Electron holography of magnetic fields in nanostructured materials and devices

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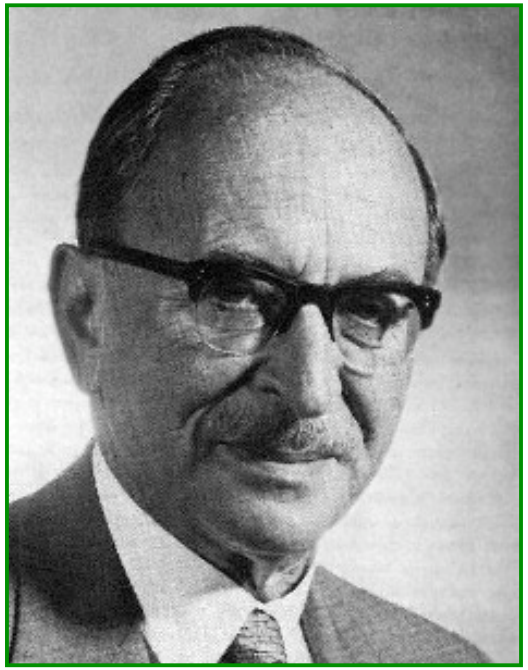
Outline

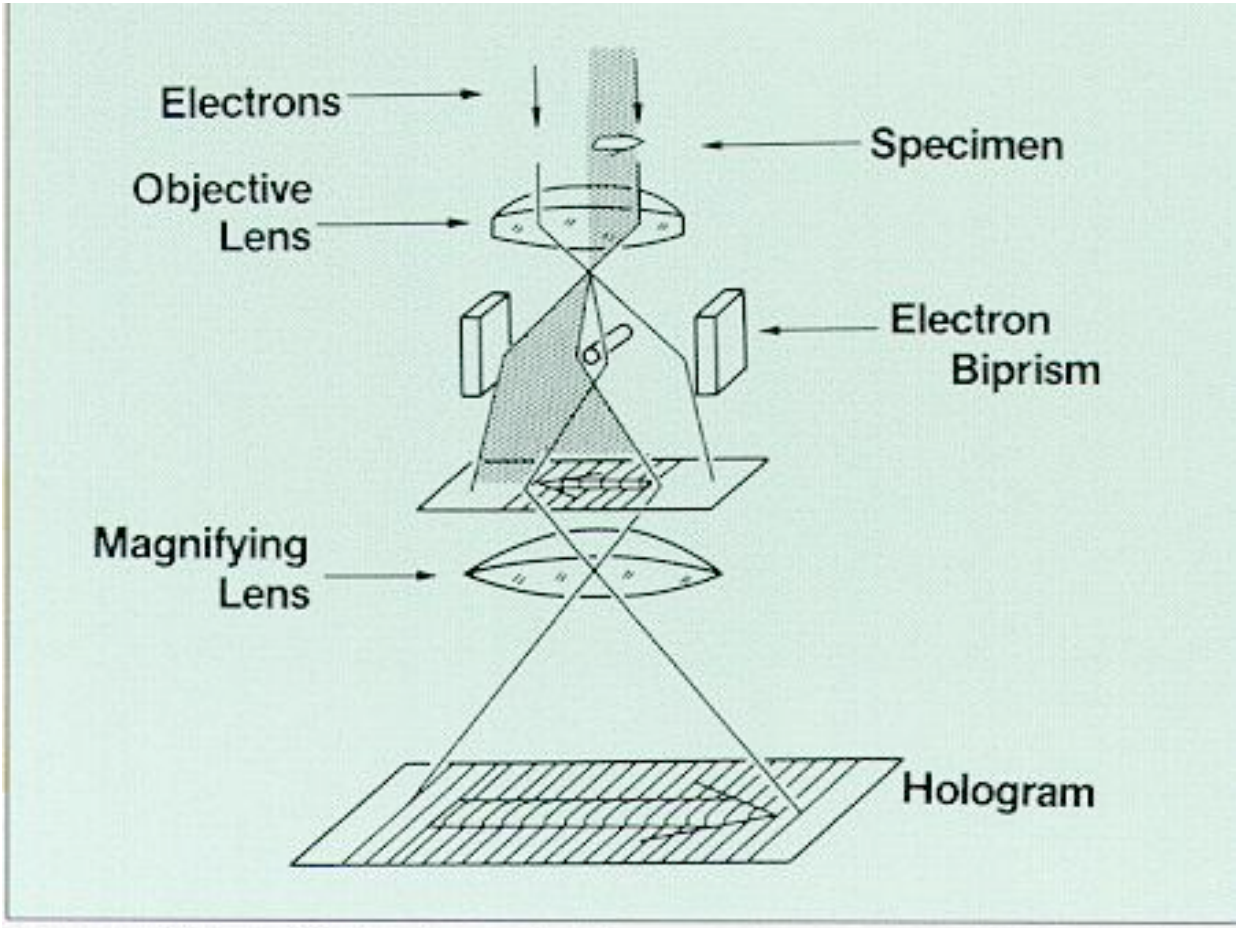
1. Brief introduction to off-axis electron holography
2. Electron holography of magnetic fields:
   - Magnetic nanocrystals
   - Magnetic devices
   - Low temperature measurements
3. Discussion:
   - Spatial resolution
   - Advantages and limitations
4. Future prospects

The origins of electron holography

1947 **Denis Gabor proposes holography ("whole writing") as a means to correct for electron microscope aberrations- Nobel prize 1971.**

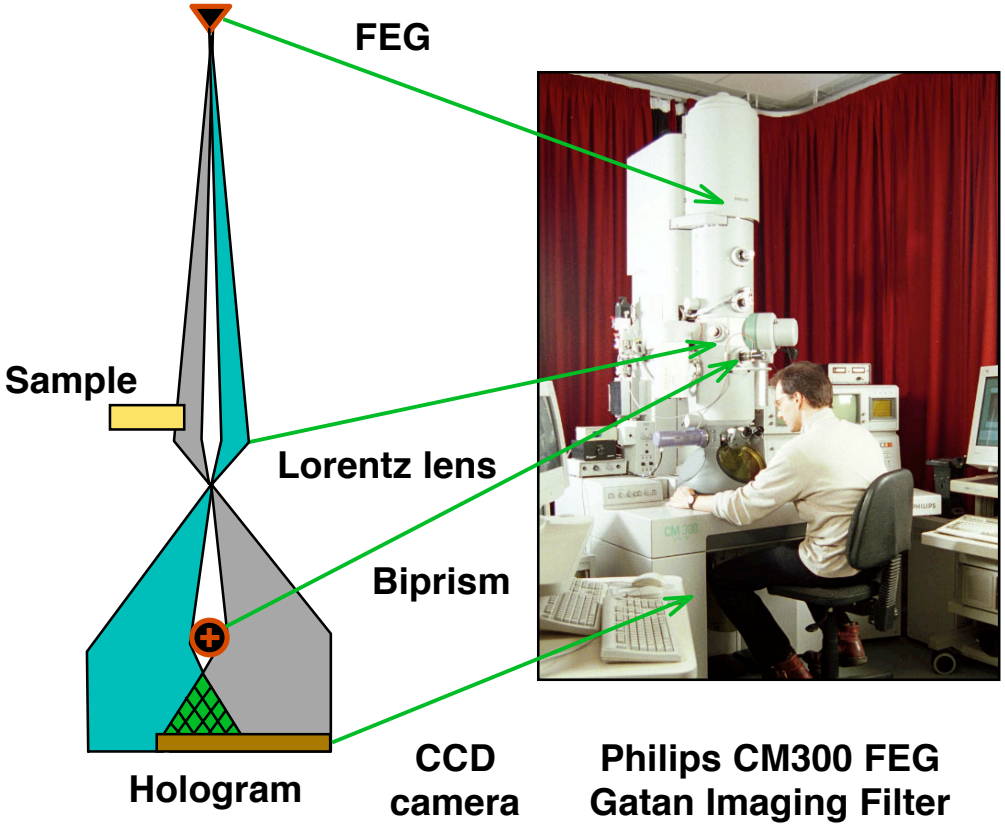


Electron holography



Formation of electron hologram

Off-axis electron holography



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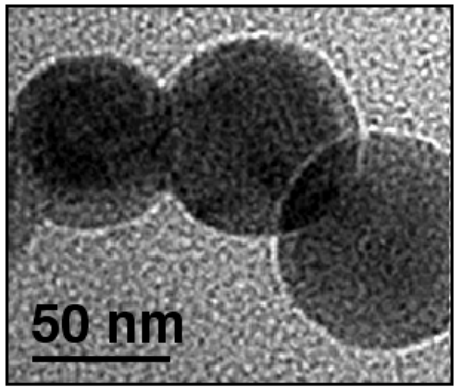
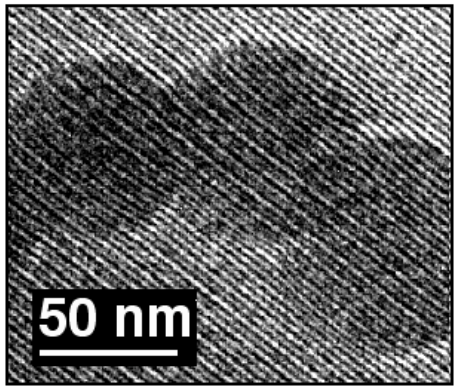
Electron holography





Off-axis electron hologram

If V(x) and B(x) do not vary in the incident electron beam direction, then :

Phase shift:

$$\phi(x) = C_E V(x) t(x) - \left(\frac{e}{\hbar}\right) \int B_{\perp}(x) t(x) dx$$

$$C_E = \left(\frac{2\pi}{\lambda}\right)\left(\frac{E + E_0}{E(E + 2E_0)}\right)$$

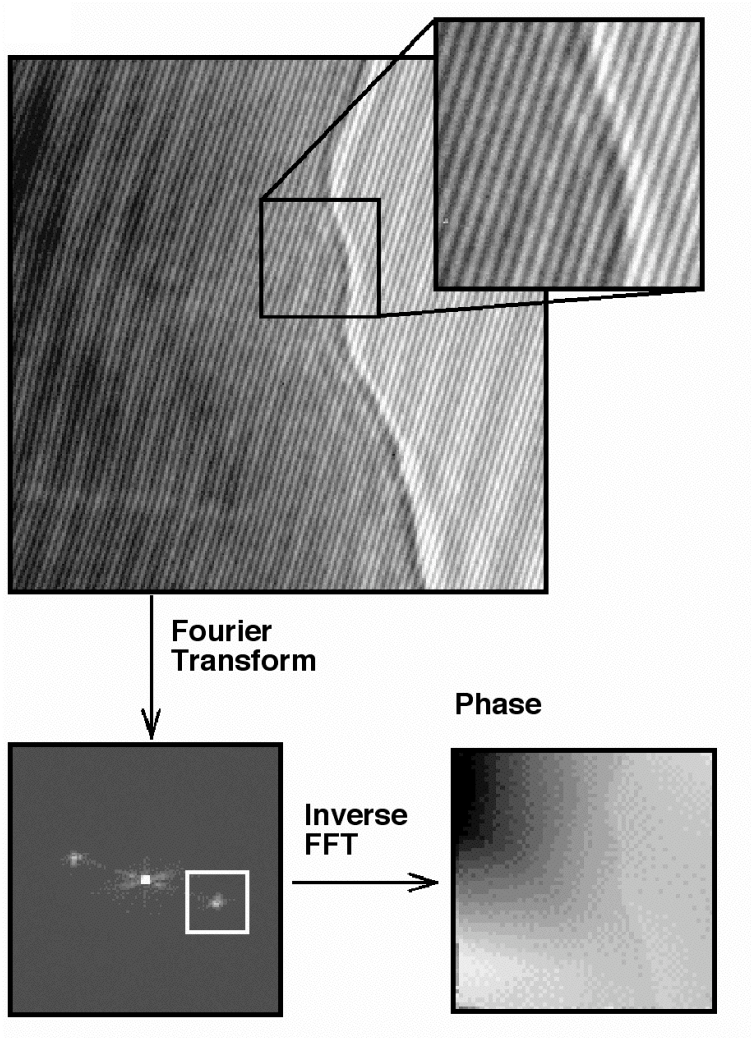
Sensitive to: **magnetic fields**

composition
density
bonding/ ionicity
electrostatic fields at depletion layers
electrostatic fringing fields outside materials

Digital reconstruction

Off-axis electron hologram from thin crystal showing interference fringes within sample

Phase image obtained from inverse Fourier transform of one 'sideband' selected from Fourier transform of hologram



Acknowledgment: M. R. McCartney

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Basis of off-axis electron holography

Why is electron holography useful?

Conventional bright-field TEM:

$$\psi(r) = A(r)\exp[i\phi(r)]$$

$$I(r) = |A(r)|^2$$

Off-axis electron holography:

$$I_{hol}(r) = |\psi(r) + \exp[2\pi i q_c.r]|^2$$

$$I_{hol}(r) = 1 + A^2(r) + 2A(r)\cos[2\pi q_c.r + \phi(r)]$$

$$FT[I_{hol}(r)] = \delta(q) + FT[A^2(r)] + \delta(q+q_c)\otimes FT[A(r)\exp[i\phi(r)]] + \delta(q-q_c)\otimes FT[A(r)\exp[-i\phi(r)]]$$

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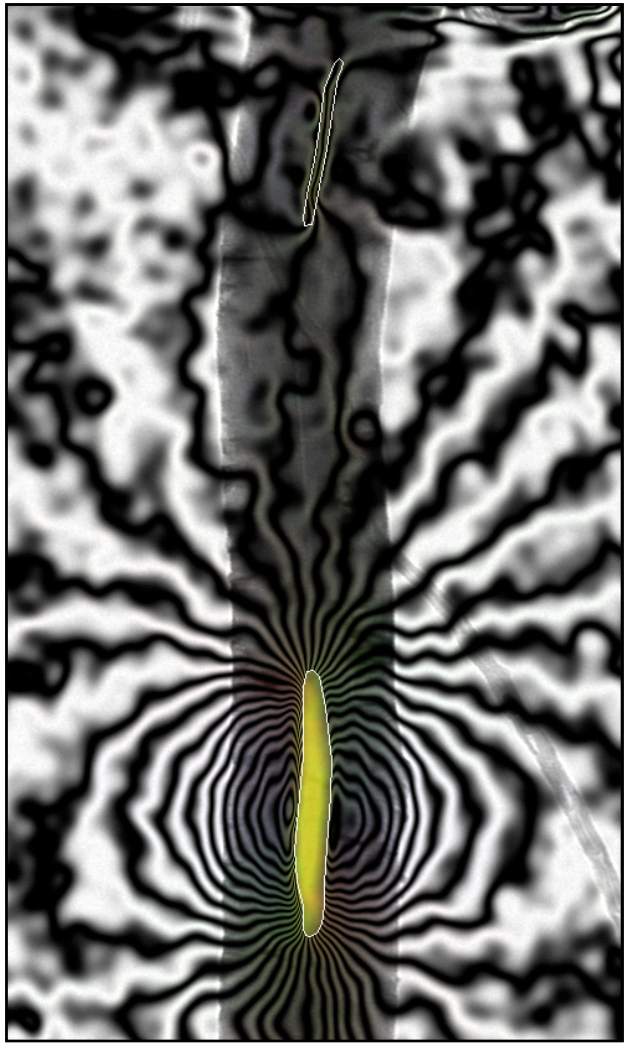
Multi-walled carbon nanotubes containing Fe



Krzys Koziol, Alan Windle

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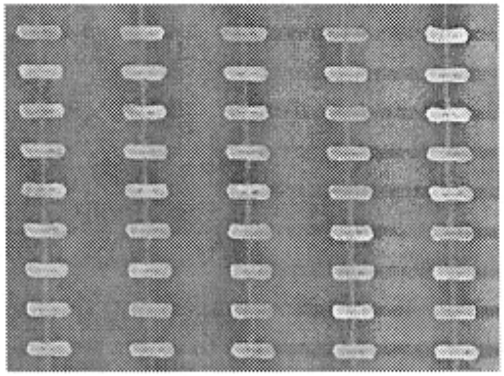
Quantitative measurements using electron holography

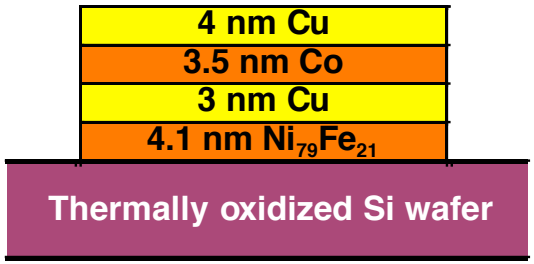
1. Magnetization
2. Coercivity
3. Magnetic moment
4. Critical sizes for SPM/ SD/ PSD/ MD transitions
5. Effects on magnetic microstructure of interactions, shape and crystallography

Magnetic nanostructures

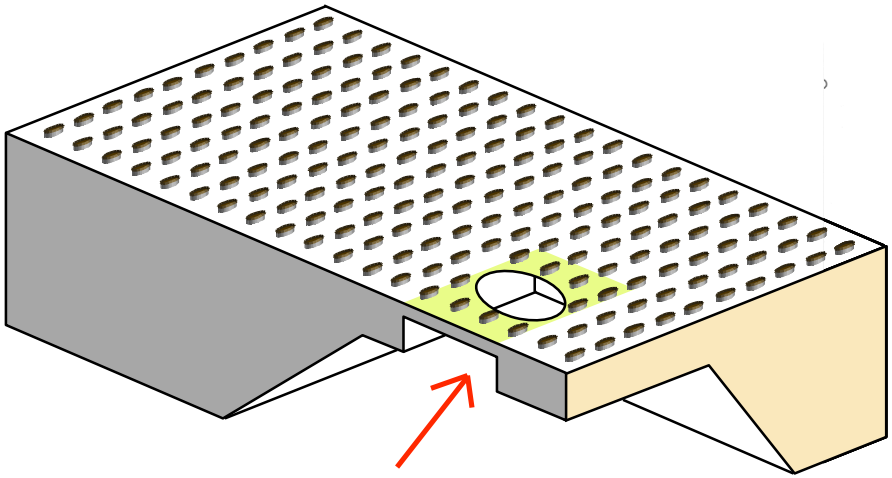
250 x 80 nm pesudo spin valve elements
fabricated on Si using interferometric lithography and examined in plan view



1 μm



Cross-section of each element



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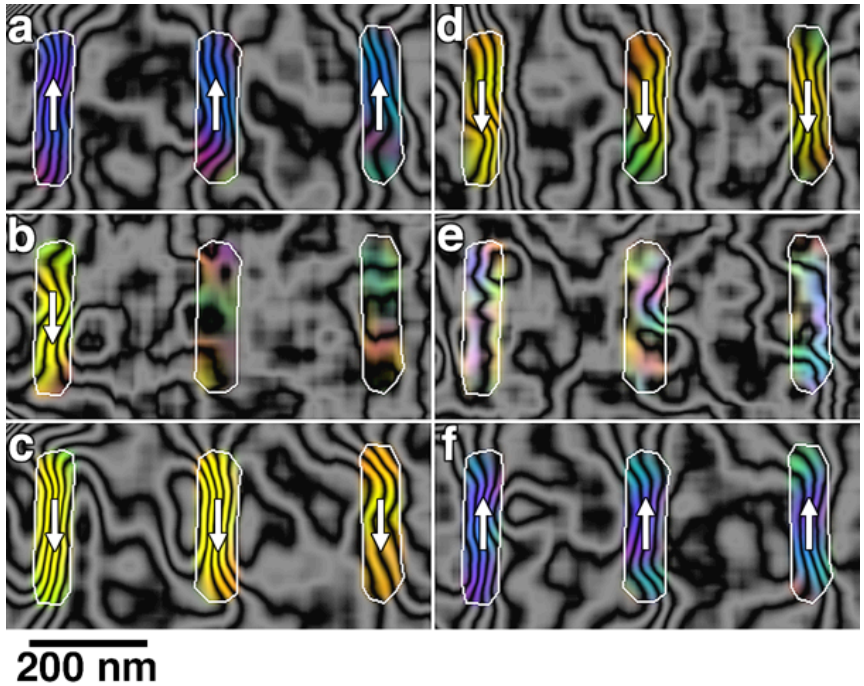
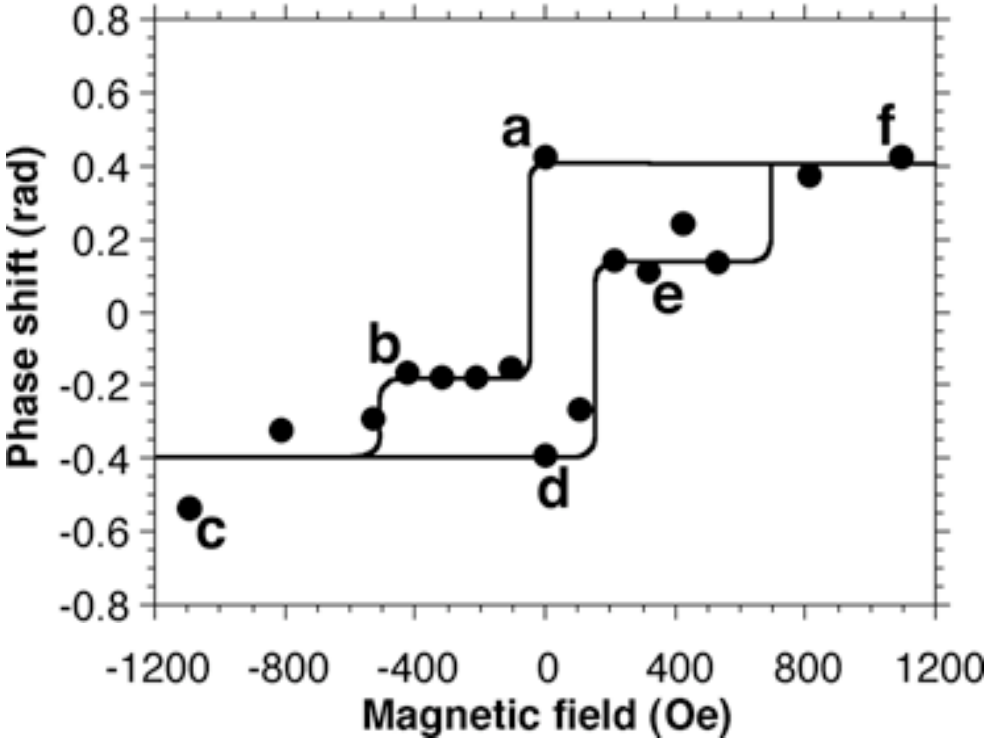
Magnetic nanostructures

Magnetic remanent states

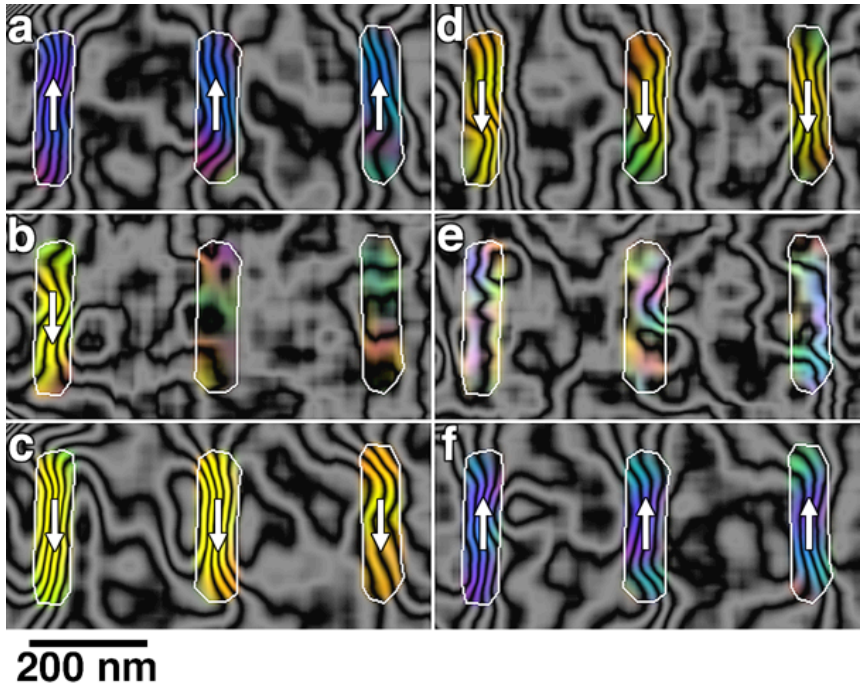


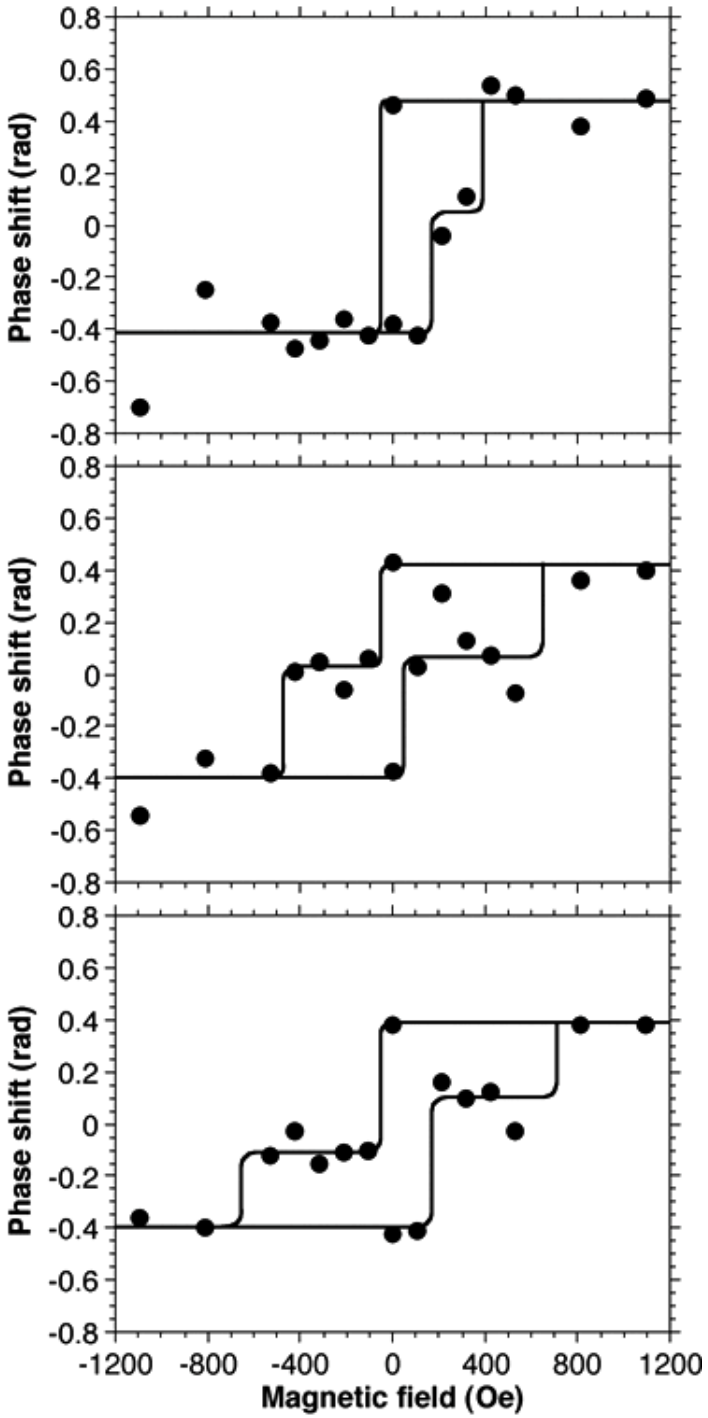
Average remanent hysteresis loop



Magnetic nanostructures

Remanent hysteresis loops from individual elements





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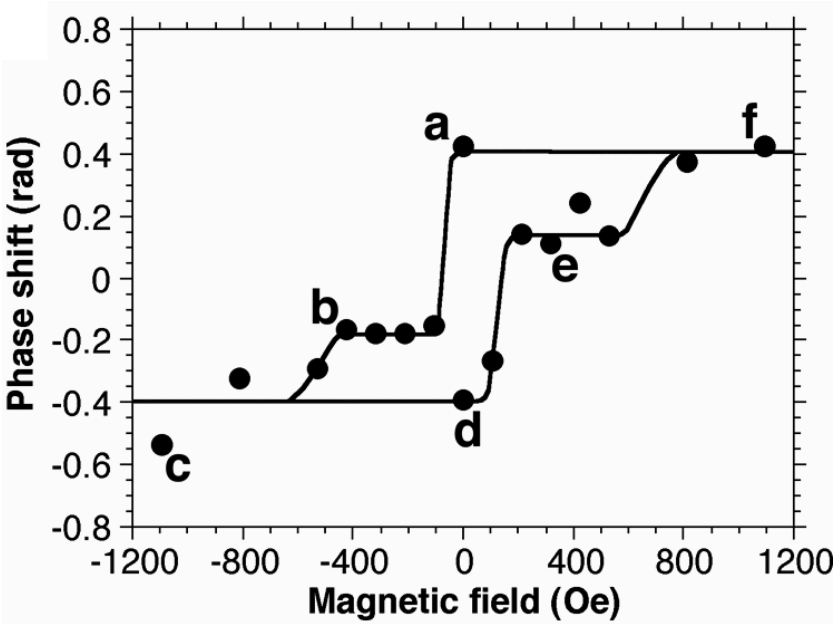
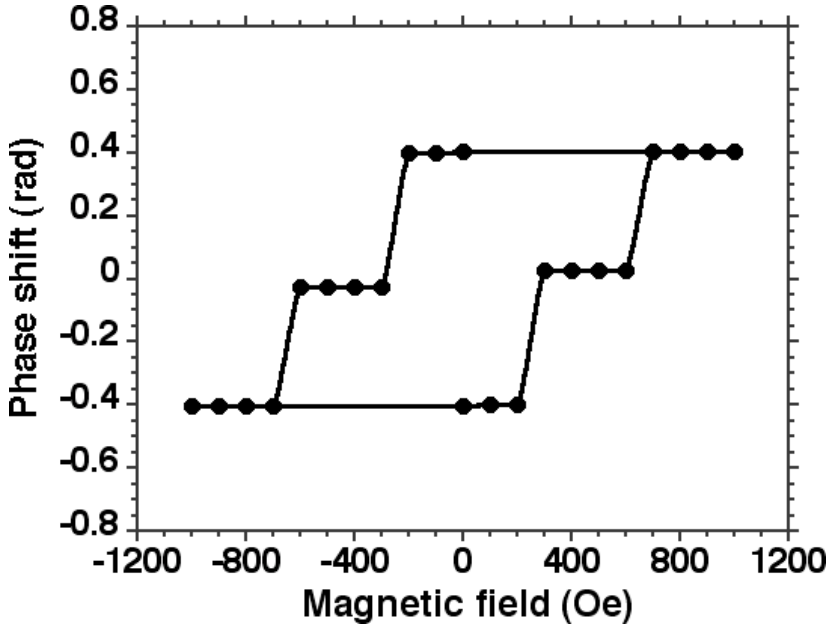
Magnetic nanostructures

Average remanent hysteresis loop



Simulated remanent hysteresis loop



Nominal parameters:
Element size 250 x 80 nm
NiFe layer thickness 4.1 nm
Co layer thickness 3.5 nm

Best-fitting parameters to measurements:
Element size 200 x 38 nm
NiFe layer thickness 4 nm
Co layer thickness 2 nm

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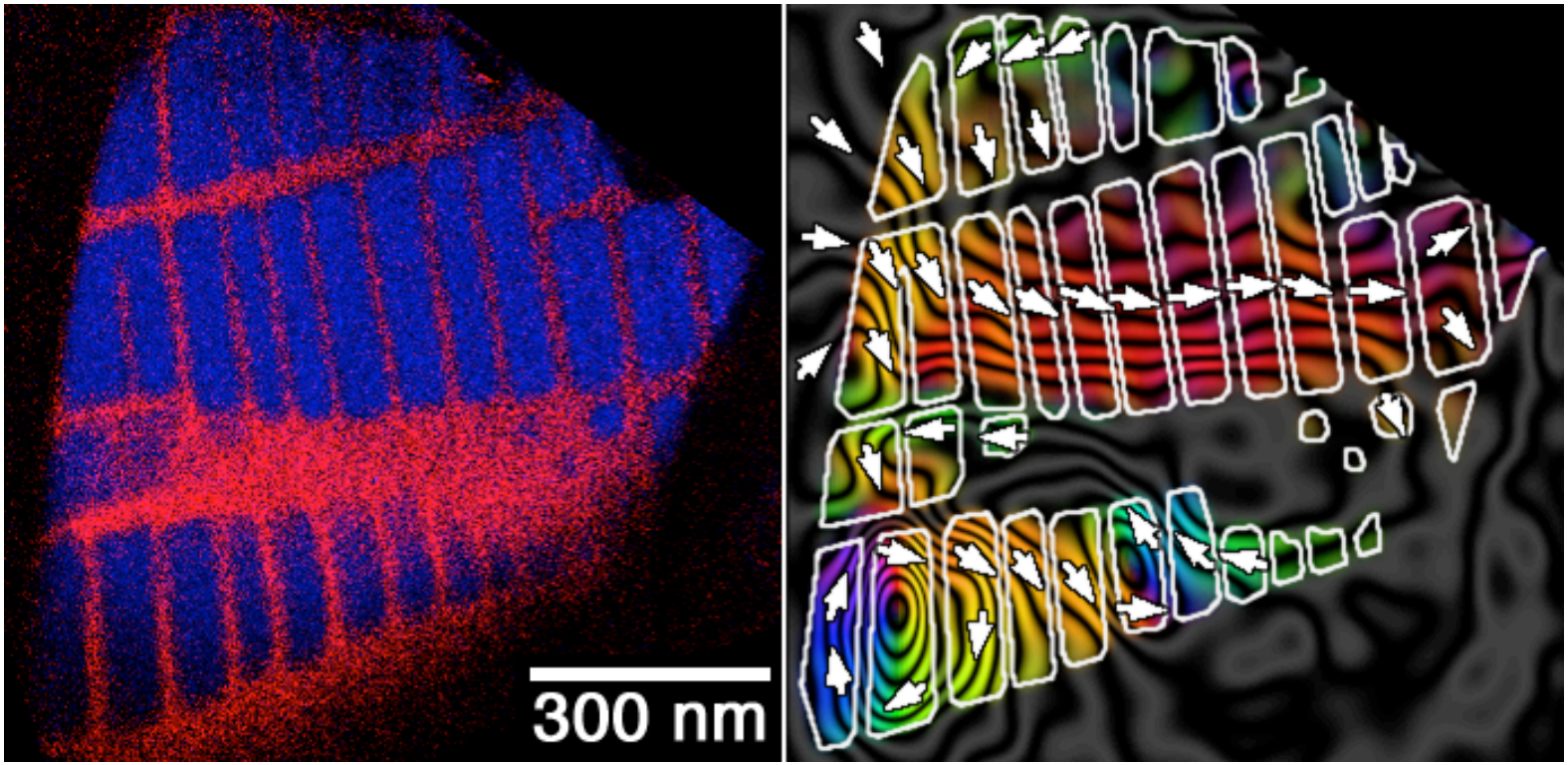
Naturally occurring magnetic crystals

Naturally occurring crystals: rock magnetism



Blue: magnetite (Fe_3O_4)
Red: ulvöspinel (Fe_2TiO_4)

Magnetic remanent state after saturating sample

Richard Harrison, Josh Feinberg

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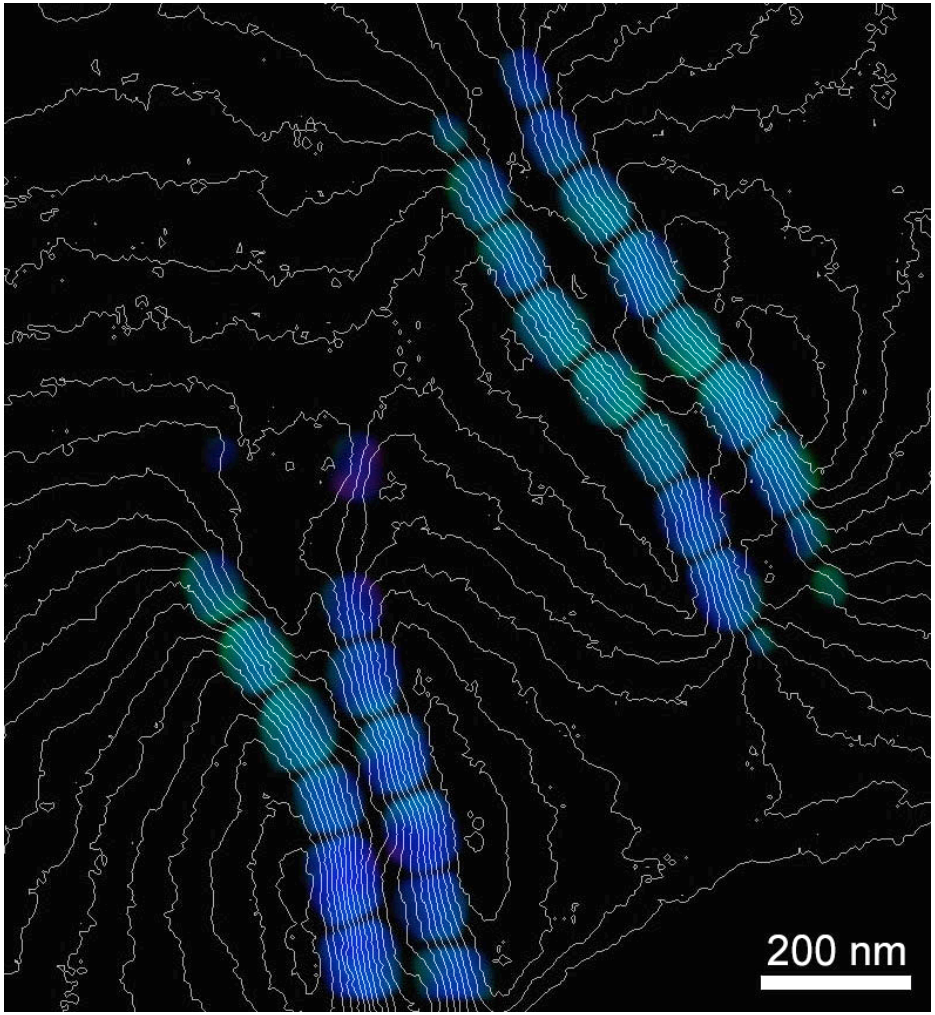
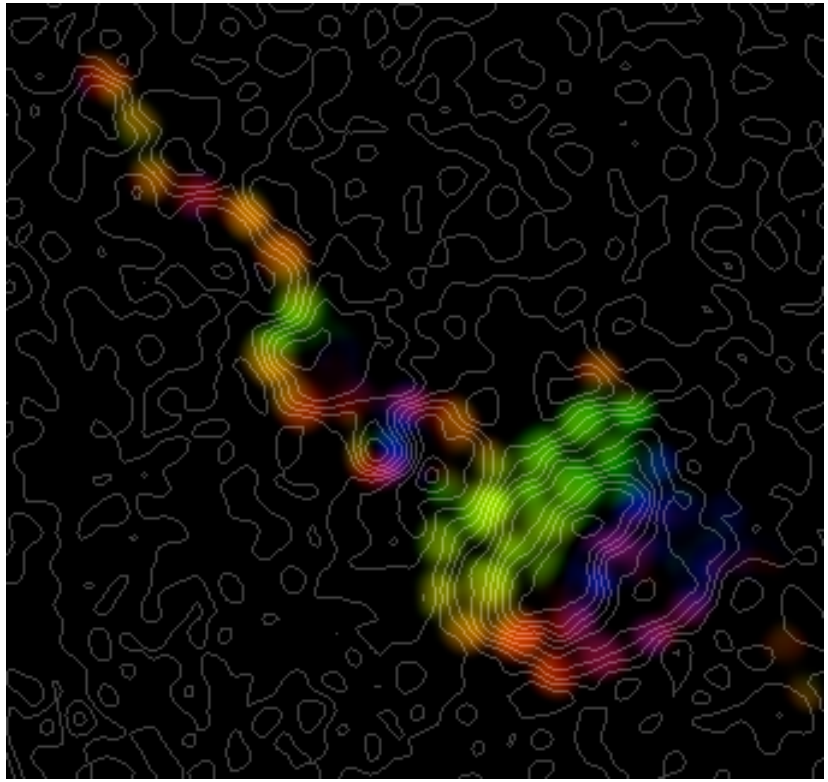
Naturally occurring crystals: magnetotactic bacteria



Four chains of magnetite crystals in a natural bacterial cell



Disrupted linear chain of magnetite crystals in a genetically modified bacterial cell

Ed Simpson, Mihaly Posfai, Dirk Schüler

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Spatial resolution of magnetic information

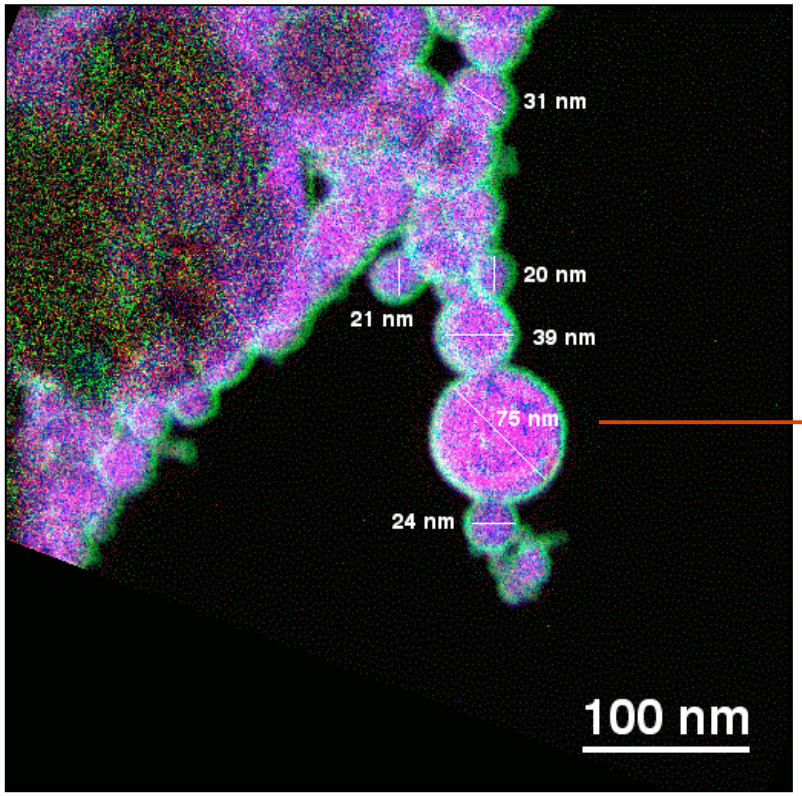
1. Electron optics of Lorentz lenses suggests that a spatial resolution of between 0.5 and 2 nm should be possible.

2. Signal to noise usually limits this to 5-10 nm at best.

3. This is not a fundamental obstacle - the practical limitation is the patience of the operator.

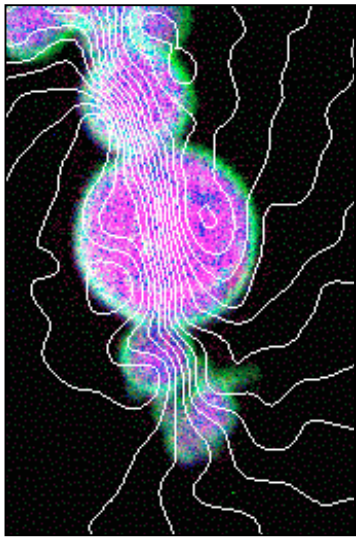
4. Automation of acquisition and analysis is possible in principle.

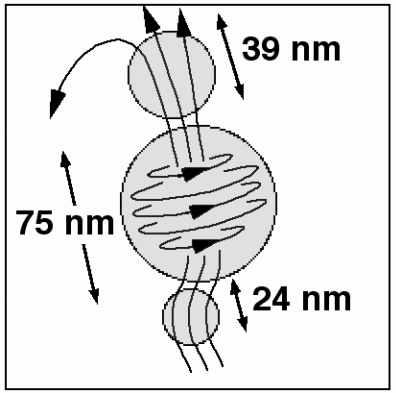
Chains of $Fe_{50}Ni_{50}$ Single Crystalline Nanoparticles



Chemical map
Green = O, Red = Ni, Blue = Fe
(Purple = magnetic, Green = non-magnetic)
Oxide around each particle is ~4 nm thick

Remanent magnetic state recorded using electron holography:





Contours are chaneled along the center of a 75 nm diameter particle by its neighbors
Contour spacing (1/6) radian.

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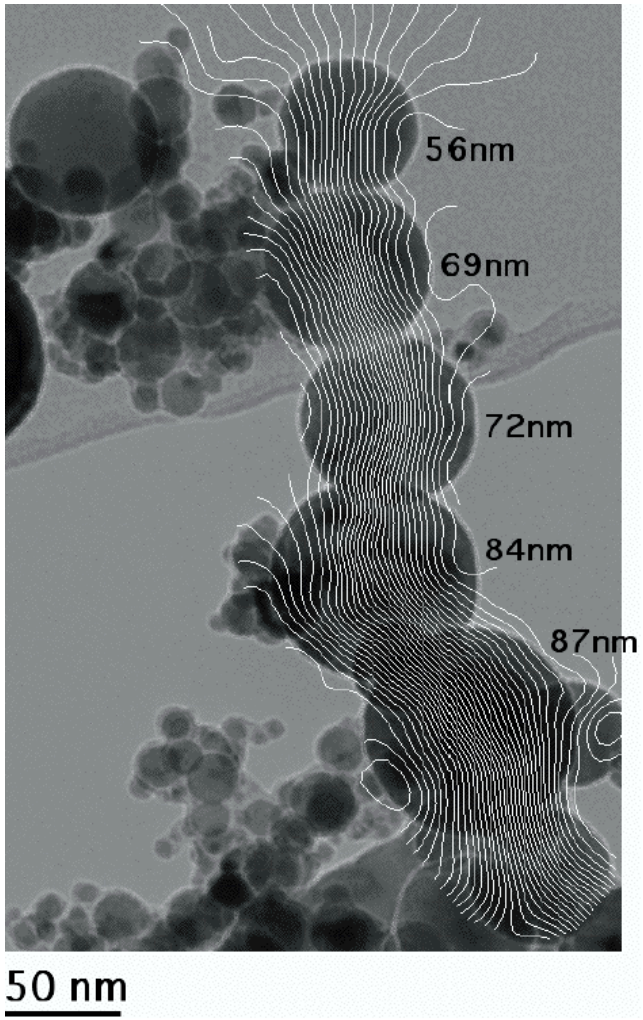
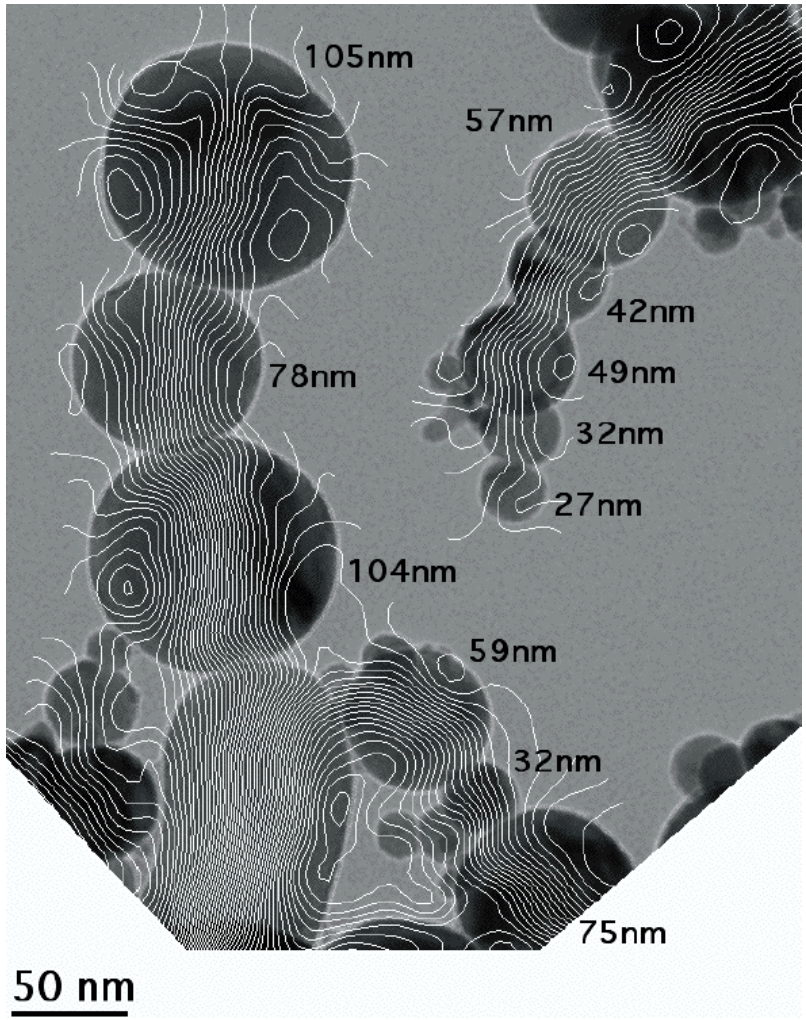
Martin Hÿtch

Chains of $Fe_{10}Ni_{90}$ Single Crystalline Nanoparticles





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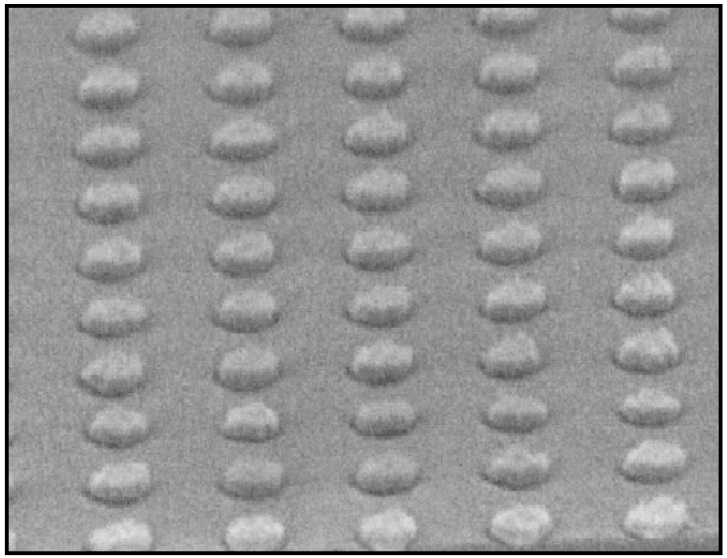
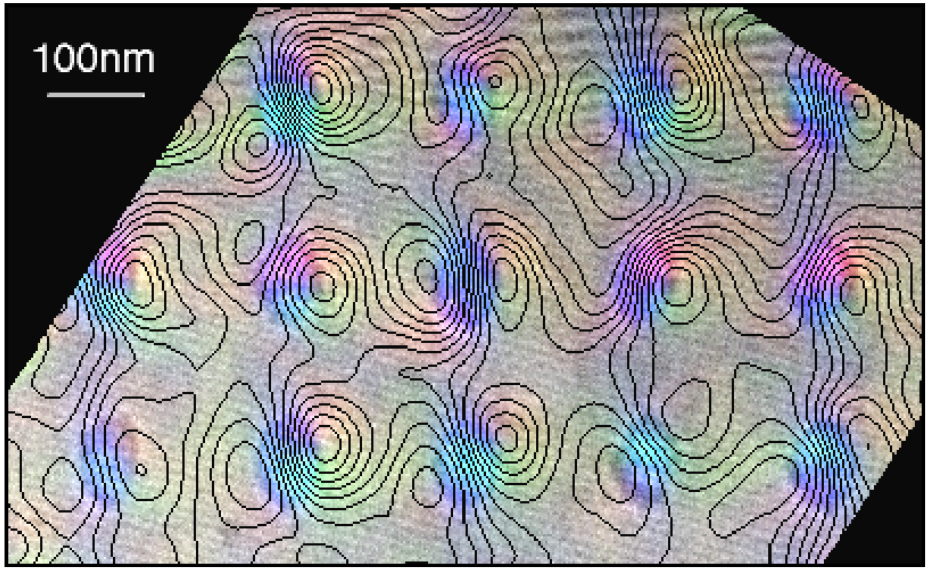
Magnetic nanostructures

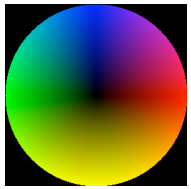
100-nm-diameter Co disks
fabricated on Si using interferometric lithography and examined in plan view



SEM image of
100 nm diameter 20 nm high
Co disks fabricated on Si
in a square array of side 200 nm



Magnetic remanent state recorded using holography
Contour spacing 0.033 radians



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Simon Newcomb

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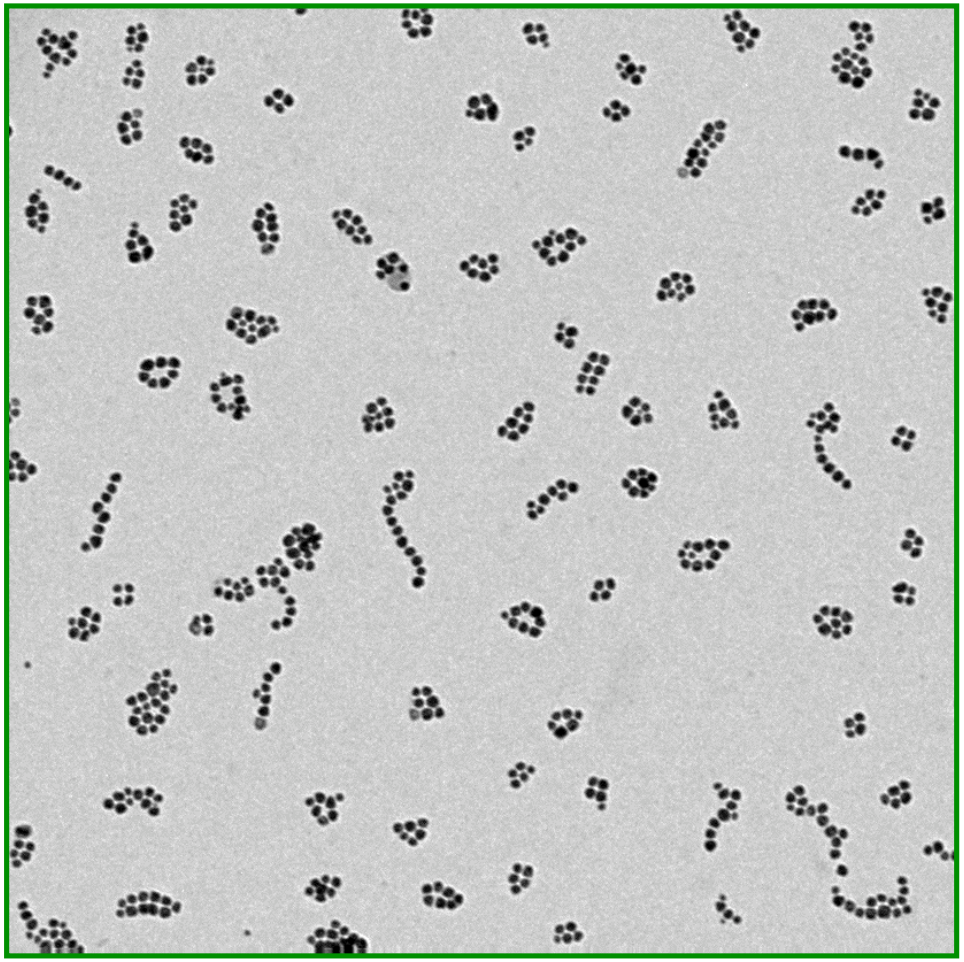
Co nanoparticle bracelets

Each Co particle is:

20 nm in size and expected to contain a single magnetic domain



Sample from Alex Wei and Steve Tripp (Purdue University)

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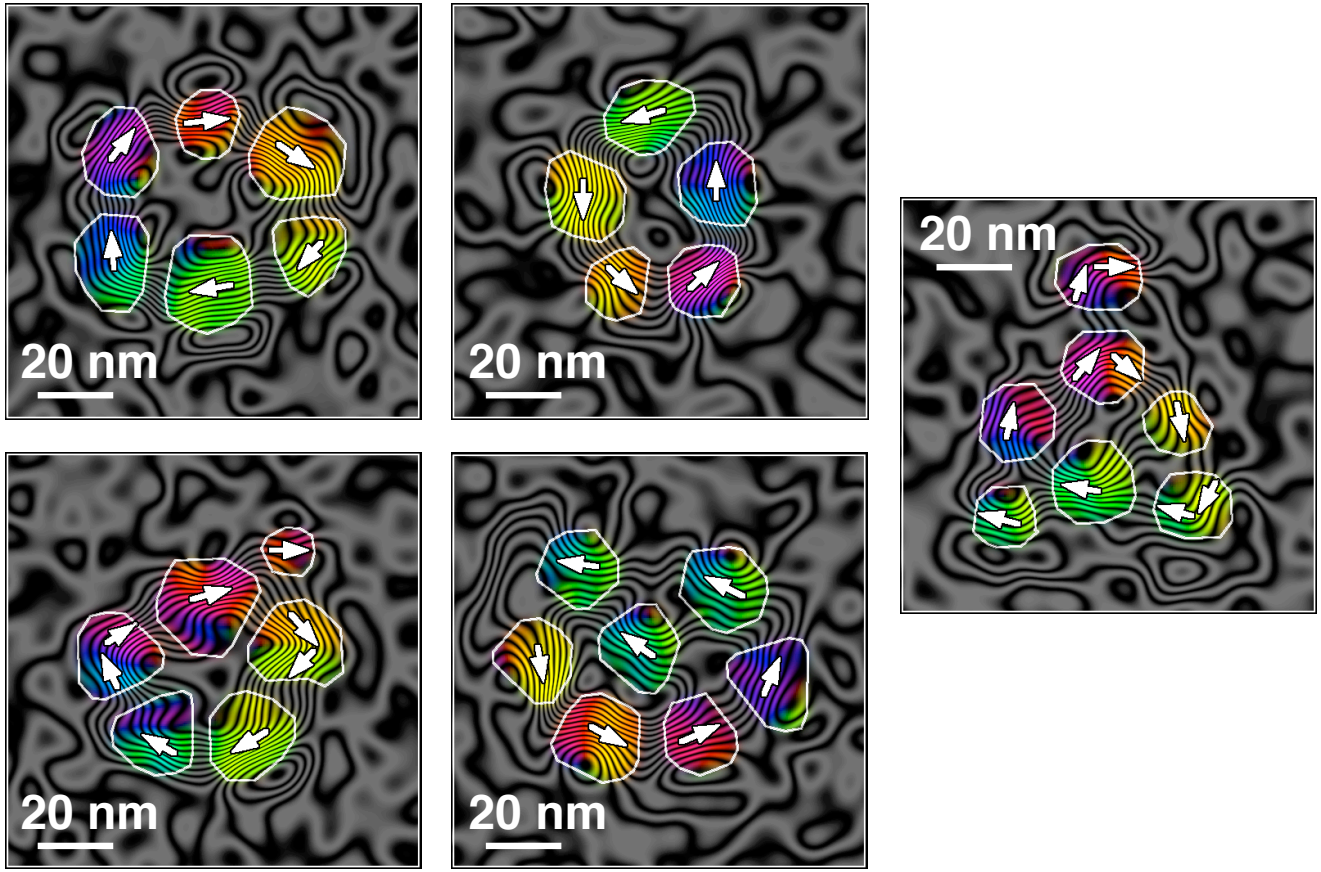
Alex Wei

Co nanoparticle bracelets - magnetic microstructure



Phase amplified magnetic contribution to measured phase

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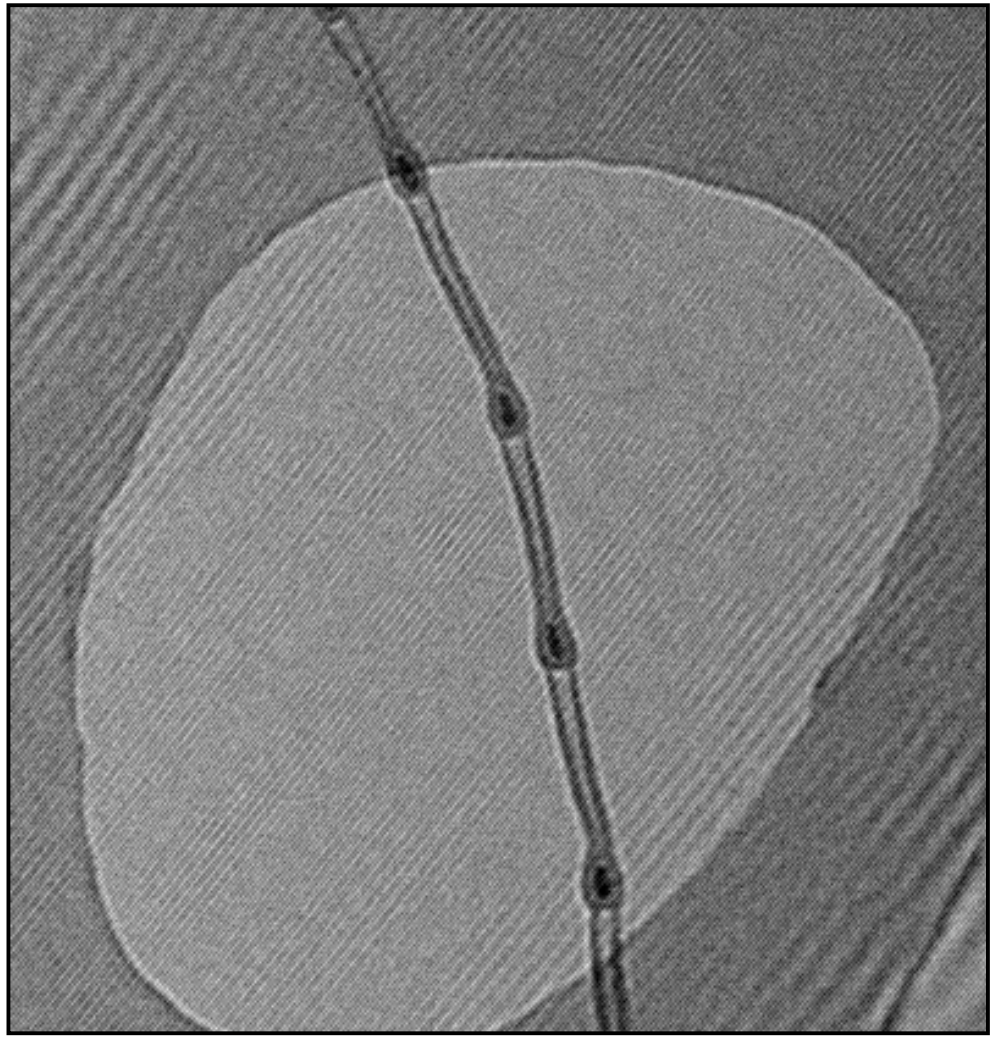
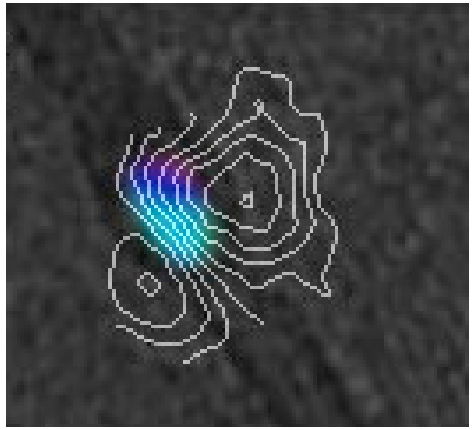
Alex Wei

10 - 20 nm magnetic nanoparticles periodically encapsulated in carbon nanotubes



100 nm



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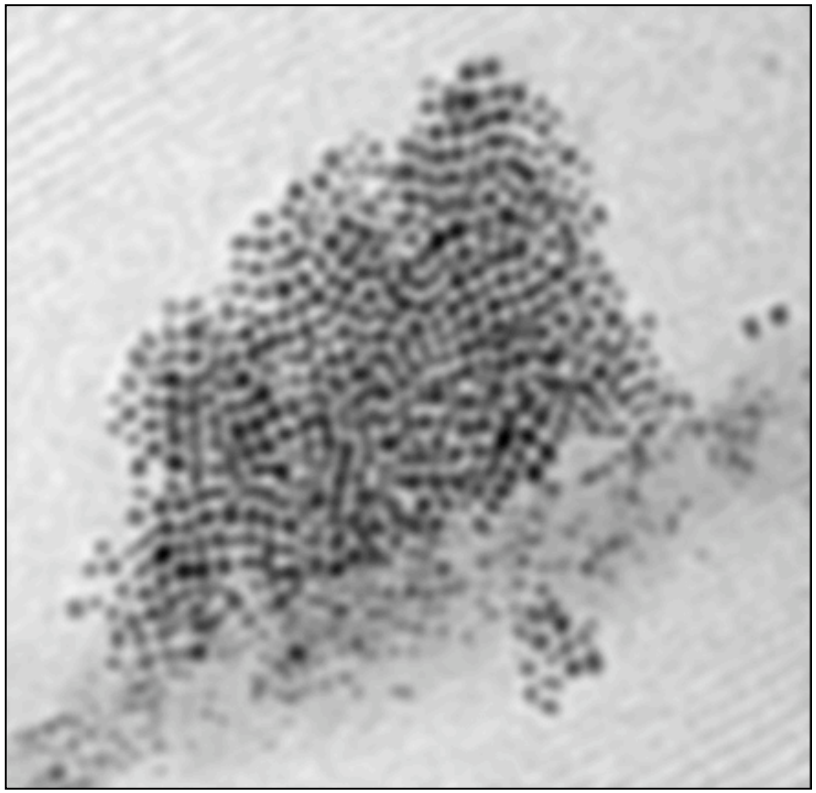
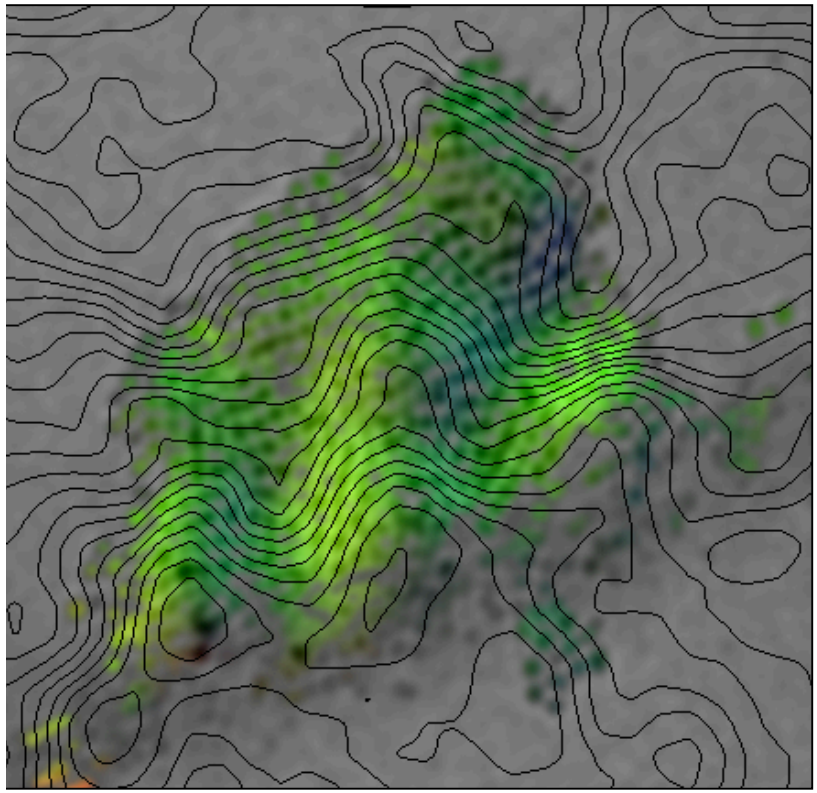
Vincent Jourdain

Two-dimensional arrays of 7 nm Fe cubes



Bright-field image



Magnetic phase contours recorded at liquid nitrogen temperature

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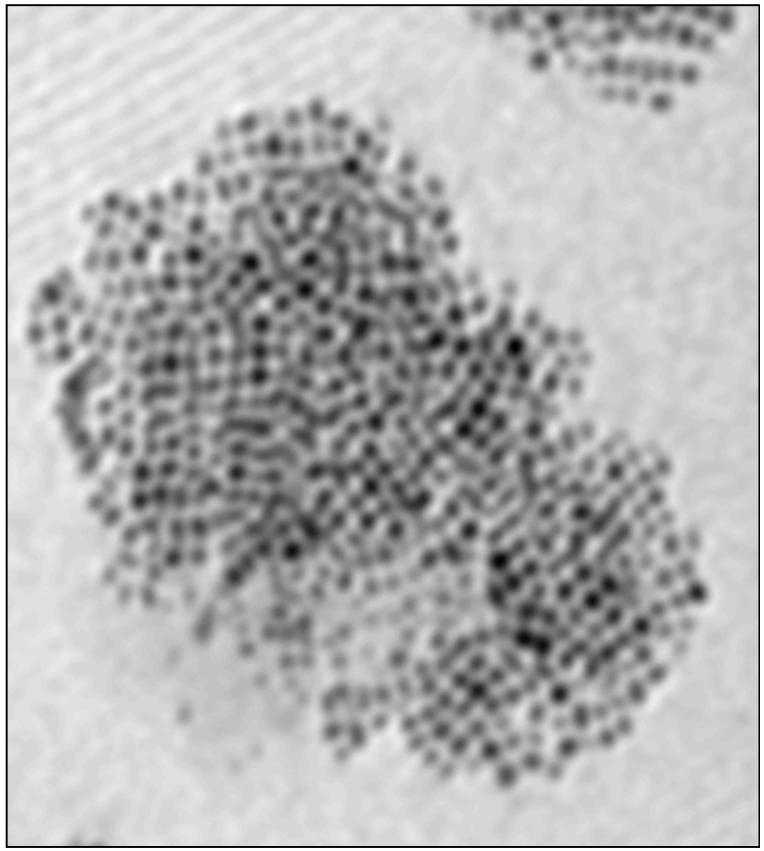
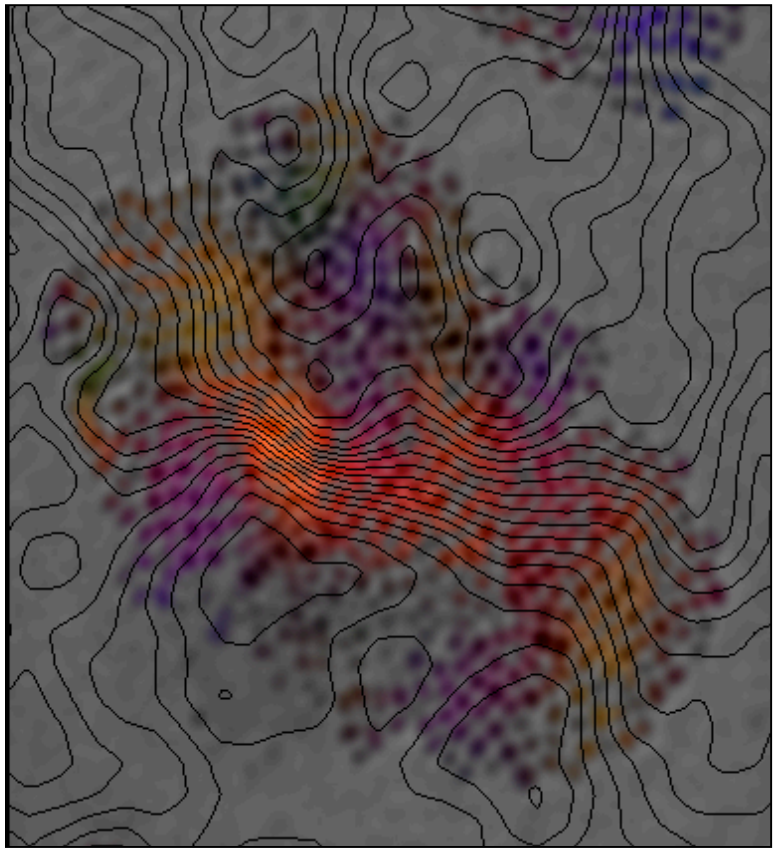
Etienne Snoeck

Two-dimensional arrays of 7 nm Fe cubes



Bright-field image



Magnetic phase contours recorded at liquid nitrogen temperature

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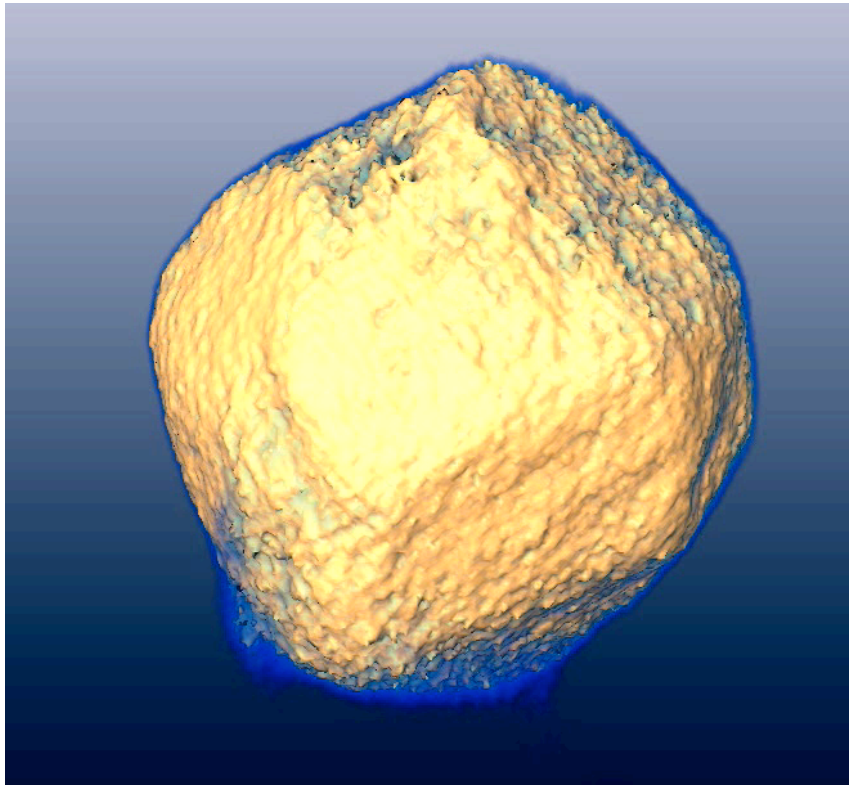
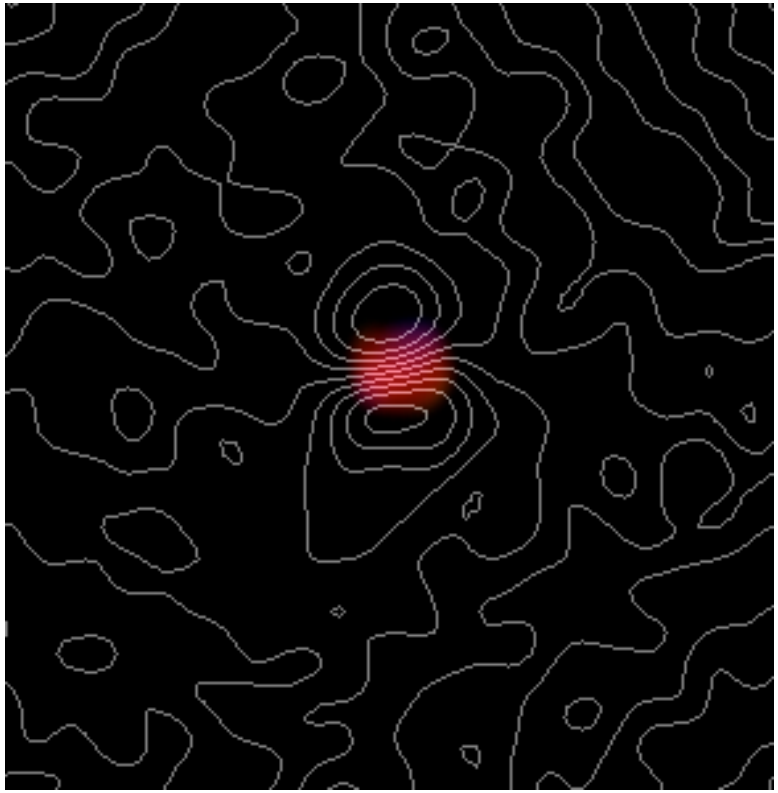
Correlation with three-dimensional microstructural information

Isolated equidimensional 30 nm magnetite nanocrystal





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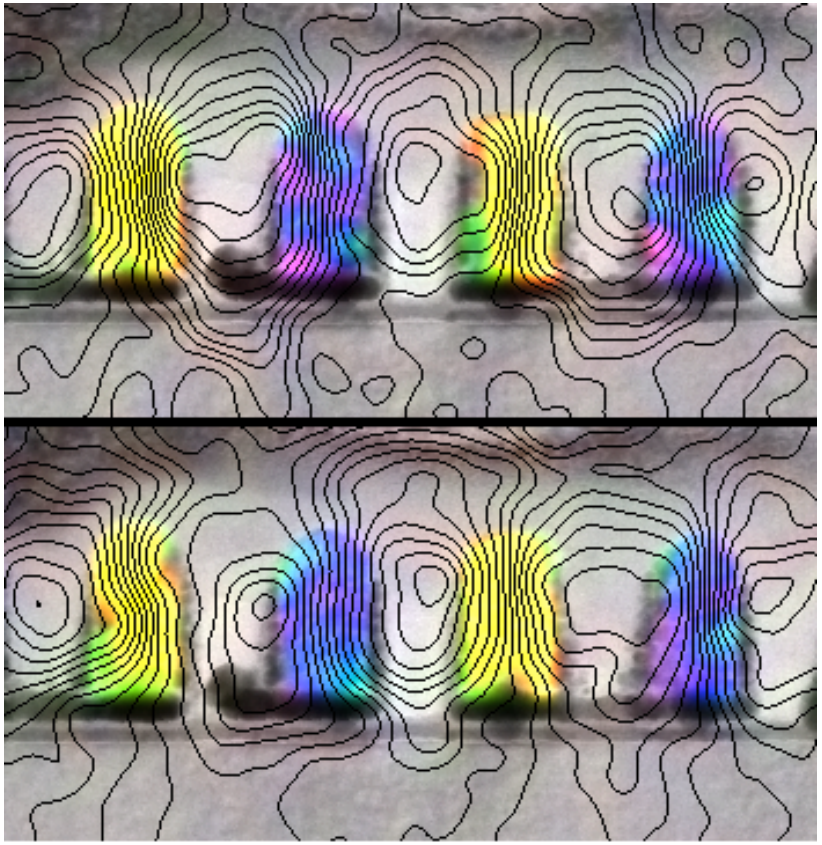
Magnetic nanostructures

150 x 50 nm Ni pillars
Fabricated on Si using interferometric lithography and examined in cross-section



100 nm

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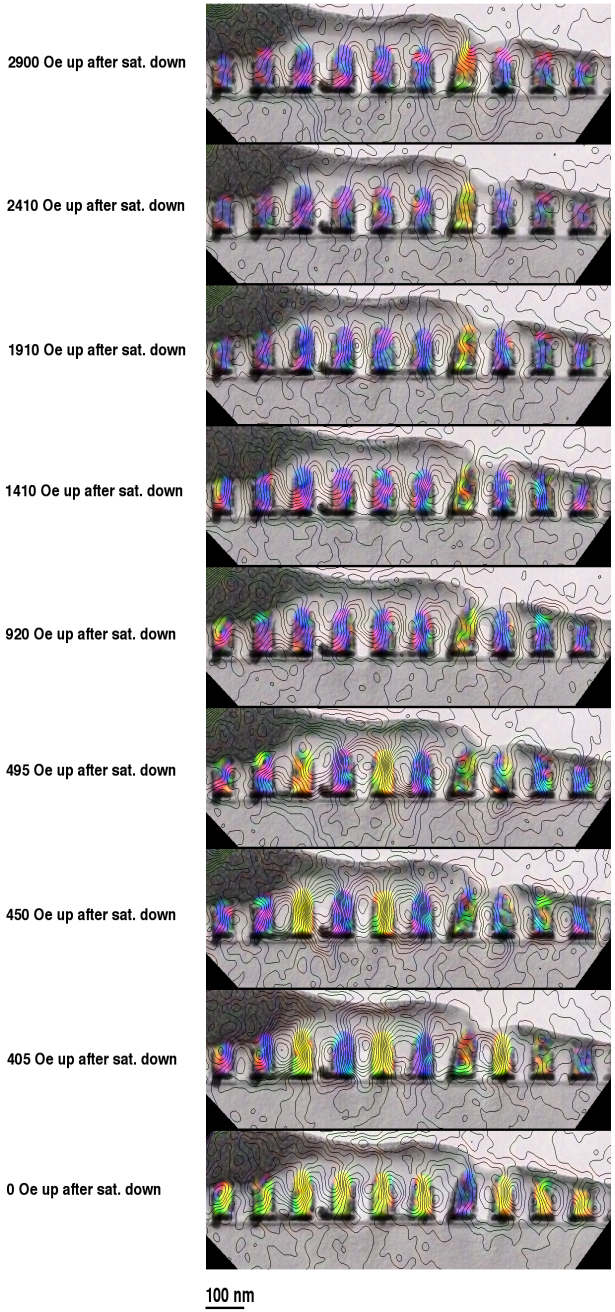
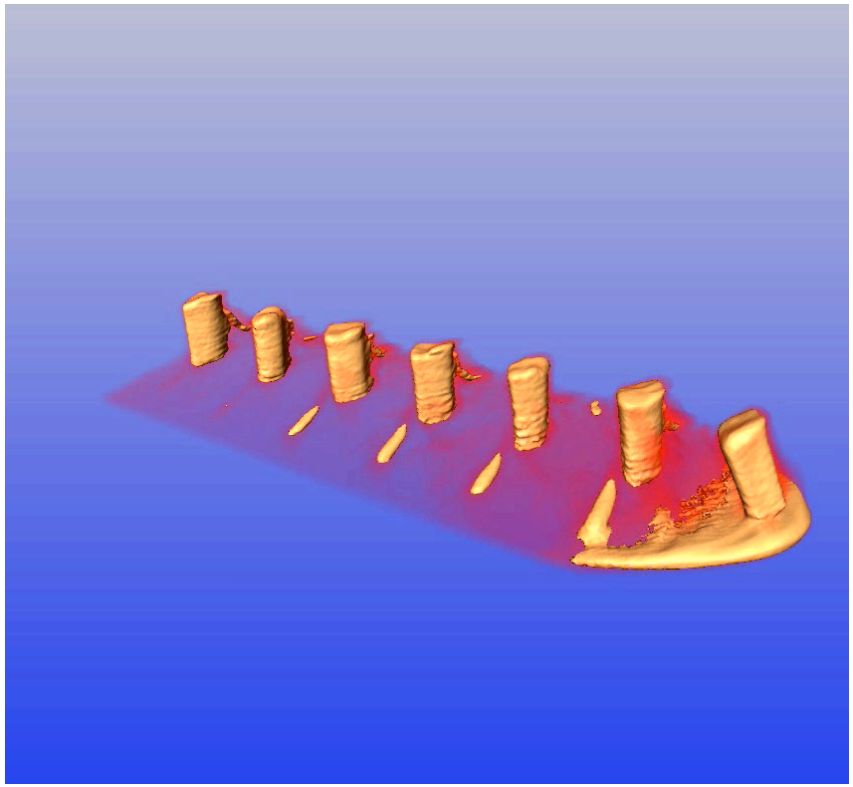
Caroline Ross, Tom Bromwich

Ni pillars. 100nm spacing. Nominally 115x57 nm.
Measured magnetic diameter 25-30 nm.
Remanent magnetic states for applied in-plane fields indicated.



Magnetic nanostructures

115 x 57 nm Ni pillars
Fabricated on Si using interferometric lithography and examined in cross-section



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Tom Bromwich

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Magnetic layers examined in cross-section

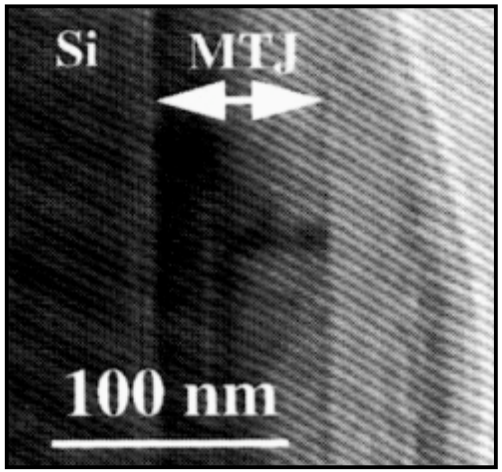
Magnetic induction mapping using off-axis electron holography

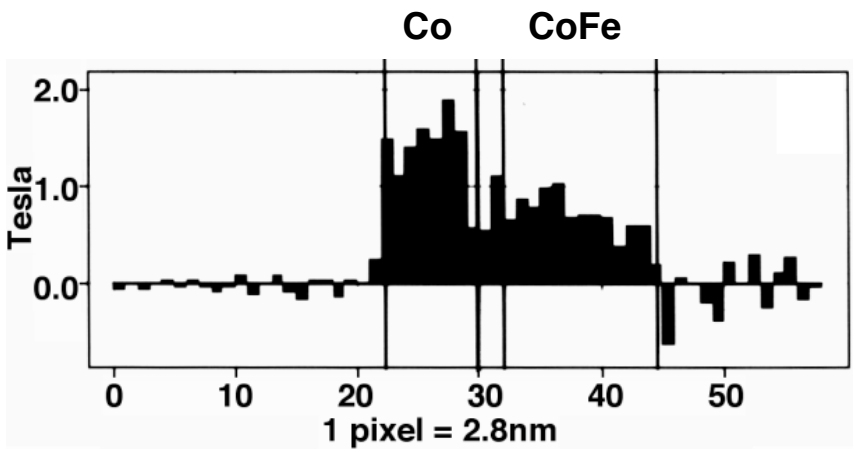
Magnetic layers in cross-section

$$B(x) = \left(\frac{\hbar C_E V_0(x)}{2e}\right)\left(\frac{\Delta\left[d\phi(x)/dx\right]}{\langle\phi(x)\rangle}\right)$$

Analysis of 2 phase images between which magnetization in specimen has been reversed

Off-axis electron hologram obtained from a magnetic tunnel junction with a 4nm HfO_2 tunnel barrier.





Molly McCartney

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Advantages of electron holography

1. Spatial resolution can approach 1 nm
2. Quantitative information can be obtained and compared with simulations
3. Non-invasive
4. Samples examined in transmission
5. Correlation with local microstructure and composition possible
6. Samples can be examined over a wide range of temperatures
7. Samples can be examined with currents passing through them

Disadvantages of electron holography

1. Region of vacuum close to area of interest required
2. Samples examined in transmission
3. In general not possible to obtain magnetic information about antiferromagnets
4. Sample preparation can affect magnetic properties and create 'dead' layers
5. Time resolution poor (seconds)
6. Difficult to interpret a 2D projection of only 2 components of a 3D field
7. Difficult to achieve a spatial resolution of better than a few nm
8. Difficult to design complicated experiments inside an electron microscope

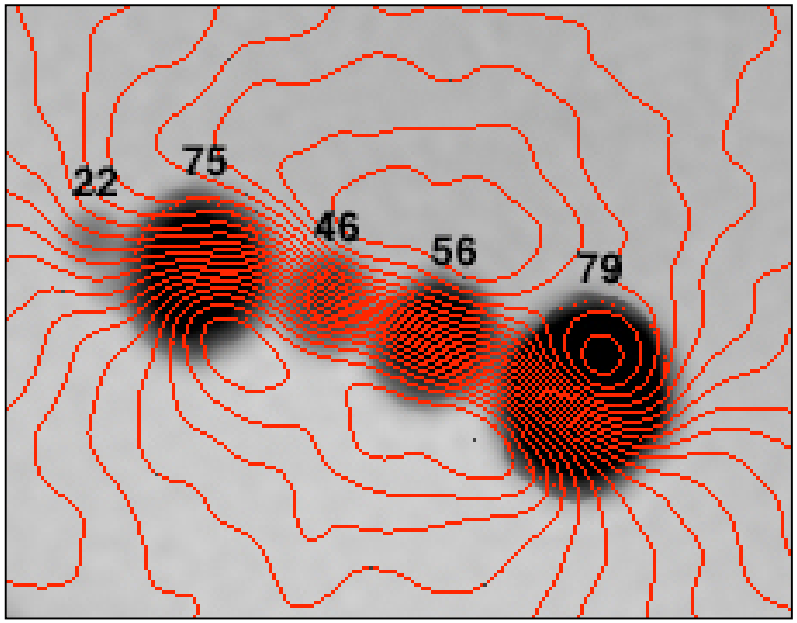
Future developments and challenges for electron holography

1. Imaging weak magnetic fields

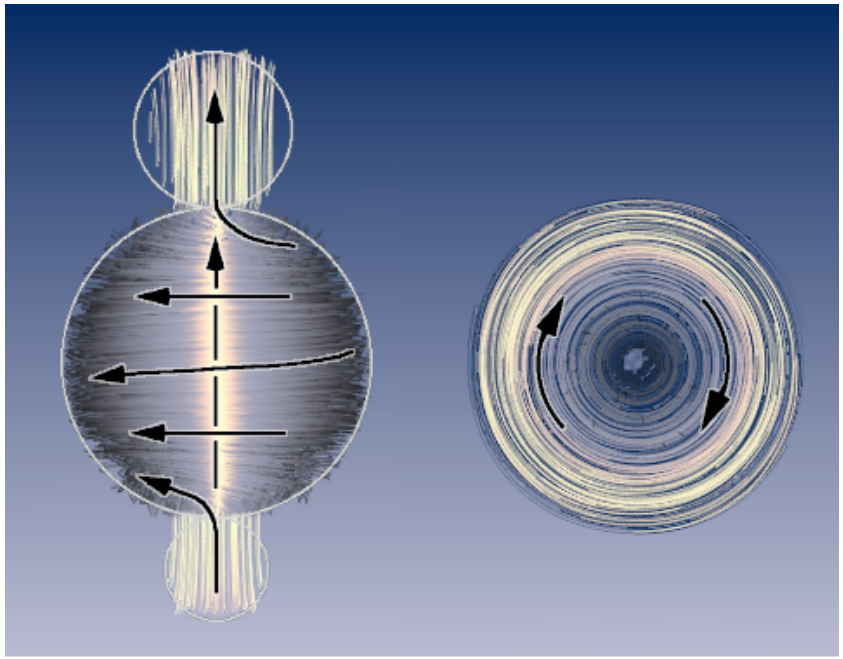
2. Imaging working devices

3. Characterizing magnetic fields inside materials in three dimensions

Chain of five $Fe_{50}Ni_{50}$ Single Crystalline Nanoparticles



Diameters in nm shown



20 nm

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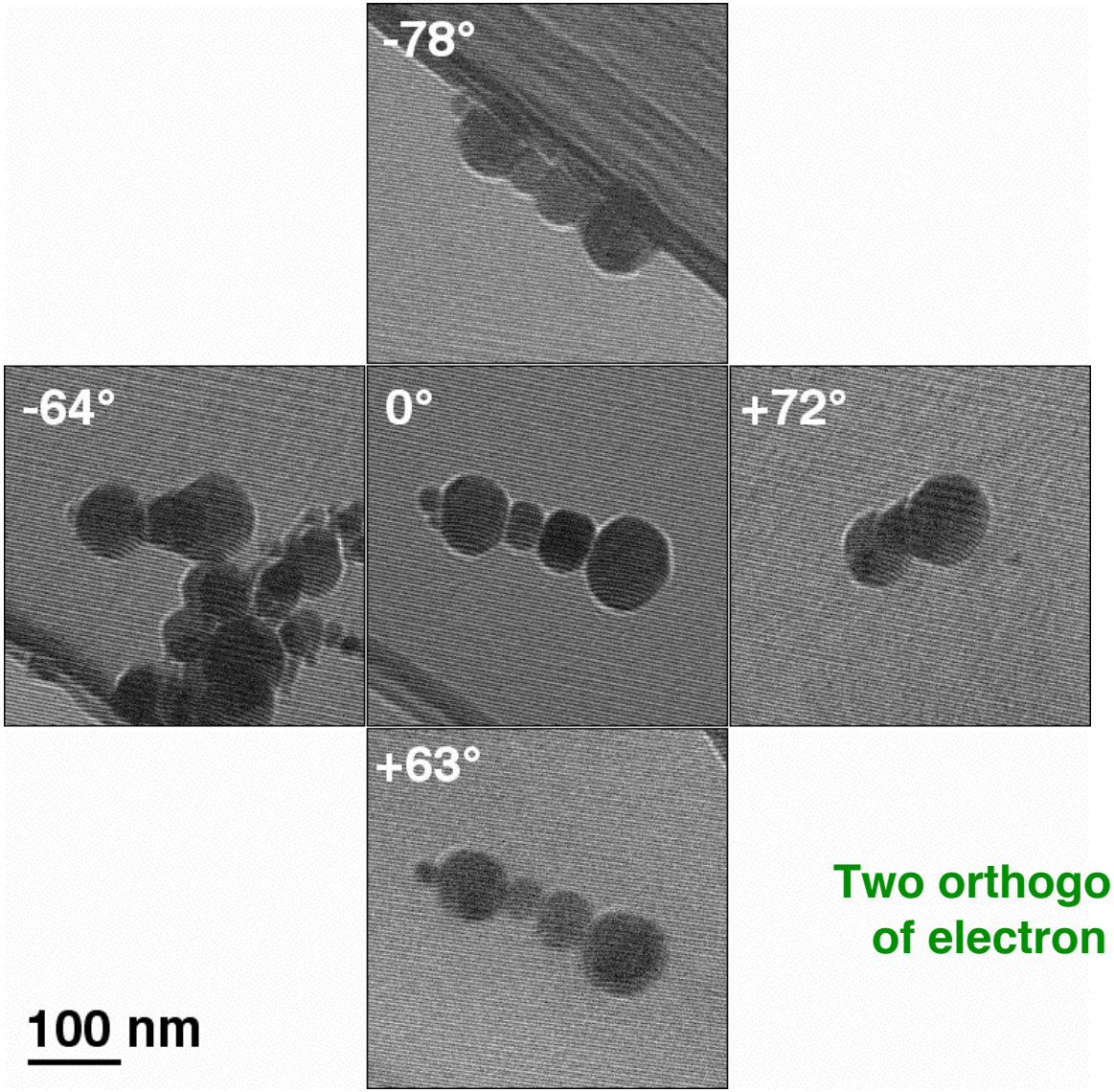
Chain of five $Fe_{50}Ni_{50}$ Single Crystalline Nanoparticles



Two orthogonal tilt series of electron holograms

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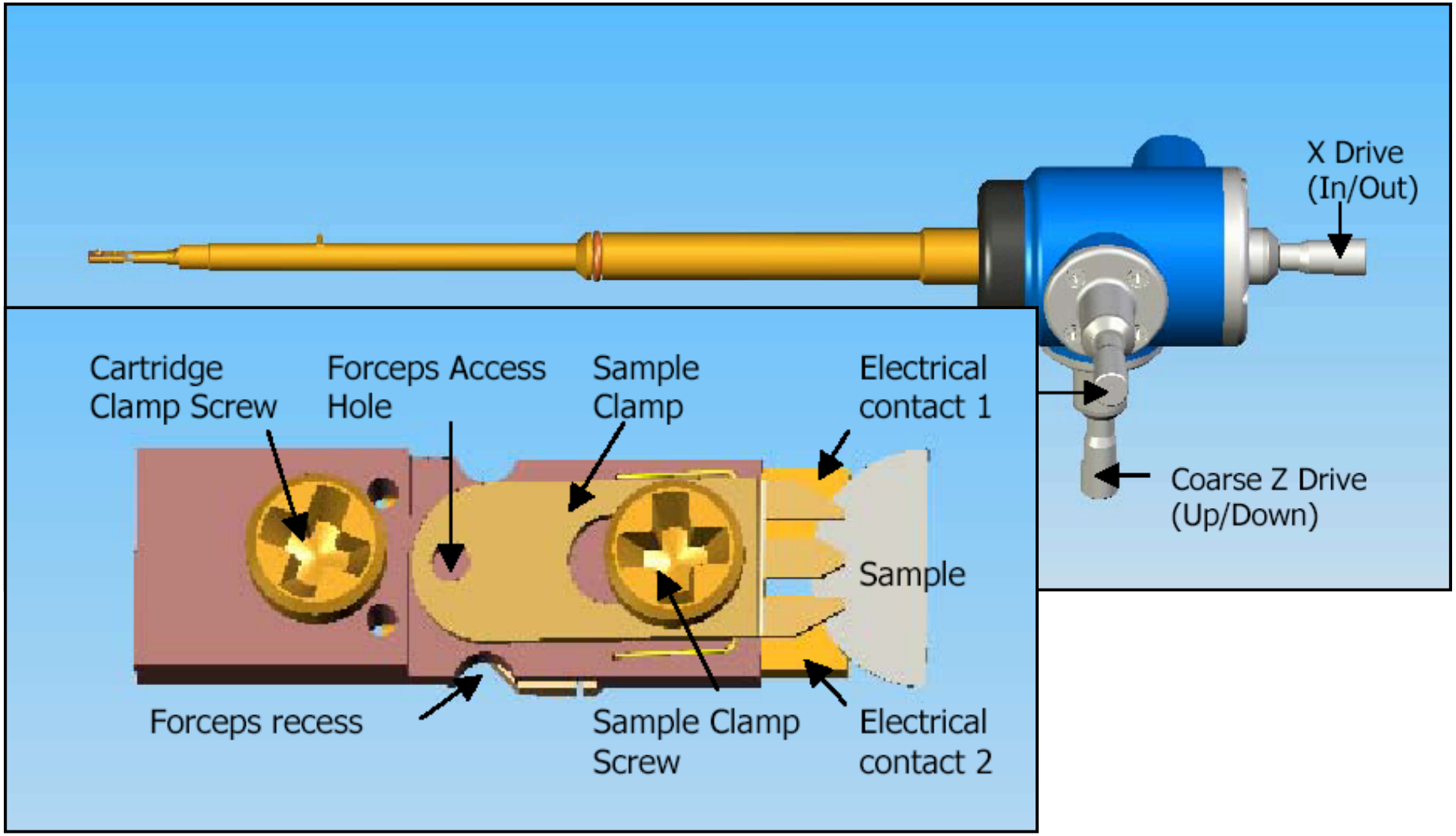
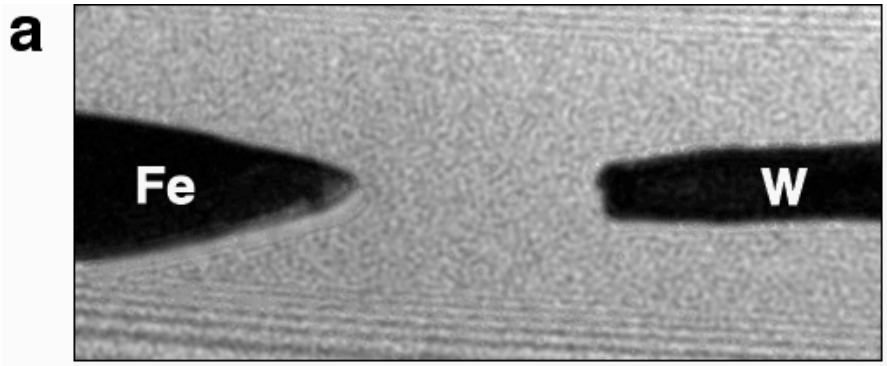
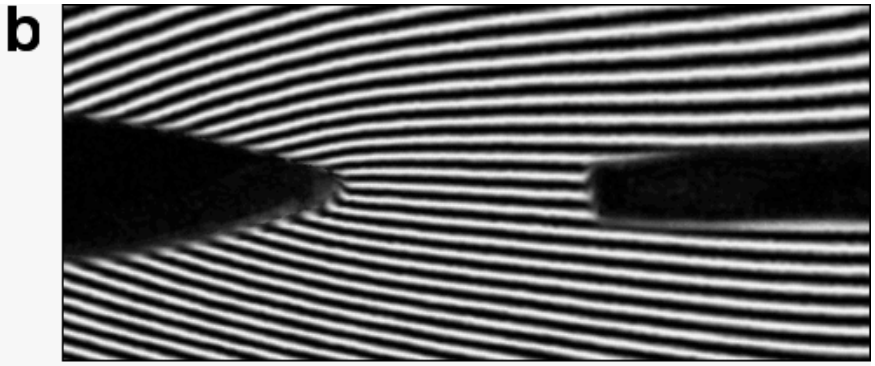
Fischione Biasing Tomography Cartridge-based Holder



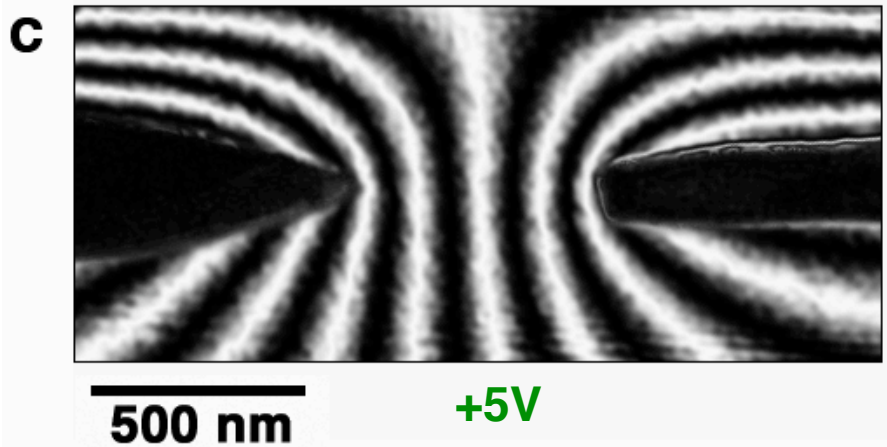
Magnetic needle with applied electric field



Amplitude image



Phase contours: 0V between needles



+5V

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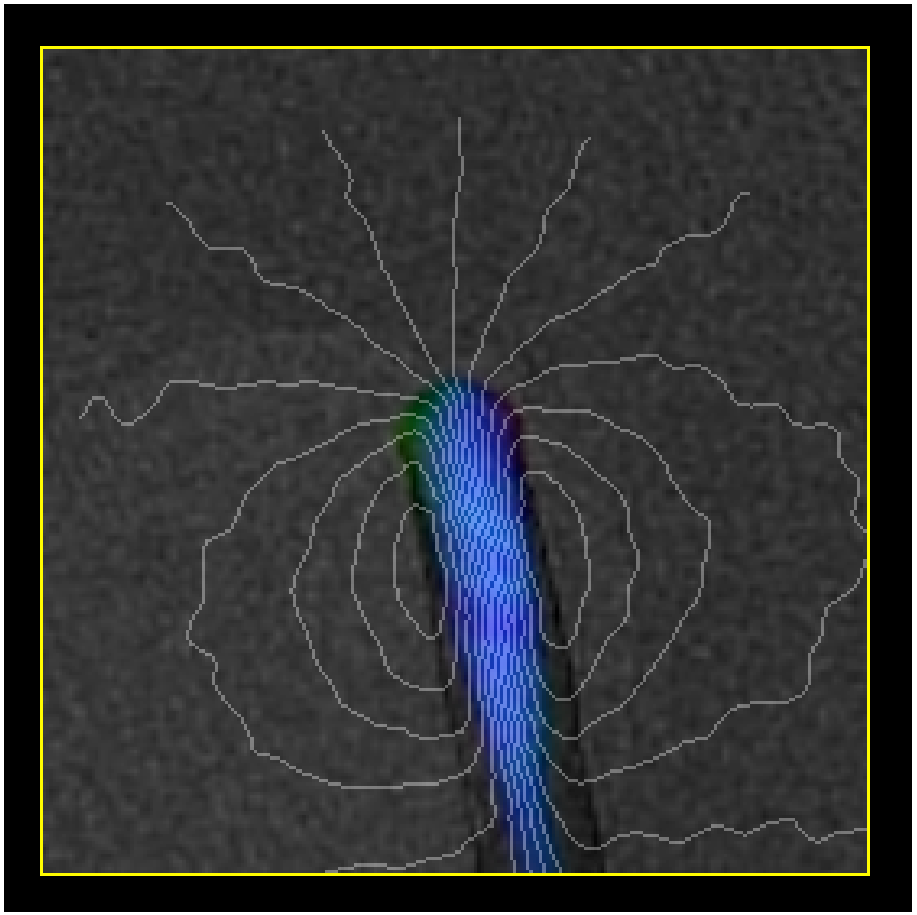
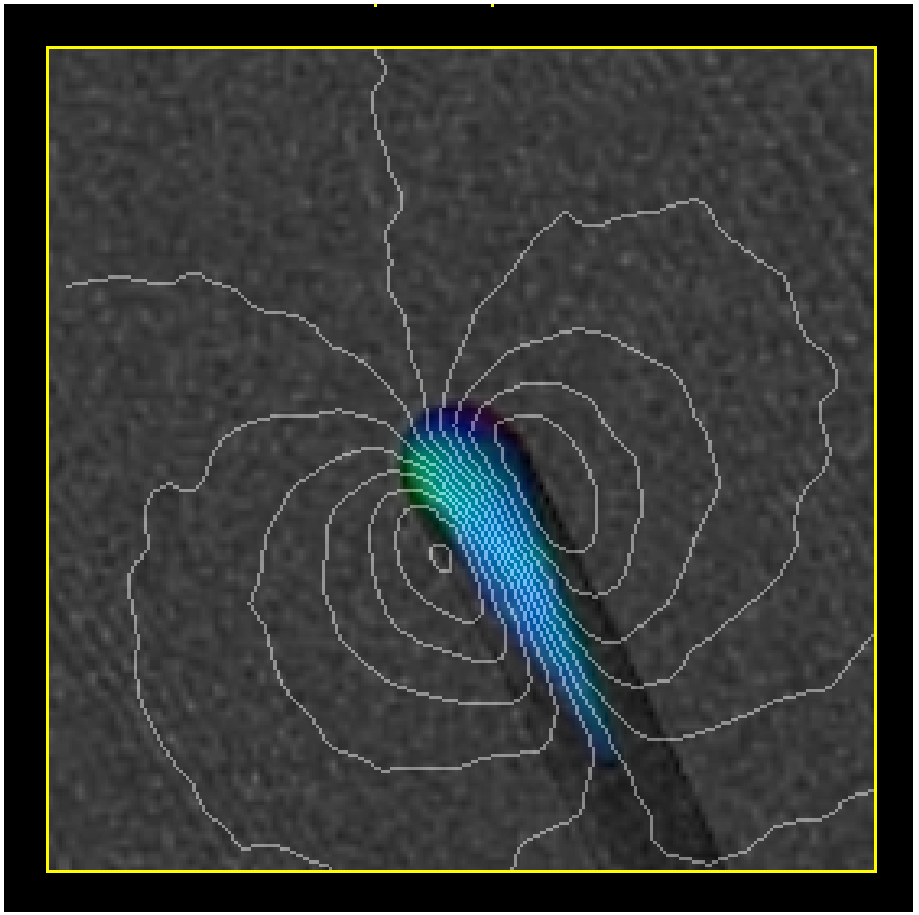
Summary

- Electron holography allows magnetic (and electric) fields in nanoscale materials and devices to be characterized.
- A spatial resolution of better than 1-2 nm for mapping the in-plane magnetic induction in a specimen can be achieved in principle.
- Electron holography may be combined with electron tomography to provides three-dimensional information about electrostatic and magnetic fields in materials.





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