Magnetism as seen with X-rays

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Magnetic materials for energy applications

Magnetic nanoparticles for biomedical applications

Magnetic thin films for information storage and processing
PERMANENT AND HARD MAGNETIC MATERIALS

Controlling grain and domain structure on the micro- and nanoscale

Engineering magnetic anisotropy on the atomic scale

NdFeB
MAGNETIC NANOPARTICLES

Optimizing magnetic nanoparticles for biomedical applications

Tailoring magnetic nanoparticles for environmental applications
MAGNETIC THIN FILMS

Magnetic coupling at interfaces

Ultrafast magnetization reversal dynamics

GMR Read Head Sensor

Magnetic domain structure on the nanometer scale

Unique magnetic phases at interfaces
+ Sensitivity to ferromagnetic and antiferromagnetic order
+ Element specificity = distinguishing Fe, Co, Ni, ...
+ Sensitivity to oxidation state = distinguishing Fe$^{2+}$, Fe$^{3+}$, ...
+ Sensitivity to site symmetry, e.g. tetrahedral, $T_d$; octahedral, $O_h$
+ Nanometer spatial resolution
+ Ultra-fast time resolution
+ Sensitivity to ferromagnetic and antiferromagnetic order
+ Element specificity = distinguishing Fe, Co, Ni, ...
+ Sensitivity to oxidation state = distinguishing Fe$^{2+}$, Fe$^{3+}$, ...
+ Sensitivity to site symmetry, e.g. tetrahedral, $T_d$; octahedral, $O_h$
+ Nanometer spatial resolution
+ Ultra-fast time resolution

**SOFT X-RAY SPECTROSCOPY AND MICROSCOPY**
SPECTROSCOPY

Photon Source → Monochromator → Sample

Intensity: wavelength, photon energy

Sample: Photon Source, Monochromator, Sample

Incoming photon is absorbed by the atom

Nucleus

Electron

Excited state

Lower energy level

Lowest energy level

Advanced Light Source
An Office of Science User Facility
Change velocity of electrons near the speed of light

⇒ Emission of wide wavelength range of electromagnetic spectrum

⇒ Tunable source in the soft x-ray range for x-ray absorption spectroscopy
Electron Storage Ring and Monochromators

Inside the ALS storage ring

+ Monochromators provide photon beams with well defined characteristics:
  - tunable energy/wavelength
  - polarization: linear, circular, ...
  - x-ray spot size ...
Endstations provide well defined environments for the interaction of samples and photons:
- precisely defined experimental geometries, i.e. angle of x ray beam to sample
- sample temperature
- external magnetic and electric fields
Magnetic fields in arbitrary directions obtained through superposition of fields generated by 4 dipole pairs in octahedral configuration.
SOFT X-RAY SPECTROSCOPY (hν ≈ 500-1000eV, λ ≈ 1-2nm)
**Electron yield:**

+ Absorbed photons create core holes subsequently filled by Auger electron emission
+ Auger electrons create low-energy secondary electron cascade through inelastic scattering
+ Emitted electrons $\propto$ probability of Auger electron creation $\propto$ absorption probability
**SOFT X-RAY ABSORPTION – PROBING DEPTH**

\[ I_t = I_o e^{-\mu t} \]

Sample

<table>
<thead>
<tr>
<th>Element</th>
<th>10eV below (L_3) (1/\mu ) [nm]</th>
<th>at (L_3) (1/\mu ) [nm]</th>
<th>40 eV above (L_3) (1/\mu ) [nm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>550</td>
<td>17</td>
<td>85</td>
</tr>
<tr>
<td>Co</td>
<td>550</td>
<td>17</td>
<td>85</td>
</tr>
<tr>
<td>Ni</td>
<td>625</td>
<td>24</td>
<td>85</td>
</tr>
</tbody>
</table>

\[ \frac{I_t}{I_0} \]

 photon energy (eV)

\~10-20 nm layer thick films supported by substrates transparent to soft x-rays
X-RAY ABSORPTION – DETECTION MODES AND PROBING DEPTH

\[ I_t = I_0 e^{-\mu t} \]

\[ I_e = I_0 \mu \]

\[ \text{electron yield (arb. units)} \]

\[ \text{photon energy (eV)} \]

\[ \text{It} / I_0 \]

Fe

Co

Ni
Electron sample depth: 2-5 nm in Fe, Co, Ni
⇒ 60% of the electron yield originates from the topmost 2-5 nm
Experimental Concept:
Monitor reduction in x-ray flux transmitted through sample as function of photon energy

$$I_0 \longrightarrow I_t = I_0 e^{-\mu t}$$

Sample

$$\Rightarrow$$ charge state of absorber Fe$^{2+}$, Fe$^{3+}$

$$\Rightarrow$$ symmetry of lattice site of absorber: $O_h$, $T_d$

$$\Rightarrow$$ sensitive to magnetic order

$$\Rightarrow$$ Absorption probability: x-ray energy, x-ray polarization, experimental geometry

$$2p_{3/2} \quad L_3$$

$$2p_{1/2} \quad L_2$$

core level $$\Rightarrow$$ atomic species of absorber Fe, Co, Ni, ....
Experimental Concept:
Monitor reduction in x-ray flux transmitted through sample as function of photon energy

⇒ charge state of absorber Fe\(^{2+}\), Fe\(^{3+}\)
⇒ symmetry of lattice site of absorber: O\(_h\), T\(_d\)
⇒ sensitive to magnetic order

⇒ Absorption probability: x-ray energy, x-ray polarization, experimental geometry

core level ⇒ atomic species of absorber Fe, Co, Ni, ....
Intensity of $L_{3,2}$ resonances is proportional to number of $d$ states above the Fermi level, i.e. number of holes in the $d$ band.
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‘White Line’ Intensity

Intensity of $L_{3,2}$ resonances is proportional to number of $d$ states above the Fermi level, i.e. number of holes in the $d$ band.
**X-Ray Absorption — Fundamentals**

**Experimental Concept:**
Monitor reduction in x-ray flux transmitted through sample as function of photon energy

- $I_t = I_o e^{-\mu t}$

- Charge state of absorber: Fe$^{2+}$, Fe$^{3+}$, ...

- Symmetry of lattice site of absorber: $O_h$, $T_d$

- Sensitive to magnetic order

- Absorption probability: x-ray energy, x-ray polarization, experimental geometry

- Core level $\Rightarrow$ atomic species of absorber Fe, Co, Ni, ....

\[e^- \rightarrow \hbar \omega \rightarrow \text{core} \rightarrow \text{symmetry} \rightarrow \text{charge state} \rightarrow \text{absorption probability} \rightarrow \text{atomic species} \]
X-RAY ABSORPTION – VALENCE STATE

Fe$^{3+}$ in LaFeO$_3$

Co$^{2+}$ in CoO

Ni$^{2+}$ in NiO

J. Stöhr, H.C. Siegmann, Magnetism (Springer)
X-ray Absorption – Valence State

Influence of the charge state of the absorber

N. Telling et al., Appl. Phys. Lett. 95, 163701 (2009)

**X-RAY ABSORPTION – ONE ELECTRON PICTURE**

**X-ray absorption:**
+ Electrons excited from core shells to unoccupied valence states through absorption of a photon determined by energy and angular momentum conservation

**Simplest model: One electron picture**
+ Photon transfers its energy and momentum to core electron
+ Core electron excited into unoccupied electronic state.

+ However: Not directly excited electrons also influenced by electron excitation, i.e. hole in core shell
**X-Ray Absorption – Configuration Model**

**Configuration model**, e.g. $L$ edge absorption:

- Excited from
  - ground/initial state configuration, $2p^63d^n$
  - to exited/final state configuration, $2p^53d^{n+1}$
- Omission of all full subshells (spherical symmetric)
- Takes into account correlation effects in the ground state as well as in the excited state
- Leads to multiplet effects/structure

$\text{Ni}^{2+}$ in $\text{NiO}$: $2p^6\ 3d^8 \rightarrow 2p^5\ 3d^9$

http://www.anorg.chem.uu.nl/CTM4XAS/

J. Stöhr, H.C. Siegmann, Magnetism (Springer)
**Experimental Concept:**
Monitor reduction in x-ray flux transmitted through sample as function of photon energy

\[ I_t = I_o e^{-\mu t} \]

- \( I_t \) = Transmitted x-ray intensity
- \( I_o \) = Initial x-ray intensity
- \( \mu \) = Attenuation coefficient
- \( t \) = Thickness of sample

\[ e^{-\mu t} \]

\[ \mu \rightarrow \text{charge state of absorber Fe}^{2+}, \text{Fe}^{3+} \]

\[ \text{symmetry of lattice site of absorber: } O_h, T_d \]

\[ \Rightarrow \text{sensitive to magnetic order} \]

\[ 2p_{3/2} \quad L_3 \]

\[ 2p_{1/2} \quad L_2 \]

\( \Rightarrow \text{Absorption probability: x-ray energy, x-ray polarization, experimental geometry} \)

\[ \Rightarrow \text{core level } \Rightarrow \text{atomic species of absorber Fe, Co, Ni, ....} \]
X-RAY ABSORPTION — LATTICE SYMMETRY

TIO₂

Rutile

Influence of lattice site symmetry at the absorber

Anatase

G. Van der Laan
**SENSITIVITY TO SITE SYMMETRY: Ti$^{4+}$ $L_{3,2}$**

- Electric dipole transitions: $d^0 \rightarrow 2p^5 3d^1$
- Crystal field splitting $10D_q$ acting on $3d$ orbitals:
  
  **Octahedral symmetry:**
  
  - $e$ orbitals towards ligands $\rightarrow$ higher energy
  - $t_2$ orbitals between ligands $\rightarrow$ lower energy

  **Tetragonal symmetry:**
  
  - $e$ orbitals $\rightarrow b_2 = d_{xy}$, $e = d_{yz}$, $d_{yz}$
  - $t_2$ orbitals $\rightarrow b_1 = d_{x^2-y^2}$, $a_1 = d_{3z^2-r^2}$

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Experimental Concept:
Monitor reduction in x-ray flux transmitted through sample as function of photon energy

\[ I_t = I_0 e^{-\mu t} \]

Sample

- \( \text{Absorption probability: } \text{x-ray energy, x-ray polarization, experimental geometry} \)
- \( \text{charge state of absorber } \text{Fe}^{2+}, \text{Fe}^{3+} \)
- \( \text{symmetry of lattice site of absorber: } \text{O}_h, \text{T}_d \)
- \( \text{sensitive to magnetic order} \)
- \( \text{core level } \Rightarrow \text{atomic species of absorber } \text{Fe}, \text{Co}, \text{Ni}, \ldots. \)
Experimental Concept:
Monitor reduction in x-ray flux transmitted through sample as function of photon energy

\[ I_t = I_0 e^{-\mu t} \]

Sample

- charge state of absorber Fe\(^{2+}\), Fe\(^{3+}\)
- symmetry of lattice site of absorber: O\(_h\), T\(_d\)
- sensitive to magnetic order

Absorption probability: x-ray energy, x-ray polarization, experimental geometry

\[ 2p_{3/2} \quad L_3 \]
\[ 2p_{1/2} \quad L_2 \]

Core level \(\Rightarrow\) atomic species of absorber Fe, Co, Ni, ....
Magnetic moments in Fe, Co, Ni well described by Stoner model: $d$-bands containing up and down spins shifted relative to each other by “exchange splitting”

- Spin-up and spin-down bands filled according to Fermi statistics
- Magnetic moment $|m|$ determined by difference in number of electrons in majority and minority bands

$$ |m| = \mu_B (n_e^{maj} - n_e^{min}) $$

J. Stöhr, H.C. Siegmann, Magnetism (Springer)
Photoexcitation of electron from $2p_{3/2}, 2p_{1/2}$ to $3d$ states by absorption of circularly polarized x rays:

- Transfer of angular momentum of incident circular polarized x ray to excited electrons (angular momentum conservation)
- Excitation from spin-orbit split $2p_{3/2}, 2p_{1/2}$ level
  - Transfer of angular momentum ($\pm \hbar$) from photon to electron spin through spin-orbit coupling
  - Spin polarization opposite for incident x rays with positive ($+\hbar$) and negative ($-\hbar$) photon spin
- $2p_{3/2}$ and $2p_{1/2}$ levels have opposite spin orbit coupling ($l+s, l-s$)
  - Spin polarization opposite for two levels
- Unequal spin-up and spin-down populations in exchange split valence shell acts as detector for spin of excited electrons
Magnitude of dichroism depends on
+ expectation value of 3d magnetic moment
+ degree of circular photon polarization, $P_{\text{circ}}$

\[
I_{\text{XMCD}} = I_{\uparrow\downarrow} - I_{\uparrow\uparrow}
\]
+ XMCD provides magnetic information resolving elements Fe, Co, ...
valence states: Fe$^{2+}$, Fe$^{3+}$, ...
lattice sites: octahedral, O$_h$, tetrahedral, T$_d$, ...

J. Stöhr, H.C. Siegmann, Magnetism (Springer)
Geobacter sulfurreducens bacteria form magnetite nanocrystals (15nm) via extracellular reduction of amorphous Fe(III)-bearing minerals.

Geobacter sulfurreducens bacteria form magnetite nanocrystals (15nm) via extracellular reduction of amorphous Fe(III)-bearing minerals.
Comparing XMCD spectra with model compounds and/or calculations

⇒ Identifying magnetic phases
The element-specificity makes XMCD measurements an ideal tool to determine induced moments at interfaces between magnetic and non-magnetic elements.

M. G. Samant et al.,
**BAND FILLING IN Ga$_x$Fe$_{1-x}$**

- Fe majority-spin filled for $x=0.3$
- $x > 0.3$: Fe moment decrease strongly, Formation of D03 precipitates


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**Graphical Elements**

1. **Ga$_x$Fe$_{1-x}$**
2. **Fe $L_{3,2}$**
3. **XMCD (arb. units)**
4. **Photograph of material**
5. **Energy DOS diagram**
6. **Equations for $S_z$**
   - $S_z = -\frac{1}{2}$
   - $S_z = +\frac{1}{2}$
7. **Filled states, electrons**
8. **Minority of electron spins**
9. **“Majority band”**
10. **“Minority band”**

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**Reference**

J. Stöhr, H.C. Siegmann, Magnetism (Springer)
Theoretically derived sum rules correlate XMCD spectra with spin and orbital moment providing unique tool for studying magnetic materials.

\[ m_S = \mu_B (-A + 2B) / C \]

\[ m_L = -2\mu_B (A + B) / 3C \]

J. Stöhr, H.C. Siegmann, Magnetism (Springer)
Separation of spin and orbital moments requires very high quality data.

\[
\frac{m_{\text{orb}}}{m_{\text{spin}}} = \frac{2q}{9p - 6q}
\]

\[
m_{\text{orb}} = -\frac{4q(10 - n_{3d})}{3r}
\]

\[
m_{\text{spin}} = -\frac{(6p - 4q)(10 - n_{3d})}{r}
\]

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C.T. Chen et al.,
Spin and orbital moment only systems have distinct XMCD spectra:

\[ m_L = -2\mu_B\langle A + B \rangle / 3C = 0 \quad \text{for} \quad A = -B \]

\[ m_S = \mu_B\langle -A + 2B \rangle / C = 0 \quad \text{for} \quad A = 2B \]
+ Strong variation of orbital and spin magnetic moment observable as change in relative $L_3$ and $L_2$ intensity in XMCD spectrum.

+ Co atoms and nanoparticles on Pt have enhanced orbital moments up to 1.1 $\mu_B$

Element-specific Magnetization Reversal
Element-specific Magnetization Reversal
Monitoring field dependence of XMCD

⇒ Element-specific information on magnetization reversal in complex magnetic nanostructures.
**Element-Specific Magnetization Reversal**

![Graph showing XMCD for Ni, Fe, and Co layers with applied fields of +0.2 and -0.2 T.](image)

**Figures:**
- Upper graph: XMCD vs. photon energy (eV) for Ni, Fe, and Co layers at 18 ML Ni, 8,10 ML Fe, and 5 ML Co.
- Lower graph: XMCD (% vs. field (mT) for 8 ML Fe layers.

**Legend:**
- Ni
- Fe
- Co

**Analysis:**
- The graph shows the XMCD signal for different layers, illustrating the magnetic properties under varying applied fields.

**Notes:**
- The XMCD signal is magnified by a factor of 3 for Fe and Ni layers.
- The XMCD signal shows a clear reversal for different layers and applied fields.

**Conclusion:**
- The element-specific magnetization reversal is observed for Ni, Fe, and Co layers under controlled magnetic fields.
Element-specific Magnetization Reversal

18 ML Ni
8,10 ML Fe
5 ML Co

XMCD (%)
field (mT)
Ni
Fe
Co

H=+0.2 T
H=-0.2 T

XMCD (arb. units)
700 750 800 850

XMCD
photon energy (eV)

H=+0.2 T
H=-0.2 T

EA (arb. units)
10 ML Fe
8,10 ML Fe
8 ML Fe

XMCD (%)
field (mT)
Ni
Fe
Co

10 ML Fe
**Magnetic Interfaces**

+ Weak Mn XMCD signal
  ⇒ Uncompensated Mn at Co/IrMn interface
+ Same sign of XMCD signal for Co and Mn
  ⇒ Parallel coupling of Co and Mn moments
+ Nominal thickness of uncompensated interface moments: (0.5±0.1)ML

Uncompensated interface moments play critical role in exchange bias phenomena = field cooling of ferromagnet/antiferromagnet bilayer inducing additional magnetic anisotropy.
MAGNETIC INTERFACES

+ Biased sample: Small vertical loop shift indicating pinned interface moments!
+ Unbiased sample: no vertical loop shift

X-Ray Linear Dichroism:

+ Difference in x-ray absorption for different linear polarization direction relative to crystalline and/or spin axis.

+ Due to the anisotropic charge distribution about the absorbing atom caused by bonding and/or magnetic order.

+ “Search Light Effect”: X-ray absorption of linear polarized x rays proportional to density of empty valence states in direction of electric field vector E.
**STRUCTURAL CHANGES IN PbZr$_{0.2}$Ti$_{0.8}$O$_3$**

+ Spontaneous electric polarization due to off-center shift of Ti$^{4+}$, Zr$^{4+}$ associated with tetragonal distortion $\Leftrightarrow$ linear dichroism

+ Reversing ferroelectric polarization changes XA $\Leftrightarrow$ Change in tetragonal distortion

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Isotropic \( d \) electron charge density \implies \text{No polarization dependence}

Magnetically aligned system \implies \text{Spin-orbit coupling distorts charge density} \implies \text{Polarization dependence}

+ \( I_{\text{XMLD}} = I_{||} - I_{\perp} \propto \langle m^2 \rangle, \langle m^2 \rangle = \text{expectation value of square of atomic magnetic moment} \)

+ XMLD allows investigating ferri- and ferromagnets as well as antiferromagnets

+ XMLD spectral shape and angular dependence are determined by magnetic order and lattice symmetry
**X-Ray Magnetic Linear Dichroism**

Isotropic $d$ electron charge density
⇒ No polarization dependence

Magnetically aligned system
⇒ Spin-orbit coupling distorts charge density
⇒ Polarization dependence

$+ I_{XMLD} = I_{||} - I_{\perp} \propto \langle m^2 \rangle$, $\langle m^2 \rangle = \text{expectation value of square of atomic magnetic moment}$

$+ $ XMLD allows investigating ferri- and ferromagnets as well as antiferromagnets

$+ $ XMLD spectral shape and angular dependence are determined by magnetic order and lattice symmetry
MAGNETIC COUPLING AT INTERFACES

La$_{0.7}$Sr$_{0.3}$MnO$_3$ (LSMO) ferromagnet
La$_{0.7}$Sr$_{0.3}$FeO$_3$ (LSFO) antiferromagnet

$\Rightarrow$ Perpendicular coupling at LSMO/LSFO interface

**PLANAR DOMAIN WALL**

**XMCD, XA (arb. units)**

- **Co XMCD (arb. units)**
  - Field (T)
  - Applied field (T)
  - Co XMCD (arb. units)

- **Ni L₂ ratio**
  - Wall angle
  - Applied field (T)

**NiO**

**Co/NiO**

**A. Scholl et al.,**
Magnetic Microscopy

Nature

- Flea
- Human hair ~30 μm wide
- Red blood cells & white cell ~ 5 μm
- Virus ~ 200 nm
- DNA helix ~3 nm width
- Water molecule
- Atom

Technology

- Head of a pin ~ 1 mm
- DVD track
- 1 μm Electrodes connected with nanotubes
- Carbon nanotube ~ 2 nm diameter
- Atomic corral ~ 14 nm diameter

The Microworld

The Nanoworld

AFM & FM domains
Recorded “bits”
Spin injection
Media grains
Nano-particles
Advanced Light Source
An Office of Science User Facility
U.S. Department of ENERGY
Office of Science
BERKELEY LAB
10-50 nm spatial resolution

J. Stöhr, H.C. Siegmann, Magnetism (Springer)
+ Images taken with left and right circularly polarized x-rays at photon energies with XMCD, i.e. Co $L_3$ edge, provide magnetic contrast and domain images.
Taking into account the geometry dependence of the Ni XMLD signal

$\Rightarrow$ Perpendicular coupling of Co and NiO moments at the interface.

First direct observation of vortex state in antiferromagnetic CoO and NiO disks in Fe/CoO and Fe/NiO bilayers using XMCD and XMLD.

Two types of AFM vortices:
- conventional curling vortex as in ferromagnets
- divergent vortex, forbidden in ferromagnets
- thickness dependence of magnetic interface coupling

J. Wu et al., Nature Phys. 7, 303 (2011)
+ BiFeO$_3$ – multiferroic = ferroelectric + antiferromagnetic
+ Compressive strain on rhombohedral phase (R-phase) induced by substrate
⇒ tetragonal-like phase (T-phase)
+ Partial relaxation of epitaxial strain
⇒ Formation of a nanoscale mixture of T- and R-phases

Q. He et al., Nature Comm. 2, 225 (2011)
Highly distorted R-phase is the source of enhanced magnetic moment in the XMCD image.

Q. He et al., Nature Comm. 2, 225 (2011)
Energy reservoirs in a ferromagnetic metal
Deposition of energy in one reservoir

⇒ Non-equilibrium distribution and subsequent relation through energy and angular momentum exchange

J. Stöhr, H.C. Siegmann, Magnetism (Springer)
Pulse length 70 ps

- 256-320 bunches for 500mA beam current
- Possibility of one or two 5mA "camshaft" bunches in filling gaps
- Bunch spacing:
  - multibunch mode: 2 ns
  - two-bunch mode: 328 ns
- Pulse length 70ps

<300 fs x ray pulses though “laser bunch-slicing technique”
**ULTRAFAST DYNAMICS OF SPIN AND ORBITAL MOMENTS**

- Orbital ($L$) and spin ($S$) magnetic moments can change with total angular momentum is conserved.
- Efficient transfer between $L$ and $S$ through spin–orbit interaction in solids
- Transfer between $L$ and $S$ occurs on fs timescales.

- $\text{Co}_{0.5}\text{Pt}_{0.5}$ with perpendicular magnetic anisotropy
- 60 fs optical laser pulses change magnetization
- Dynamics probed with XMCD using 120fs x-ray pulses

- Linear relation connects $\text{Co} \ L_3$ and $L_2$ XMCD with $L_z$ and $S_z$ using sum rules

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**Ultrafast Dynamics of Spin and Orbital Moments**

- **Thermalization:** Faster decrease of orbital moment
- **Theory:** Orbital magnetic moment strongly correlated with magnetocrystalline anisotropy
- **Reduction in orbital moment**
  \[ \Leftrightarrow \text{Reduction in magnetocrystalline anisotropy} \]
- **Typically observed at elevated temperatures in static measurements as well**

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**Graphical Representation:**
- **Spin and orbital moments**
- **Ratio**
- **Delay (ps)**

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**C. Boeglin, et al.,**
*Nature 465, 458 (2010)*

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**Electron-phonon relaxation time**

**Electron-spin relaxation time**

**Spin-lattice relaxation time**
J. Stöhr, H.C. Siegmann
Magnetism—From Fundamentals to Nanoscale Dynamics
Springer

D. Attwood
Soft X-Rays and Extreme Ultraviolet Radiation: Principles and Applications
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DOE-BES
**Magnetism as seen with X-Rays**

- X-ray absorption, XA
- X-ray magnetic circular dichroism, XMCD
- X-ray magnetic linear dichroism, XMLD
- X-ray magnetic microscopy
- Magnetization Dynamics

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**FeCo-GaAs**


**Co$_{0.5}$Pd$_{0.5}$**


**CoO**


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**CoO-Co**