Measurement of magnetic fields by transmission electron microscopy with nm scale resolution

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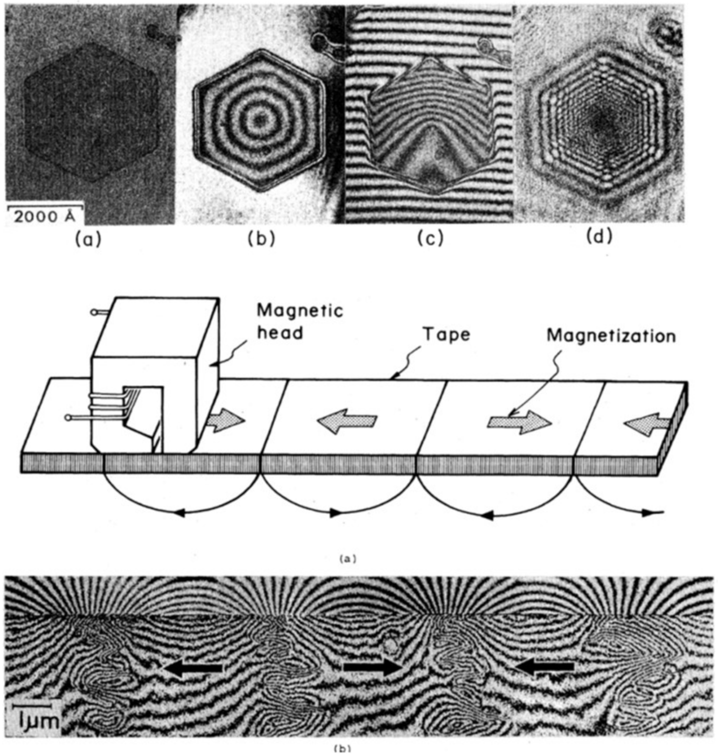
*Presenting author

Contents of this presentation

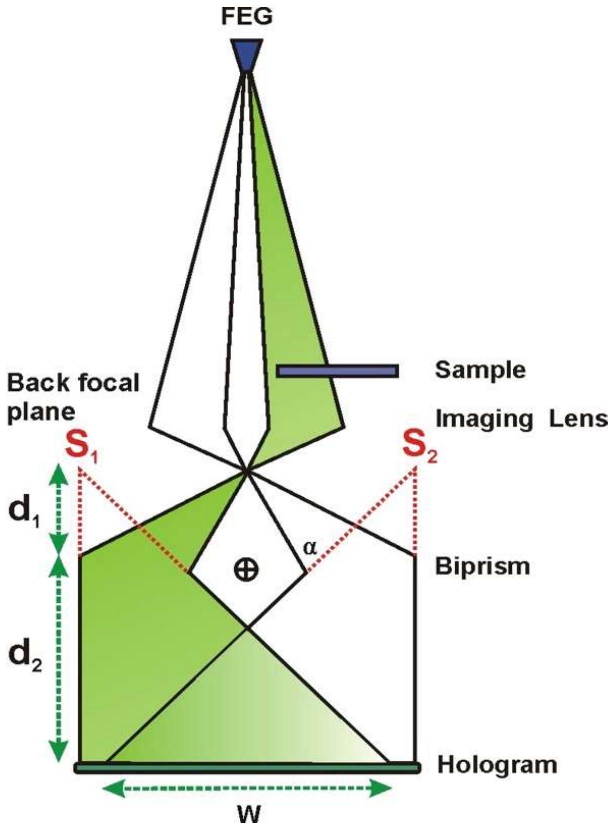
- **Introduction of electron holography** for the mapping of electromagnetic fields
- **Improving** spatial resolution and sensitivity

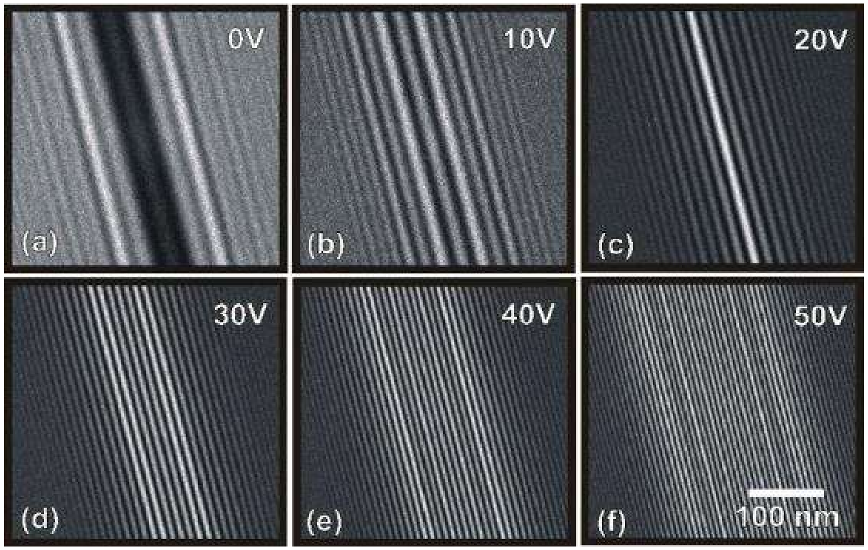
- Demonstration of **quantitative holography** on a simple NiFe Nanowire
- Electron holography of **STT-MRAM and PSA-STT-MRAM devices at elevated temperature.**



Applications of electron holography
Akira Tonomura, *Rev. Mod. Phys*. 59, 639 (1987)

Off-axis electron holography – Phase mapping of electrons



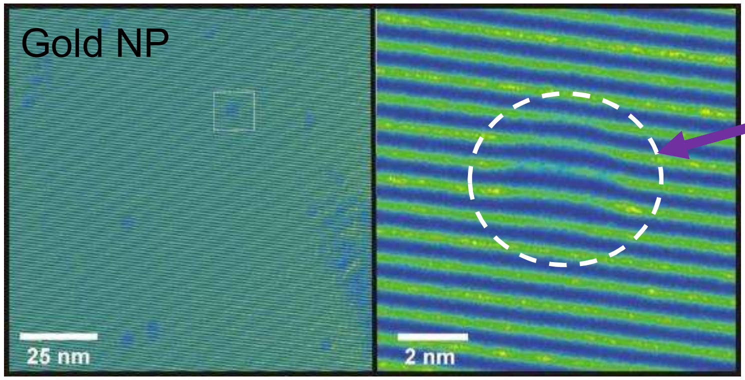


Spatial resolution

⇕

Fringes frequency

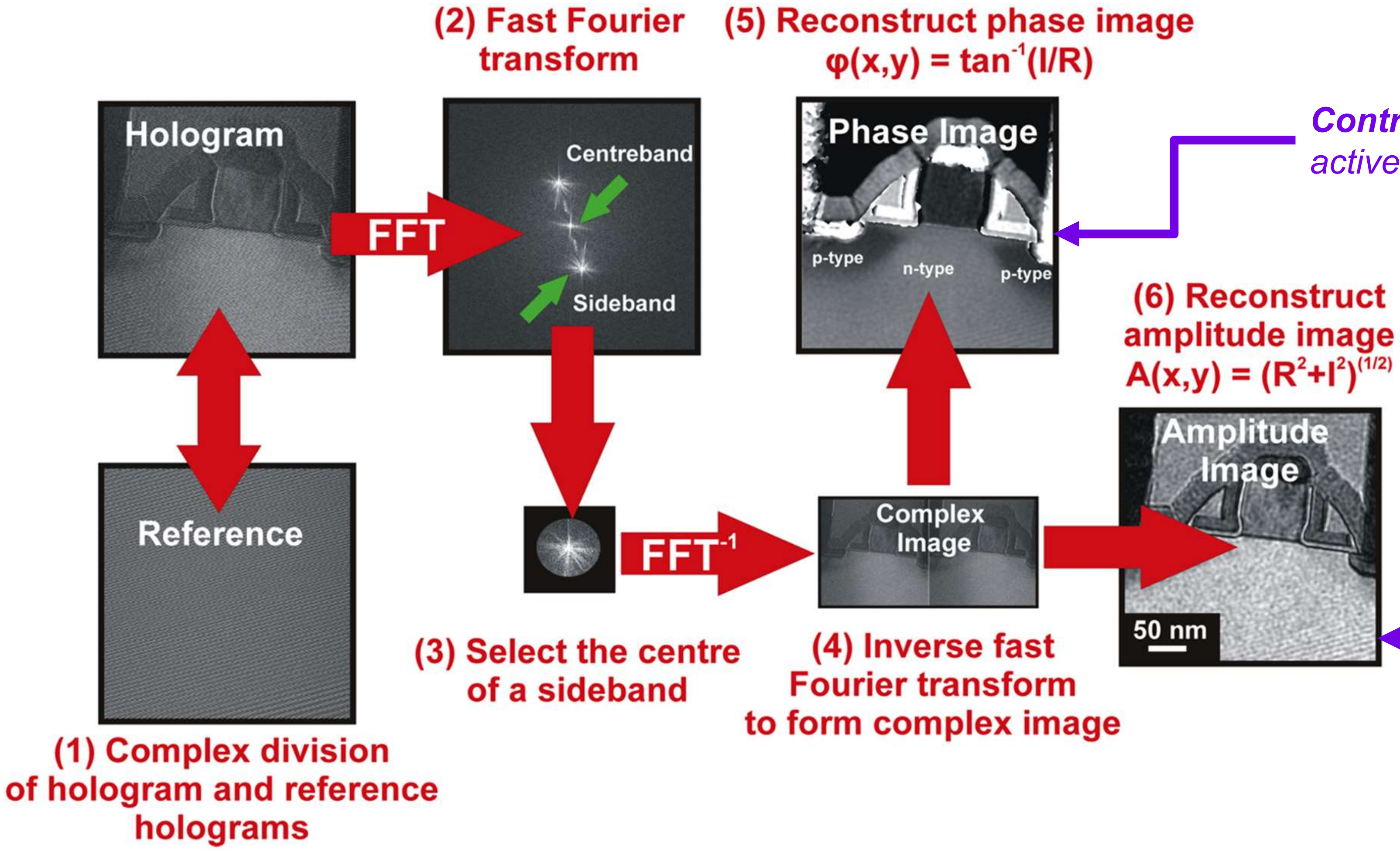
contrast issue
@high frequency !



Shifted fringes:
Thickness
Electrostatic field

$$\varphi(x) = C_E \int \boxed{V(x,z)} dz - \frac{e}{h} \int \int \boxed{B \perp (x,z)} dx, dz$$

Off-axis electron holography – Hologram reconstruction - *p*MOS device



(1) Complex division of hologram and reference holograms

(2) Fast Fourier transform

(3) Select the centre of a sideband

(4) Inverse fast Fourier transform to form complex image

(5) Reconstruct phase image $\varphi(x,y) = \tan^{-1}(I/R)$

(6) Reconstruct amplitude image $A(x,y) = (R^2+I^2)^{(1/2)}$

Contrast due to *active dopants*

No contrast

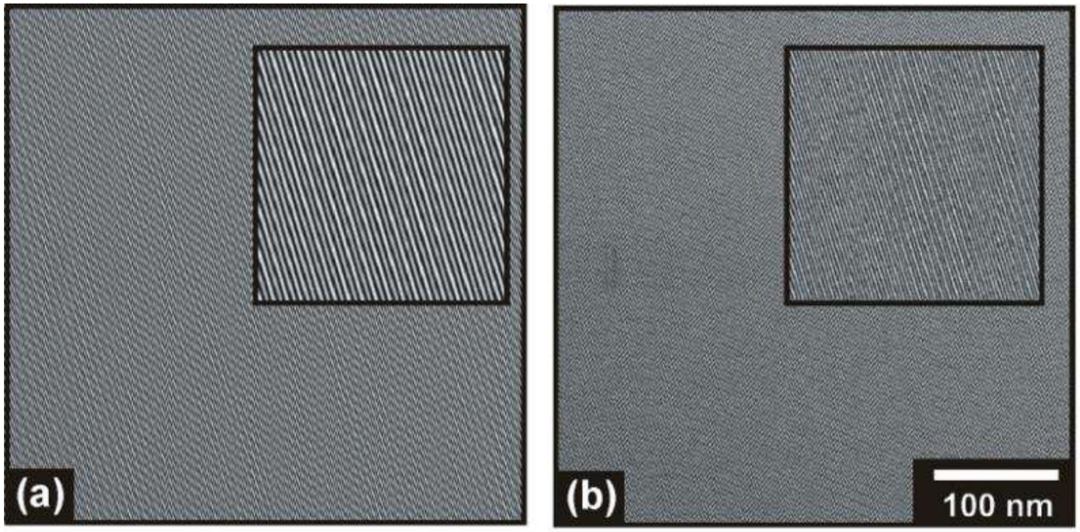
Off-axis electron holography – New Developments

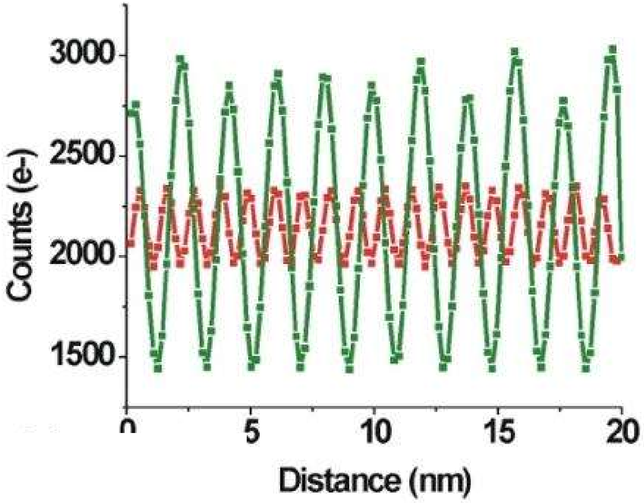
Improving the spatial resolution:
Finer interference fringes @higher frequency

Phase sensitivity: $$\delta\phi = \frac{1}{N\mu^2}$$

Hologram fringe contrast.

Electron counts



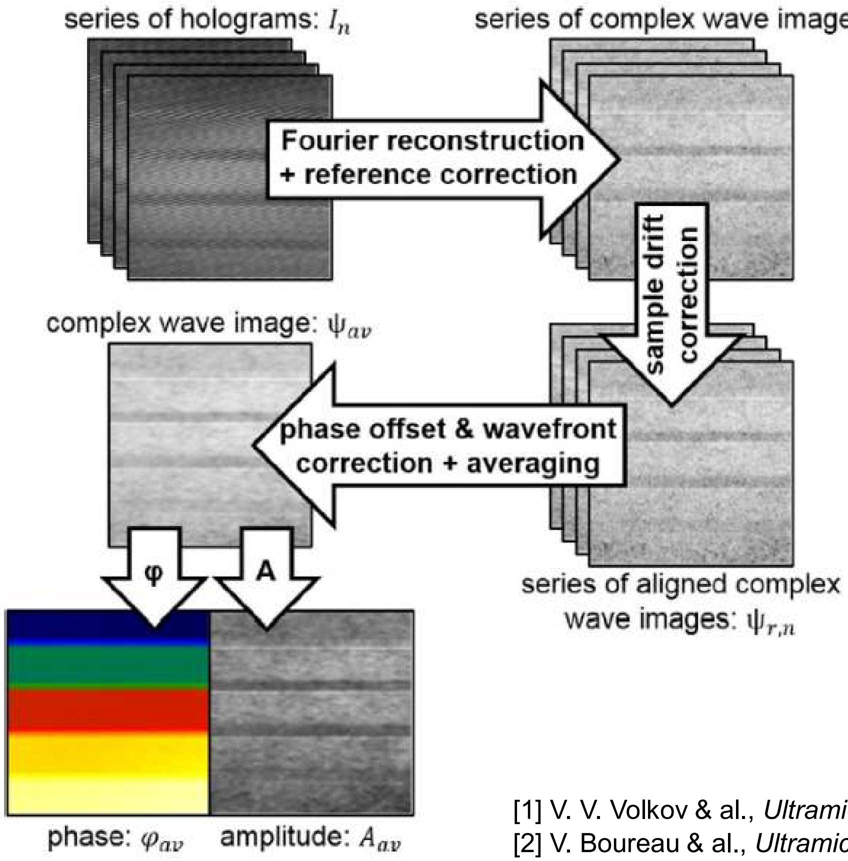


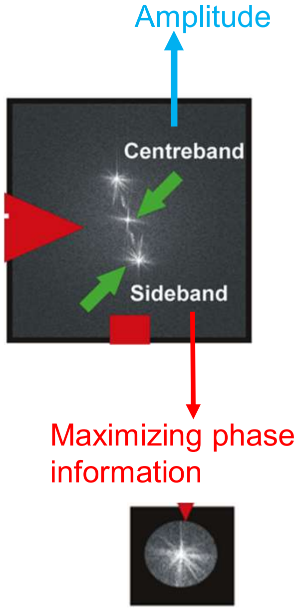
- If we have finer fringes then we lose contrast !!
- Solution is to ***add together a large series of holograms*** → *not trivial*

[1] V. Boureau & al., *Ultramicroscopy*, 193, 52-63 (2018)

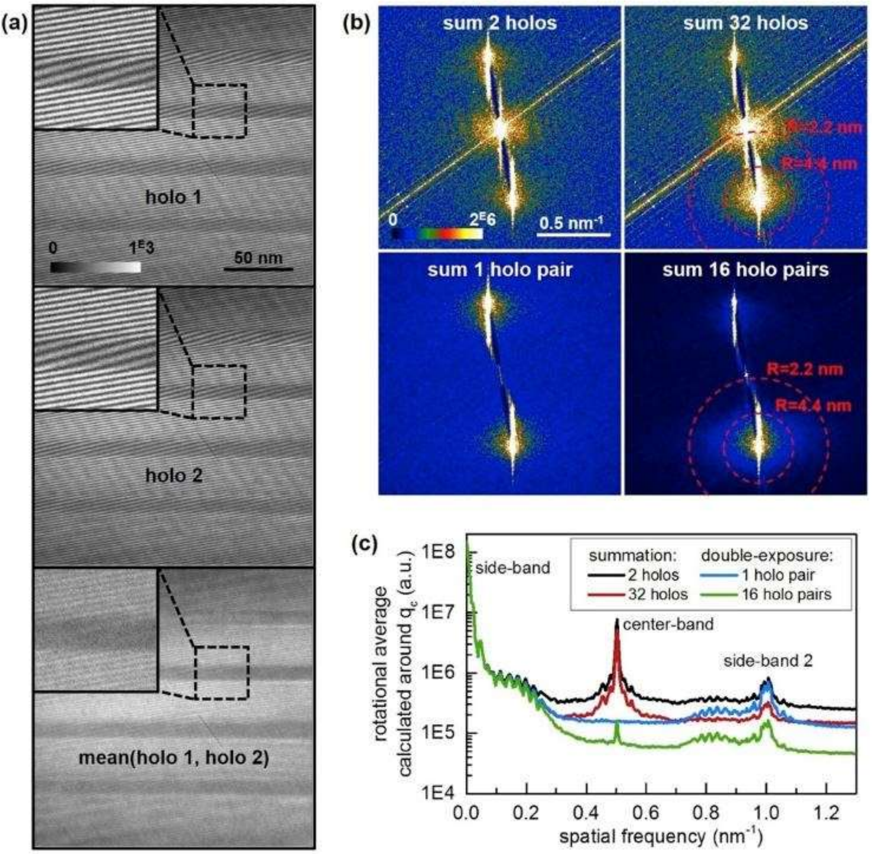
Off-axis electron holography – π phase shifting to remove centreband

1. Aligning stacked holograms





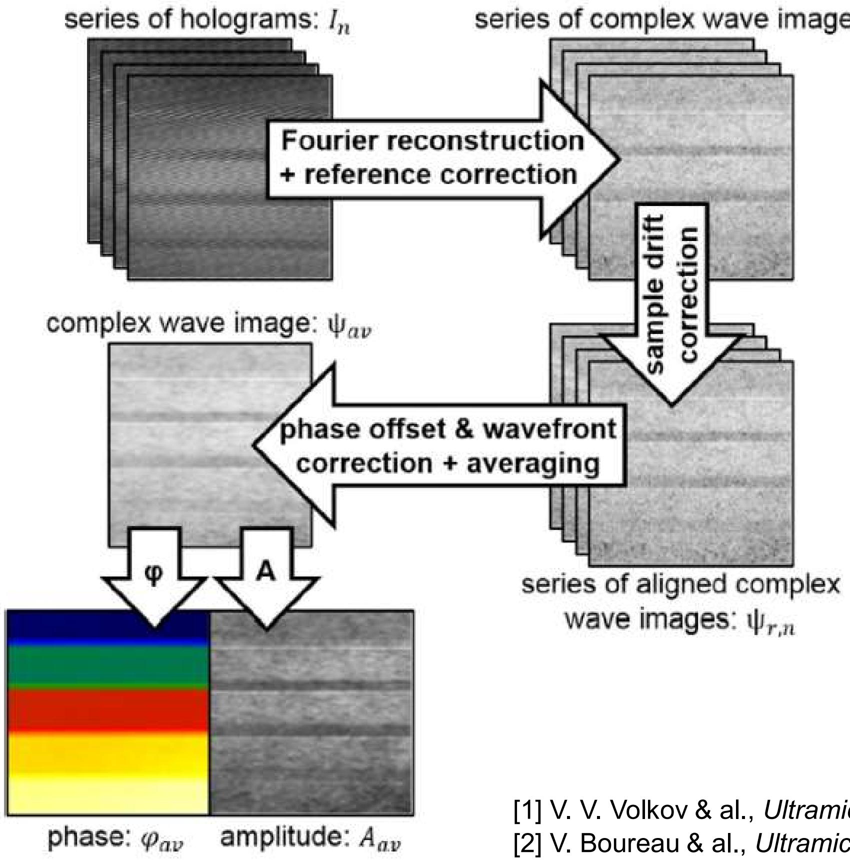
2. Summation with π phase shifting

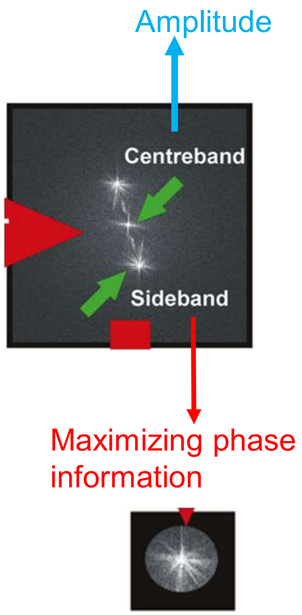


[1] V. V. Volkov & al., *Ultramicroscopy* 134, 175 (2013)
[2] V. Boureau & al., *Ultramicroscopy* 193, 52-63 (2018)

Off-axis electron holography – π phase shifting to remove centreband

1. Aligning stacked holograms





Amplitude

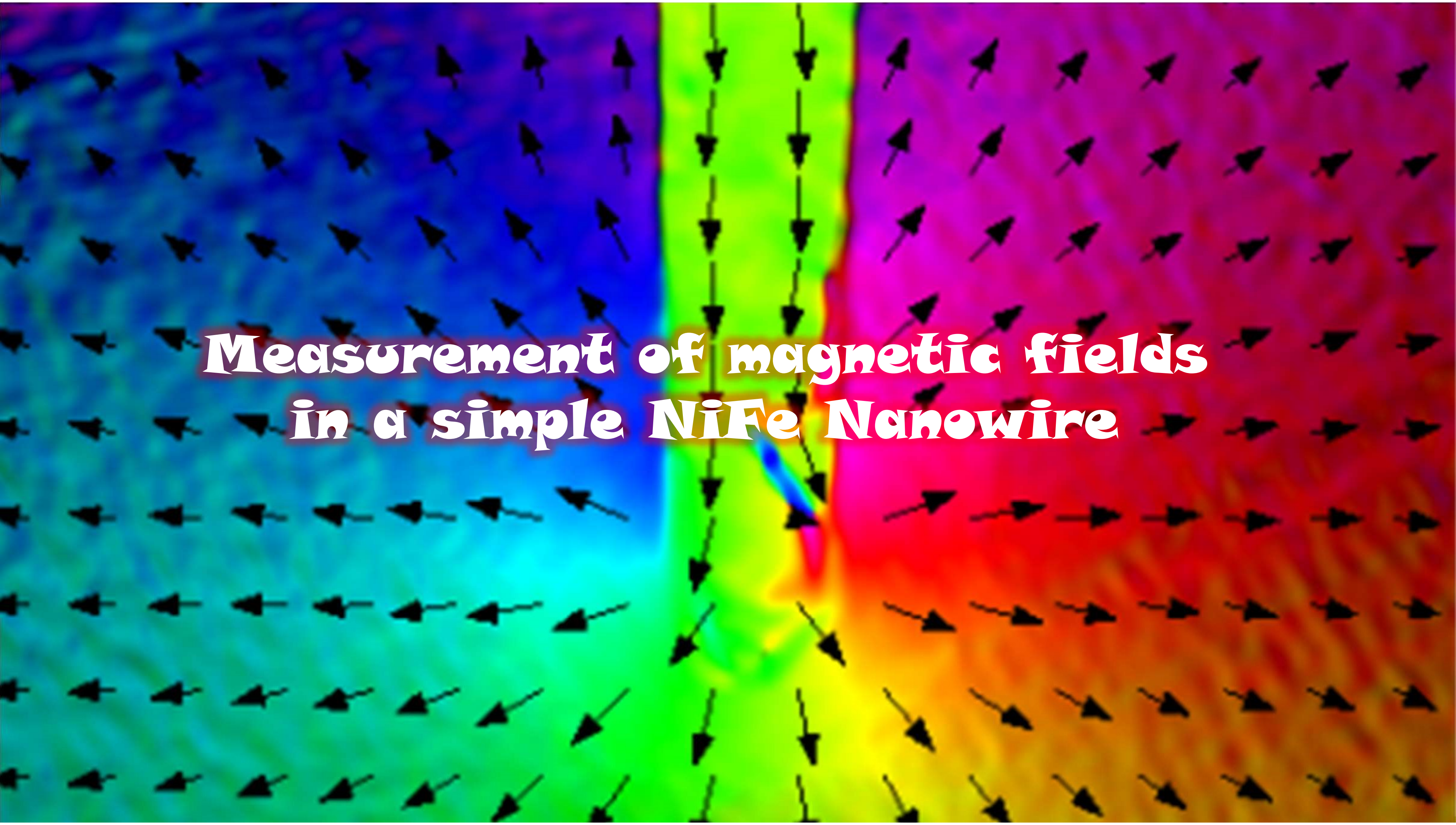
Maximizing phase information

[1] V. V. Volkov & al., *Ultramicroscopy* 134, 175 (2013)
[2] V. Boureau & al., *Ultramicroscopy* 193, 52-63 (2018)

3. Application to strain mapping in SiGe/Si superlattices

- π phase shifting
- 32 holograms stacked

→ 1 nm resolution



Measurement of magnetic fields in a simple NiFe Nanowire

Quantitative electron holography on a NiFe Nanowire

$$\begin{aligned} I_{holo} &= |\Psi_0 + \Psi|^2 \\ &= {A_0}^2 + A^2 + 2\mu A_0 A \cos(2\pi \overrightarrow{q_c}.\vec{r} + \varphi) \end{aligned}$$

Phase reconstruction (Fourier routine) → $\boxed{\varphi = \varphi^{MIP} + \varphi^{E} + \varphi^{M}}$

Mean Inner Potential

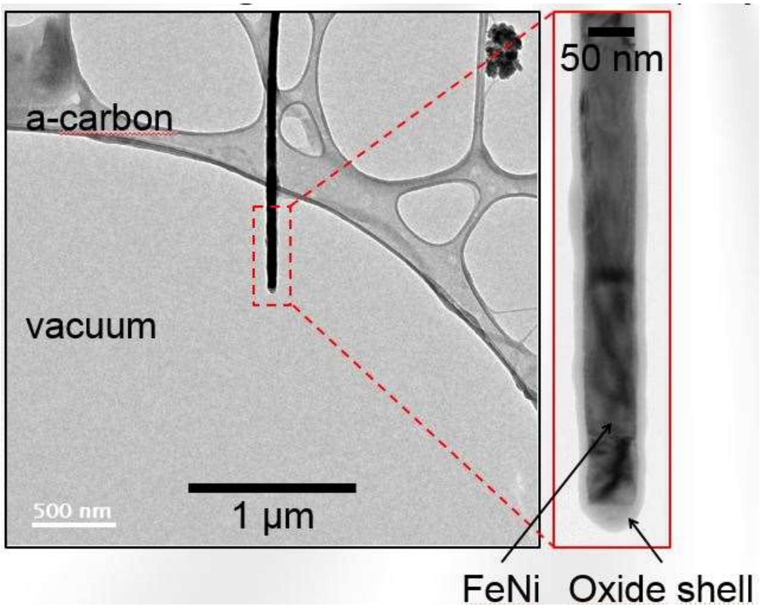
$$\varphi^{MIP} = C_E V_0 t$$

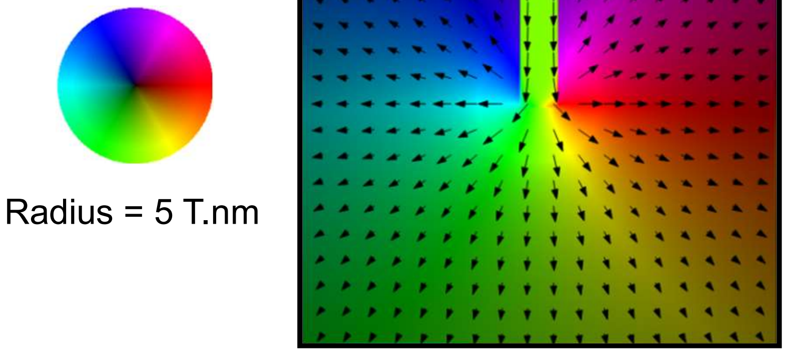
Electric

$$\varphi^{E} = C_E \int V dz$$

Magnetic

$$\varphi^{M} = -\frac{e}{\hbar} \int A_z dz$$

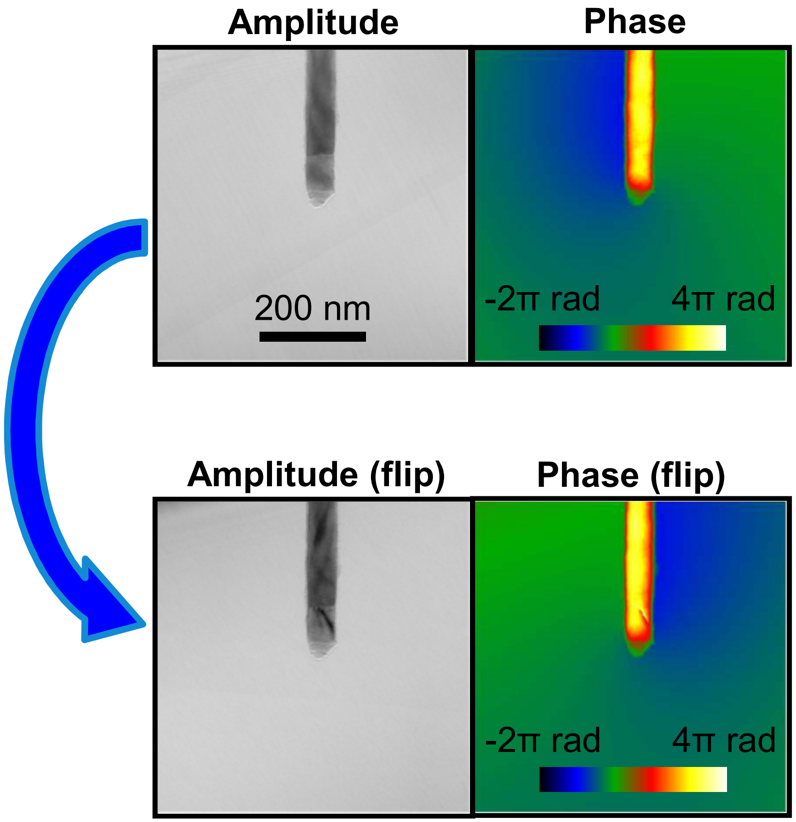




Magnetic induction: modeling

Quantitative electron holography on a NiFe Nanowire

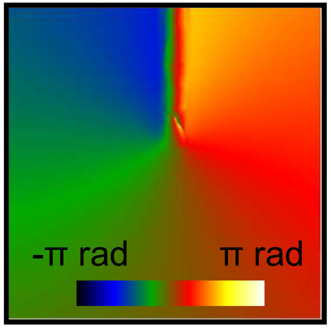
Reverse sample in the TEM
or
Reversing the field



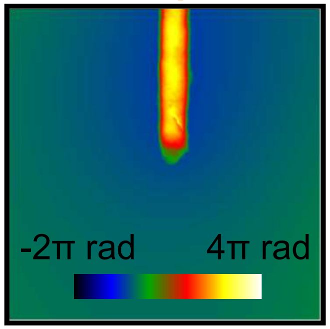
- Difference

- Sum

Magnetic phase



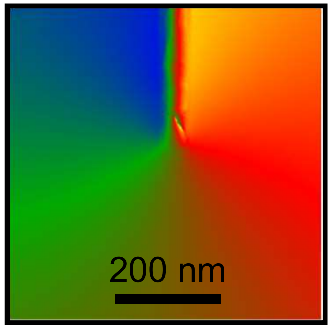
Electric phase



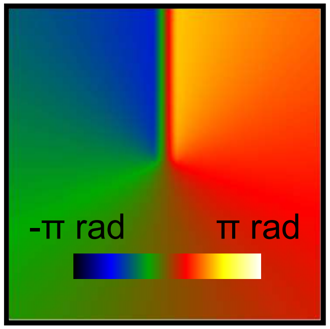
Sensitivity = 2π/1150 rad
Resolution = 3.5 nm

Quantitative electron holography on a NiFe Nanowire

Phase: holo

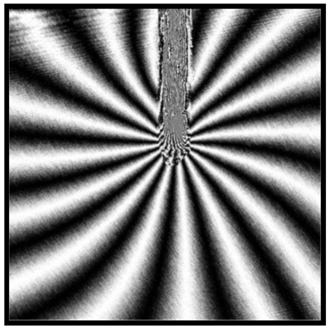


Phase: modeling

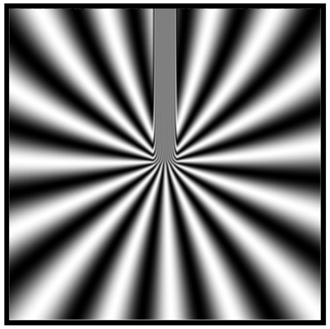


Magnetization of the FeNi alloy (0.9 T) calibrated with holography

Cos(30*phase): holo



Cos(30*phase): modeling



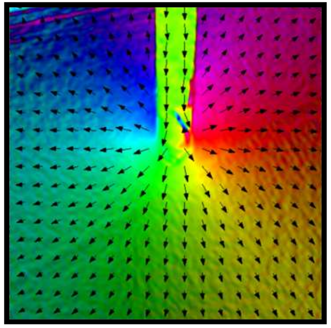
Very good agreement with modeling

Magnetic induction

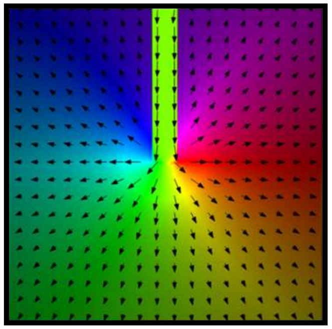
$$\vec{B} = \vec{\nabla} \times \vec{A}$$

Validation of the method !

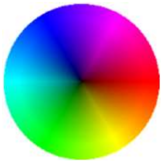
Magnetic induction: holo



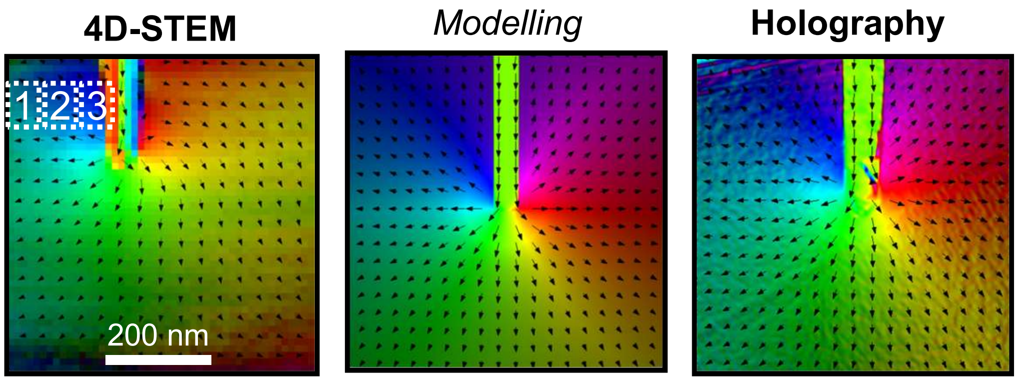
Magnetic induction: modeling

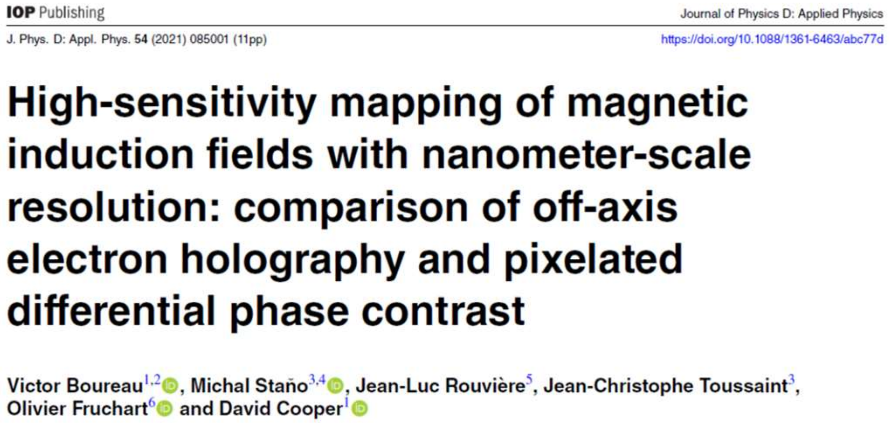


Radius = 5 T.nm



Comparison of pixelated STEM and Holography in the FEI Titan Ultimate




IOP Publishing
Journal of Physics D: Applied Physics
J. Phys. D: Appl. Phys. 54 (2021) 085001 (11pp)
https://doi.org/10.1088/1361-6463/abc77d

High-sensitivity mapping of magnetic induction fields with nanometer-scale resolution: comparison of off-axis electron holography and pixelated differential phase contrast

Victor Boureau[1,2], Michal Staňo[3,4], Jean-Luc Rouvière[5], Jean-Christophe Toussaint[3], Olivier Fruchart[6] and David Cooper[1]

- ***Electron holography vs pixelated STEM for magnetic imaging:*** much better sensitivity !!

  ✓ OK for < 1 µm FoV specimens

  ☺ **Quick data acquisition** required (<10 min)

  ☺ Image aligment required but **easy with our method**

  Spatial resolution ≥ 1 nm

- ***4D STEM issues:***

  ☹ Dynamic charging.

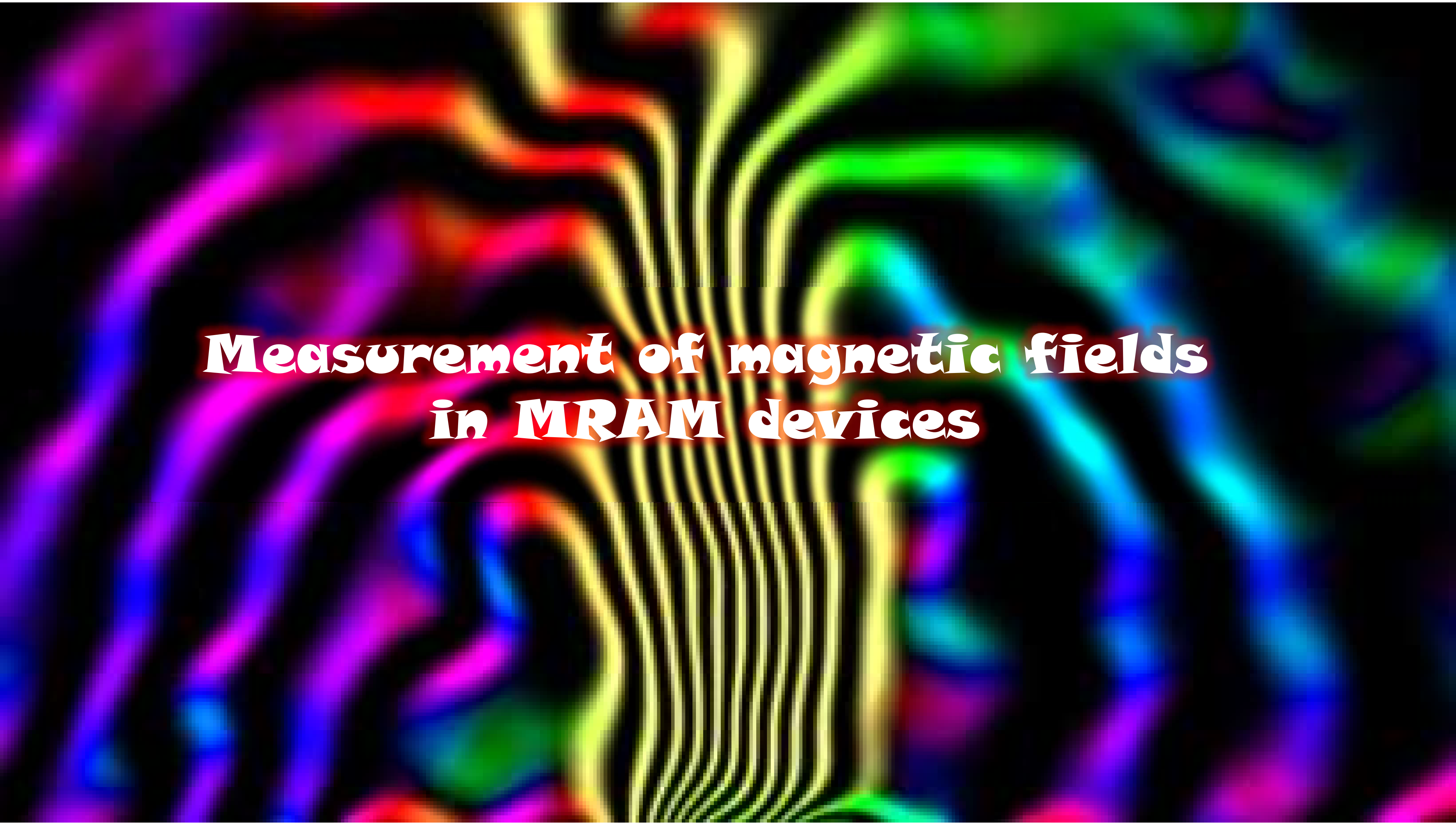
  ☹ Artefacts from the tails of the electron probe.

  ☹ Data processing is long (→ LiberTEM).

  ☹ Poor diffraction contrast with convergent beam

  4D STEM + objective lens off:
  Best resolution = 10 nm

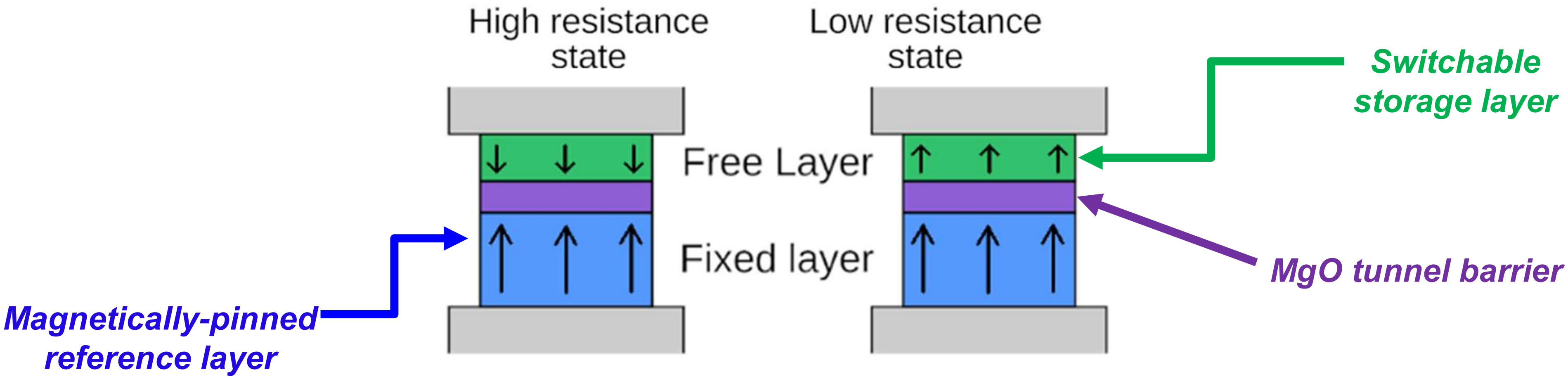
Measurement of magnetic fields in MRAM devices



CMOS-compatible magnetic data storage

- Magnetic tunnel junctions (MTJ)
- **STT-RAM devices:** reduce MTJ size for enhancing bit density → thermal stability issues

➡ *Increase free layer thickness*

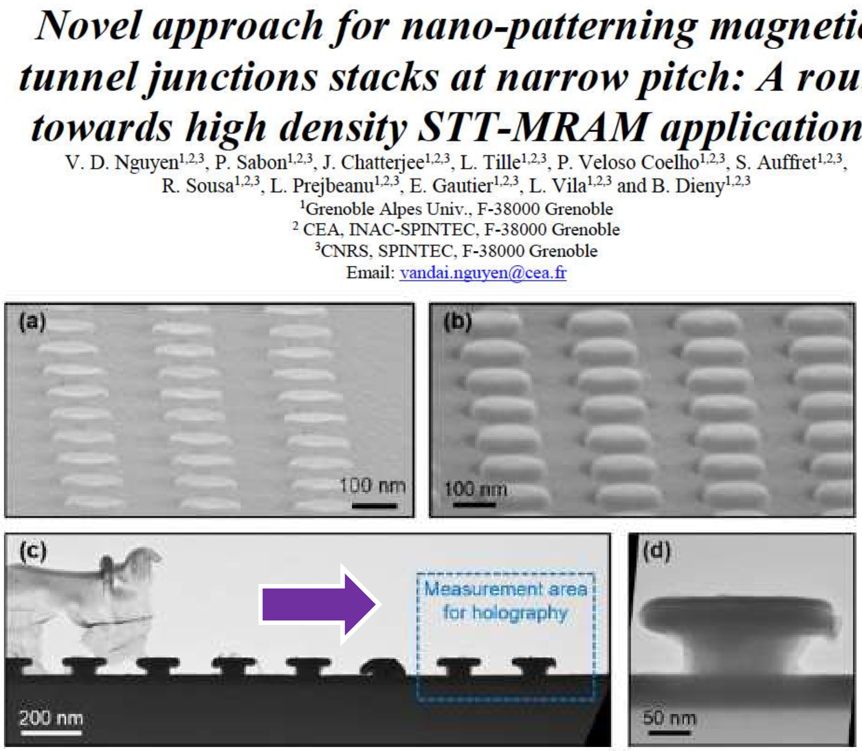


Switchable storage layer

MgO tunnel barrier

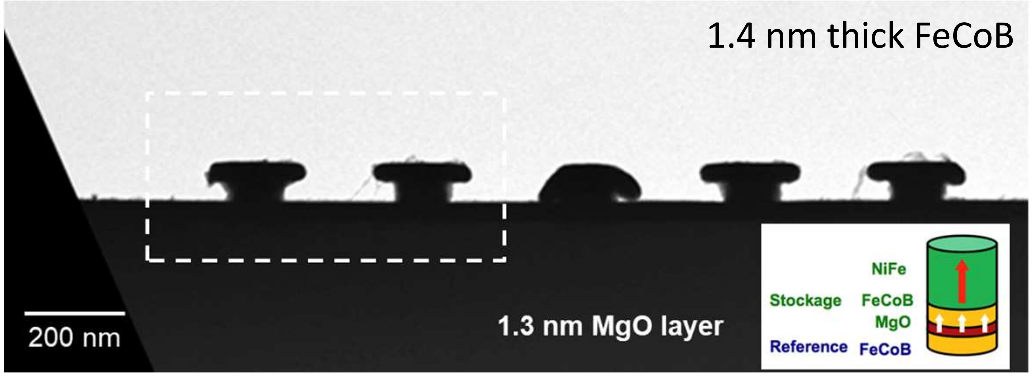
Magnetically-pinned reference layer

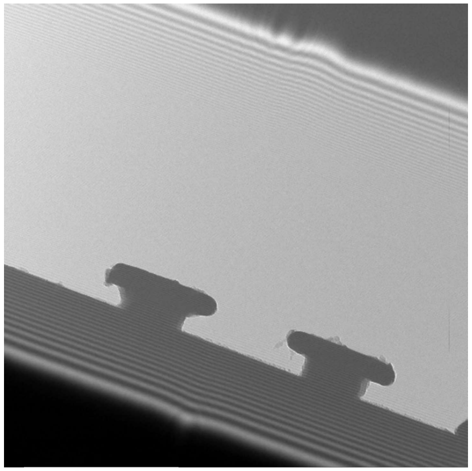
Magnetic fields mapping in CMOS compatible STT-MRAM test pillars



Novel approach for nano-patterning magnetic tunnel junctions stacks at narrow pitch: A route towards high density STT-MRAM applications

V. D. Nguyen[1,2,3], P. Sabon[1,2,3], J. Chatterjee[1,2,3], L. Tille[1,2,3], P. Veloso Coelho[1,2,3], S. Auffret[1,2,3],
R. Sousa[1,2,3], L. Prejbeanu[1,2,3], E. Gautier[1,2,3], L. Vila[1,2,3] and B. Dieny[1,2,3]
[1]Grenoble Alpes Univ., F-38000 Grenoble
[2] CEA, INAC-SPINTEC, F-38000 Grenoble
[3]CNRS, SPINTEC, F-38000 Grenoble
Email: vandai.nguyen@cea.fr





Measuring thermal stability with holography

Behaviour of magnetic field ?

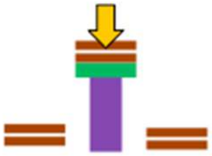
Inter-pillars interactions ?

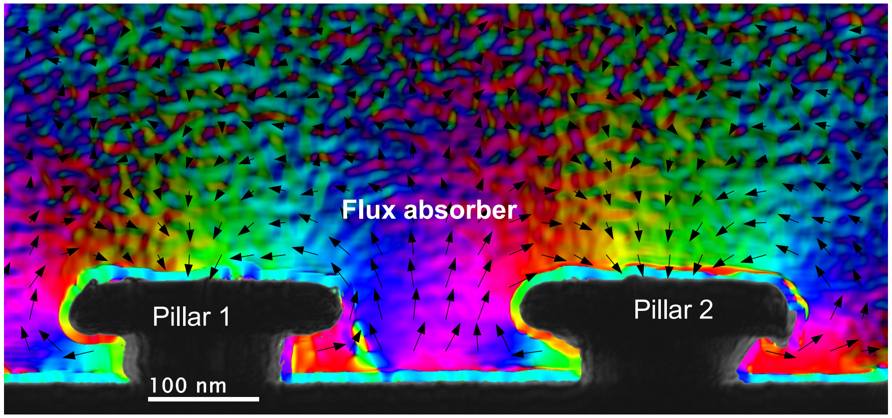
→ ***Expecting low signals***

- 32 holograms, 8s each
- Magnetization reversed w/ objective lens
- Alignment with Holoview software

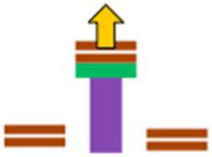
Mapping the magnetic induction in MRAM devices

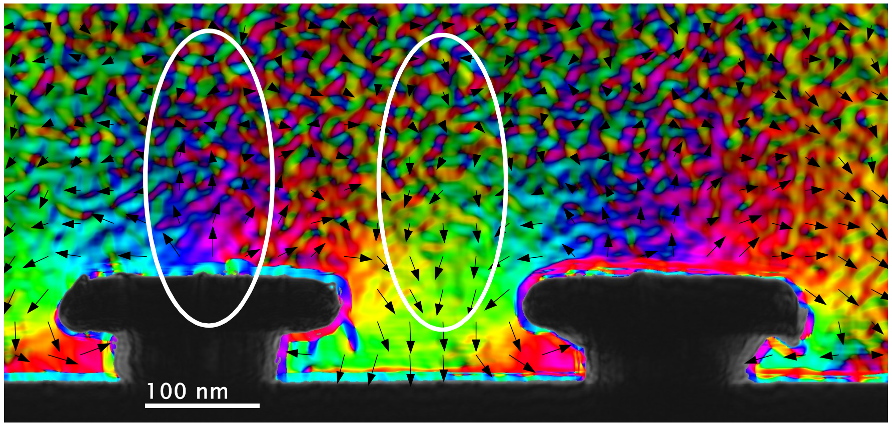
After **negative** out-of-plane saturation





After **positive** out-of-plane saturation





Interestingly, the magnetization of the deposit in the trenches is coupled antiparallel to the one on top of the neighboring dot.

→ **Flux absorber**

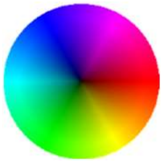
Excellent signal to noise in the phase image

High-quality magnetic induction mapping

Magnetic induction

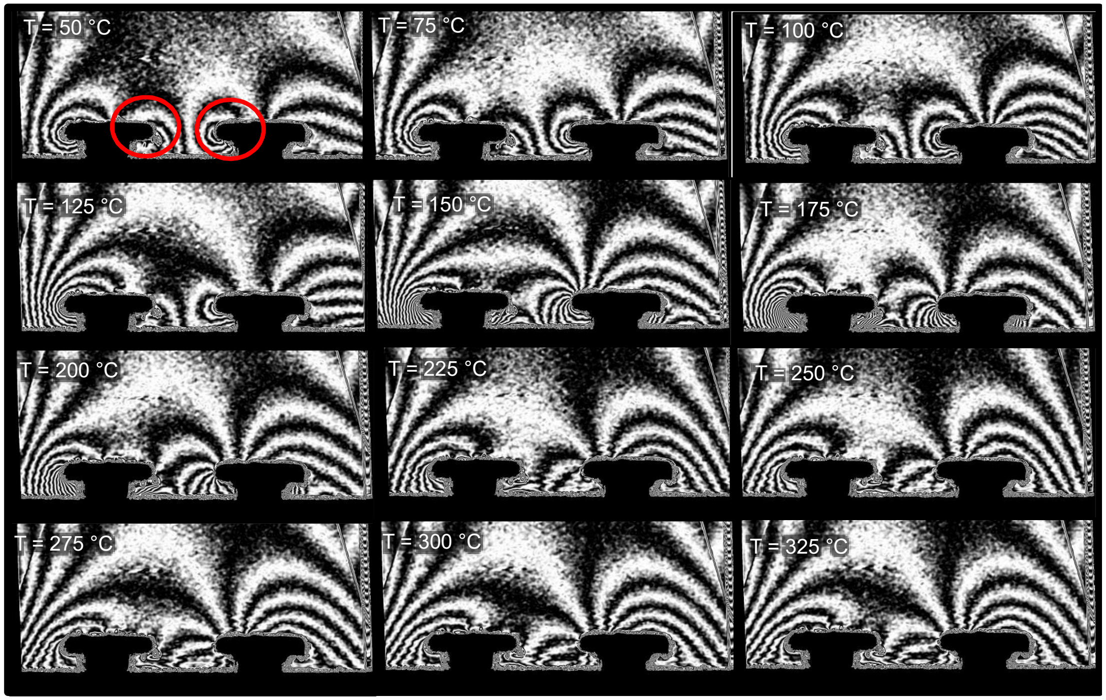
$$\vec{B} = \vec{\nabla} \times \vec{A}$$

Radius = 5 T.nm

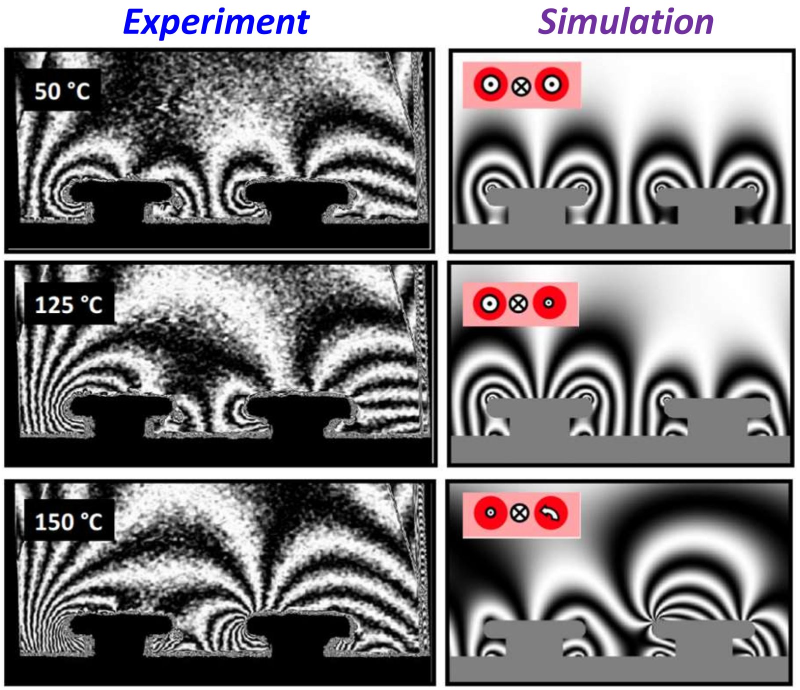


V. Boureau *et al.*, *Nanoscale* **12**, 17312–8 (2020)

Thermal behavior of MRAM devices



Specimen heated up to 325°C *in situ* in the TEM



To show better the changes we use contour maps (Cosine φ)

- Big change between 125 and 150°C → switch in direction of magnetization?
- Smooth change between 175 and 225°C → damping of magnetization?

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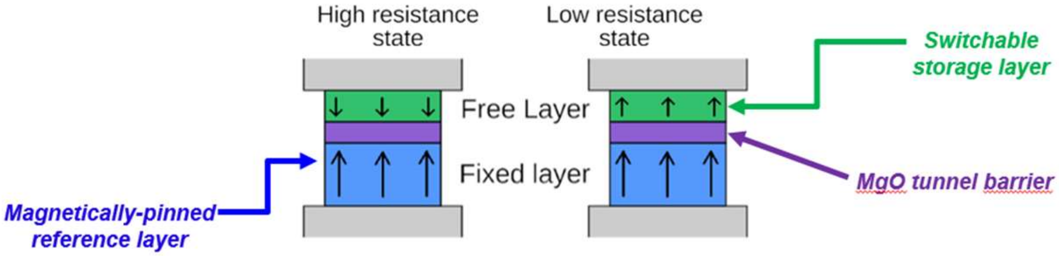
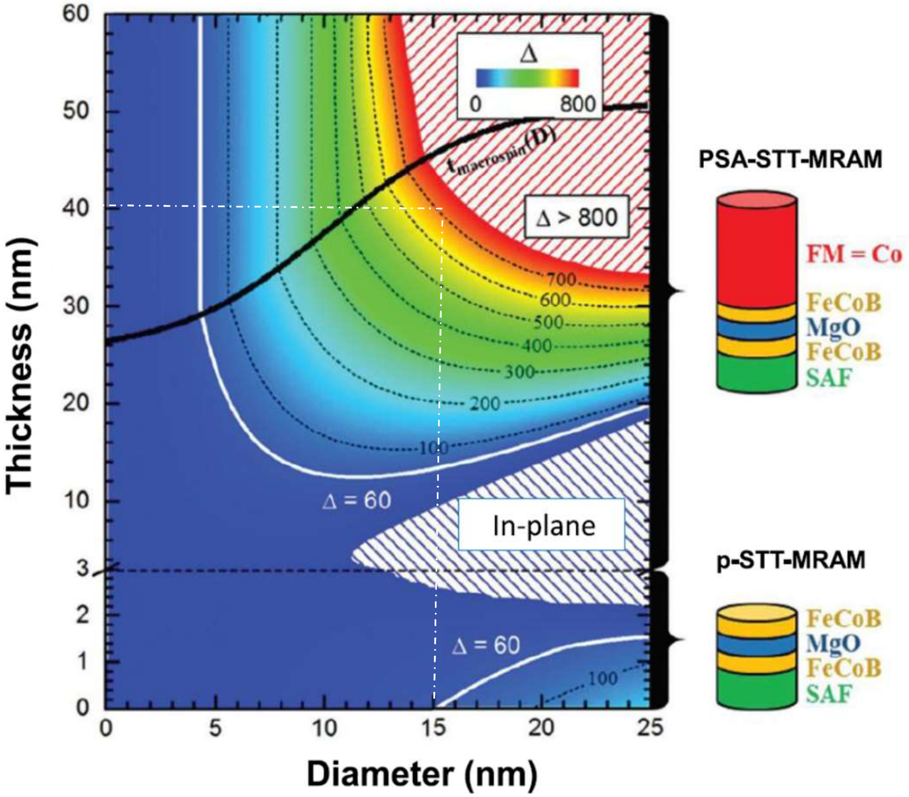
An electron holography study of perpendicular magnetic tunnel junctions nanostructured by deposition on pre-patterned conducting pillars

V. Boureau, [a] V. D. Nguyen,[b] A. Masseboeuf,[b] A. Palomino,[b] E. Gautier,[b] J. Chatterjee, [b] S. Lequeux, [b] S. Auffret,[b] L. Vila,[b] R. Sousa,[b] L. Prejbeanu,[b] D. Cooper[a] and B. Dieny *

Stability of perpendicular STT-MRAM

- Conventional perpendicular STT-MRAM vs ***perpendicular shape anisotropy (PSA)* STT-MRAM**
- Δ = magnetic stability, as high as possible for use in MRAM
- 15 nm diameter pillar → ***thick FeCoB storage layer***





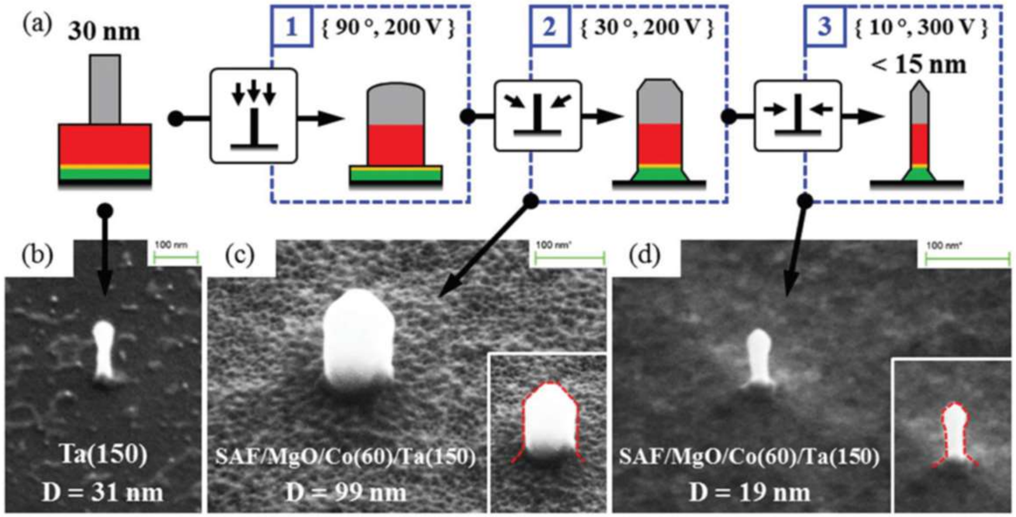
Stability of perpendicular shape anisotropy STT-MRAM

A highly thermally stable sub-20 nm magnetic random-access memory based on perpendicular shape anisotropy

Nanoscale

PAPER

N. Perrissin,[a] S. Lequeux,[a] N. Strelkov,[a,b] A. Chavent,[a] L. Vila,[a] L. D. Buda-Prejbeanu,[a] S. Auffret,[a] R. C. Sousa,[a] I. L. Prejbeanu[a] and B. Dieny*[a]



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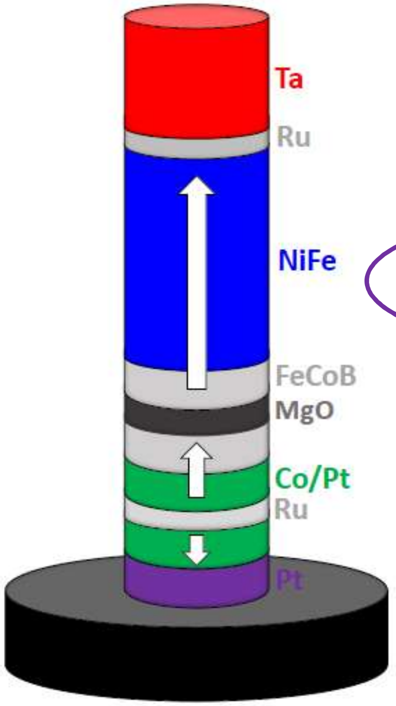


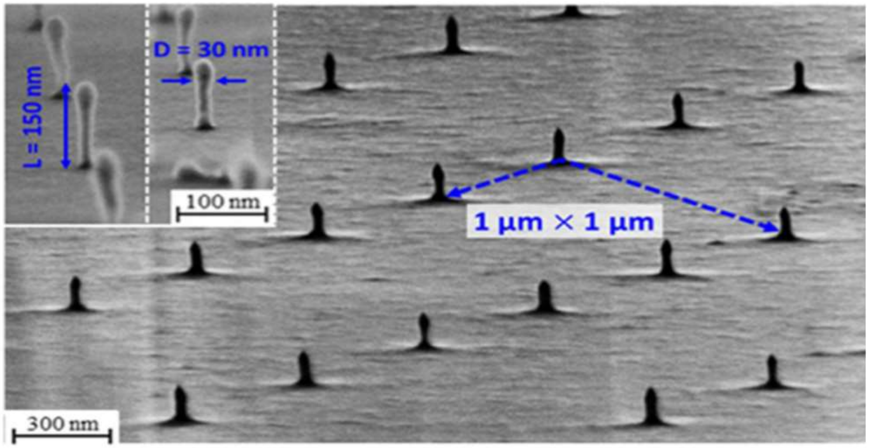
Main Objectives

1) New methodology of sample preparation
2) 20 nm diameter PSA-STT-MRAM devices by electron holography
3) Show the thermal stability and effect of annealing.

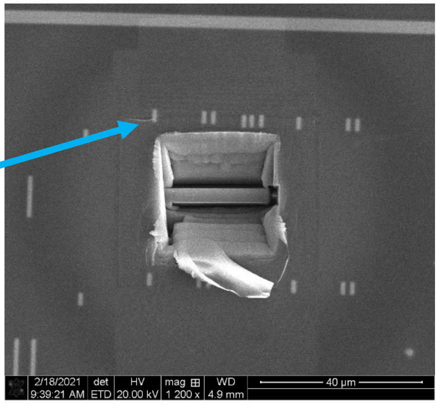
Often indirect measurements

Magnetoresistance
+ micromagnetic modelling

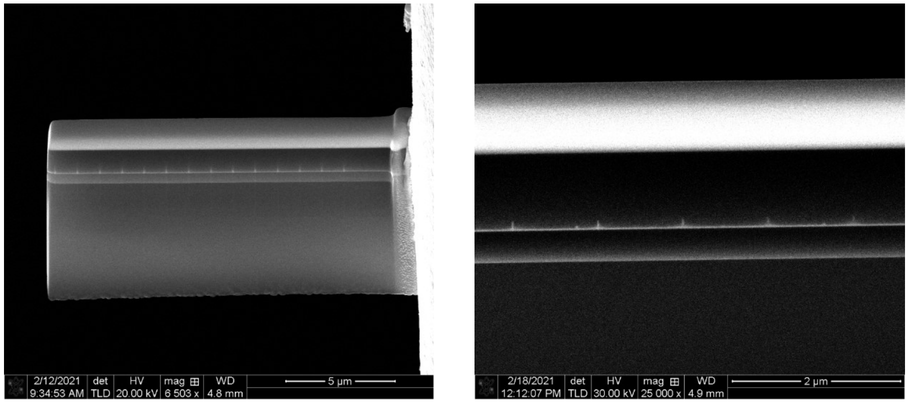
Sample preparation for TEM analysis



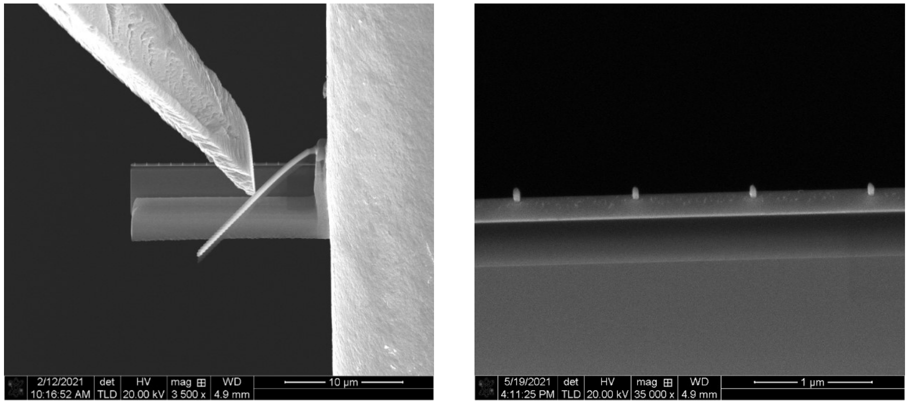
To prevent ion-beam damage, a ***protective layer of organic resin*** is spin-coated on the nano-pillar array



Nano-pillars are visible when milling lamella to ~ 300 nm

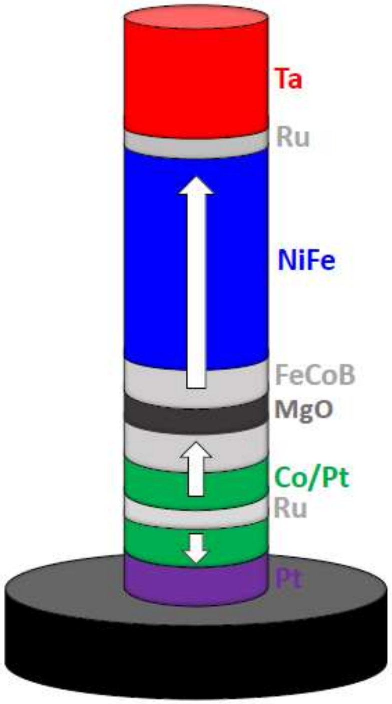


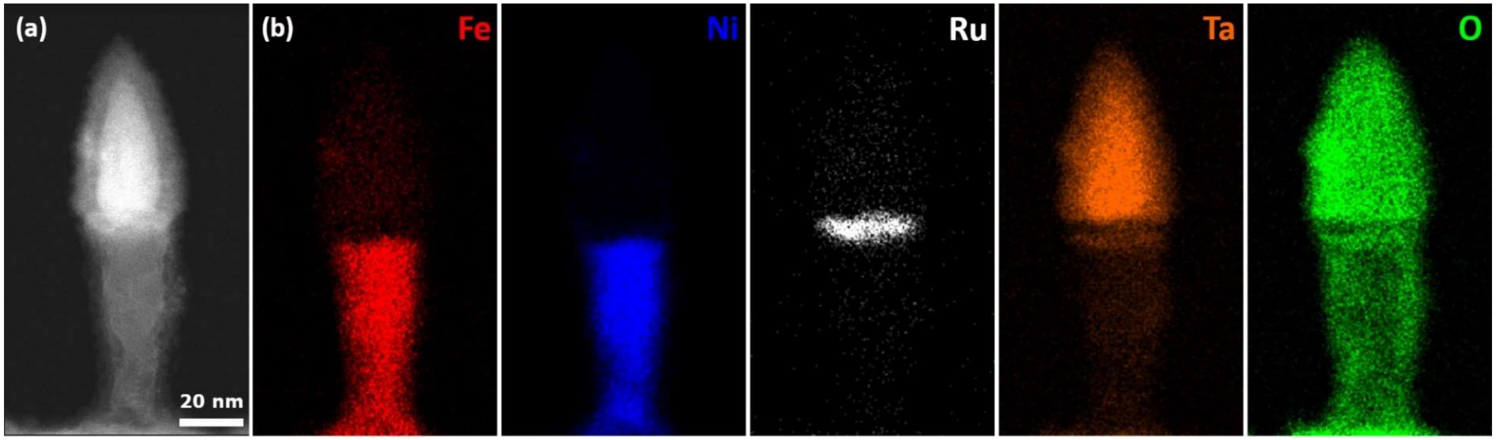
Plasma etching and manipulator remove the resin and Pt

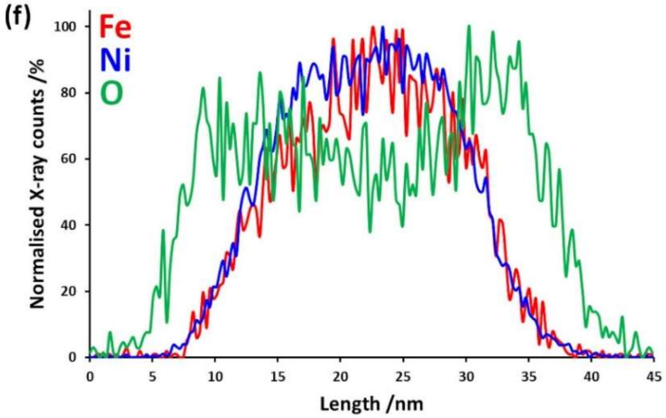


Chemical and structural characterisation

Pillars integrity from chemical analysis in the TEM

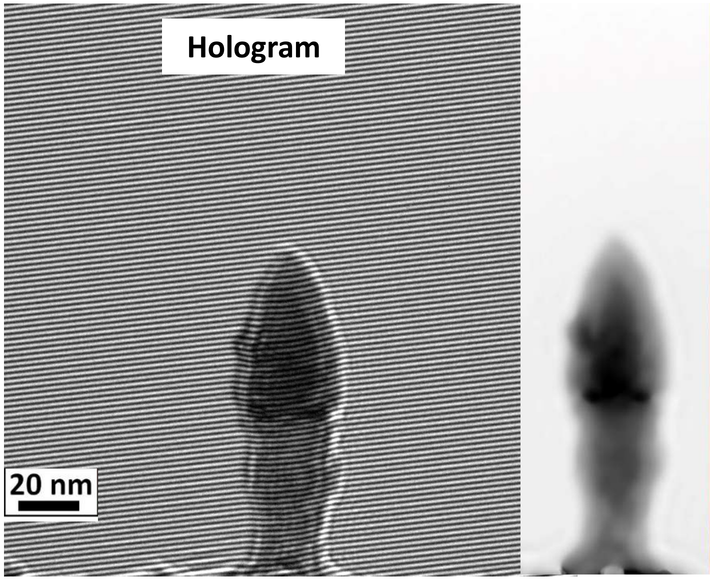


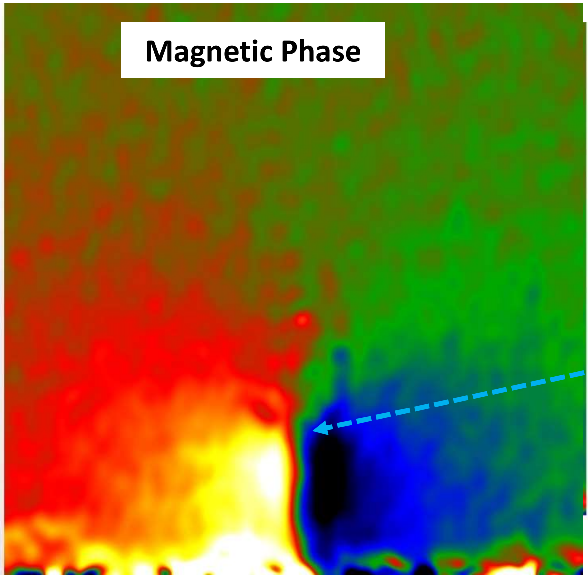


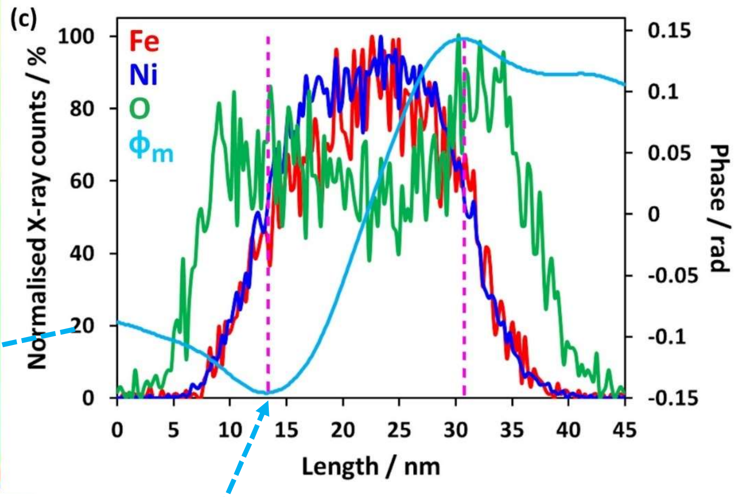


S. Lequeux *et al.*, *Nanoscale* **12,** 6378–6384 (2020)

Electron holography and magnetic measurements







- 16 holograms
- 8s each

Clear magnetic signal !

Electrostatic component of MIP:
Dimensional details.

$$|\vec{B}| = \frac{\hbar \Delta\boldsymbol{\phi}_m}{e\pi r^2}$$

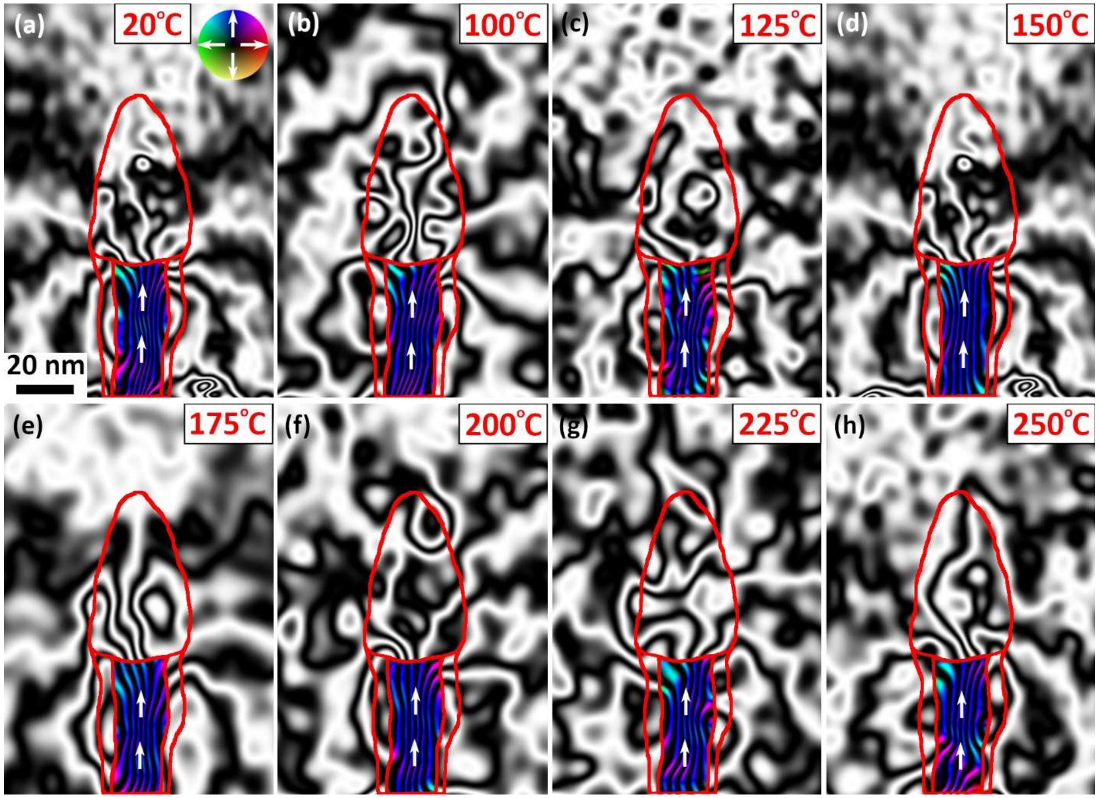
$$|\vec{B}| = \frac{\hbar * 0.29\text{rad}}{e\pi(8.6nm)^2}$$

$$|\vec{B}| = 0.82\ \text{T}$$

$|\vec{B}|$ *for NiFe is normally* ~ 0.8 – 1.0 T

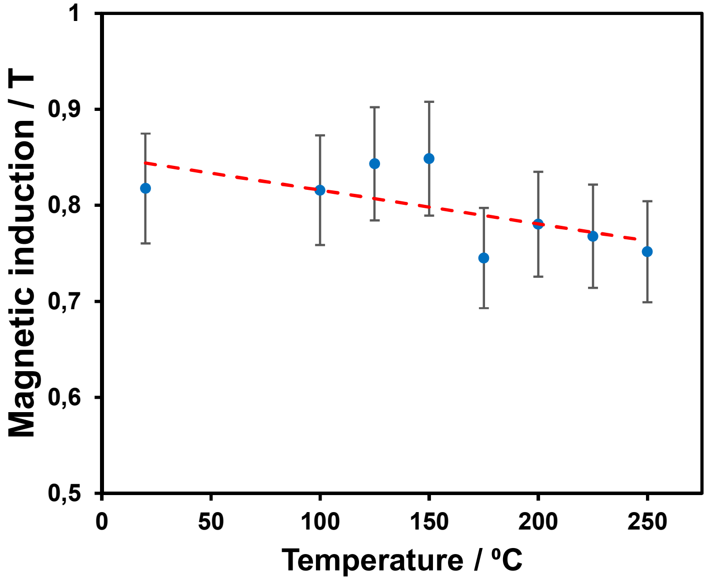
Direct imaging and analysis of thermal stability

$|\vec{B}|$ maps reveal ⊥ shape anisotropy in NiFe segment is stable with temperature

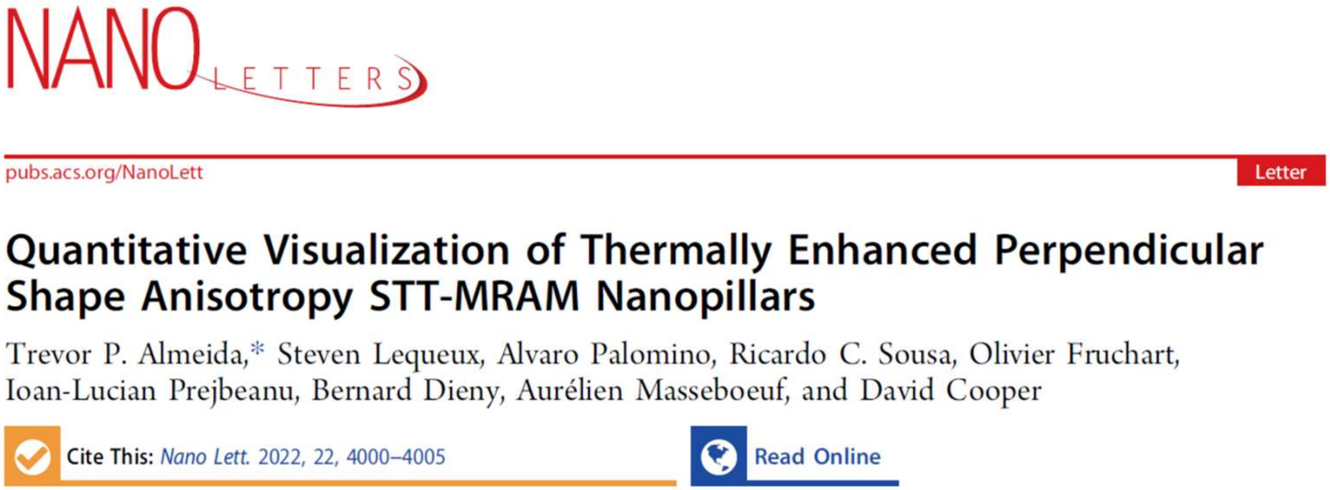


For construction of maps, the cosine is amplified by x150.

Measured $|\vec{B}|$ as a function of temperature



S. Lequeux, T. Almeida *et al.*, 2021 *IEEE International Memory Workshop (IMW)*, pp. 1-4 (2021), doi: 10.1109/IMW51353.2021.9439609.


NANO LETTERS

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Letter

Quantitative Visualization of Thermally Enhanced Perpendicular Shape Anisotropy STT-MRAM Nanopillars

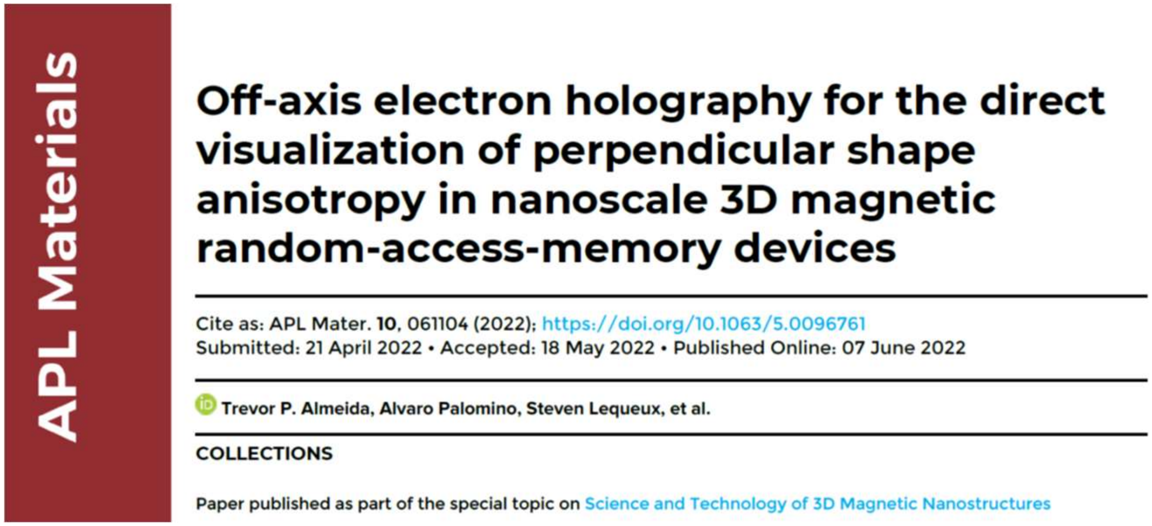
Trevor P. Almeida,* Steven Lequeux, Alvaro Palomino, Ricardo C. Sousa, Olivier Fruchart, Ioan-Lucian Prejbeanu, Bernard Dieny, Aurélien Masseboeuf, and David Cooper

Cite This: *Nano Lett.* 2022, 22, 4000–4005

Read Online

For a discussion of the PSA-STT-MRAM

For a discussion of the holography methods


APL Materials

Off-axis electron holography for the direct visualization of perpendicular shape anisotropy in nanoscale 3D magnetic random-access-memory devices

Cite as: APL Mater. **10**, 061104 (2022); https://doi.org/10.1063/5.0096761
Submitted: 21 April 2022 • Accepted: 18 May 2022 • Published Online: 07 June 2022

Trevor P. Almeida, Alvaro Palomino, Steven Lequeux, et al.

COLLECTIONS

Paper published as part of the special topic on Science and Technology of 3D Magnetic Nanostructures

Conclusions

- *Off-axis electron holography has been demonstrated as a **quantitative method** for measuring the magnetic fields with **nm-scale resolution.***

- *In an FEI microscope, **holography is our method of choice <u>for magnetic and electrostatic imaging</u>.***

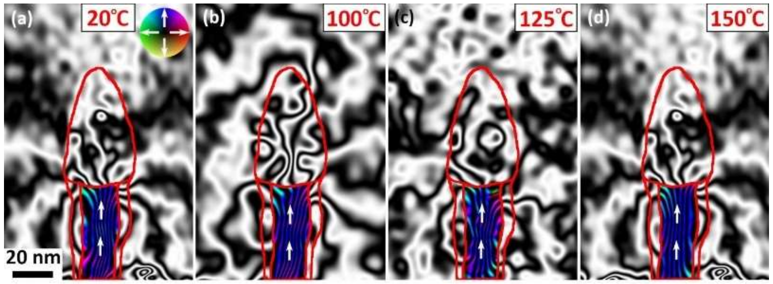
***<u>For strain mapping</u> pixelated STEM (PED) is preferred** (as the diffracted beams have larger displacements compared to the transmitted beam).*

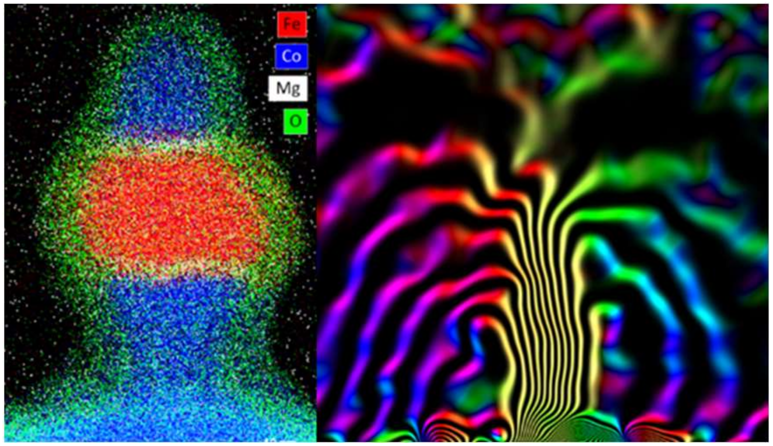
- ***Improvements in methods means that electron holography is today capable of measuring the magnetic fields in 20 nm diameter devices, revealing their PSA.***

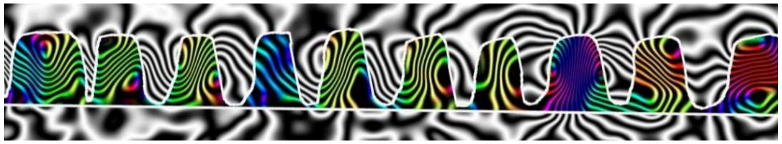
- ***In-situ annealing of 20 nm NiFe nano-pillars confirms their PSA is stable up to 250°C.***

- ***Already there are many other examples of magnetic holography being used to solve materials issues at LETI / Spintec not shown here.***

Today, electron holography is a technique that is applicable to an industrial environment using suitable set-up & software







PlatForm for NanoCharacterisation

Characterisation of advanced materials and components supporting disruptive innovation

This unique tool in the world, supported by three **CEA** Grenoble institutes (**CEA-Leti**, **CEA-Irig** and **CEA-Liten**), aims to develop new characterisation techniques for micro and nanotechnologies, nanomaterials, materials for energy, etc. ; and to carry out the characterizations needed for the advancement of the programs of the CEA and its academic or industrial partners. The expertise of the PFNC covers eight competence centres, including one dedicated to sample preparation, a key step for observations at the nanoscale.


MAGNETIC RESONANCE

Liquid and solid state NMR, DNP


SCANNING PROBE MICROSCOPY

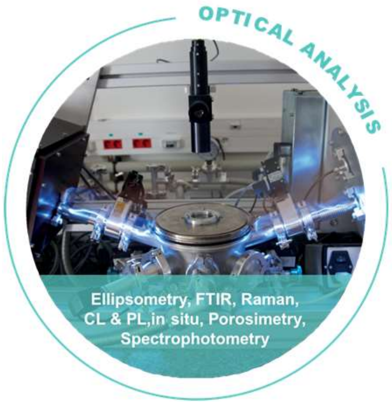
AFM, SSRM, SCM, KFM, Nano-DMA


ION BEAM ANALYSIS

SIMS, TOF-SIMS, RBS, Atomic Probe Tomography


SURFACE ANALYSIS

XPS, HAXPES, XPEEM, Auger nanoprobe, synchrotron


OPTICAL ANALYSIS

Ellipsometry, FTIR, Raman, CL & PL,in situ, Porosimetry, Spectrophotometry


X-RAY ANALYSIS

(HR)XRD, XRR, XRF, SAXS, WAXS, operando, tomography, synchrotron


SAMPLE PREPARATION

FIB (Ga, Xenon), polishing, cleaving, cutting tools, chemistry…


ELECTRON MICROSCOPY

SEM, EBSD, TEM, HR-(S)TEM,EDX, EELS, in situ, P-NBED, 3D, Holography

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