

Electron Holography studies of the magnetic Nanowires

Etienne Snoeck, Christophe Gatel,
Luis Alfredo Rodriguez, David Reyes, Aurélien Masseboeuf,
Bénédicte Warot, Nicolas Biziere



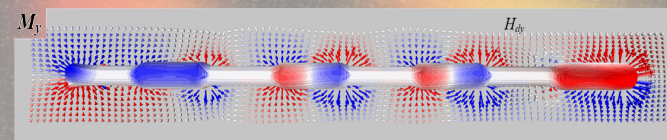
CEMES - CNRS
Toulouse - France



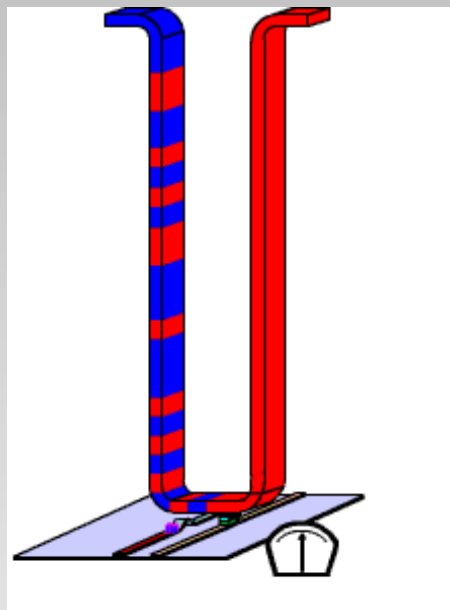
C. Bran, M. Vázquez, and A. Asenjo



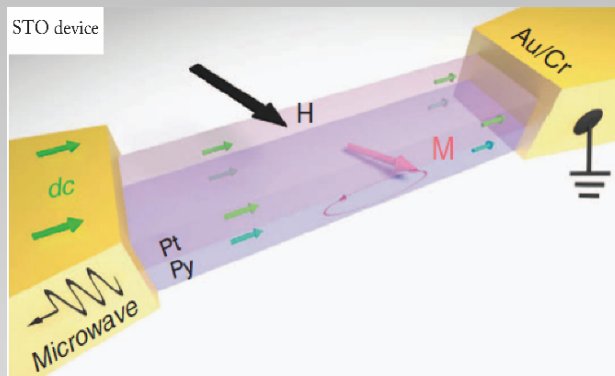
ICMM-CSIC
Madrid - Spain



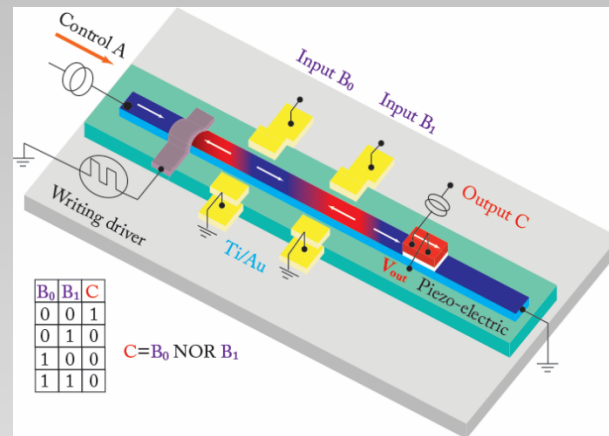
MOTIVATION : Domain Wall (DW) manipulation information storage technologies, logic systems and sensors.



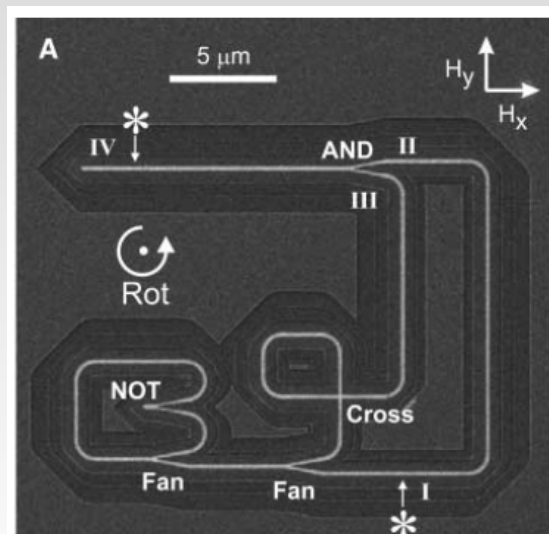
→ Magnetic Racetrack Memory
Stuart Parkin. *Science* 320 (2008)



→ Nanowire spin torque oscillator
Z. Duan et al., *Nat. Commun.* (2014)



→ NOR gate by strain controlled magnetic domain wall propagation
N. Lei et al., *Nat. Commun.* (2013)



→ Magnetic Logic Circuit
D. Allwood et al. *Science* (2005)

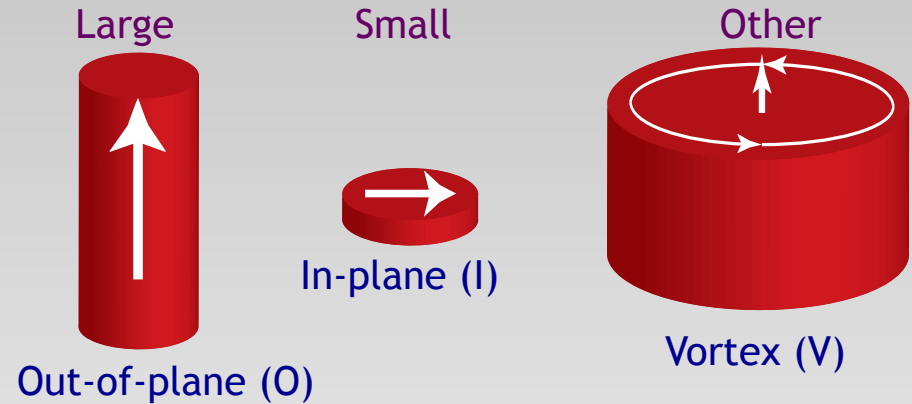
Why Nanowires ?

- DWs motion is the fastest (*even if it depends on the DW conf.*)
- Very high density
- Low cost (*compared to lithography processes*)

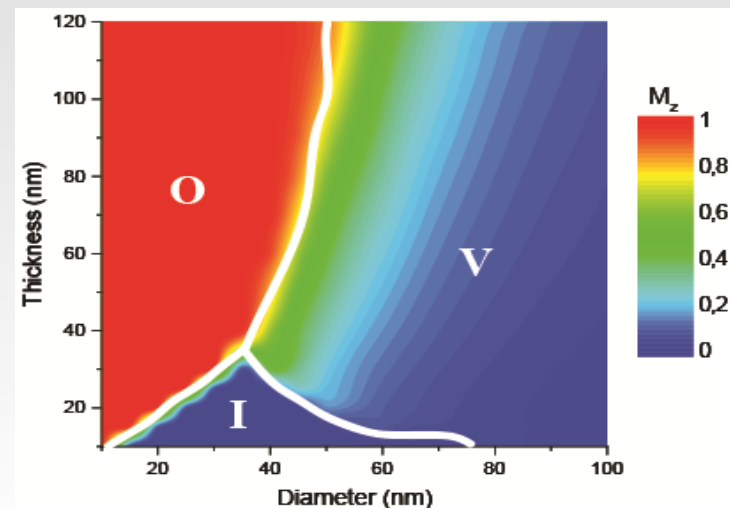
- Cylindrical symmetry of NWs => High propagation velocity of DWs
- DW configuration depends on anisotropy (shape, magnetocrystalline), magnetisation (materials) exchange constant (materials)

Effects on the magnetic configuration and DW structure in NWs of

- Crystallography
- Diameter & aspect ratio (length/diameter)
- Morphology
- Composition
- Defects
- ...



*Phase diagram for permalloy nanocylinders
Chung et al.*



MOTIVATION : Domain Wall (DW) manipulation information storage technologies, logic systems and sensors.

2nd Need: Efficient methods to control DWs in the NWs

→ DWs' fine structure knowledge is required → EH

→ Finding efficient DWs pinning centres ?

In planar nano-ribbons, DWs can be controlled by **changing their lateral dimensions** (*notches, anti-notches, periodic modulations, ...*)

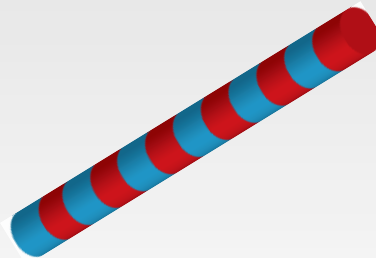
→ *Are similar microstructural defects good pinning centers in NWs as well ?*



Opt 1 *Modulated-diameter NWs*



Opt.2 *[FM/NM] multilayers NWs*



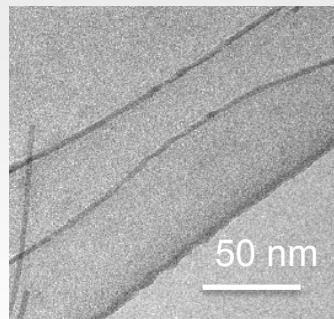
→ EH can provide valuable info at the nm scale

- Cylindrical symmetry of NWs => High propagation velocity of DWs
- DW configuration depends on anisotropy (shape, magnetocrystalline), magnetisation (materials) exchange constant (materials)

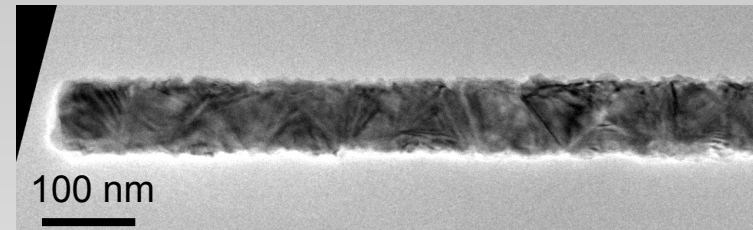
Effects on the magnetic configuration and DW structure in NWs of

- Crystallography
- Diameter & aspect ratio (length/diameter)
- Morphology
- Composition
- Defects
- ...

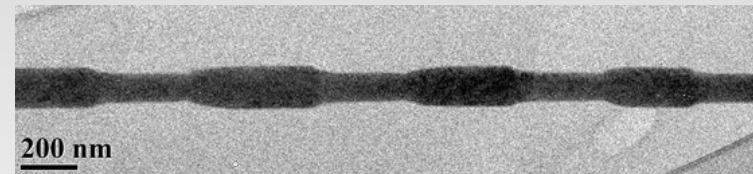
- *Very narrow single crystal Co nanowire (4 nm diameter)*



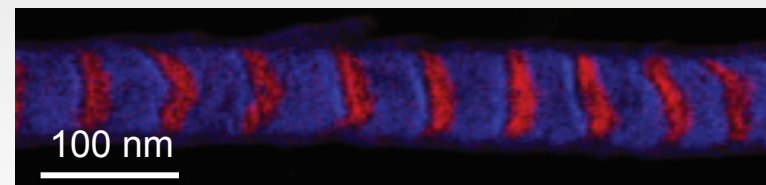
- Polycrystalline Ni nanowires



- Modulated-diameter polycrystalline Co nanowire




- Multilayer Co/Cu nanowire

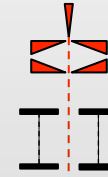




I2TEM: TEM dedicated to holography and “in-situ” TEM

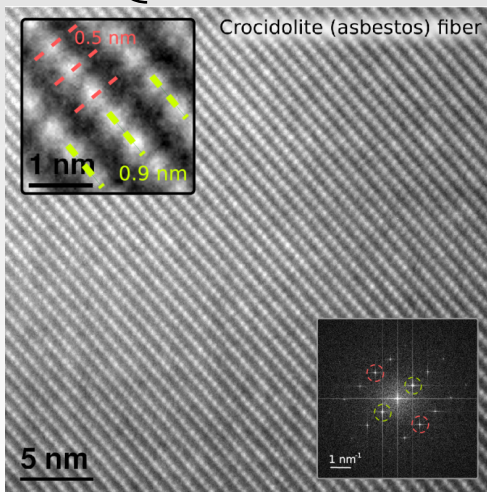
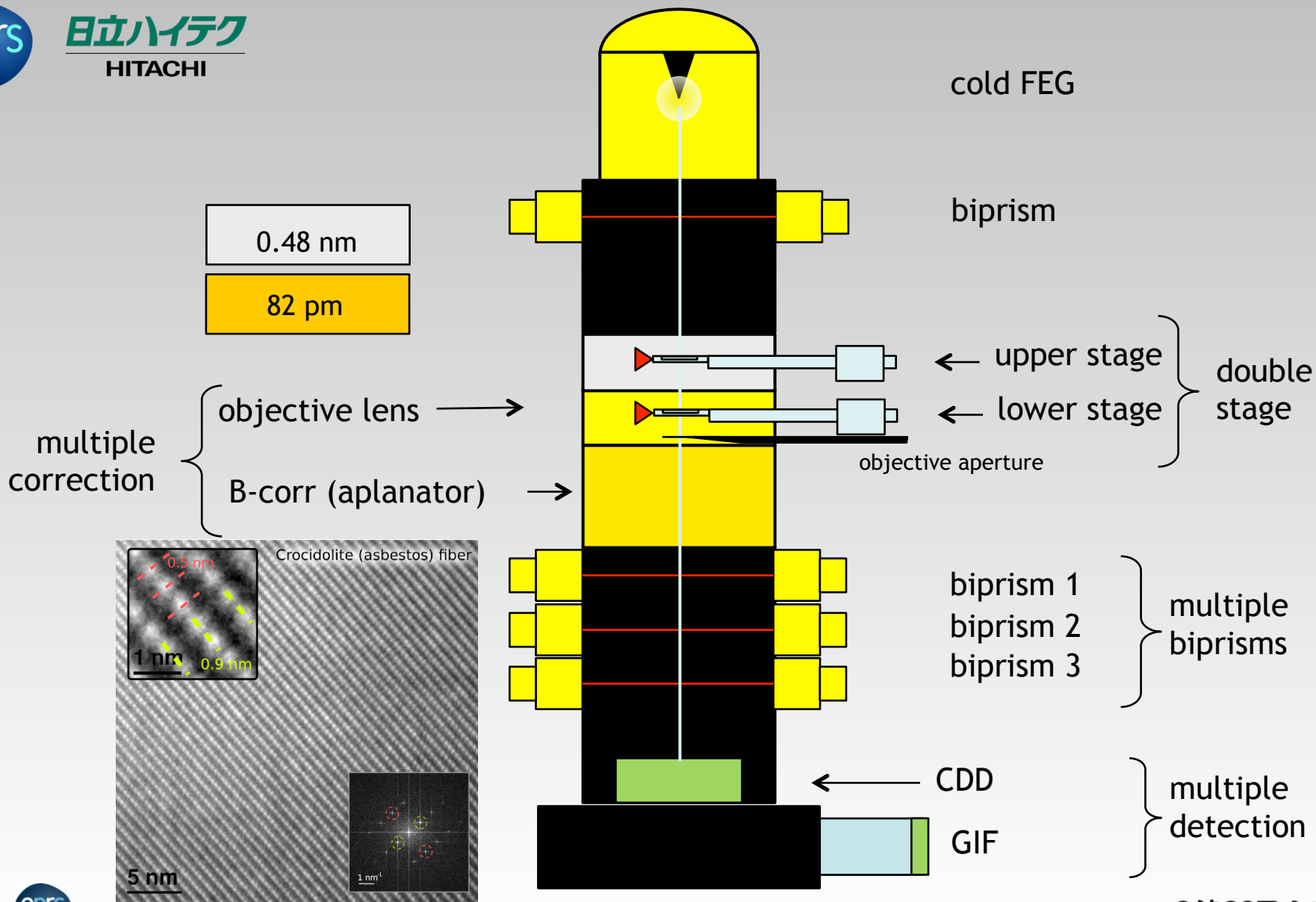
Materials oriented project : Studying “In situ” the dynamical physical phenomena process in material under external constraints (*mechanical stress, magnetic and electric fields*)

- New column design
- B-corr: correct for radial and azimuthal off-axial coma  CEOS
Corrected Electron Optical Systems GmbH
- Two goniometers
- Multibiprisms
- One View Camera (Gatan)



Biorism 1





Very first EH work on magnetic nanowires performed with RDB in Cambridge (2003) - CM30 FEG

APPLIED PHYSICS LETTERS VOLUME 82, NUMBER 1 6 JANUARY 2003

Quantitative magnetization measurements on nanometer ferromagnetic cobalt wires using electron holography

E. Snoeck
CEMES-CNRS, 29 rue J. Marvig, BP4347, 31055 Toulouse, France

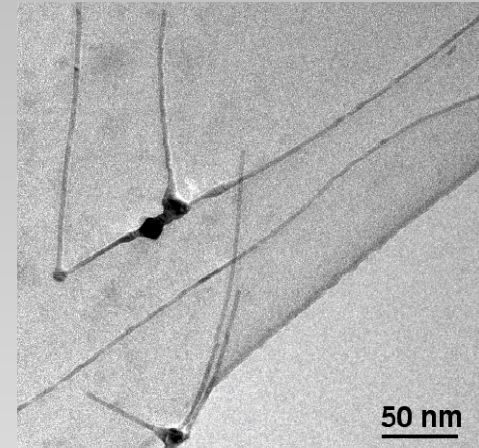
R. E. Dunin-Borkowski⁽¹⁾
Department of Materials Science and Metallurgy, University of Cambridge, Pembroke Street, Cambridge CB2 3QZ, United Kingdom

F. Dumestre⁽²⁾ and P. Renaud
Digital DNA Labs, Semiconductor Products Sector, Motorola, le Mirail B.P. 1029, 31023 Toulouse Cedex, France

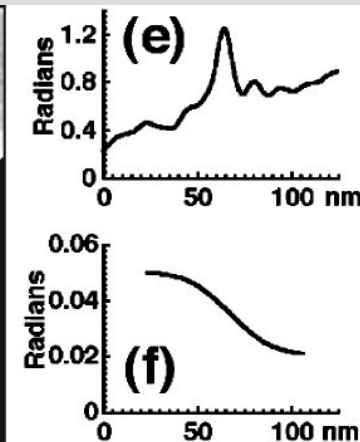
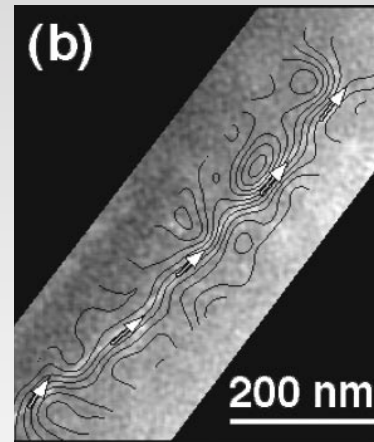
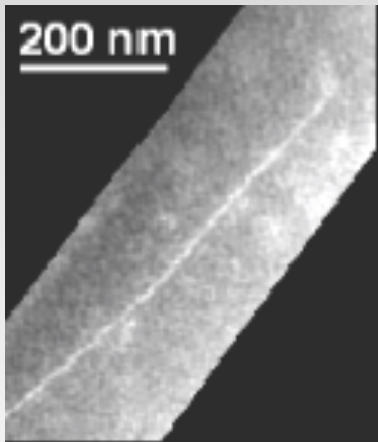
C. Amlens and B. Chaudret
LCC-CNRS, route de Narbonne, 31055 Toulouse, France

P. Zürcher
Digital DNA Laboratories, Semiconductor Products Sector, Motorola, 2100 E. Elliot Road, Tempe, Arizona 85284

(Received 19 September 2002; accepted 1 November 2002)



Single crystal 4 nm diameter Co NWs



$$\Delta\phi_{\text{MIP}} = 0.65 \text{ rad } (\pm 0.005)$$

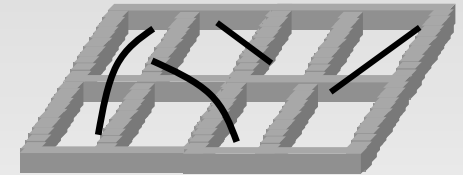
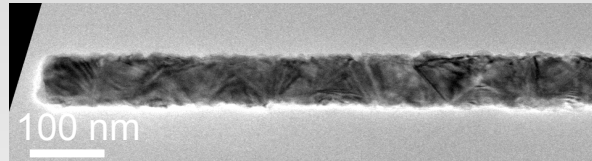
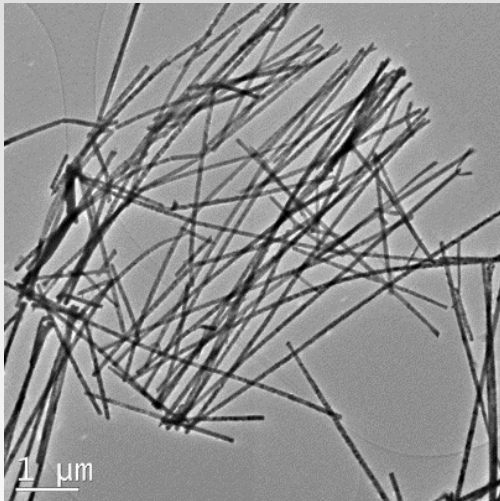
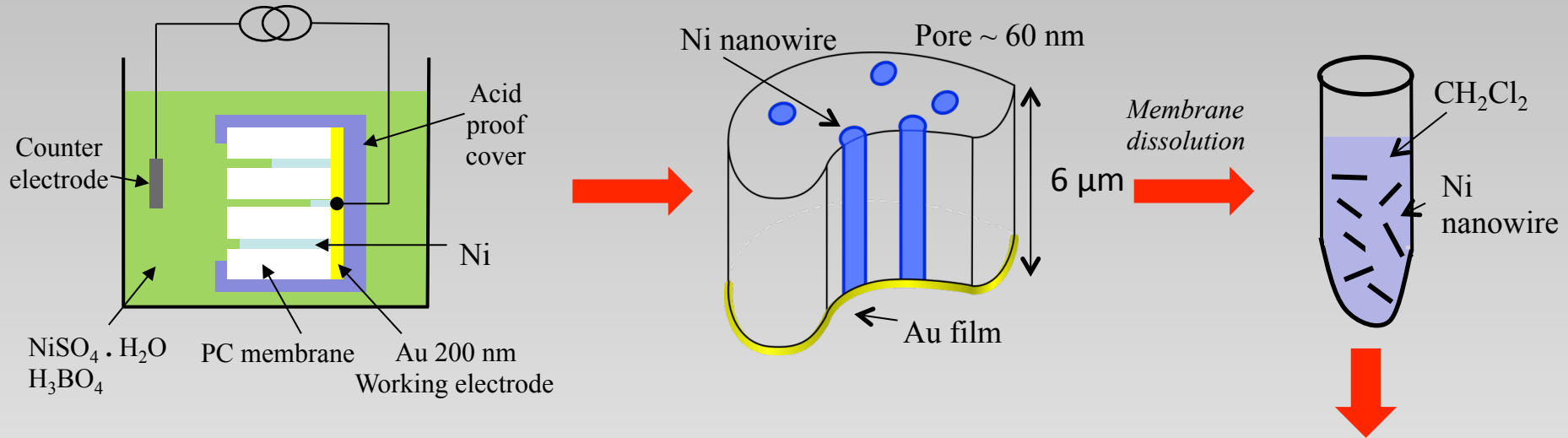
$$\Delta\phi_{\text{Mag}} = 0.03 \text{ rad } (\pm 0.005)$$

$$\Rightarrow B = 1.7 \text{ Tesla}$$

E. Snoeck, R. E. Dunin-Borkowski, et al. Appl. Phys. Lett. 82, p 88 (2003).

E. Snoeck – CEMES-CNRS Toulouse (France)

Electro-deposition in commercial Poly-Carbonate (PC) membranes



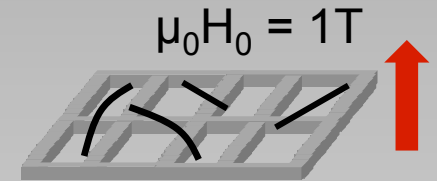
TEM Carbon grid

→ Ni NWs of mean diameter : 50, 80 or 110 nm

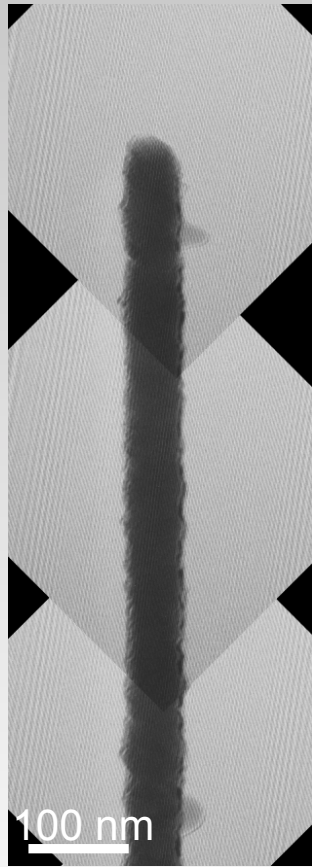
Easy and low cost method

Polycrystalline Ni NWs

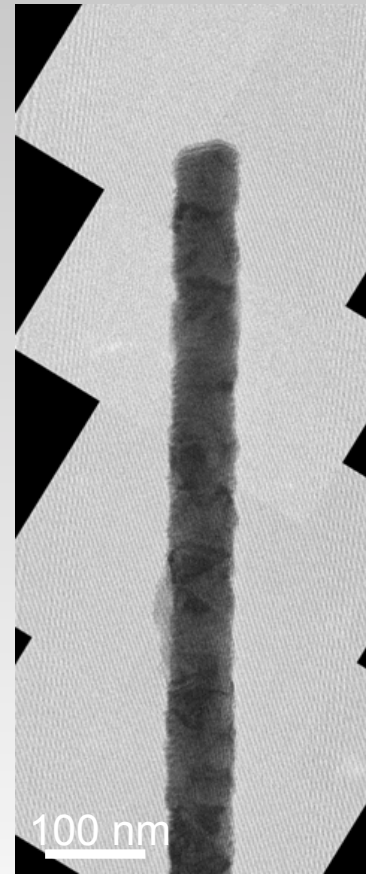
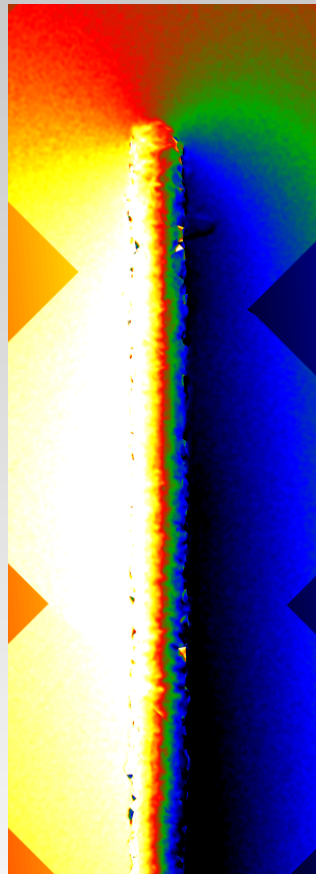
No DWs in as deposited NWs (DW very unstable)



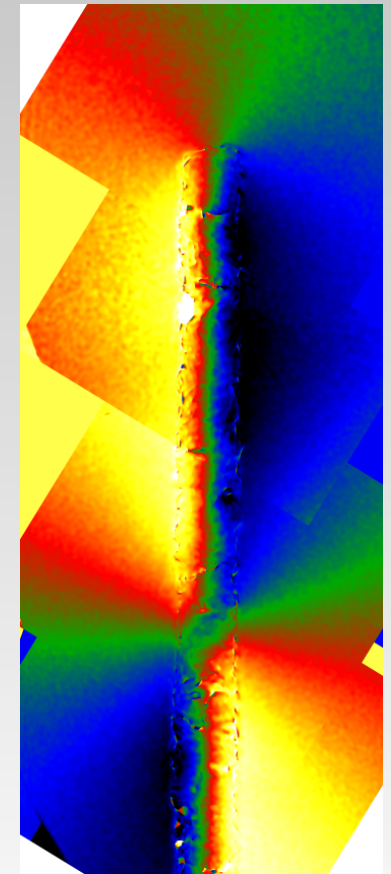
→ Apply B field (objective) to switch the magnetization and then create DWs



no DW

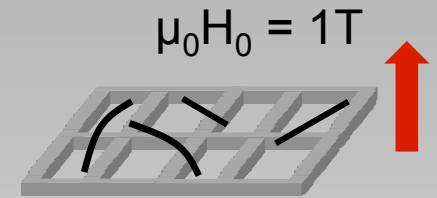


DW



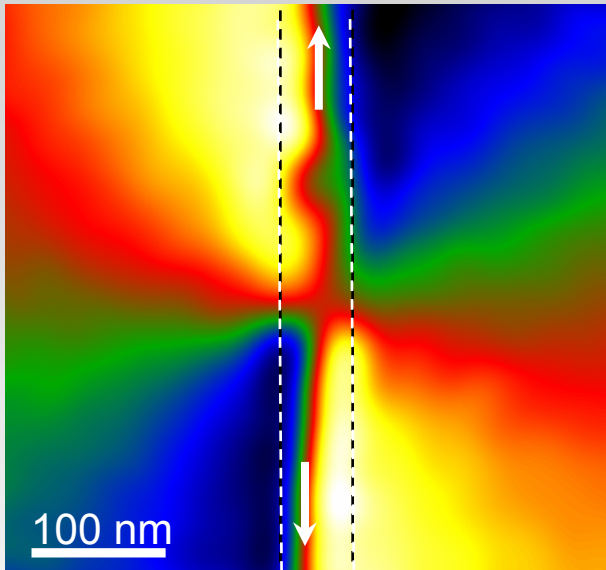
Collaborations: - School of Physics, Trinity College, Dublin Ireland
- LSI/CEA/CNRS/Ecole Polytechnique, Paris, France

No DWs in as deposited NWs (DW very unstable)

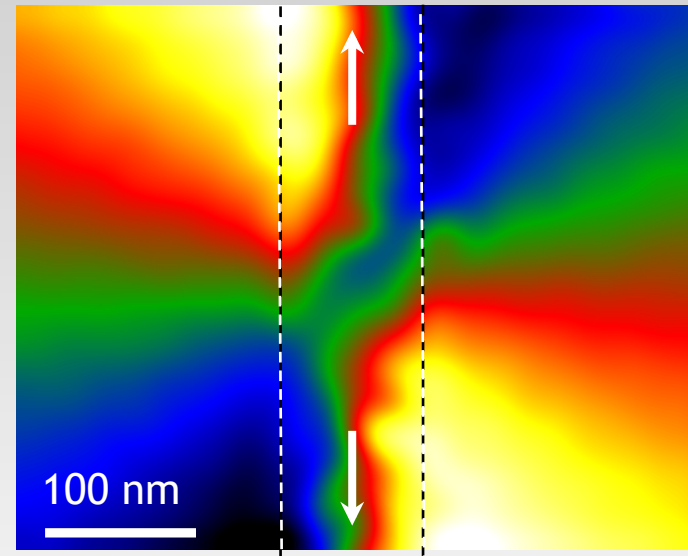


→ Apply B field (objective) to switch the magnetization and then create DWs

→ Two types of DWs



Diameter = 55 nm



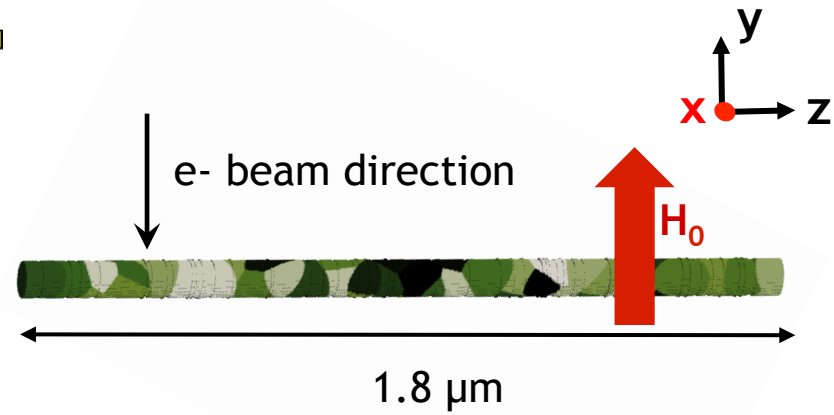
Diameter = 85 nm

Polycrystalline Ni NWs

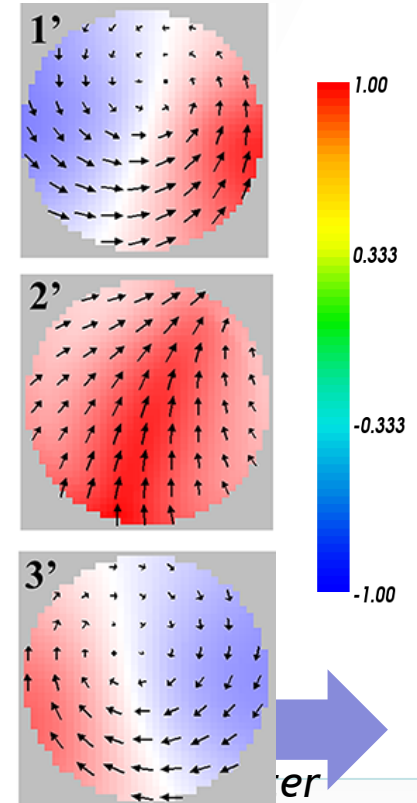
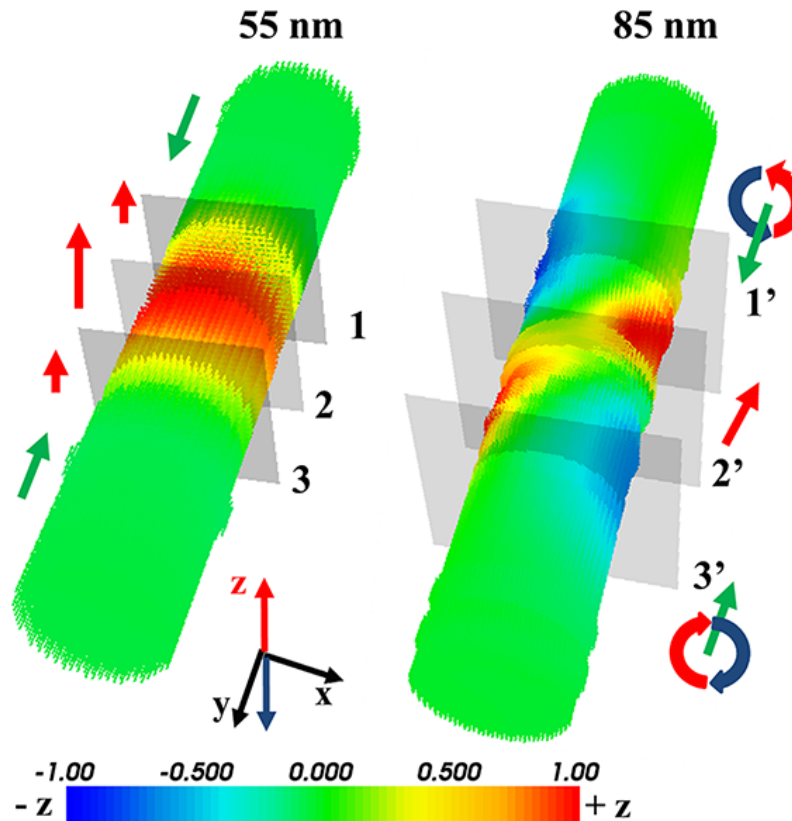
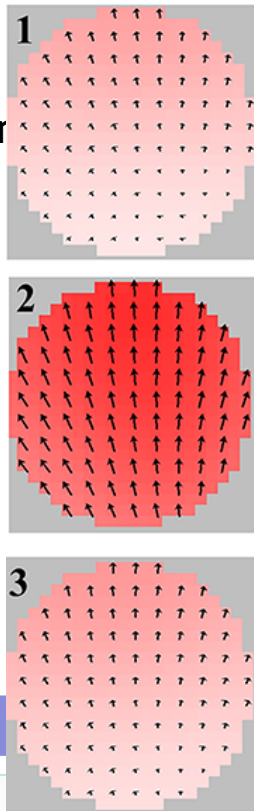


3D OOMMF software:

- Cell size $2.5 \times 2.5 \times 2.5 \text{ nm}^3$
- Total size $500 \times 500 \times 2000 \text{ nm}^3$
- Generation of grains with random orientation
- $\mu_0 M_s = 0.485 \text{ T}$,
- Magnetocrystalline anisotropy $K_1 = -5.7 \text{ e}^{-3} \text{ J/m}^3$,
- Exchange constant $A = 9 \cdot \text{e}^{-12} \text{ J/m}$



M_y componer

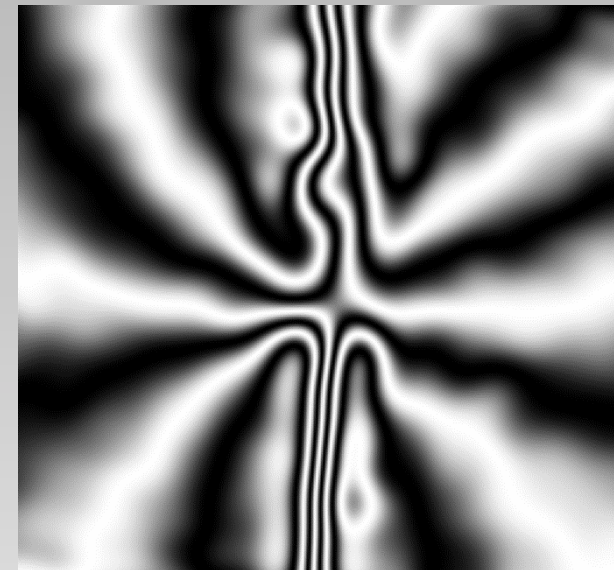
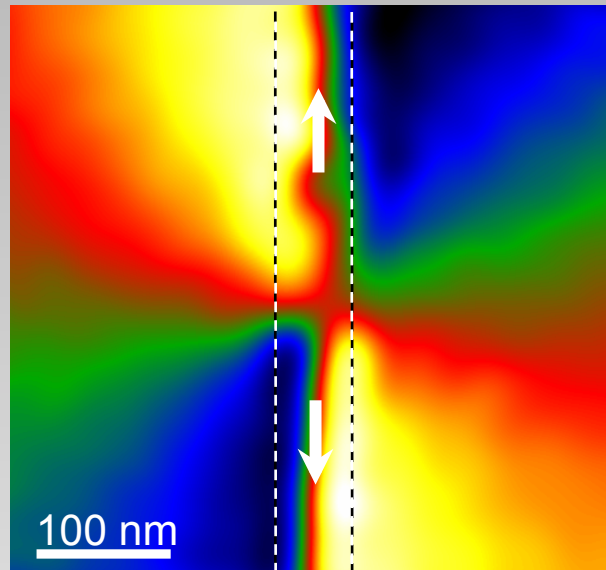


Polycrystalline Ni NWs

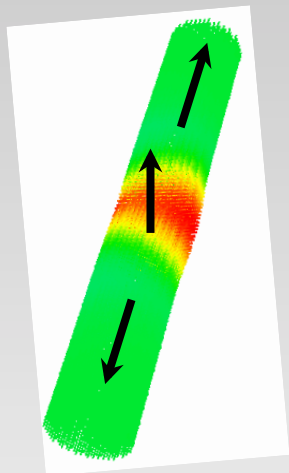
Phase

Cos(phase)

Experimental

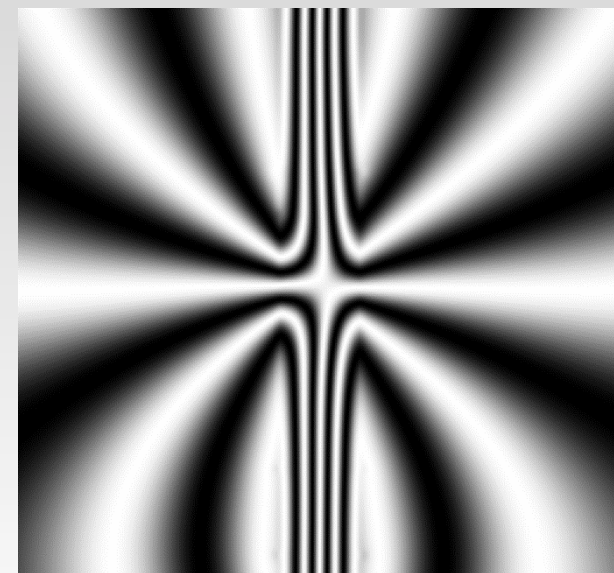
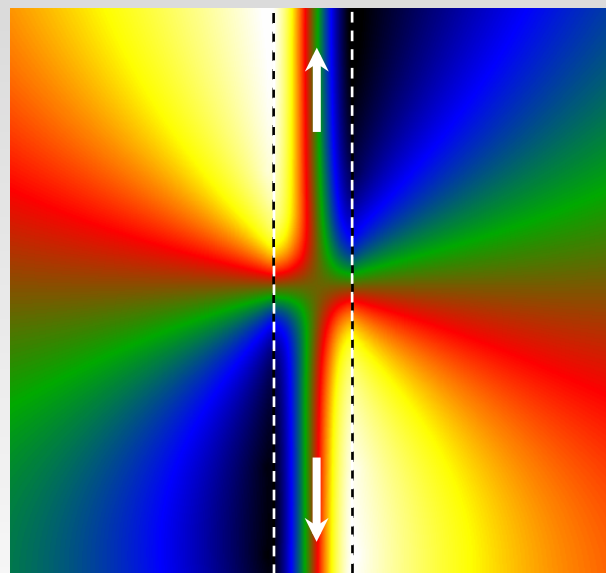


Diameter = 55 nm



Transverse wall

Simulated



Very good agreement between simulation and experiments

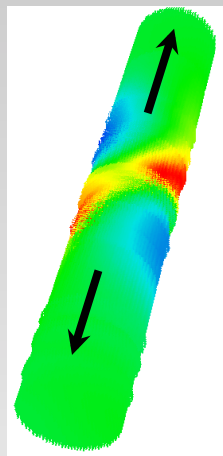
Polycrystalline Ni NWs

Phase

Vecs (phase map)

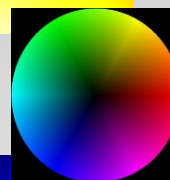
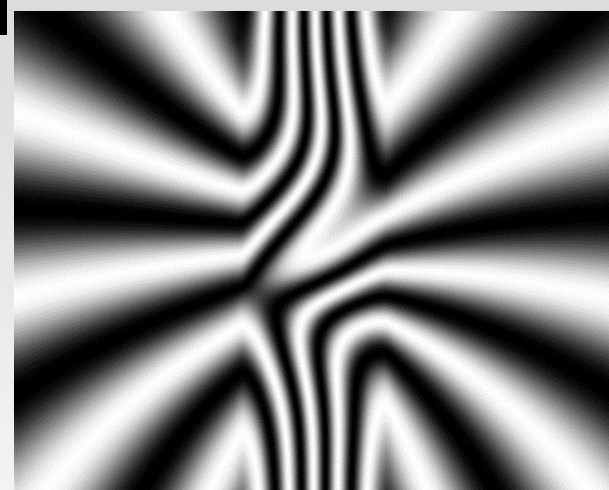
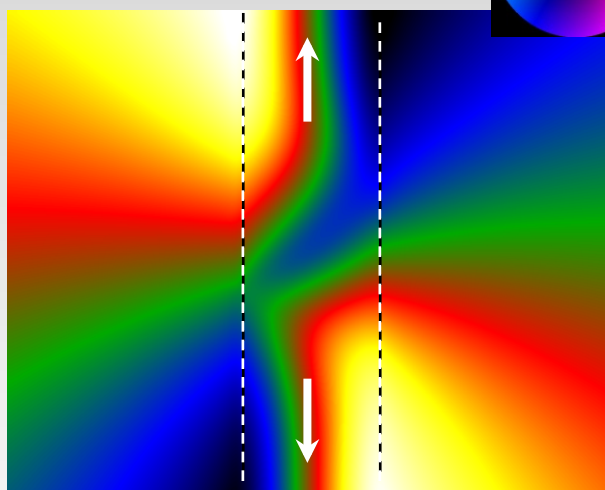
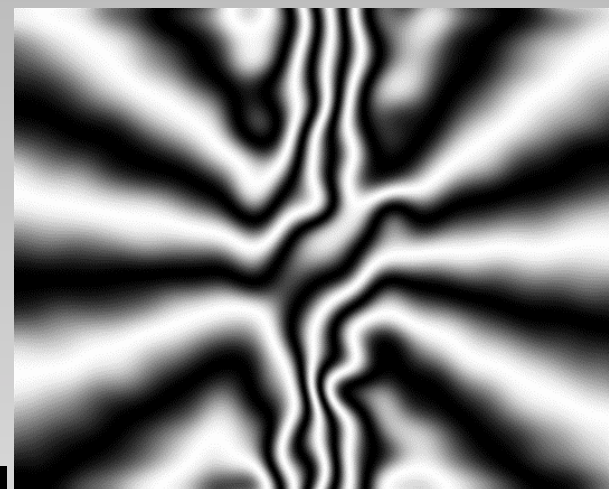
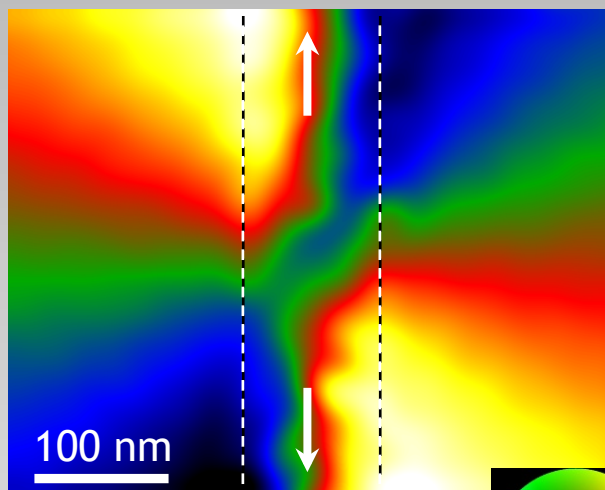
Experimental phase

Diameter = 85 nm



Hybrid wall

Simulated phase



Excellent agreement between simulation and experiments

N. Biziere, C. Gatel, R. Lassalle-balier, M.C. Clochard, J.E. Wegrowe, and E. Snoeck
Nano Letters, 13 (5), pp 2053-2057 (2013)

MOTIVATION : Domain Wall (DW) manipulation information storage technologies, logic systems and sensors.

Need: 1/ Magnetic nanocylinders

2/ Efficient methods to control DWs in the NWs

→ DWs' fine structure is required → EH can provide valuable info at the nm scale

→ Finding efficient DWs pinning centres ?

In planar NWs, DWs can be controlled by changing their lateral dimensions (*notches, anti-notches, periodic modulations, ...*)

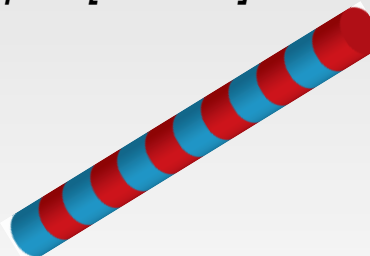
→ Are similar topological defects good pinning centres in NWs as well ?



Opt. 1 Modulated-diameter NWs

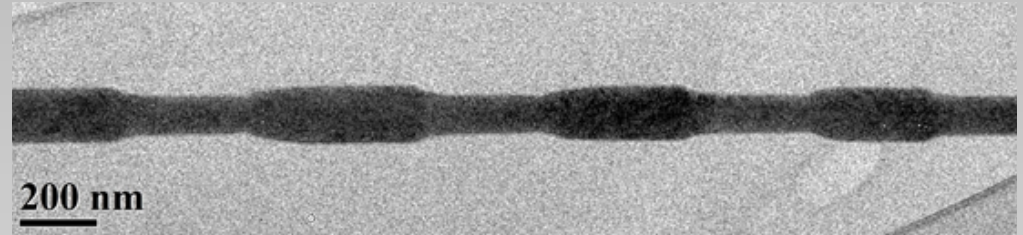
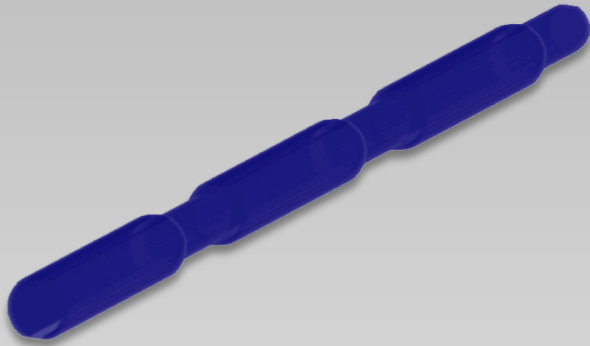


Opt.2 [FM/NM] multilayers NWs

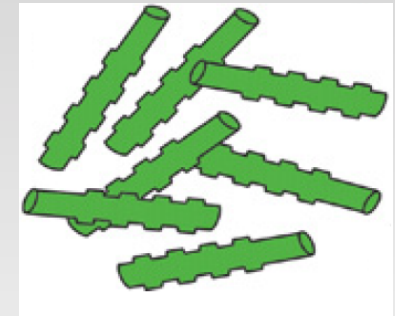
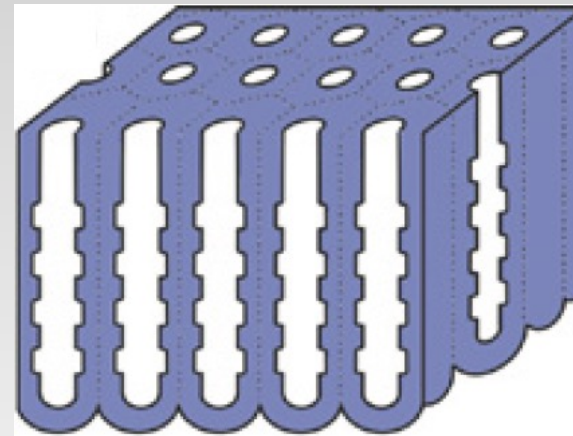
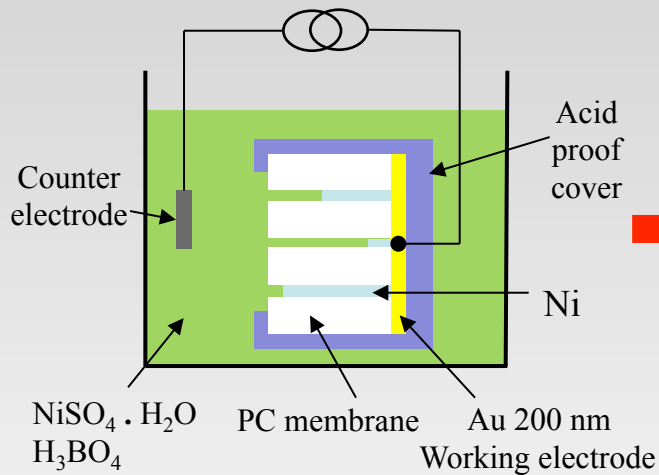


→ EH can provide valuable info at the nm scale

→ MOTIVATION : Finding efficient DWs pinning centres ?



Manuel Vasquez and Agustina Asenjo
Intituto de Ciencia de Materiales de Madrid (ICMM-CSIC)



Fabrication of the anodic aluminum oxide (AAO) template in two-steps: **Mild and hard anodization process.**

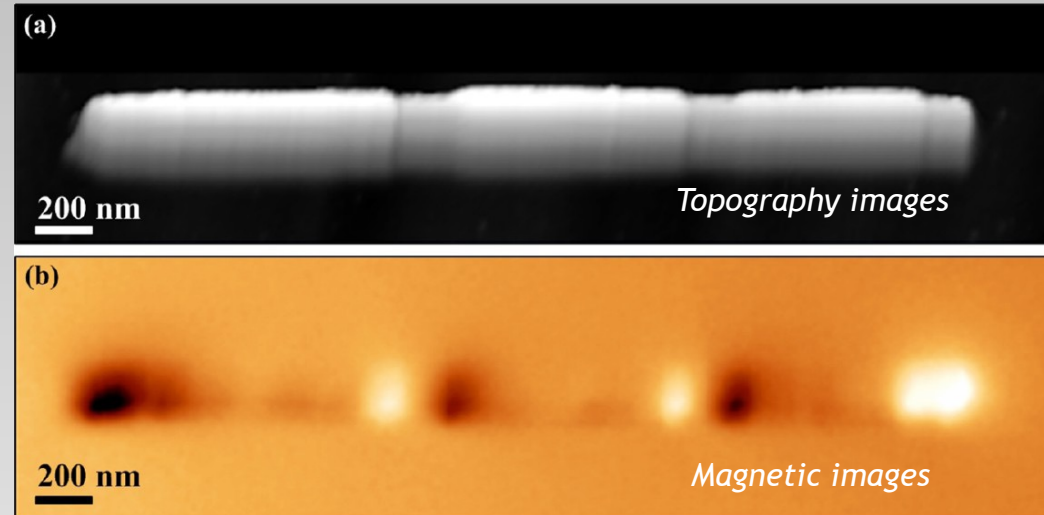
G. D. Sulka et al. *Electrochimical Acta* **56** (2011) 4972 - 4979

Diameter modulated FeCoCu NWs

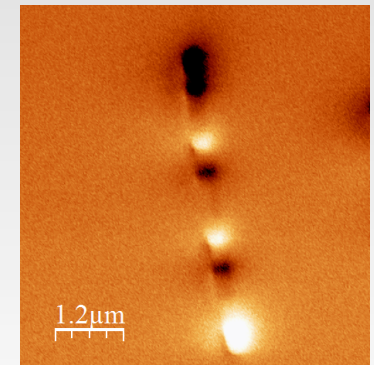
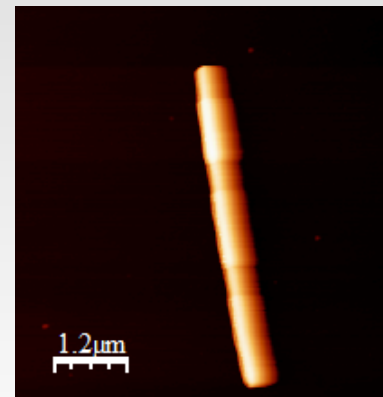
First MFM results

M Vasquez and A. Asenjo

→ Diameter-modulated (D-M) FeCoCu NWs present a series of periodic magnetic contrasts located at the diameter change position.



Are they domain walls?



Morphology and crystal structure

- The longest NWs are about 10 μm length
- Alternating segments of large (LD) and small diameters (SD).

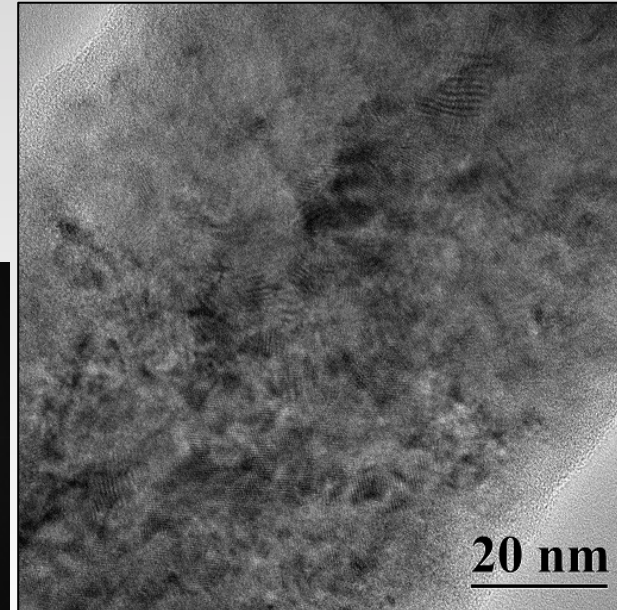
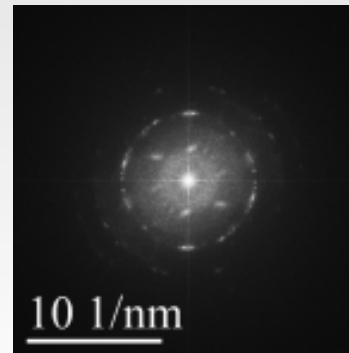
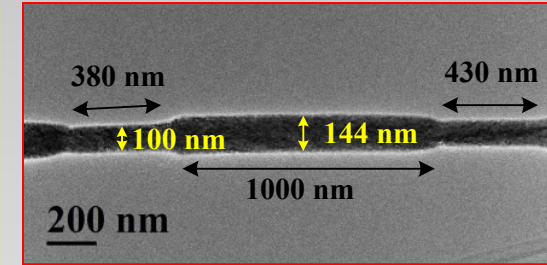
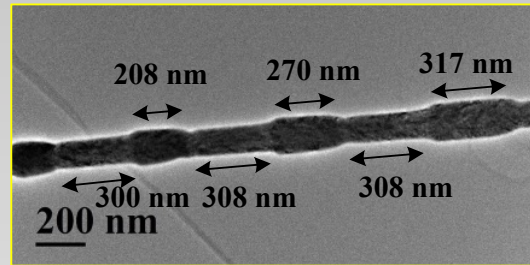
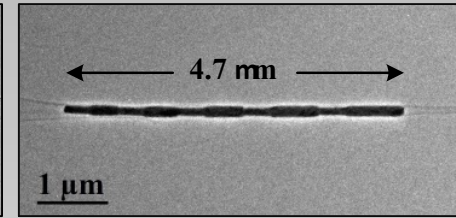
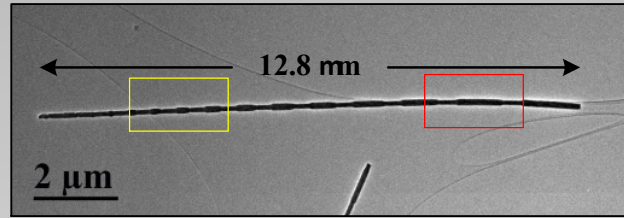
Smallest diameter = 100 nm

Largest diameter = 144 nm

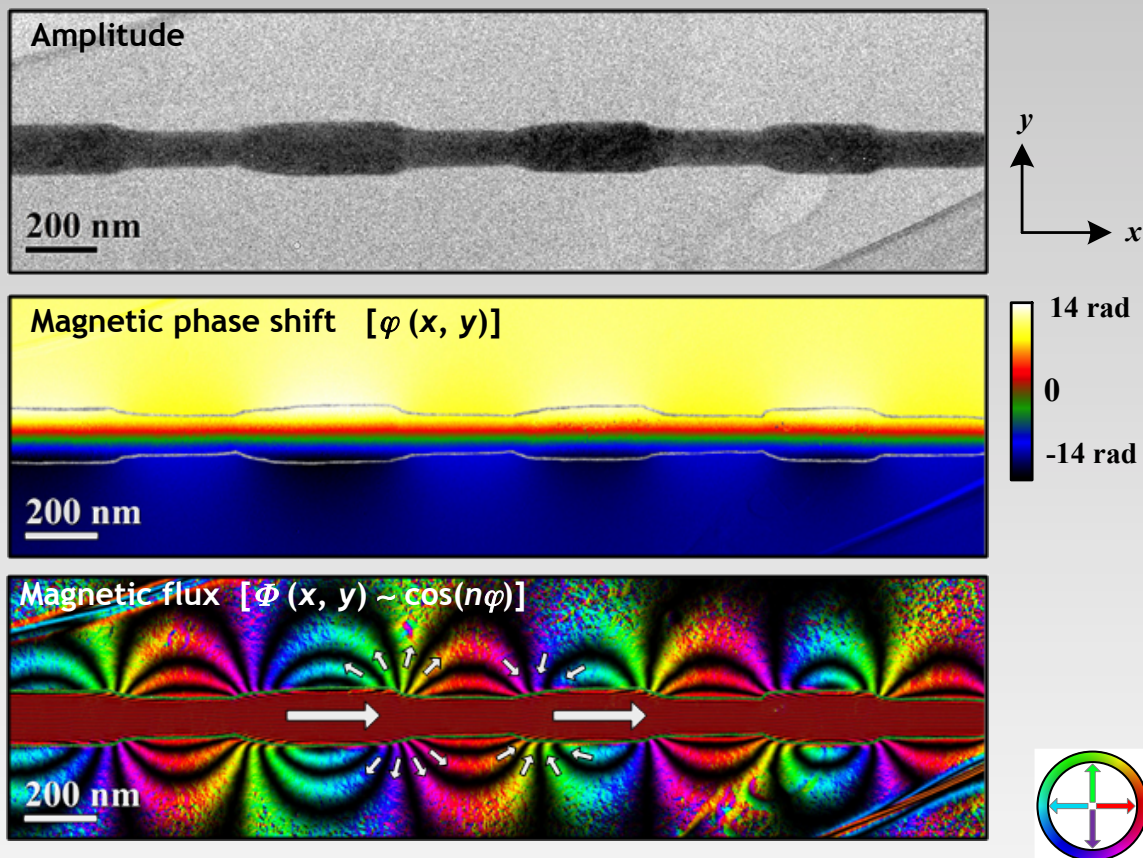
SD segment = 430 nm - 280 nm

LD segment = 1000 nm - 200 nm

→ FeCoCu NWs have a polycrystalline structure



Remanent magnetic configuration: EH analysis



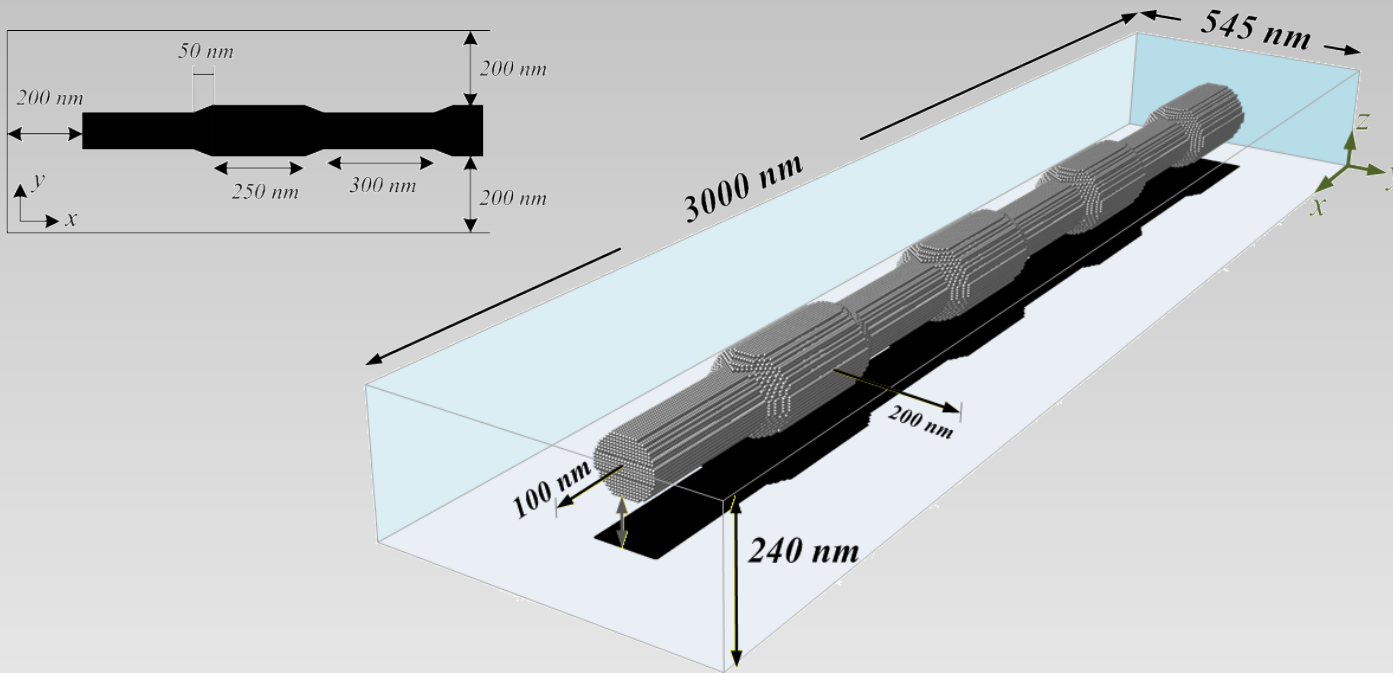
EH at remanence :

- → **single-domain state.**
- → **complex local stray field**
- The Diameter Modulation crossovers act like **magnetic poles**

For a 3D analysis:

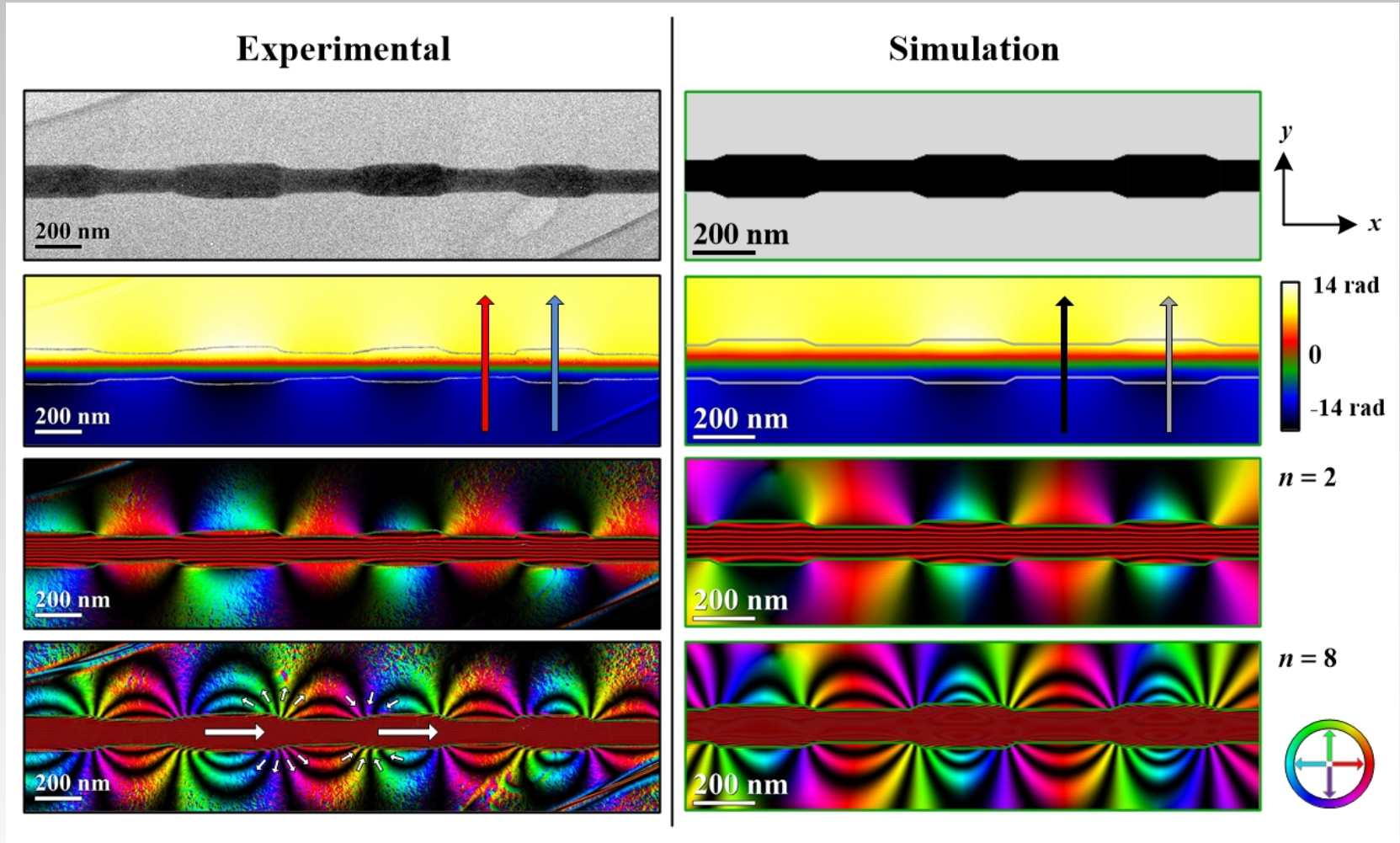
→ **Micromagnetic simulations**

Remanent magnetic configuration



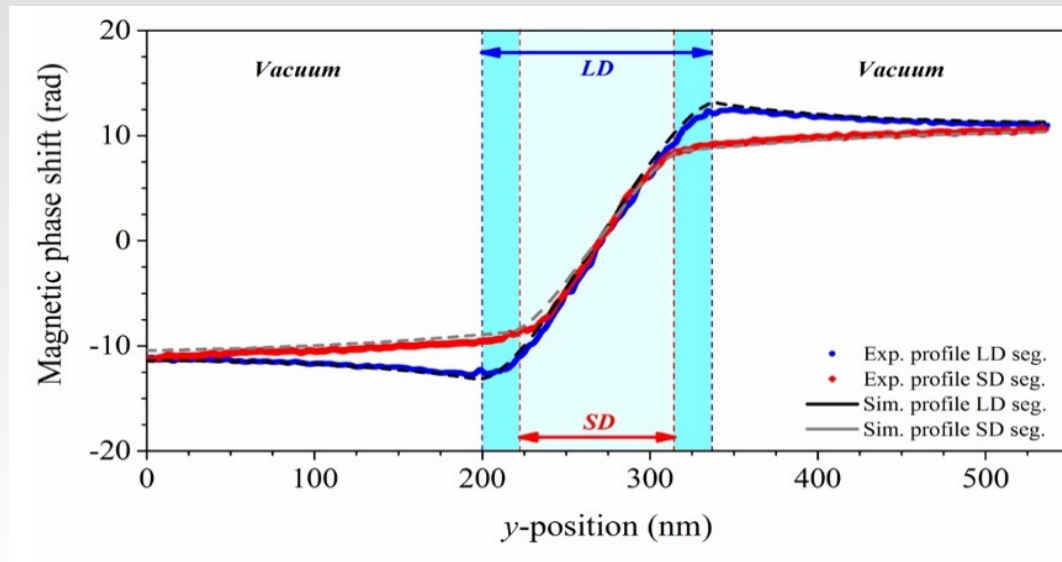
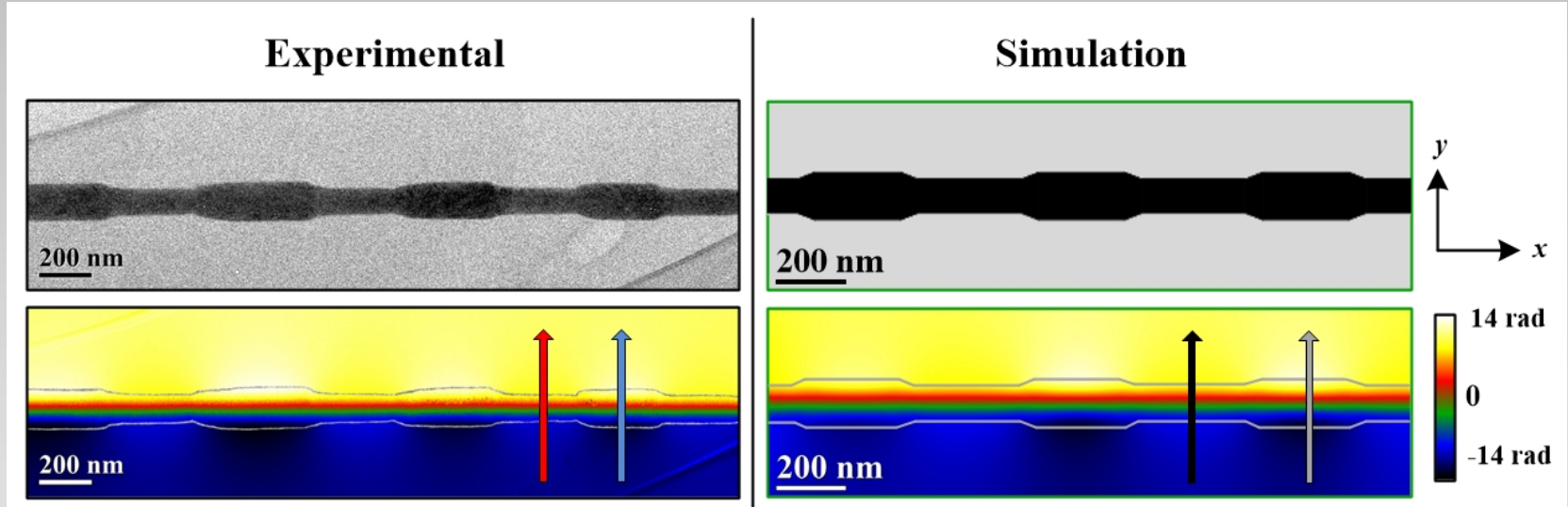
Unit Cell = $5 \times 5 \times 5 \text{ nm}^3$
 $M_S = 1060 \text{ kA/m} \rightarrow 1.33 \text{ T}$
 $A = 26 \times 10^{-12} \text{ J/m}$
 $K = 0$ (polycrystalline structure)

Remanent magnetic configuration: EH and Simulations

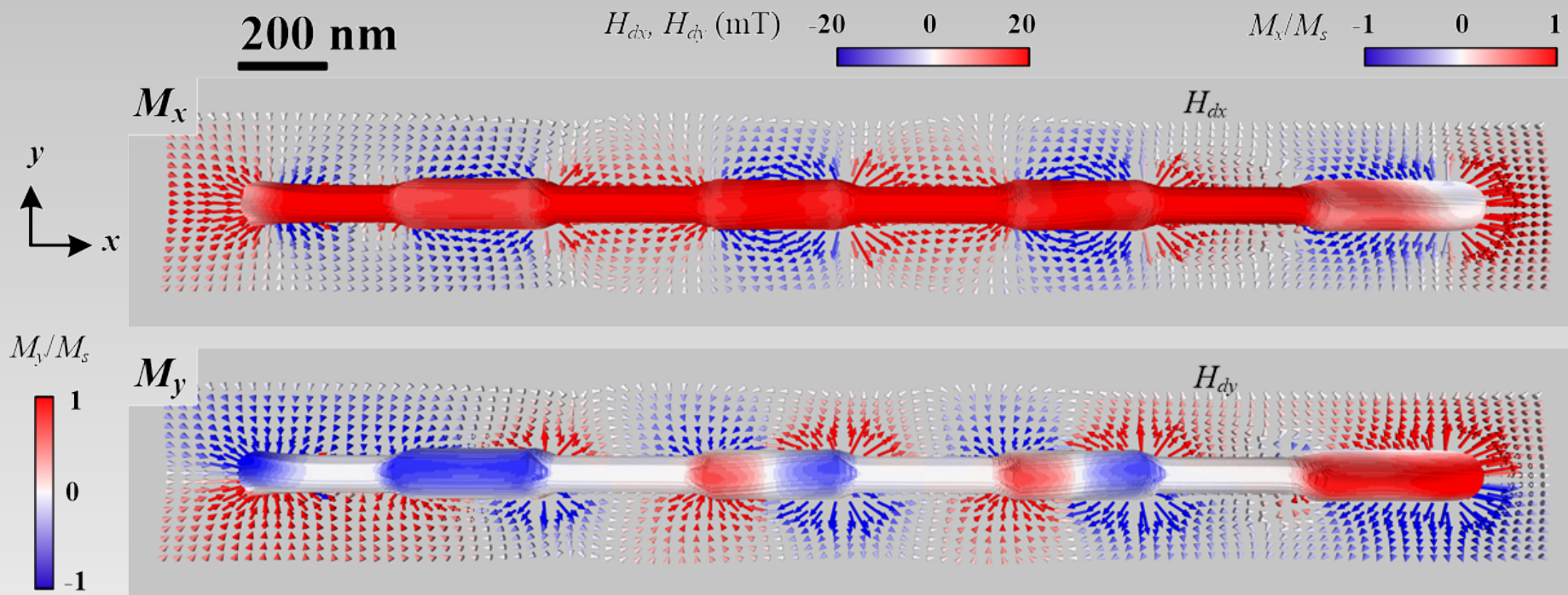


→ excellent agreement between EH experiments and simulations

Remanent magnetic configuration: EH and Simulations



Remanent magnetic configuration: Simulations



x-component of the magnetization (M_x)



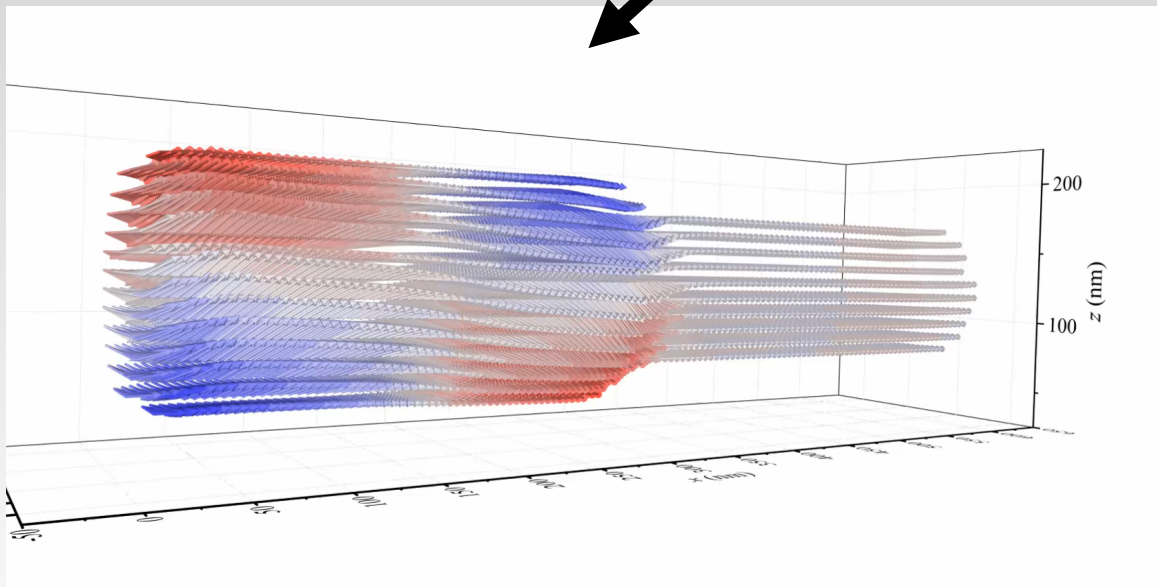
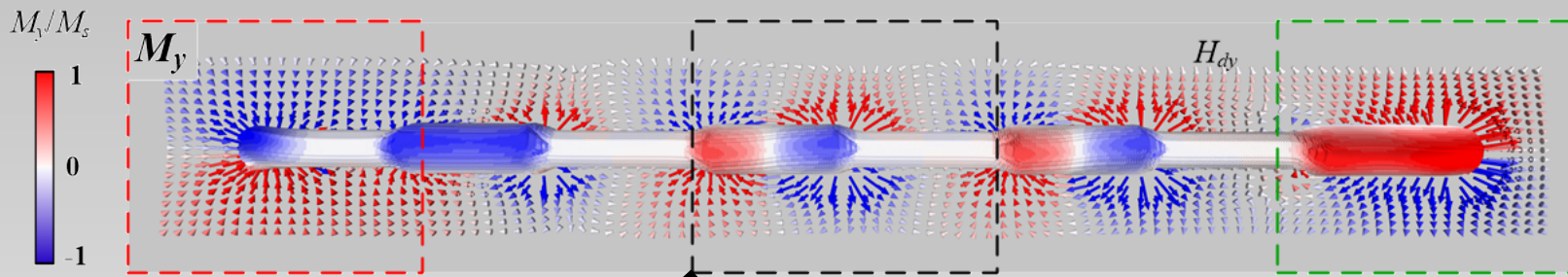
Indicates a **single-domain state**

y-component of the magnetization (M_y)



shows a more complex **vortex-like configuration**

Remanent magnetic configuration: Simulations

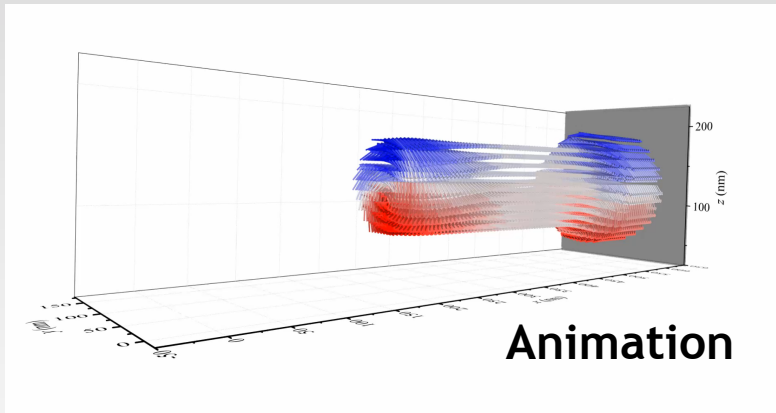
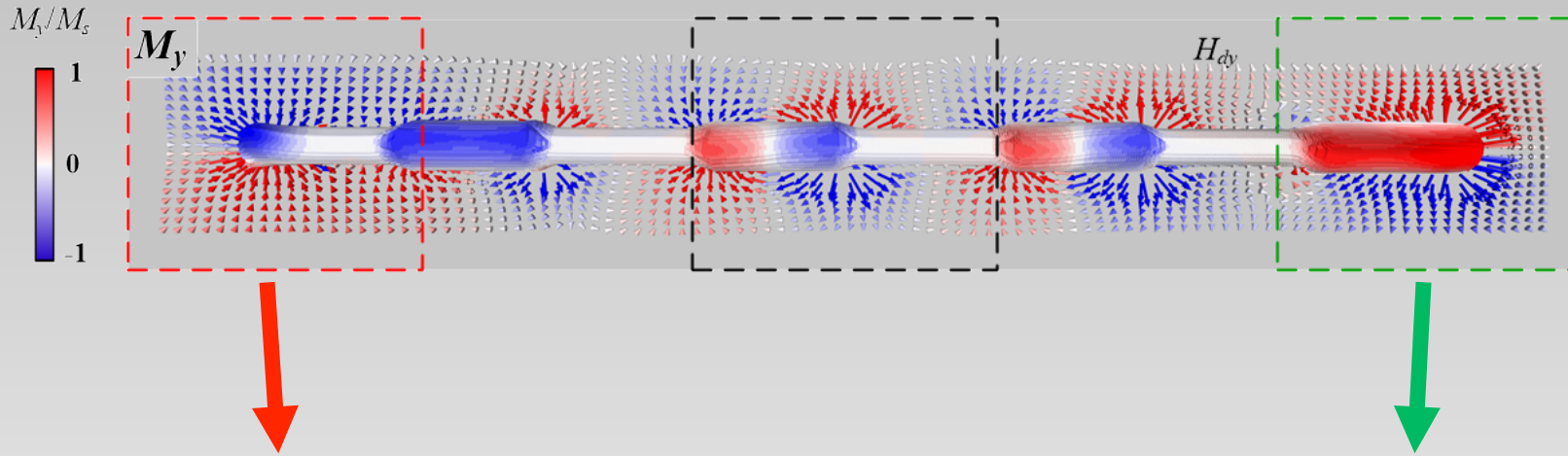


Animation

The diameter-modulated geometry of the NW induces:

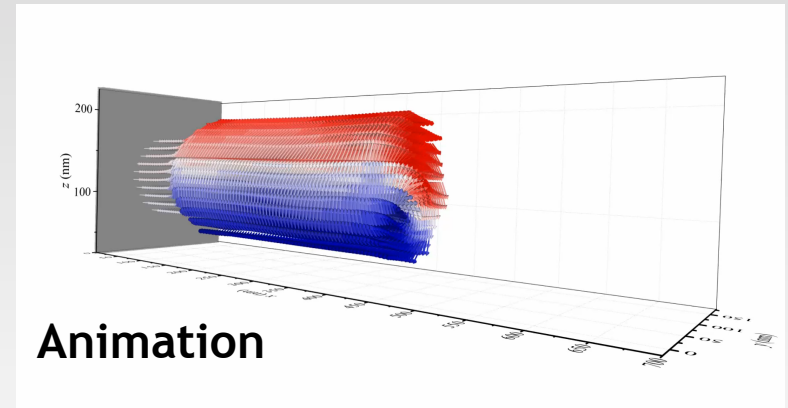
- **Vortex-like states** in the DM transitions and at the ends of the NWs
- **longitudinal alignment** of the spins in the thin segments.
- **Torsional deformed longitudinal alignment** of the spins in the thick segments.

Remanent magnetic configuration: Simulations



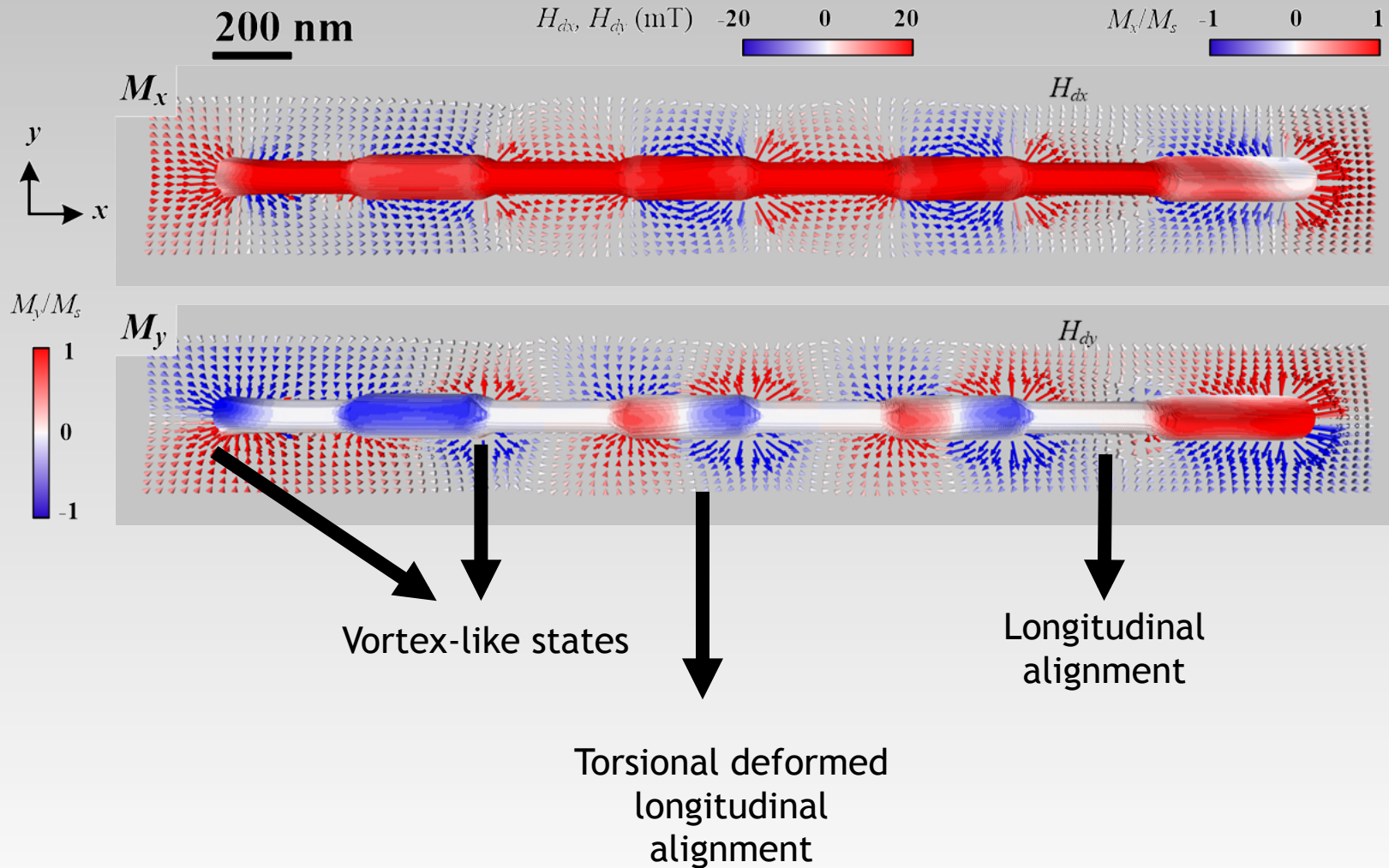
Thin ends:

- Vortex-like state at the tip
- Axial state close to the DM crossover



Thick ends: - Vortex-like state at the tip

Remanent magnetic configuration: Full description

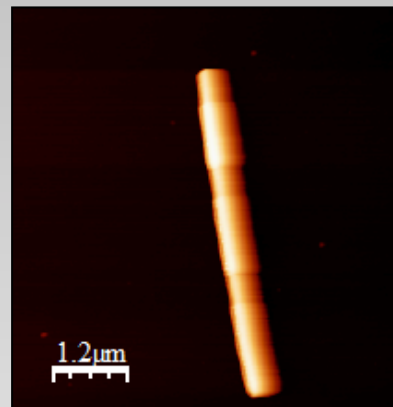


Remanent magnetic configuration

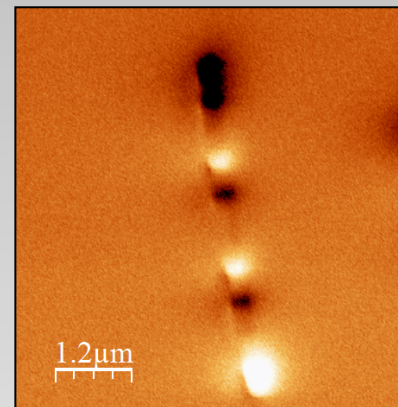
Interpretation of MFM images ?

Bright and dark magnetic contrasts along the wire is due to the magnetic stray fields resulting on the diameter-variation crossover.

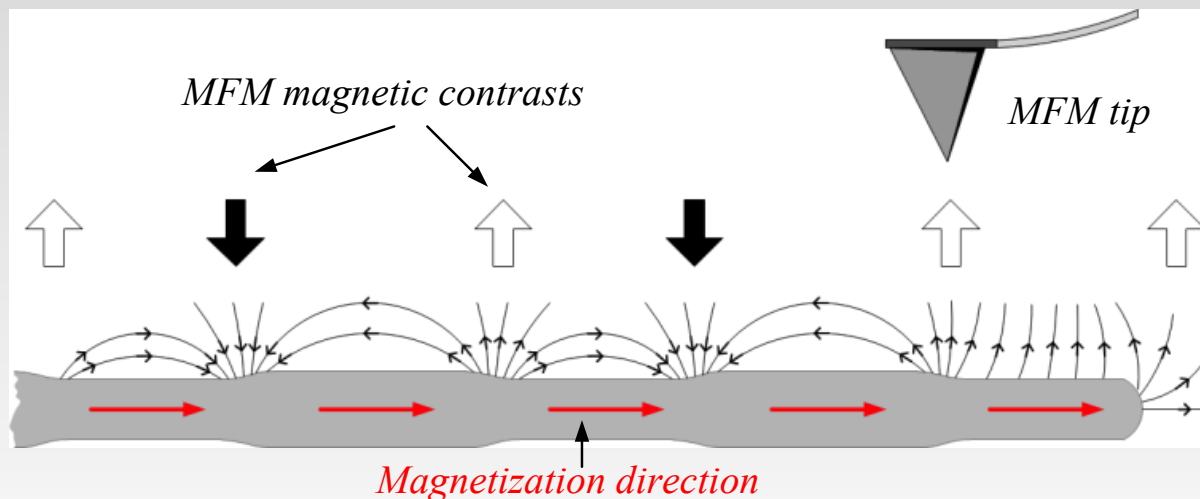
Topography images



Magnetic images



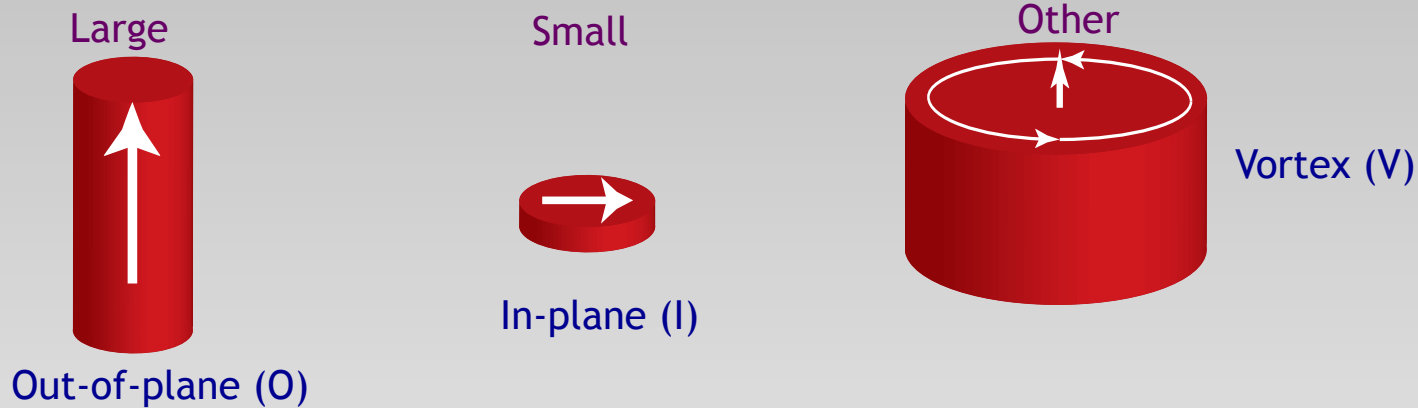
There are not DWs



L. A. Rodriguez, C. Bran, D. Reyes, E. Berganza, M. Vázquez, C. Gatel, E. Snoeck, A. Asenjo, *ACS Nano*, 2016, 10 (10), pp 9669-9678

Magnetic configurations in multilayers-nanocylinders ?

Magnetic state in single NW depends on its **aspect ratio** (thickness/diameter)



Magnetic state in FM/NM multilayers NWs depends on :

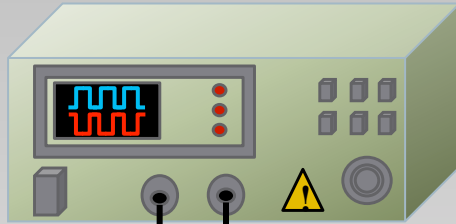
- Dipolar coupling between Co layers across Cu ones
- Shape anisotropy

*and also : crystal anisotropy
exchange constant
magnetization ...*

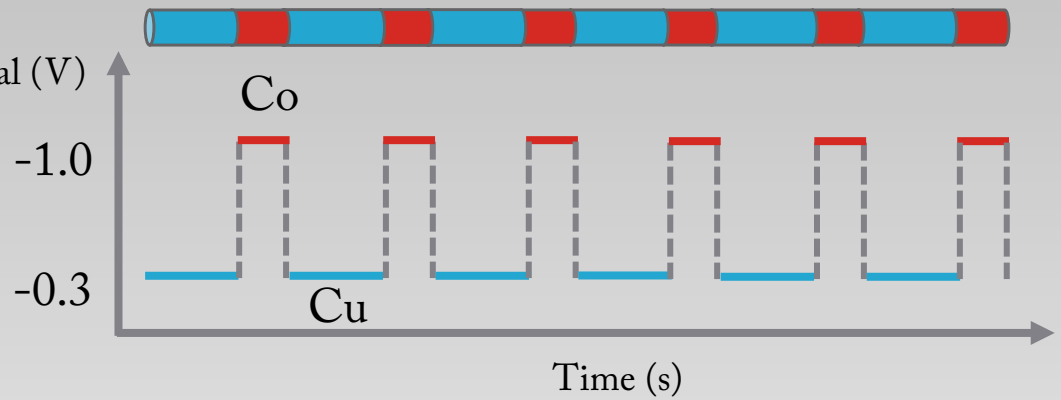


Polycrystalline [Co/Cu] NWs

Potentiostat



Potential (V)



Ag/AgCl
Electrode

Aluminium
piece

Counter
electrode

Membrane
Aqueous
solution

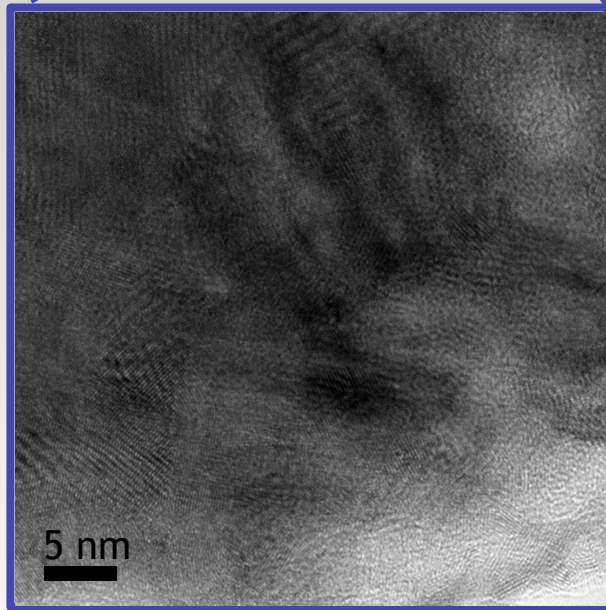
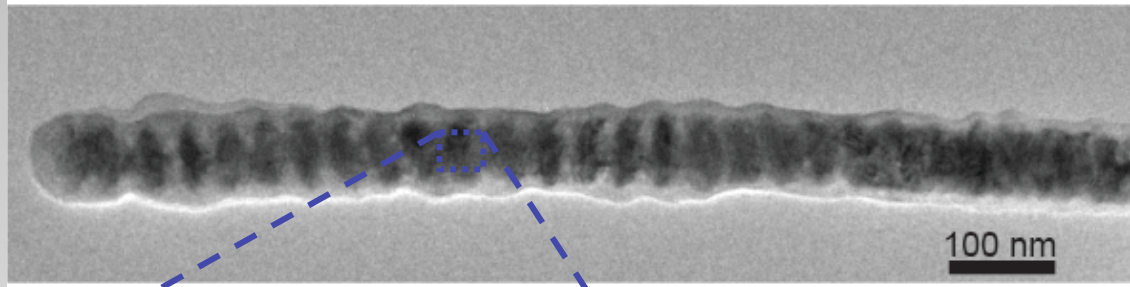
Pulsed
Electrodeposition

Chemical species	Molar concentration (mol/L)
$\text{CoSO}_4 \cdot \text{H}_2\text{O}$	0.43
CuSO_4	0.01
H_3BO_4	0.58

Collaborations: - School of Physics, Trinity College, Dublin Ireland
- LSI/CEA/CNRS/Ecole Polytechnique, Paris, France

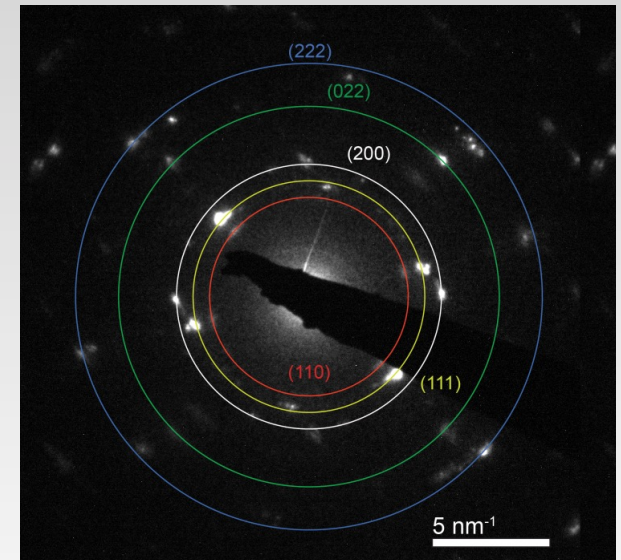
Polycrystalline [Co/Cu] NWs

- Cu/Co NwW of 50-90 nm diameter
- Polycrystalline



Grain size 7 ± 2 nm

FCC Cu and Co

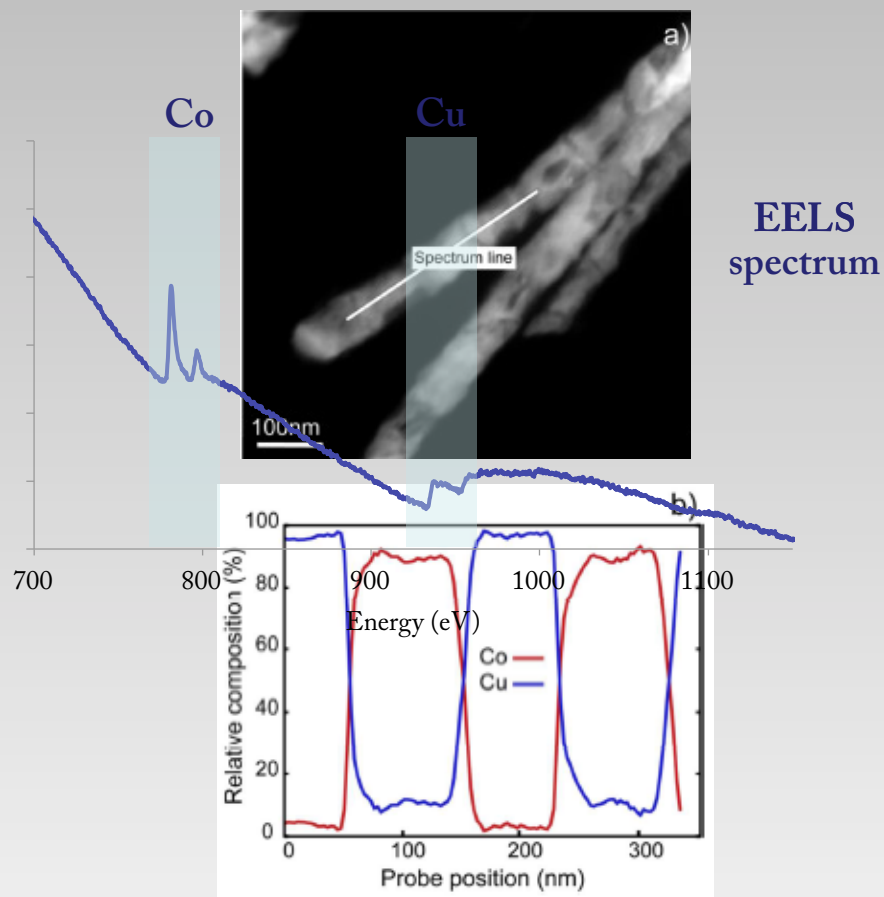
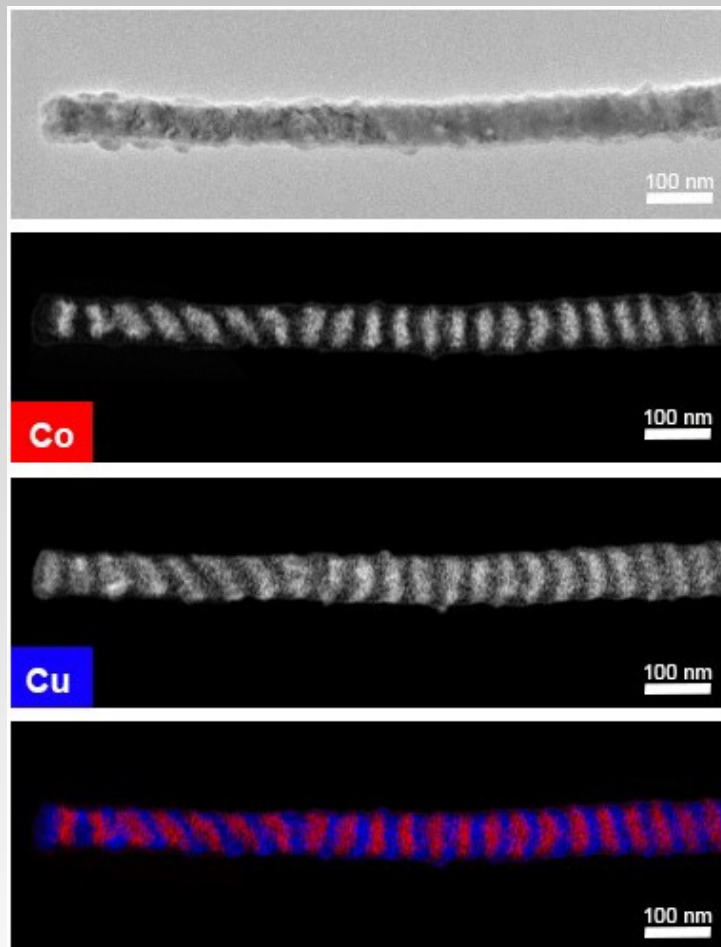


Diffraction pattern

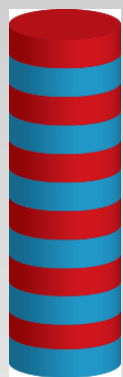
Polycrystalline [Co/Cu] NWs

- Cu/Co NWs of 50-90 nm diameter
- Polycrystalline

Energy filtered TEM (EFTEM)



15% of Cu in the Co layers



Co/Cu
NW

	Sample M1		
	Time (s)	Co	Cu
	Thickness (nm)	25	15
	Sample M2		
	Time (s)	Co	Cu
	Thickness (nm)	25	45
	Sample M3		
	Time (s)	Co	Cu
	Thickness (nm)	50	50
	Sample M4		
	Time (s)	Co	Cu
	Thickness (nm)	100	100
	Sample M5		
	Time (s)	Co	Cu
	Thickness (nm)	7500	0.00



Effect of:
Dipolar coupling

Effect of:
Shape anisotropy



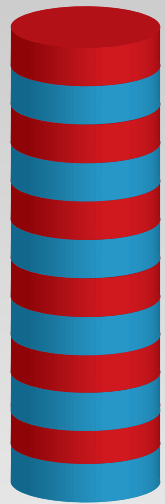
Reference sample:
Saturation
magnetization (M_S)

Sample : Travis Wade, LSI, Paris

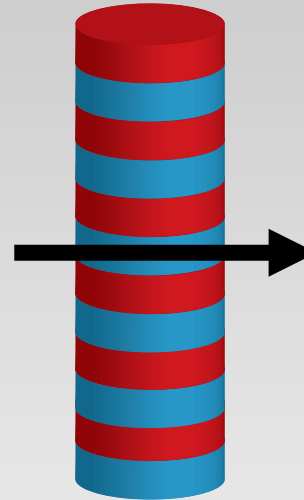
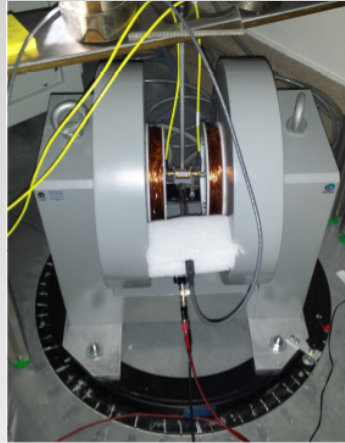
Experimental procedure (magnetic history)

1. Parallel (PL)

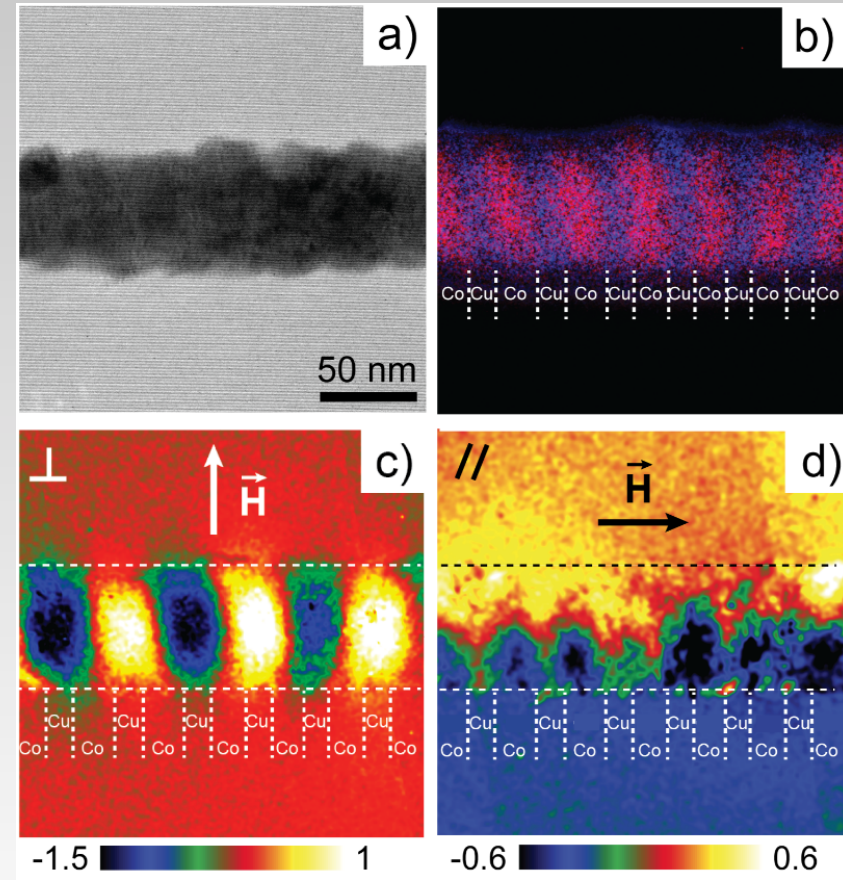
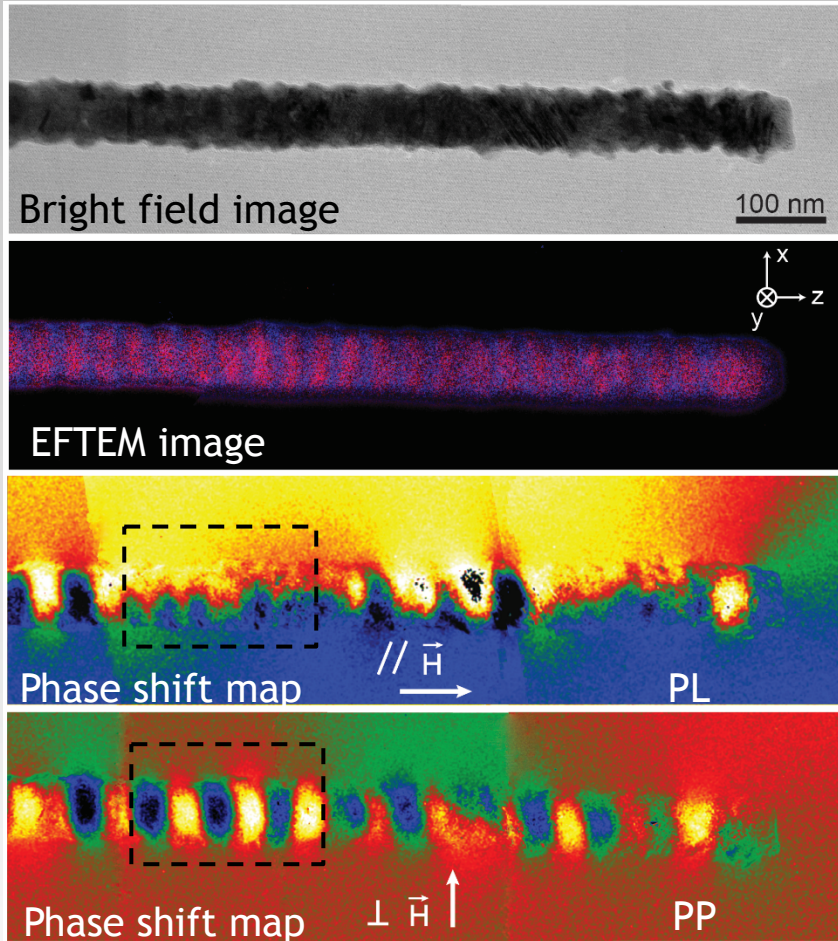
2. Perpendicular (PP)



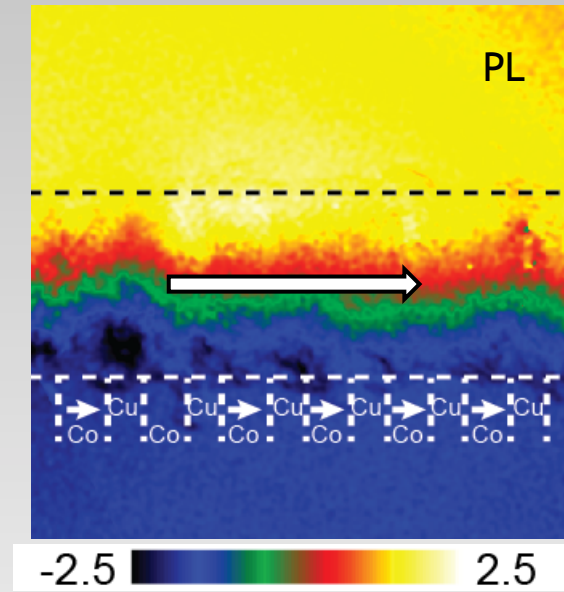
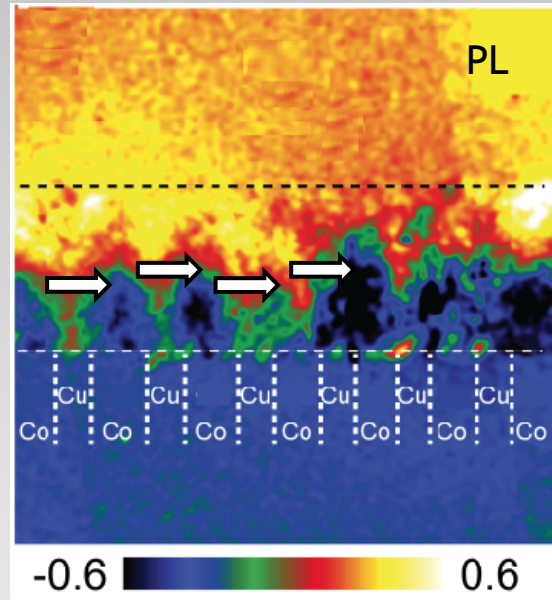
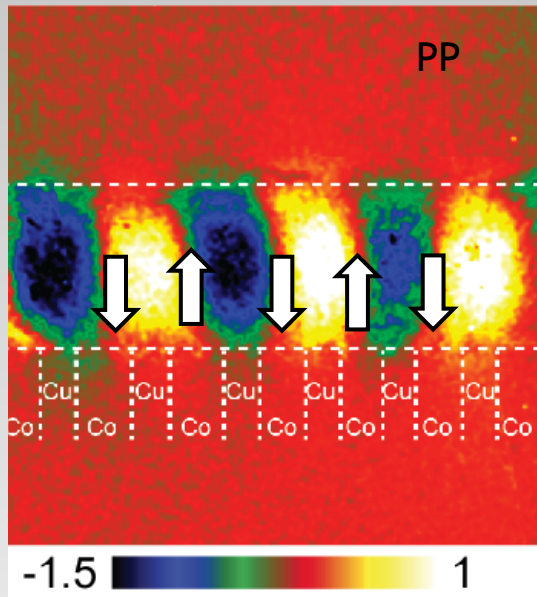
Electromagnet



Co₂₅Cu₁₅ nanowires



Co₂₅Cu₁₅ nanowires



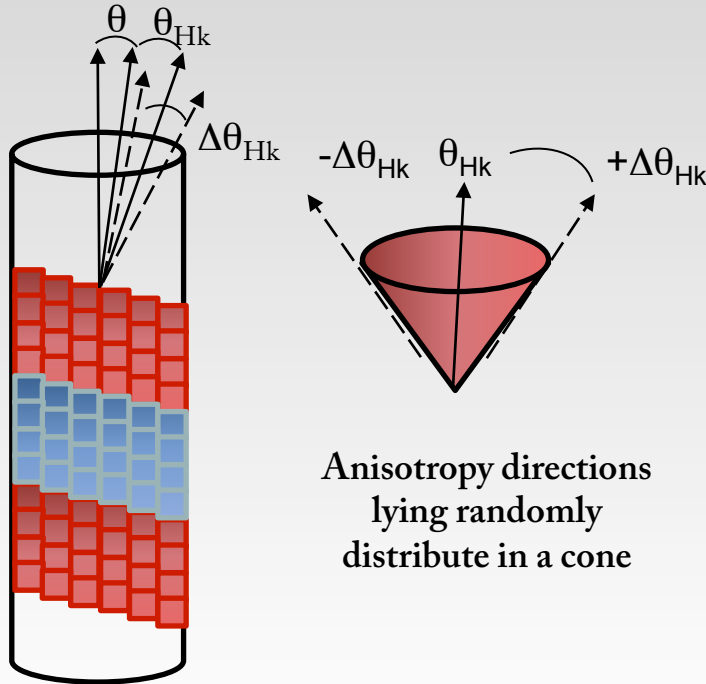
about 10 % NWs

Micro-magnetic calculations

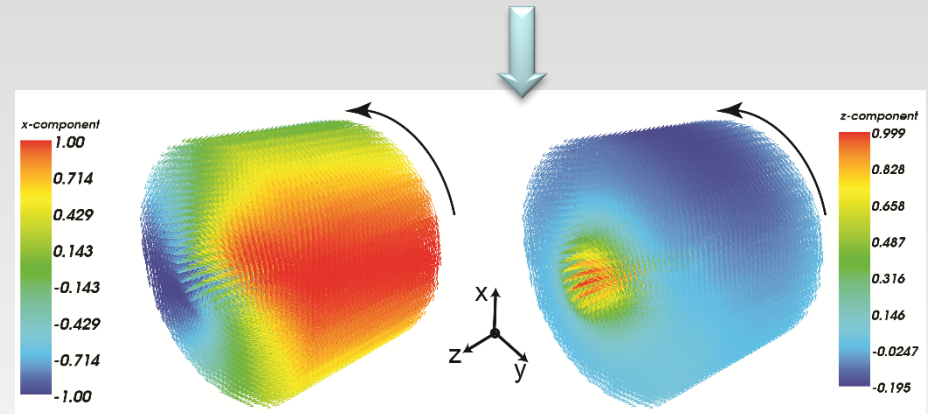
- Universe simulated: $1000 \times 500 \times 500 \text{ nm}^3$
- Cell size: 2 nm
- Magnetization of saturation: $1200 \cdot 10^3 \text{ A/m}$ (1.5 T)
(from reference Co(Cu) NW)
- *Dipolar coupling*
- ➔ Uniaxial anisotropy (K) with orientation distribution around the NW axis
- ➔ Exchange Ct^e (A) ($22 \cdot 10^{-12} \text{ J} \cdot \text{m}^{-1}$)
- ➔ Applied field direction (PP, PL)



3D Representation



Anisotropy directions lying randomly distribute in a cone

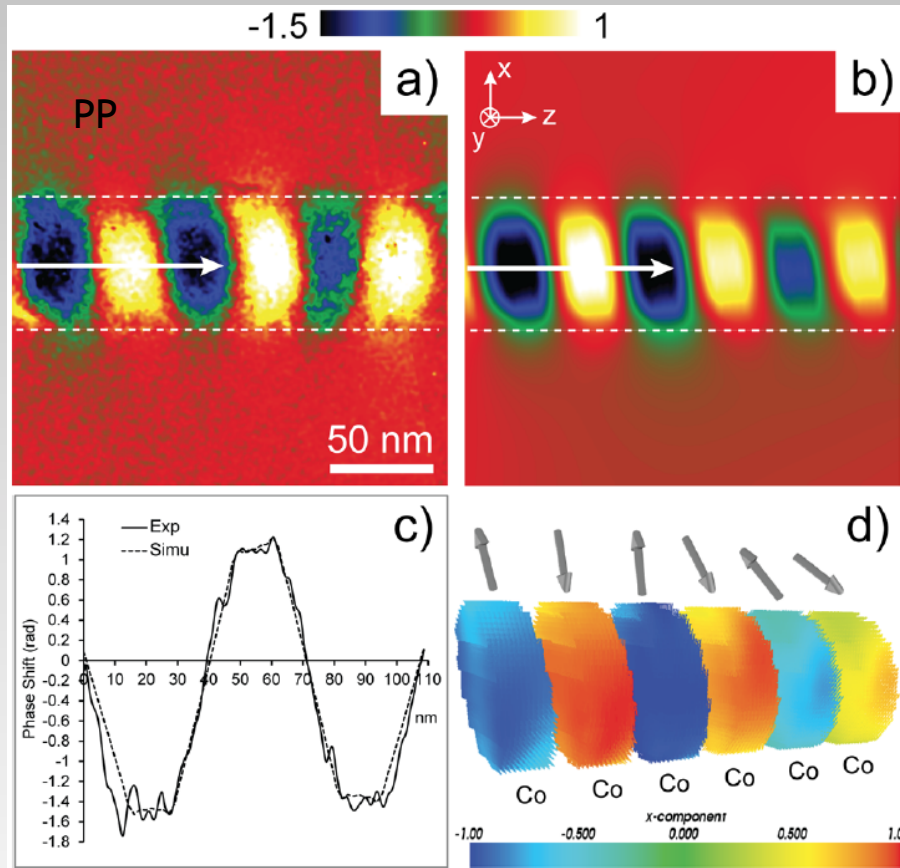


x-component

z-component

Vortex state

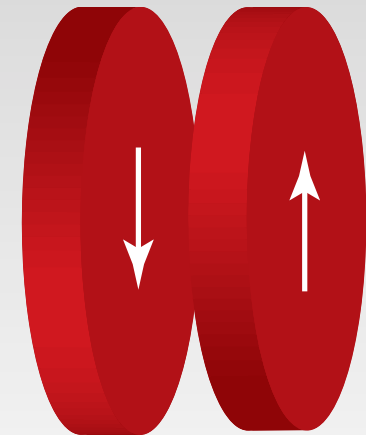
Magnetic configurations Co₂₅/Cu₁₅



Diameter = 64 ± 2 nm

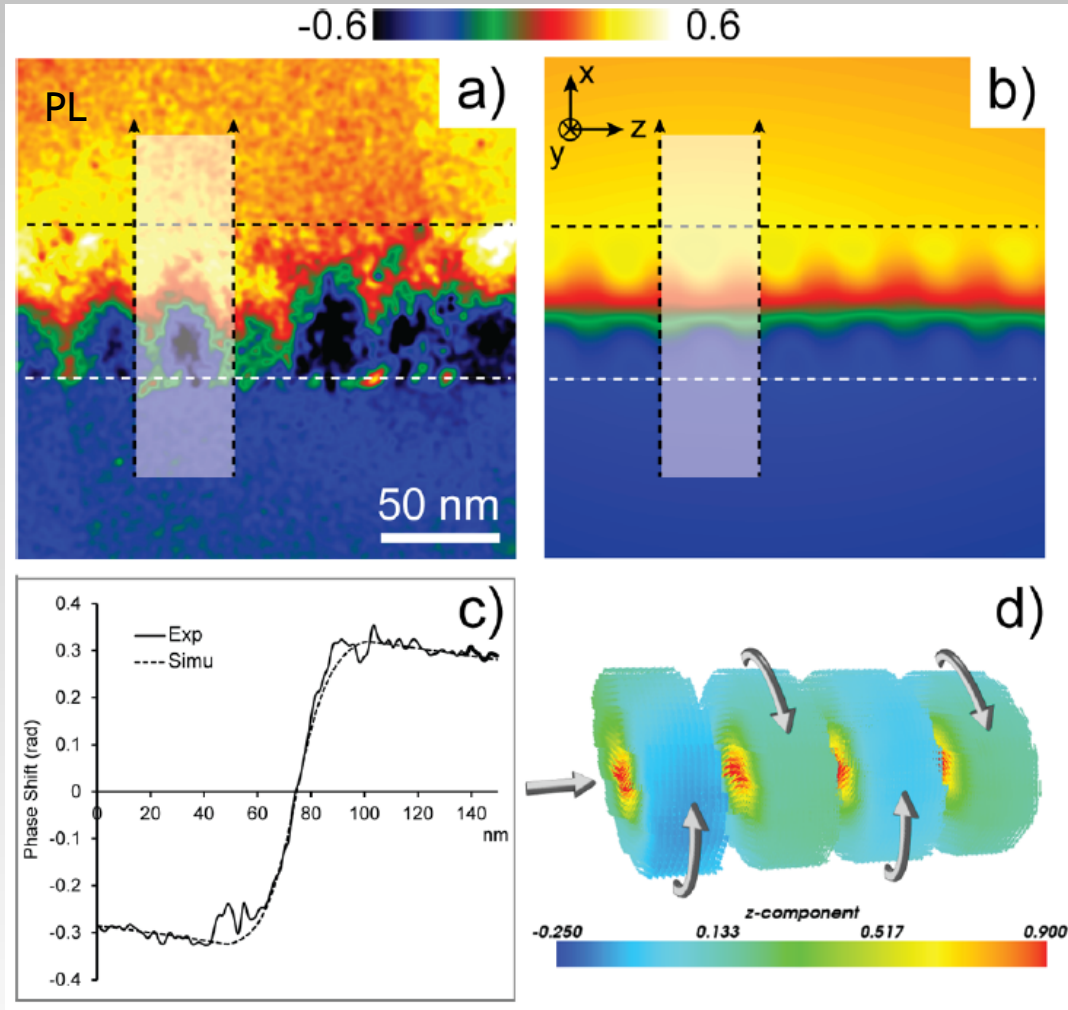
Projection of the magnetic phase shift obtained from the 3D calculations

→ Antiparallel coupling between the Co layers.

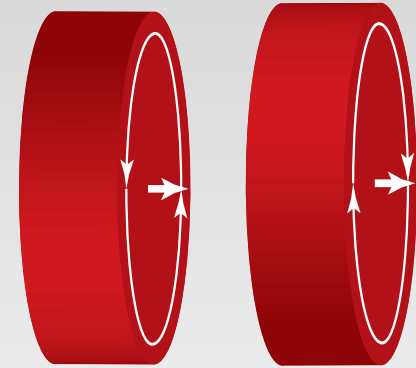


In-plane (I)

Magnetic configurations $\text{Co}_{25}/\text{Cu}_{15}$



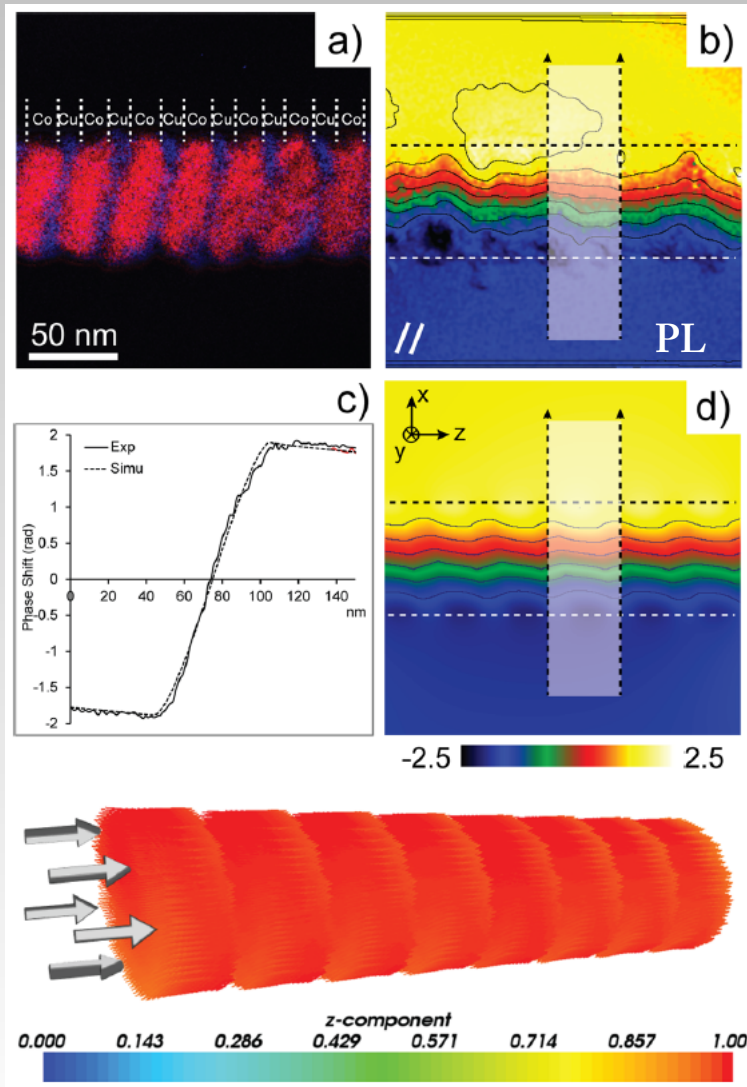
→ Coupled vortices:
different chirality



Vortex (V)

Diameter = 64 ± 2 nm

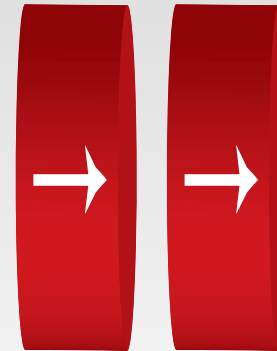
Magnetic configurations $\text{Co}_{25}/\text{Cu}_{15}$



In few NWs in PL with:

- $K = 200 \cdot 10^3 \text{ J} \cdot \text{m}^{-3}$ randomly orientated around the normal of the NWs axis in a 20° cone
- Diameter = $76 \pm 2 \text{ nm}$

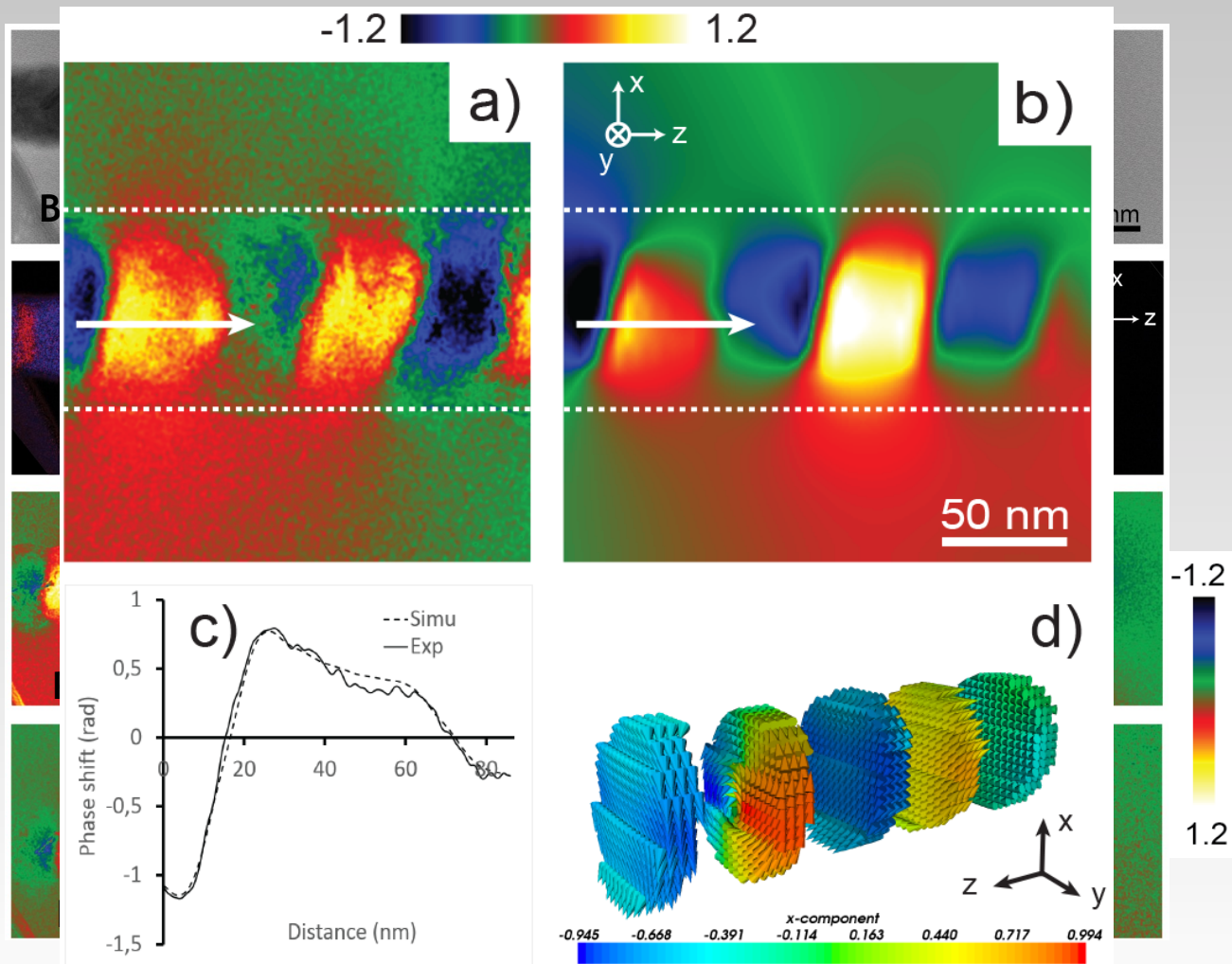
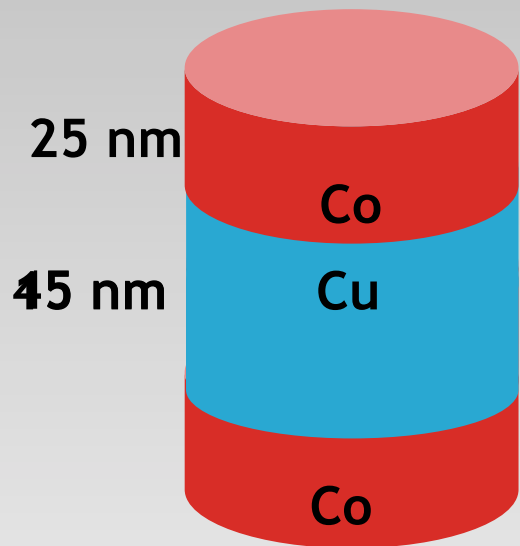
→ Mono domain-like state



Out-of-plane

Magnetic configurations Co₂₅/Cu₄₅

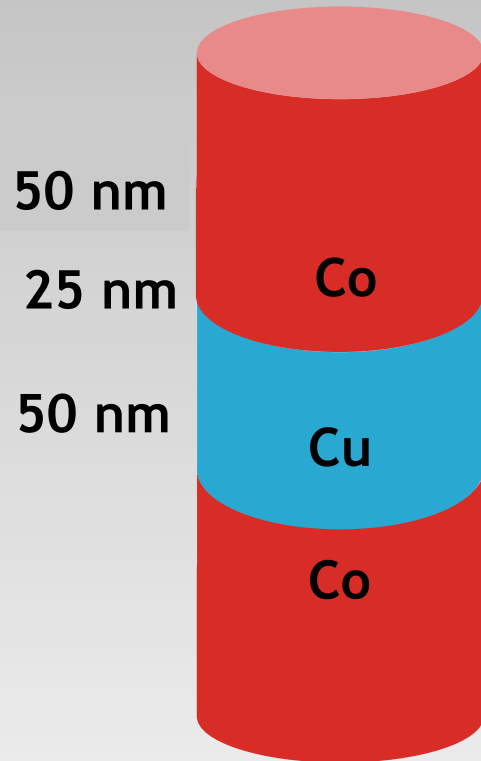
Diameter = 80 ± 2 nm



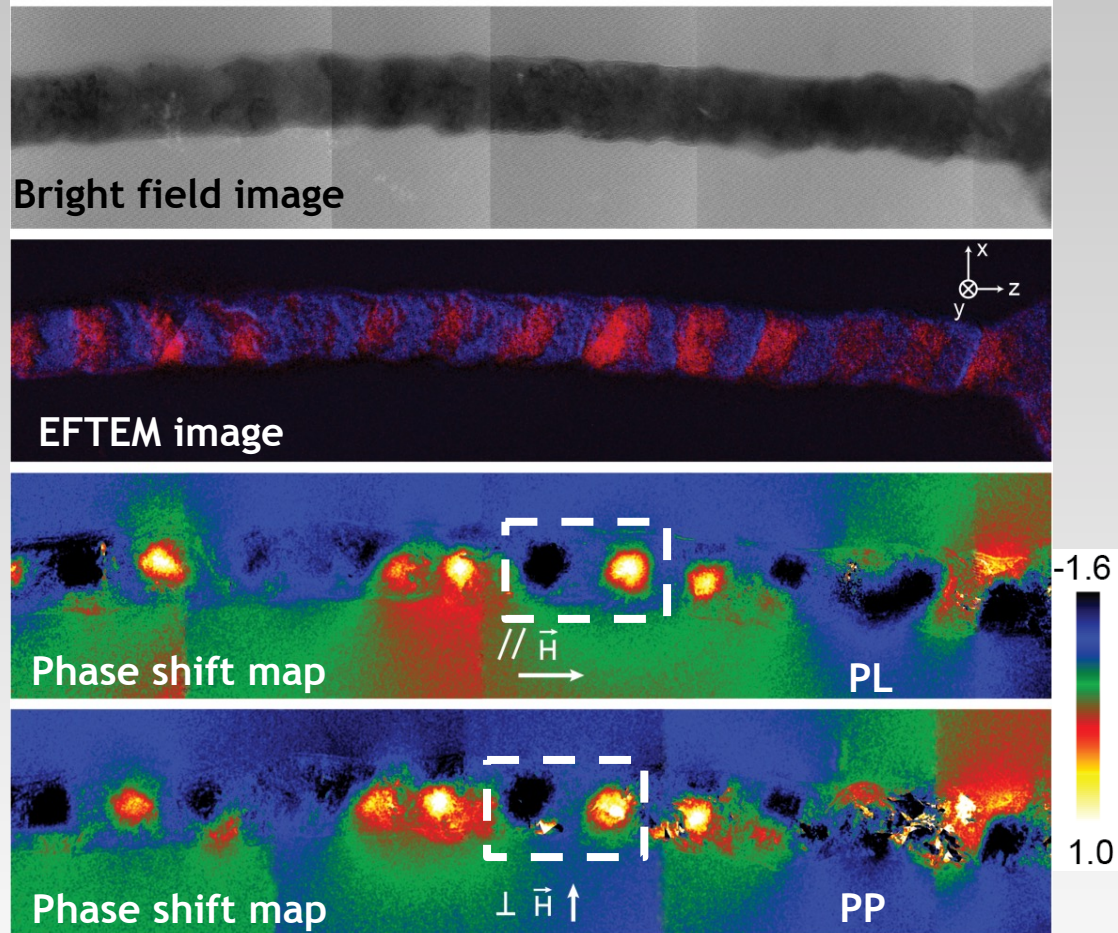
AP + V

Magnetic configurations $\text{Co}_{50}/\text{Cu}_{50}$

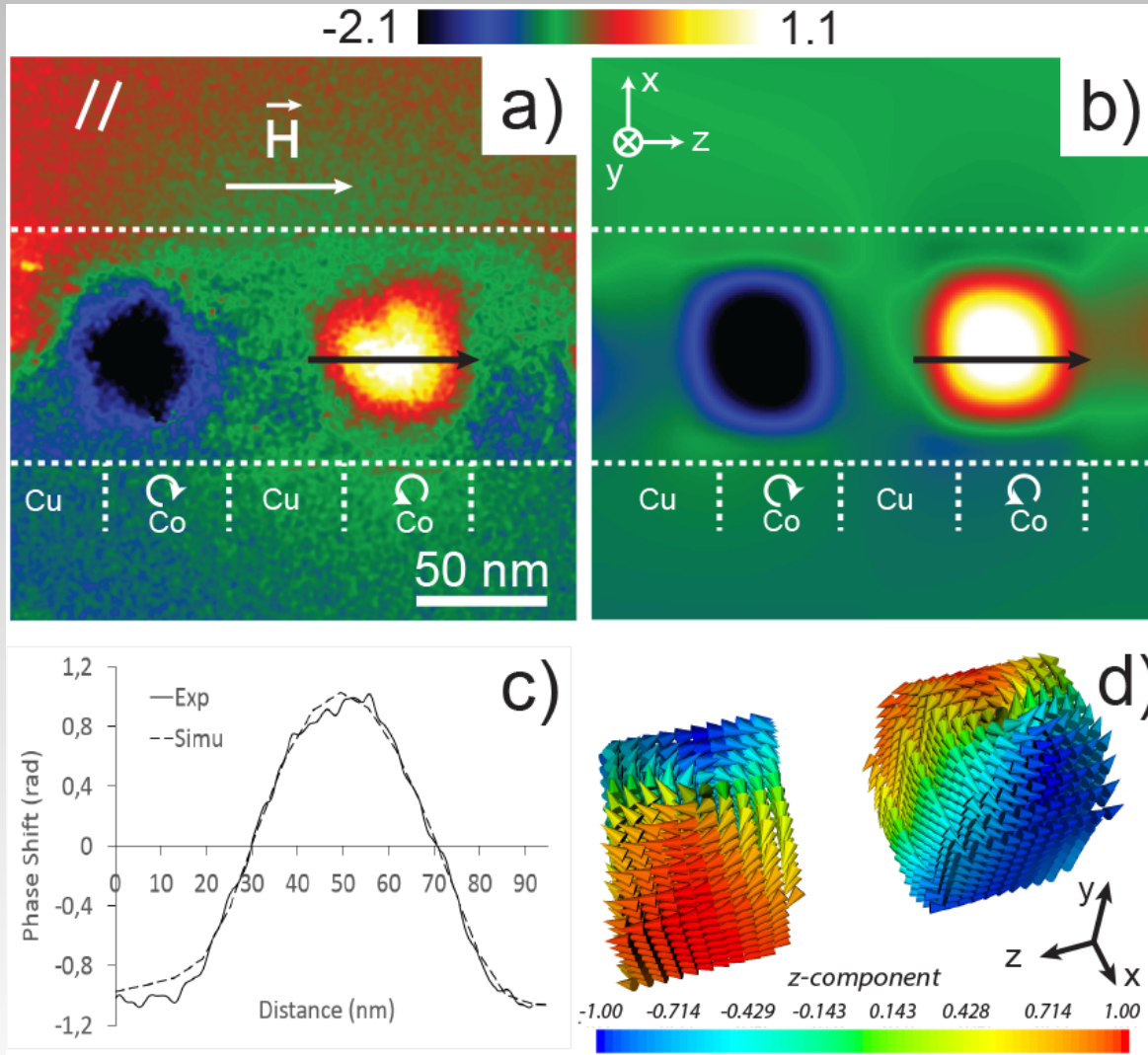
Diameter = 90 ± 2 nm



→ shape anisotropy



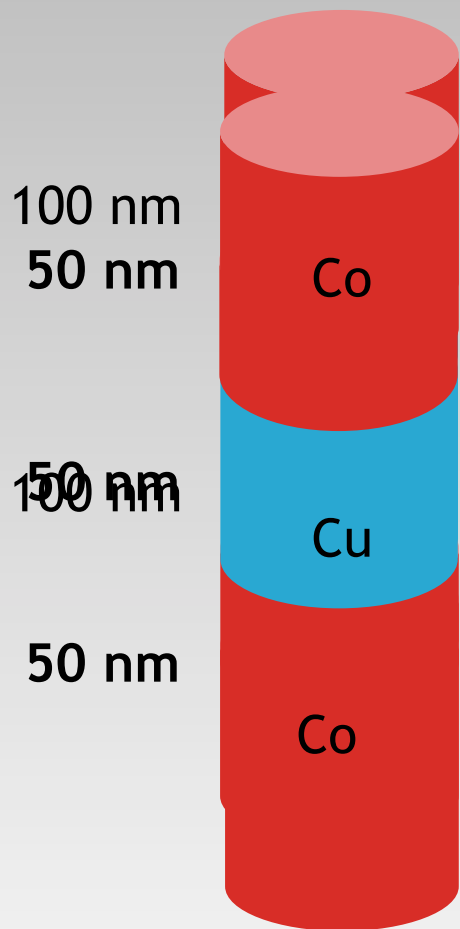
Magnetic configurations Co₅₀/Cu₅₀



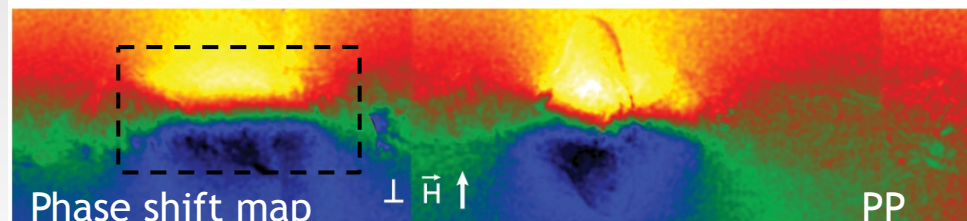
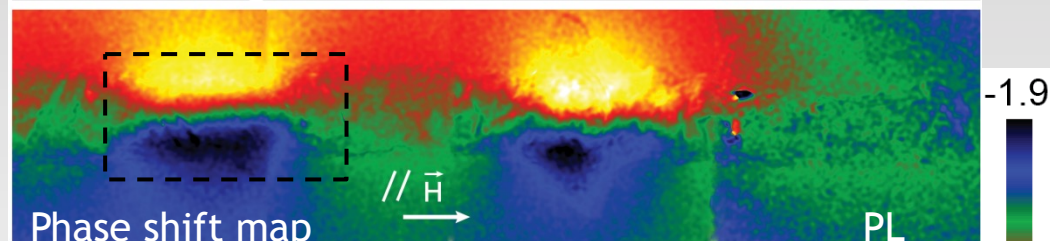
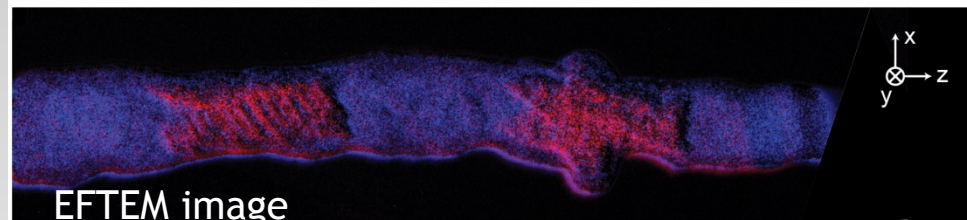
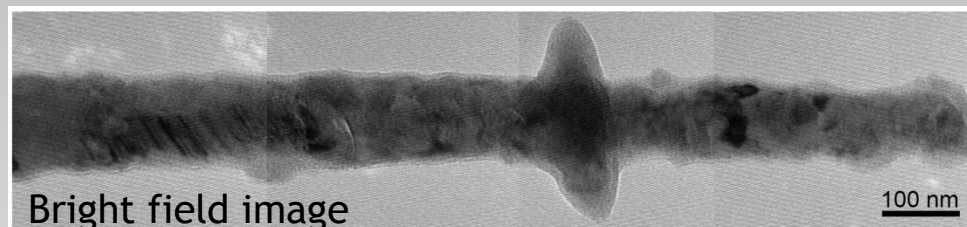
→ Only vortex state with opposite chirality and perpendicular to the wire axis.

Magnetic configurations $\text{Co}_{100}/\text{Cu}_{100}$

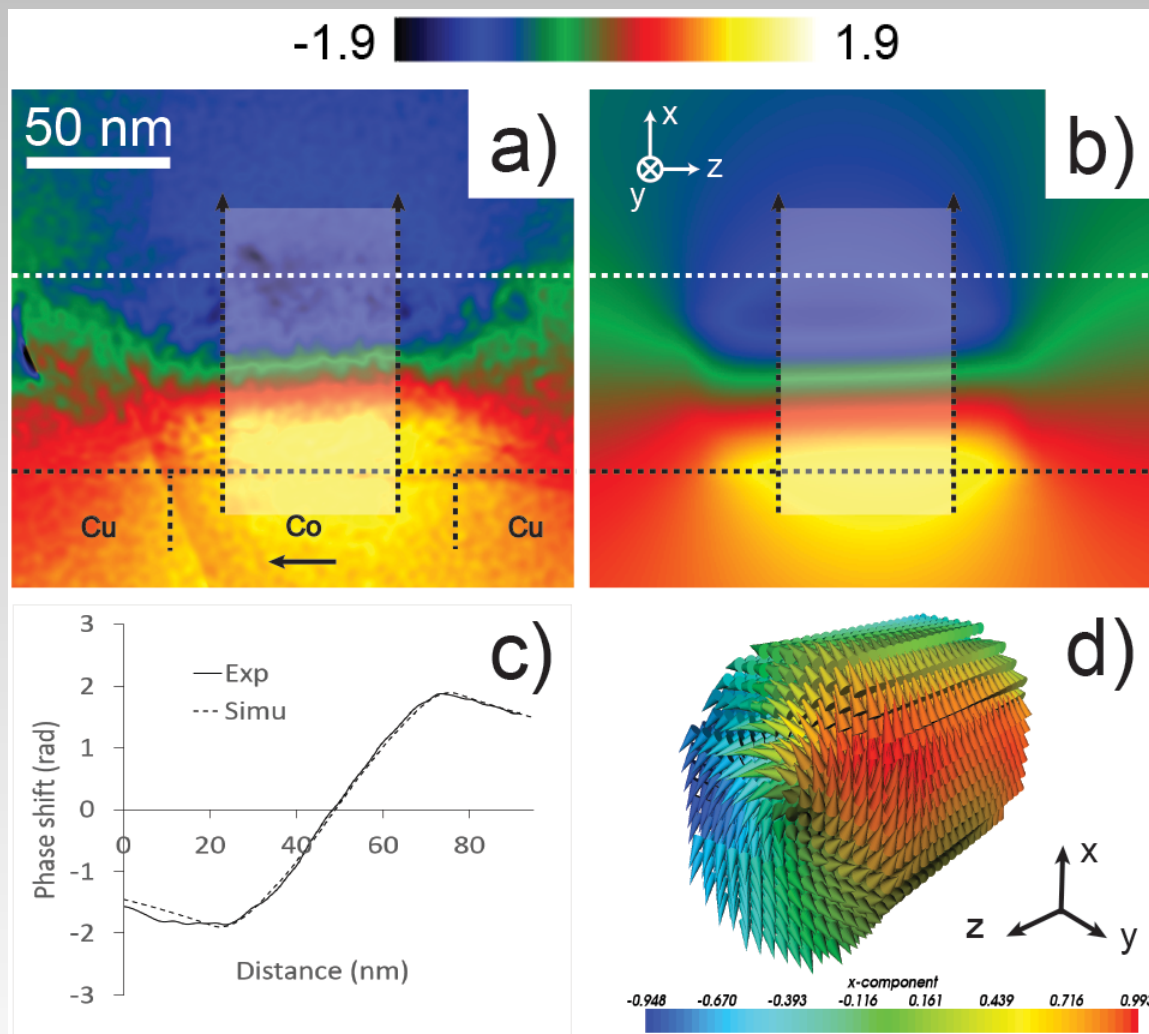
Diameter = 65 ± 2 nm



→ shape anisotropy



Magnetic configurations Co₁₀₀/Cu₁₀₀



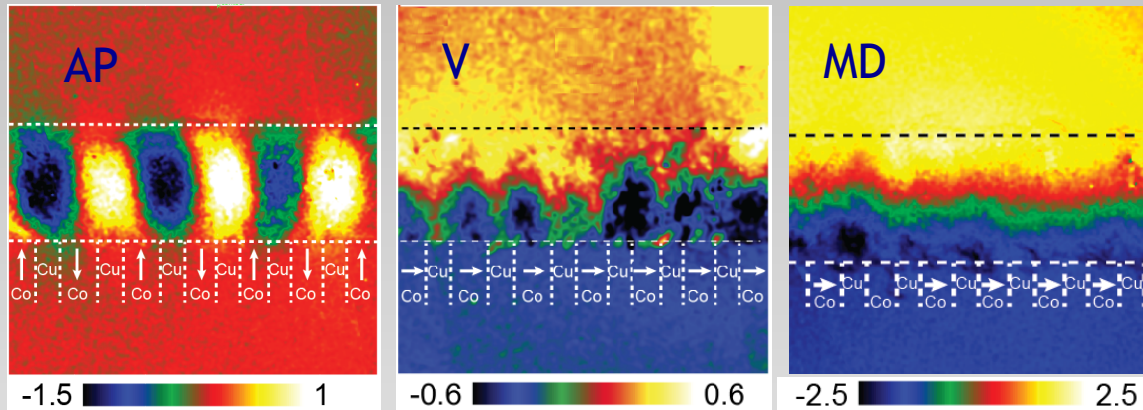
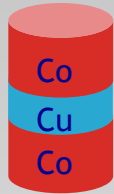
→ Vortex state (V)



Summary magnetic configurations Co/Cu

Dipolar coupling & anisotropy intensity and direction

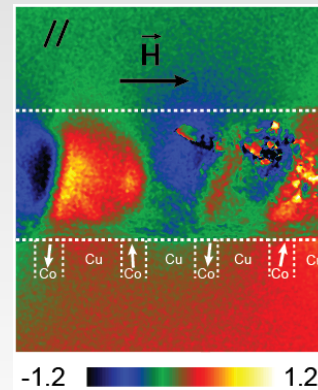
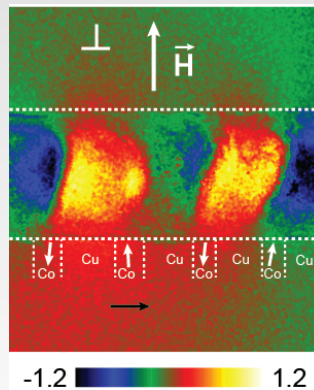
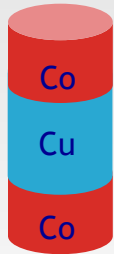
25 nm
15 nm



Transition AP to V

Coupling between Co layers

25 nm
45 nm

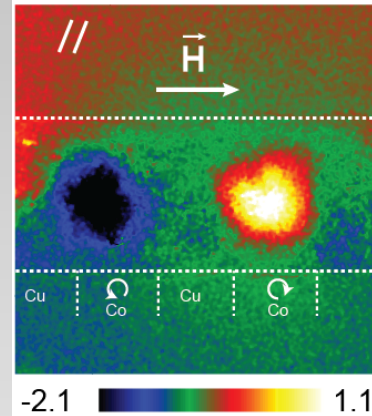
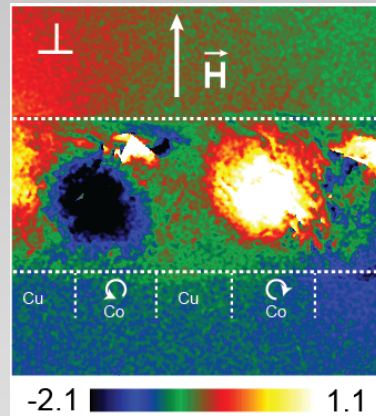
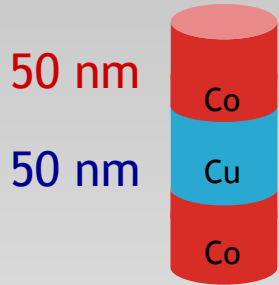


AP+ V

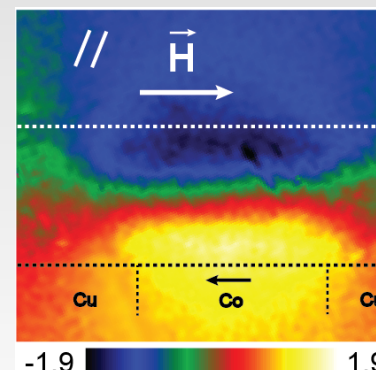
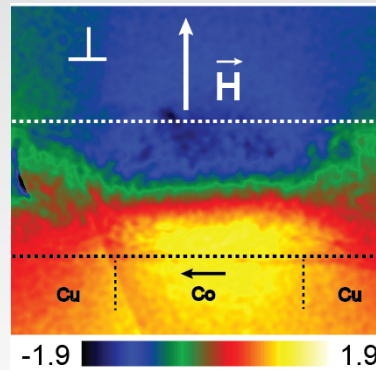
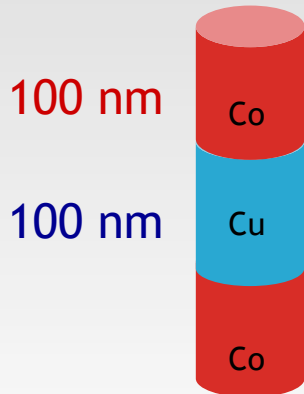
- Same remnant state
- No monodomain state

Summary magnetic configurations Co/Cu

Shape anisotropy



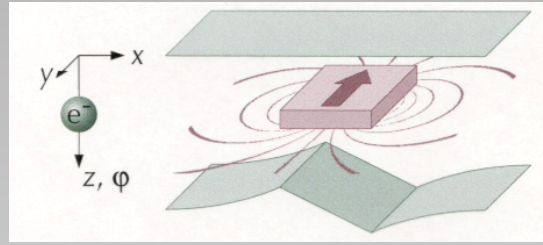
- Same remnant state (Vortex perpendicular to the NW axis)
- No “strong” Coupling



- Same remnant state (Vortex // to the NW axis)
- No Coupling

D. REYES, N. BIZIERE, B. WAROT-FONROSE, T. WADE and C. GATEL
Nanoletters 2016, 16 (2), pp 1230-1236

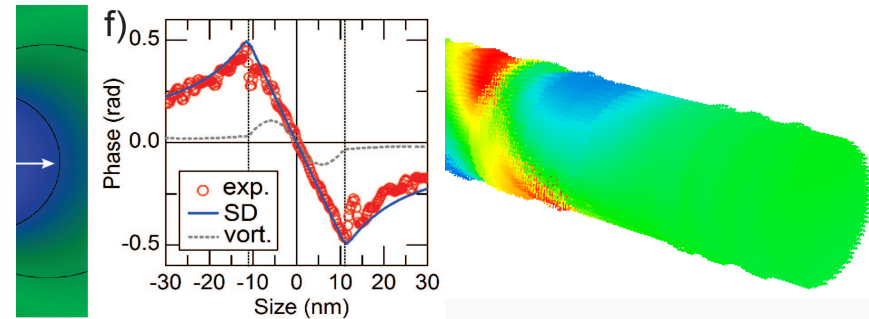
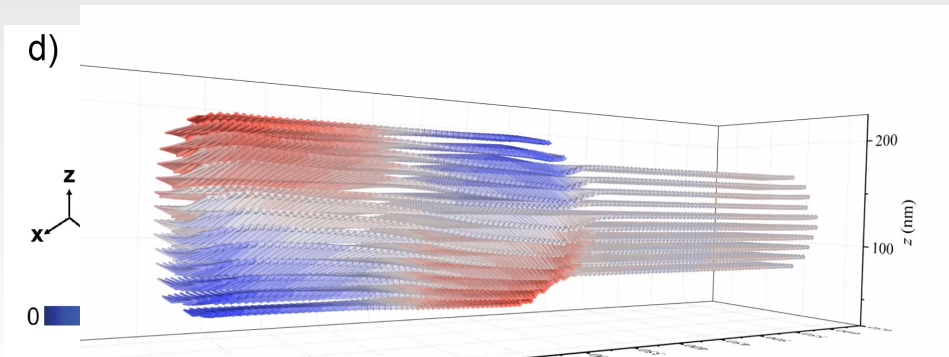
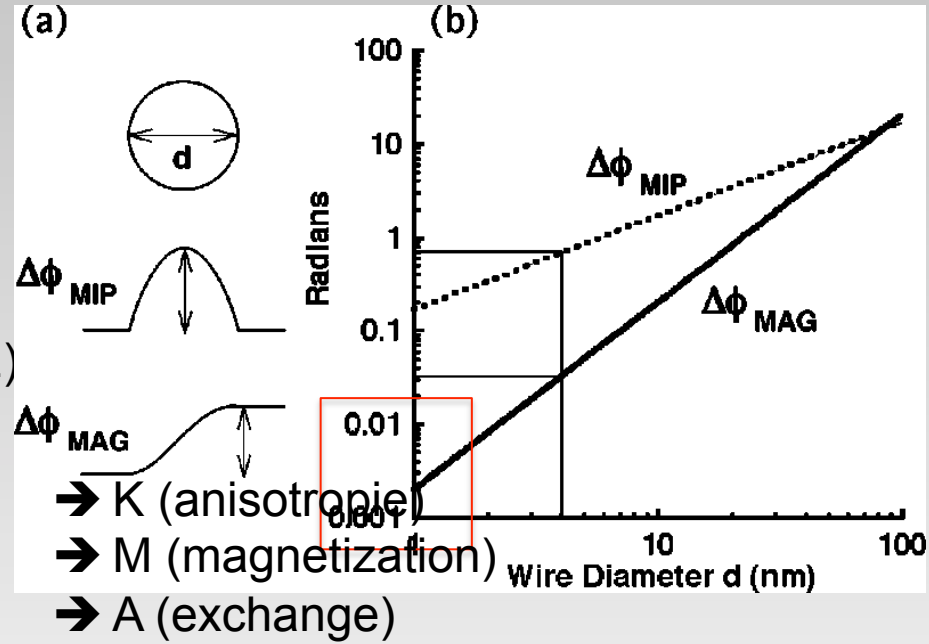
Summary



$$\phi_{Mag}(x) = -\frac{e}{\hbar} \iint B_{\perp}(x,z) \cdot dx \cdot dz$$



- Spatial resolution
- Phase sensitivity (high brightness sources, stability, good detectors (DQE))





Centre d'Elaboration de Matériaux et d'Etudes Structurales
BP 4347, F-31055 Toulouse Cedex 4

Two postdoctoral positions in advanced electron microscopy at CEMES

Description

The CEMES laboratory in Toulouse is looking for two post-doctoral scientists to join their electron microscopy team. We are currently working on several aspects of transmission electron microscopy: in-situ experiments (applied fields), electron holography, data analysis, new instrumentation and computer-controlled microscopy.

Developments will particularly concern our unique instrument designed for in-situ interferometry, the I2TEM microscope: Hitachi HF-3300 TEM equipped with a cold-field emission gun (C-FEG) operating at 300kV, two specimen stages (standard stage within the pole-piece gap of the objective lens and upper-stage placed above the objective lens for field-free imaging), an image corrector (CEOS B-COR for correcting off-axial aberrations) and four biprisms (three post-specimen and one in the condenser lens system). The microscope is also equipped with a GIF (Quantum) and high-speed pre-GIF 4K CCD detector (Gatan OneView). <https://sites.google.com/site/equipxmimetis/>

The CEMES coordinates the European Infrastructure for Electron Microscopy (ESTEEM2) and is founding member of the French Microscopy Network (METS).

Requirements

- PhD in electron microscopy
- Experimental or computer analysis expertise
- Modelling skills such as finite-element method a plus
- Working language: English or French

Job conditions

Starting salary: 2500 €/month gross as a function of experience

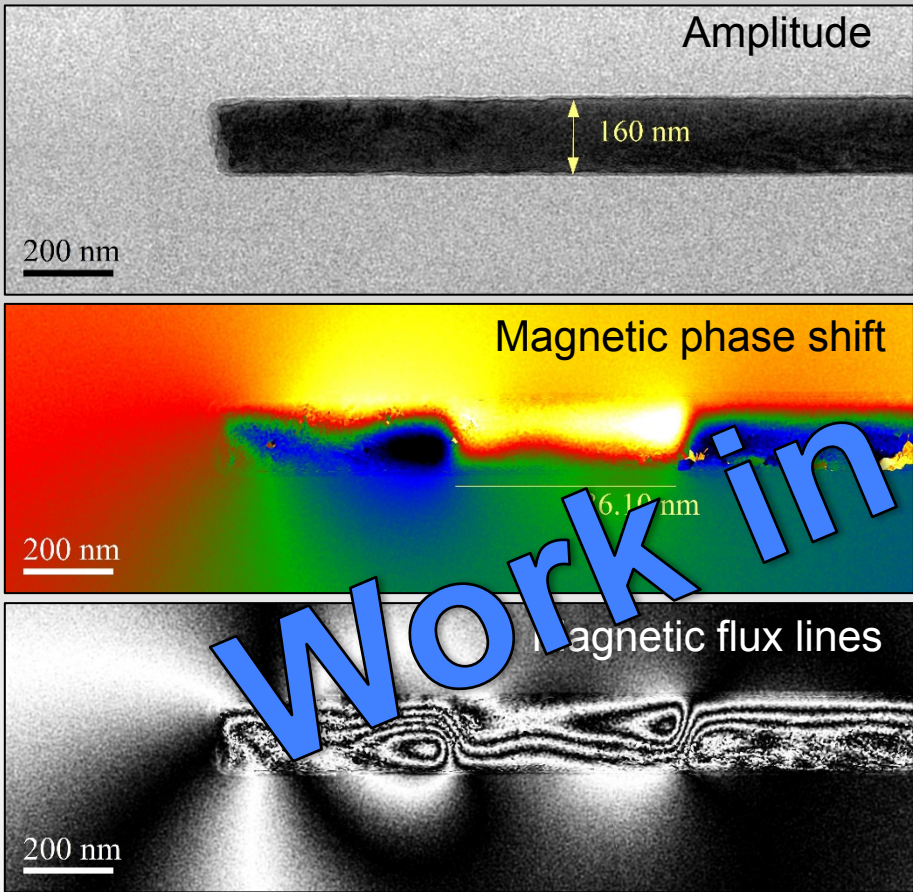
Starting date: from June 2017 onwards

Contact:

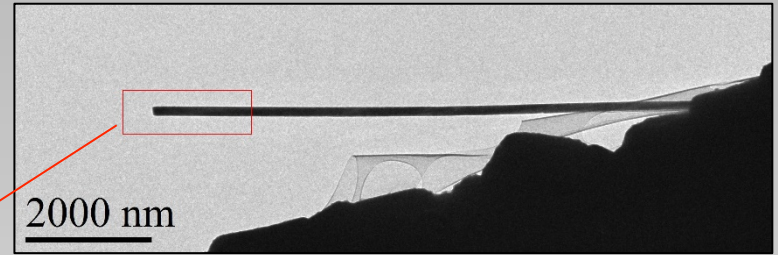
Dr Etienne Snoeck	or Dr Martin Hÿtch
Director of CEMES-CNRS 29 rue Jeanne Marvig 31055 Toulouse France email: snoeck@cemes.fr	CEMES-CNRS 29 rue Jeanne Marvig 31055 Toulouse France email : hÿtch@cemes.fr

**Thank you for your
attention**

« c » axis perpendicular to the NW ?
 → Competition between shape and
 cristallographic anisotropy

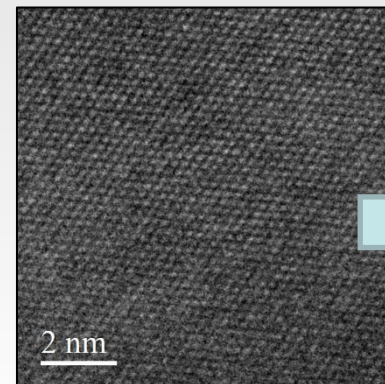
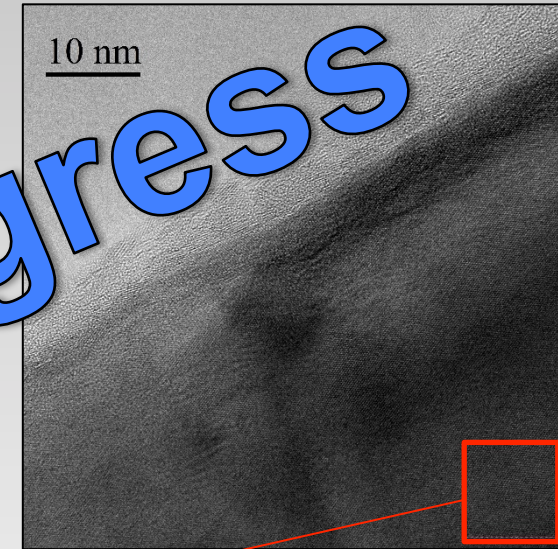


Low-magnification bright field TEM image of a
 isolated $\text{Co}_{85}\text{Ni}_{15}$ NW.

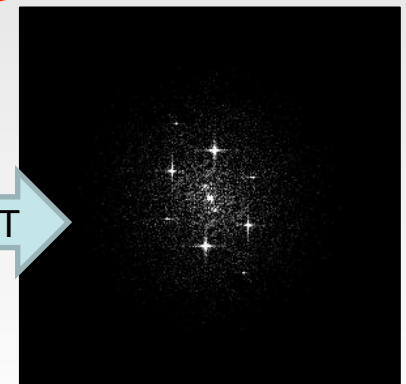


work in progress

→ Long-range
 single-crystal
 structure



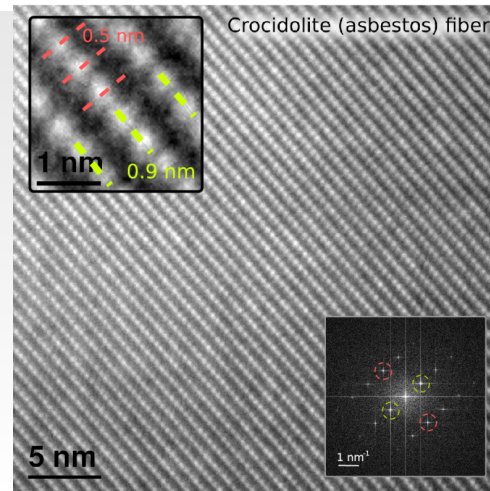
FFT



I2TEM: Spatial Resolution

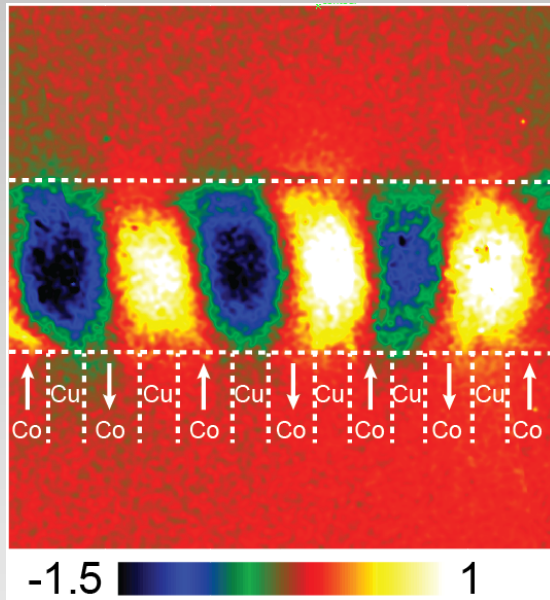
Observation modes	Aberration	Stage	Focusing	Cs	Cc	Resolution	Mag. at SA	Dark field
OBJ off observation Lorentz mode	Uncorrected	Standard	Int 1	770 mm	50 mm	1.0 nm	-	NA
	Corrected (Aplanatic)	Standard	TL11	0 mm	164 mm	0.56 nm	8.8	NA
Double stage (single focus Lorentz mode)	Uncorrected	Upper	Objective (weak)	3.2 m	63 mm	1.43 nm	3.8	NA
	Corrected (Aplanatic)	Upper	Objective (weak)	0 m	115 mm	0.48 nm	13	NA
Double stage (double focus Lorentz mode)	Uncorrected	Upper	Objective (strong)	241 m	1.1 m	4.2 nm	4.1	Available
	Corrected (Aplanatic)	Upper	Objective (strong)	0 m	1.7 m	1.8 nm	3.8	Available
High resolution mode	Uncorrected	Standard	Objective	2.3 mm	2.4 mm	0.24 nm	61	Available
	Corrected (Aplanatic)	Standard	Objective	0 mm	3.7 mm	0.084 nm	99 (Changeable)	Available

→ 0.48 nm spatial resolution confirmed experimentally in field free mode (B-Corr)

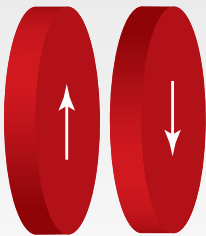


Magnetic configurations Co₂₅/Cu₁₅

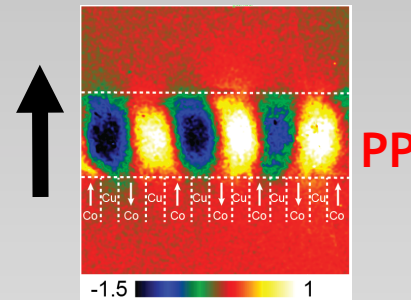
Antiparallel (AP)



PP

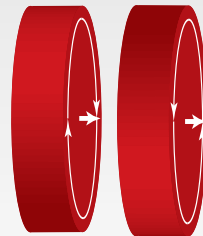
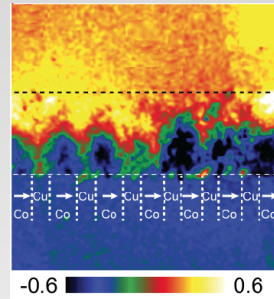


Transition from AP to V (V)

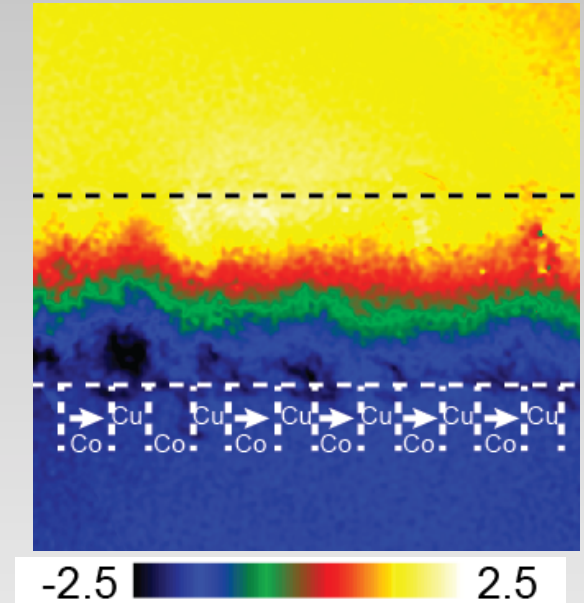


PP

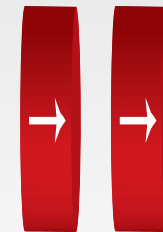
PL



Monodomain-like (MD)



PL



D. Reyes et al., *Nano Lett.*, 16 (2), pp 1230-1236, (2016)