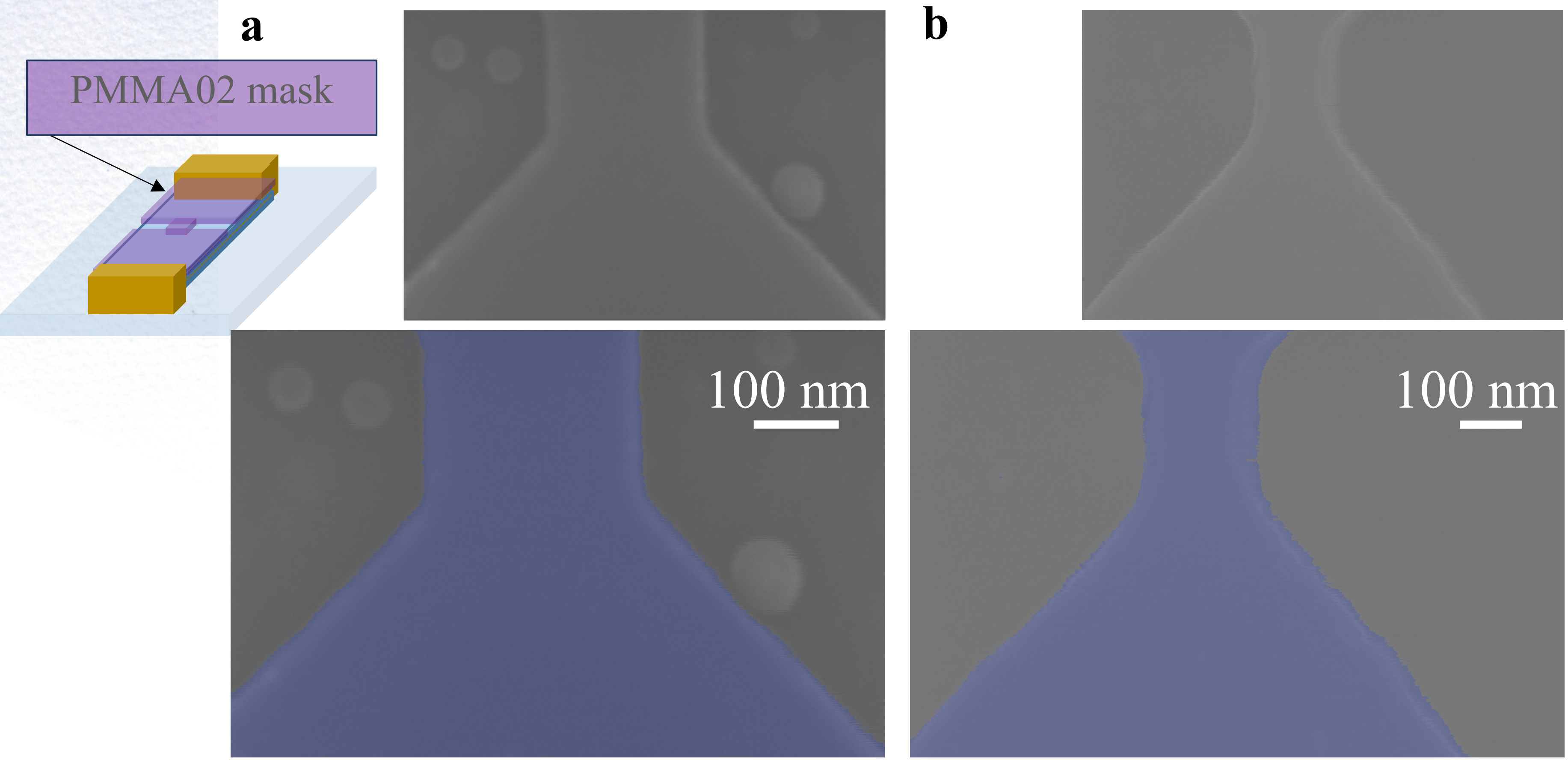
<u>Highly suitable for nanoscale applications</u>

# Qualitative Comparison with no cryoetched GNC



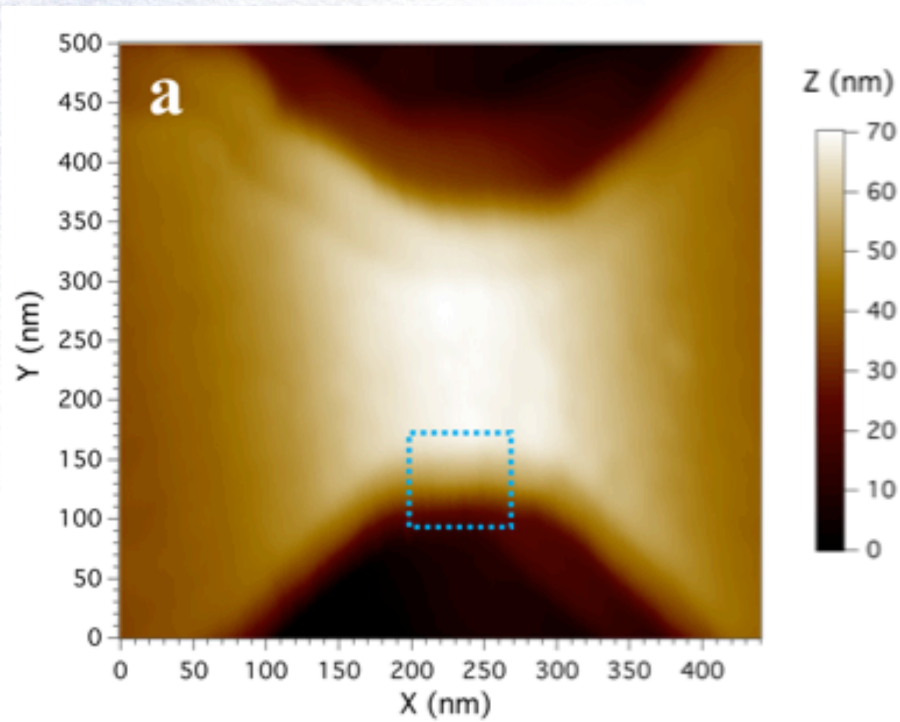
a. cryo-etched at -110K

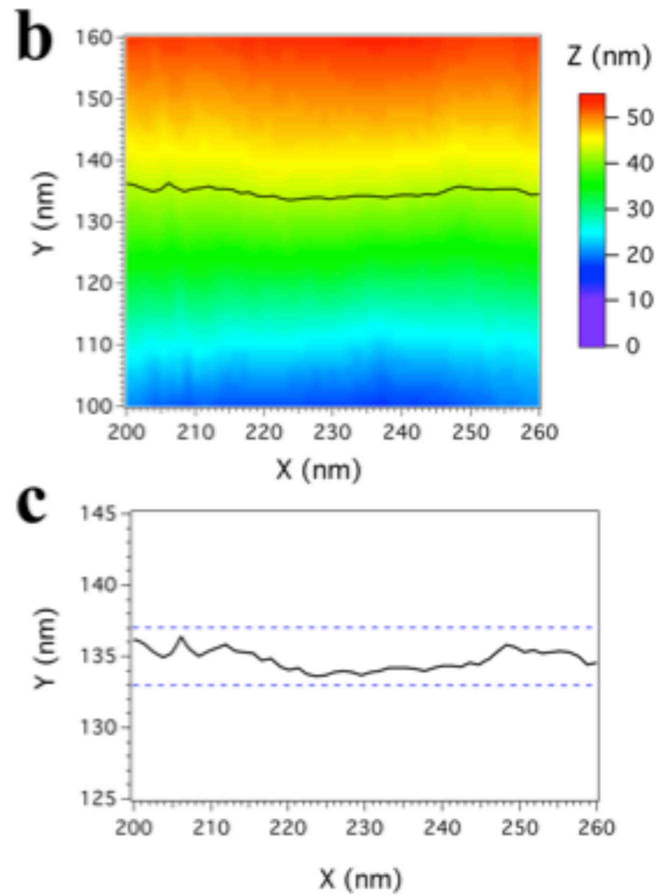
b. etched at 293K




We introduce also Ar for a more vertical etching

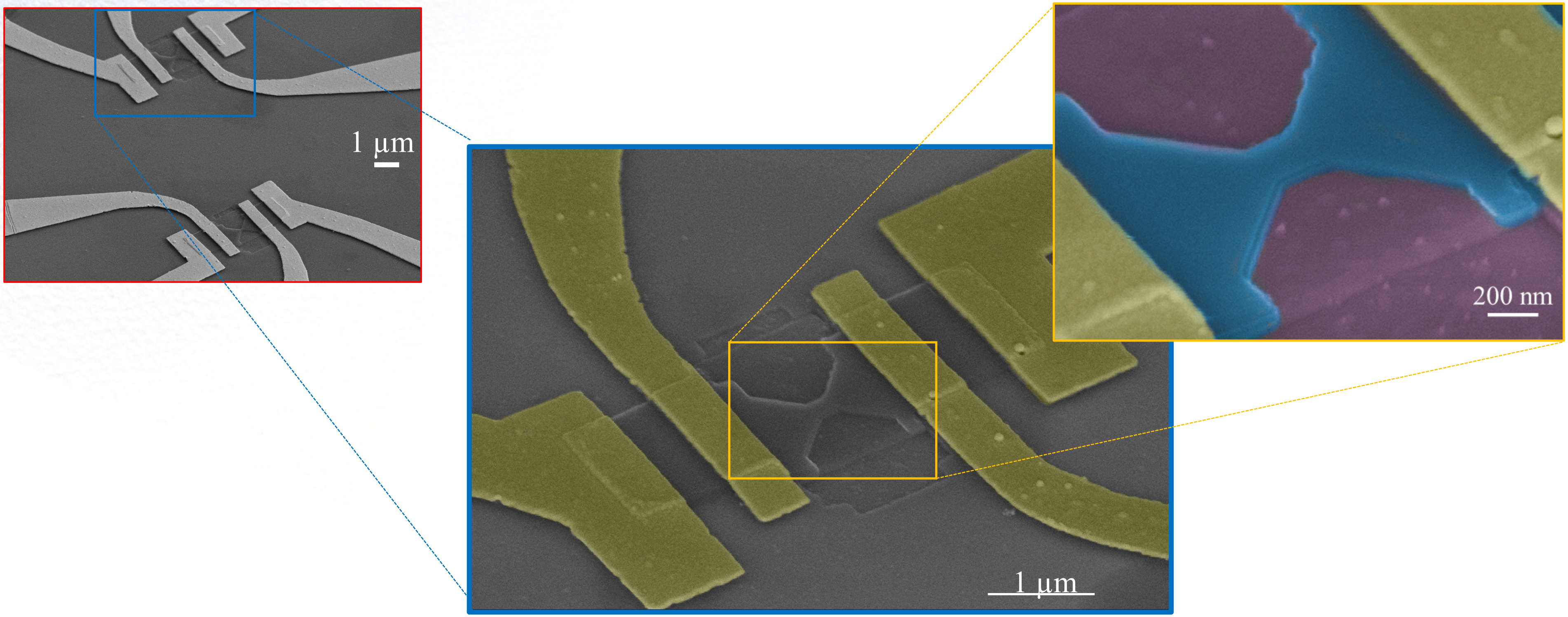
# Quantitative Measurement: AFM



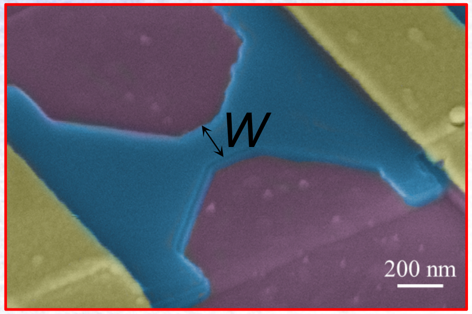




Low roughness < ±2nm

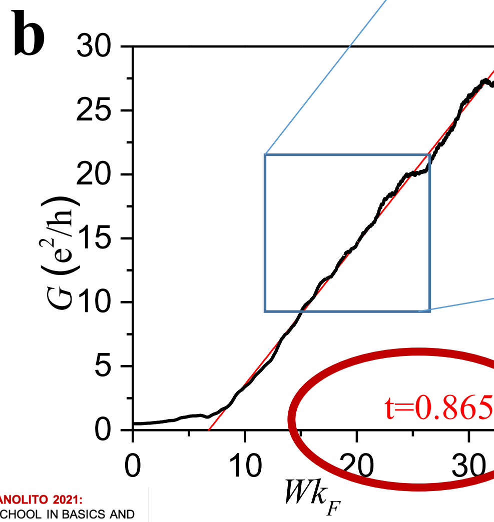
# Typical Device

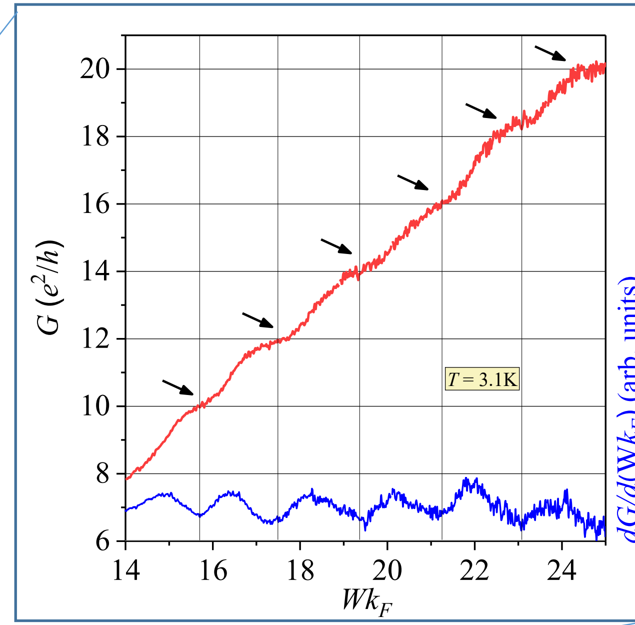


# NC with W=200 nm




$$G = \left(2\frac{e^2}{h}\right)\frac{2Wk_F}{\pi}t$$




Quantized Conductance of $\frac{2e^2}{h}$

Transmission coefficent close to 1:low roughness

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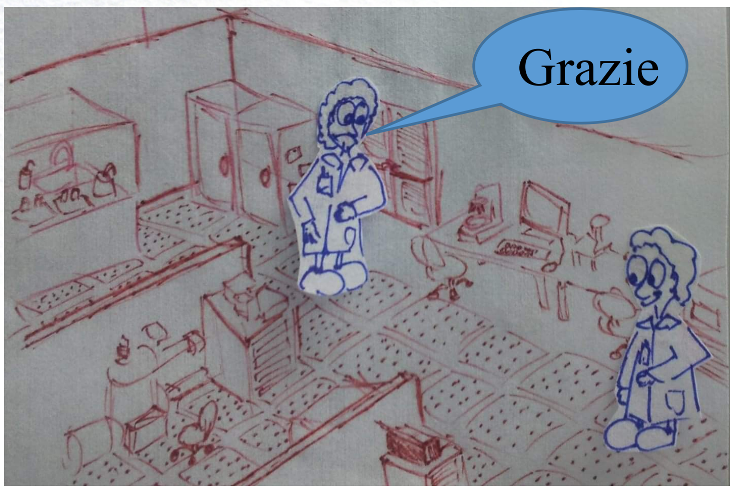
# ACKNOWLEDGEMENTS

## Thank you to all the people of NEST (Pisa)




Drawing on Texwipe of CR at NEST(Pisa) by Dr. María José Martínez-Pérez

## Thank you to all the people of Nanolab group


This is only the more 'complete' photo ☺

# Thank you for your attention and enjoy Salamanca ☺

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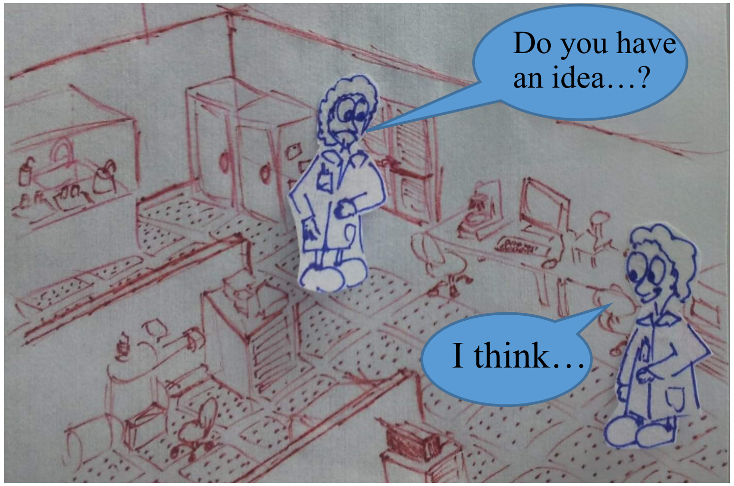
# Knowledgement of a process (in CR)

0. No Knowledgment

1. Study/have idea of a process

2. See ‘somebody’ doing the process

3. Do the process by yourself




Drawing of CR at NEST(Pisa) by Dr. María José Martínez-Pérez

# Other detectors



## - Backscattered Electron Detector (AsB):

sensitive to the variation of atomic number in the elements present in the simple (used to observe changes in the chemical composition of the specimen). The AsB detector comprises four quadrants and permits selection of images with topographic and compositional contrast. Moreover, its high-angle working mode allows us to observe structural contrast in crystalline samples.

## - Backscattered Electron In-lens Detector (EsB):

provides a pure backscattered signal with no secondary electron contamination and very low acceleration potential. It gives a higher Z-contrast than any other backscattered detector, and it is the only one that can select the electrons according to their energy, enabling differentiation between elements which are only distinguished by a few atoms. It is also capable of working at very low voltage (in the same range as the secondary in-lens detector), which makes it ideal for sensitive samples.

## - X-Ray Dispersive Energy Detector (EDS):

receives x-rays from each surface point the electron beam passes over. As the x-rays scattered energy is a characteristic of each chemical element, it provides qualitative and quantitative analytical information about selected points, lines, or areas on the surface of the sample.



[http://www.upv.es/entidades/SME/info/859071normali.html](http://www.upv.es/entidades/SME/info/859071normali.html)

# Resist: Contrast and Sensitivity

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**Contrast** and **Sensitivity**

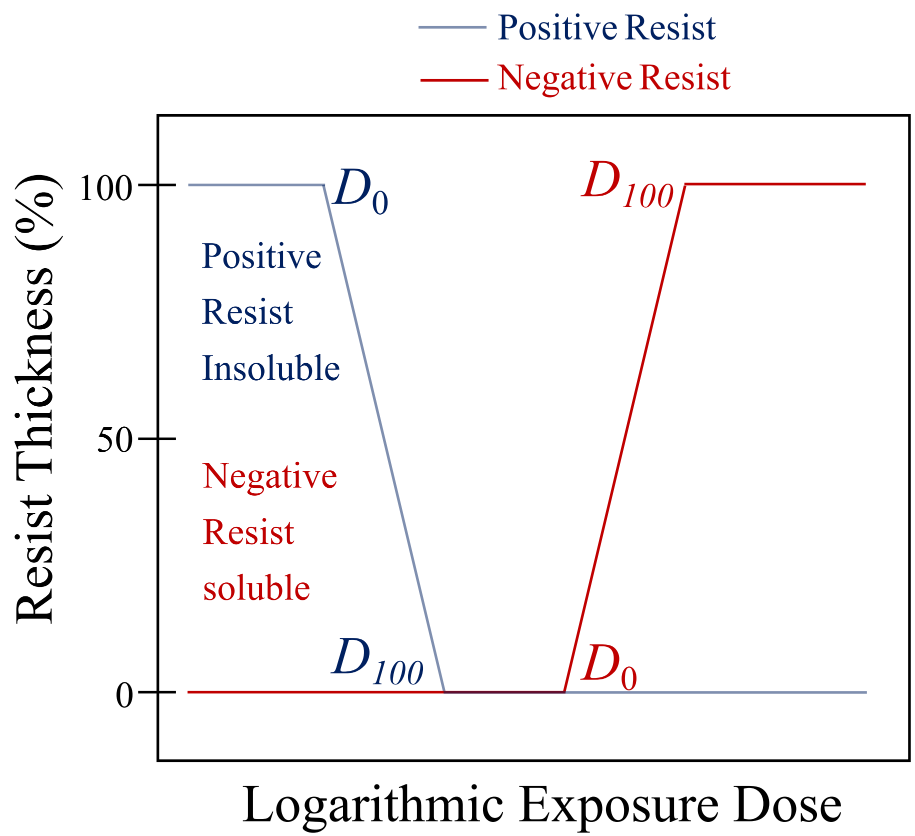
correlated:

$$\gamma = \frac{1}{\log \frac{D_{100}}{D_0}}$$

Contrast value

Highest exposition Dose (not affecting resist)

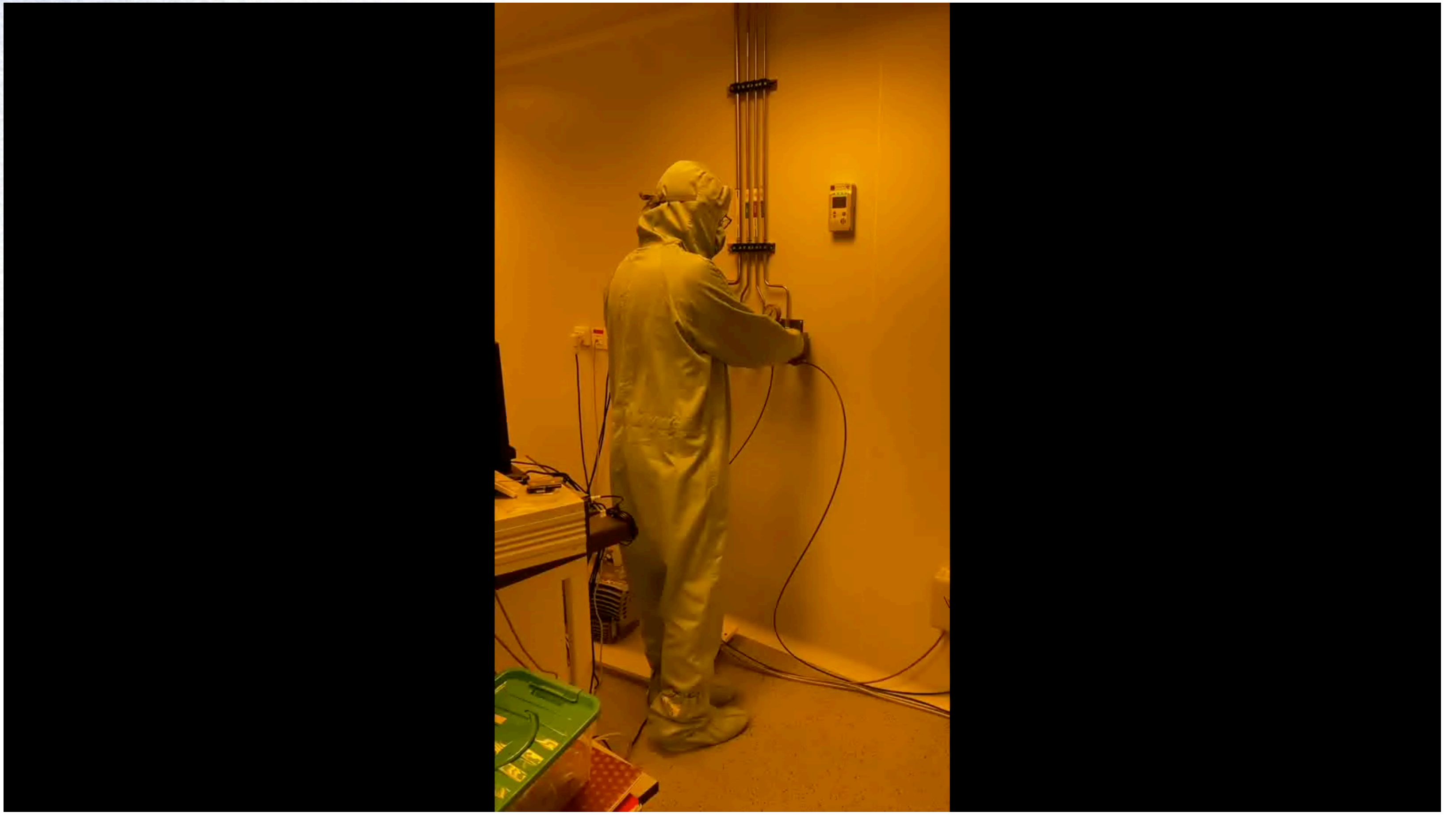
Minimum dose for a complete exposition of the resist






Zheng Z 2008 Nanofabrication by charged beams Nanofabrication Principles, Capabilities and Limits (New York: Springer) ch 3

# Beginning a SEM imaging (or EBL)

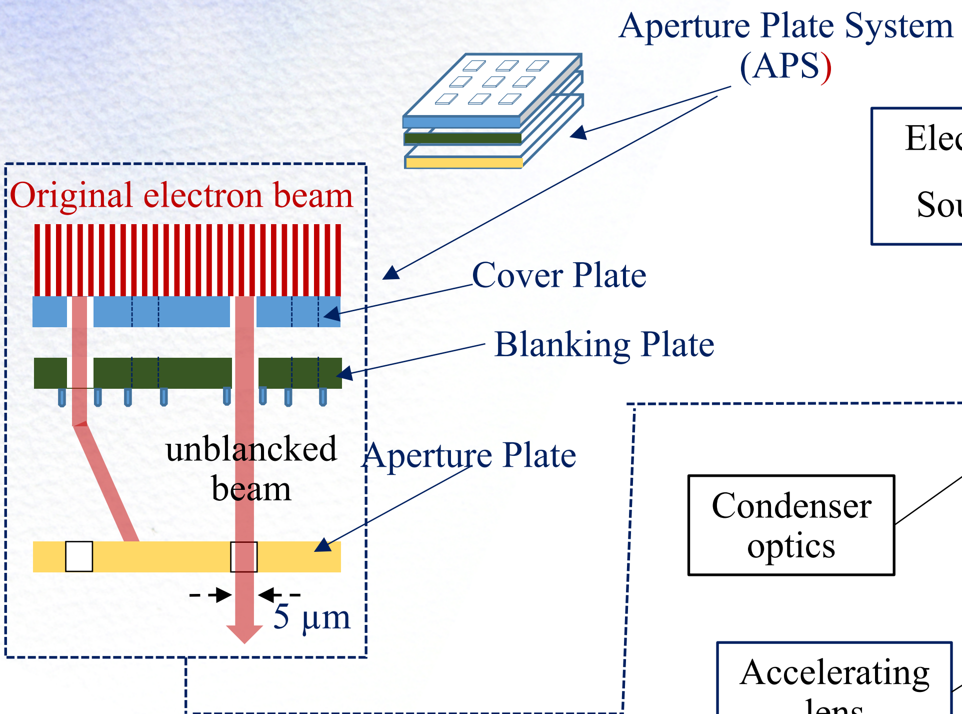


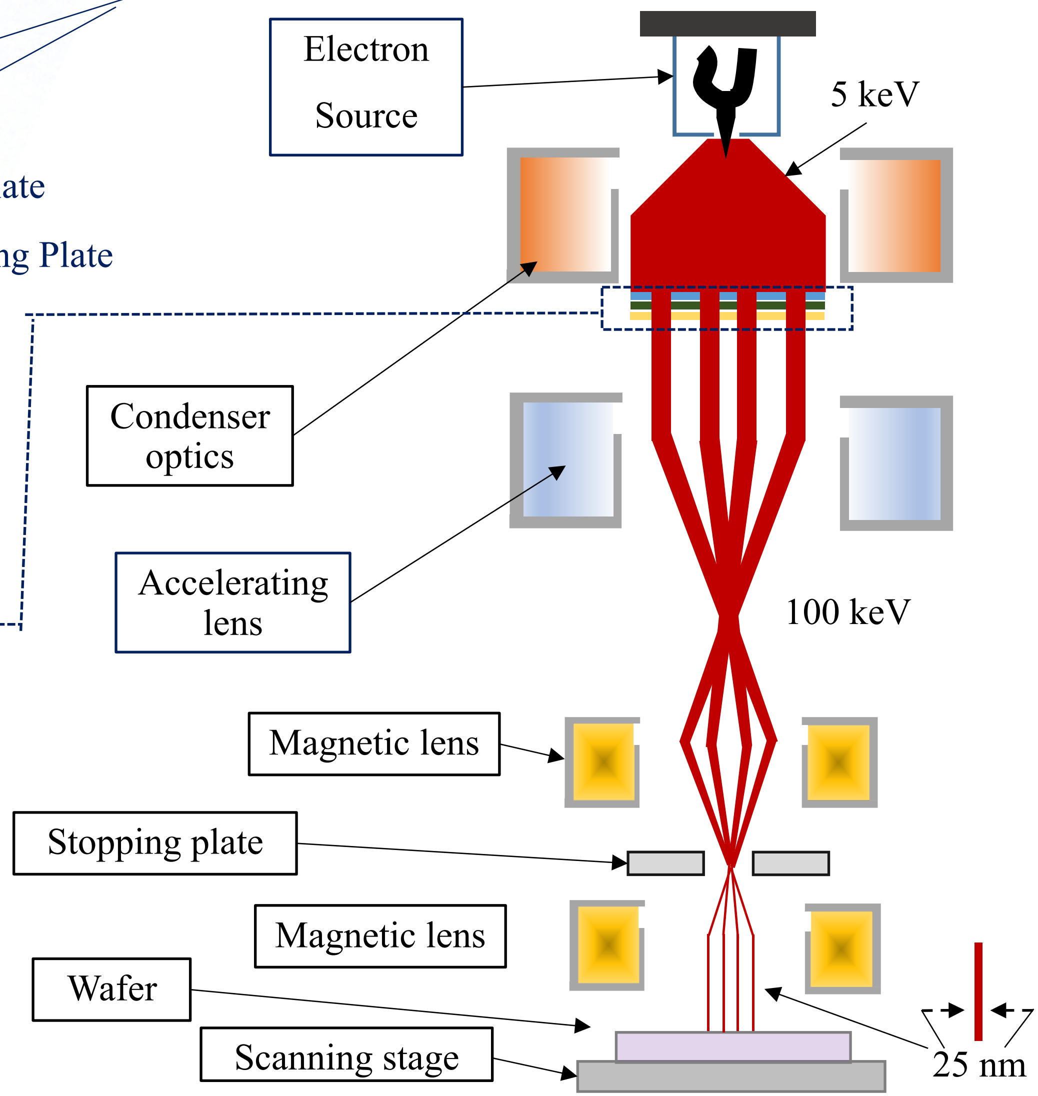
# Ending a SEM imaging

- Turn off the EHT
- Move the stage down
- Venting
- Remove the sample holder
- Pump again the system

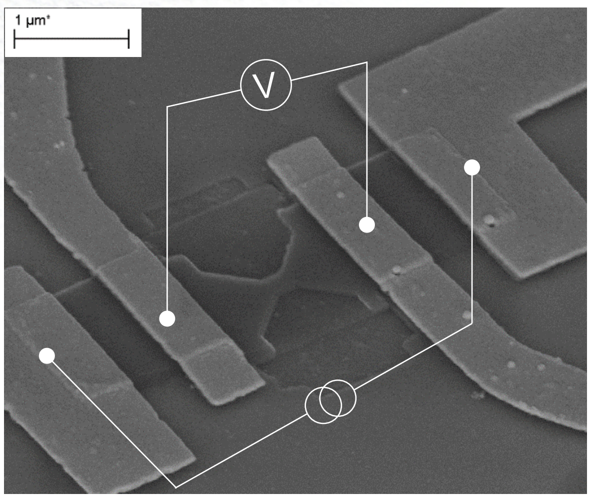
Same procedure when you finish an EBL process

# Fixed-beam-moving-stage (FBMS) strategy

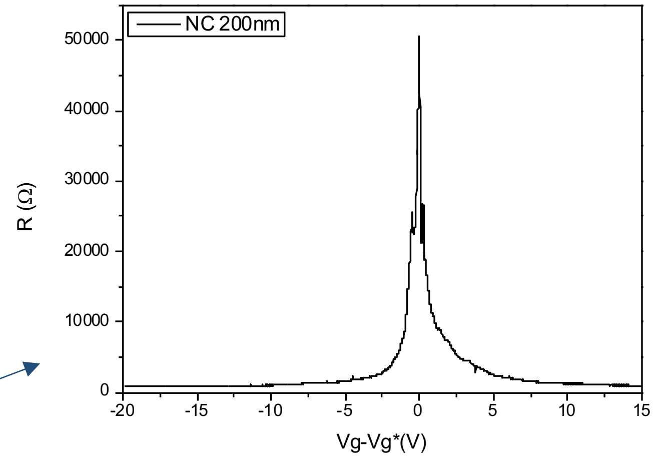


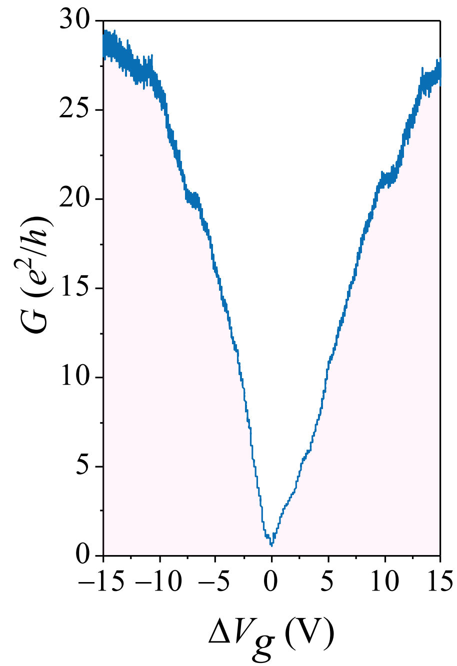

Khodadad I 2017 Electron beam lithography using fixed beam moving stage J. Vac. Sci.Technol. B 35 05160



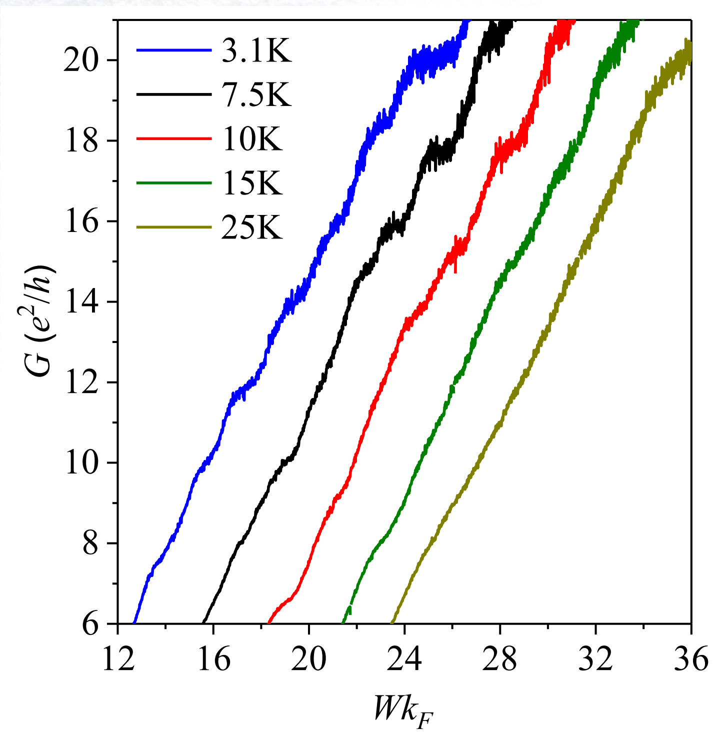

4 Probes measurement (**R**)

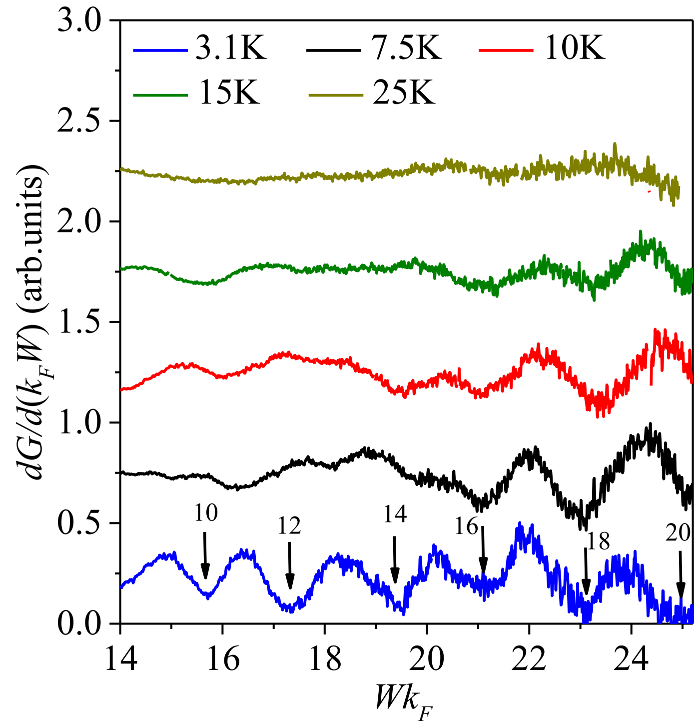



**G=1/R**



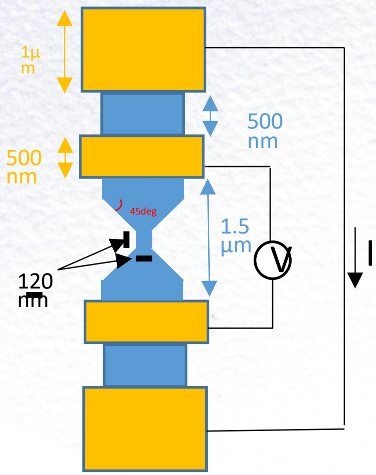
# T dependence

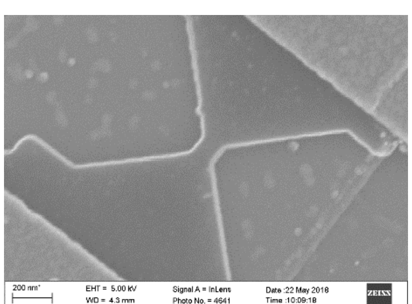


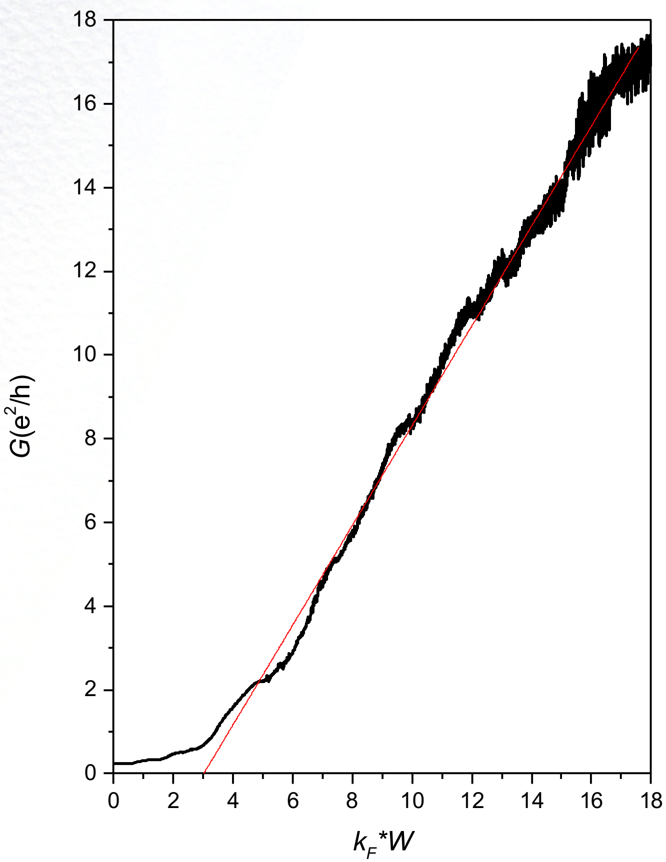



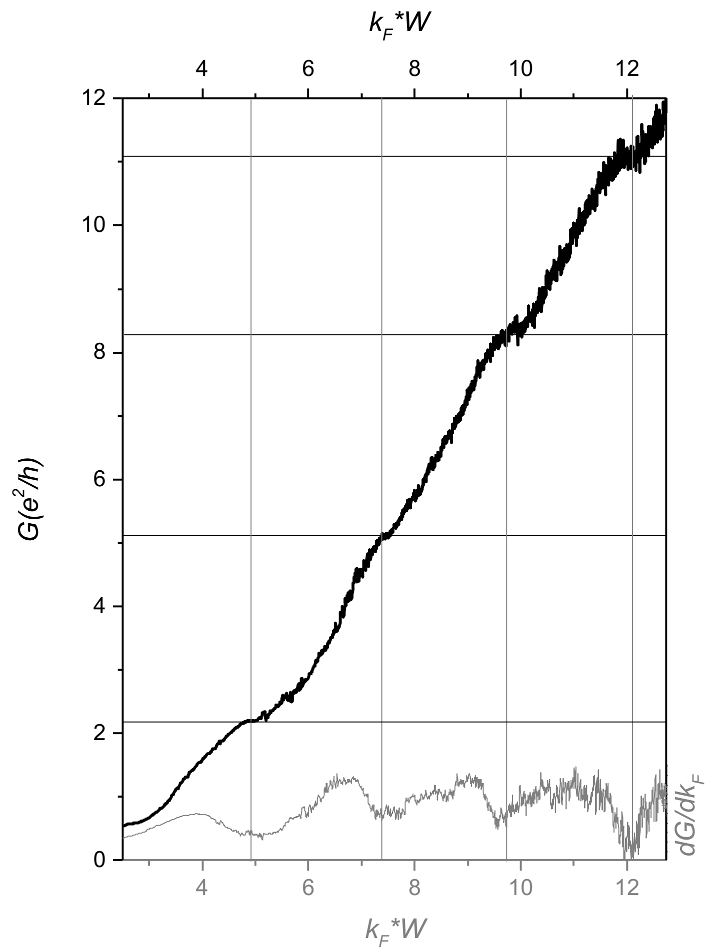
# NC 120nm (G vs kF*W)






GNC_hBN