

NV-center microscopy a versatile tool to study magnetism

Aurore Finco

Laboratoire Charles Coulomb
Team Solid-State Quantum Technologies (S2QT)

CNRS and Université de Montpellier, Montpellier, France

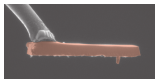
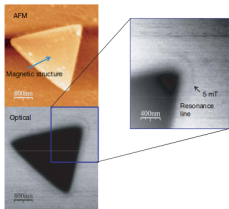
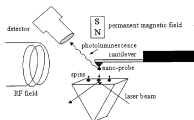


**UNIVERSITÉ DE
MONTPELLIER**

ITN-SPEAR, FTS5: Skyrmions, February 28th 2024, Hamburg

slides available at <https://magimag.eu>

A very short history



1997: First detection of single NV centers

 A. Gruber *et al. Science* 276 (1997), 2012

2004: Proposal of the scanning NV center magnetometer

 B. M. Chernobrod and G. P. Berman. *J. Appl. Phys.* 97 (2004), 014903

2008: First demonstration of scanning NV imaging

 G. Balasubramanian *et al. Nature* 455 (2008), 648–651

2012: All-diamond probes

 P. Maletinsky *et al. Nat. Nano.* 7 (2012), 320–324

2020: Commercial scanning NV microscopes available

Further reading

Reviews about NV center magnetometry:

- S. Hong *et al.* *MRS Bulletin* 38 (2013), 155–161
- L. Rondin *et al.* *Reports on Progress in Physics* 77 (2014), 056503
- F. Casola *et al.* *Nature Reviews Materials* 3 (2018), 17088
- A. Laraoui and K. Ambal. *Applied Physics Letters* 121 (2022), 060502
- Y. Xu *et al.* *Photonics Research* 11 (2023), 393–412
- A. Finco and V. Jacques. *APL Materials* 11 (2023), 100901

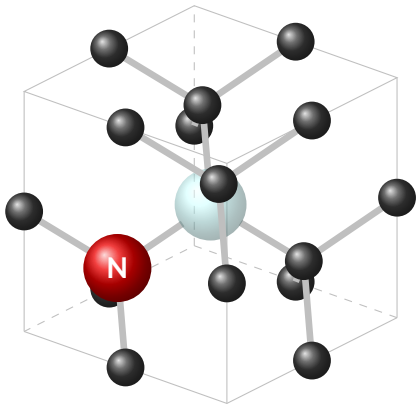
Outline

1. The NV center in diamond as a quantum sensor
2. Dismantling the scanning NV microscope
3. Quantitative ODMR experiments
 - Principle of the measurement
 - The need for a proper calibration
 - Example 1: analyzing domain walls
 - Example 2: the spin cycloid in bismuth ferrite
 - Example 3: van der Waals magnets
4. Taking a step back: PL quenching effects
 - Strong off-axis magnetic fields
 - Magnetic noise!
5. Relaxometry: sensing via the relaxation time
6. Coherent control of the NV center using spin waves
7. Going further: other sensors and sensing methods

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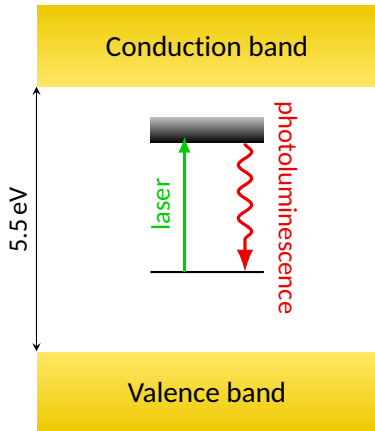
The NV center in diamond



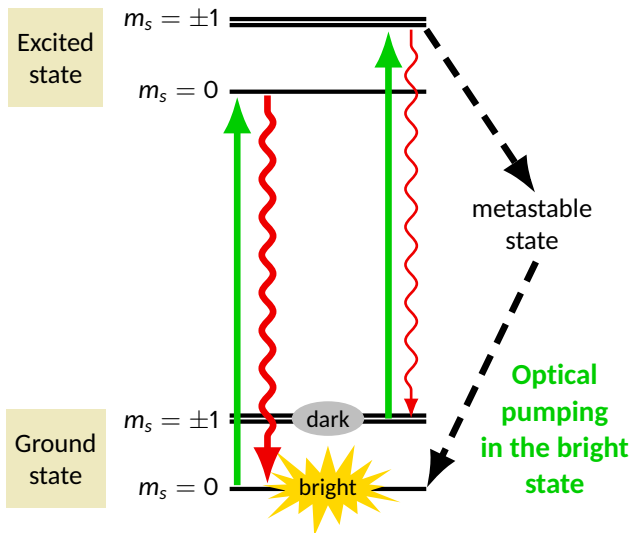
- Artificial atom: energy levels in the diamond bandgap
- Photostable defect
- Spin $S=1$
- Individual defects can be isolated/implanted
- Ambient conditions

Optical properties

Artificial atom
in the diamond band gap

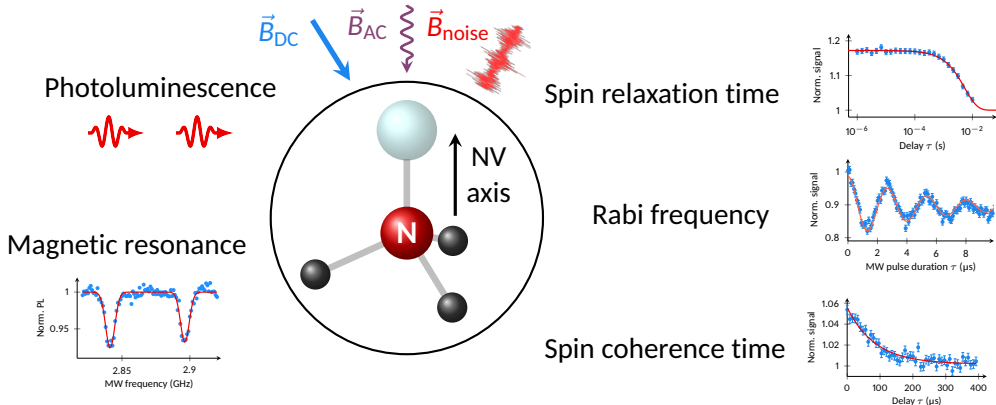


Spin dependent photoluminescence



The NV center as a quantum sensor

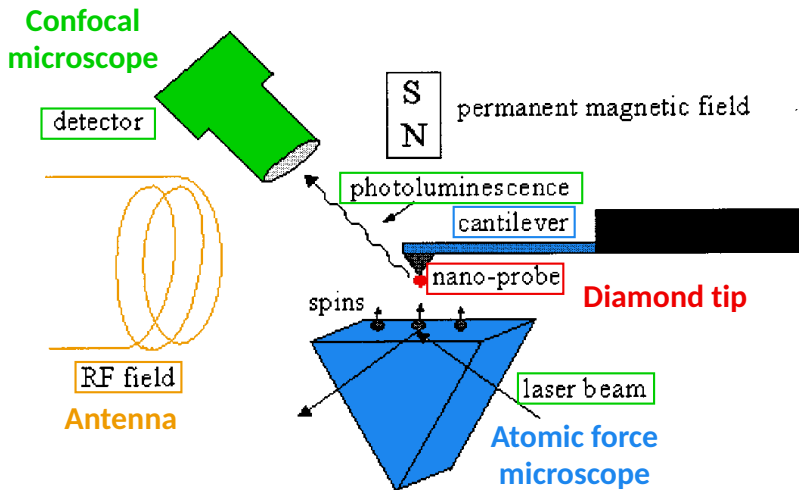
$$\mathcal{H}_{\text{gs}} = h \left[D_{\text{NV}} \hat{S}_z^2 + \gamma_{\text{NV}} \hat{\mathbf{S}} \cdot \vec{\mathbf{B}} \right] \text{ with } \begin{cases} D_{\text{NV}} \simeq 2.87 \text{ GHz} \\ \gamma_{\text{NV}} \simeq 28 \text{ GHz T}^{-1} \end{cases}$$



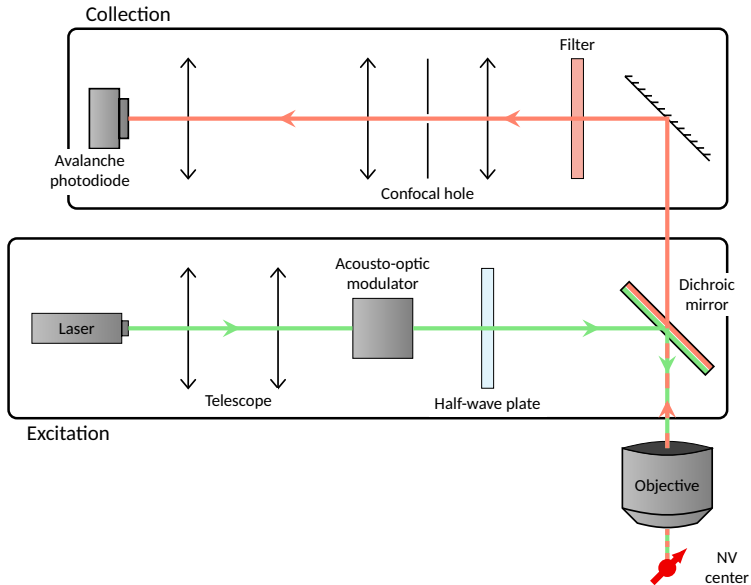
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Dismantling the scanning NV microscope



The confocal microscope



The atomic force microscope

Akiyama probes

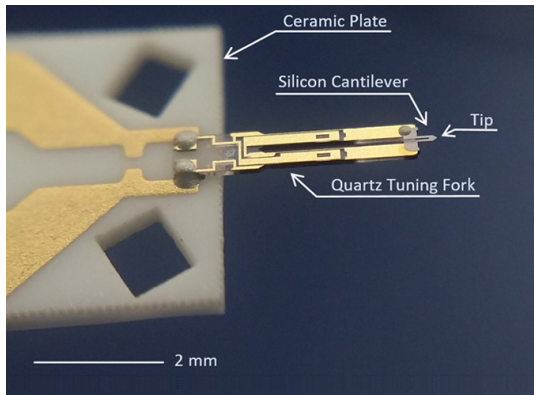


Image from www.akiyamaprobe.com

- The quartz tuning fork oscillates at about 32 kHz.
- The tip and cantilever are removed and the diamond tip glued on one of the arms.
- Excitation and reading of the mechanical resonance with piezo elements.
- Feedback loop onto the frequency or the amplitude of the mechanical resonance.

The diamond probes

All-diamond probes developed in 2012

 P. Maletinsky et al. *Nat. Nano.* 7 (2012), 320–324

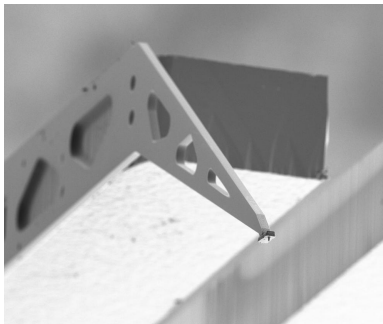


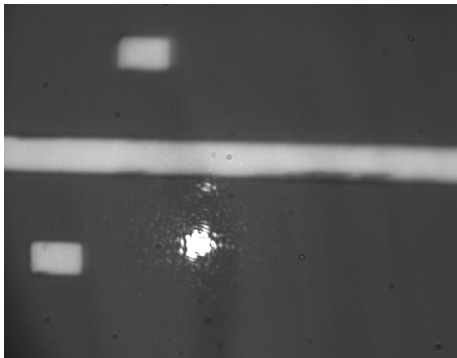
Image from www.qzabre.com

- High-purity diamond
→ Good coherence properties
- Hosting single NV centers at 50-100 nm from the sample surface
→ Spatial resolution of the microscope
- Usually made from (001) diamond
→ Tilted NV axis
- Diamond pillar as guide for the emitted light
→ Optimized signal collection

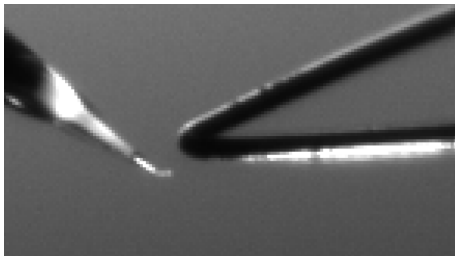
The microwave antenna

We need to deliver a microwave signal very close to the NV center. Several options:

Patterned gold line on the sample



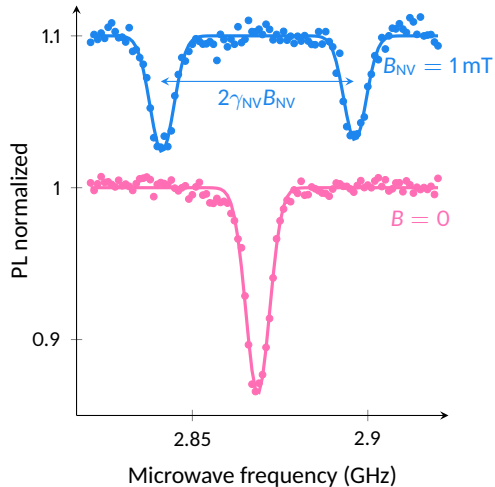
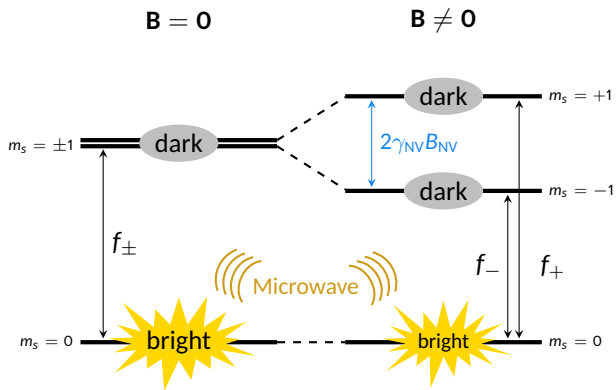
Use a wire, close to the tip



Outline

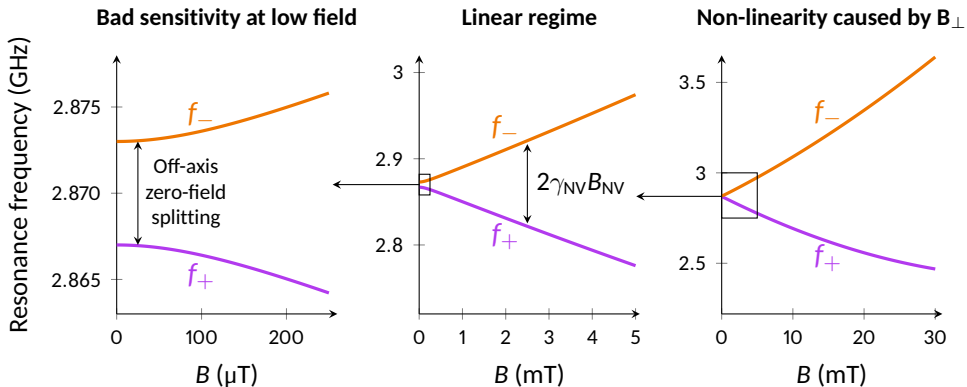
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Optically detected magnetic resonance (ODMR)



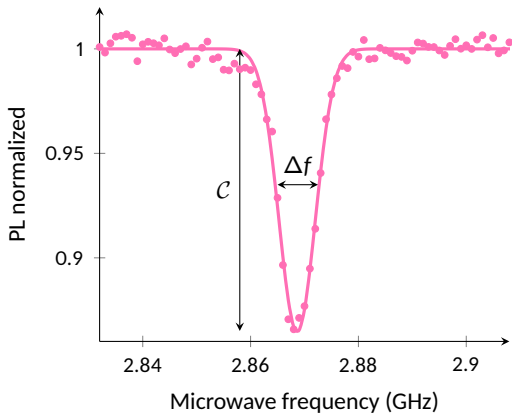
The different regimes

Calculation of f_- and f_+ , with a 45° angle between \vec{B} and the NV axis



- We apply a bias field roughly aligned with the NV axis (also to extract the sign of B_{NV})
- Quantitative measurements of very strong fields impossible

Magnetic field sensitivity



$$\eta_B = \frac{4}{3\sqrt{3}} \frac{\Delta f}{\gamma_{\text{NV}} C \sqrt{\mathcal{R}}}$$

- Linewidth Δf : tunable with the **microwave** power
 - Contrast \mathcal{C} : tunable with the **laser** and the **microwave** power
 - Off-resonance count rate \mathcal{R} : tunable with the **laser** power
- Optimal (P_{laser} , P_{MW}) values
→ Sensitivity: a few $\mu\text{T}/\sqrt{\text{Hz}}$

📄 A. Dréau et al. *Physical Review B* 84 (2011), 195204

Understanding stray field maps

$$\vec{B} = \mu_0 \vec{\nabla} \phi$$

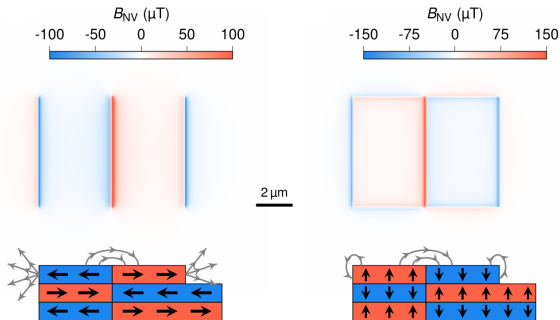
where ϕ is a scalar potential which verifies the Poisson equation:

$$\Delta \phi = \vec{\nabla} \cdot \vec{M}$$

Same equation as for the electrostatic potential V

$$\Delta V = -\frac{\rho}{\epsilon_0}$$

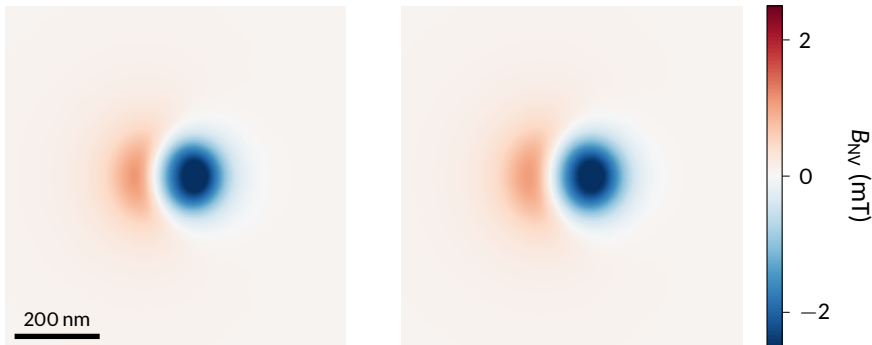
$\Rightarrow \vec{\nabla} \cdot \vec{M}$ can be seen as the magnetic field source



Field sources: edges, domain walls, skyrmions, etc.

We need to calibrate the NV flying distance!

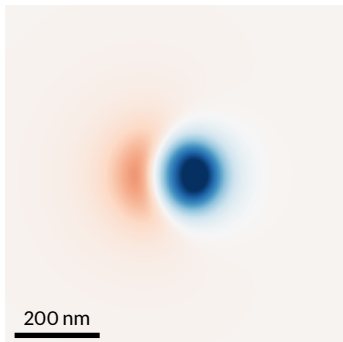
Simulated stray field maps from skyrmions of diameter 150 nm in a 0.5 nm thick film with $M_s = 1 \text{ MA m}^{-1}$



We need to calibrate the NV flying distance!

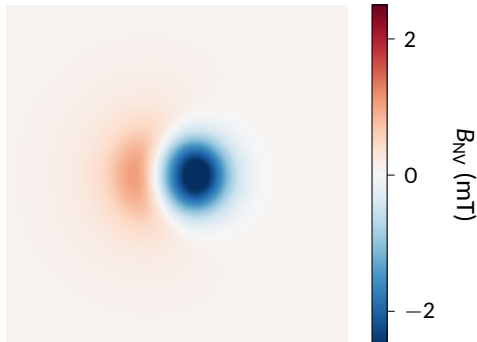
Simulated stray field maps from skyrmions of diameter 150 nm in a 0.5 nm thick film with $M_s = 1 \text{ MA m}^{-1}$

Clockwise Néel skyrmion



NV height: $d_{\text{NV}} = 50 \text{ nm}$
DW width: $w = 15 \text{ nm}$

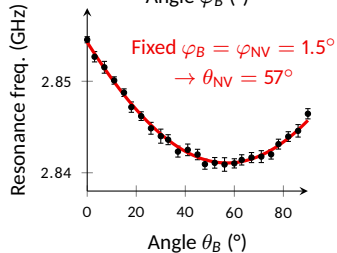
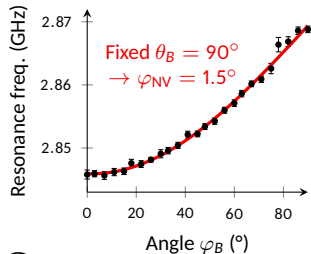
Counter clockwise Néel skyrmion



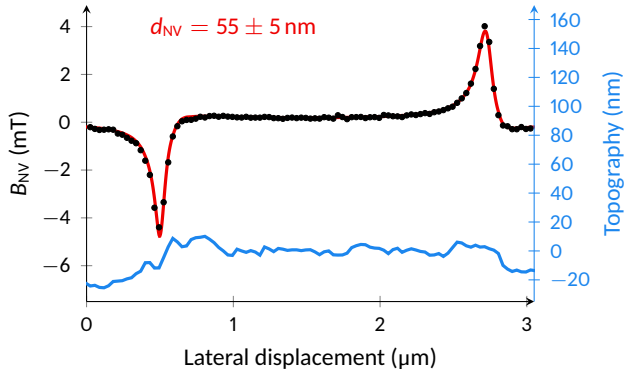
NV height: $d_{\text{NV}} = 80 \text{ nm}$
DW width: $w = 30 \text{ nm}$

The calibration procedure

1. Find the NV axis orientation



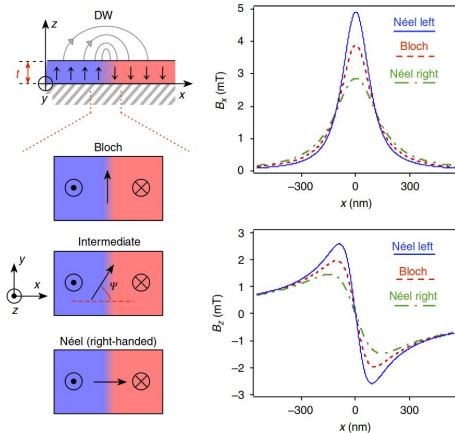
2. Find the NV height d_{NV} with a well-known ferromagnetic stripe



T. Hingant et al. *Phys. Rev. Appl.* 4 (2015), 014003

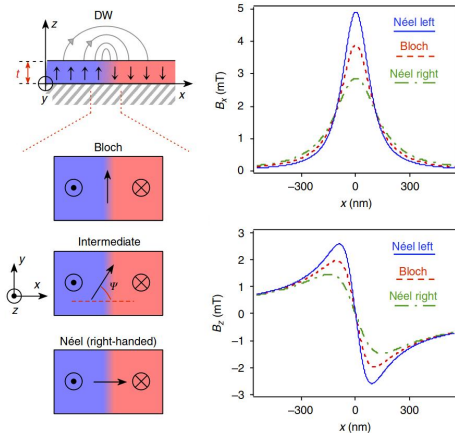
Example 1: analysis of domain walls

Analytical expression of the stray field

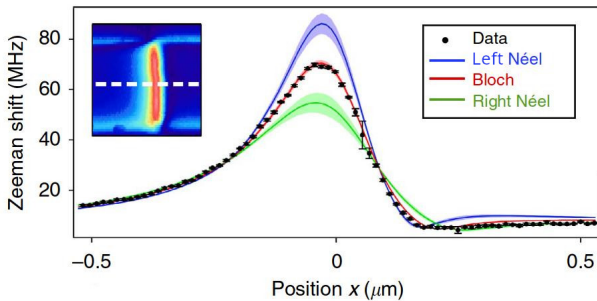


Example 1: analysis of domain walls

Analytical expression of the stray field



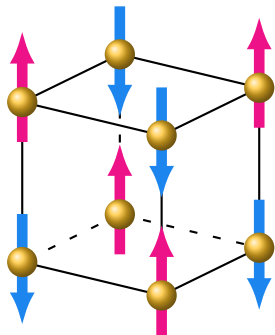
Ta/CoFeB/MgO stripe



→ With a precise calibration, we can determine the internal texture of the domain wall

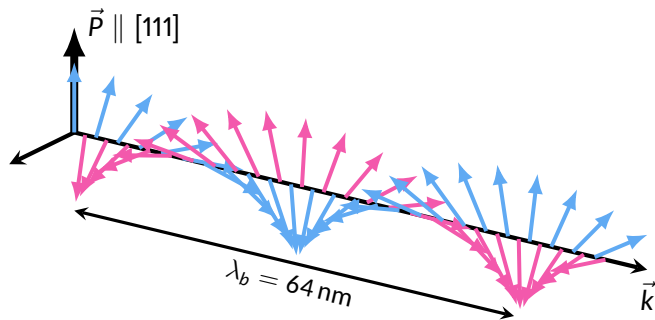
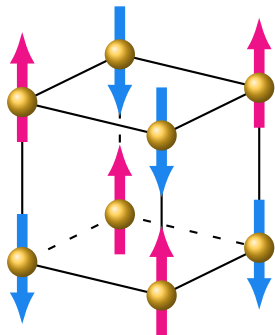
Example 2: the cycloid in bismuth ferrite

G-type antiferromagnet



Example 2: the cycloid in bismuth ferrite

G-type antiferromagnet

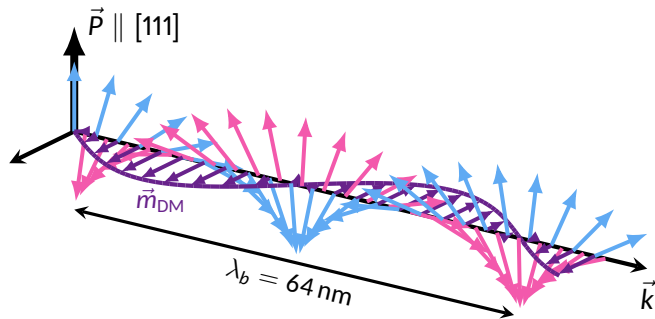
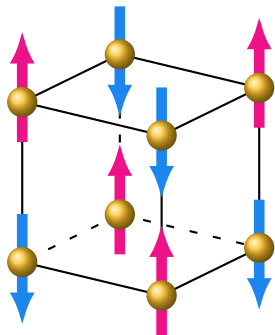


Fully compensated cycloid

→ **No stray field!**

Example 2: the cycloid in bismuth ferrite

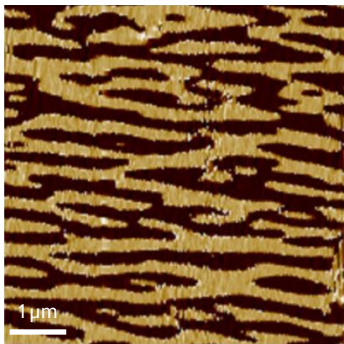
G-type antiferromagnet



Spin density wave
Weak uncompensated moment
→ **Small stray field**

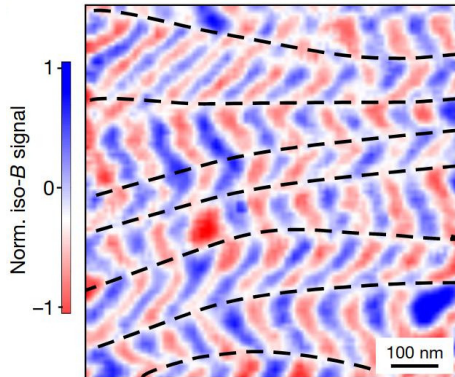
First real-space images of the cycloid

Piezoresponse force microscopy image
Ferroelectric domains



Reminder: The wavevector \vec{k} of the cycloid is always **perpendicular** to \vec{P}

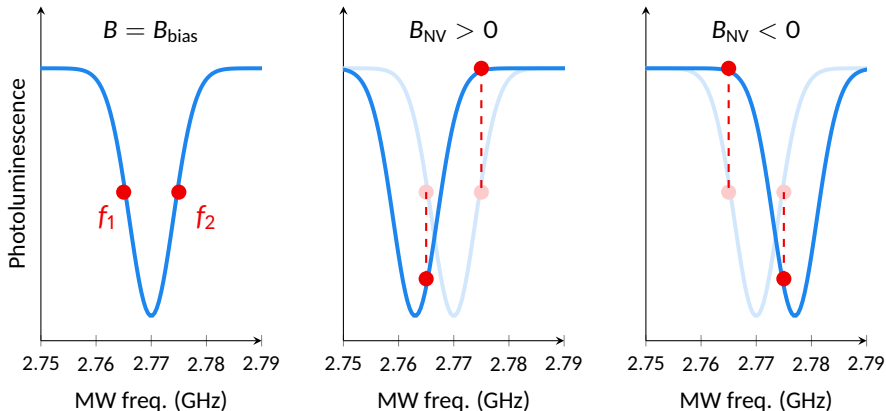
NV image
Field from the spin density wave



I. Gross et al. *Nature* 549 (2017), 252-256

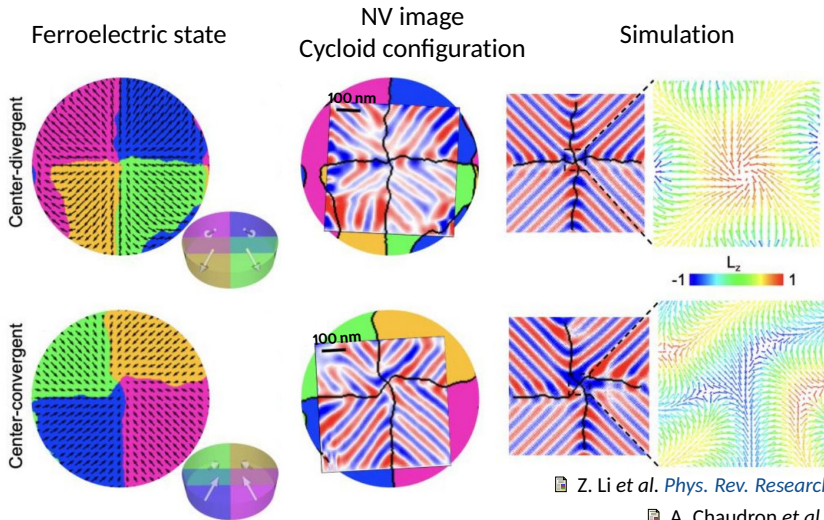
Note: the iso-B mode

$$\Delta\text{PL} = \text{PL}(f_2) - \text{PL}(f_1)$$



Creating "topological" multiferroic states in BiFeO_3

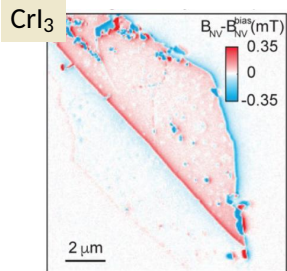
Collaboration: Lab. A. Fert, Paris (A. Chaudron, K. Bouzheouane, S. Fusil, V. Garcia)



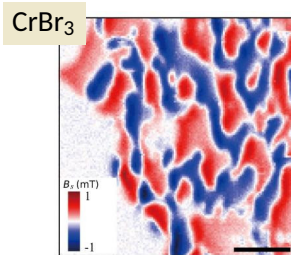
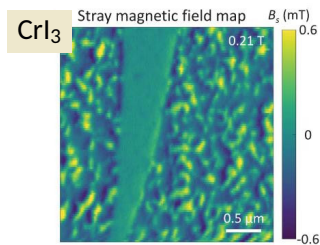
Z. Li et al. *Phys. Rev. Research* 5 (2023), 043109

A. Chaudron et al. *submitted* (2024)

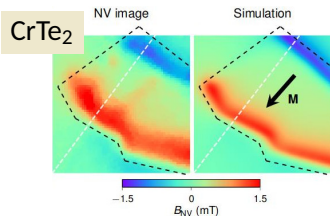
Example 3: van der Waals magnets



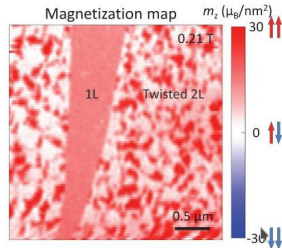
L. Thiel et al. *Science* 364 (2019), 973–976



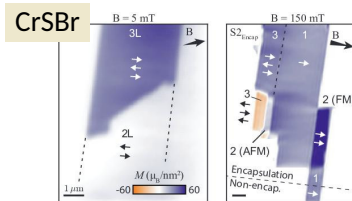
Q.-C. Sun et al. *Nat. Commun.* 12 (2021), 1989



F. Fabre et al. *Phys. Rev. Mater.* 5 (2021), 034008

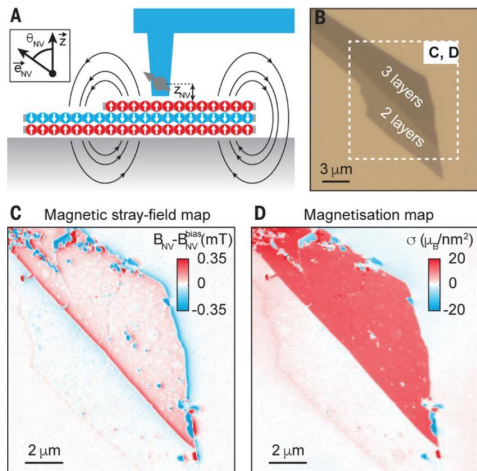


T. Song et al. *Science* 374 (2021), 1140



M. A. Tschudin et al. *arXiv* 2312.09279 (2023)

Reverse propagation to compute M maps



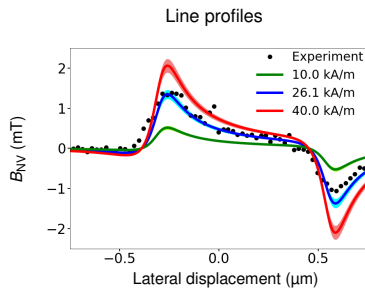
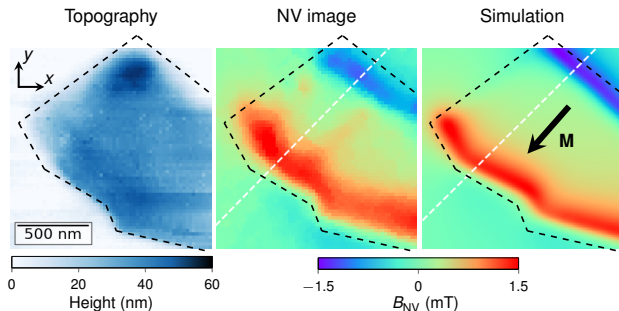
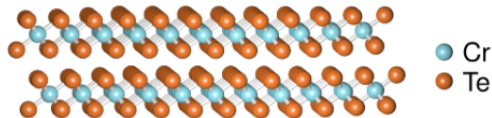
- Reconstruction of ferromagnetic magnetization density maps
- Fourier space calculation
- \vec{M} needs to be aligned along a single direction (α, β)
- Mostly reliable for out-of-plane magnetized samples

$$\begin{pmatrix} \tilde{B}_x \\ \tilde{B}_y \\ \tilde{B}_z \end{pmatrix} = \frac{-\mu_0}{2 e^q z_{NV}} \begin{pmatrix} \frac{q_x^2}{q} & \frac{q_x q_y}{q} & i q_x \\ \frac{q_x q_y}{q} & \frac{q_y^2}{q} & i q_y \\ i q_x & i q_y & -q \end{pmatrix} \begin{pmatrix} \sin \alpha \cos \beta \tilde{M}(q_x, q_y) \\ \sin \alpha \sin \beta \tilde{M}(q_x, q_y) \\ \cos \alpha \tilde{M}(q_x, q_y) \end{pmatrix}$$

Working with a model

Collaboration: Institut Néel, Grenoble (A. Purbawati, J. Coraux, N. Rougemaille)

2D ferromagnet at room temperature
with in-plane magnetization

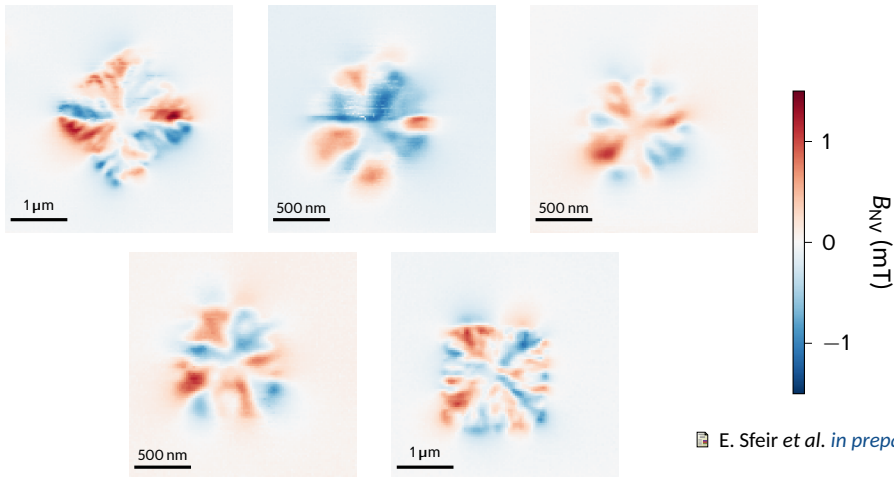


F. Fabre et al. *Phys. Rev. Mater.* 5 (2021), 034008

Vortices!

Collaboration: Spintec, Grenoble (F. Bonnell, M. Jamet)

At zero field, we observe vortices in microstructured Fe_5GeTe_2 (also an in-plane RT magnet):



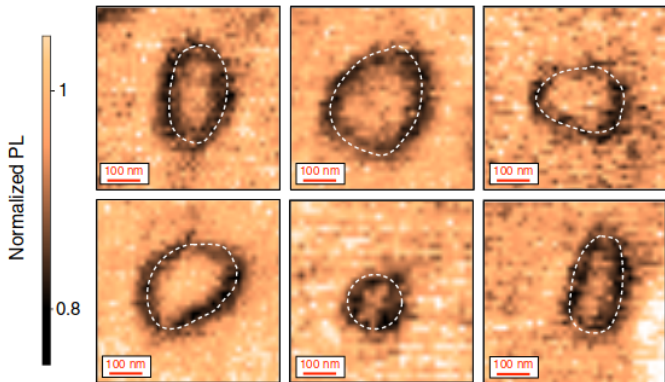
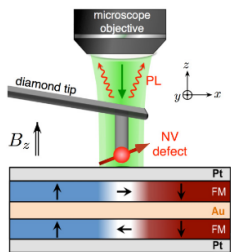
E. Sfeir et al. *in preparation* (2024)

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Simpler, faster but qualitative: measure the PL

Photoluminescence maps, under $B_z = 3$ mT

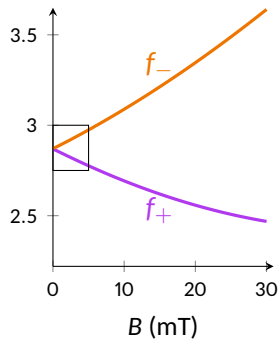


The effect of perpendicular magnetic field

“Strong” field not oriented along the NV axis

→ Mixing of the states $m_s = 0, \pm 1$, which are not eigenstates anymore

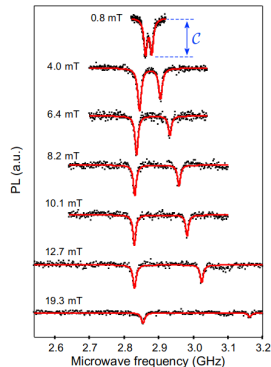
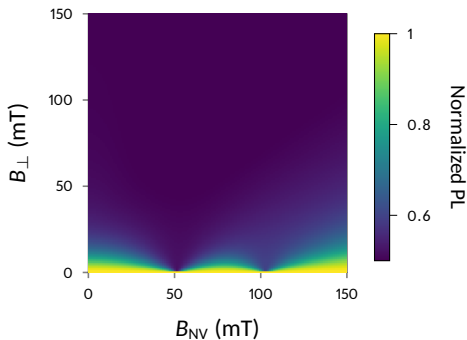
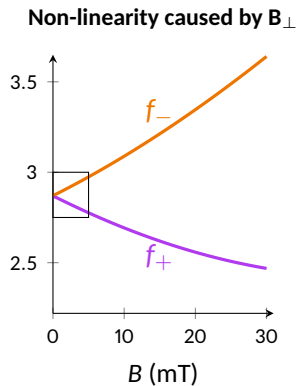
Non-linearity caused by B_{\perp}



The effect of perpendicular magnetic field

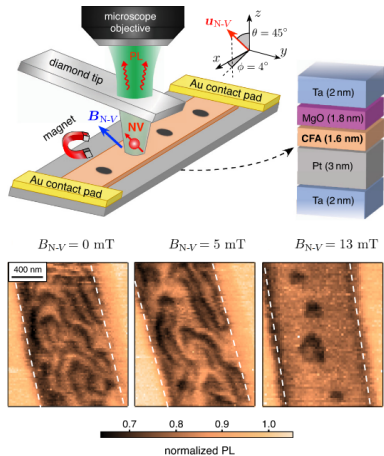
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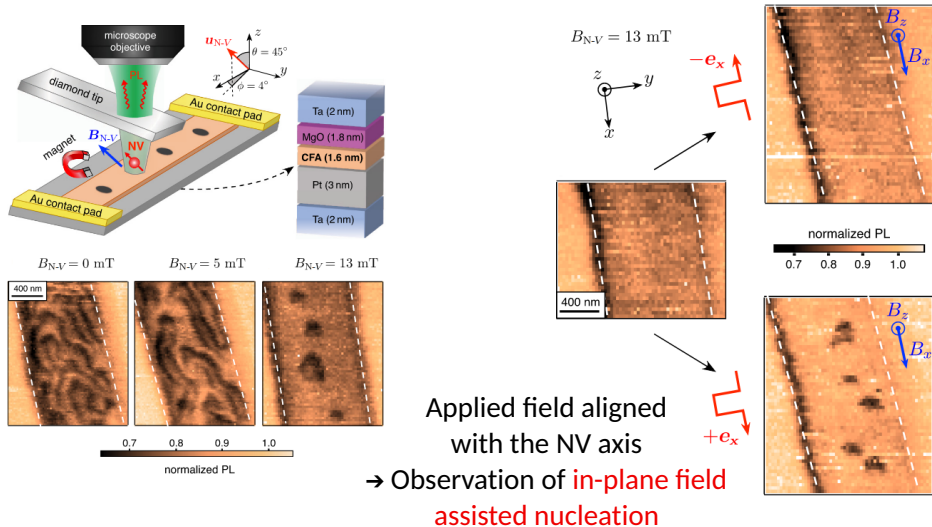


J.-P. Tetienne *et al.* *New J. Phys.* 14 (2012), 103033

Skyrmion nucleation in PL quenching mode

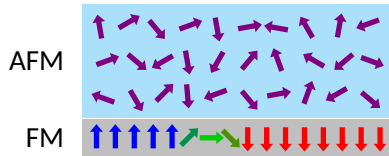


Skyrmion nucleation in PL quenching mode

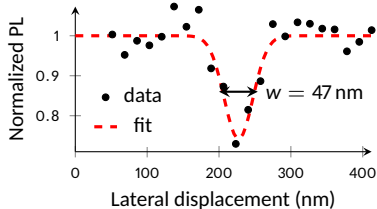
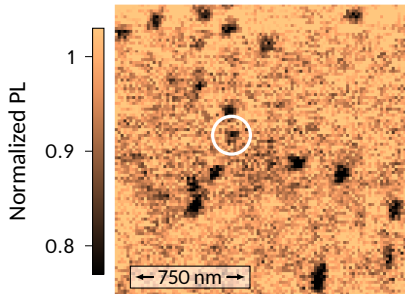
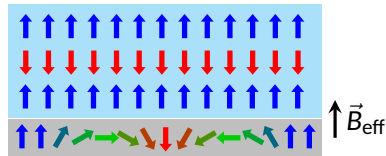


Skyrmions stabilized by exchange-bias

Blocking T below RT

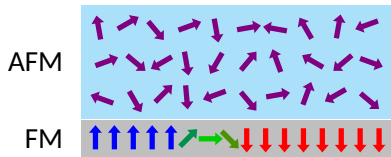


Blocking T above RT

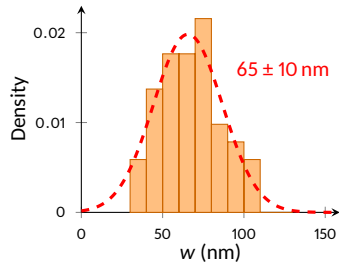
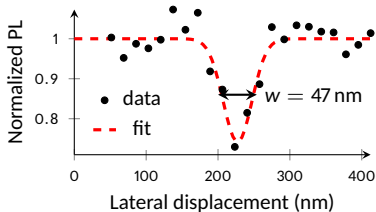
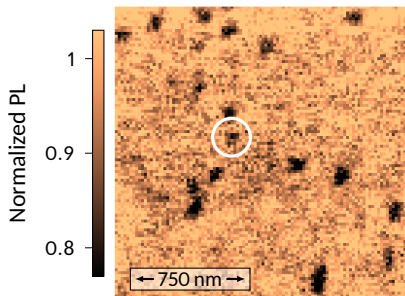
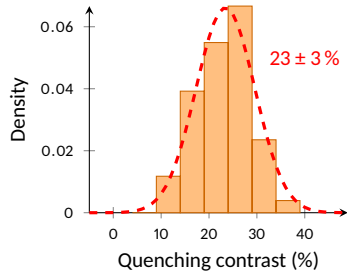
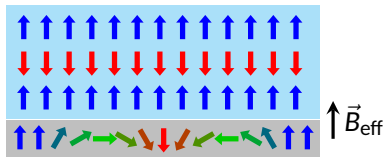


Skyrmions stabilized by exchange-bias

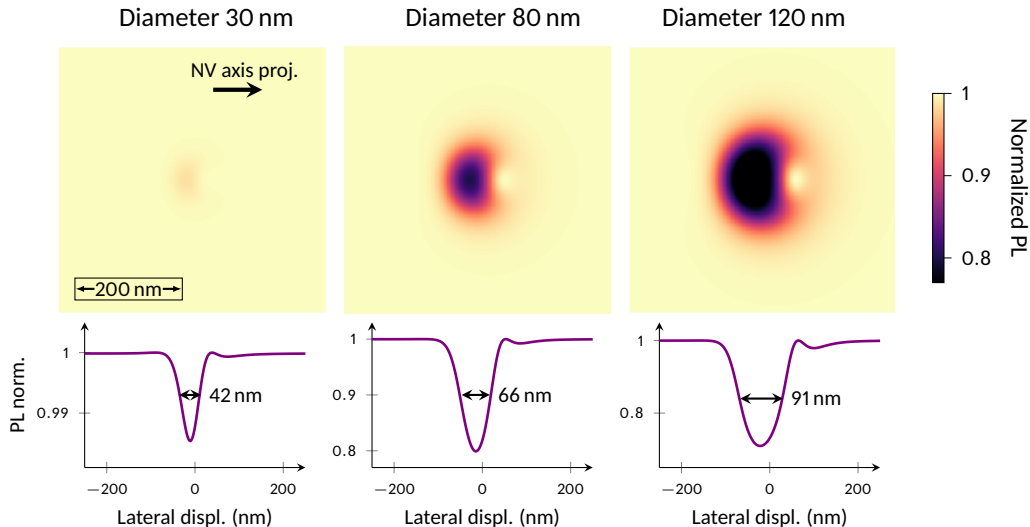
Blocking T below RT



Blocking T above RT



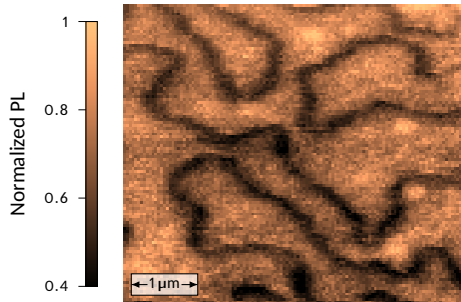
Trying to extract quantitative information



Noise: another source of PL quenching

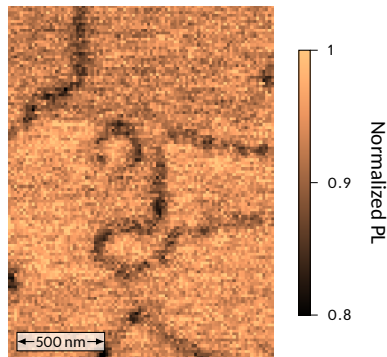
Thick ferromagnet

[Pt / Co (0.65 nm)]₄



Synthetic antiferromagnet

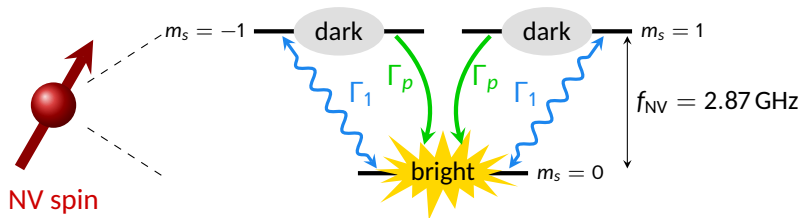
Pt / Co (0.65 nm) / Ru / Pt / Co (0.65 nm) / Ru



Outline

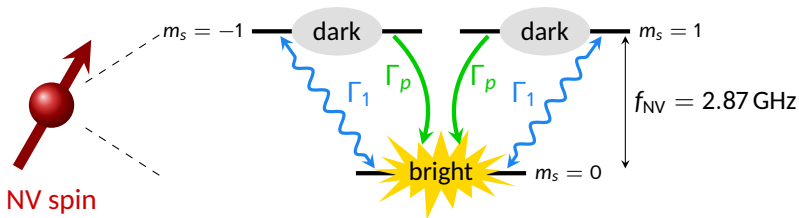
1. The NV center in diamond as a quantum sensor
2. Dismantling the scanning NV microscope
3. Quantitative ODMR experiments
 - Principle of the measurement
 - The need for a proper calibration
 - Example 1: analyzing domain walls
 - Example 2: the spin cycloid in bismuth ferrite
 - Example 3: van der Waals magnets
4. Taking a step back: PL quenching effects
 - Strong off-axis magnetic fields
 - Magnetic noise!
5. **Relaxometry: sensing via the relaxation time**
6. Coherent control of the NV center using spin waves
7. Going further: other sensors and sensing methods

Effect of magnetic noise on the NV center

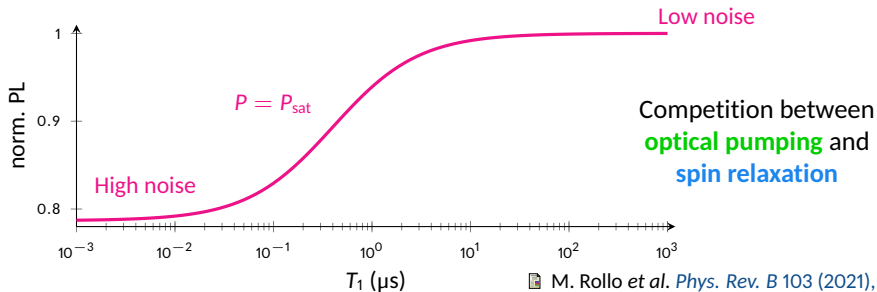


Relaxation rate $\Gamma_1 \propto S_{B_\perp}(f_{\text{NV}})$ magnetic field spectral density at the resonance frequency f_{NV}

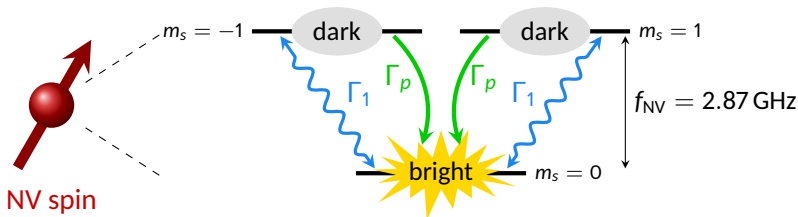
Effect of magnetic noise on the NV center



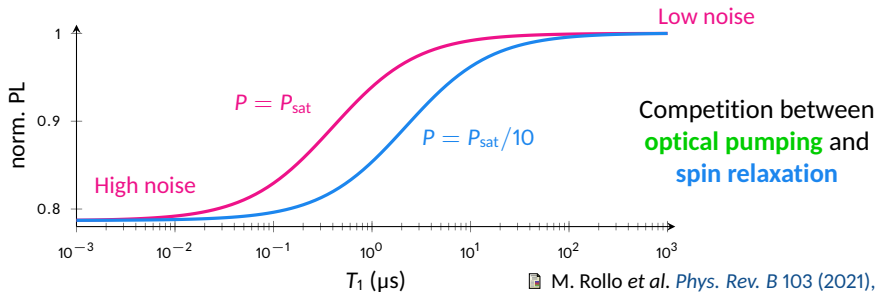
Relaxation rate $\Gamma_1 \propto S_{B_{\perp}}(f_{NV})$ magnetic field spectral density at the resonance frequency f_{NV}



Effect of magnetic noise on the NV center

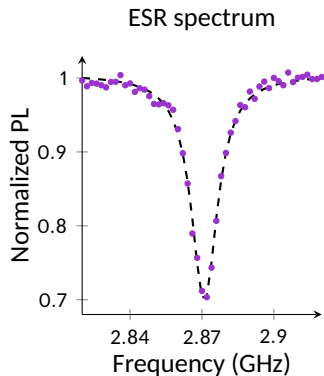
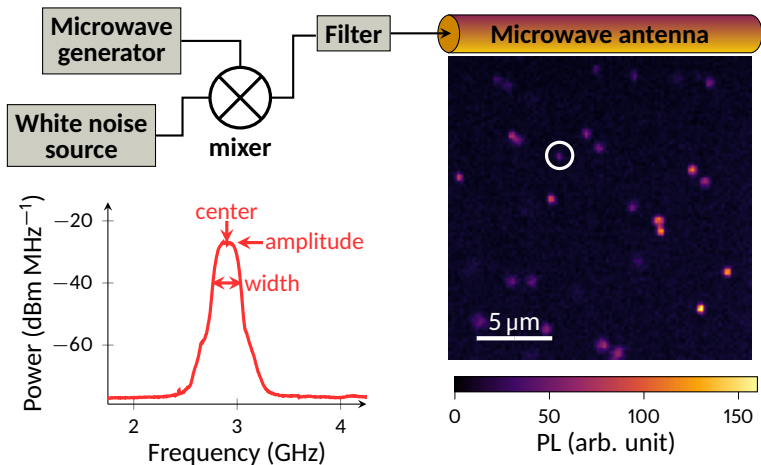


Relaxation rate $\Gamma_1 \propto S_{B_{\perp}}(f_{\text{NV}})$ magnetic field spectral density at the resonance frequency f_{NV}

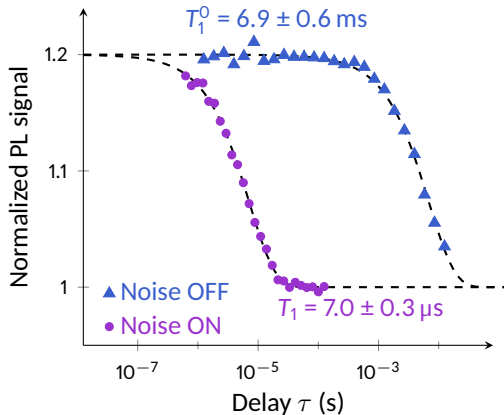
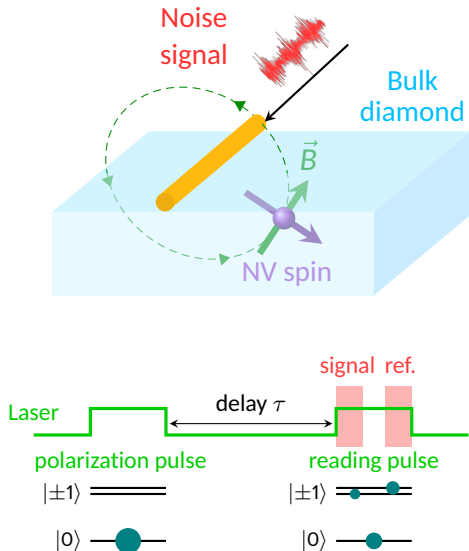


Experimental investigation

Collaboration: C2N, Palaiseau (Thibaut Devolder)



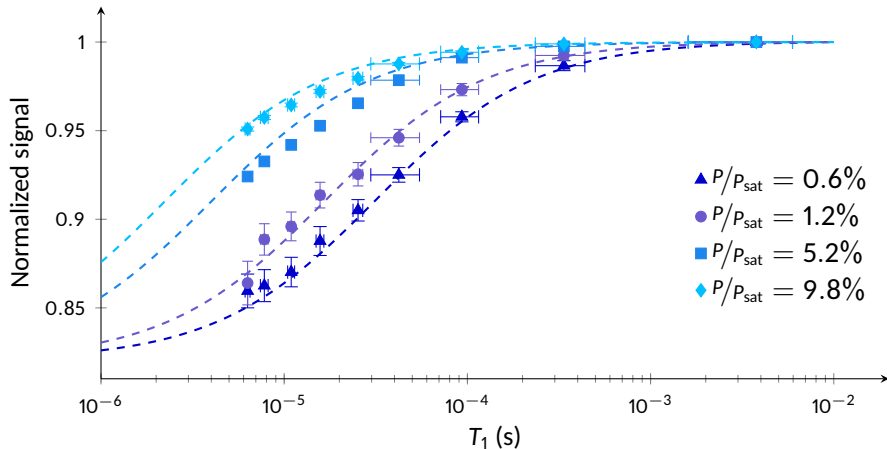
Acceleration of the relaxation with noise



Noise spectrum centered
at the NV transition frequency

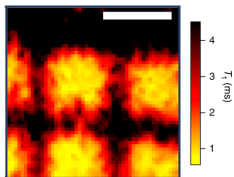
M. Rollo et al. *Phys. Rev. B* 103 (2021), 235418

Evolution of the PL with noise



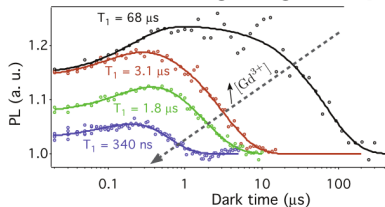
Applications of NV relaxometry

Measurement of Johnson noise: conductivity



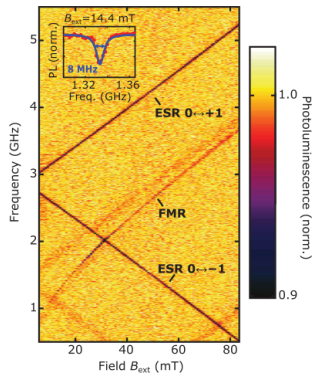
A. Ariyaratne *et al.* *Nat. Commun.* 9 (2018), 2406

Detection of fluctuating magnetic particles



J.-P. Tetienne *et al.* *Phys. Rev. B* 87 (2013), 235436

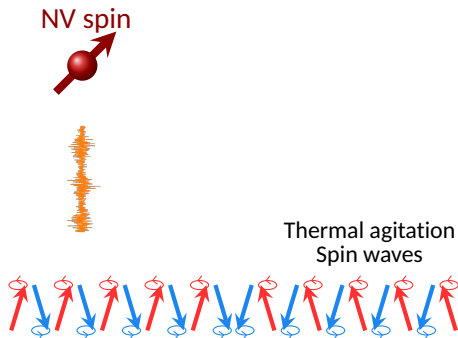
Investigation of spin waves



C. Du *et al.* *Science* 357 (2017), 195

Relaxometry to image antiferromagnets

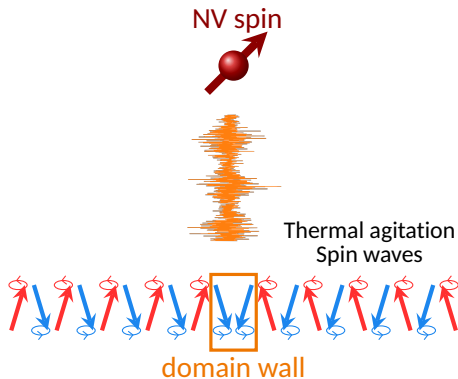
Principle: spin waves have different frequencies when they are confined inside domain walls



B. Flebus *et al.* *Phys. Rev. B* 98 (2018), 180409

Relaxometry to image antiferromagnets

Principle: spin waves have different frequencies when they are confined inside domain walls

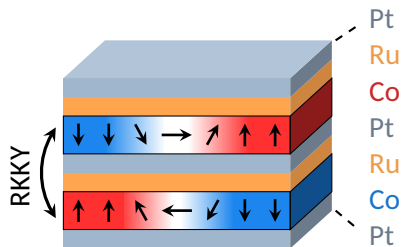


B. Flebus *et al.* *Phys. Rev. B* 98 (2018), 180409

Synthetic antiferromagnets

Collaboration: LAF, Palaiseau (W. Legrand, K. Bouzehouane, N. Reyren, V. Cros)

Two **ferromagnetic** layers coupled **antiferromagnetically**

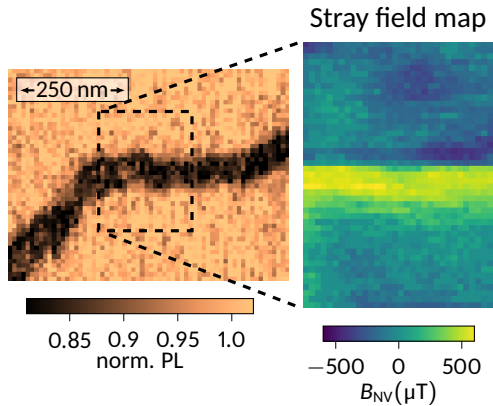
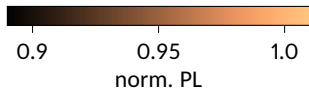
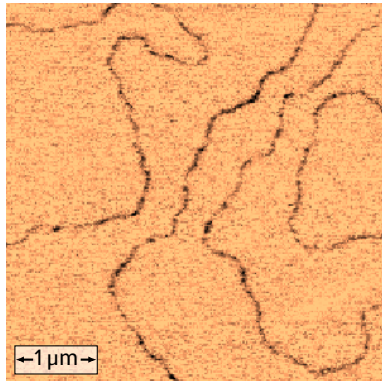


W. Legrand *et al.* *Nat. Mater.* 19 (2020), 34

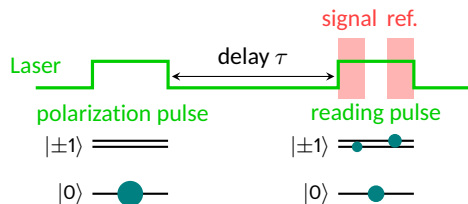
- No net magnetic moment
- Small stray field (vertical shift)
- Highly tunable properties
- Spin wave frequencies in the few GHz range

→ Perfect **test system**
for noise imaging!

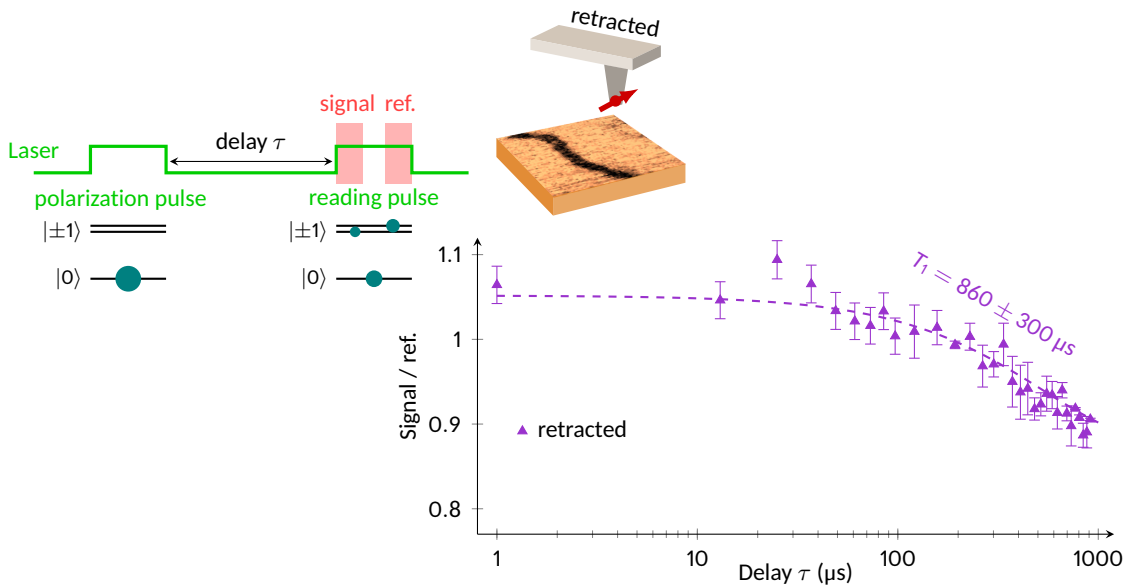
Detection of domain walls by relaxometry



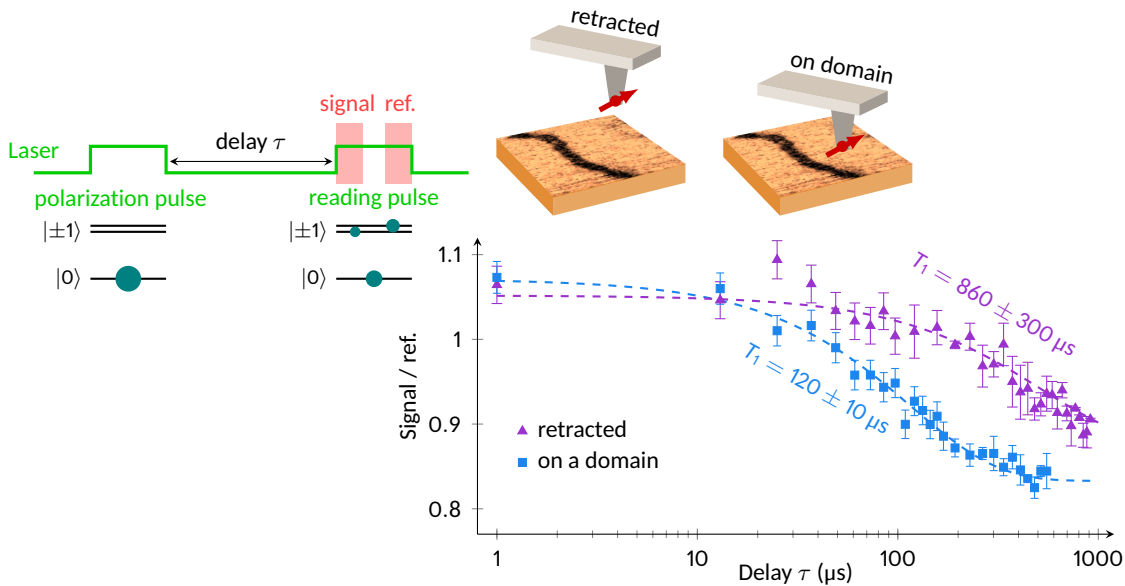
Local variation of the relaxation time



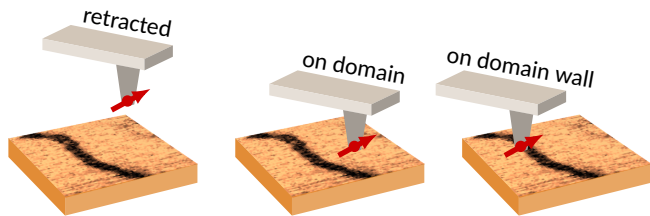
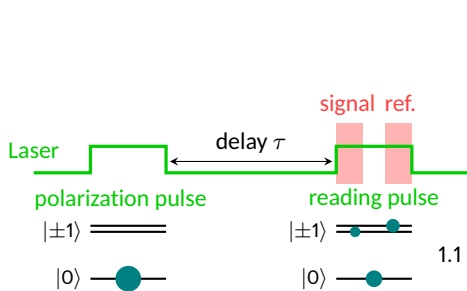
Local variation of the relaxation time



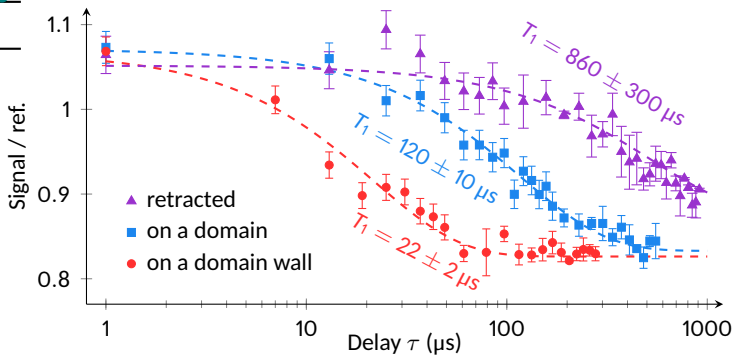
Local variation of the relaxation time



Local variation of the relaxation time

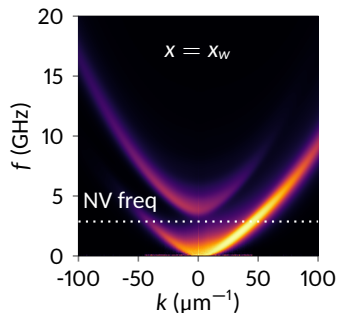
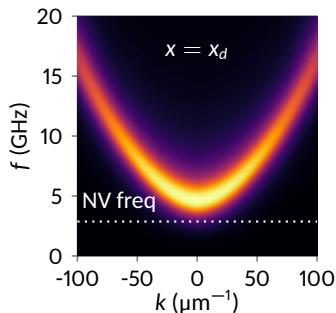
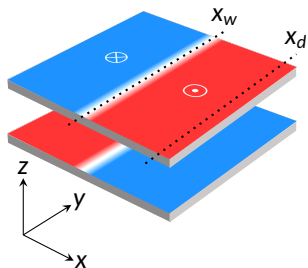


Clear diminution of T_1
→ **Enhancement of the spin relaxation**



Origin of the noise: spin waves

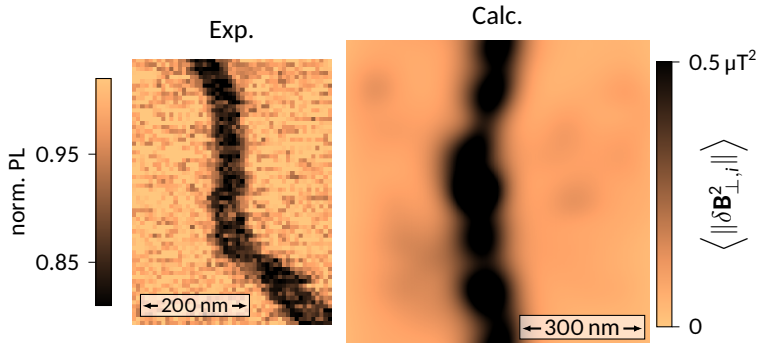
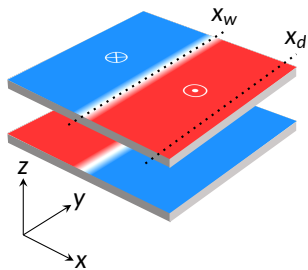
Collaboration: C2N, Palaiseau (J.-P. Adam, J.-V. Kim)



- NV frequency slightly below the gap, in the tail of power spectral density, which is the reason why we detect some noise when approaching the tip.
- No gap in the domain walls, presence of modes at the NV frequency: **the NV center is more sensitive to the noise from the walls!**

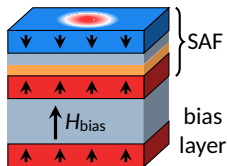
Origin of the noise: spin waves

Collaboration: C2N, Palaiseau (J.-P. Adam, J.-V. Kim)

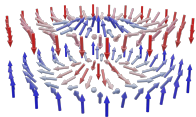


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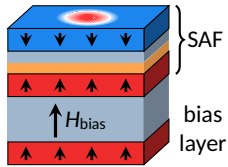
Skyrmions stabilized by a bias layer



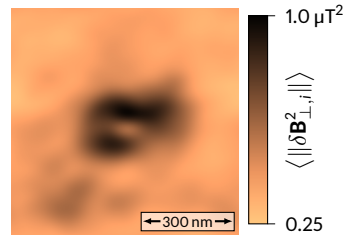
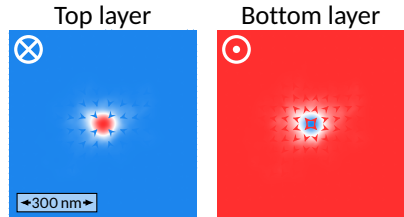
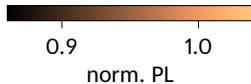
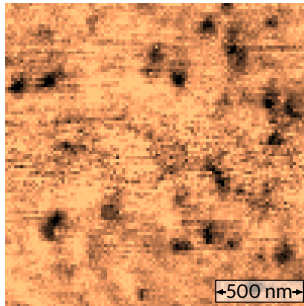
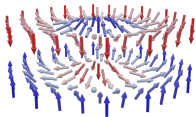
 W. Legrand *et al.* *Nat. Mater.* 19 (2020), 34



Skyrmions stabilized by a bias layer



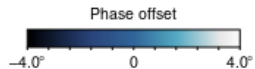
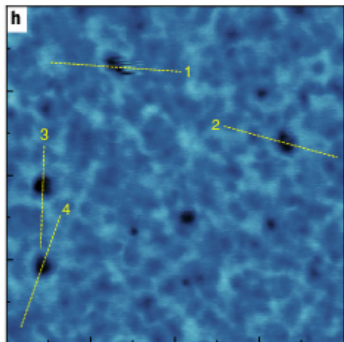
W. Legrand *et al. Nat. Mater.* 19 (2020), 34



We are not probing the internal modes but the scattering of spin waves on the skyrmions

Are these really skyrmions?

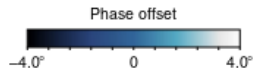
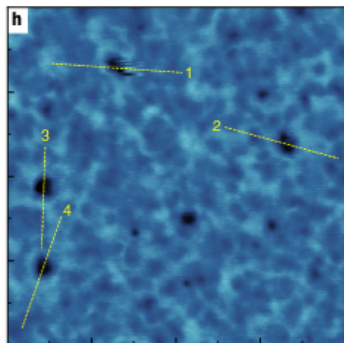
MFM under oop field 110 mT



W. Legrand *et al.* *Nat. Mater.* 19 (2020), 34

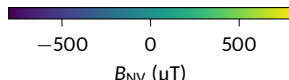
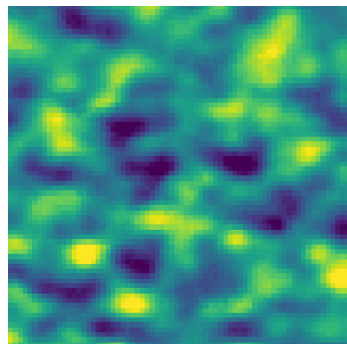
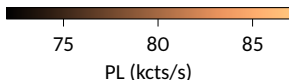
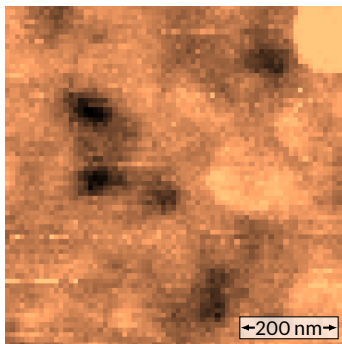
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MFM under oop field 110 mT



W. Legrand et al. *Nat. Mater.* 19 (2020), 34

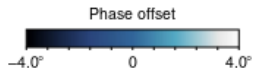
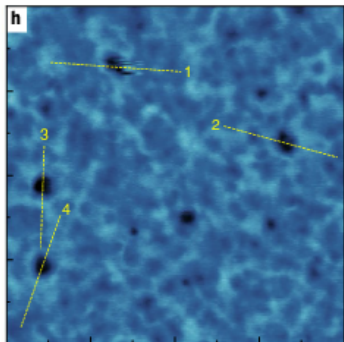
NV images (zero field)



Large background fluctuations (roughness of the film)

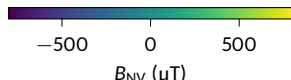
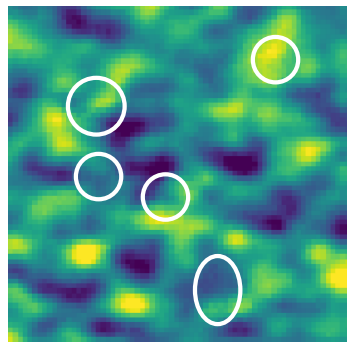
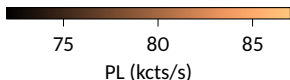
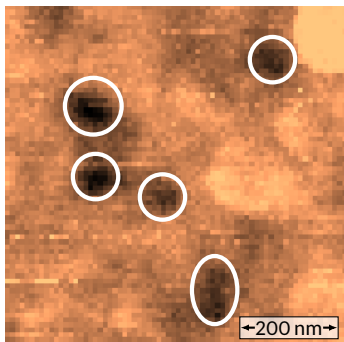
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W. Legrand et al. *Nat. Mater.* 19 (2020), 34

NV images (zero field)

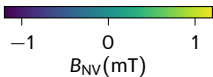
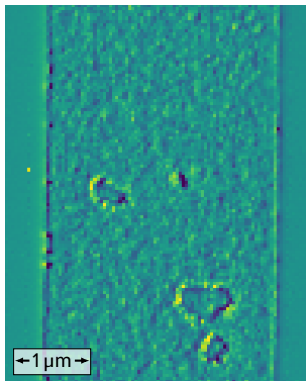


Large background fluctuations (roughness of the film)

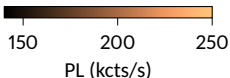
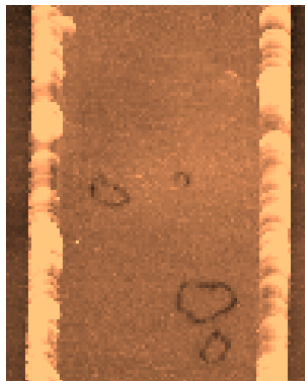
Other samples without bias layer

Collaboration: Spintec, Grenoble (V.-T. Pham, J. Urrestarazu, O. Boulle)

NV stray field map

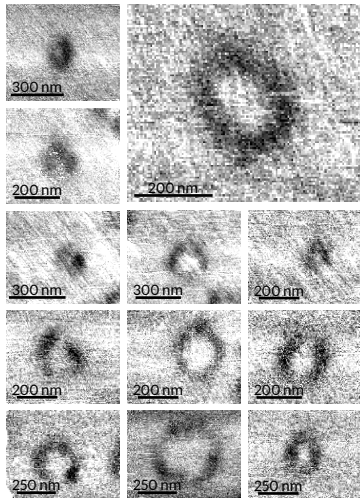


Noise (PL) map

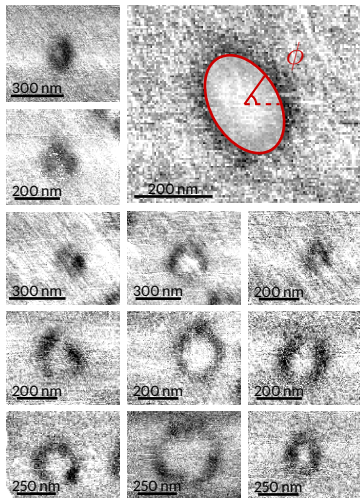


- Oop field of about 150 mT applied for nucleation
- Less background fluctuations

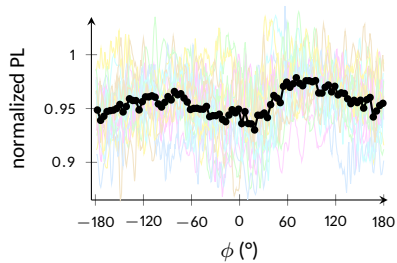
Statistics on Néel left skyrmions



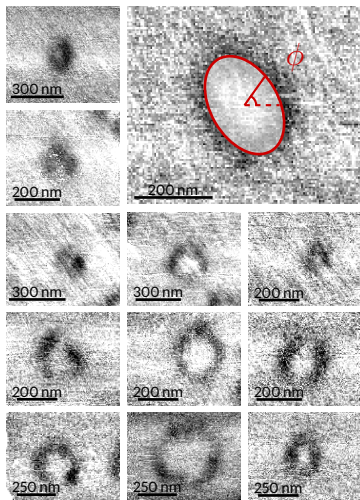
Statistics on Néel left skyrmions



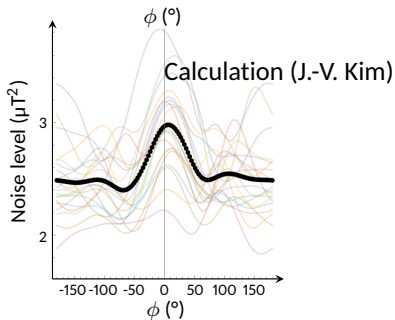
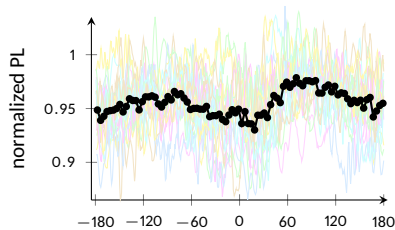
Angular variation of PL



Statistics on Néel left skyrmions

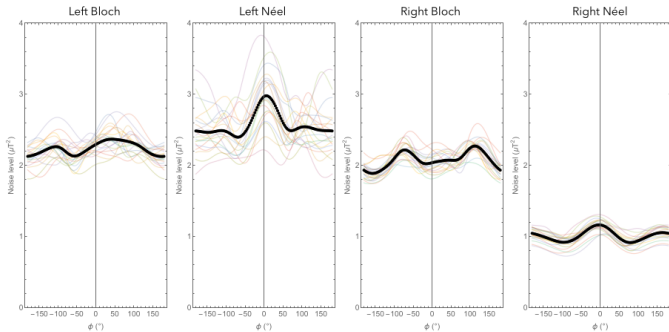


Angular variation of PL

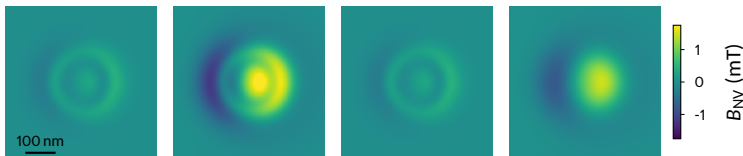


Expected pattern on other skyrmion types

Simulated noise distribution along the contour

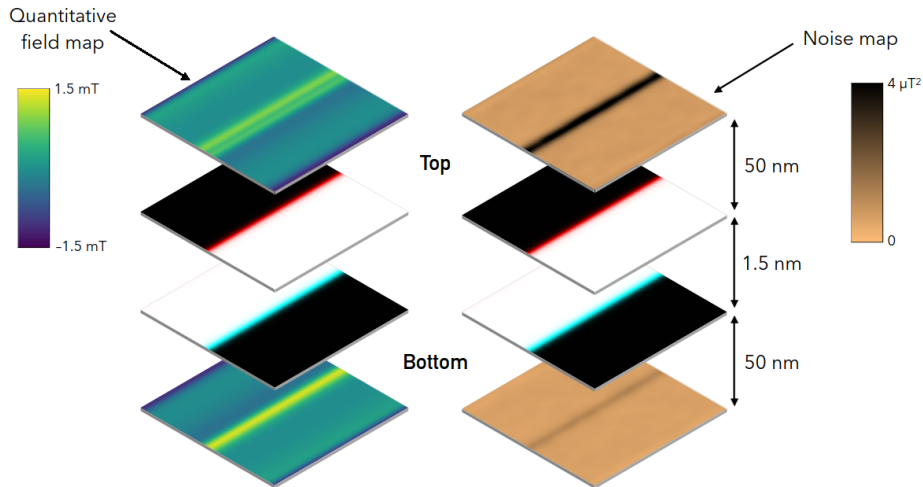


Simulated stray field maps



Expected noise level for each domain wall chirality

Calculation: C2N, Palaiseau (J.-V. Kim)

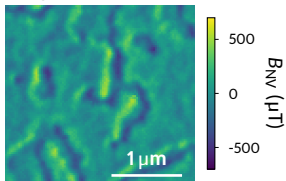


Experiment: looking at both sides of the film

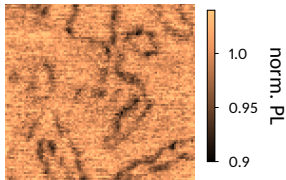
Initial stack: Néel left

TaOx 3 nm
Ru 0.6 nm
Co 1.5 nm
Pt 0.5 nm
Ru 0.8 nm
Co 1.5 nm
Pt 3 nm
Ta

Magnetic field map



Noise map

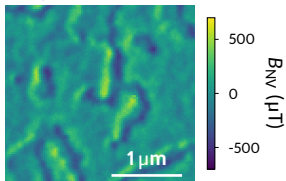


Experiment: looking at both sides of the film

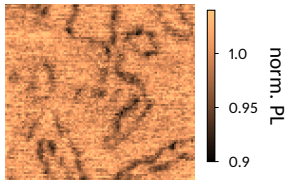
Initial stack: Néel left



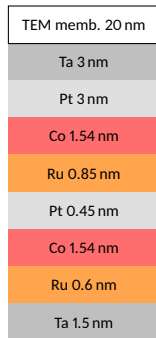
Magnetic field map



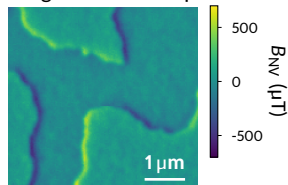
Noise map



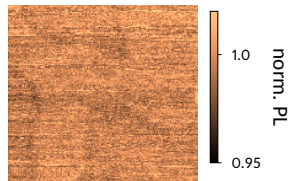
Inverted stack: Néel right



Magnetic field map



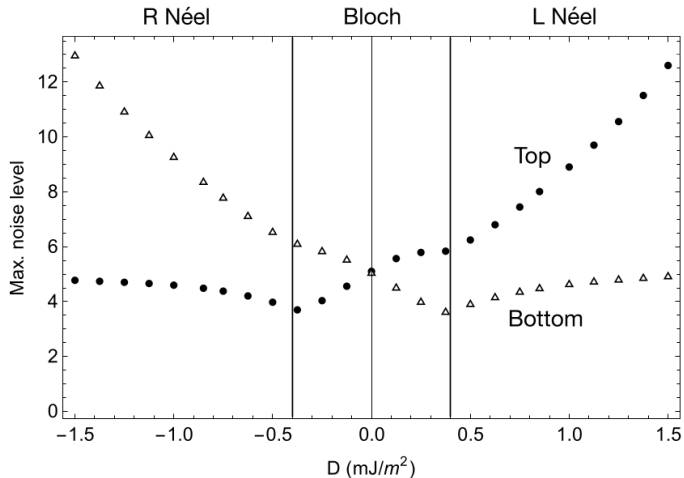
Noise map



A DMI-related effect

Calculation: C2N, Palaiseau (J.-V. Kim)

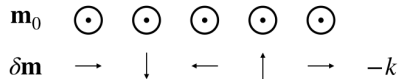
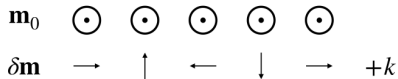
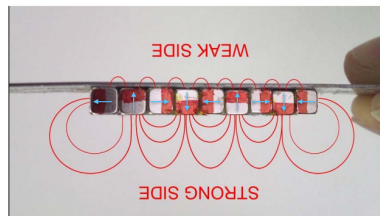
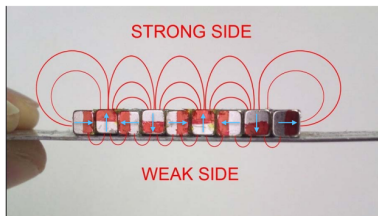
Calculation made for a **single** ferromagnetic layer



Spin waves are like fridge magnets!

©Thibaut Devolder, C2N, Palaiseau

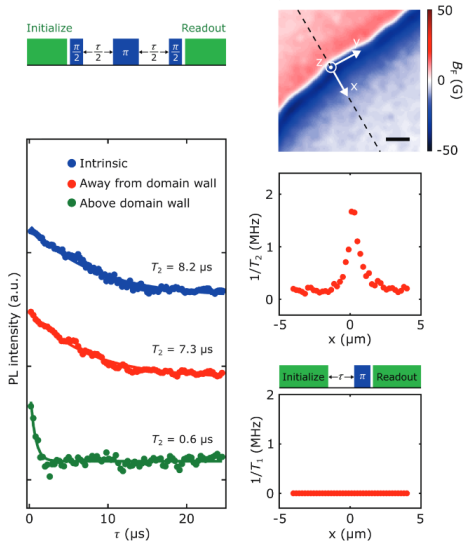
... and the DMI is selecting a propagation direction!



J. Mallinson. *IEEE Trans. on Mag.* 9 (1973), 678

T. Devolder. *Phys. Rev. Appl.* 20 (2023), 054057

Decrease of T_2 at ferromagnetic domain walls



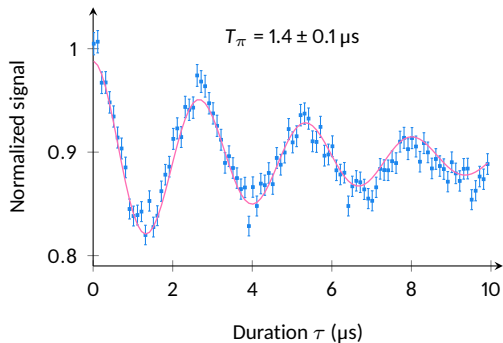
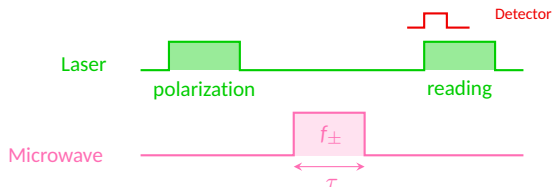
- Co/Ni/Co/Ni ferromagnetic layer
- Coherence time T_2 of the NV center decreased above the domain wall
- Here T_1 is not modified!
- Effect attributed to the gapless magnetic excitation of the wall

N. J. McLaughlin et al. *ACS Nano* 17 (2023), 25689

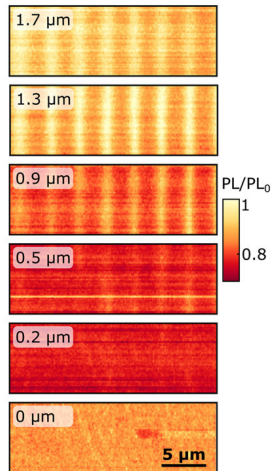
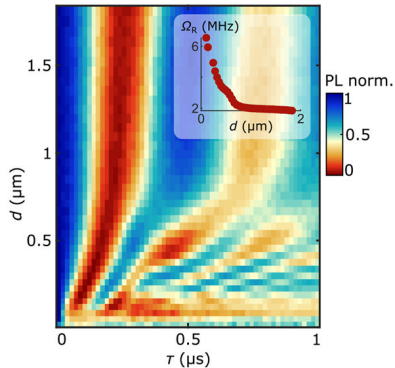
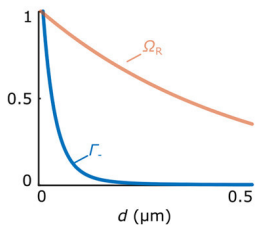
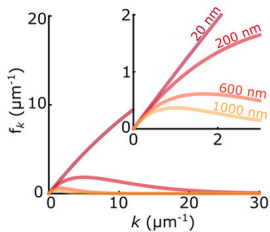
Outline

1. The NV center in diamond as a quantum sensor
2. Dismantling the scanning NV microscope
3. Quantitative ODMR experiments
 - Principle of the measurement
 - The need for a proper calibration
 - Example 1: analyzing domain walls
 - Example 2: the spin cycloid in bismuth ferrite
 - Example 3: van der Waals magnets
4. Taking a step back: PL quenching effects
 - Strong off-axis magnetic fields
 - Magnetic noise!
5. Relaxometry: sensing via the relaxation time
6. Coherent control of the NV center using spin waves
7. Going further: other sensors and sensing methods

Measurement of a Rabi oscillation

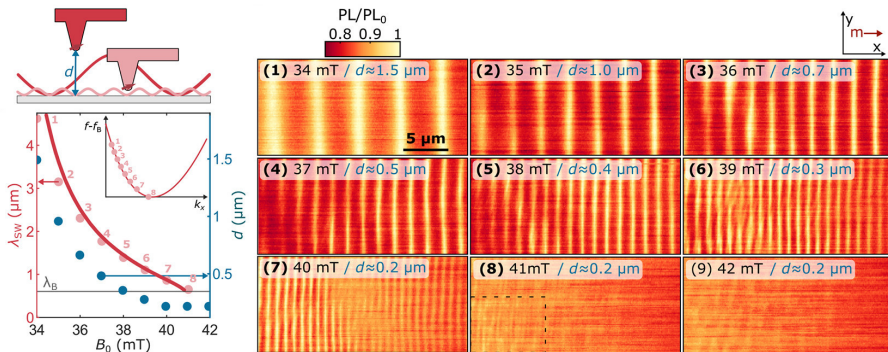


Rabi oscillation driven by a spin wave



Wavelength filter with d_{NV}

- Spin waves excited in YIG with a microwave stripline
- The wavelength of the spin wave at f_{\pm} is tuned with magnetic field
- The flying distance d_{NV} is optimized to observe it in the ODMR contrast map



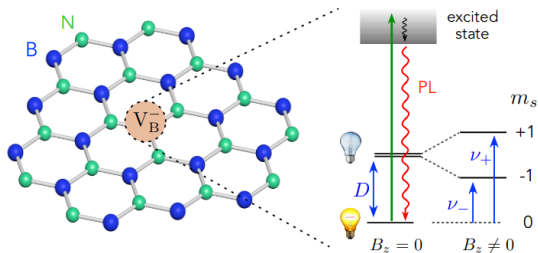
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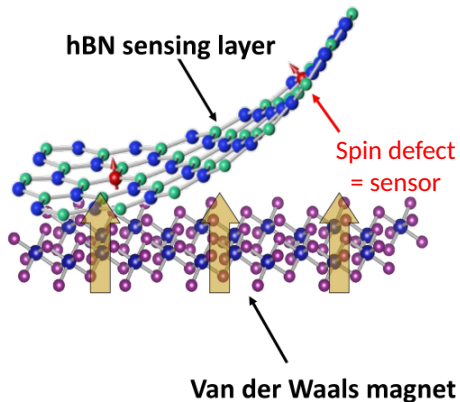
Boron vacancies in h-BN

Optically-active spin defect in h-BN found in 2020

A. Gottscholl et al. *Nat. Mater.* 19 (2020), 540

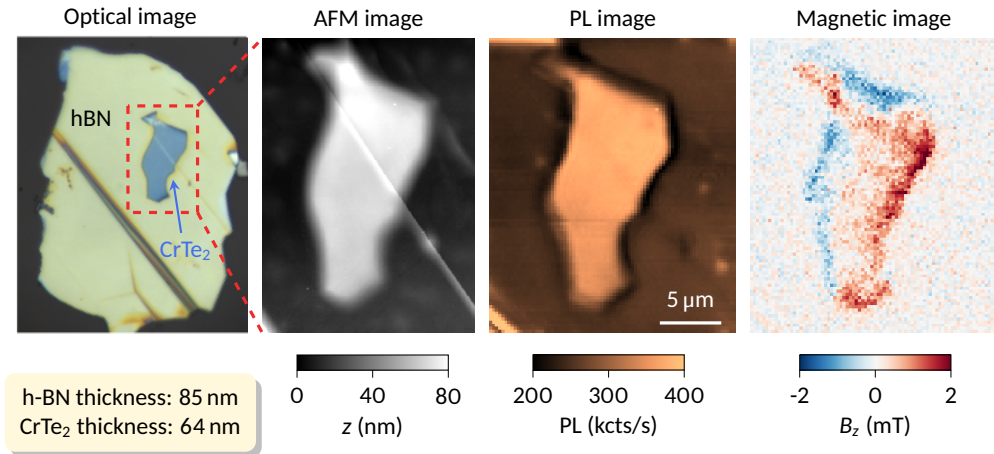


P. Kumar et al. *Phys. Rev. Appl.* 18 (2022), L061002



Imaging with boron vacancies

Collaboration: Institut Néel, Grenoble and LPCNO, Toulouse



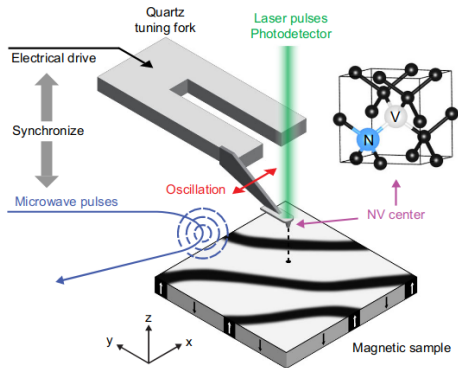
P. Kumar et al. *Phys. Rev. Appl.* 18 (2022), L061002

M. Huang et al. *Nat. Commun.* 13 (2022), 5369

A. J. Healey et al. *Nat. Phys.* 19 (2023), 87

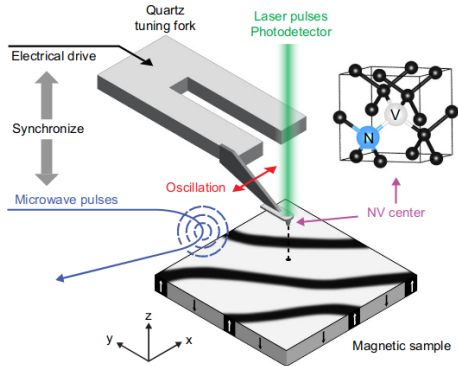
Advanced mode: gradiometry

Use a spin echo sequence to improve the magnetic sensitivity

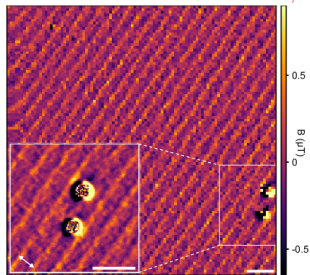
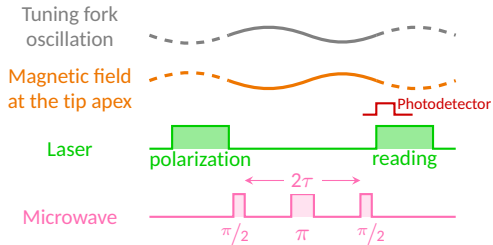


Advanced mode: gradiometry

Use a spin echo sequence to improve the magnetic sensitivity



W. S. Huxter *et al.* *Nat. Commun.* 13 (2022), 3761

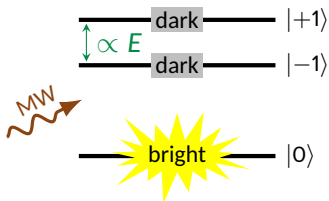


Stray field from atomic steps visible in the A-type antiferromagnet Cr_2O_3

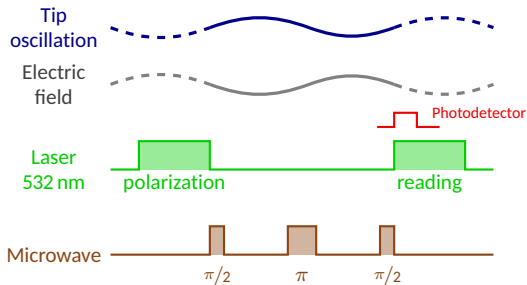
Electric field sensing

To investigate ferroelectrics!

Stark shift



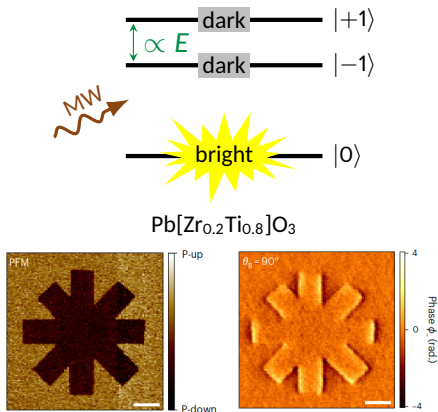
- Need to apply off-axis field to avoid that Zeeman effect dominates
- Electric susceptibilities rather small
→ spin echo sequences



Electric field sensing

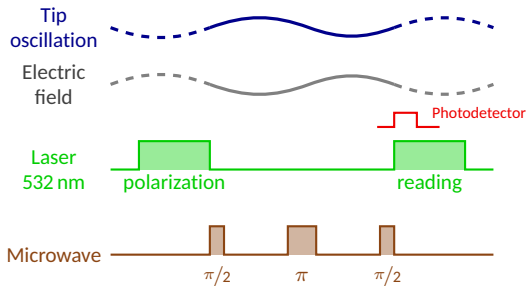
To investigate ferroelectrics!

Stark shift



W. S. Huxter et al. *Nat. Phys.* 19 (2023), 644

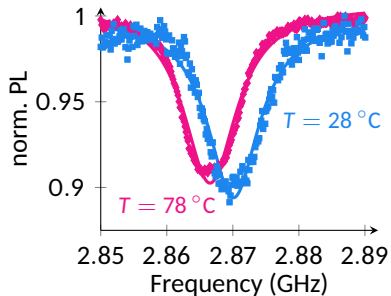
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Z. Qiu et al. *npj Quantum Info.* 8 (2022), 107

Temperature sensing

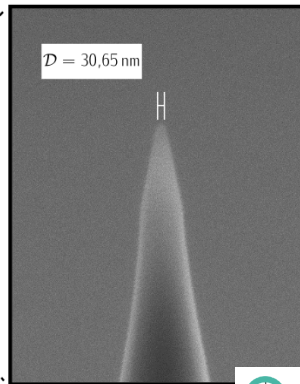
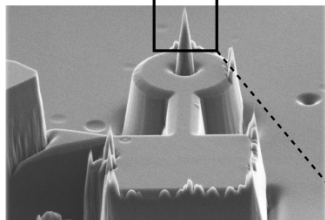
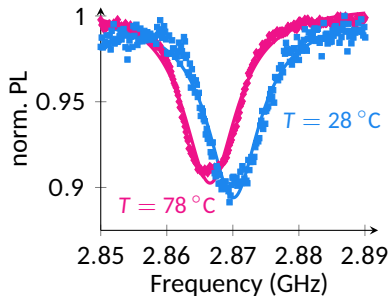
Crystal dilatation \rightarrow Shift of the zero-field splitting



Temperature sensing

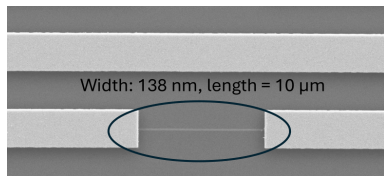
Crystal dilatation \rightarrow Shift of the zero-field splitting

Specifically designed probe
to ensure a perfect
thermalization of the sensor

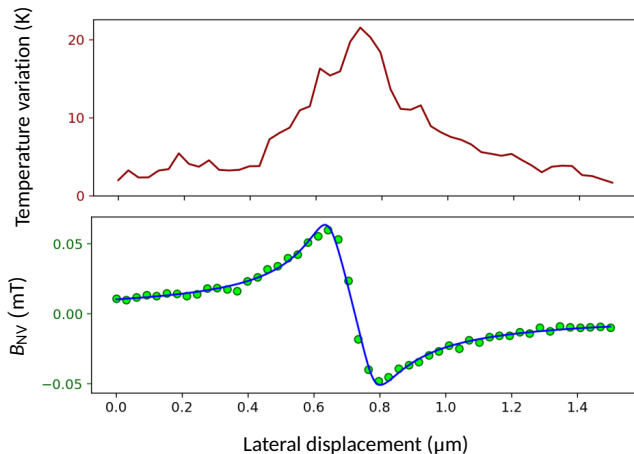


Sensitivity $\sim 1\text{K}/\sqrt{\text{Hz}}$

First results on thermometry



Nanowire of doped Si from CEA Grenoble
 $I = 60 \mu\text{A}$



A versatile microscope to probe condensed matter

Magnetic field

(Anti)ferromagnetic textures
Currents

Magnetic noise

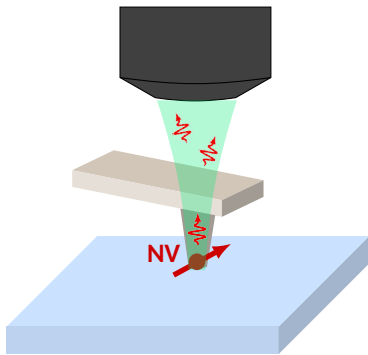
Spin waves
Conductivity

Electric field

Ferroelectric textures

Temperature

Localized hot spots



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