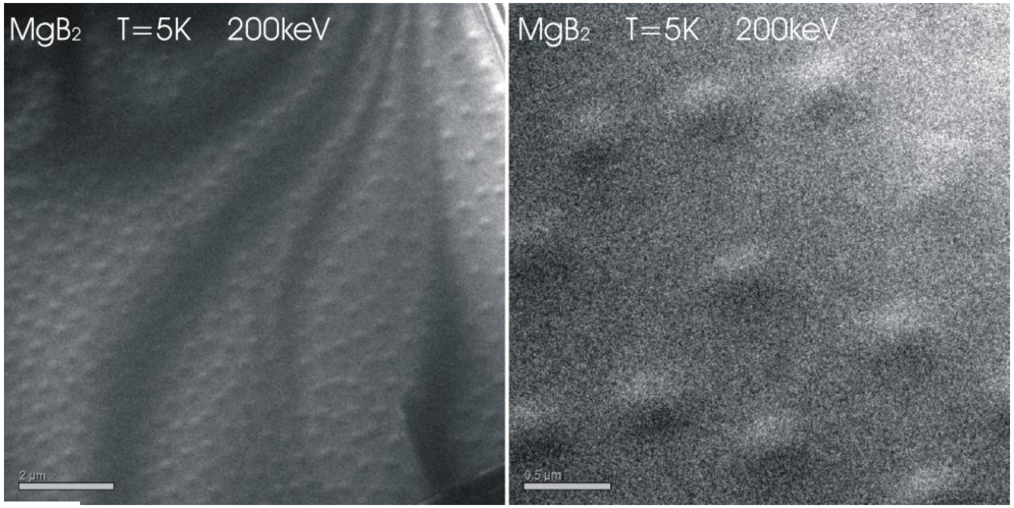
ÉCOLE POLYTECHNIQUE FÉDÉRALE DE LAUSANNE

F. Carbone

Nimes, October 2012

Transmission Electron Microscopy resolved in space, energy and time



LUMES
Laboratory for Ultrafast Microscopy and Electron Scattering

PAUL SCHERRER INSTITUT
PSI

IDES INTEGRATED DYNAMIC ELECTRON SOLUTIONS

SIGILL. COLL. TRIN. SANC.

CiMe
Centre Interdisciplinaire de Microscopie Électronique

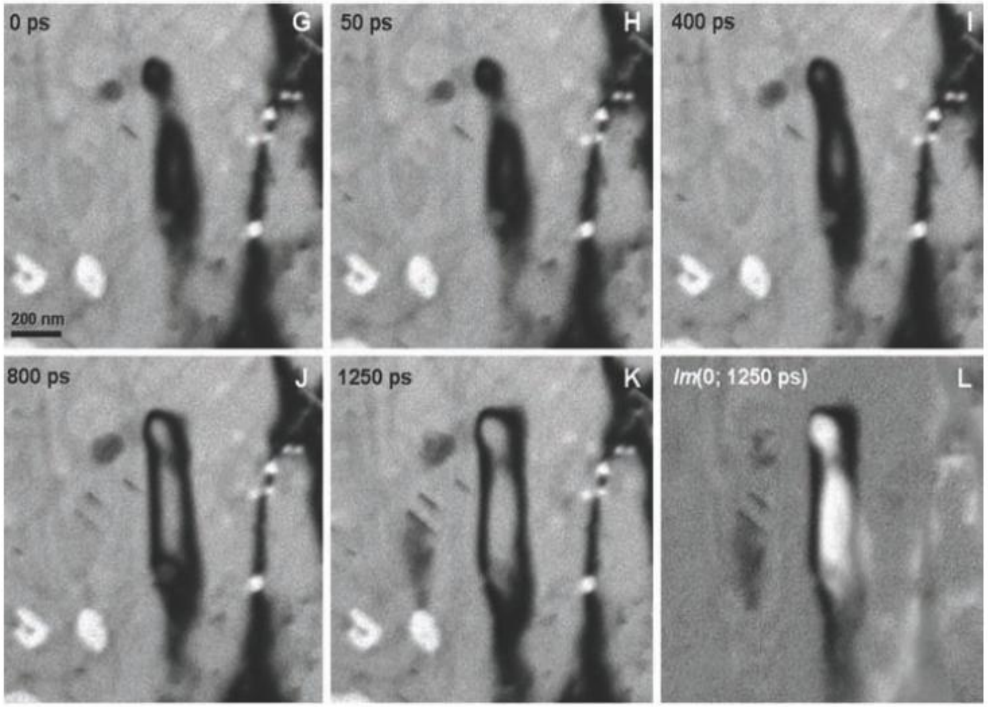
erc

Lawrence Livermore National Laboratory

Why time-resolved?

Ultrafast TEM - Motivation

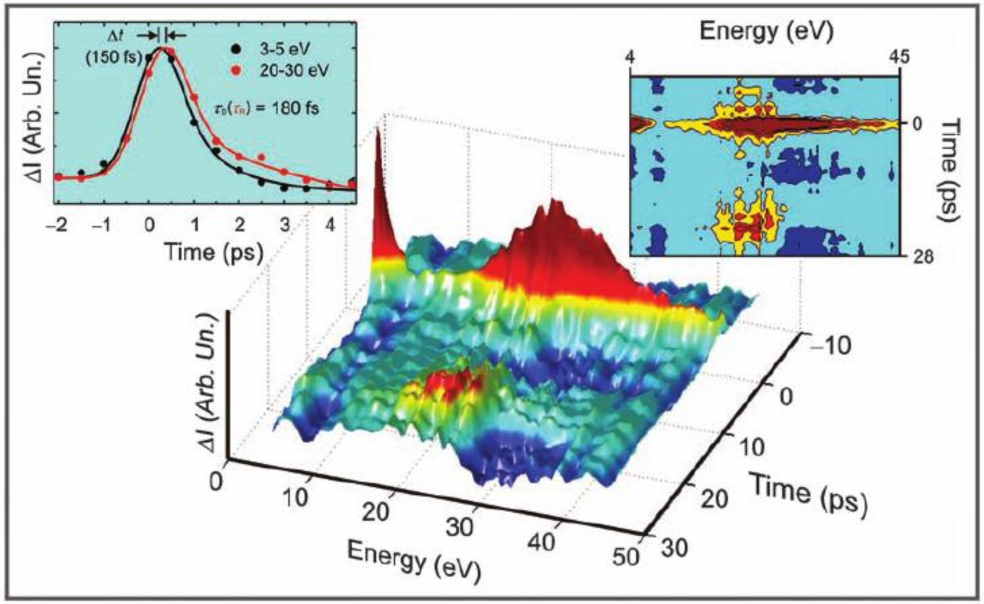
- Ultrafast Imaging



B. Barwick, Science (2008)

Ultrafast TEM - Motivation

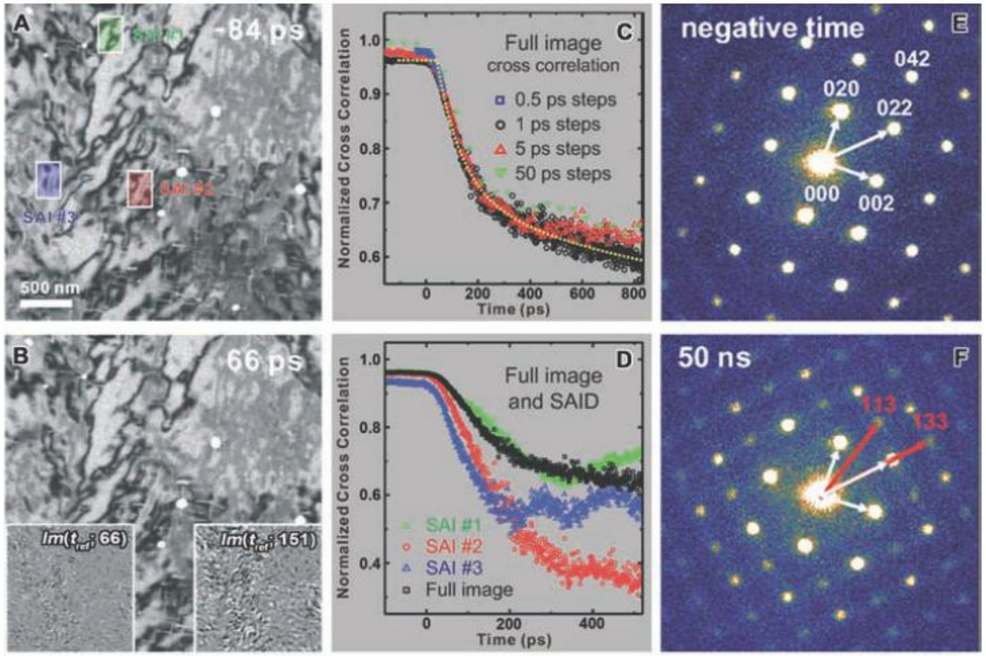
- Ultrafast Imaging
- Ultrafast Spectroscopy



F. Carbone, Science (2009)

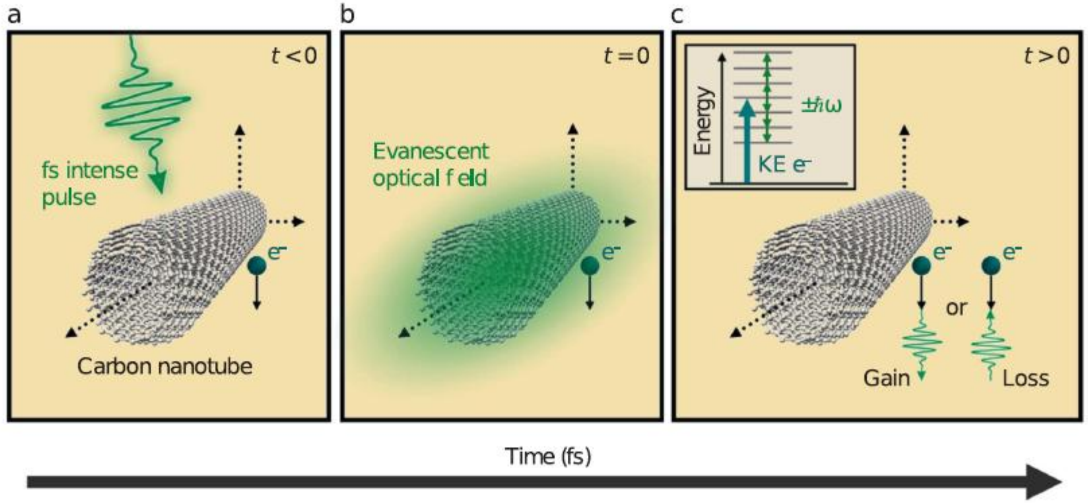
Ultrafast TEM - Motivation

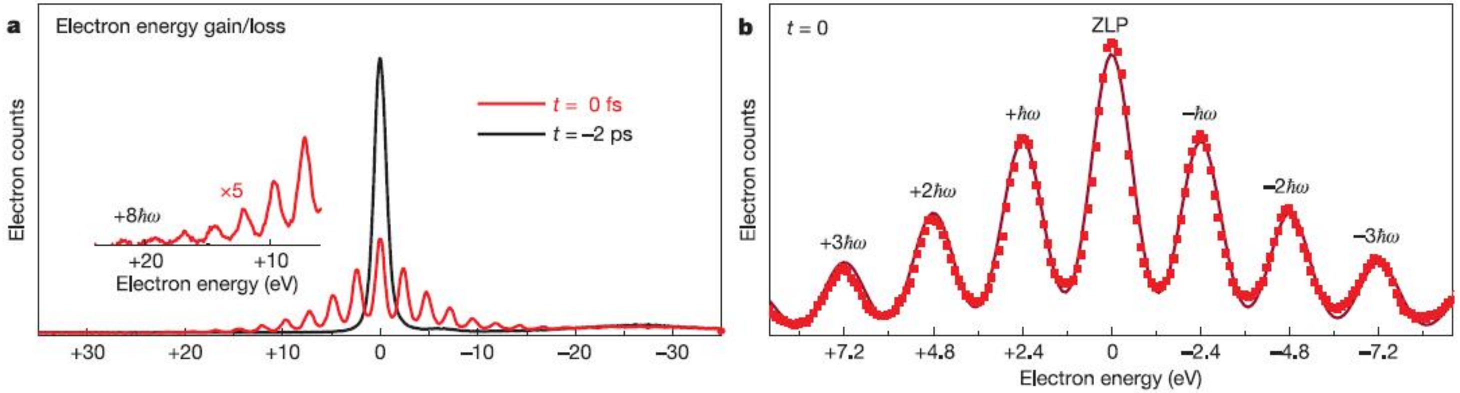
- Ultrafast Imaging
- Ultrafast Spectroscopy
- Ultrafast Diffraction



B. Barwick, Science (2008)

Photon-Induced Near-field Electron Microscopy

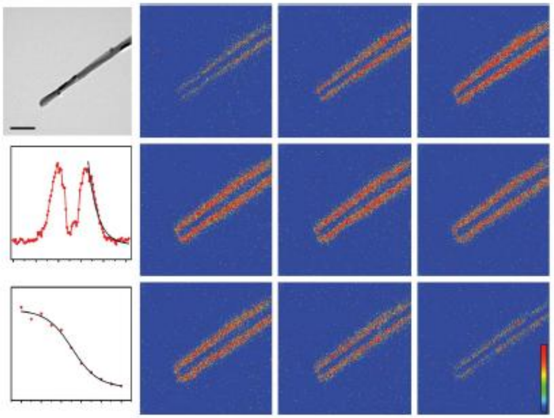




(B. Barwick, Nature (2009))

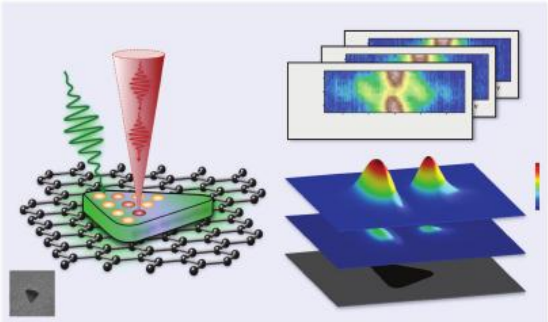
PINEM - Applications

Carbon Nanotubes



(B. Barwick, Nature (2009))

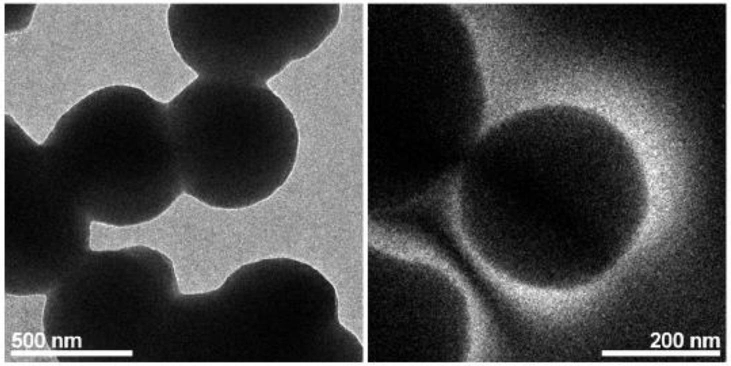
Silver Nanoparticles



(A. Yurtsever, Science (2012))

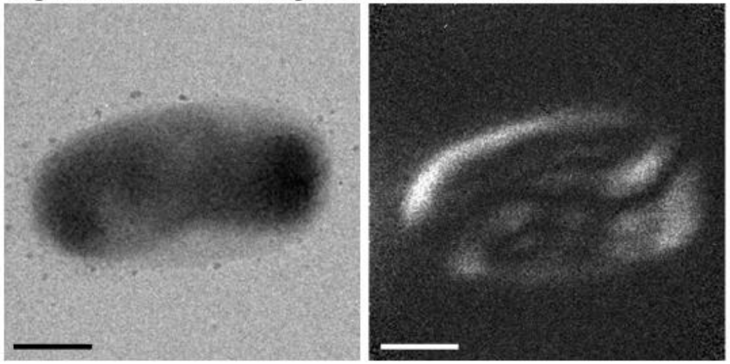
Protein Vesicles

Bright-field and PINEM of protein vesicles



Escherichia coli

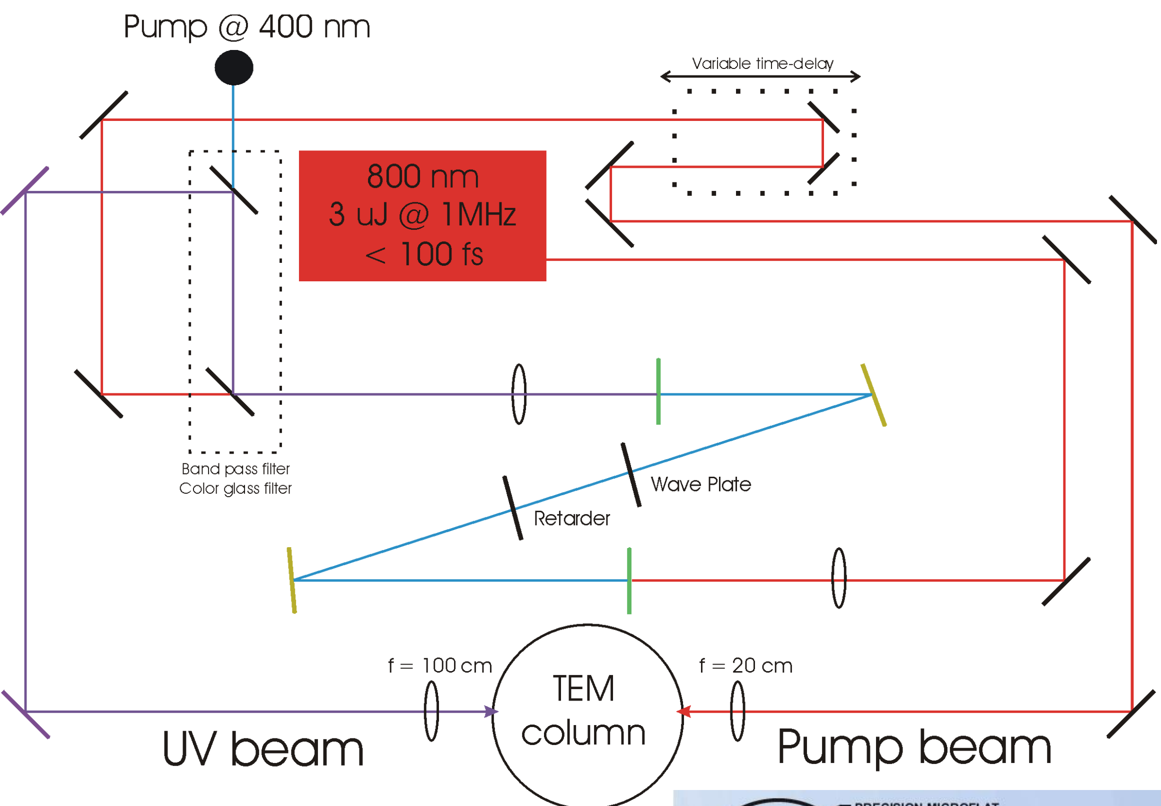
Bright-field and PINEM images of whole unstained, unfixed *E. coli*



(D. Flannigan, PNAS (2010))

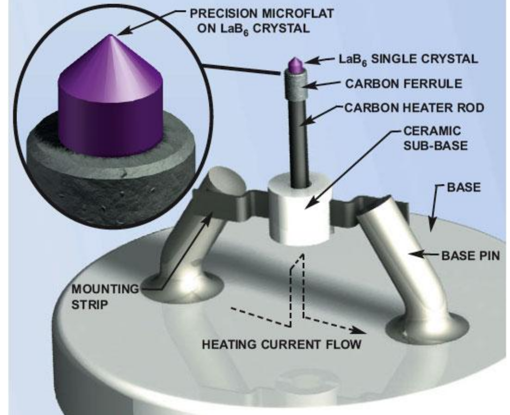
Set-up implementation

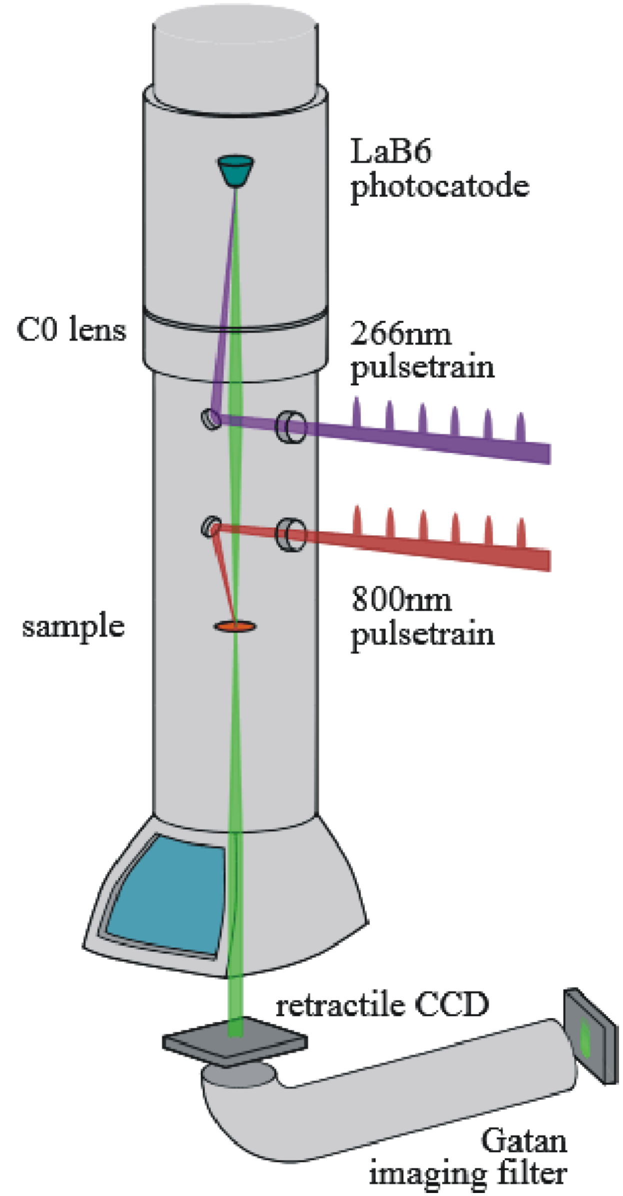
General Introduction: **D-TEM**



ϕ = 2.5 eV

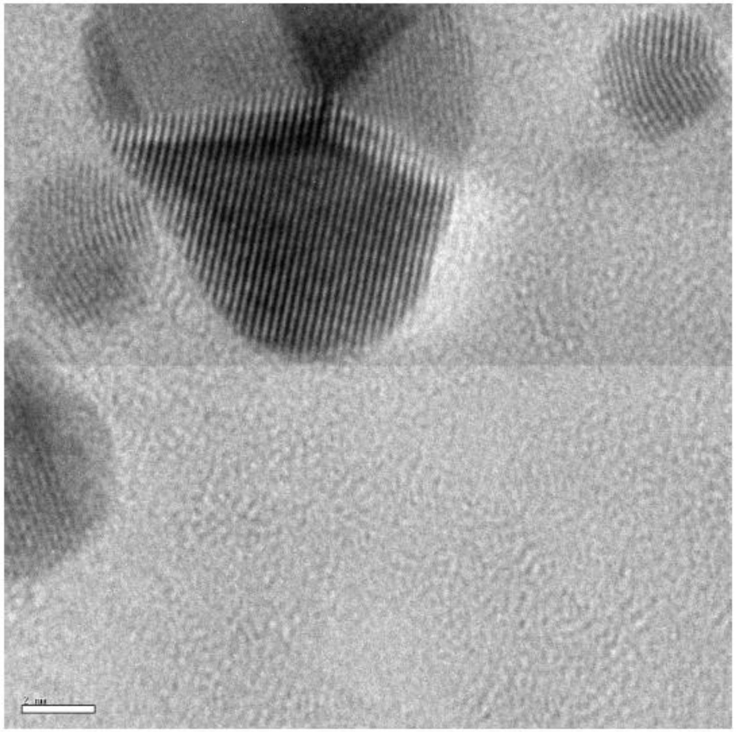
266 nm = 4.6 eV



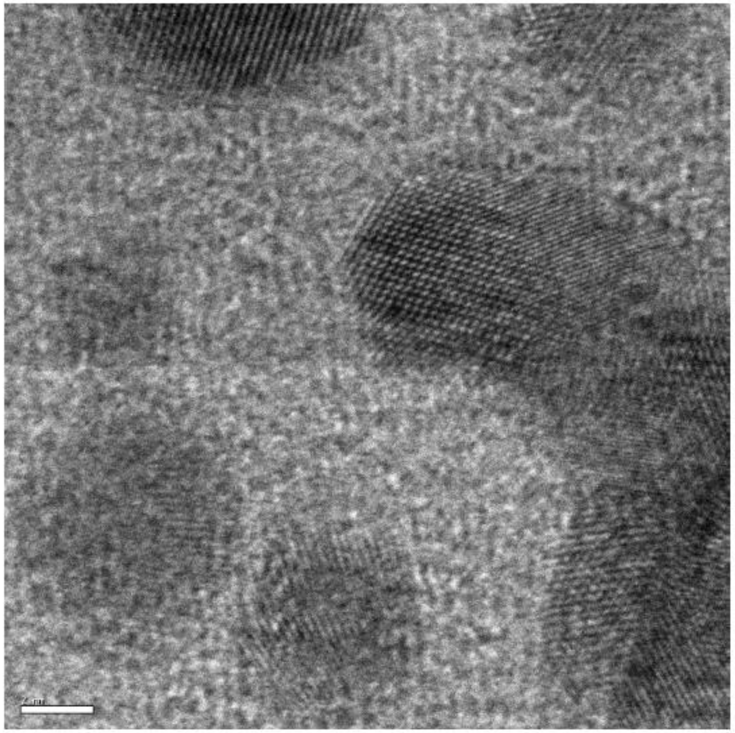


Modified JEOL 2100

Static Performances - spatial resolution

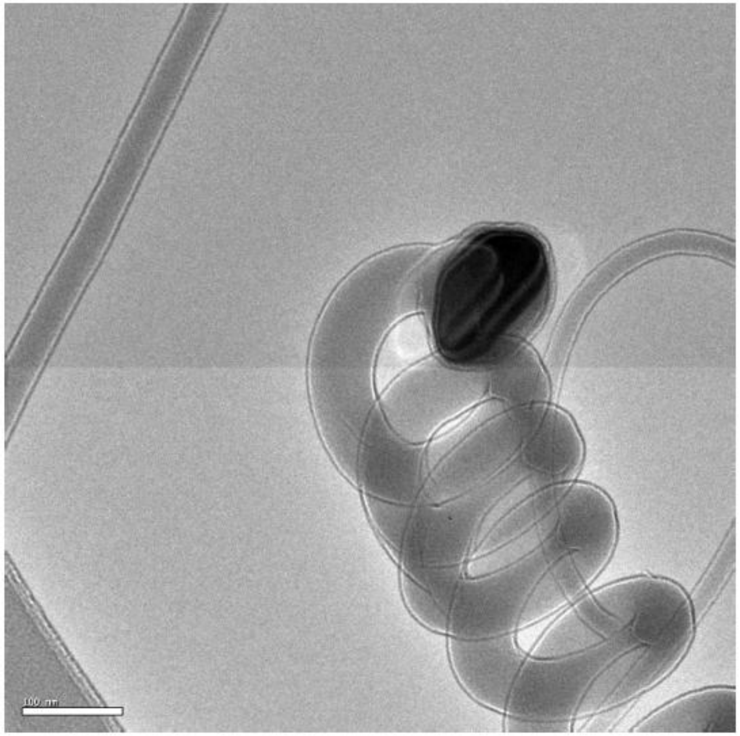


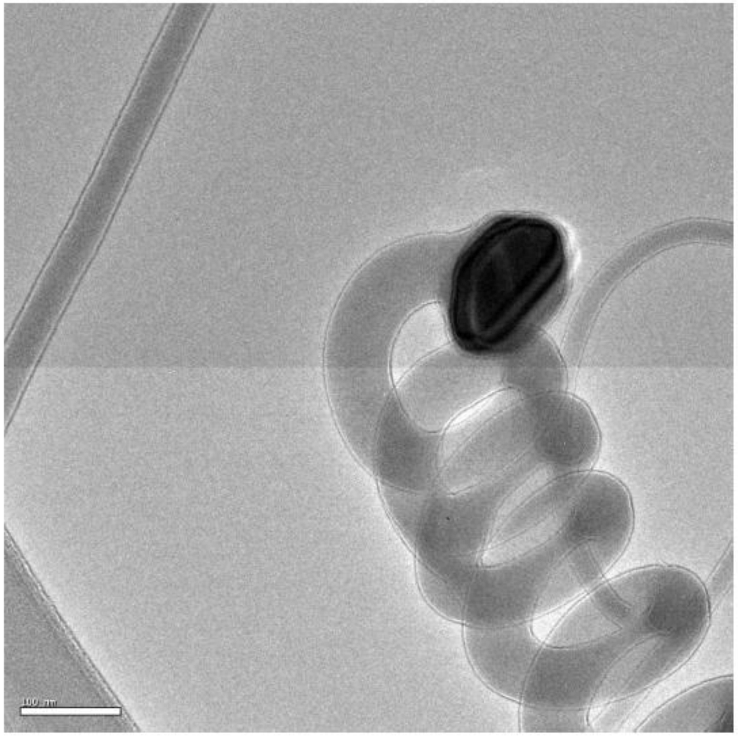
Before modification



After modification

Static Performances - Lorenz Microscopy



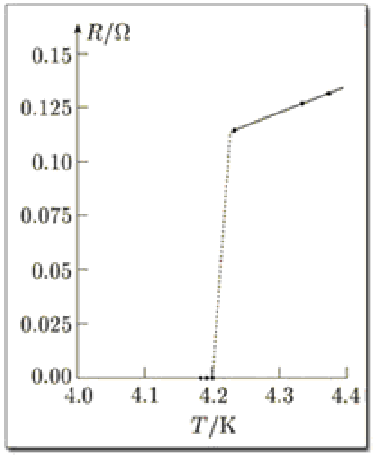


Si nanocoils, Cobalt magnetic head, free lens control

Lorentz imaging of superconducting vortexes

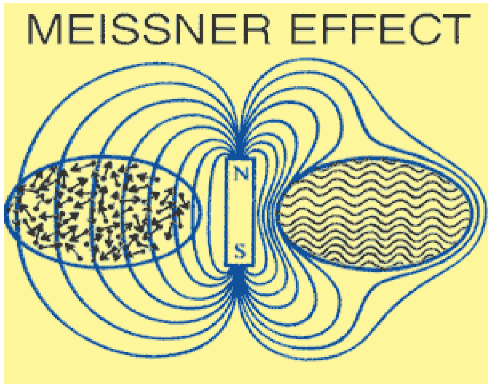
Superconductivity

- **1911:** First observation of superconductivity

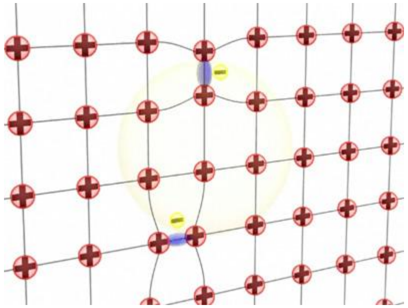


Initial data from Onnes's resistance measurements on mercury showing a precipitous fall in resistance around T_C = 4.2 K

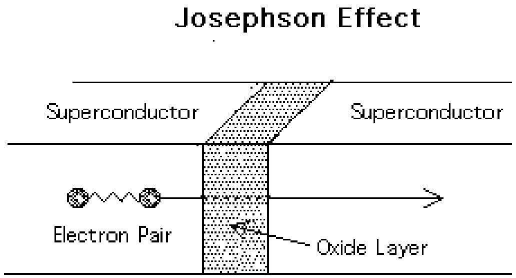
- **1933:** Meissner effect



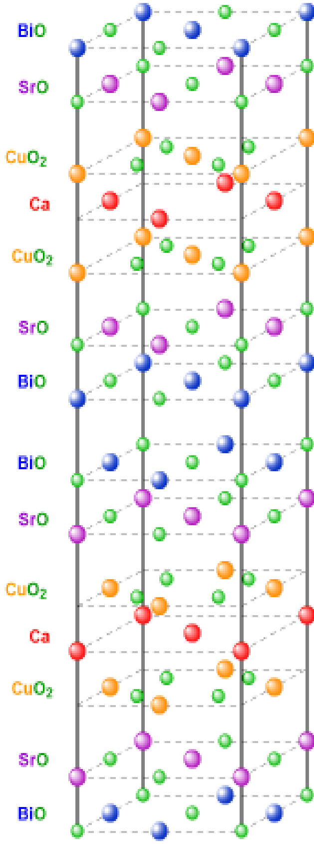
- **1957:** B.C.S. Theory



- **1962:** Josephson effect



- **1986:** Superconductivity in LaBaCuO / High Temperature Superconductors



J. Bardeen *et al*. Phys. Rev. (1957)
B.D. Josephson Physics Letters (1962)
A. Schilling *et al*. Nature (1993)

D-TEM investigation of superconductors across the phase transition:

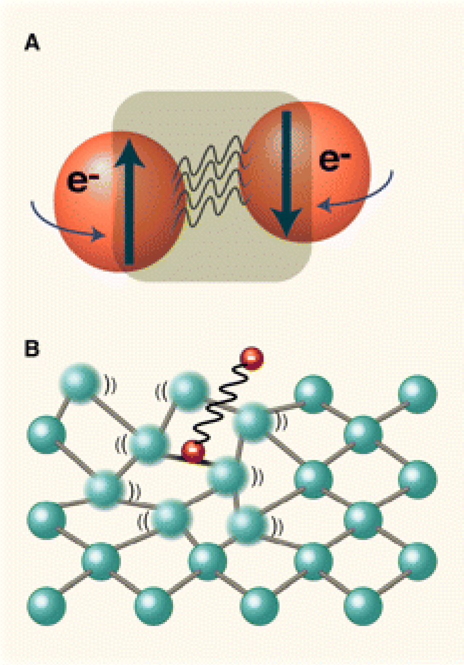
Dynamical electronic properties at *q=0* (ultrafast optics)
Dynamical electronic properties at *q≠0* (ultrafast EELS)
Dynamical structural properties (ultrafast electron diffraction)
Vortex dynamics (ultrafast imaging)

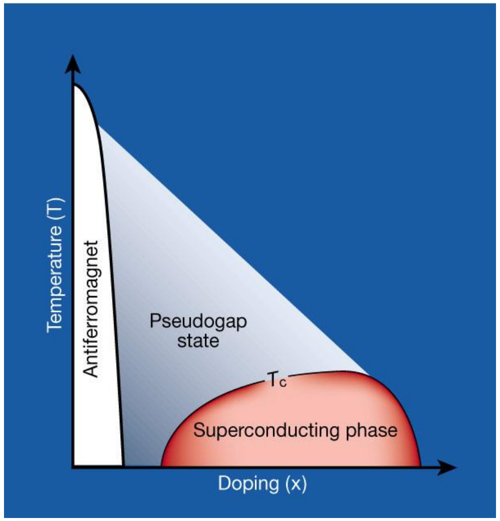
Quantitative information on:

Electron-phonon coupling parameter
Pair breaking and recombination dynamics
time-evolution of the superfluid density (vortexes)

Understanding:

Pairing mechanism
Phase diagram



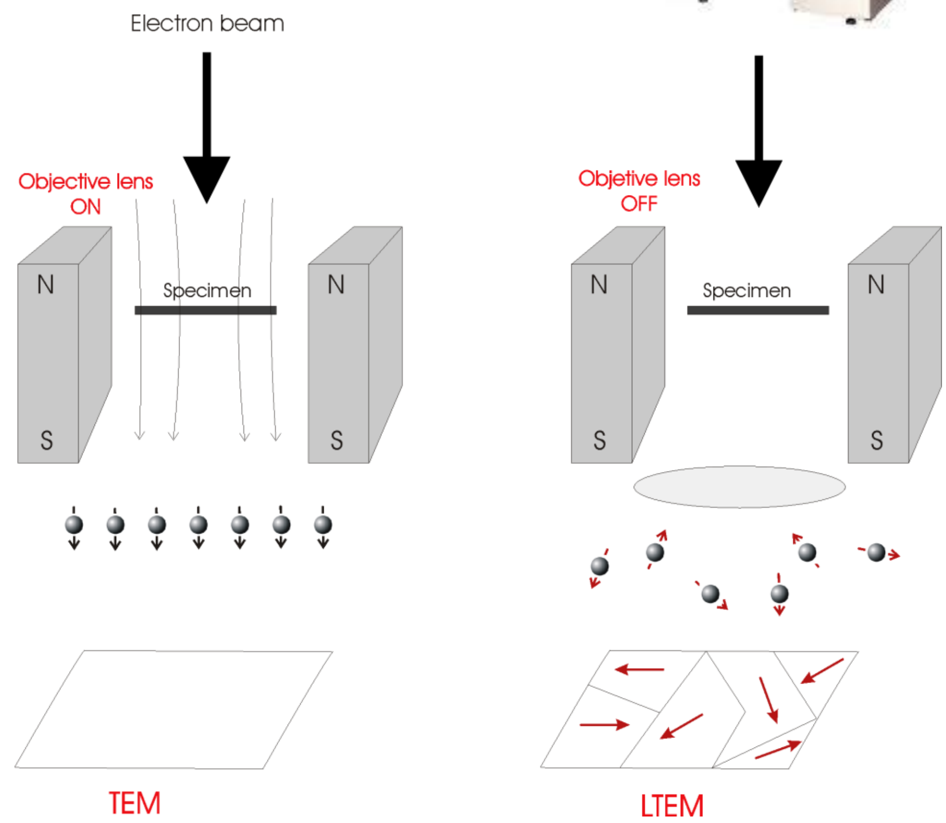


Lorentz Microscopy:

<u>Lorentz microscopy = Phase contrast microscopy</u>

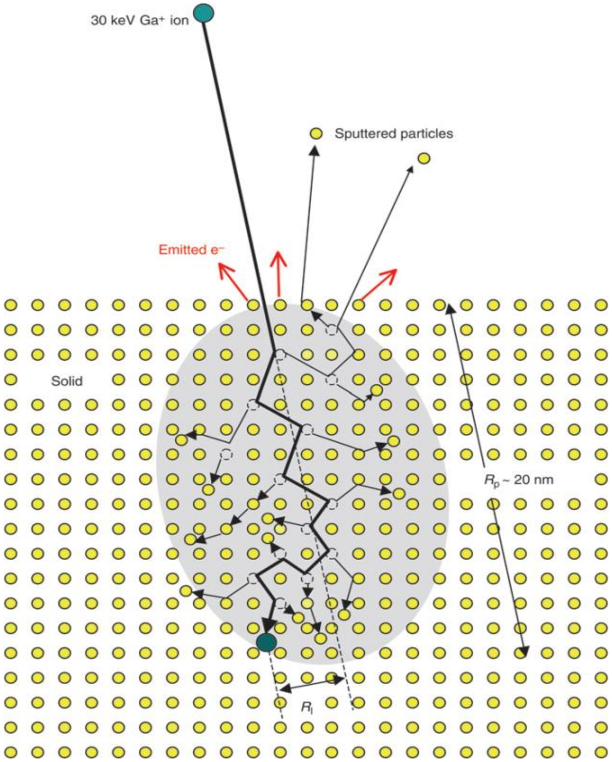
Foucault mode:
Electron beam deviated by domains
Splitting of diffraction spots
Quantitative analysis
In focus

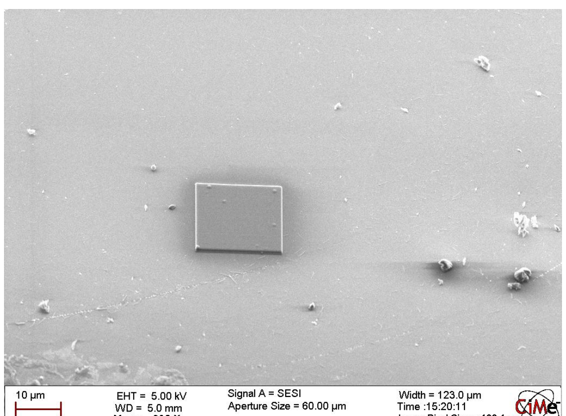
Fresnel mode:
Domain walls rather than domains
Out of Focus

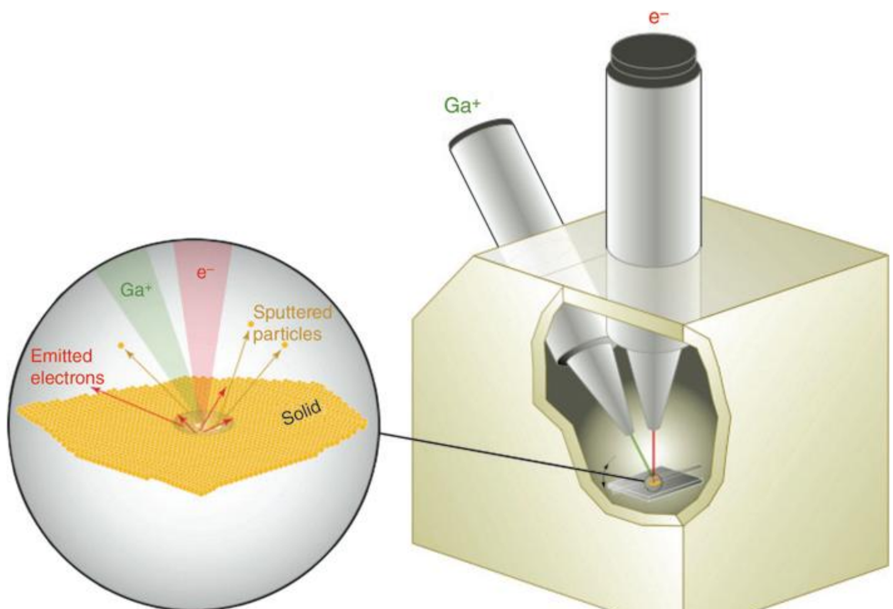


D.B. Williams and C. Barry Carter – Transmission Electron Microscopy – Springer

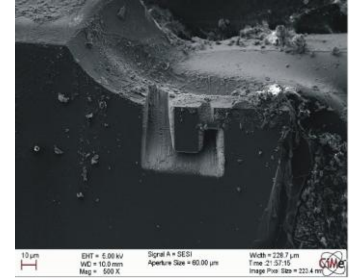
Sample preparation: MgB2

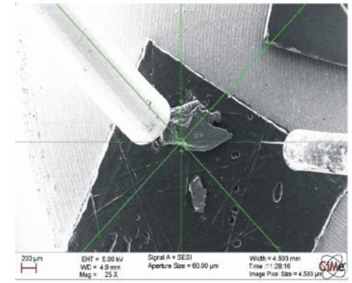


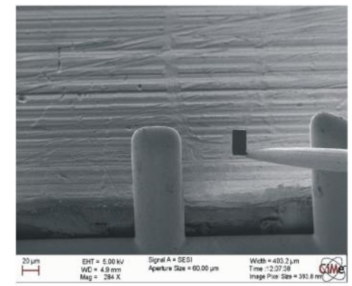


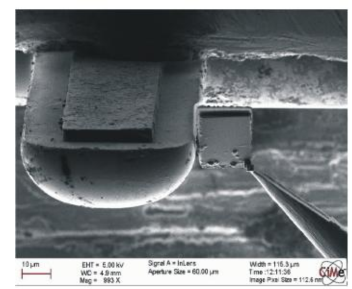


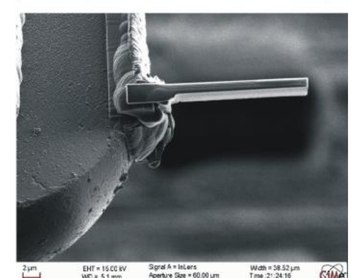
Schematic illustration of a dual-beam FIB–SEM instrument



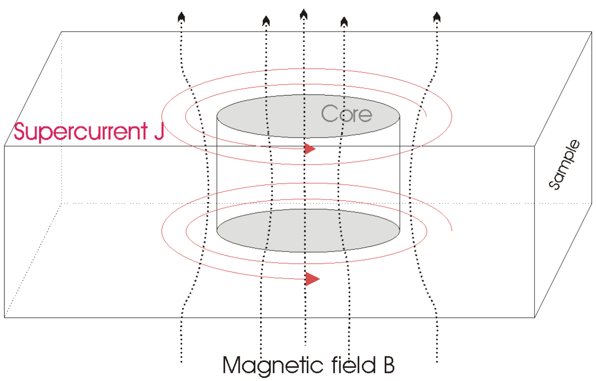


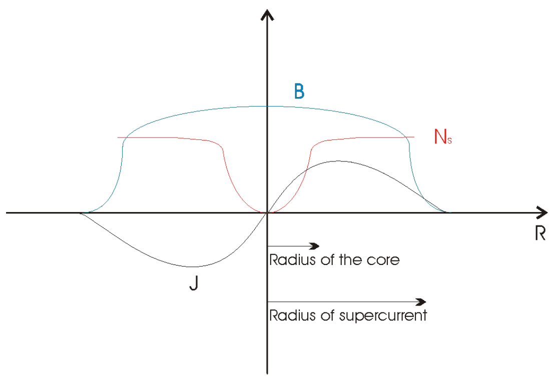


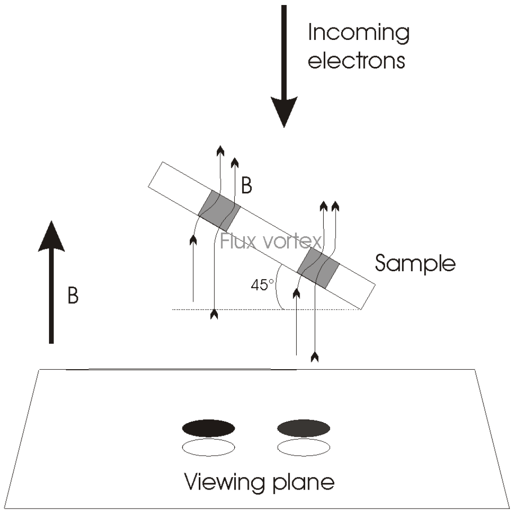


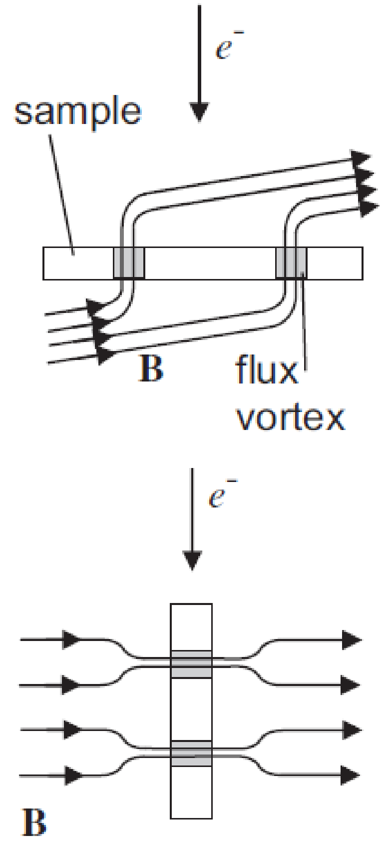


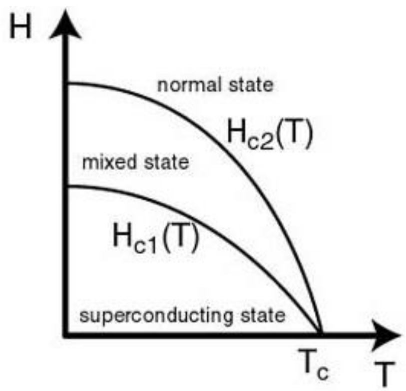
Imaging Vortices











J.C. Loudon *et al*. Physica C (2011)

First results: **Vortices in MgB_2**

J.C. Loudon *et al*. Physica C (2011)

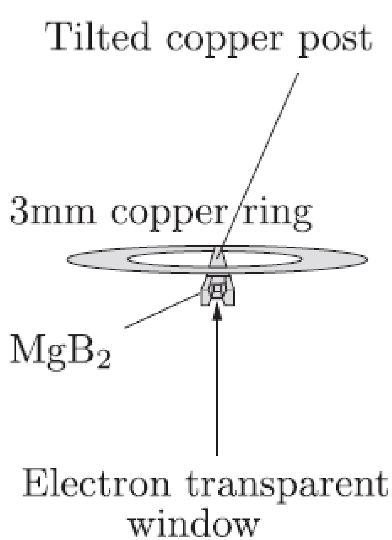
Philips CM300 @ 300kV

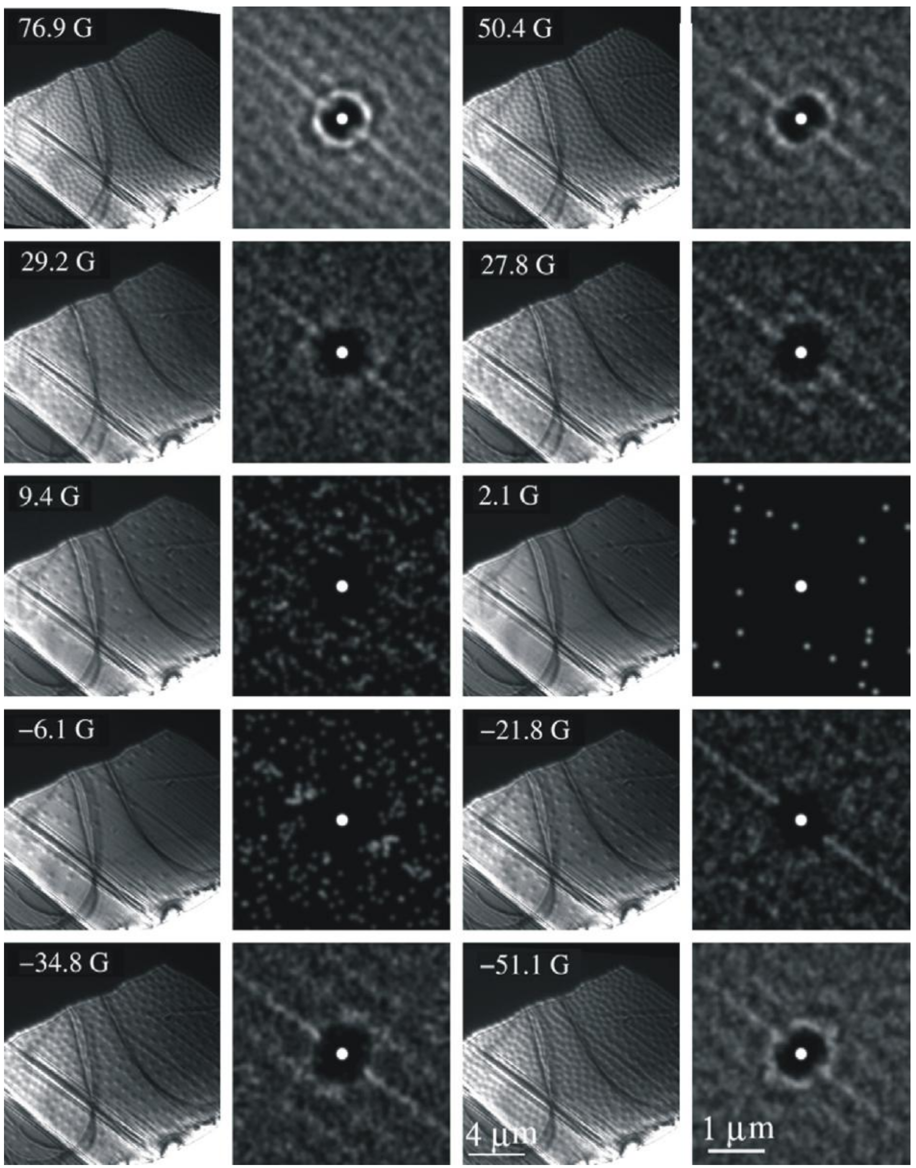
250nm in the *c*-direction (FIB)

T = 10.8K

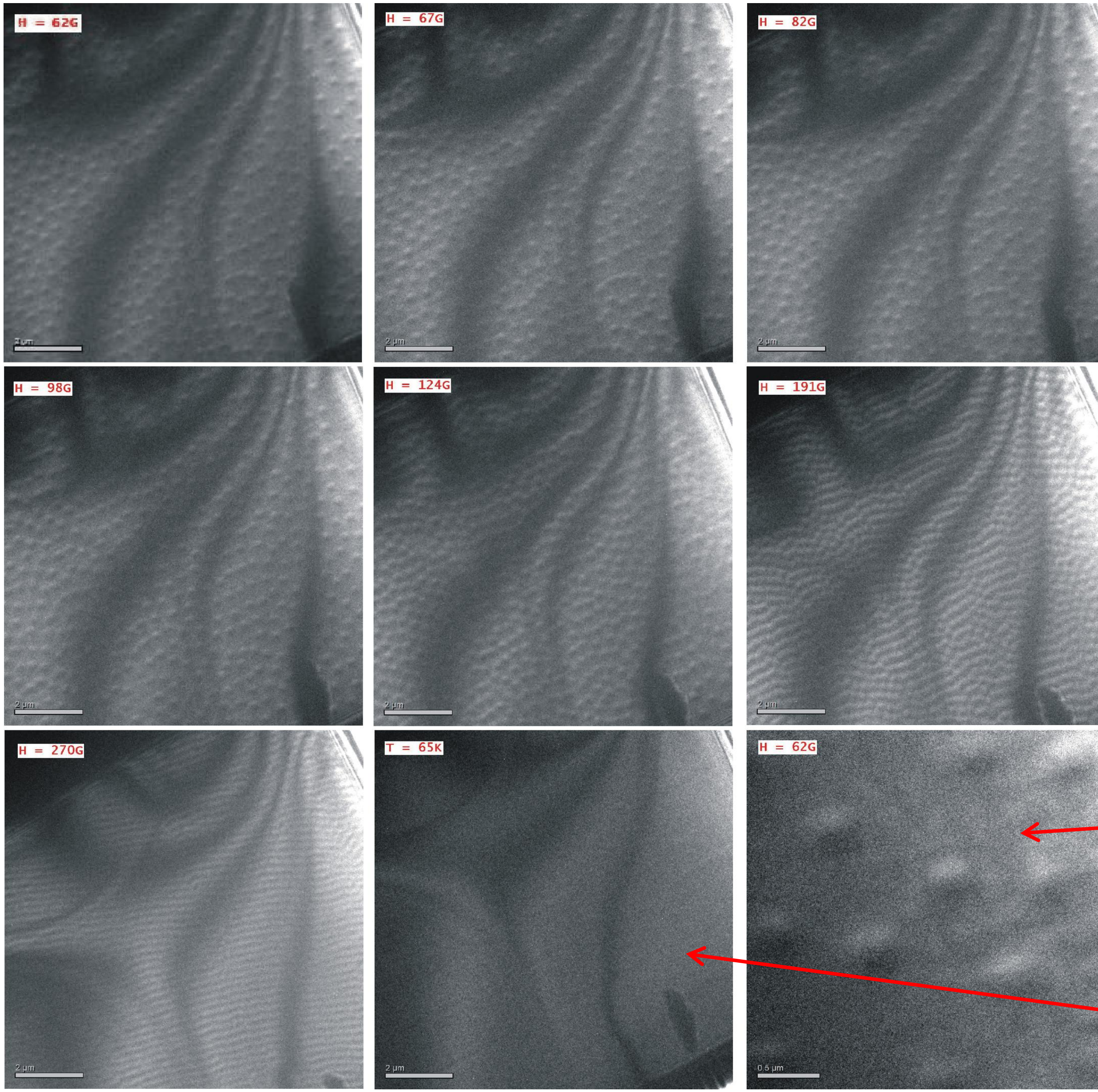
Attached to copper post (α = 45° ± 1°)

Longitudinal undulations: pinning landscape





Vortices in MgB_2



Microscope: JEOL 2200FS
Magnification: 2000
Imaging mode: LowMag
Probe size: 2nm
Voltage: 200kV

T = 5K

$$\emptyset_0 = \frac{h}{2e} = 2.07\ 10^{-15}\ W$$

$$H = n\emptyset_0$$

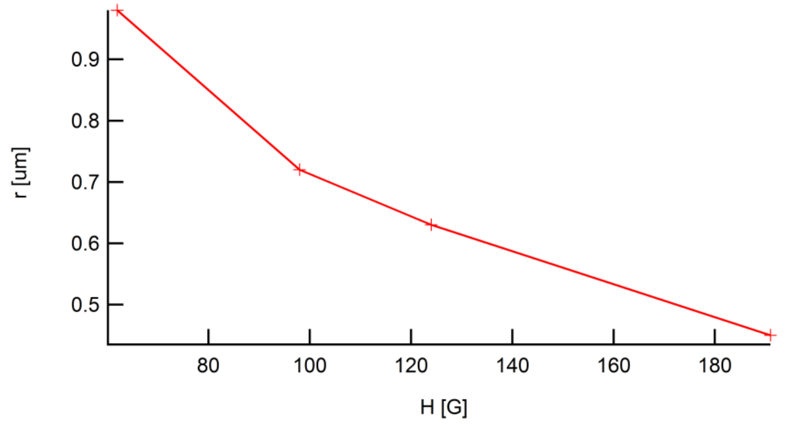
$$H_{\perp} = \frac{\sqrt{2}}{2}H \qquad (\alpha = 45°)$$

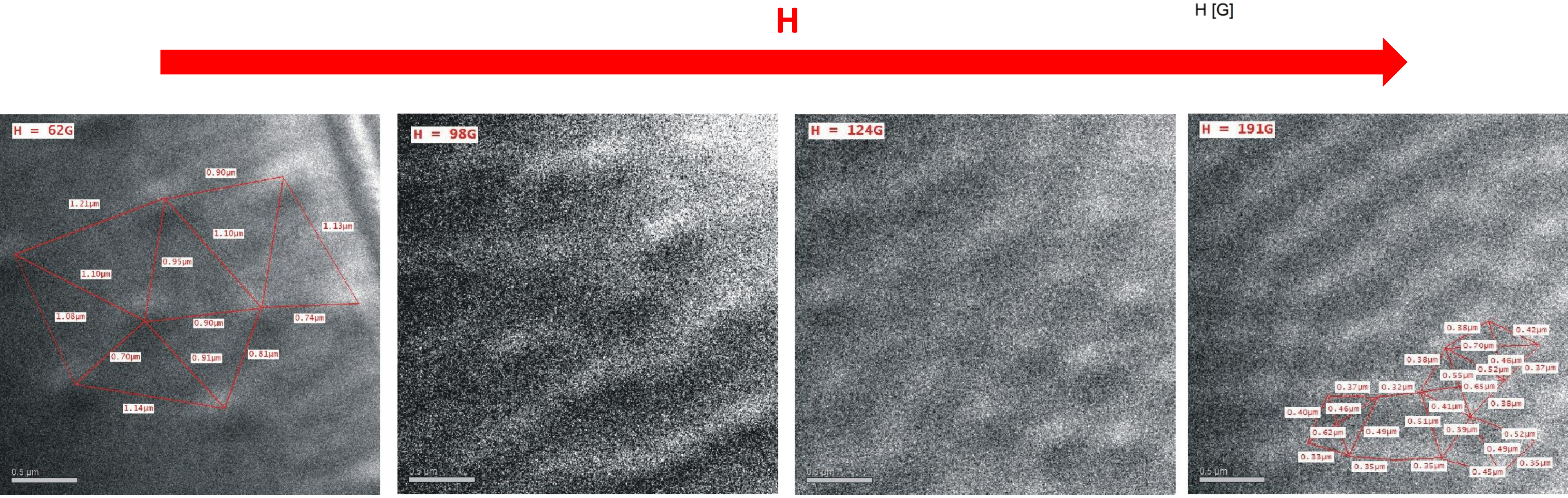
Magnification: 8000

T = 65K

First results: **Vortices in MgB_2**

H [G]	62	98	124	191
r [µm]	0.98	0.72	0.63	0.45





The Abrikosov lattice in MgB_2

C-H. Sow *et al*. Phys. Rev. L.ett. (1997)
D.R. Nelson et al. Phys. Rev. B. (1979)

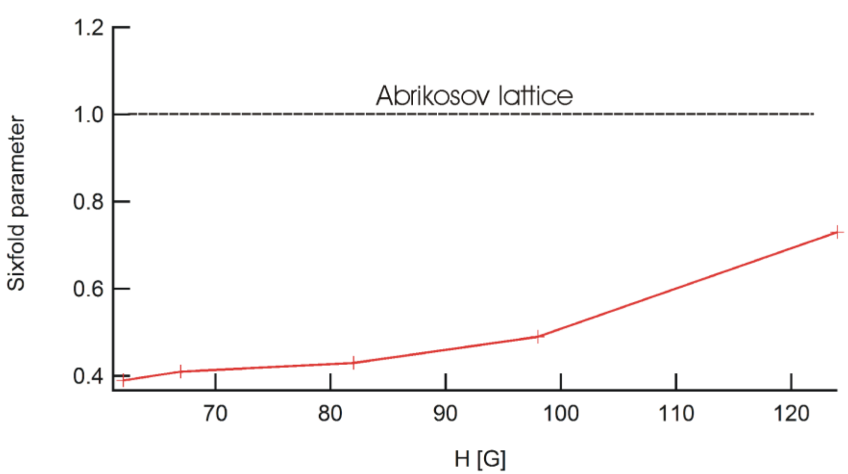
The sixfold order parameter: deviation from Abrikosov lattice

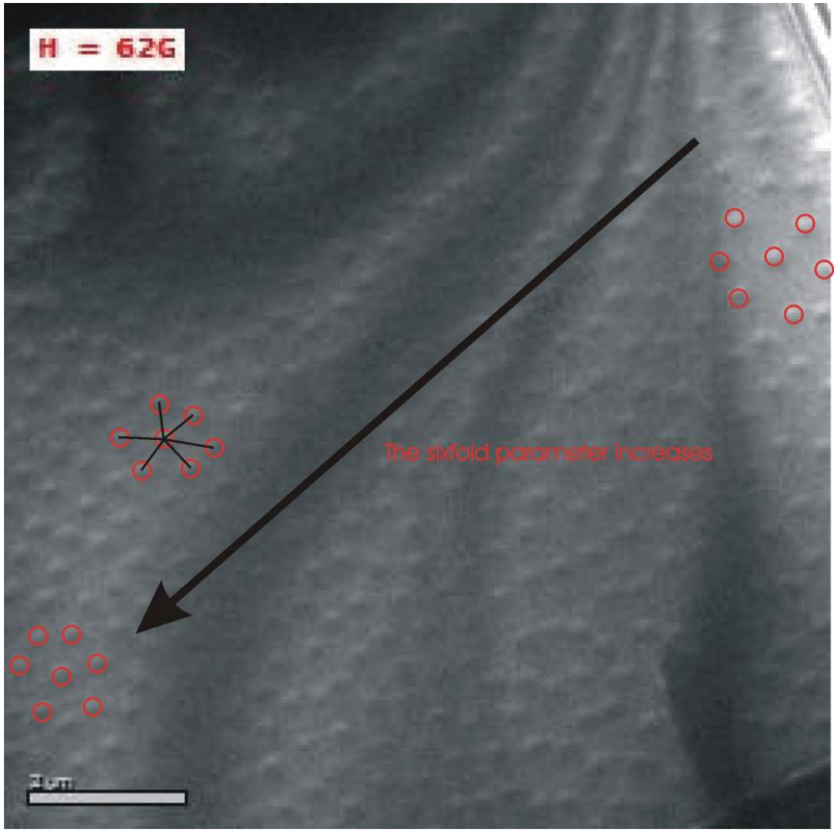
$$\Psi_6(r_i,t) = \frac{1}{n_i}\sum_{j=0}^{n_i} \exp(6i\theta_{ij})$$

$$\cos(6 * 60°) = \cos(360°) = 1 \qquad \textit{Real part}$$

$$\Psi_6(r_i,t) = 1 \qquad \textit{Abrikosov lattice}$$

Ψ_6	0.39	0.41	0.43	0.49	0.73
H [G]	62	67	82	98	124





Conclusion and future planning

Conclusion

- Direct observation of the formation of the Abrikosov lattice in MgB_2
- Possibility to study liquid/solid transition by following the single costituents in real space and time (msec)
- Possibility to follow the phase transition along the H axis of the phase diagram

Future planning

- Ultrafast study of the Abrikosov lattice dynamics (ns to fs)
- Spanning the phase diagram of supercondutors as a function of photoexcitation, temperature, magnetic field and chemical composition

Acknowledgements

LUMES

Laboratory for
Ultrafast Microscopy and
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- Eugenia Minikus

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- Bryan Reed
- Dan Masiel

- Brett Barwick

IDES INTEGRATED DYNAMIC ELECTRON SOLUTIONS

CIME Centre Interdisciplinaire de Microscopie Électronique

PAUL SCHERRER INSTITUT PSI

SIGILL. COLL. TRIN. SANC. PRO ECCLESIA ET PATRIA