CIC nanoGUNE
nanoscience cooperative research center

INTERNATIONAL SUMMER SCHOOL ON NANOFABRICATION AND TRANSMISSION ELECTRON MICROSCOPY

Universidad Zaragoza

JACA 18th-22nd July 2016

Electron-beam lithography and its applications to spintronics

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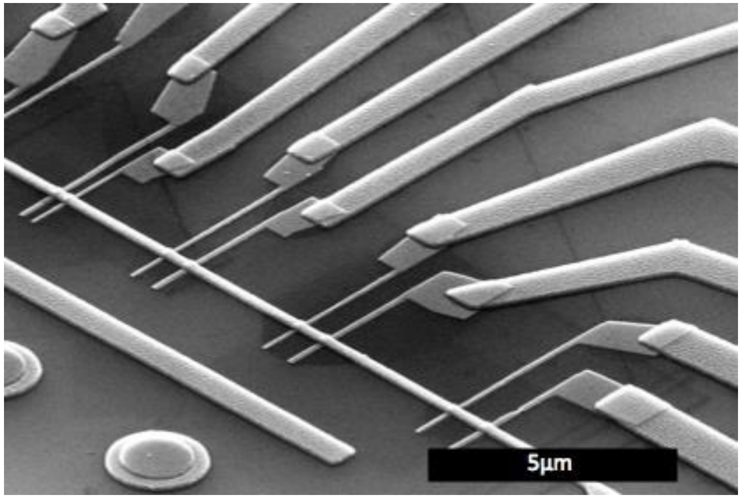

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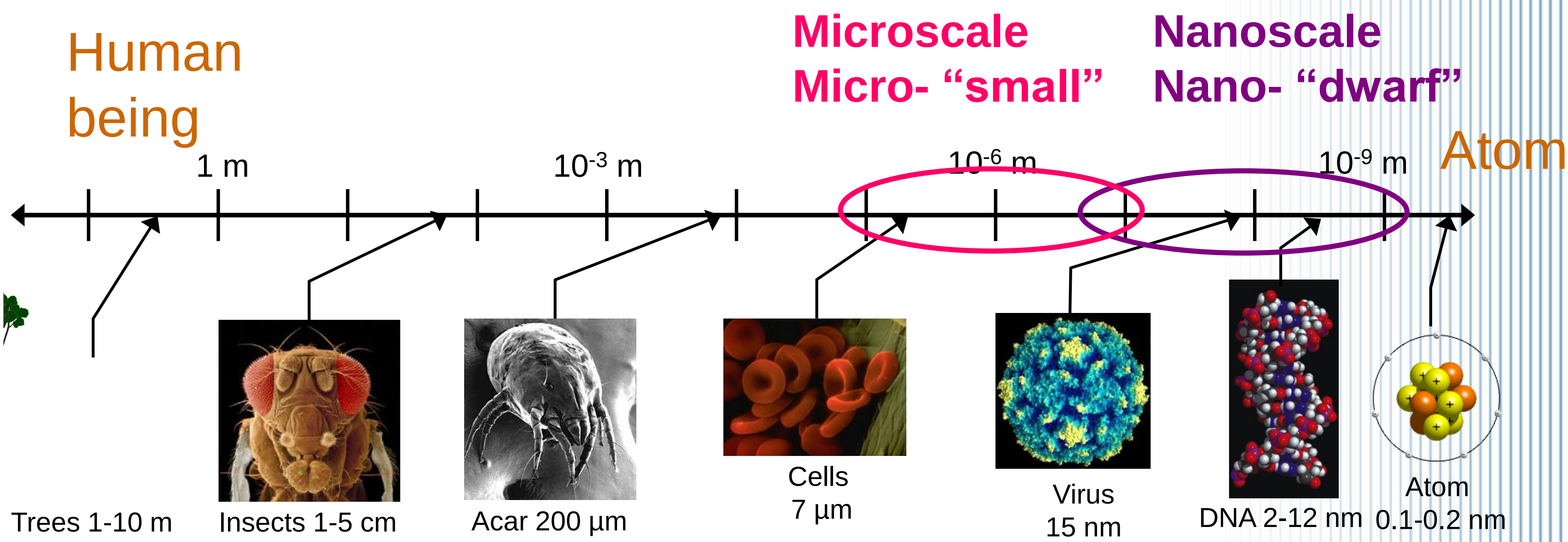
THE NANOSCALE



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THE NANOSCALE

FIELD	PROPERTY	SCALE LENGTH
Electronics	Electronic Wavelength Inelastic mean free path Tunneling	10 - 100 nm 1 - 100 nm 1 - 10 nm
Magnetism	Domain wall Spin-flip scattering	10 - 100 nm 1 - 100 nm
Optics	Quantum well Evanescent wave decay length Metallic skin depth	1 - 100 nm 10 - 100 nm 10 - 100 nm
Superconductivity	Cooper pair coherence length Meissener penetration depth	0.1 - 100 nm 1 - 100 nm
Mechanics	Dislocation interactions Grain Boundaries Crack tip radi Nucleation/growth defect Surface corrugation	1 - 1000 nm 1 - 10 nm 1 - 100 nm 0.1 - 10 nm 1 - 10 nm
Catalysis	Surface topology	1 - 10 nm
Supramolecules	Kuhn length Secondary structure Tertiary structure	1 - 100 nm 1 - 10 nm 10 - 1000 nm
Inmunology	Molecular recognition	1 - 10 nm

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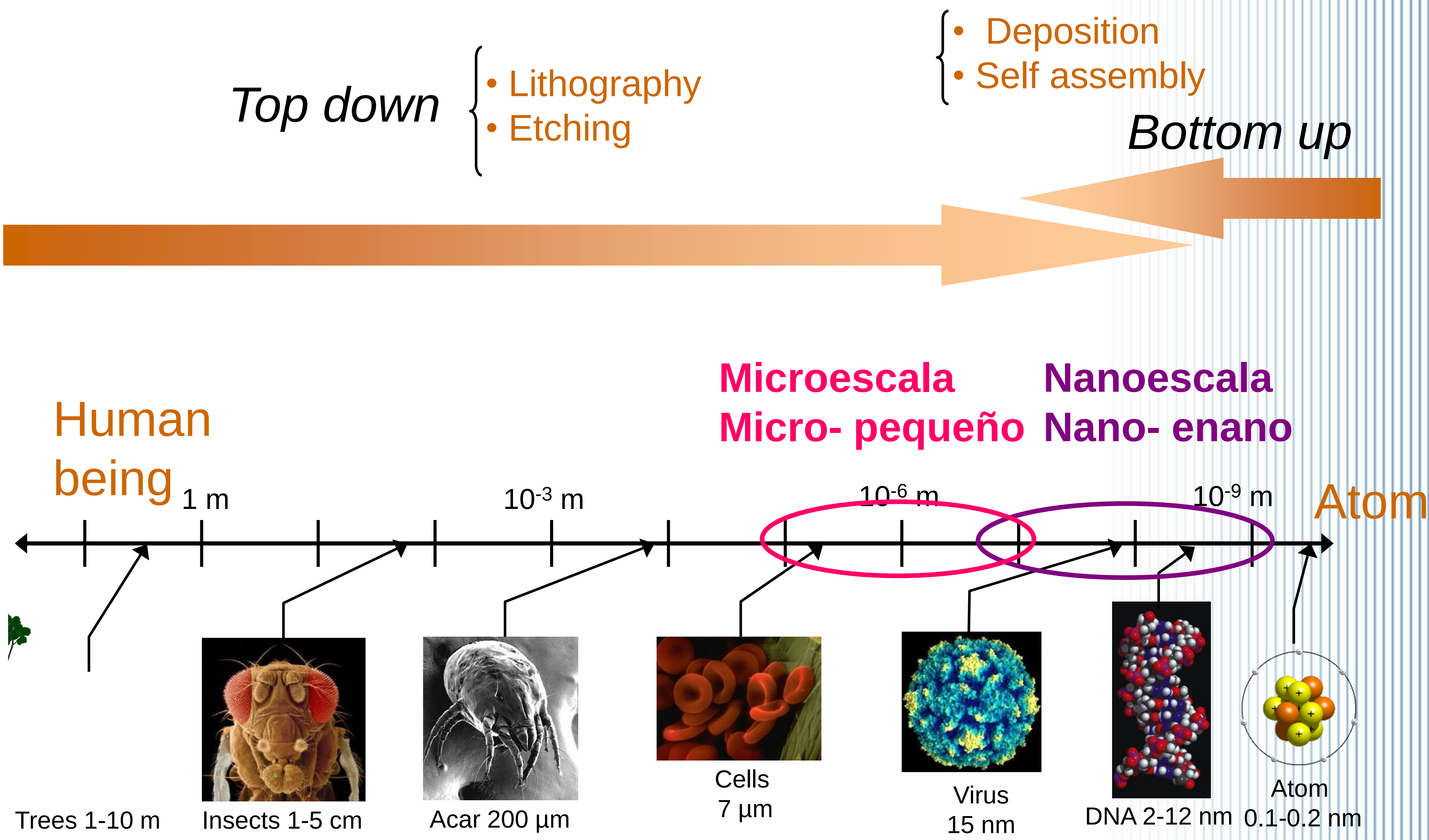
NANOFABRICATION TECHNIQUES



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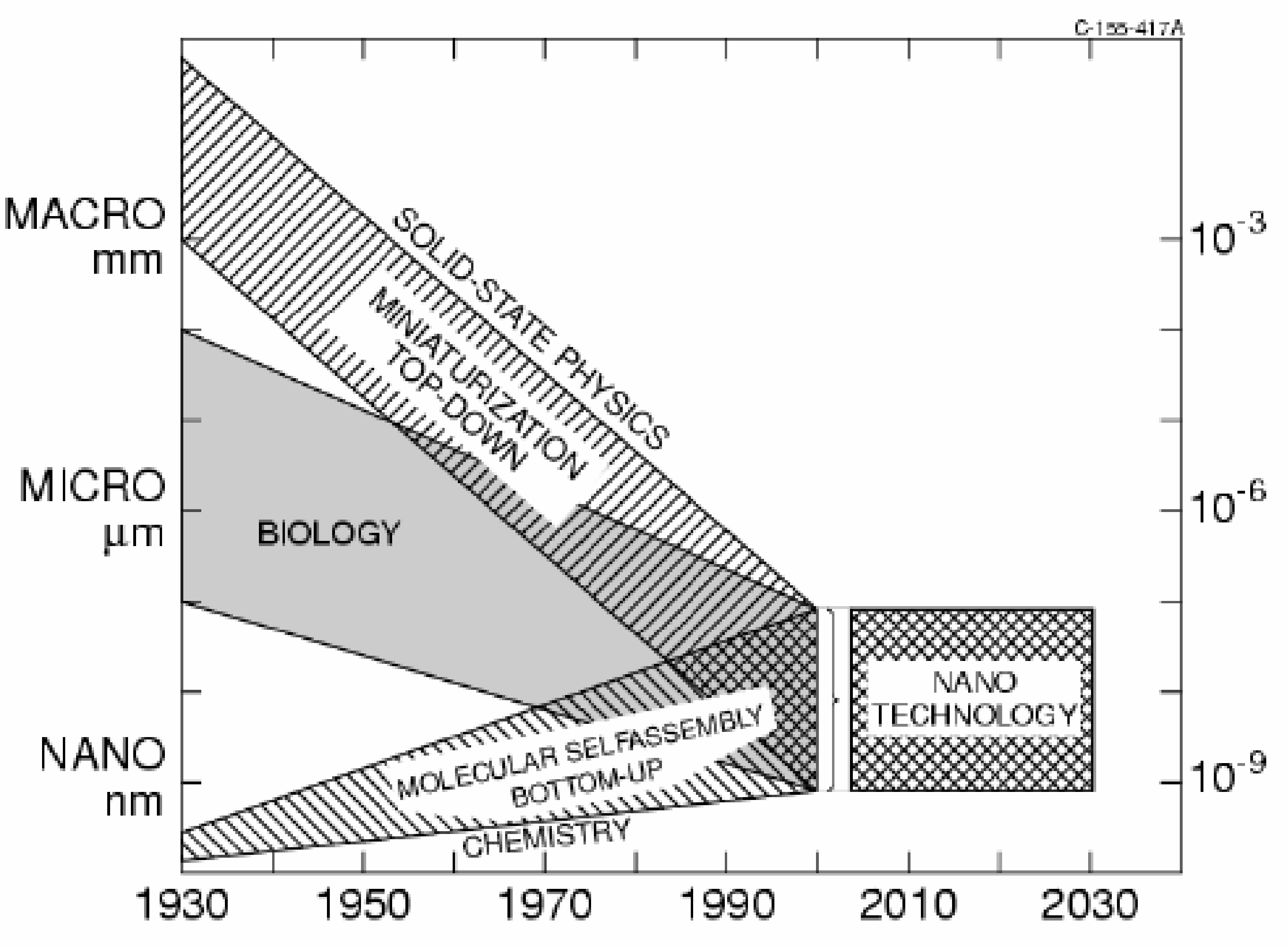
NANOFABRICATION TECHNIQUES



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OUTLINE

- Introduction to electron-beam lithography
  - Working principle
  - Complete process
  - Key parameters
  - Proximity effect

- Common problems
  - Sharp edges
  - Redeposition
  - Insulating substrates

- Spintronics applications
  - Lateral spin valve devices (for spin transport)
  - Spin absorption devices (for spin Hall effect)

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LITHOGRAPHY

Lithography (from Ancient Greek *λίθος (lithos)*, meaning "stone", and *γράφειν* (*graphein)*, meaning "to write") is a method of printing originally based on the immiscibility of oil and water. The printing is from a stone (lithographic limestone) or a metal plate with a smooth surface. It was **invented in 1796** by German author and actor **Alois Senefelder** as a cheap method of publishing theatrical works.Lithography can be used to print text or artwork onto paper or other suitable material.

From Wikipedia

Anasazi people, s. X-XI A.C.
Monument Valley (Arizona)

Nabatean people, s. III B.C.-II A.D.
Wadi Rum (Jordan)

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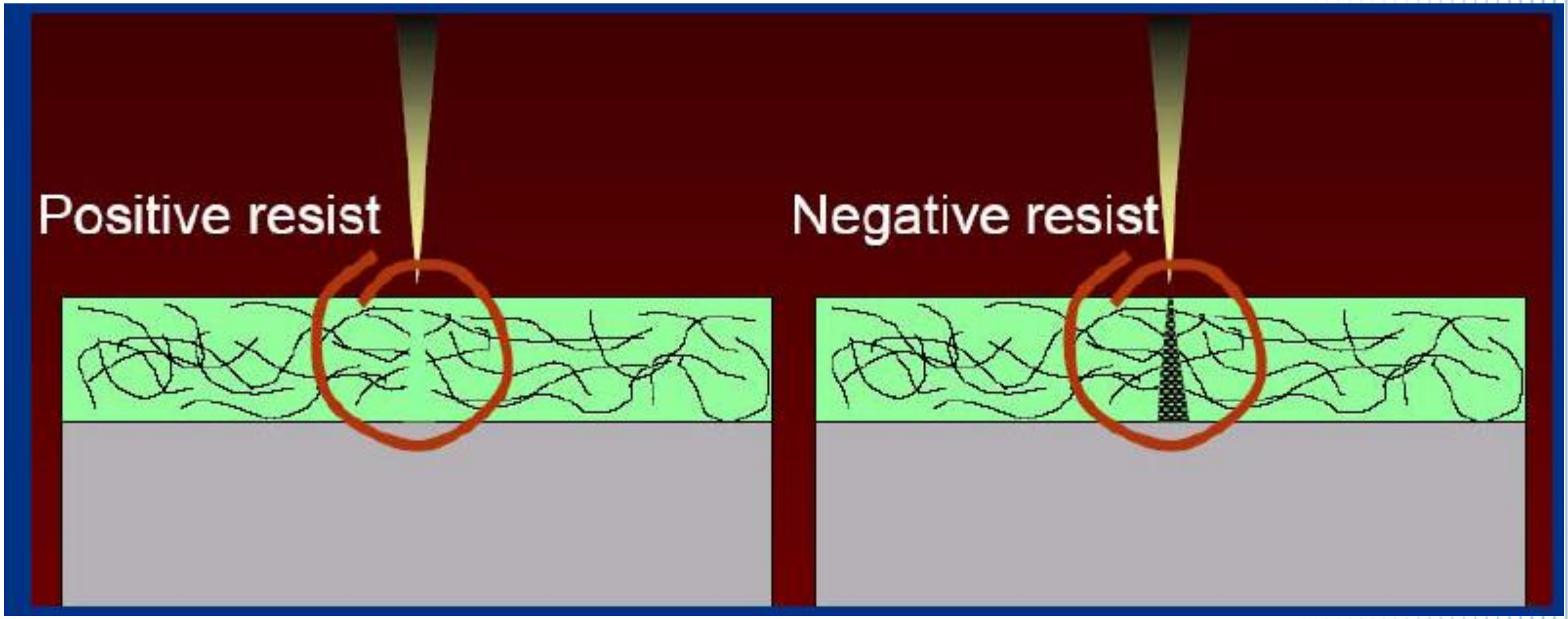
LITHOGRAPHY

•Negative Resist

Cross-linking: adjacent polymer chains cross-link to form complex 3D structures with higher molecular weigth and, thus, lower solubility.

•Positive Resist

Chain-scission: polymer chains break to form chains with lower molecular weight and, thus, higher solubility



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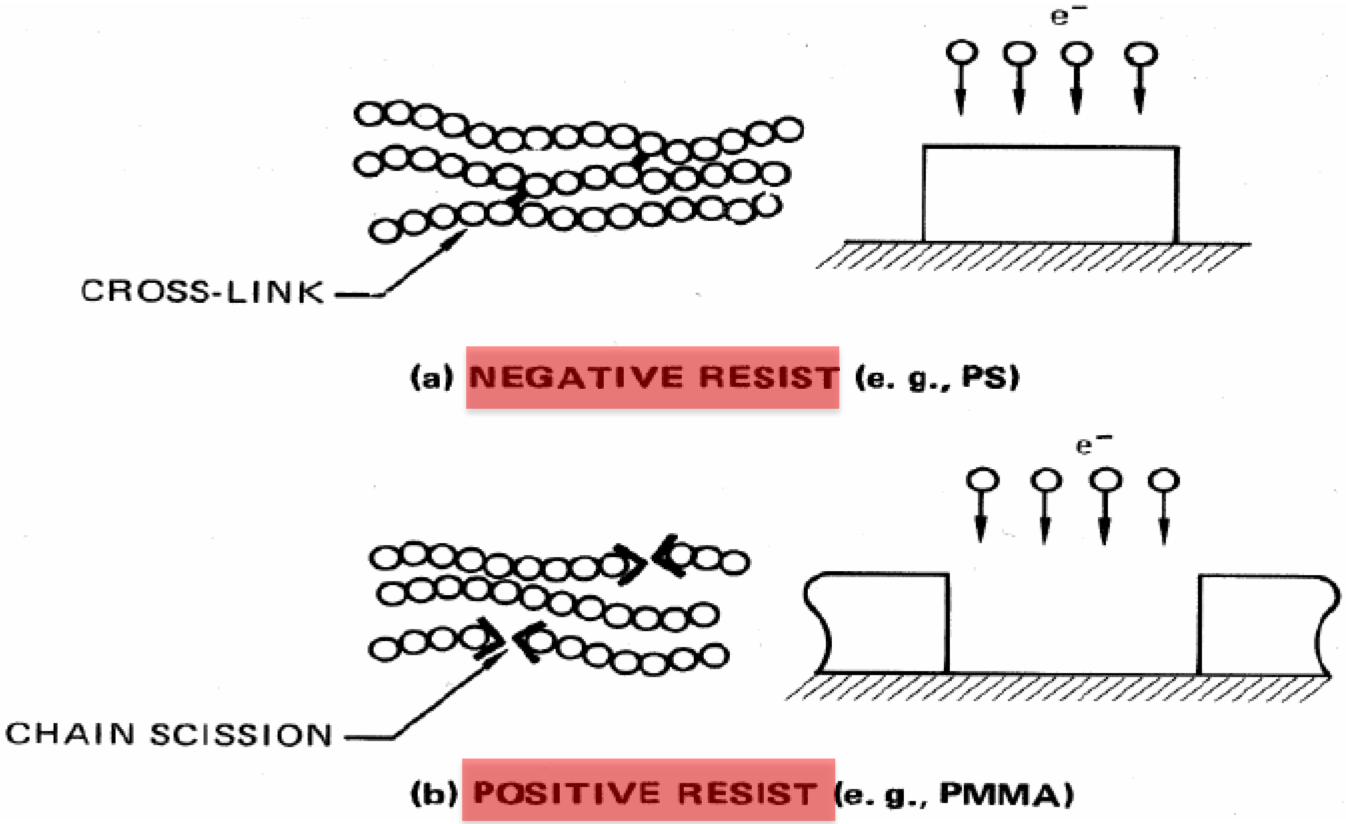
E-BEAM LITHOGRAPHY

Electron-sensitive resists



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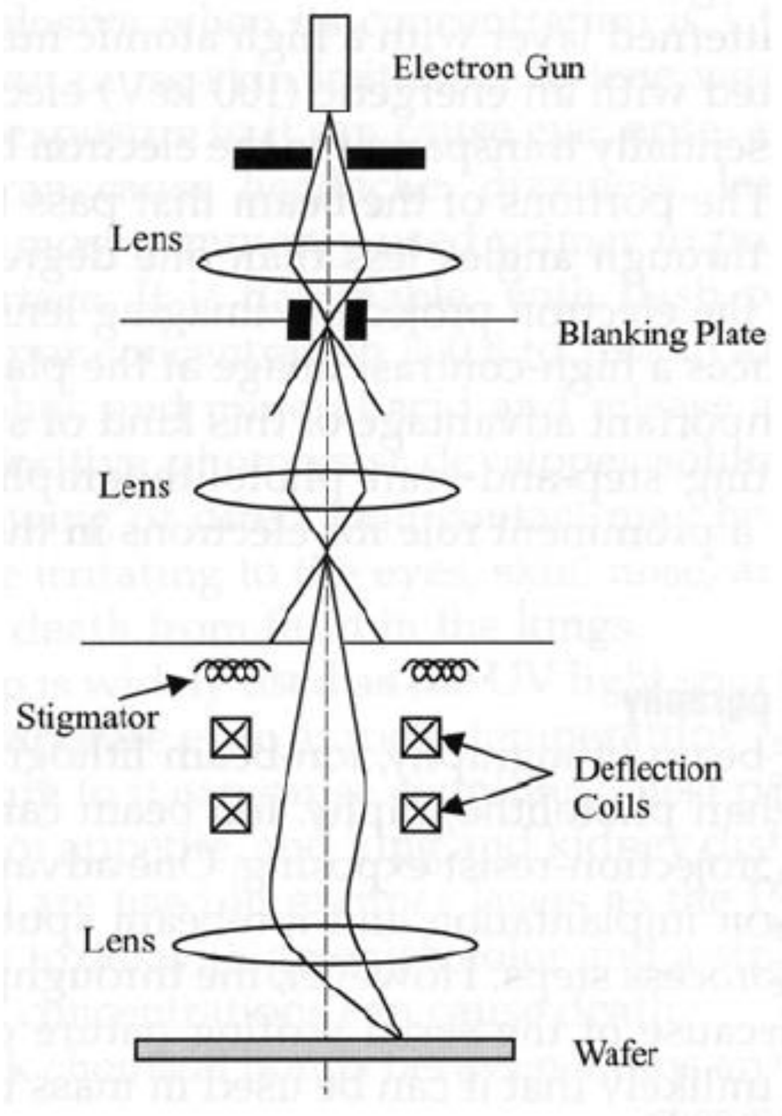
E-BEAM LITHOGRAPHY

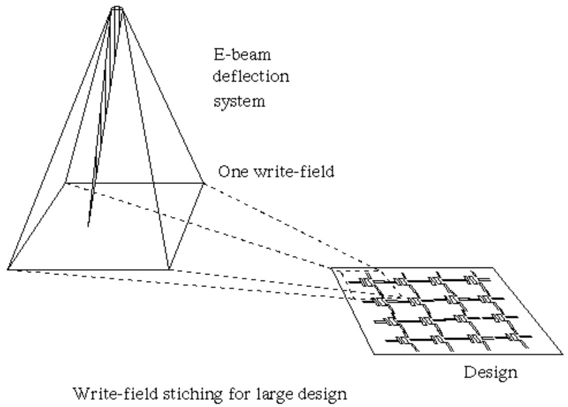


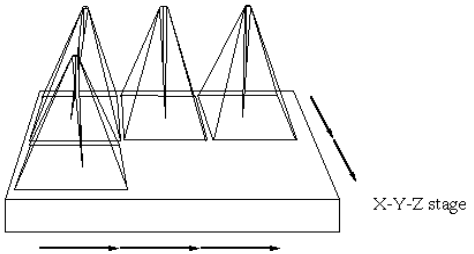
1) Scanning electron microscopy (SEM):

Electron beam column and optics

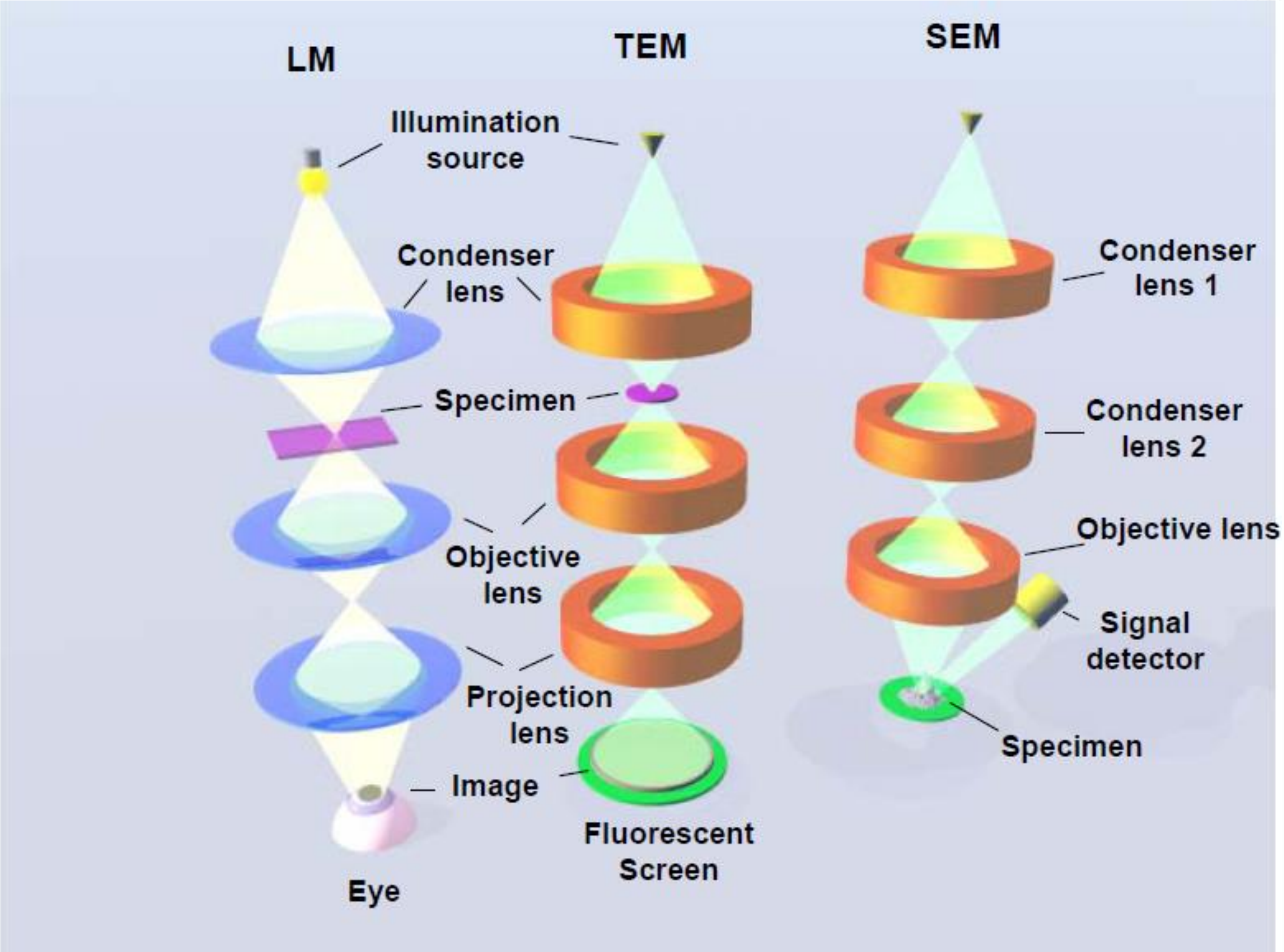
- Electron source Schottky field emitter ZrO/W.
- Beam energy range : 100 V to 30 kV in 10 V steps.
- Beam current range : selectable between approximately 5 pA – 20 nA.
- Beam size : • 2 nm at 20 kV at 3 mm working distance.
  • 4 nm at 1 kV at 3 mm working distance.
- Gaussian beam.

2) Laser interferometric stage





ELECTRON MICROSCOPY



Transmission electron microscopy (TEM) studies the **inner structure** of objects
Scanning electron microscopy (SEM) visualizes the **surface** of objects

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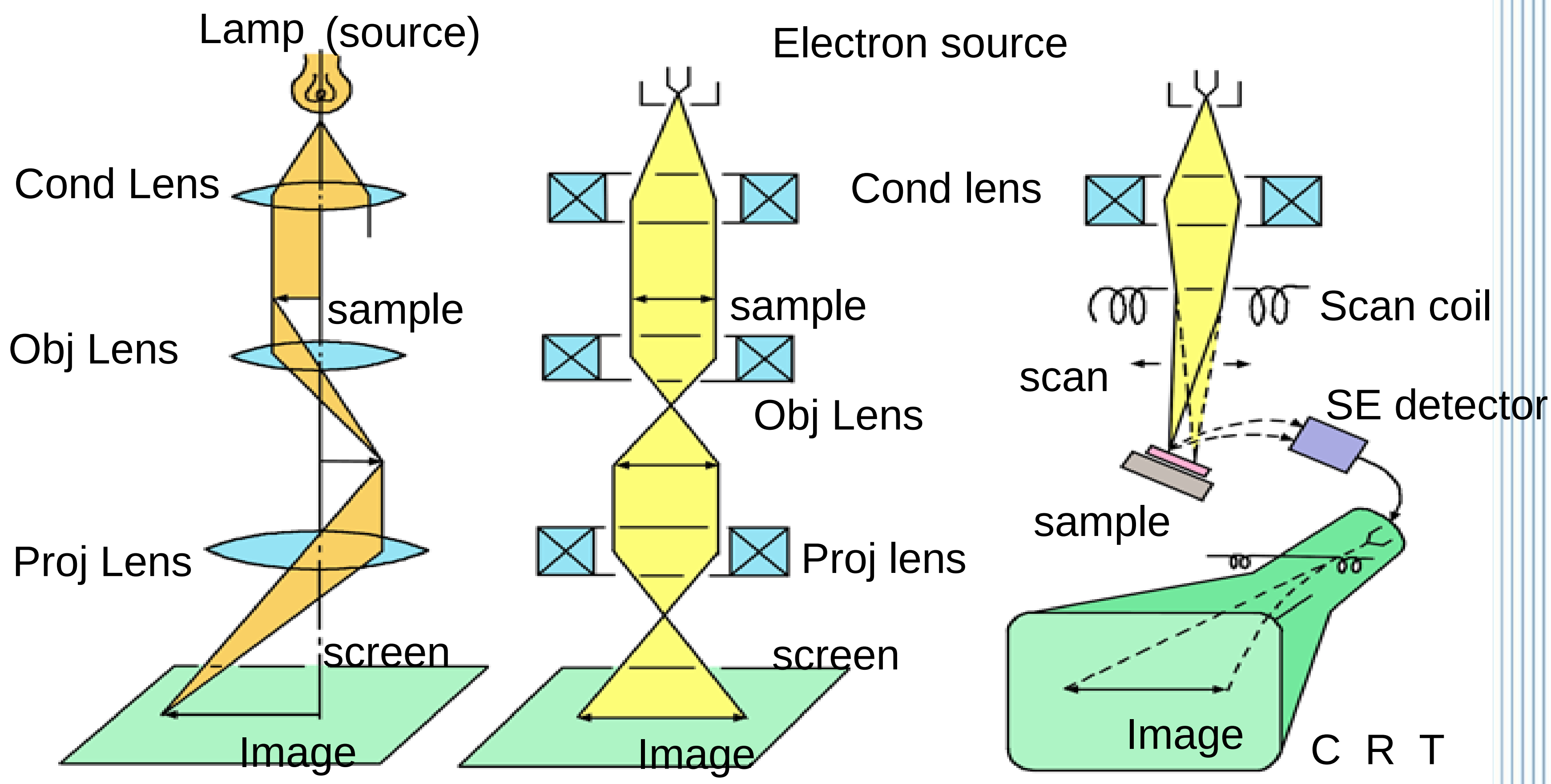
ELECTRON MICROSCOPY

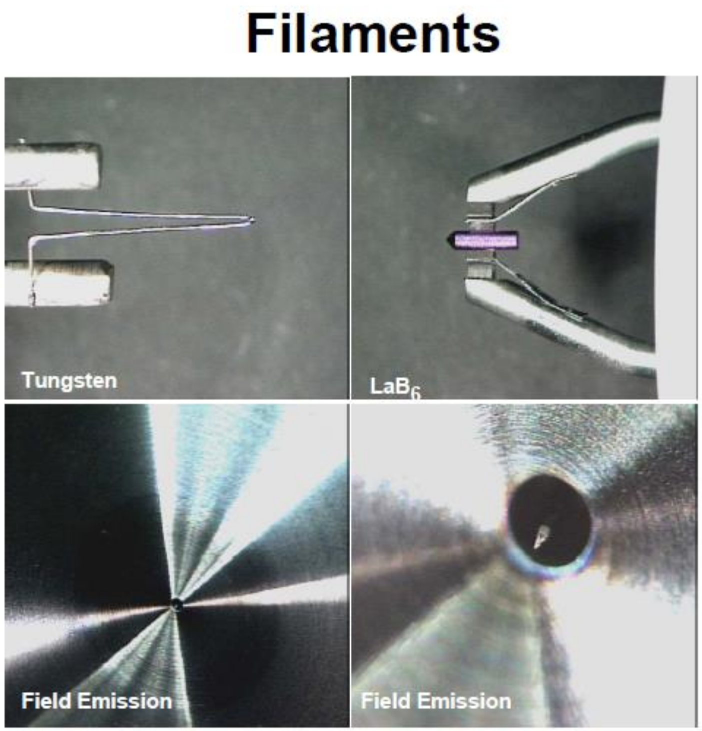


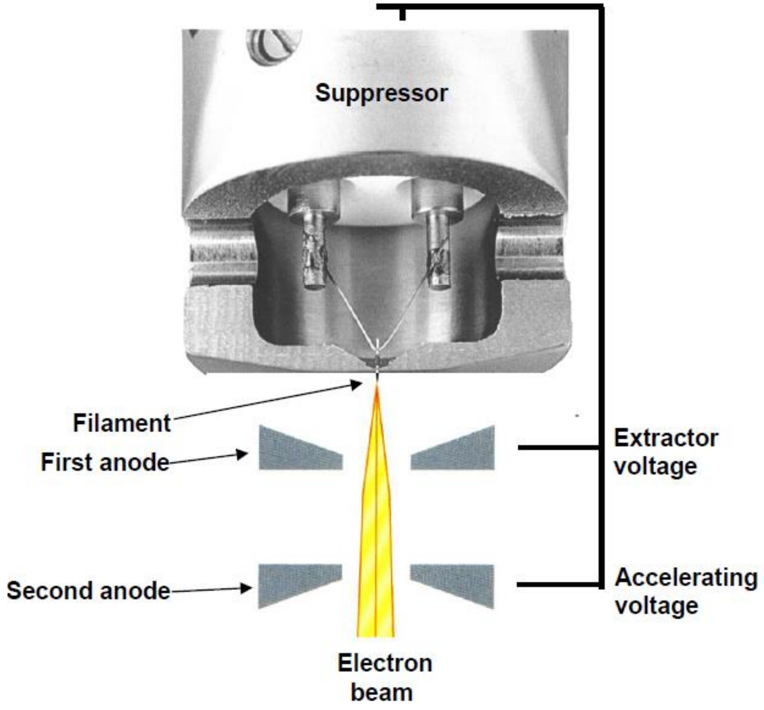
Light microscopy (LM) **TEM** **SEM**

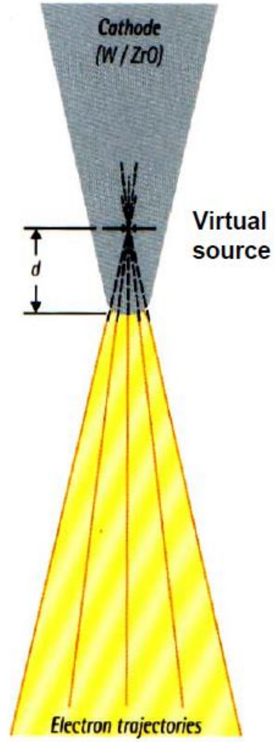
Difference between LM, TEM and SEM

ELECTRON MICROSCOPY

Electron sources







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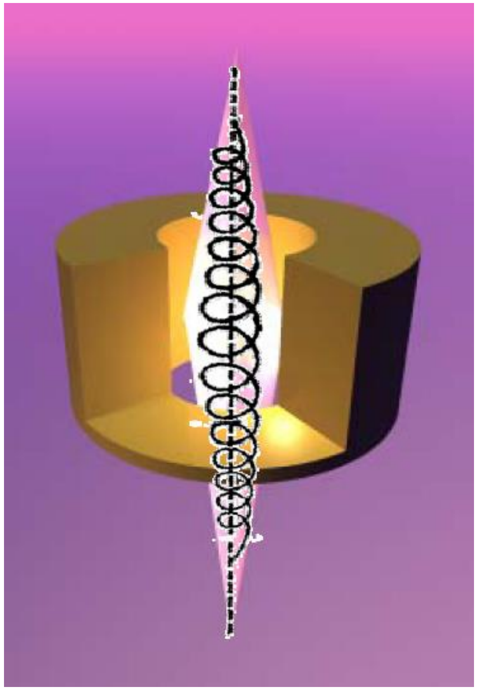
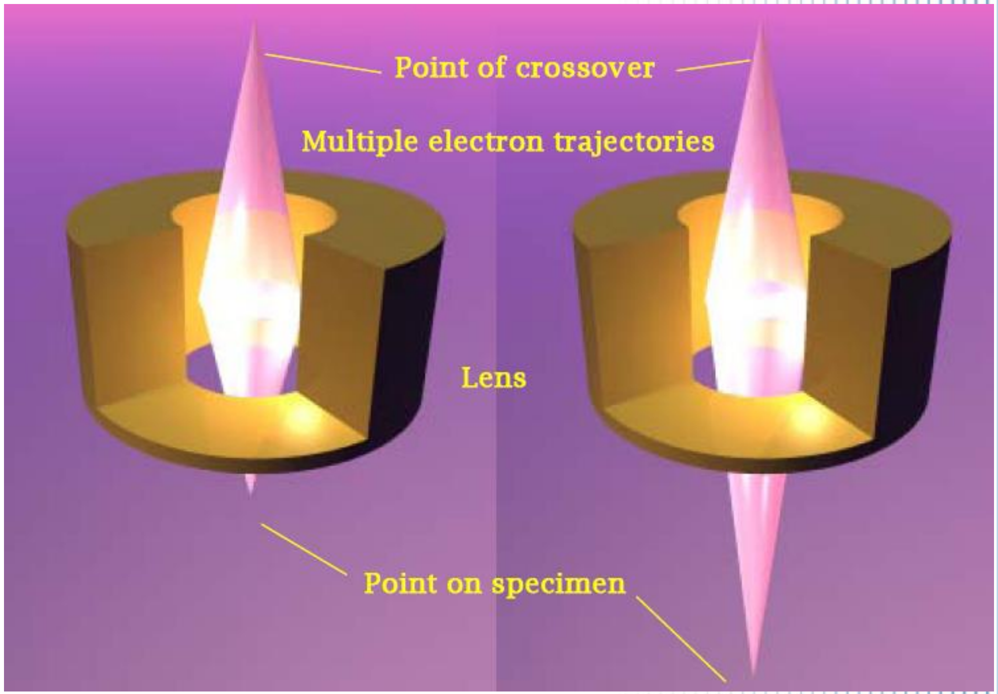
ELECTRON MICROSCOPY

Electromagnetic lenses





F = -e (B x v)

Electrons move through the lens in a helicoidal trajectory, not in a straight line

The focal length of the lens can be tuned with the DC current applied to the coils.

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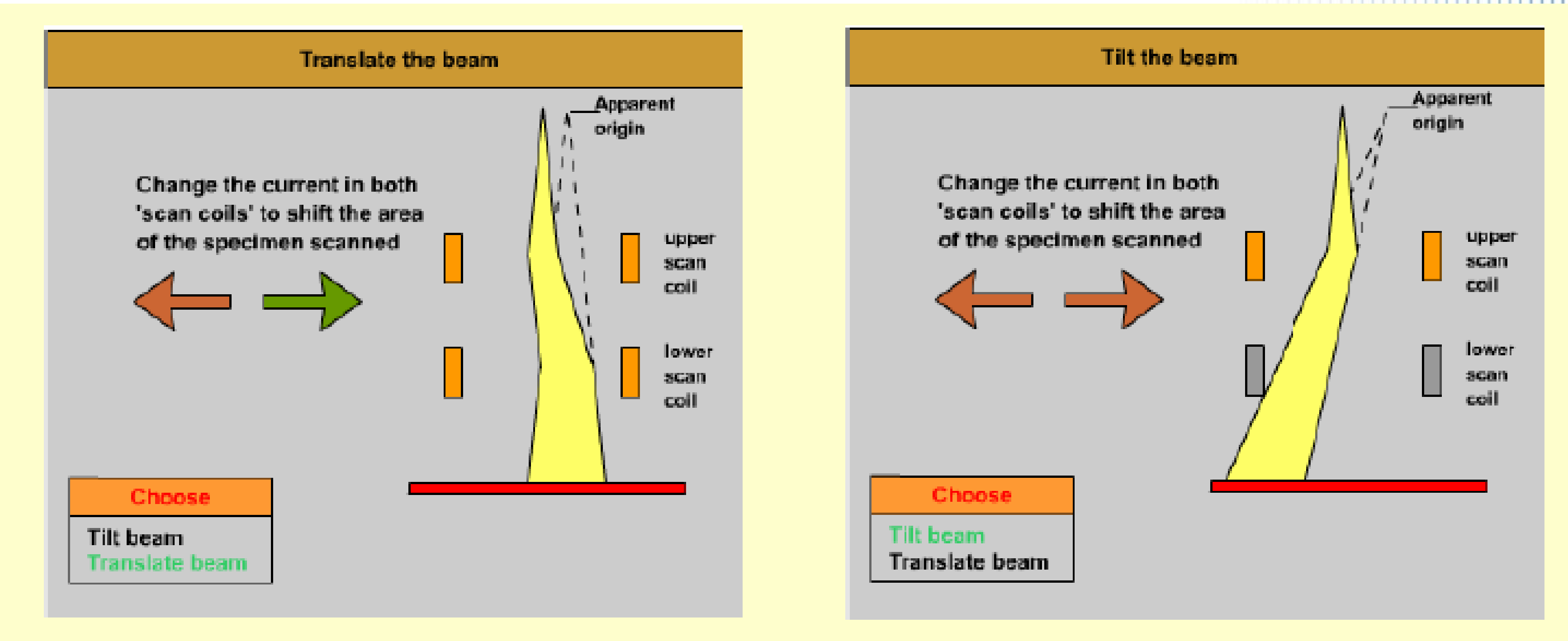
SCANNING ELECTRON MICROSCOPY (SEM)

Scanning



Double scanning coil set-up allows to:

1) Translate the beam without changing its angle
2) Tilt the beam without changing its position on the object

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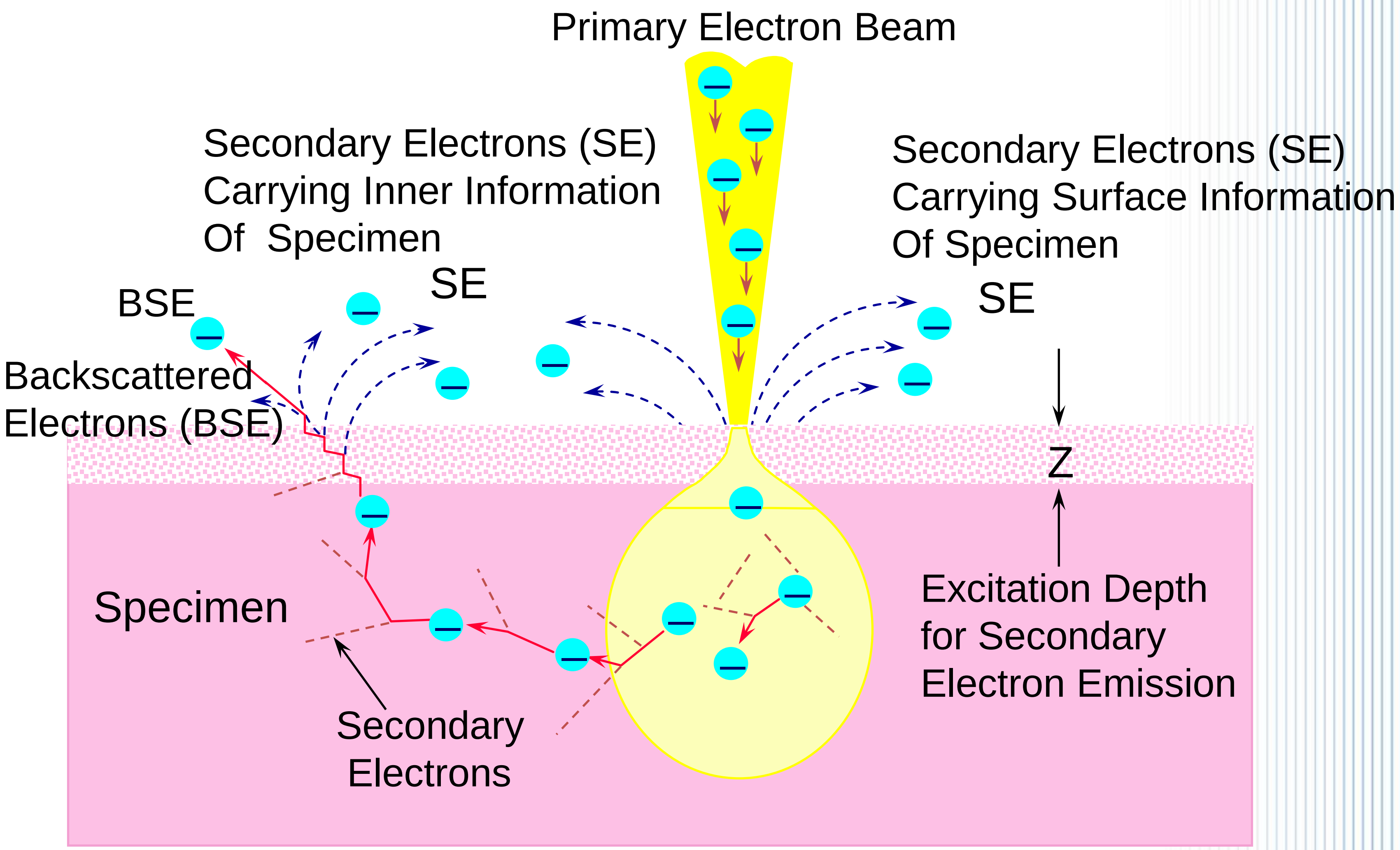
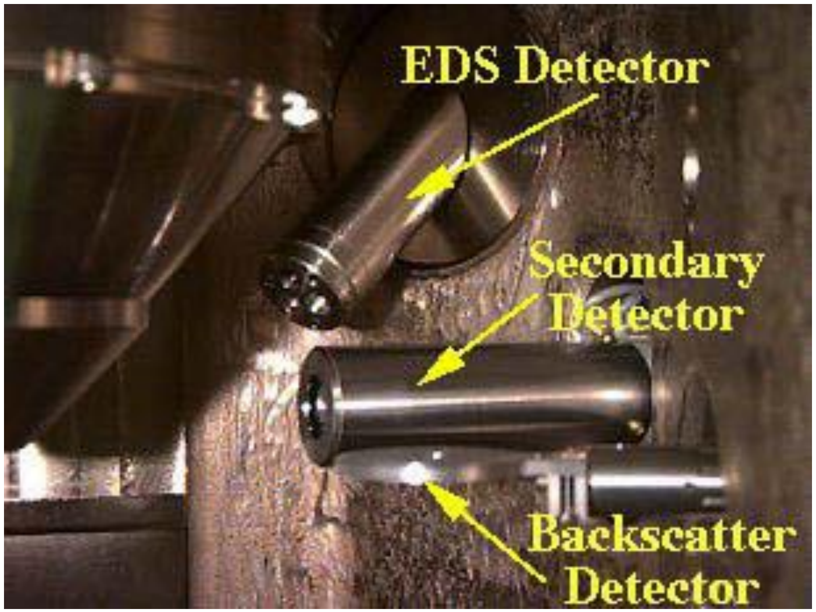
SCANNING ELECTRON MICROSCOPY (SEM)

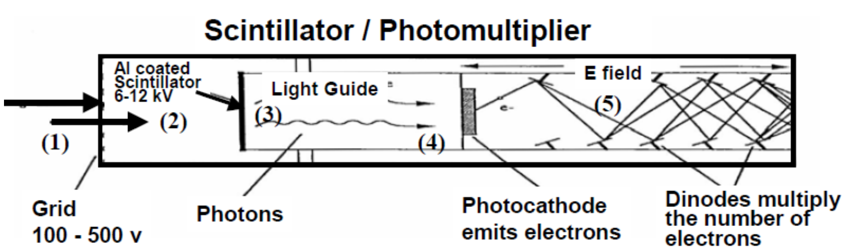


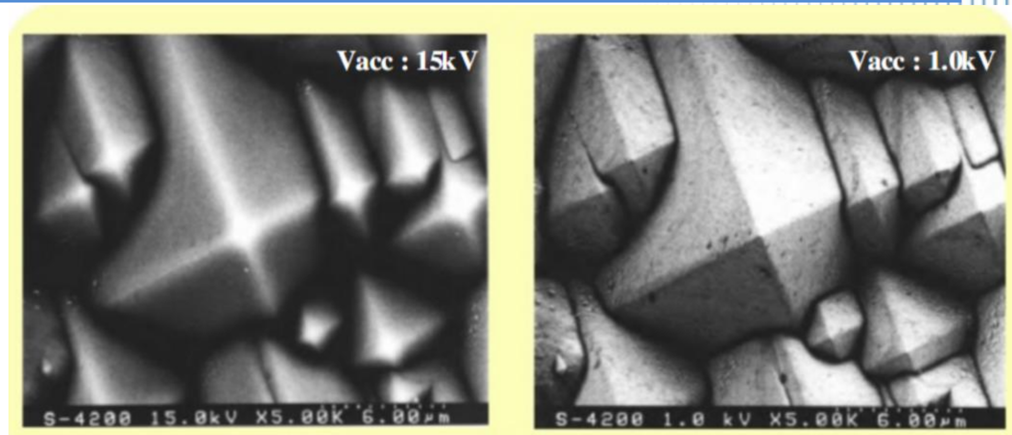
SE and BSE emitted from solid sample

SCANNING ELECTRON MICROSCOPY (SEM)

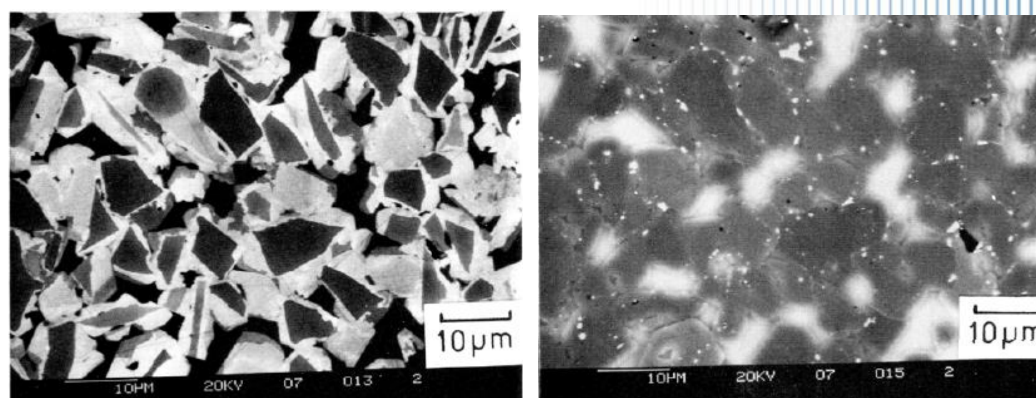
Electron detectors







Secondary electrons: low energy, topografic information, + depth at + Vacc



Backscattered electrons: high energy, topografic information and atomic number

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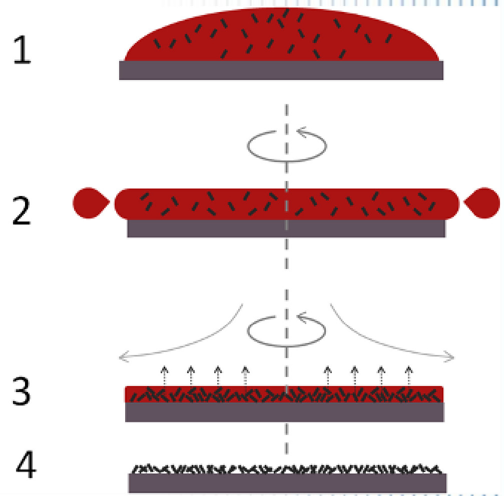
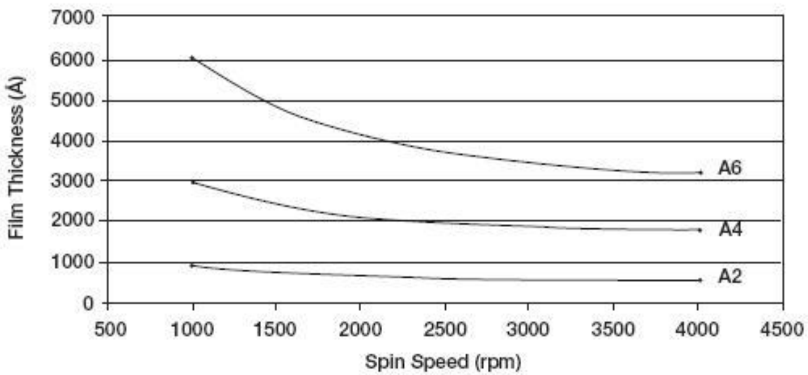
COMPLETE PROCESS:

- Spinning resist
- Exposing resist
- Develop resist
- Etch / Deposit
- Remove resist

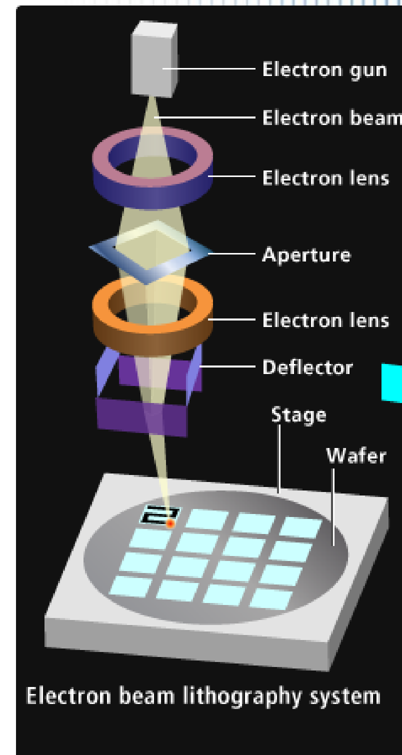


495PMMA A Resists
Solids: 2% - 6% in Anisole

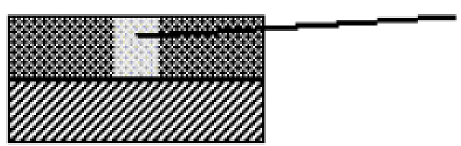


E-BEAM LITHOGRAPHY

COMPLETE PROCESS:



- Spinning resist
- Exposing resist
- Develop resist
- Etch / Deposit
- Remove resist

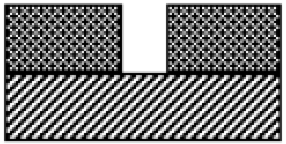


Photosensitive materials properties change only where exposed to radiation

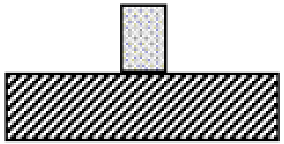
E-BEAM LITHOGRAPHY

COMPLETE PROCESS:

- Spinning resist
- Exposing resist
- Develop resist
- Etch / Deposit
- Remove resist



a) Positive resist, developer solution removes exposed material



b) Negative resist, developer solution removes unexposed material

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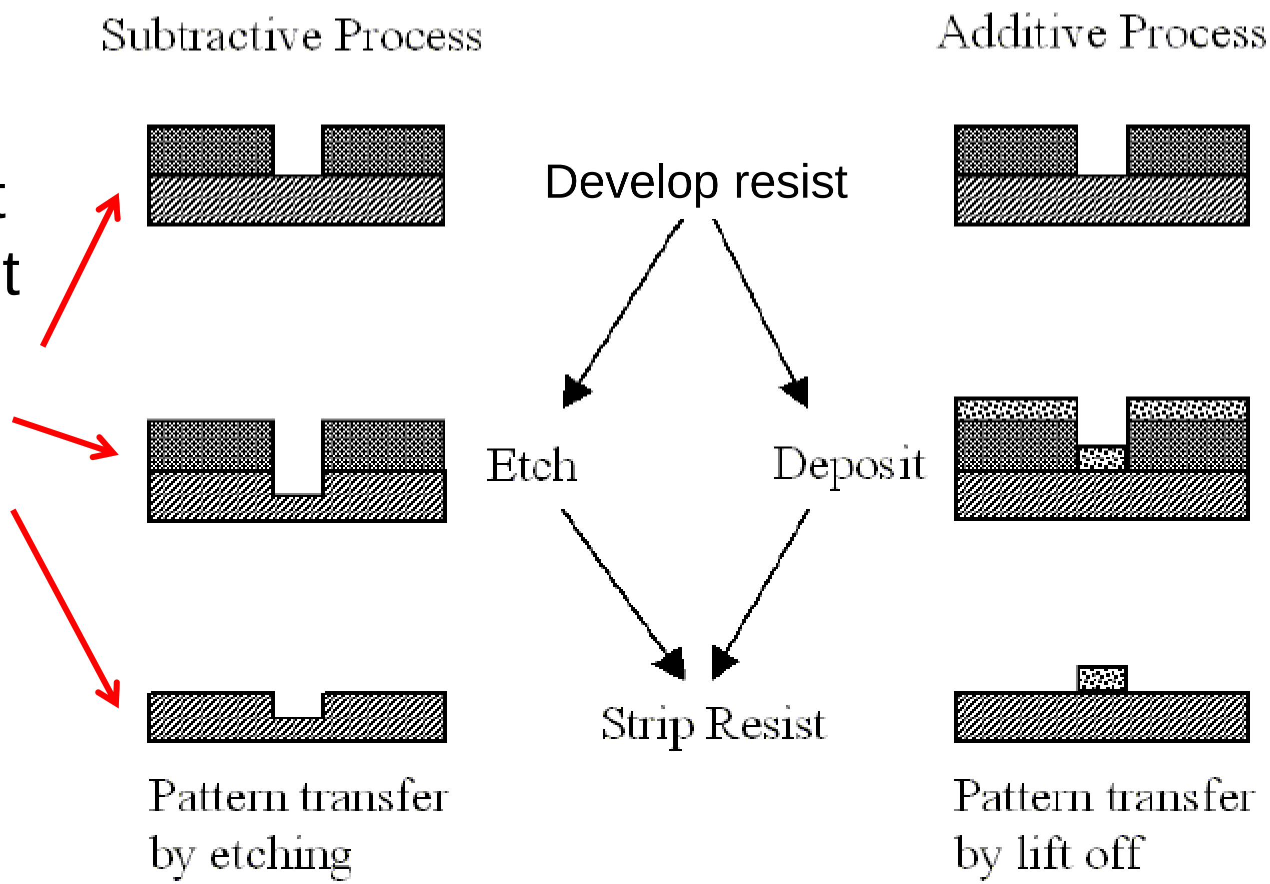
E-BEAM LITHOGRAPHY

COMPLETE PROCESS:

Example with positive resist

- Spinning resist
- Exposing resist
- Develop resist
- Etch / Deposit
- Remove resist



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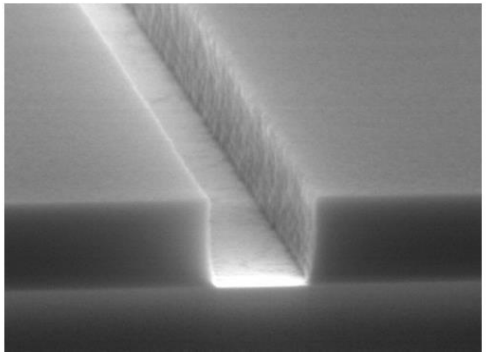
KEY PARAMETERS:

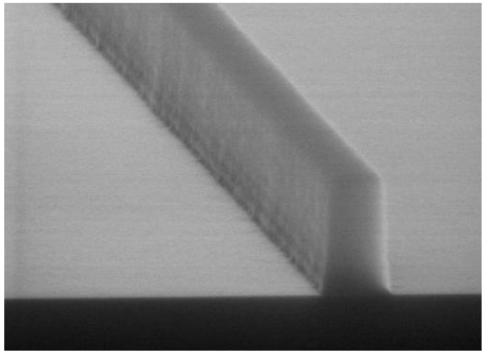
- Resist tone
- Resist performance
- Resist thickness
- Acceleration voltage

Positive or negative: depends on which will give a minimum area to be exposed

Positive resist (trench)



Negative resist (line)



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KEY PARAMETERS:

Resist performance with respect to:
resolution, sensitivity, etching stability

- Resist tone
- Resist performance
- Resist thickness
- Acceleration voltage

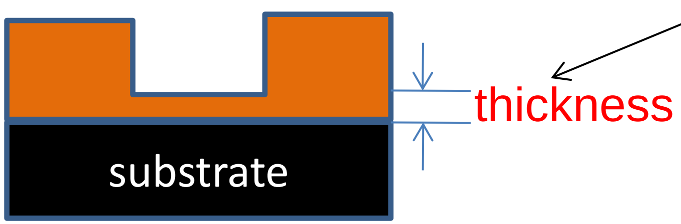
Table-3.8. Conventional e-beam resists and properties

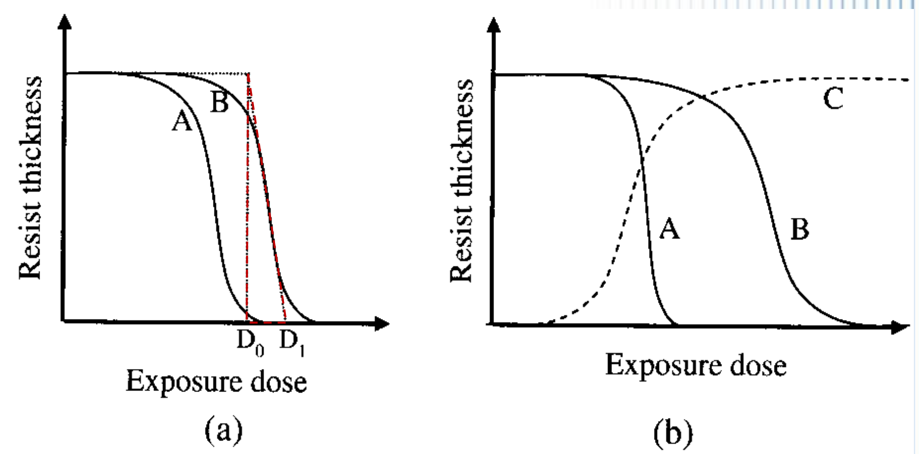
More popular ones: PMMA, ZEP-520, ma-N 2400

	resist tone	resolution / nm	sensitivity*	developer
PMMA	+	10	100	MIBK:IPA
ZEP-520	+	10	30	xylene : p-dioxane
ma-N 2400	–	80	60	MIF726
EBR-9	+	200	10	MIBK:IPA
PBS	+	250	1	MIAK: 2-pentanone 3:1
COP	–	1,000	0.3	MEK : ethanol 7:3

* sensitivity measured at 20 keV beam energy, unit: $\mu C/cm^2$.

Resist development curves:
(a) Resist A is of higher sensitivity than B.
(b) A is of higher contrast than B; C is negative resist.





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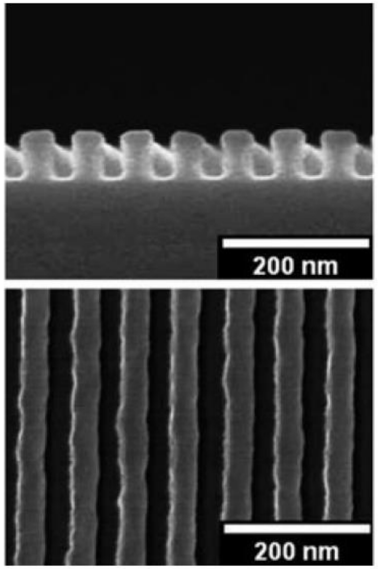
KEY PARAMETERS:

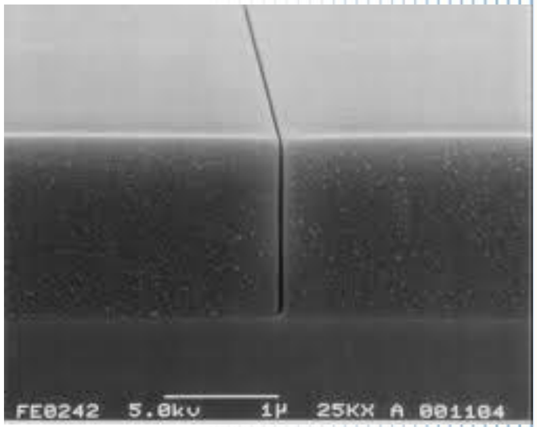
- •Resist tone
- •Resist performance
- •Resist thickness
- •Acceleration voltage

-Thinner resist: will give you higher lateral resolution (polymer size limits aspect ratio)

-Thicker resist: if a thick metal or a long etching is needed



PMMA (aspect ratio 3:1)



ZEP 520 (aspect ratio 30:1)

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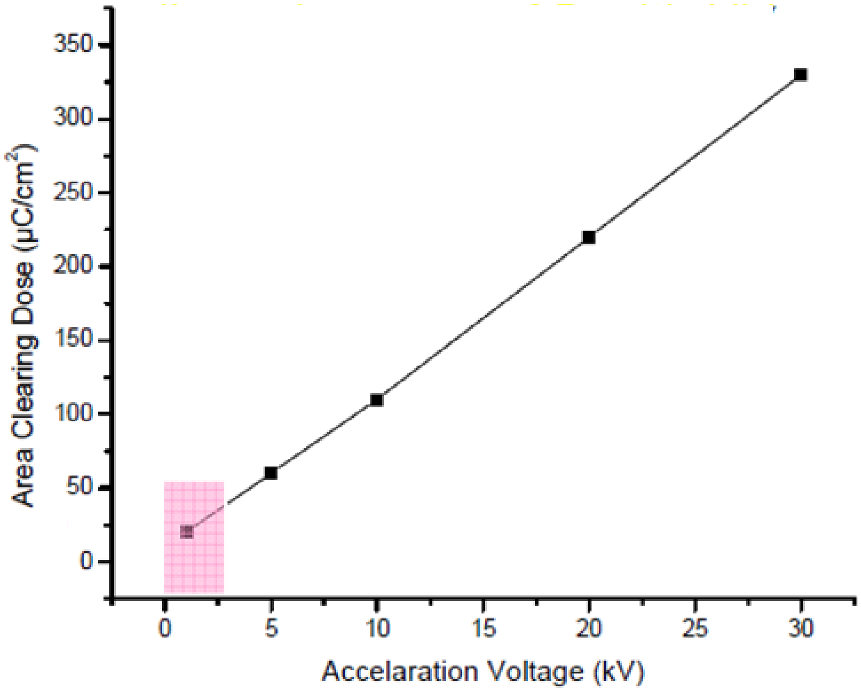
E-BEAM LITHOGRAPHY

KEY PARAMETERS:

Linear increase of exposure dose with the acceleration voltage (for all resists)

- Resist tone
- Resist performance
- Resist thickness
- Acceleration voltage



-Higher voltage gives higher resolution

-Higher voltage requires higher exposure dose (and therefore more time)

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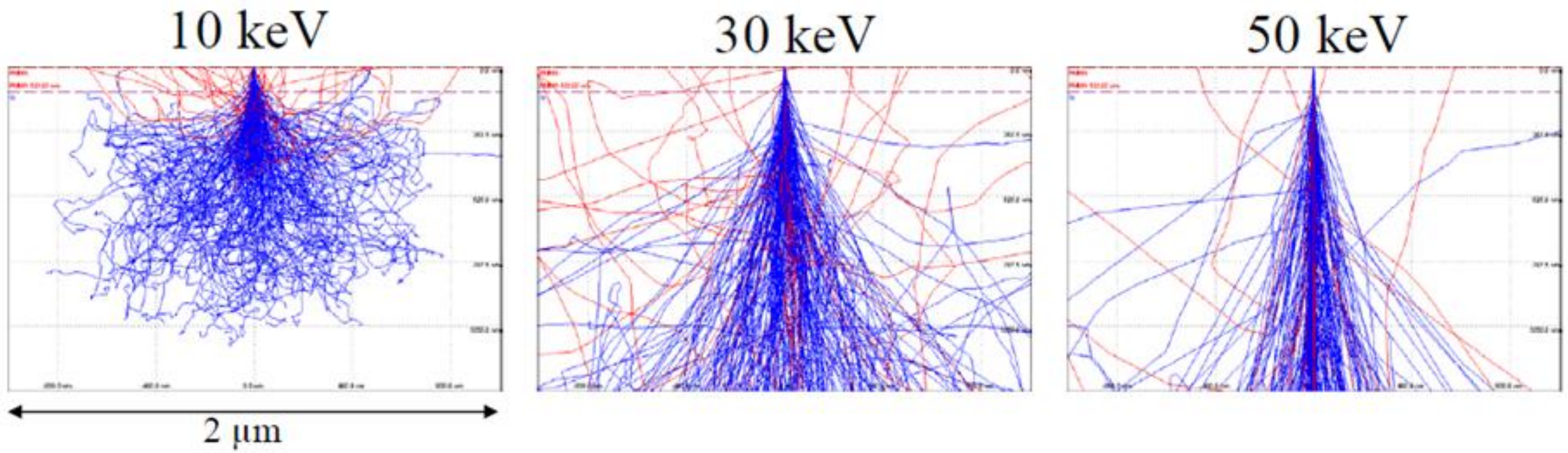
E-BEAM LITHOGRAPHY

KEY PARAMETERS:

- Resist tone
- Resist performance
- Resist thickness
- Acceleration voltage

Linear increase of exposure dose with the acceleration voltage (for all resists)

Secondary electrons (created by forward scattering) are the ones that mainly interact with the resist.



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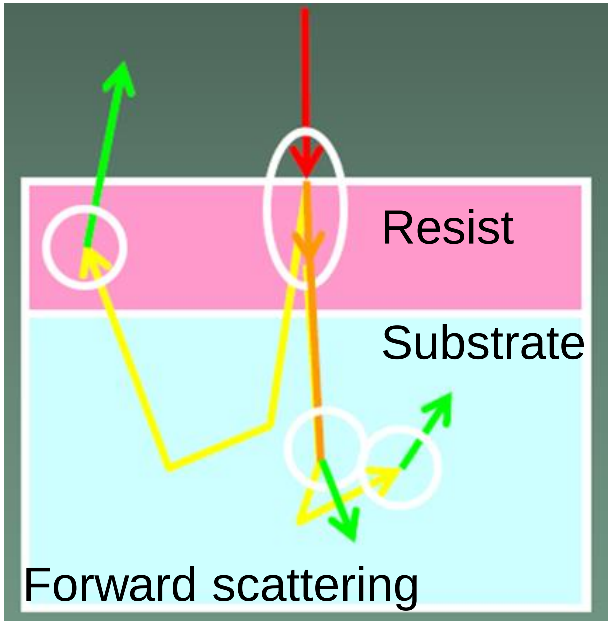
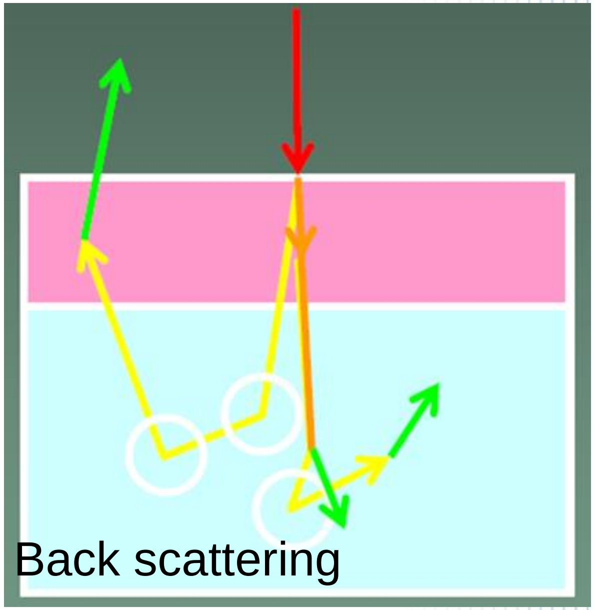
E-BEAM LITHOGRAPHY

Scattering: spreading of the beam, loss of resolution





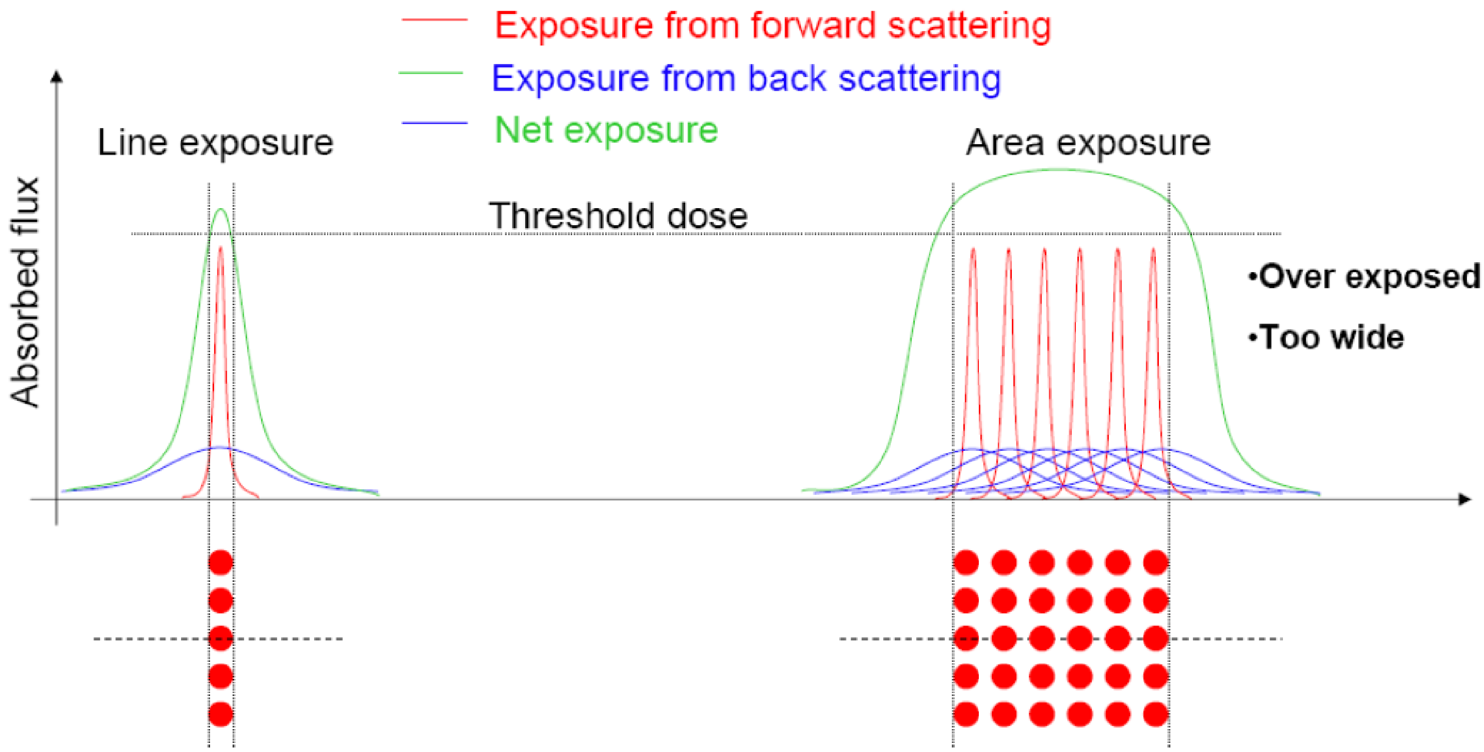
Properties:
Very often
Small angle
Very inelastic (i.e. lose energy)
Generation of SE with low energy.

Properties:
Occasionally (collision with nucleus)
Large angles, thus mainly elastic
High energy, same range as primary electrons.
Large travel length, cause proximity effect.

SE with few eV are responsible for most resist exposure. Such SE diffuses laterally few nm.
Backscattering is responsible for resist exposure far from incidence (proximity effect).

E-BEAM LITHOGRAPHY

PROXIMITY EFFECT



- Proximity effect is negligible for isolated/sparse fine features.
- It is good for *areal* exposure (e.g. pad >>1μm), since pixel can be much larger than beam spot size (right figure). E.g., beam step size (pixel) of 50nm is usually enough even with a beam spot size only 5nm.
- Proximity effect is worst for dense and fine patterns, such as grating with sub-50nm pitch.

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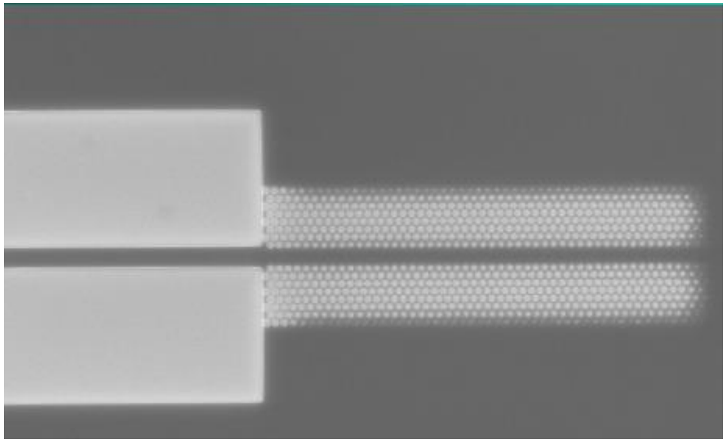
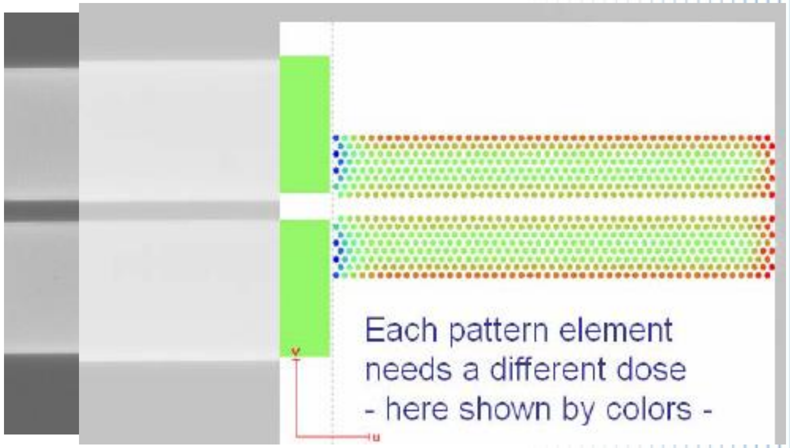
E-BEAM LITHOGRAPHY

PROXIMITY EFFECT How to beat it?

- Use small energy beam
  - Backscattered distance below 100 nm
  - Resolution limited by forward scattering : only for thin resist layer
- Use high energy beam
  - Dilution of proximity effect on large area
- Dose correction
  - Optimization of the dose for regular array of small objects
  - Empirical correction (higher doses for smaller structures)
  - Simulation of proximity effect and software dose optimization





E-BEAM LITHOGRAPHY

RESOLUTION LIMIT

beam:

- Thick resists (forward scattering)
- Thin resists (~0.5 nm by diffraction, de Broglie wavelength)
- SE range (5-10 nm)

resist:

- Polymer size (5-10 nm)
- Chemically Amplified Resists (acid diffusion ~50 nm)

In practice, best achievable resolution:
in polymer resists ~ 20 nm
(in inorganic resists, currently impractical, ~ 5 nm)

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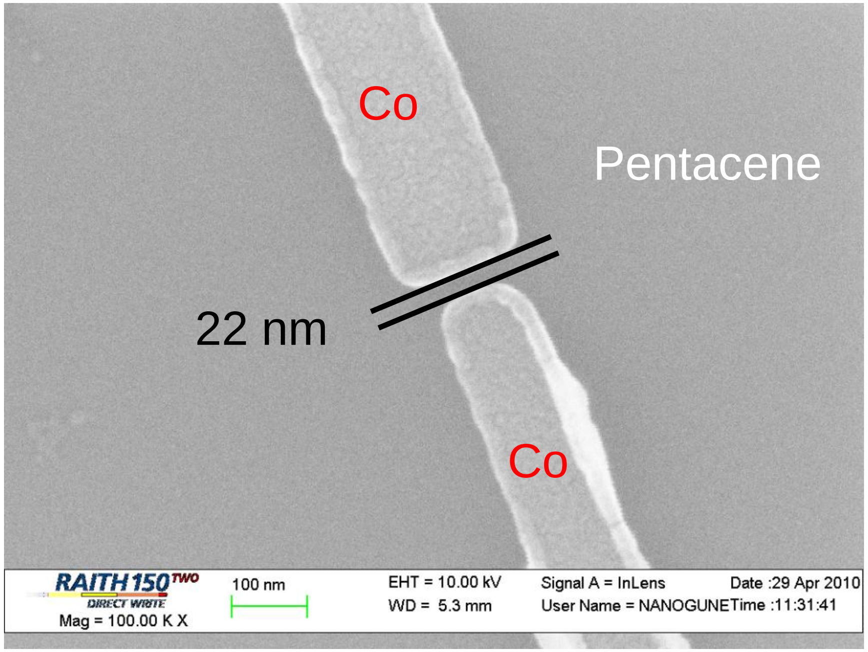
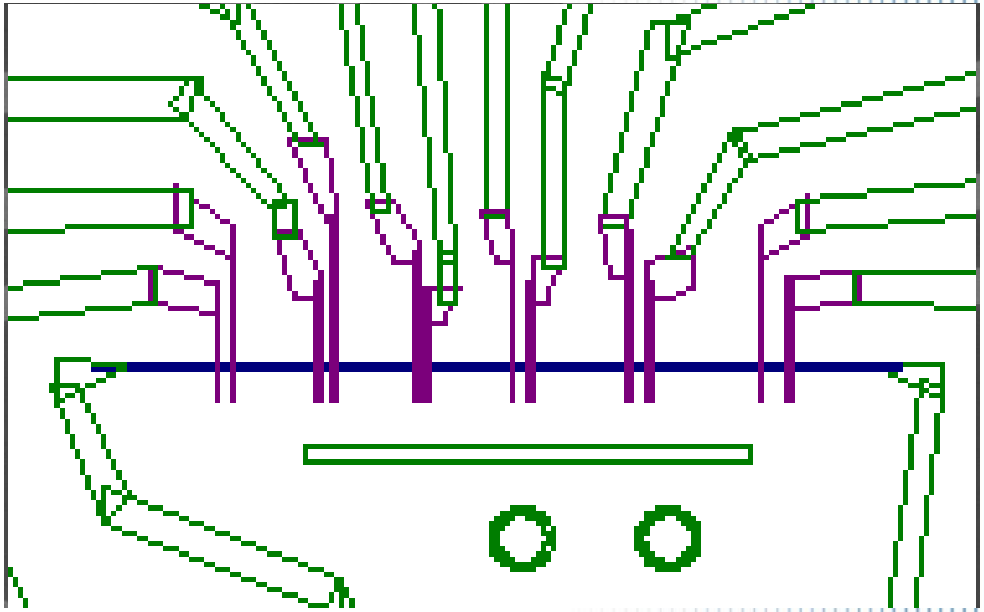
E-BEAM LITHOGRAPHY

Pros:
•Resolution ~10-20 nm
•Easy to change pattern (ideal for research)
•Multiple steps for complex nanostrucutres

Cons:
•Low throughput (the beam writes continuously)





OUTLINE

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- Common problems
  - Sharp edges
  - Redeposition
  - Insulating substrates
- Spintronics applications
  - Lateral spin valve devices (for spin transport)
  - Spin absorption devices (for spin Hall effect)

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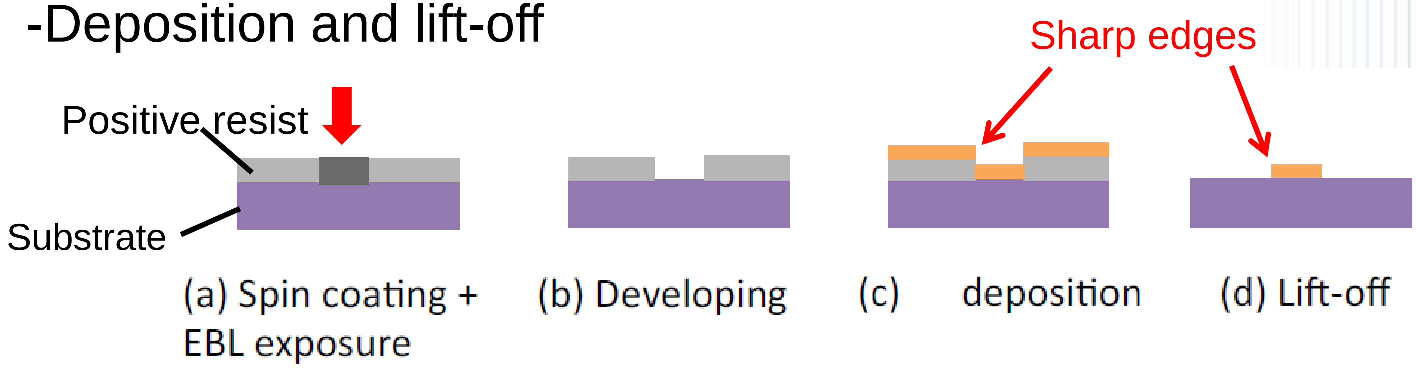
COMMON PROBLEMS

After e-beam lithography:

-Deposition and lift-off

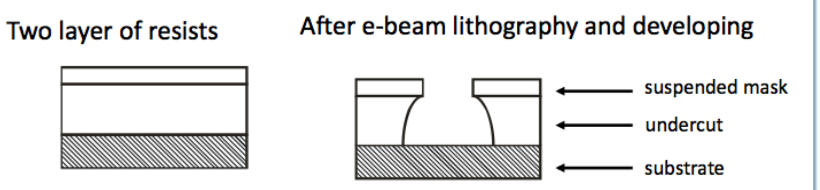


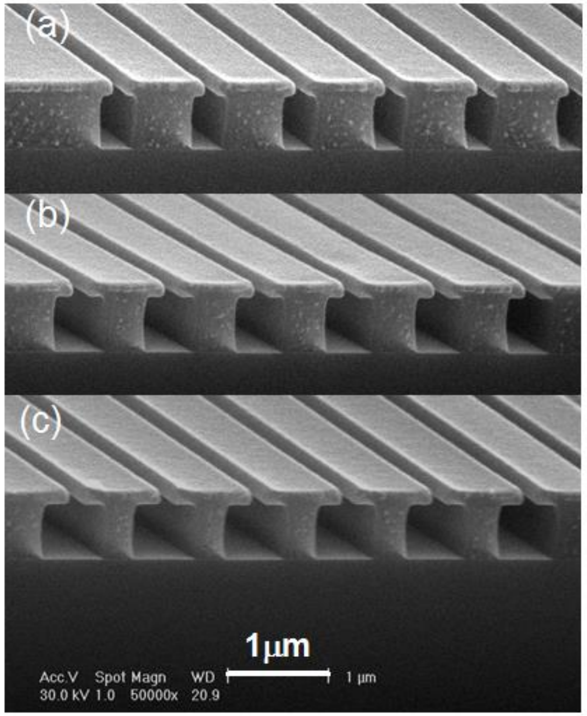
COMMON PROBLEMS: sharp edges

- Appearence of sharp edges depends on:

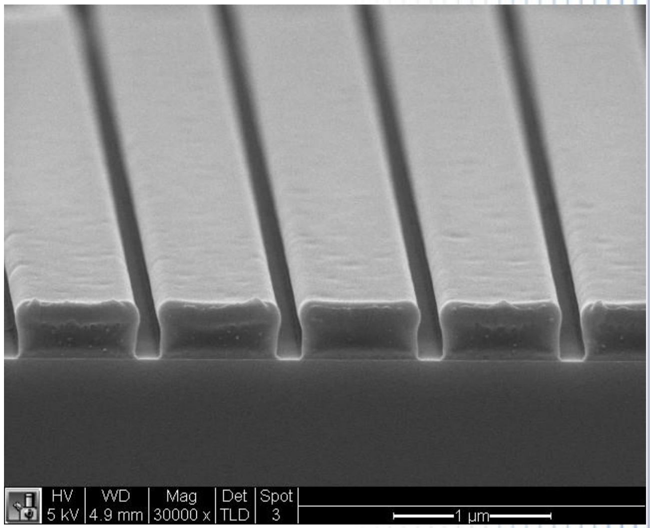
-Hardness of material to deposit (Au vs. magnetic materials)

-Single layer resist vs. Double layer resist





LOR / 950 PMMA A2



495 PMMA A2 / 950 PMMA A2

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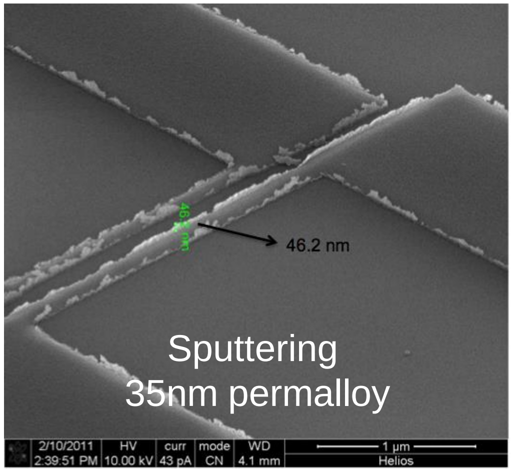
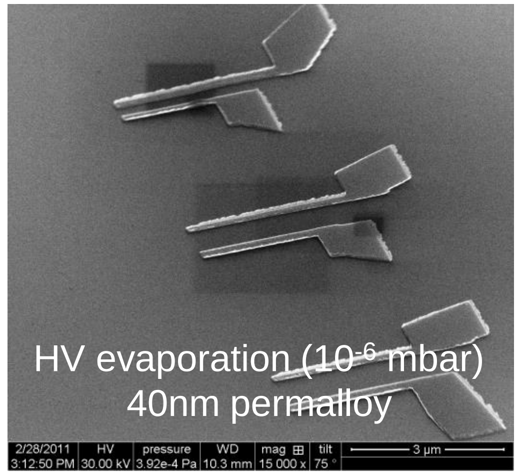
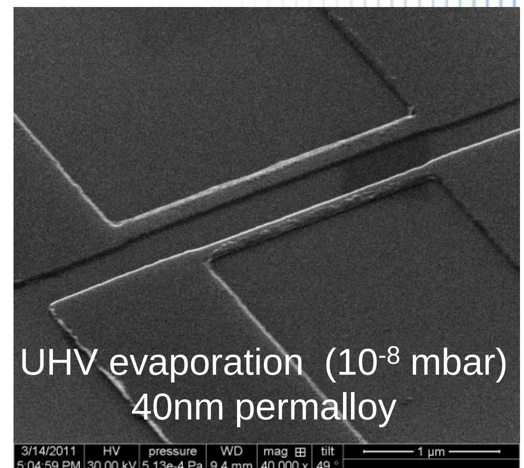
COMMON PROBLEMS: sharp edges

-Deposition type (sputtering vs. evaporation)

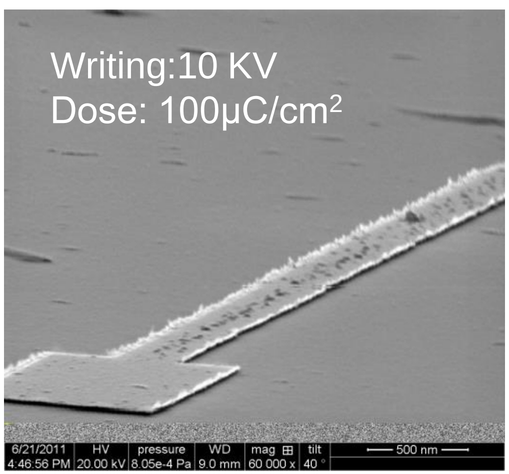
Writing: 20kV and ≈50pA
Dose:100-475μC/cm^2

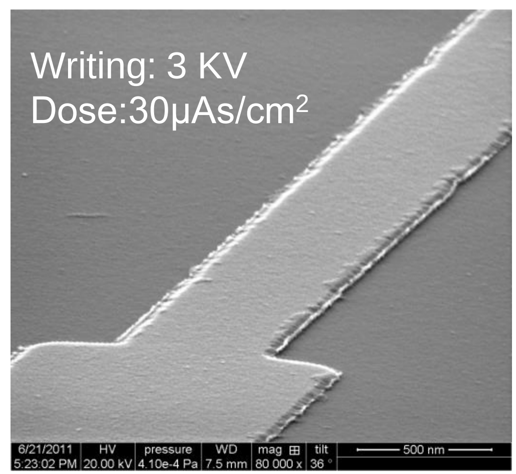






-E-beam exposure conditions





HV evaporation (10^{-6} mbar)
20nm permalloy

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COMMON PROBLEMS: sharp edges

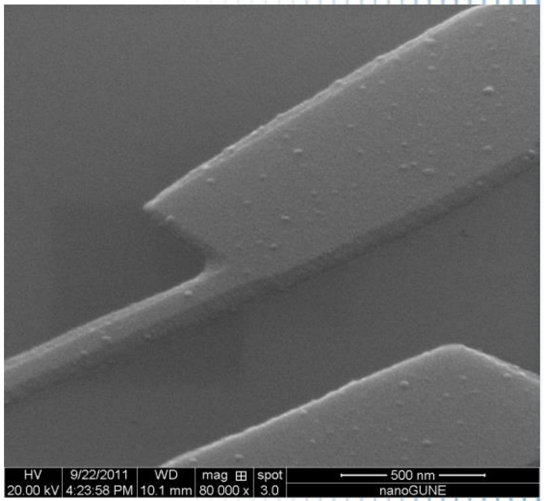
-Combination of resists

Double PMMA (495/950)

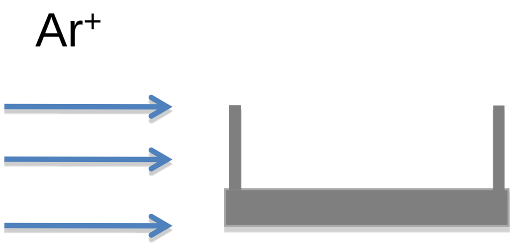
-same developer and remover
-small undercut
-cleaner

LOR /PMMA

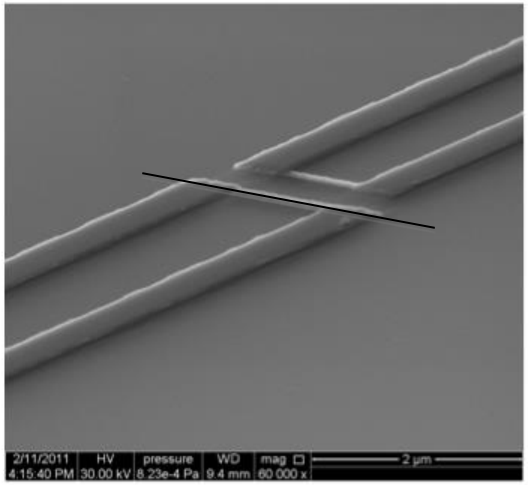
-2 developers and 2 removers
-large undercut
-dirtier

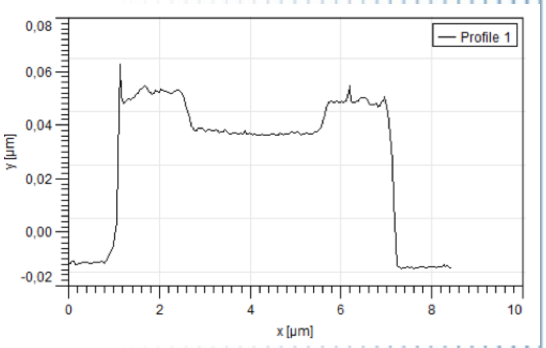


- Can be improved with subsequent Ar-ion milling



-Parameters: ion energy, etching time
-Not useful if edges are too tall
-Narrower regions are more etched!





Atomic Force Microscopy profile

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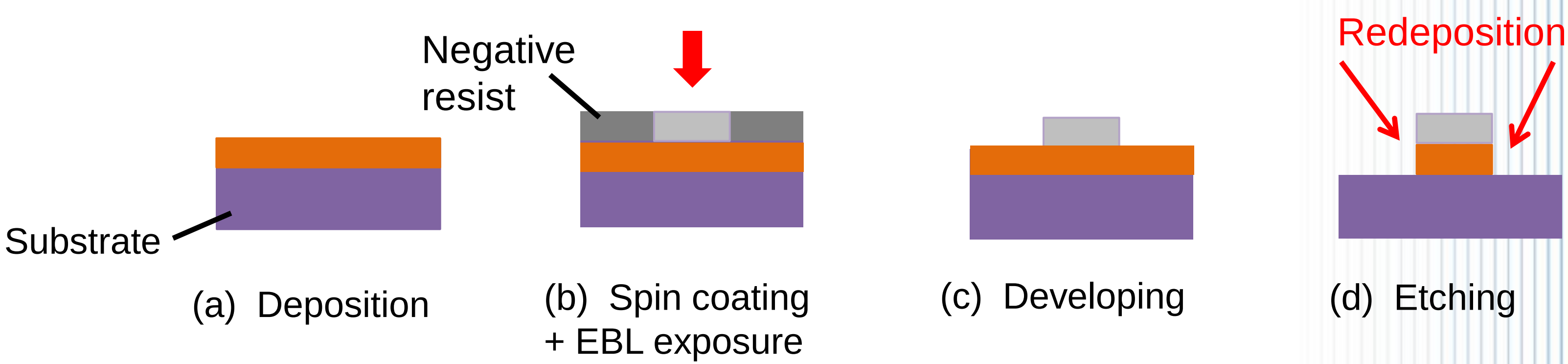
COMMON PROBLEMS

After e-beam lithography:

-Deposition and lift-off

-Etching



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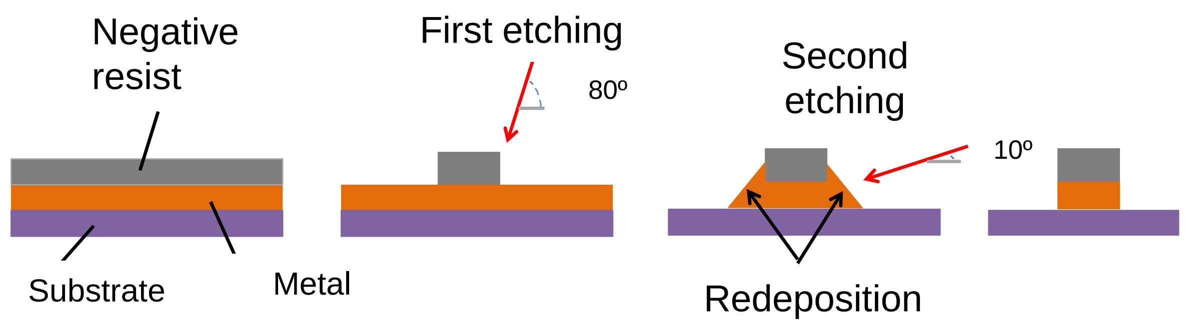
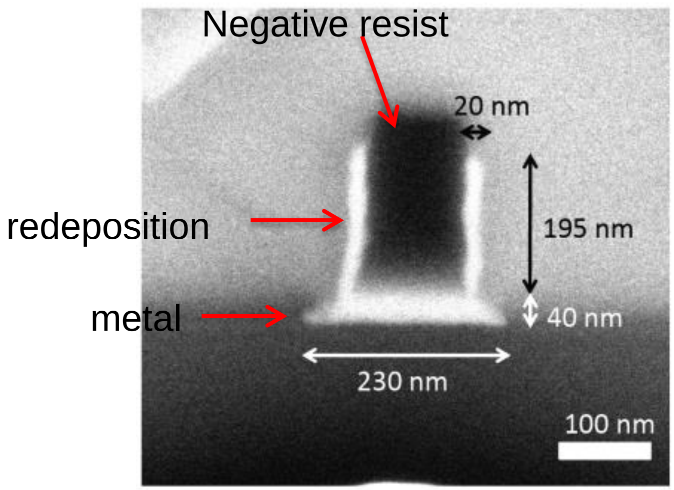
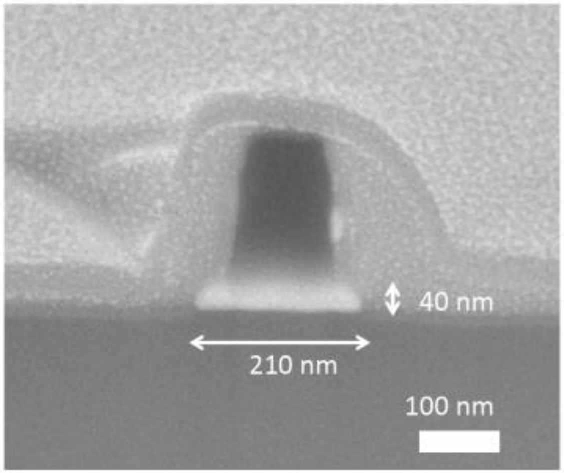
COMMON PROBLEMS: redeposition

- Redeposition during etching:







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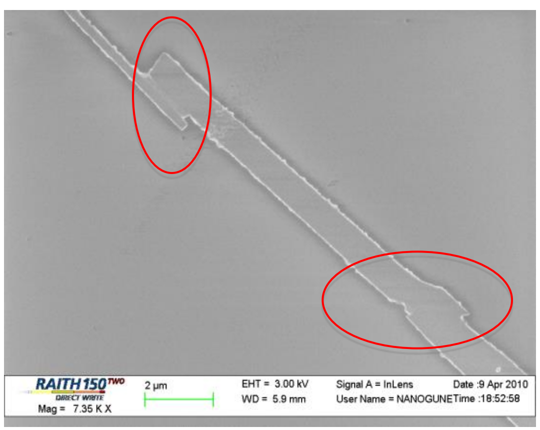
COMMON PROBLEMS: insulating substrates

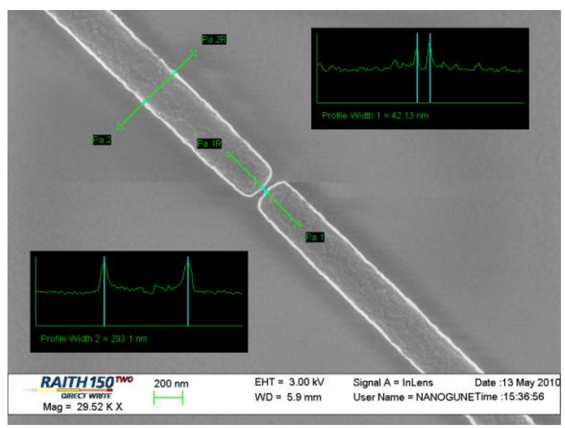
Problem: Substrate gets negatively charged and deflects e-beam

Solution: Optimization of conductive overlayers (typically Au) on PMMA for charge dissipation

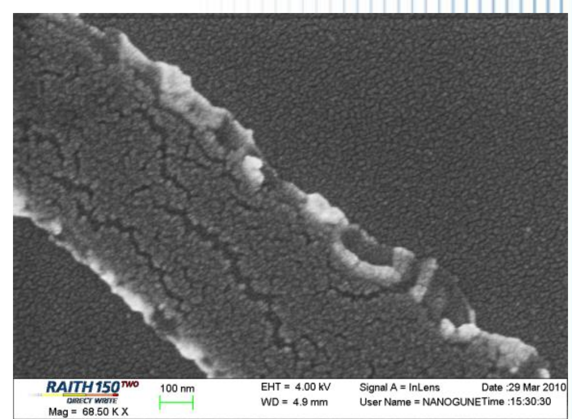
1) Au deposition (1.5 nm) by sputtering on top of PMMA before e-beam exposure
2) Au etching with KI + I_2 (aq) before development



No Au



1.5 nm Au



3 nm Au

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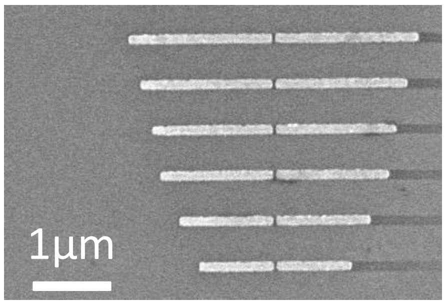
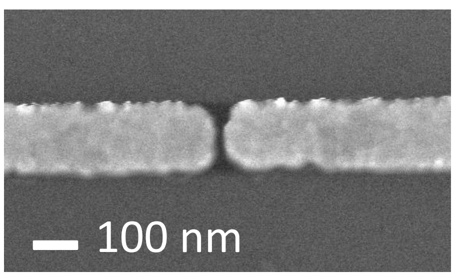
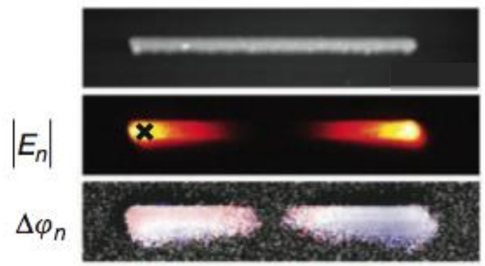
✓ Application: nano-optics, plasmonic nanoantennas

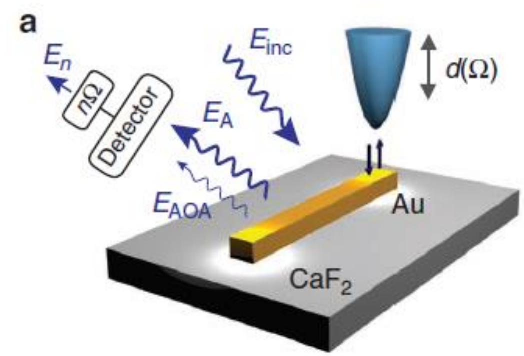
Need of regular arrays of elements on insulating substrates (CaF_2, glass)



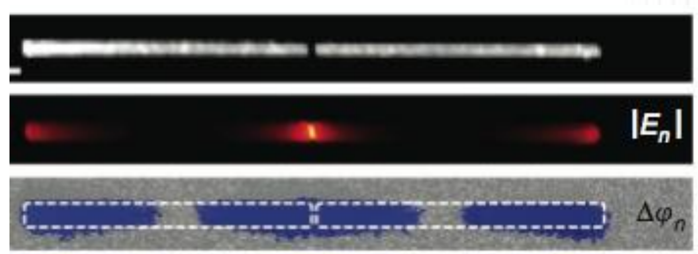




Resonance modes appear when nanorod length = λ/2



Measured with s-SNOM



Enhanced hot spots in nanogaps

P. Alonso-Gonzalez et al., Nature Comm. **3**, 684 (2012)

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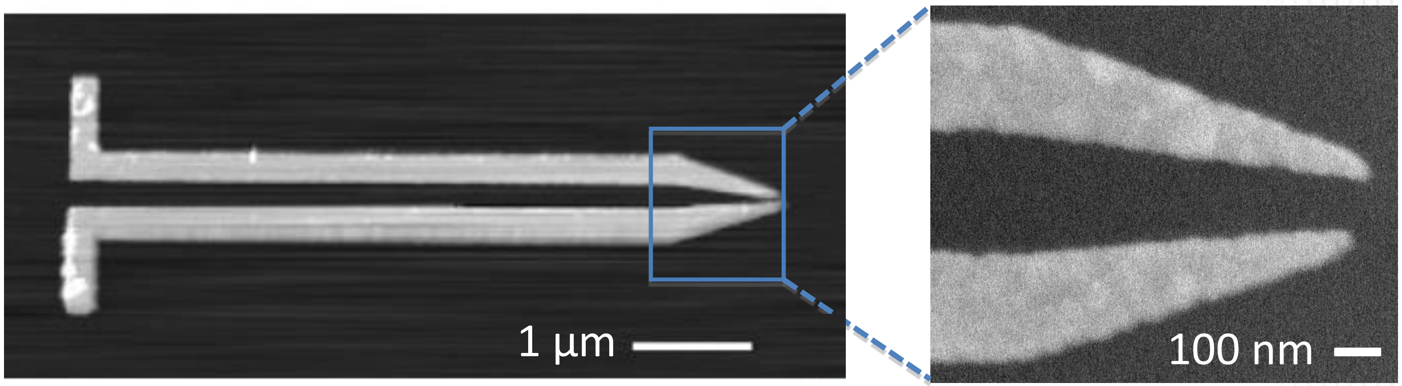
EXAMPLES: Nano-optic devices on insulating substrates

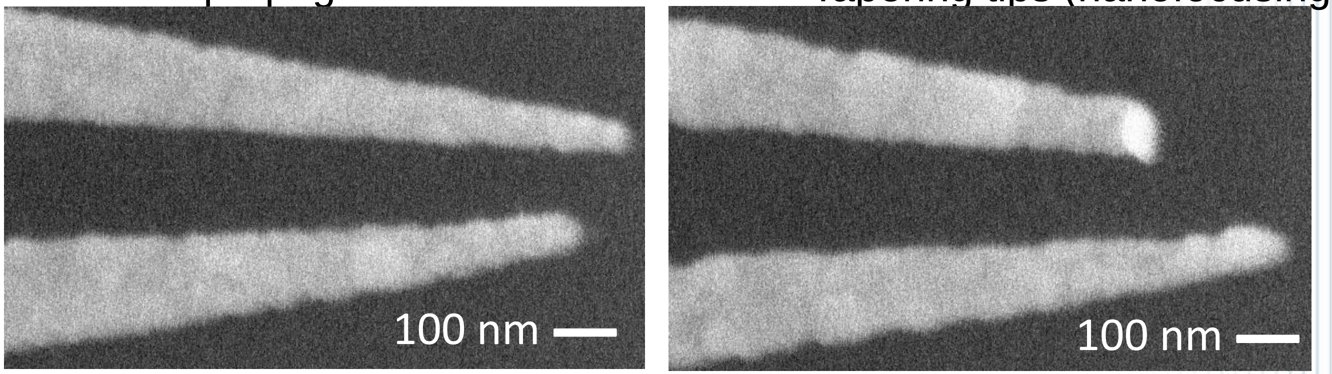
✓ Application: nano-optics, infrared waveguides

Need of combination of very different feature sizes on insulating substrates (CaF_2, glass)



Resonance and propagation of the E field

Tapering tips (nanofocusing of E field)



M. Schnell et al., Nature Photonics 5, 283 (2011)
P. Sarriugarte et al., Optics Comm. 285, 3378 (2012)

Control of evaporation angle!

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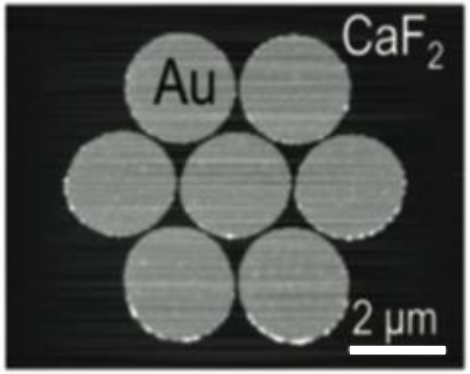
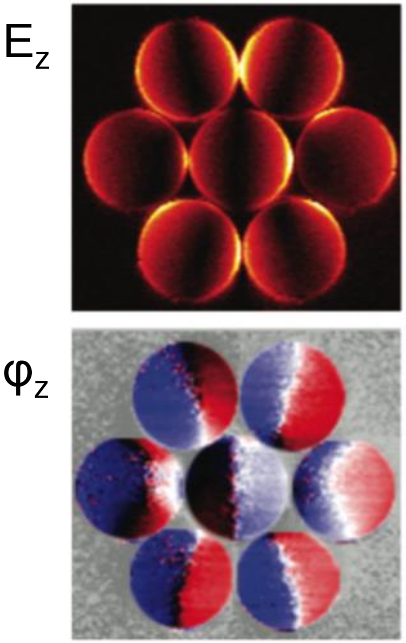
EXAMPLES: Nano-optic devices on insulating substrates

✓ Application: nano-optics, heptamers

Need of structures with large area/small separation on insulating substrates (CaF_2, glass)



Playing with dose and distance –
proximity effect corrections not used



Observation of the interference of resonance modes

P. Alonso-Gonzalez et al., Nano Lett. 11, 3922 (2011)

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OUTLINE

- Introduction to electron-beam lithography
  - Working principle
  - Complete process
  - Key parameters
  - Proximity effect
- Common problems
  - Sharp edges
  - Redeposition
  - Insulating substrates
- Spintronics applications
  - Lateral spin valve devices (for spin transport)
  - Spin absorption devices (for spin Hall effect)

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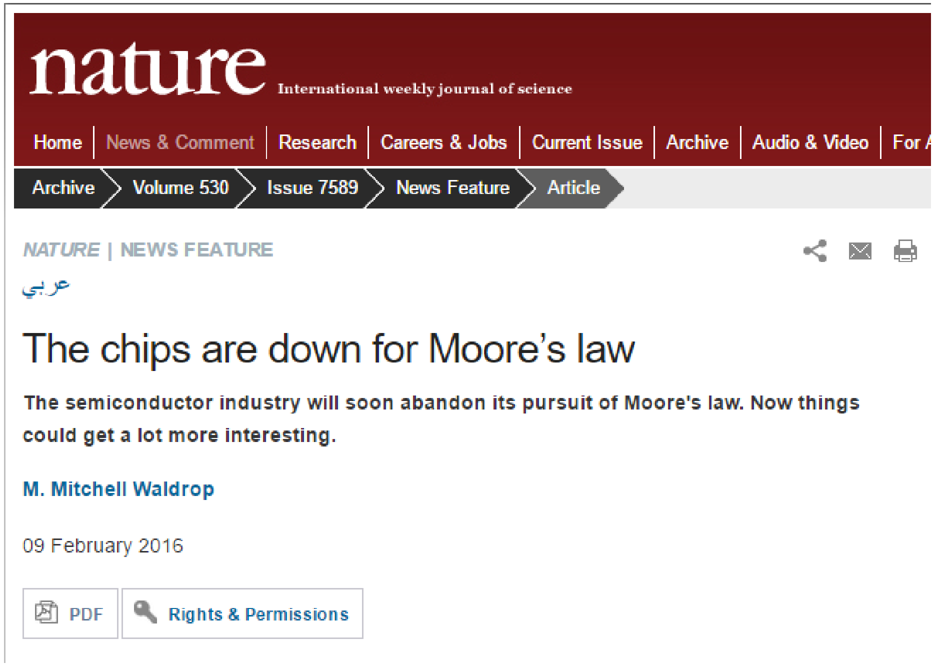
MOTIVATION

Moore’s law



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Archive > Volume 530 > Issue 7589 > News Feature > Article

NATURE | NEWS FEATURE

عربي

The chips are down for Moore’s law

The semiconductor industry will soon abandon its pursuit of Moore's law. Now things could get a lot more interesting.

M. Mitchell Waldrop

09 February 2016

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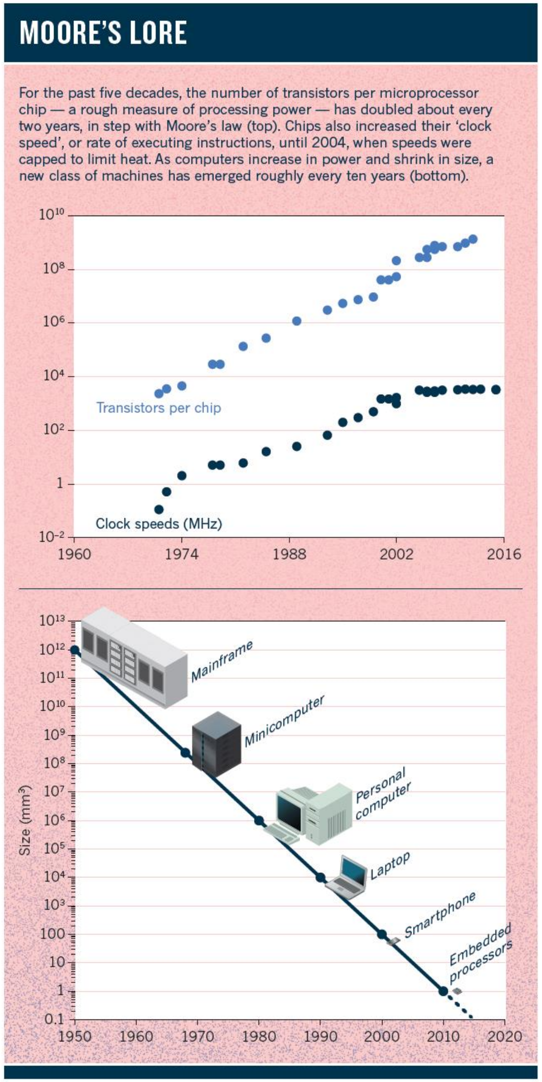

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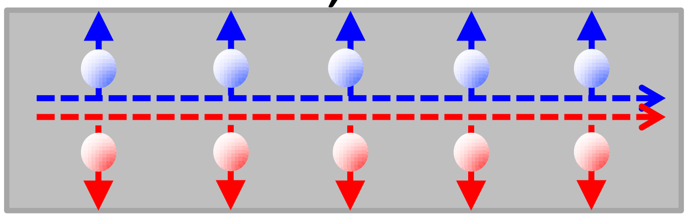
MOTIVATION

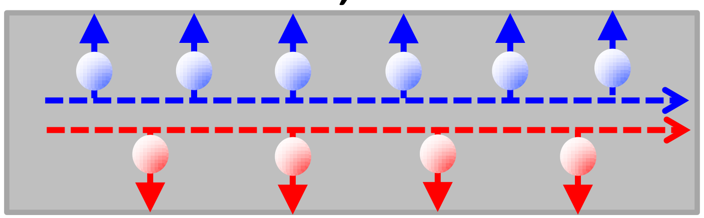
$I_c = I_{\uparrow} + I_{\downarrow}$, $I_s = I_{\uparrow} - I_{\downarrow}$

$I_c \neq 0, \; I_s = 0$



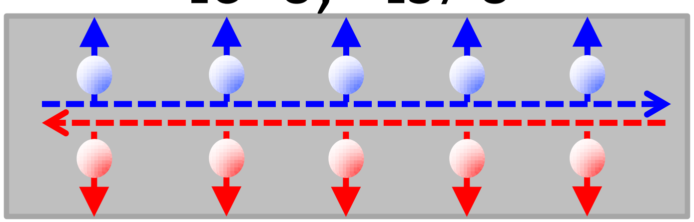
Charge current

$I_c \neq 0, \; I_s \neq 0$



Spin polarized current

$I_c = 0, \; I_s \neq 0$



Pure spin current

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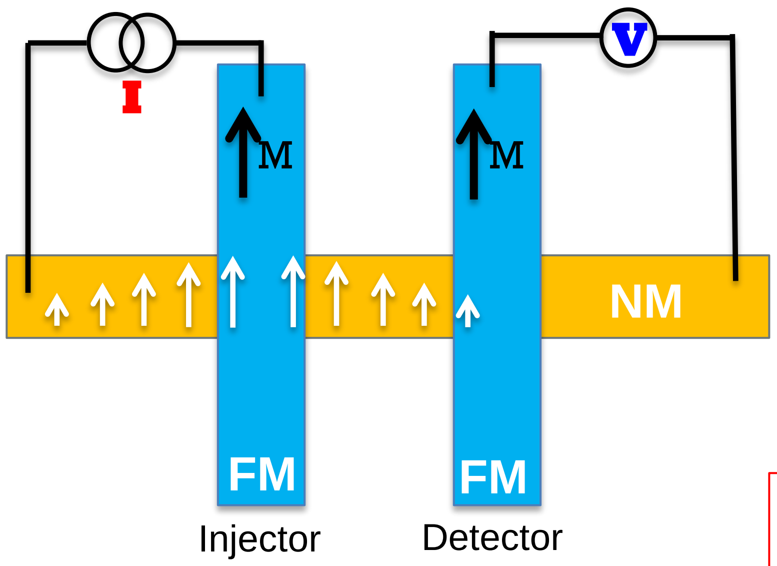
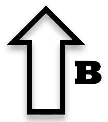
Non-local measurement



$$L < \lambda_N$$



Decoupling of spin current from charge current

Nanoscale fabrication required

F. J. Jedema et al., Nature **410**, 345 (2001)
S. O. Valenzuela et al., APL **85**, 5914 (2004)
T. Kimura et al., PRB **72**, 014461 (2005)
Y. Ji et al., J. Phys. D: Appl. Phys. **40**, 1280 (2007)
F. Casanova et al., PRB **79**, 184415 (2009)
P. Laçzkowski et al., APEX **4**, 063007 (2011)

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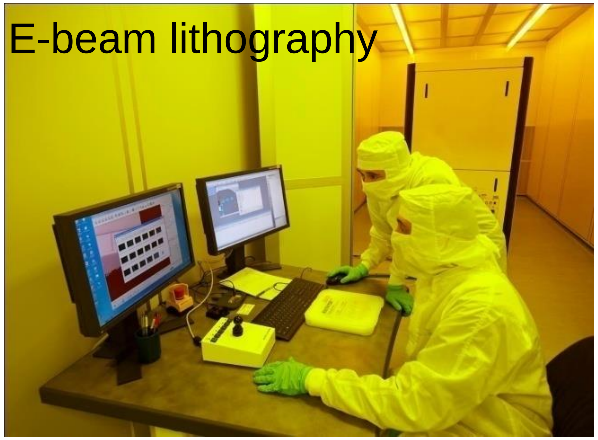
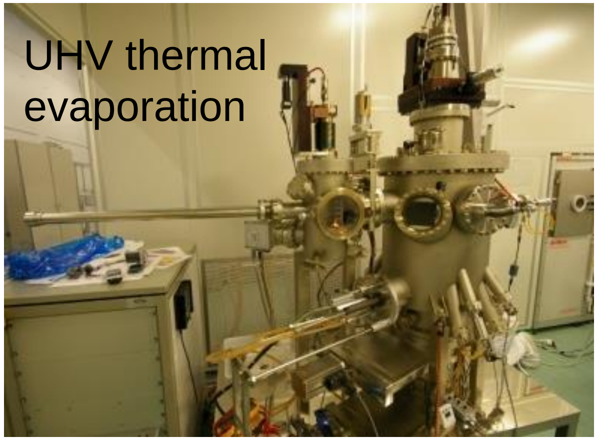
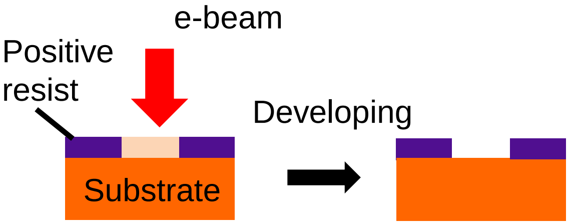
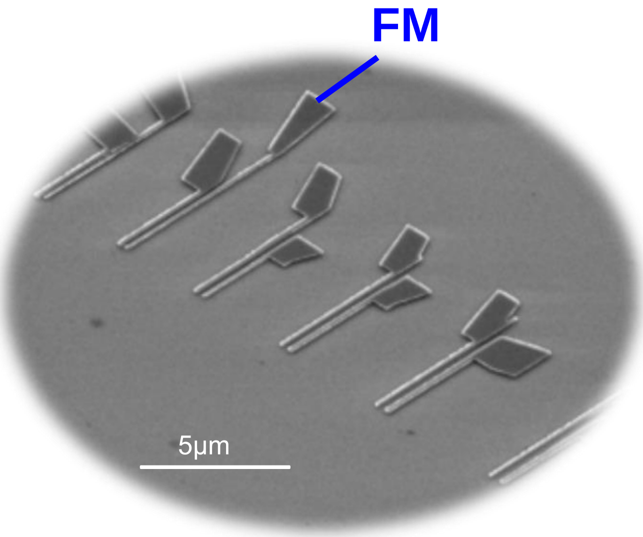
SPINTRONICS: Metallic lateral spin valves

Two-step lithography and deposition process









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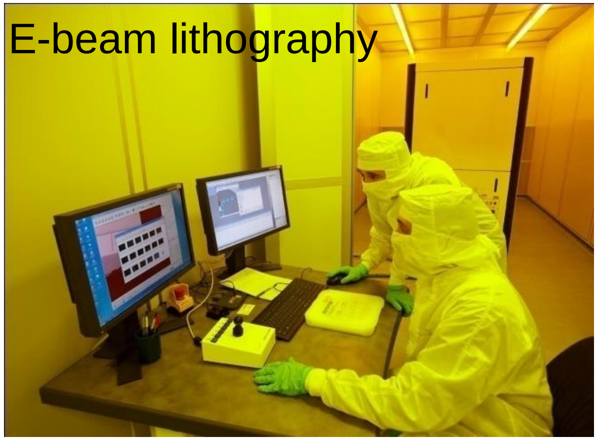
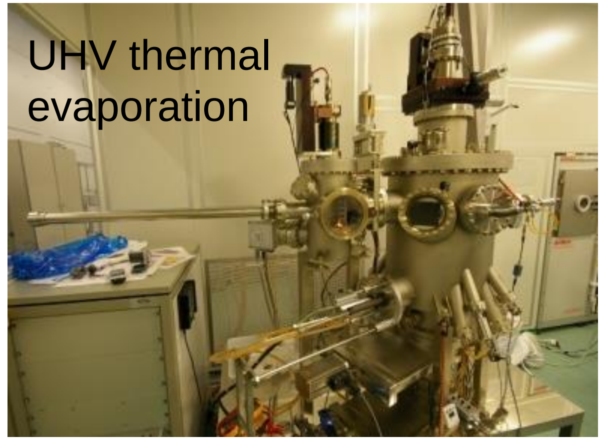
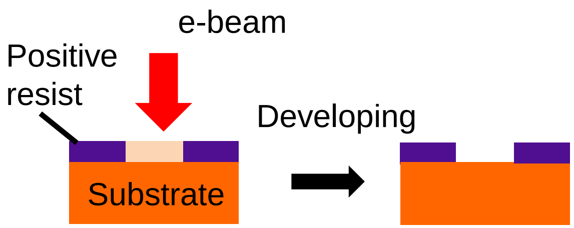
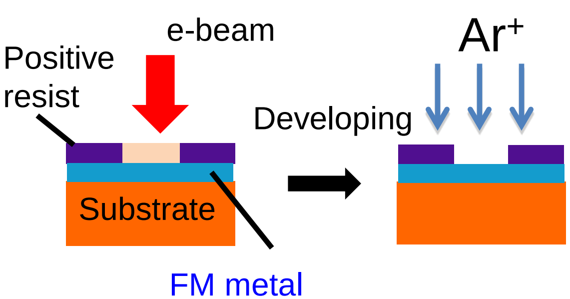
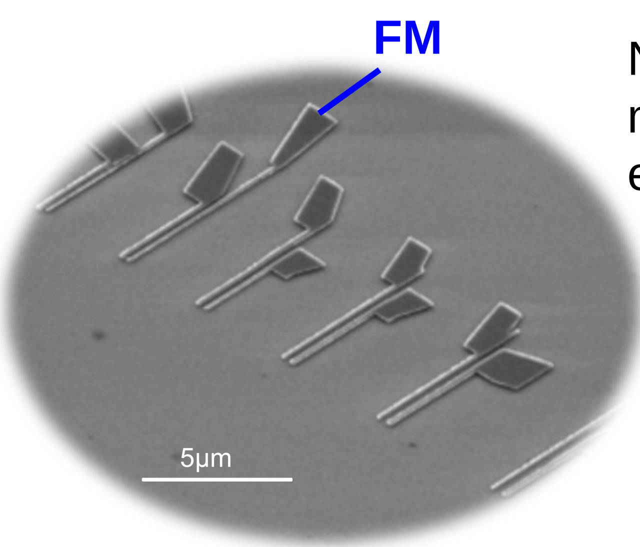
Two-step lithography and deposition process











Need of clean and smooth metal-metal interfaces for an efficient spin injection

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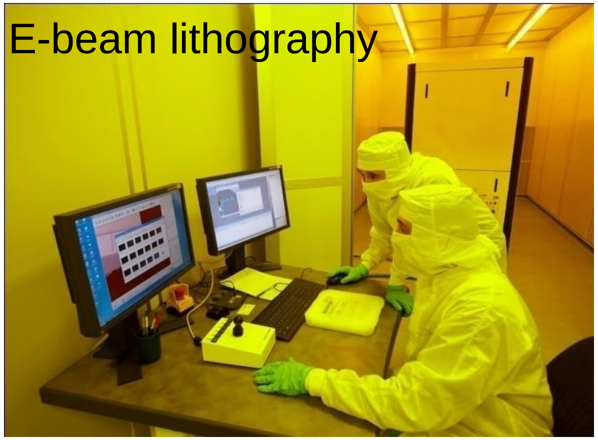
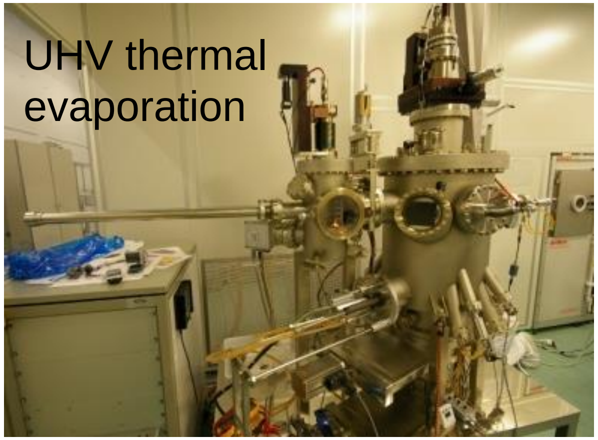
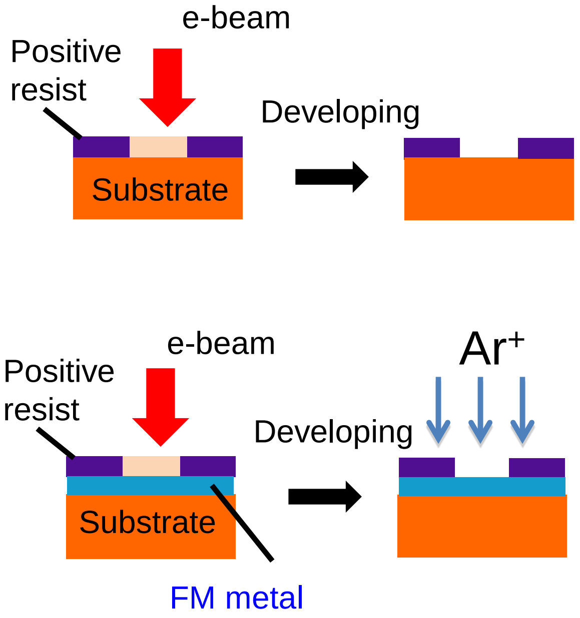
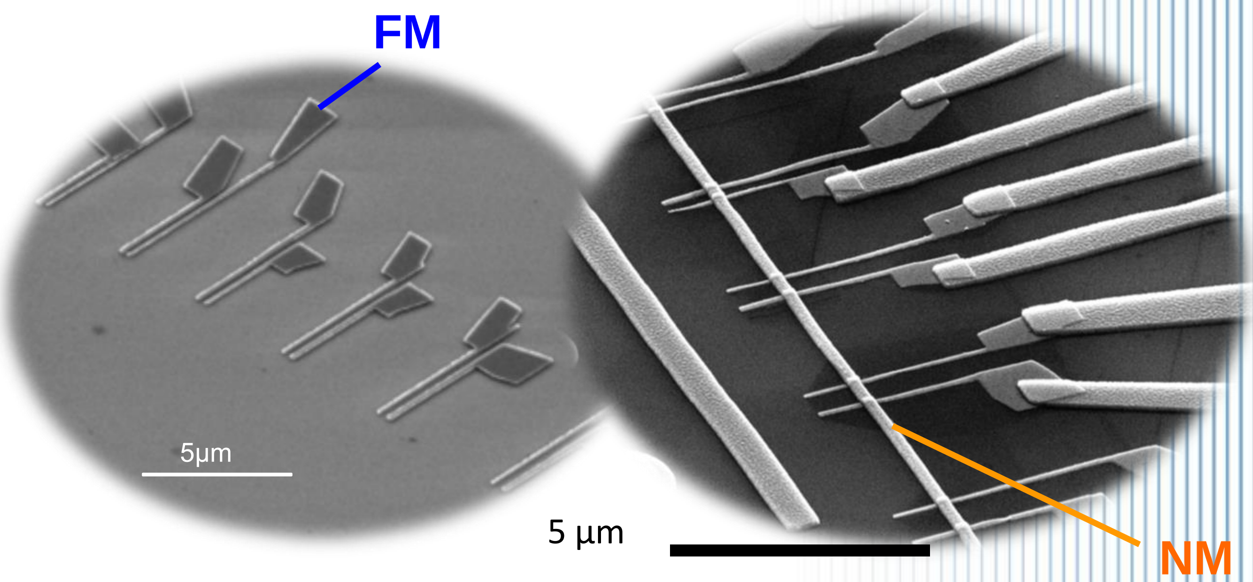
SPINTRONICS: Metallic lateral spin valves

Two-step lithography and deposition process









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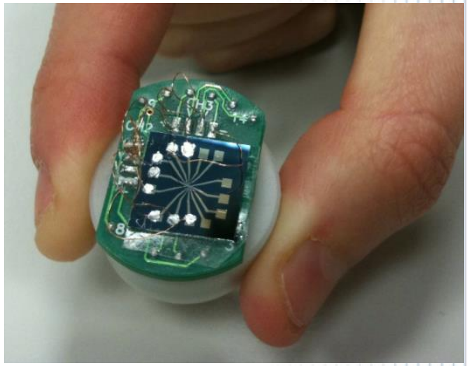
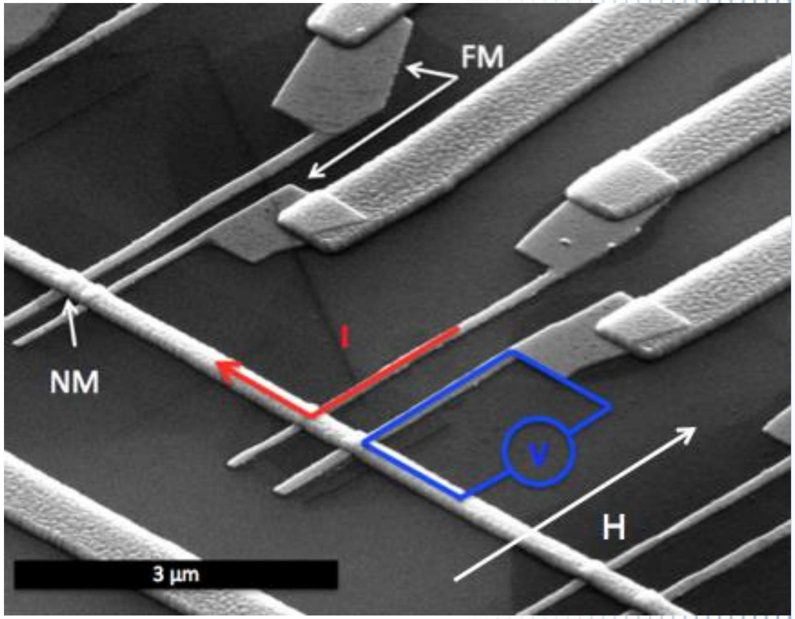
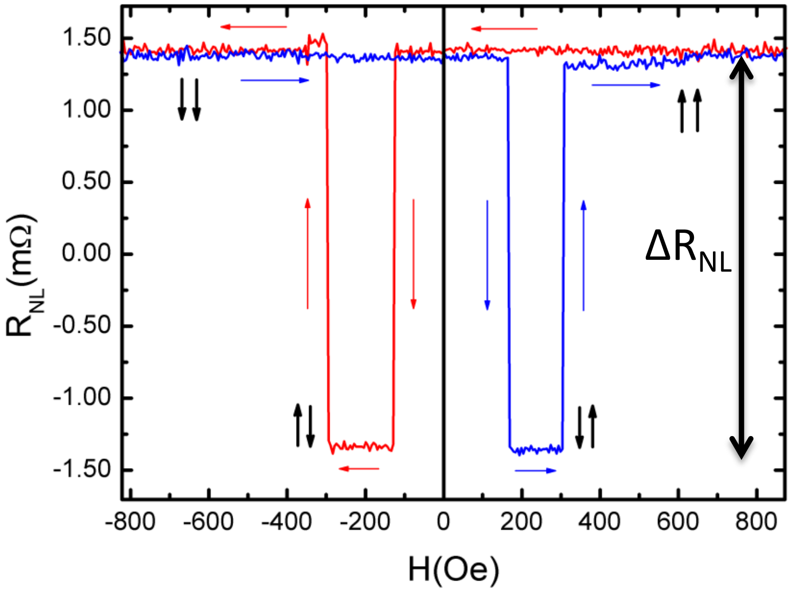
Non-local measurement

- Performed in a liquid He cryostat under an applied external magnetic field
- Non-local resistance is measured: $R_{NL} = \frac{V}{|I|}$
- Spin signal: $\Delta R_{NL} = \frac{V_p - V_{AP}}{|I|}$







E. Villamor *et al.*, PRB **87**, 094417 (2013)

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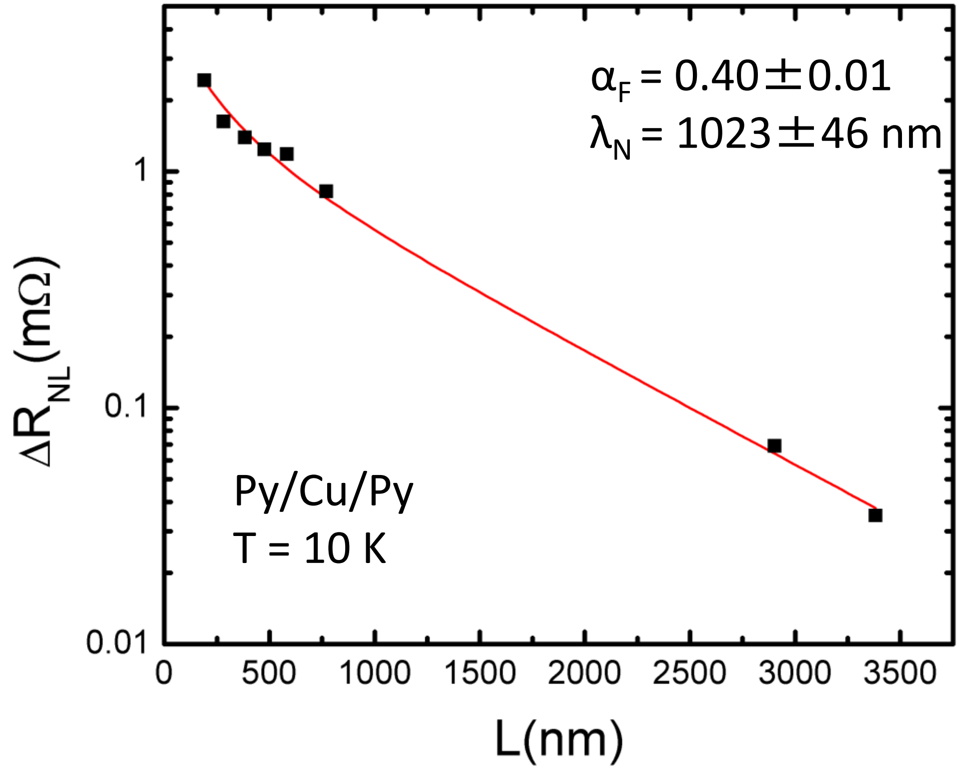
SPINTRONICS: Metallic lateral spin valves

From the one dimensional spin-diffusion model:

$$\Delta R_{NL} = \frac{\Delta V}{|I|} = \frac{\alpha_F^2 R_N}{\left(2+\frac{R_N}{R_F}\right)^2 e^{L/\lambda_N} - \left(\frac{R_N}{R_F}\right)^2 e^{-L/\lambda_N}}$$



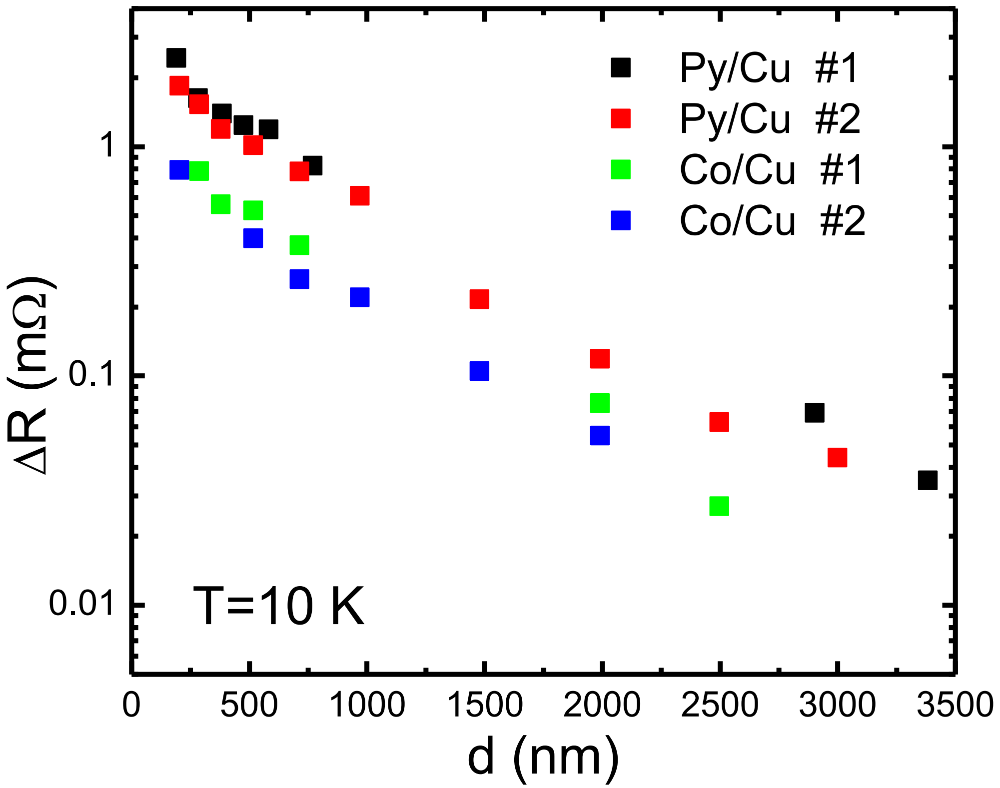
α_F → Spin polarization of **FM** (Spin injection)

λ_N → Spin diffusion length of **NM** (Spin transport)

L → Distance between **FM** injector and detector

E. Villamor *et al.*, PRB **87**, 094417 (2013)
E. Villamor et al., PRB **88**, 184411 (2013)

SPINTRONICS: Metallic lateral spin valves



High reproducibility:

-Same α_F for the same **FM** and milling time

-Different α_F for a different **FM** (Py vs Co)

-Same λ_N for the same **NM** channel (Cu) and different **FM**

Large dispersion in the literature:

-Interface quality (α_F)

-NM channel purity (λ_N)

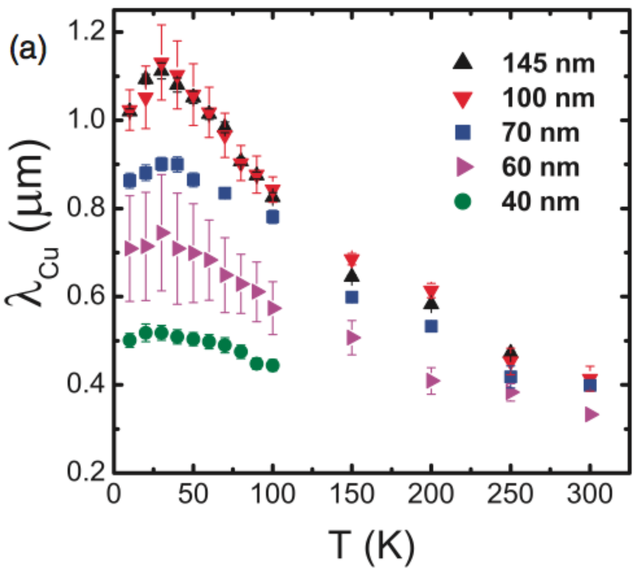
E. Villamor *et al.*, PRB **87**, 094417 (2013)
E. Villamor et al., PRB **88**, 184411 (2013)

SPINTRONICS: Metallic lateral spin valves

Long spin diffusion length in Cu, Ag, Al

Enhance the spin transport properties controlling **spin relaxation** mechanisms

- **Grain boundaries**
- Impurities
- Phonons
- Surface



Control the Ag growth:

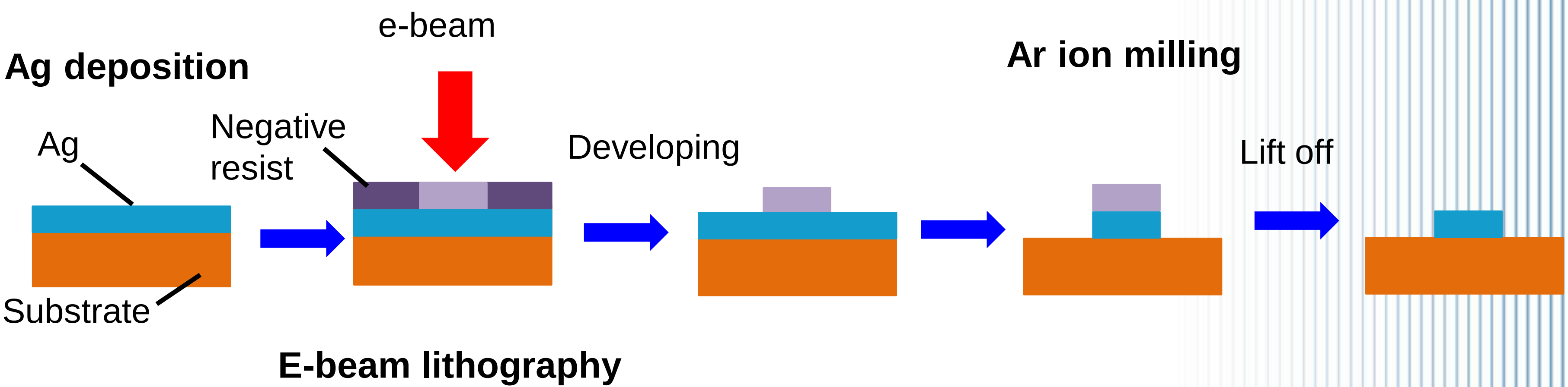
- Epitaxial Ag
- Polycrystalline Ag

G. Mihajlovic *et al.*, PRL **104**, 237202 (2010)
Y. Fukuma *et al.*, Nature Mater. **10**, 527 (2011)

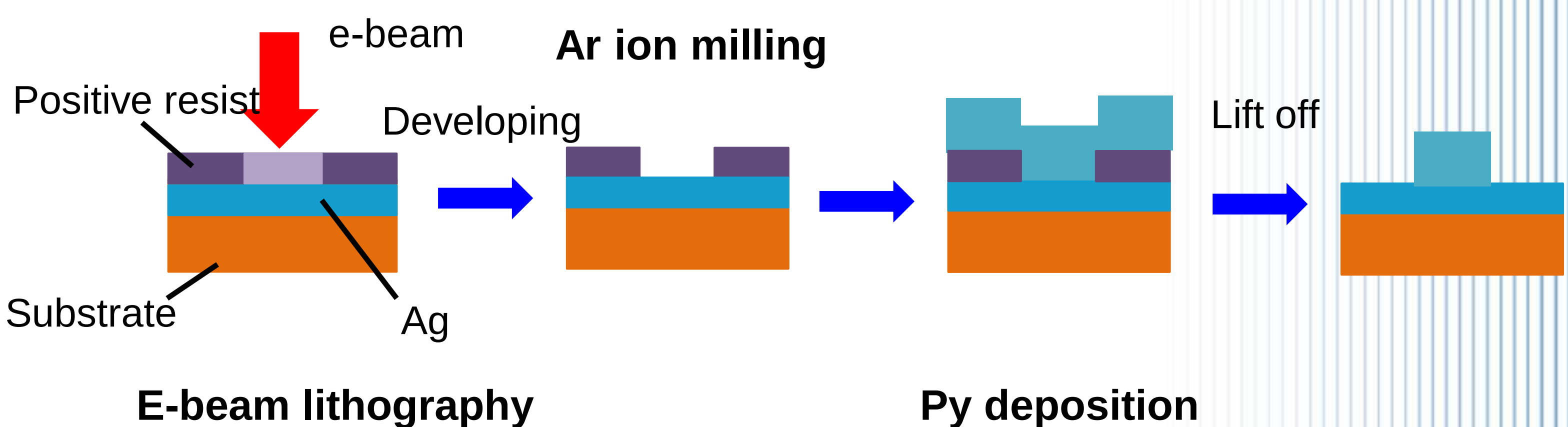
E. Villamor *et al.*, PRB **87**, 094417 (2013)

Alternative two-step lithography process

- 1st step: NM channel



- 2nd step: FM electrodes



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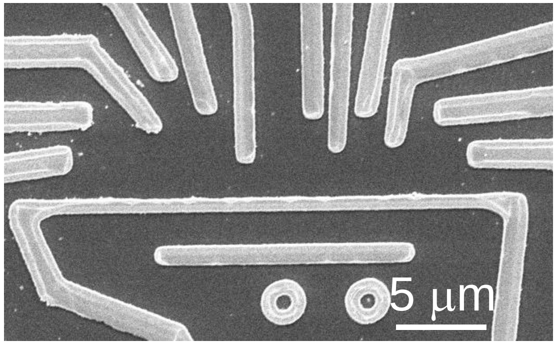
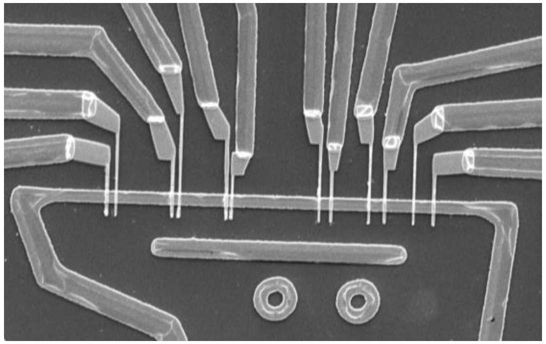
Alternative two-step lithography process

- 1st step: NM channel



- 2nd step: FM electrodes



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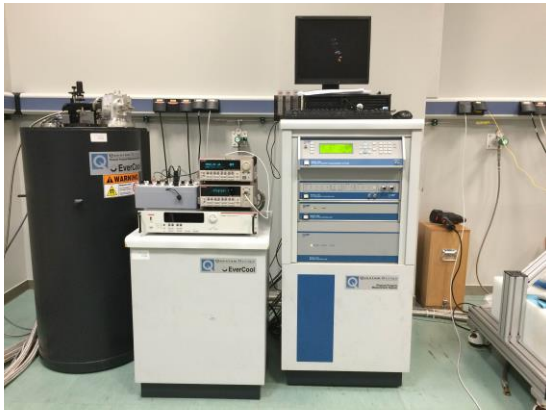
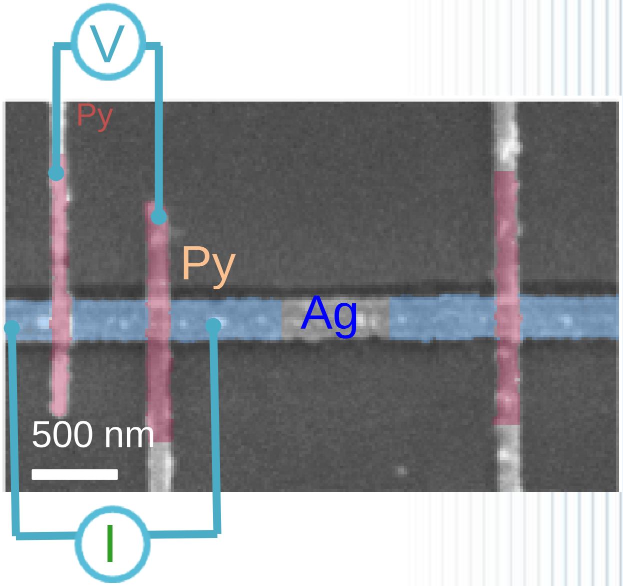
SPINTRONICS: Metallic lateral spin valves

Electrical measurements





Resistivity

- $\rho_{epitaxial}$= 1.06 $\mu\Omega$ cm
- $\rho_{polycrystalline}$= 2.22 $\mu\Omega$ cm

M. Isasa *et al.*, J. Phys. D: Appl. Phys. **48**, 215003 (2015)

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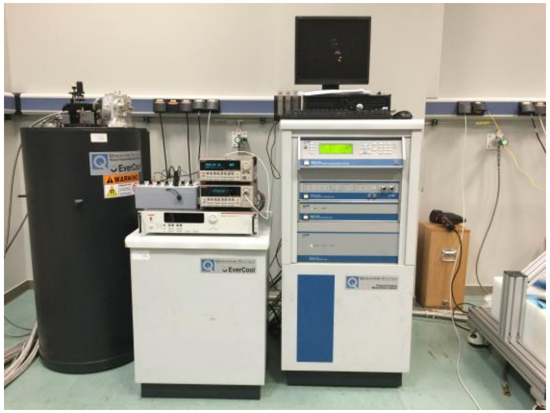
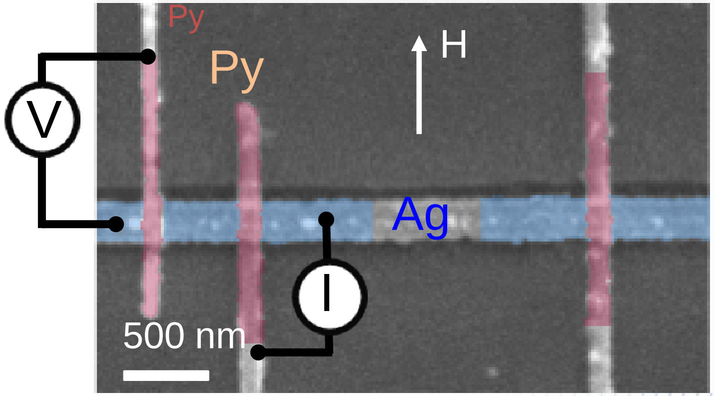


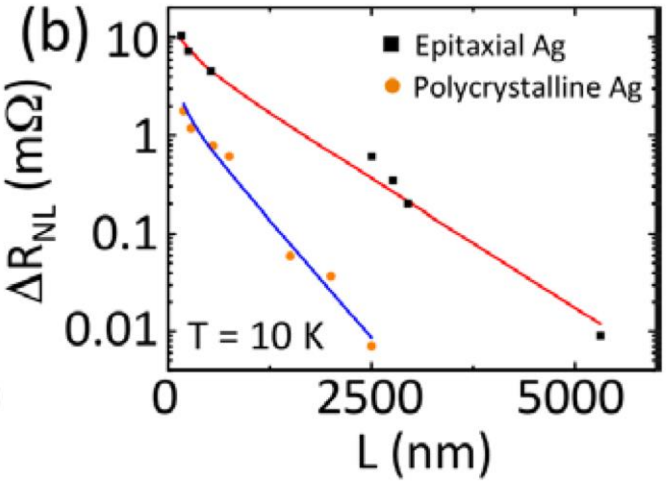


Resistivity

- $\rho_{epitaxial}$= 1.06 $\mu\Omega$ cm
- $\rho_{polycrystalline}$= 2.22 $\mu\Omega$ cm

Spin transport properties

- $\lambda_{epitaxial}$= 823 $\pm$ 59 nm
- $\lambda_{polycrystalline}$= 449 $\pm$ 30 nm



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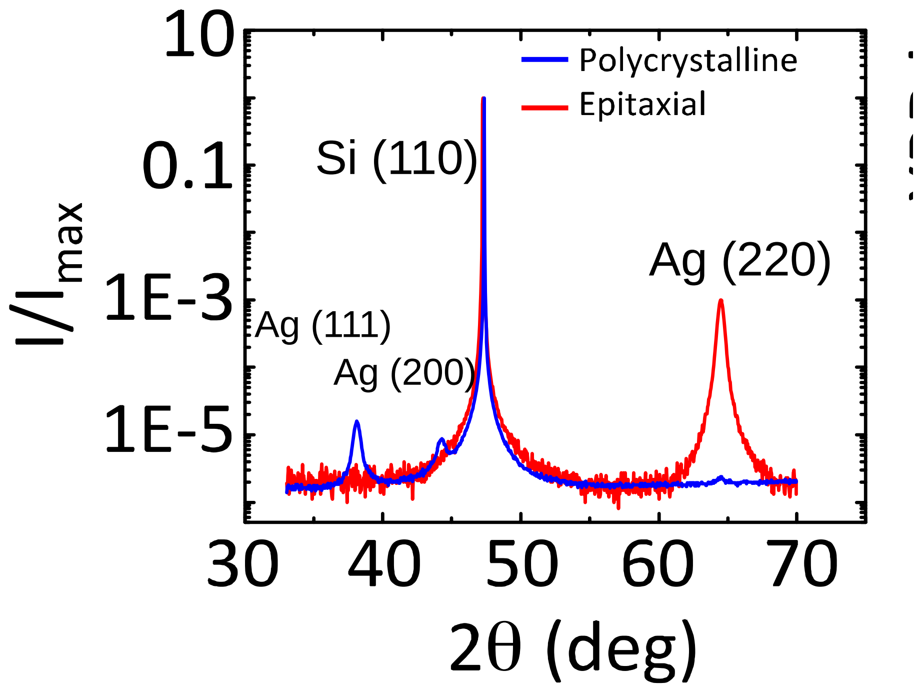
XRD measurements



Epitaxial Ag

- Grain size = 41 ± 4 nm
- Less grain boundaries

Polycrystalline Ag

- Grain size = 19 ± 6 nm
- More grain boundaries

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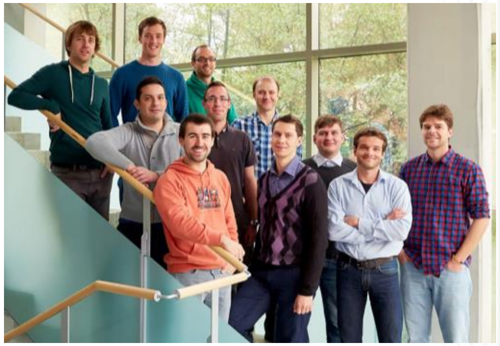
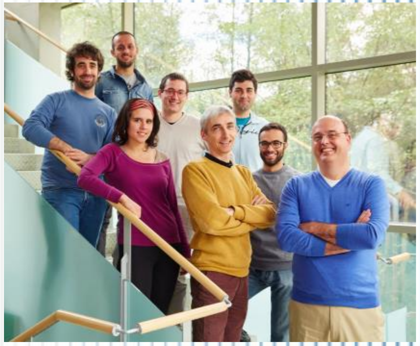
ACKNOWLEDGMENTS

Nanodevices

Nanooptics



Nanomagnetism



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MARIE CURIE ACTIONS

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