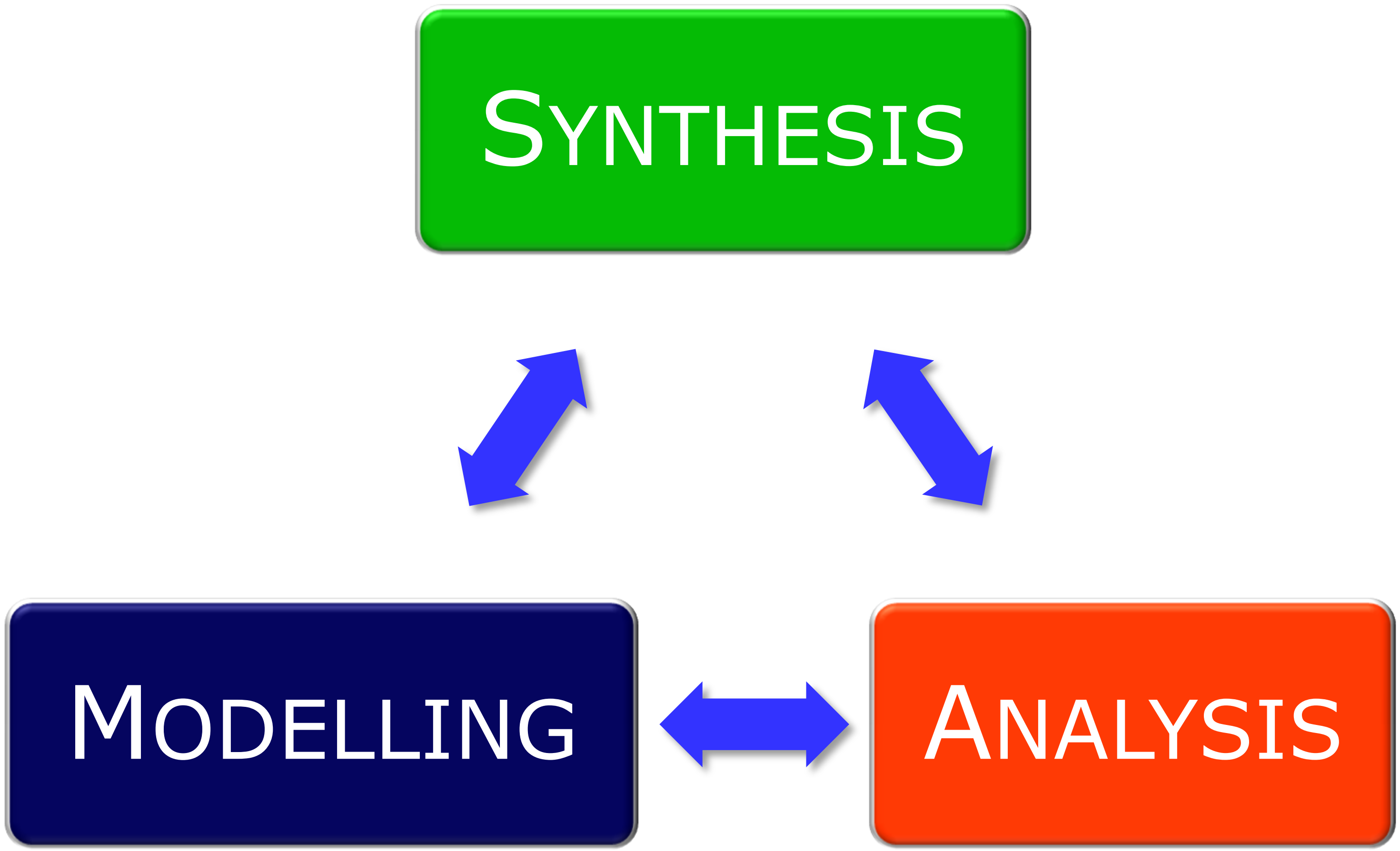
Advanced Soft X-Ray Microscopy for Nanomaterials Sciences

Peter Fischer
CXRO/MSD

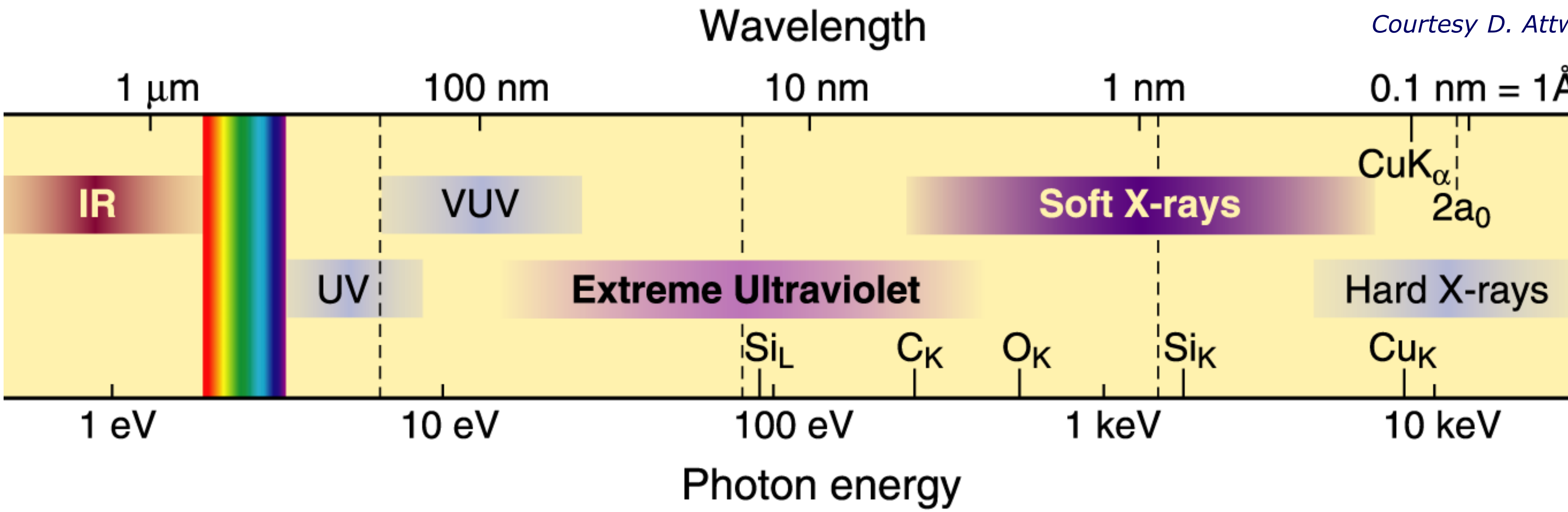
- Materials aspects in nanosciences
- Soft X-ray microscopies as valuable tools
- Current achievements and future directions

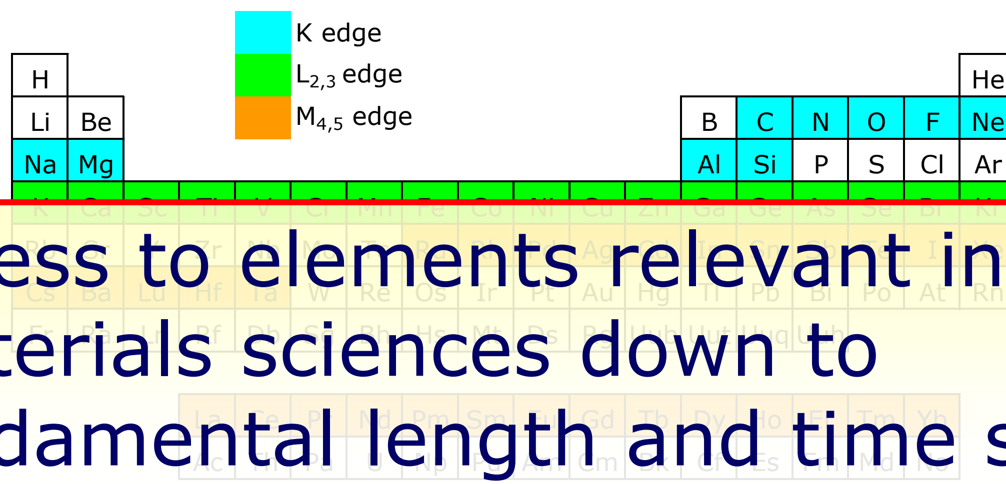
Key elements to nanoscience



Why soft X-rays?

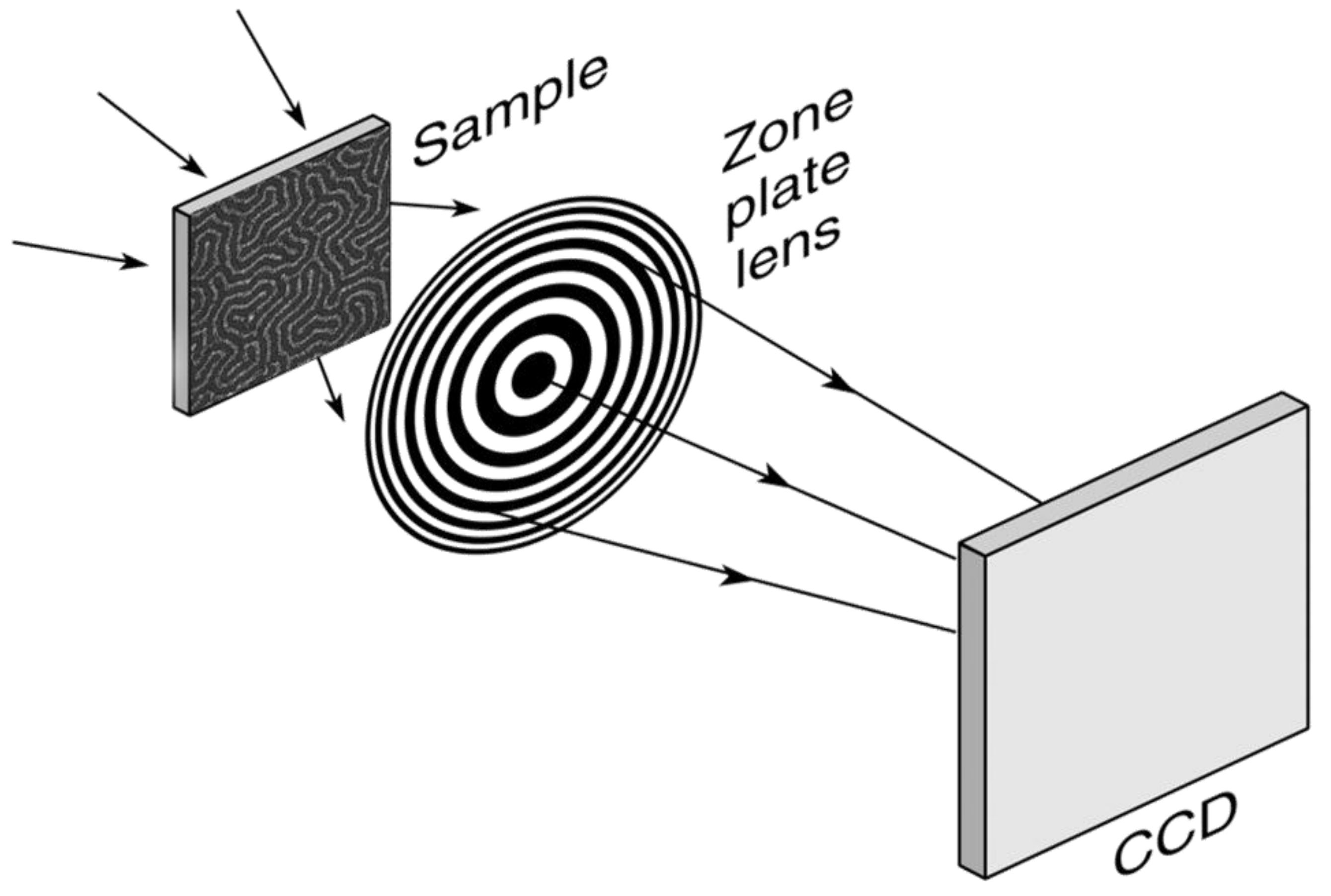
Courtesy D. Attwood





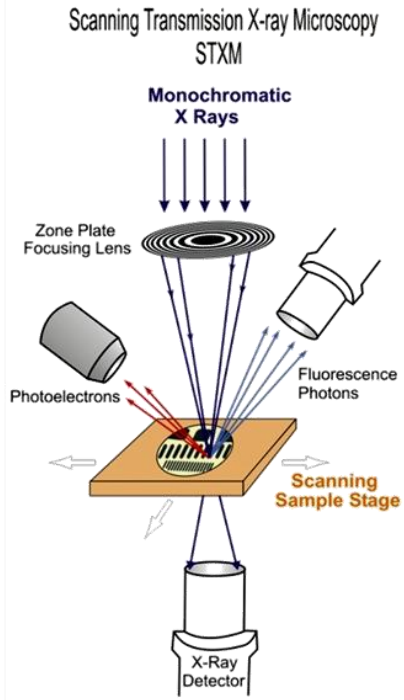
⇒ access to elements relevant in materials sciences down to fundamental length and time scales

Imaging with soft X-rays

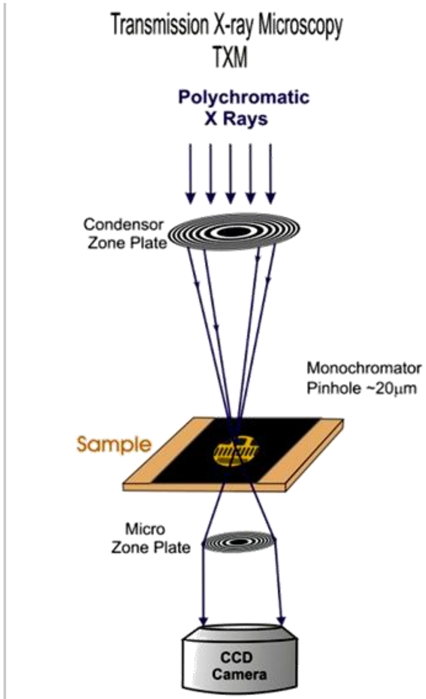


Various X-ray imaging techniques

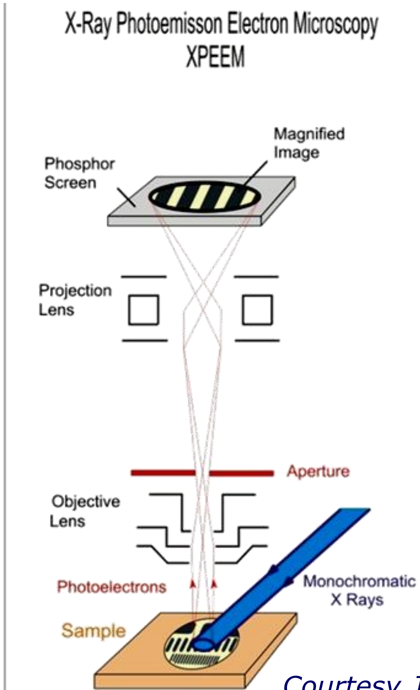
in real space



J.B. Kortright, et al., in: X-Ray Microscopy, AIP **507** 49 (2000)



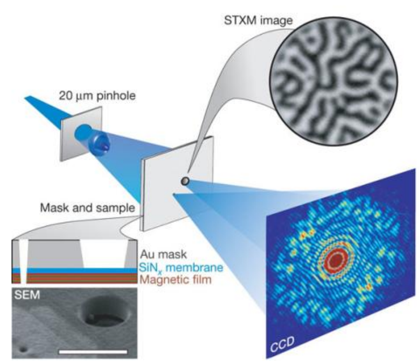
P. Fischer, et al., Z. f. Phys. **B101** 313 (1996)



Courtesy J. Stöhr

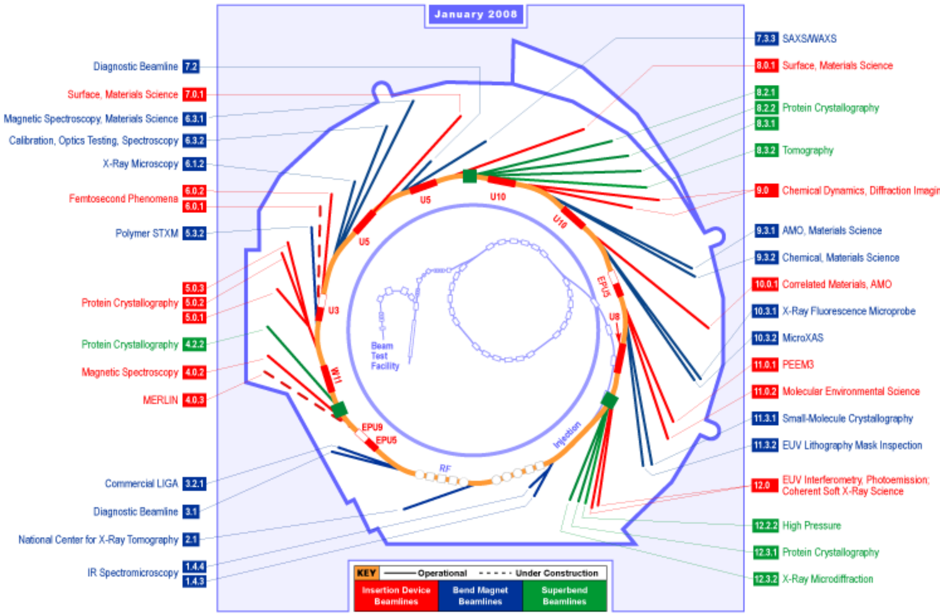
J. Stoehr, et al., *Science* **259** 658 (1993)

in reciprocal space



S. Eisebitt, et al. *Nature* **432**, 885 (2004)

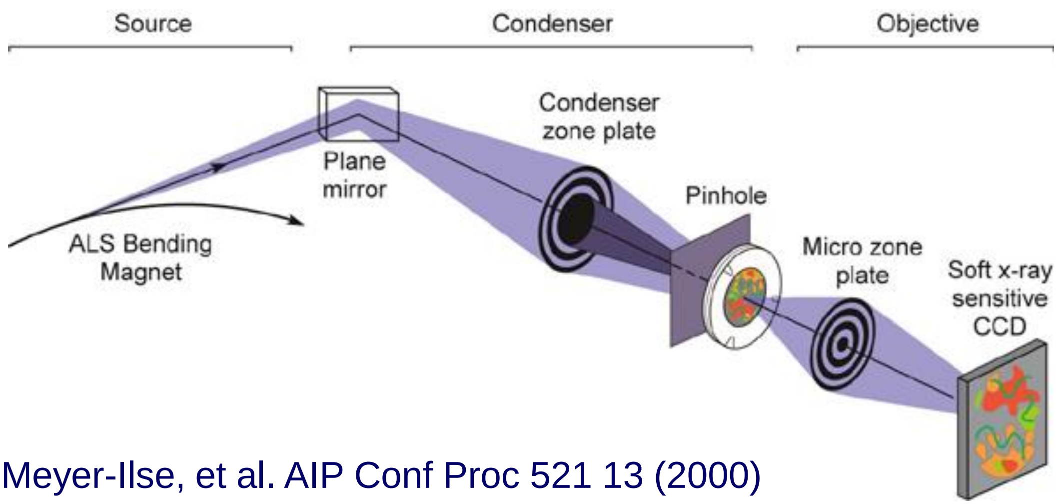
Suite of X-ray microscopes at ALS



- PEEM-3 (11.0.1)
- STXM (11.0.2)
- STXM (5.3.2)
- Coherent/diffractive imaging (9.0.1)
- XM-2 (2.1)
- XM-1 (6.1.2) (operated since 1994)

Full-field TXMs

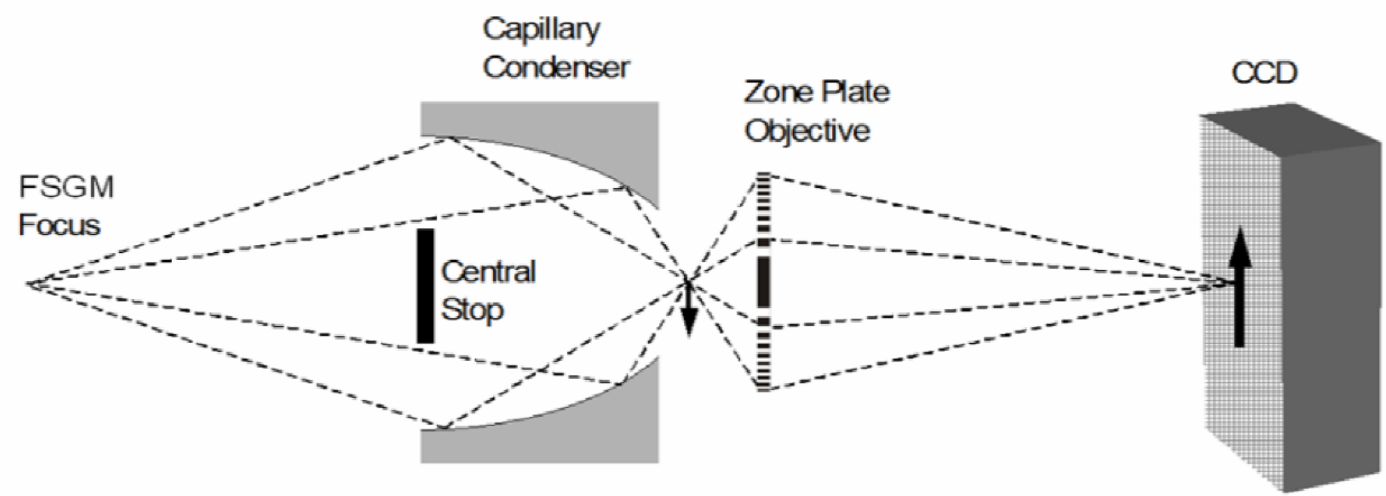
ALS



W. Meyer-Ilse, et al. AIP Conf Proc 521 13 (2000)

BESSY II

U41



P. Guttmann et al, JOP Conf Ser. 186 012064 (2009)

From XM-1 to XM-3

	XM-1	XM-3
Spectral resolution	400-600 (elemental mapping)	2000-4000 (true microspectroscopy)
Photon E-range	500-1200eV	280-2000eV
Polarization	elliptical polarisation via movable aperture	EPU: circular and linear
Sample environment	magnetic field <0.2T ambient T	magnetic fields 2T variable T (4-600K) variable pressure In-situ experiments
Spatial resolution	25nm (standard) 10nm (demo)	<15nm (standard) <<10nm (demo)
Time resolution	<100ps	<100ps
tomography	(yes) limited capabilities	YES, optimized for materials science
reflection geometry	NO	YES, access to interfaces

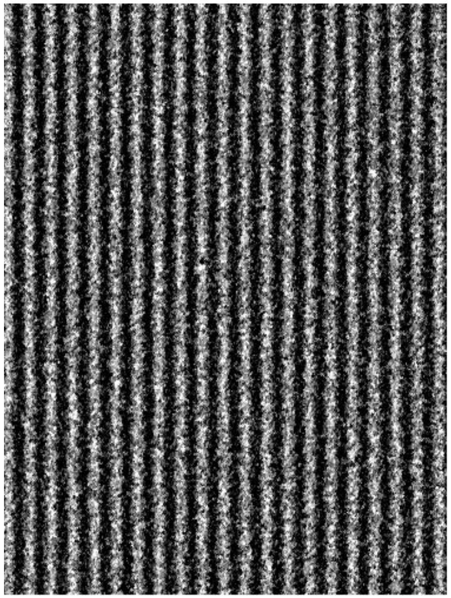
Progress in spatial resolution

BERKELEY LAB

Halbach Award 2010

W. Chao, CXRO

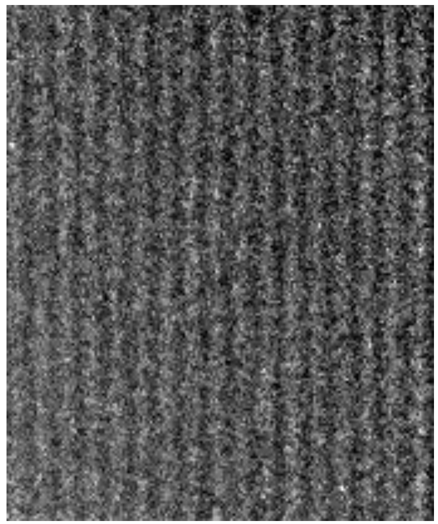
15 nm



2005

W. Chao, et al, Nature ***435*** 1210 (2005)

12 nm

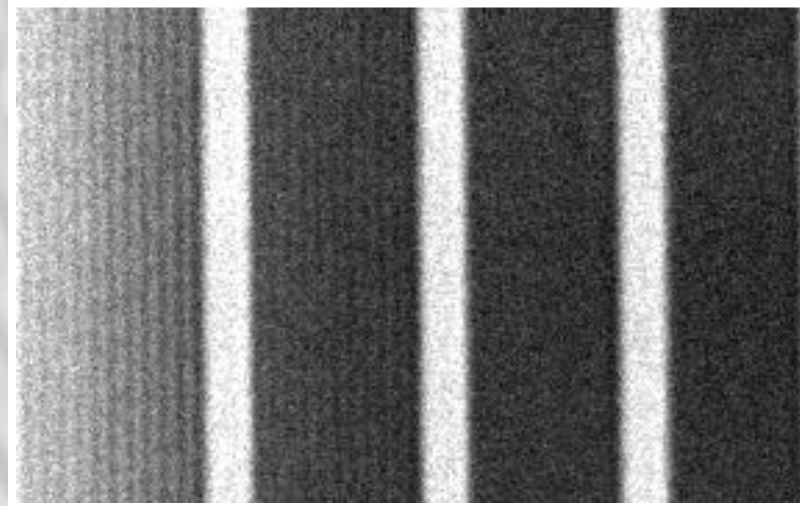


2009

W. Chao, et al, Optics Express, ***17****(20) 17669 (2009)*

10 nm

11 nm | 10 nm | 9 nm | 8 nm



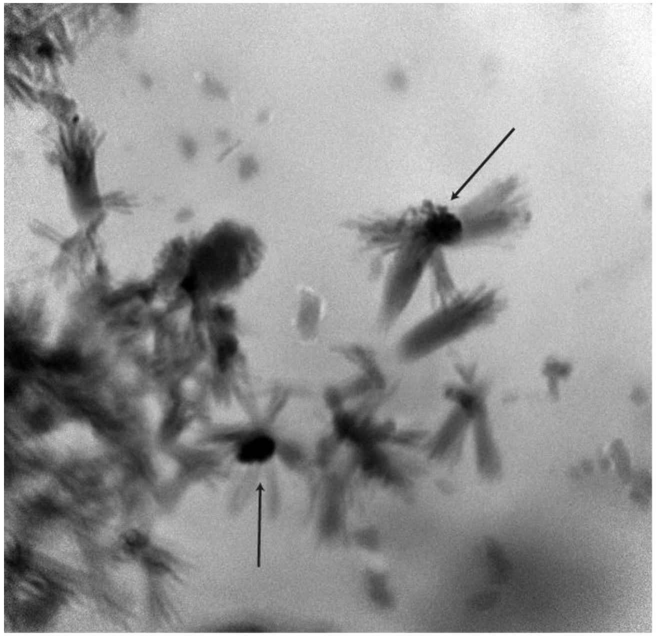
2010

W. Chao, et al, (2010) in preparation

Development of green cements

M. Mancio, UCB

Problem to our environment: The production of Portland cement is responsible for 5-8% of the CO_2 generation worldwide.
Challenge: New cements have to demonstrate long-term stability and durability for over 120 years.



This 2d TXM image seems to show that the "sheet of wheat" have a core which acts as a nucleation point (see arrows)

This assumption is wrong as verified by rotating the sample (done at BESSY II)

Work with Brisard, Levitz and Chae

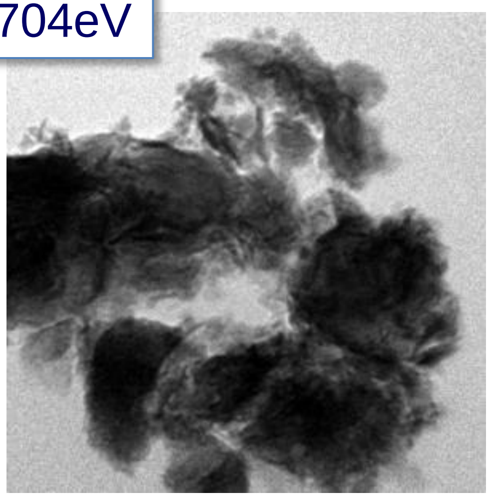
Colloids in soil science

J. Thieme NSLSII

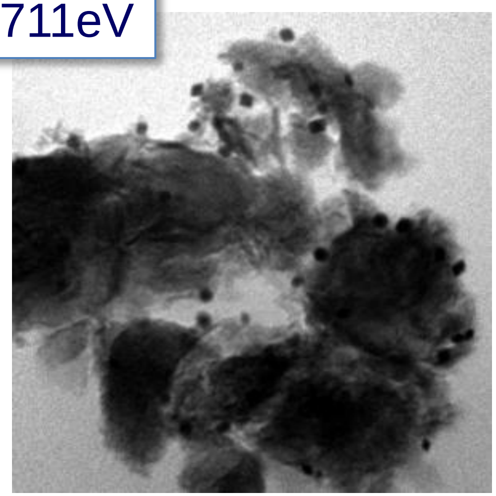
- Fe is abundant in soils and groundwater aquifers, but occurs in various oxidation states
- Problem: Too much iron in water used in engineering purposes may cause corrosion

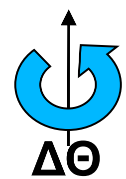
XM-1 Elemental mapping Stereomicroscopy

704eV

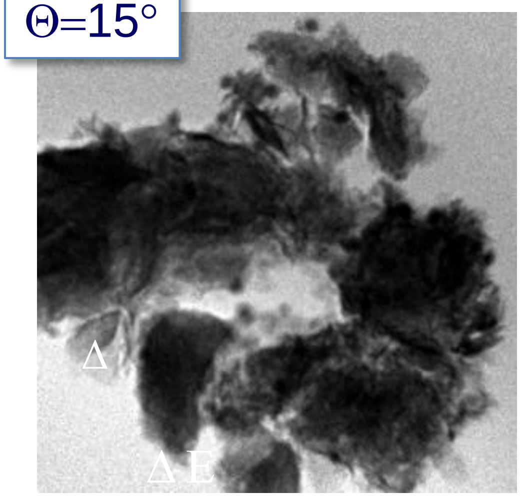


711eV





Θ=15°



S Gleber et al. J. of Microscopy 235 199 (2009)

But where exactly are the elements and what oxidation state are they in?

Atomically controlled synthesis of Mono and Bimetallic clusters in catalysis

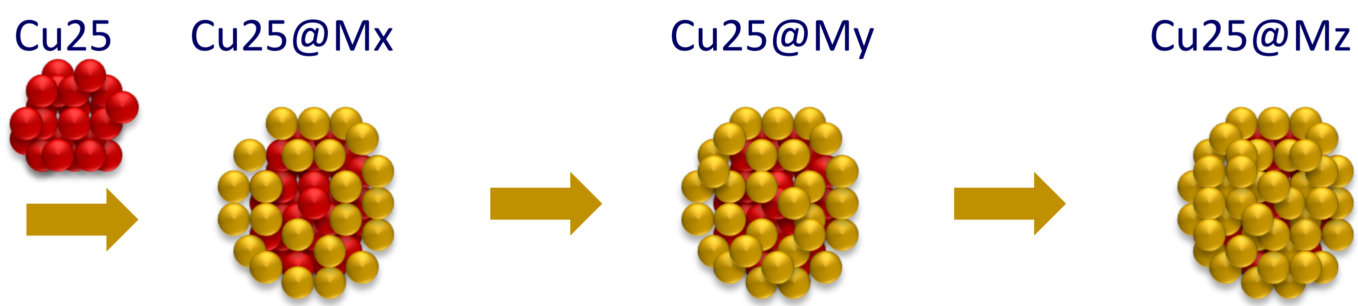
Center for Atomic-Level Catalyst Design

DOE Energy Frontier Research Center

C. Kumar LSU

Core-shell nanoparticles and Nanostructured Mesoporous Materials (NMMs) offer a great opportunity to modulate the properties of materials.
Delineating the size, size distribution, shape, core-shell dependent electronic and geometric properties of nanomaterials allows to tune specific properties such as magnetism, heat capacity, optical, photoconductivity and charge transfer, electronic, melting point, catalytic by varying the size/shape of the nanomaterials.

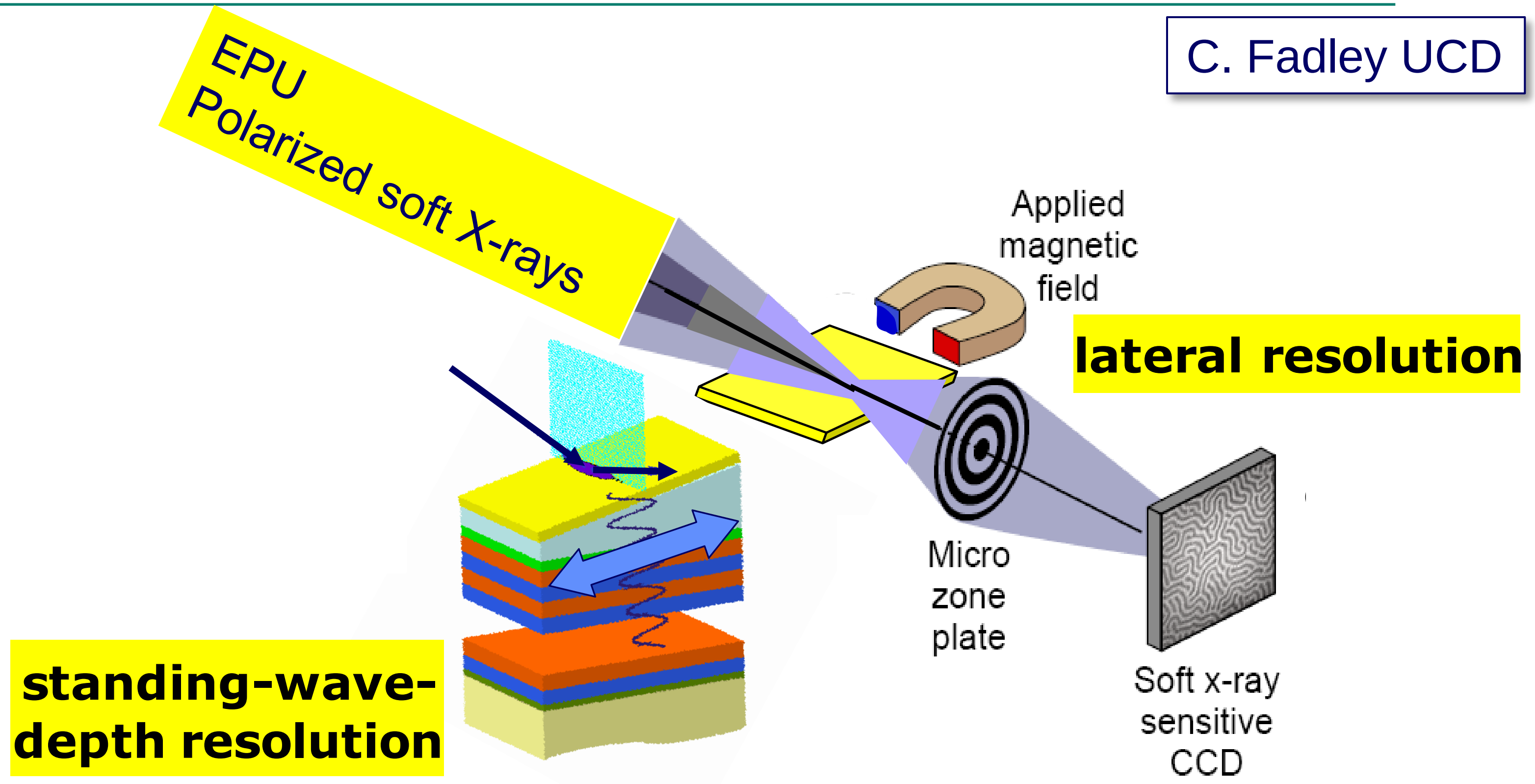
NMMs is a new class of catalysts and are one of the key class of materials investigated by the DOE funded EFRC at LSU.



Soft X-ray nanotomography is an essential tool to characterize these 3-dim structures.

Imaging buried interfaces

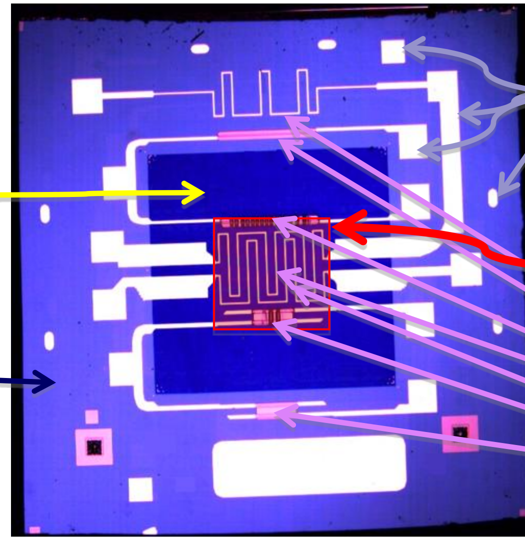
C. Fadley UCD



- Combining in a unique way high lateral resolution with depth resolution ⇒ access to magnetic interface
- Goal: combining with time resolution to study the dynamics at interfaces

Nanocalorimetry of Magnetic Thin Films

C. Baldasseroni, UCB



thin a-SiN_x membrane

Si frame

Contacts, leads, and test areas

Sample area 2 mm

Heater and thermometers (sample area and matching on frame)

Calorimetry: powerful technique, hard to do on thin films!

- Membrane-based micro/nanocalorimetry, calorimeter-on-a-chip; 0.3-800K, 0-8T; ~2% accuracy for <1μg, 20 nm thick films
- Electron, phonon, magnon density of states; crystal field levels; phase transitions; entropy, enthalpy
- Basis for proposed integrated *in situ* ALS heater/ sample imaging stage

2006 APS Keithley Instrumentation Award

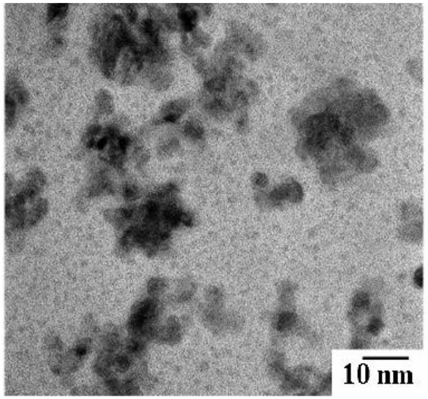
D.R. Queen and F. Hellman, Rev. Sci. Instrum. 80, 063901 (2009)

Membrane heater stage for in situ soft x-ray microscopy

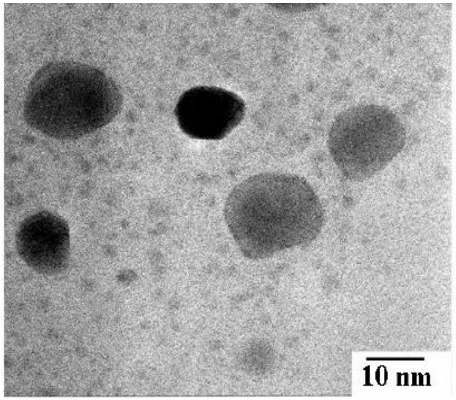
F. Hellman, UCB

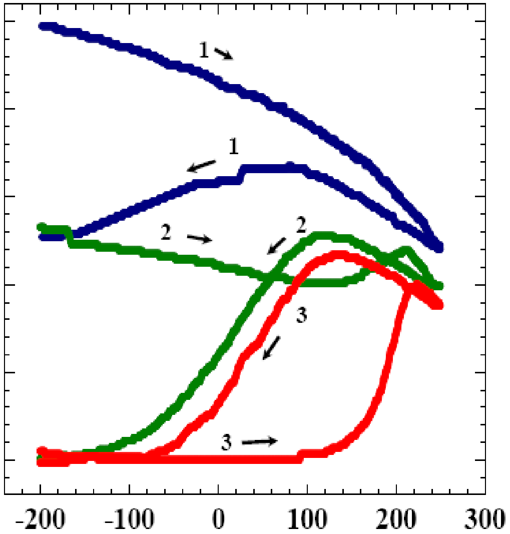
Example: high anisotropy $Fe_{38}Rh_{62}$ nanoparticles produced from amorphous alloy by annealing
GOAL: image DURING heating to understand the evolution
Longer term goal: measure thermodynamics of process

as-deposited



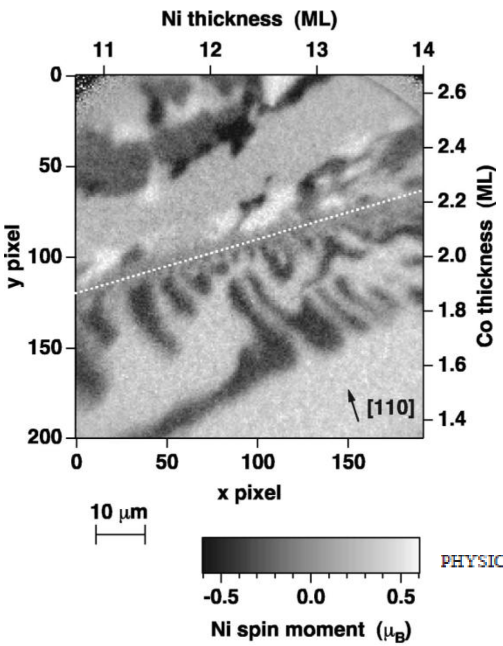
after annealing

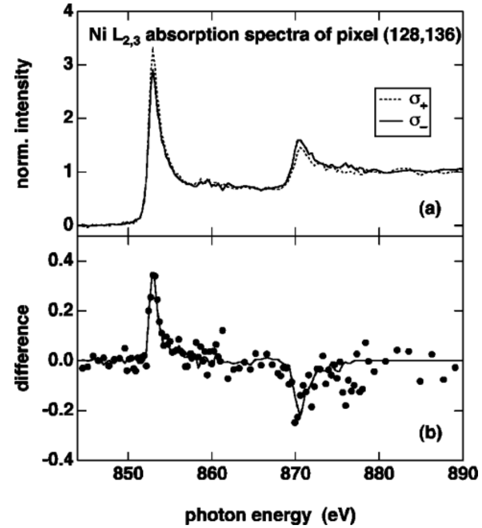


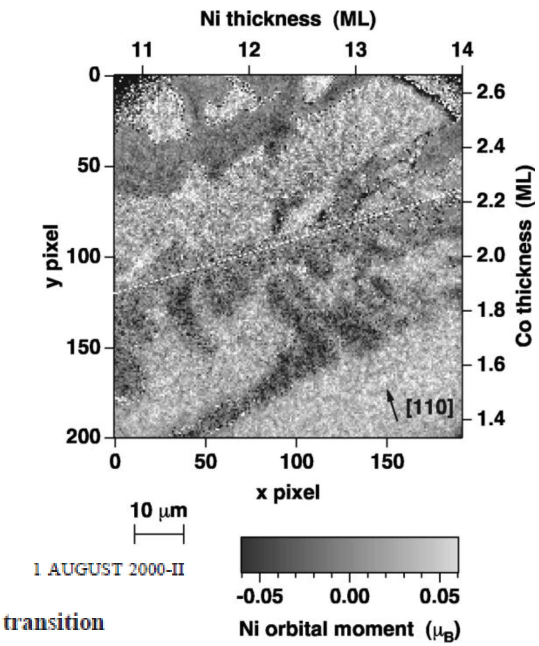


courtesy T. Suzuki/H.Yu Yu Ko

Mapping spin and orbital moments







PHYSICAL REVIEW B VOLUME 62, NUMBER 6 1 AUGUST 2000-II

Magnetic-circular-dichroism microspectroscopy at the spin reorientation transition in Ni(001) films

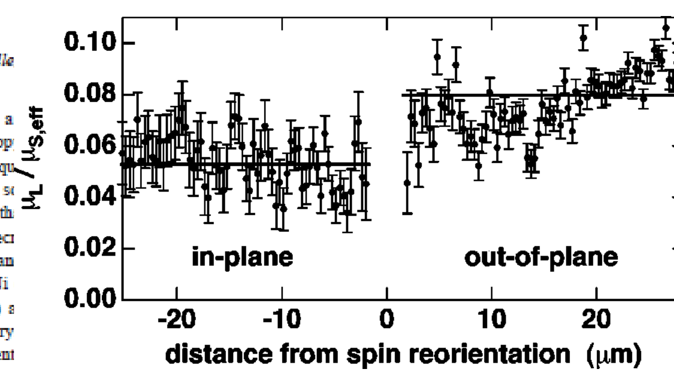
W. Kuch, J. Gilles, and S. S. Kang
Max-Planck-Institut für Mikrostrukturphysik, Weinberg 2, D-06120 Halle, Germany

S. Imada and S. Suga
Osaka University, Graduate School of Engineering Science, 1–3 Machikaneyama, Toyonaka 560-8531, Japan

J. Kirschner
Max-Planck-Institut für Mikrostrukturphysik, Weinberg 2, D-06120 Halle

(Received 10 November 1999)

The spin reorientation transition in fcc Co/Ni/Cu(001) epitaxial ultrathin films as a film thickness is studied by the combination of photoelectron emission microscop circular-dichroism spectroscopy at the Ni $L_{2,3}$ edge. This microspectroscopic techniqu local quantitative information about the Ni magnetic properties on a submicrometer sc the thickness range of 1.4–2.6 atomic monolayers (ML) Co and 11–14 ML Ni show th occurs as a function of both Co and Ni thicknesses. Increasing the Co thickness or decr leads to a switching of the magnetic easy axis from [001] out-of-plane to ⟨110⟩ in-plan effective Ni spin moment similar to the bulk magnetic moment is observed. The Ni ratio shows distinctly different values for out-of-plane magnetization (0.080±0.005) a tion (0.053±0.005). This is discussed in terms of the connection to the Ni magnetocry domain density of the perpendicular magnetization increases towards the spin reorient

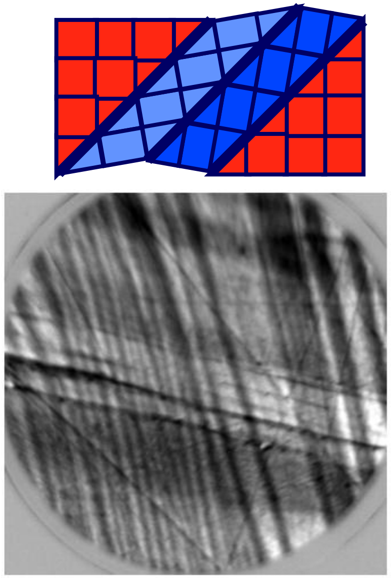


Heusler alloys:
Shape memory phase transitions in Ni_2MnGa

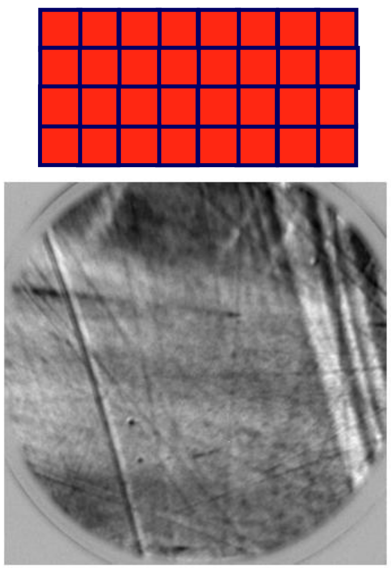
C. Jenkins, ALS

Heusler compounds are a plethora of many spintronics and multiferroic applications incl. MTJs and spin momentum transfer devices.

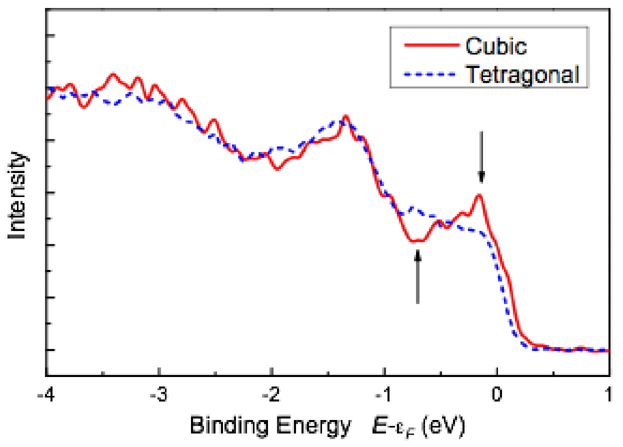
Tetragonal (low T)



Cubic (high T)

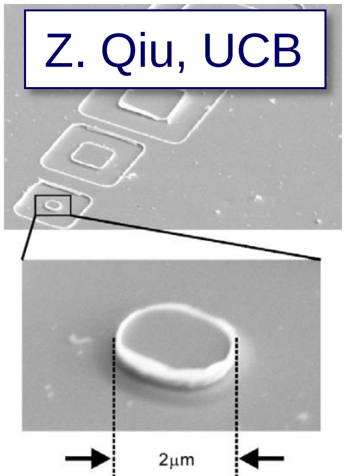


Photoemission

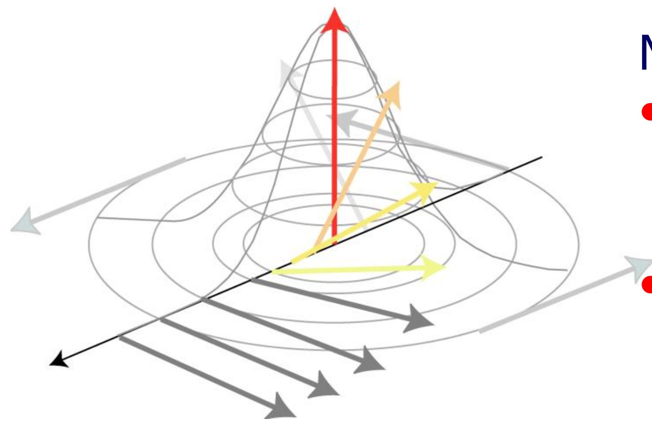


- Martensitic phase transition between cubic and tetragonal phase
- Magnetic easy axis coupled to tetragonal distortion
  ⇒ ferromagnetically driven, ferroelastic actuation
- Anisotropy vs. orbital moments in Heusler alloys

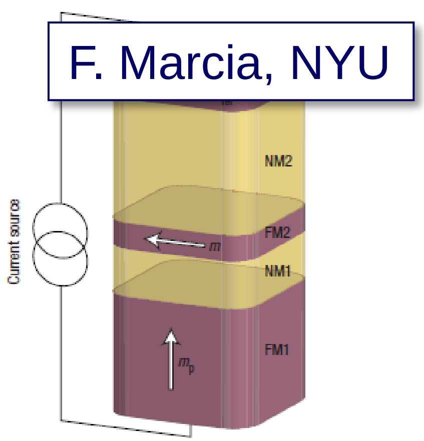
Fast dynamics in nanostructures



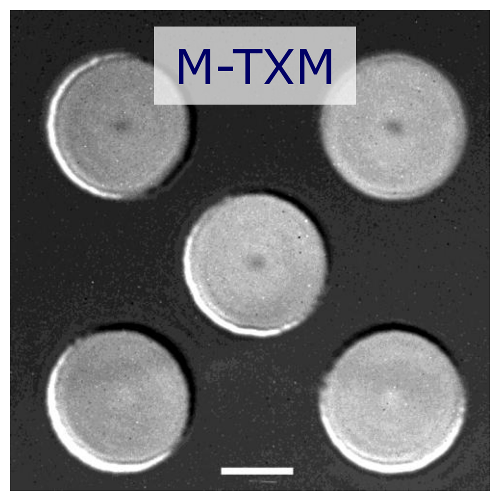
J. Wu et al, J. Phys.: Condens. Matter **22** (2010) 342001



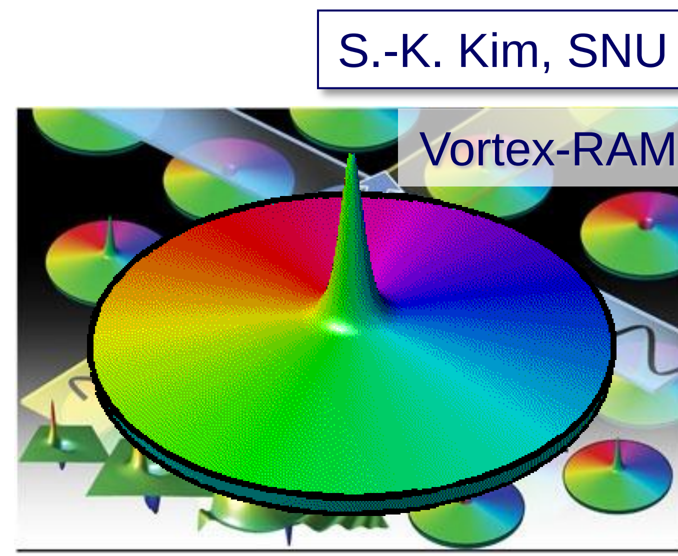
Magnetic vortices
- Chirality
  in-plane circular domain structure
- Polarity
  out-of-plane component of magnetization



A. Kent, Nature Mat, **6** 399 (2007)



P. Fischer, S. Kasai, A. Thiaville et al., in preparation (2010)

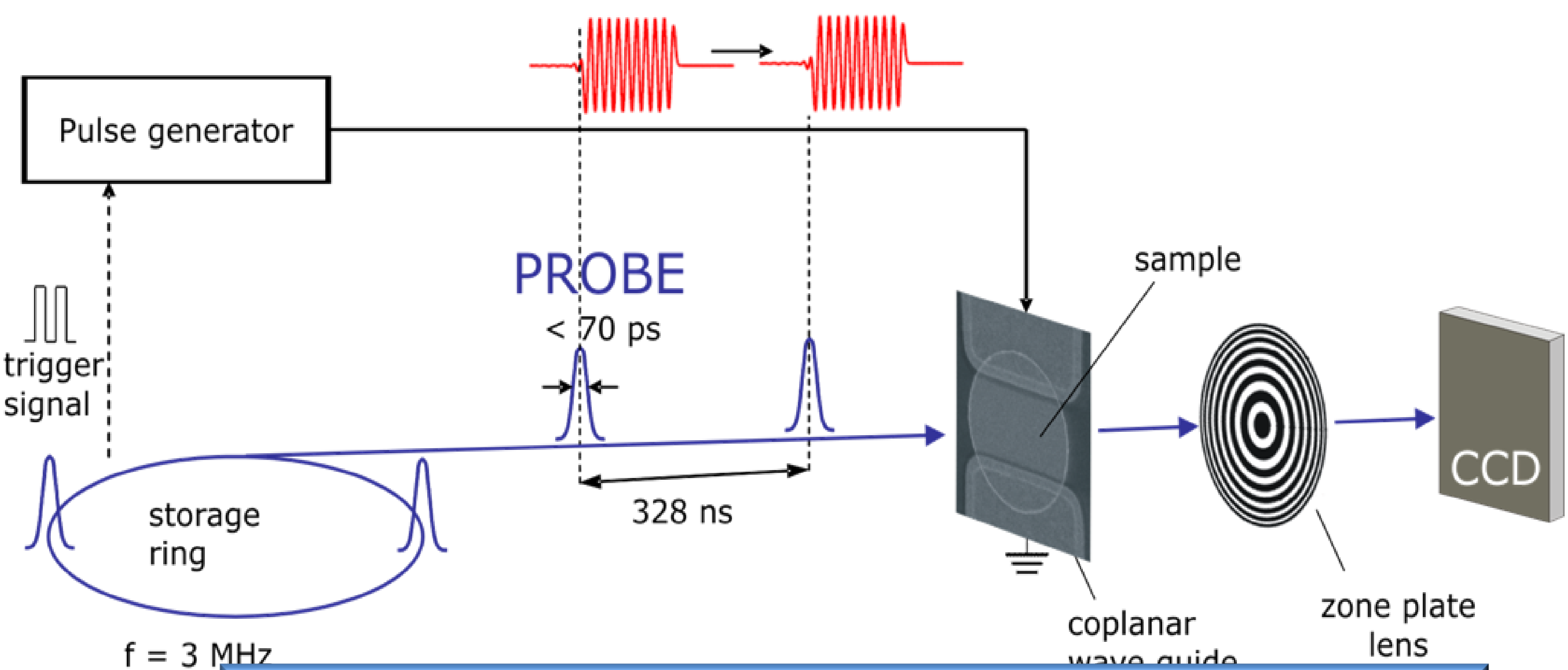


S.-K. Kim et al. APL 92, 022509 (2008)

Courtesy S.-K. Kim

Stroboscopic pump-probe setup for time resolved soft X-ray microscopy

- $j=1.3\times10^{11}$ A/m^2
- $10^{8\text{-}9}$ pump-probe events/image

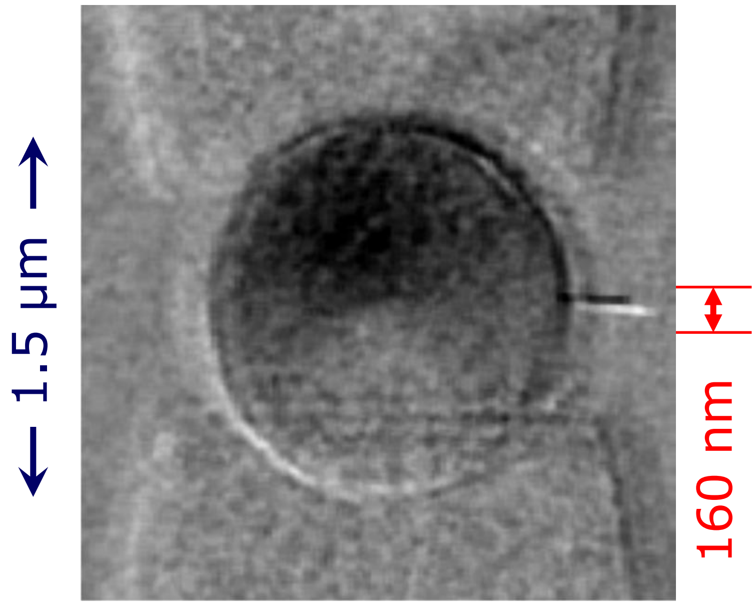


- perfect repeatability of the dynamics required
- no access to non-deterministic components e.g. spin fluctuations

S. Kasai, P. Fischer, M.-Y. Im, K. Yamada, Y. Nakatani, K. Kobayashi, H. Kohno, T. Ono, Phys Rev Lett 101, 237203 (2008)

Deriving spin polarisation of currents P

Permalloy ($Fe_{80}Ni_{20}$)
t=40 nm
f=220 MHz
Δt=0…9ns

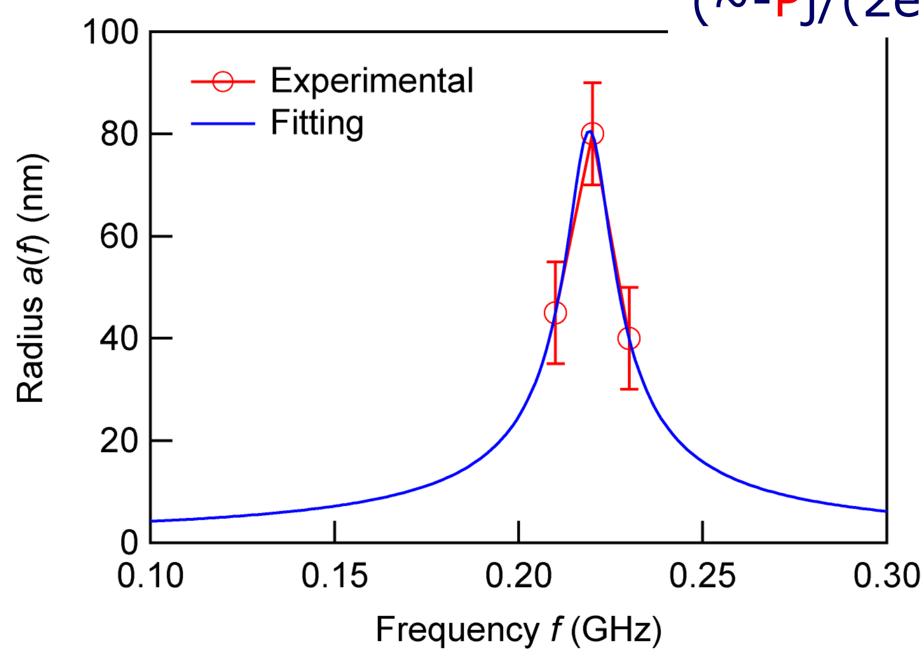


- CW rotation ⇒ polarity = -1
- gyration radius R~80 nm

Solving Thiele eqn with STT for u

$$\vec{G}\times\vec{v}+\alpha D\vec{v}+k\vec{X}-\vec{G}\times\vec{u}=0$$

drift velocity ($\sim$-Pj/(2eM$_s$))

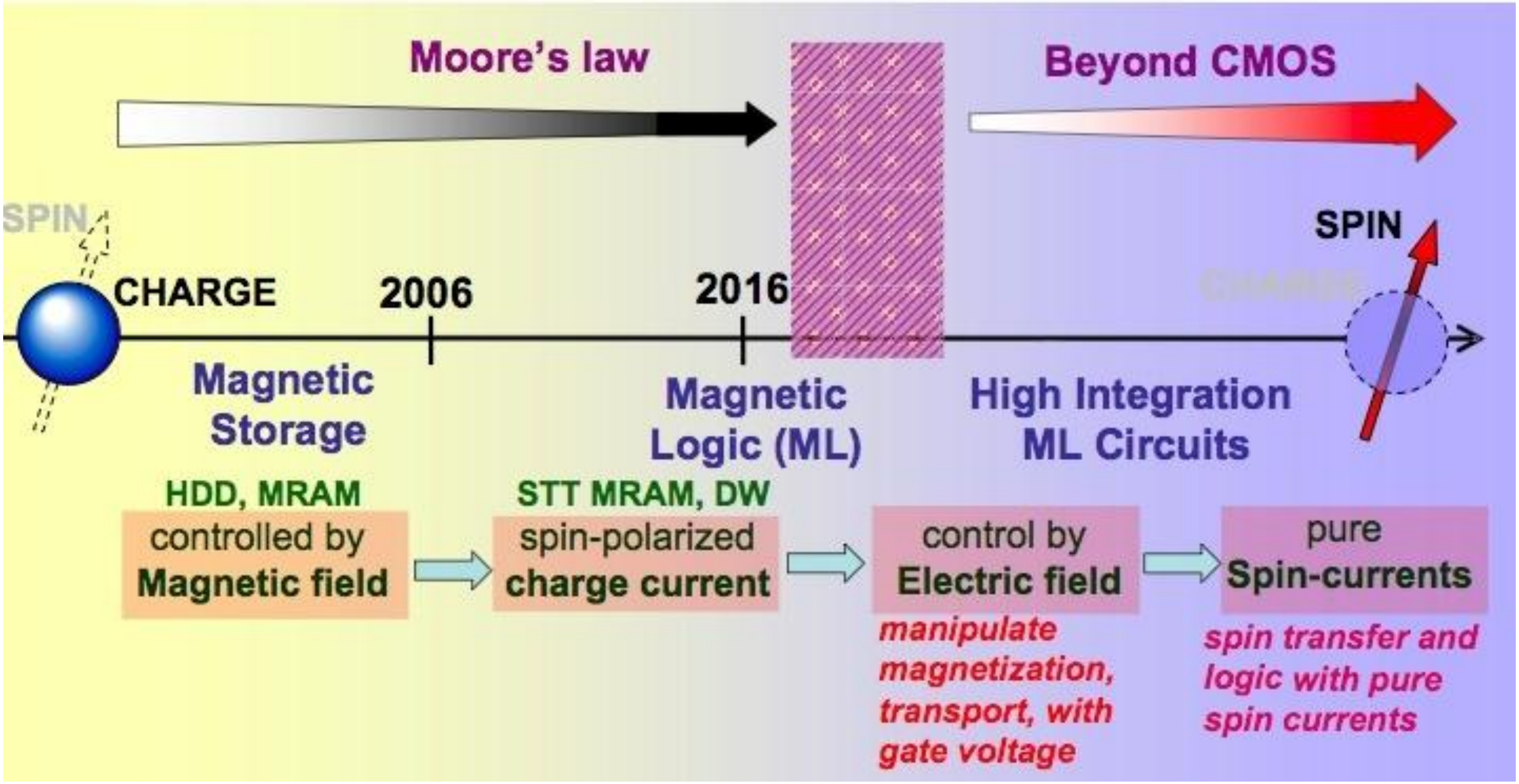


measuring R(ω) ⇒ *P*=0.67±0.15

S. Kasai, P. Fischer, M.-Y. Im, K. Yamada, Y. Nakatani, K. Kobayashi, H. Kohno, T. Ono, Phys Rev Lett 101, 237203 (2008)

The road to spin currents

A. Hoffmann, ANL

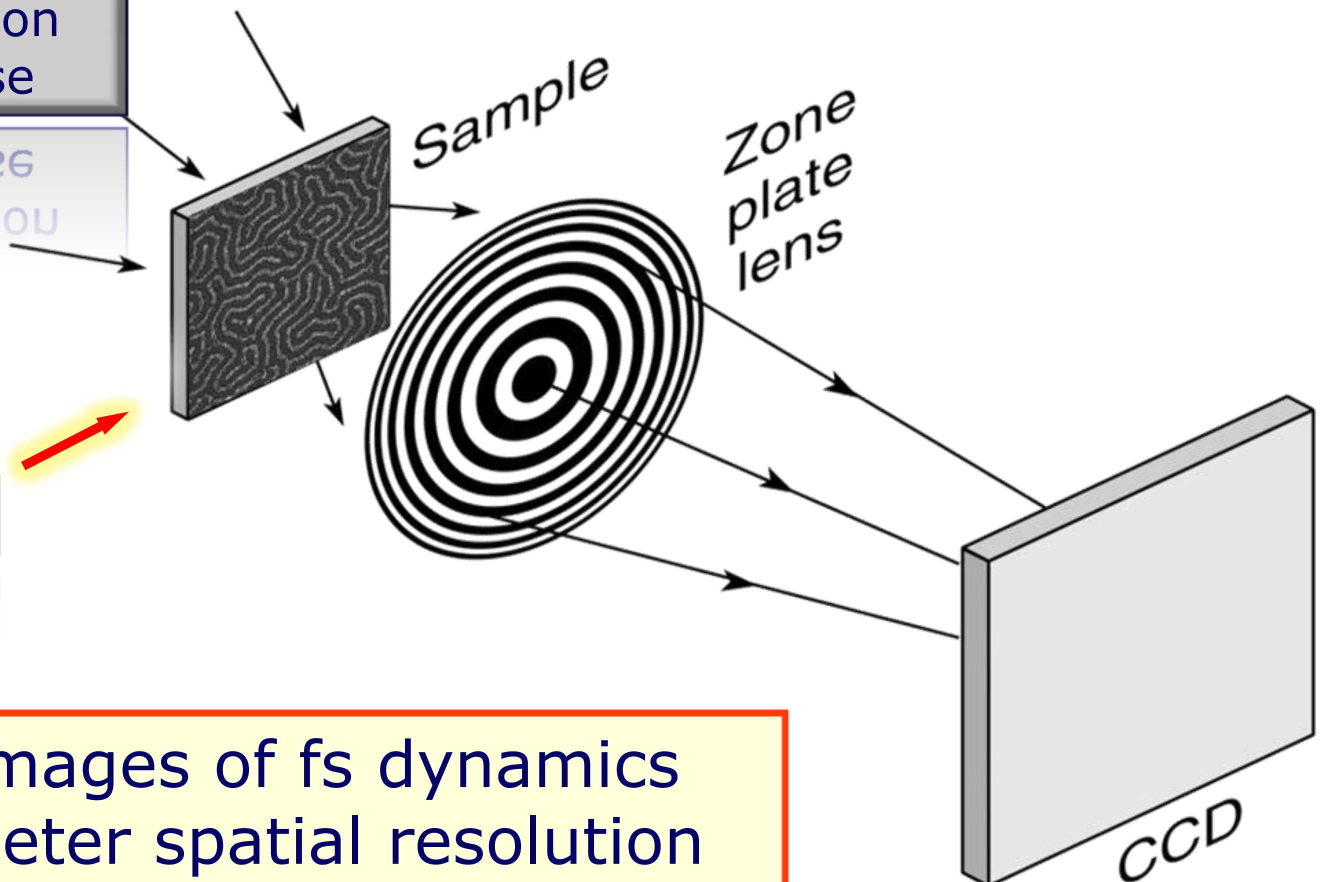


Courtesy C. Chappert

Future goal: imaging at the fsec scale

soft X-ray source
- fsec pulse length
- variable polarization
- high photons/pulse

high photons/pulse
variable polarization



pump stimulus

pump stimulus

snapshot images of fs dynamics
with nanometer spatial resolution

Agenda

10:00	P. Fischer (CXRO/LBNL)	"Overview of recent advances and future opportunities in soft X-ray microscopy"
10:30	A. Hoffmann (ANL)	"New Insights into Spin Relaxation from Pure Spin Currents"
11:00	S.-K. Kim (Seoul Natl. U)	"Soft x-ray microscopy study of vortex dynamics in soft magnetic dots at XM-1"
11:30	F. Marcia (NYU)	"Spin-wave interference patterns created by spin-torque oscillators"
12:00	LUNCH	
13:15	K. Jenkins (ALS/LBNL)	"Design and application of multifunctional manganese intermetallics"
13:45	C. Baldasseroni (UCB)	"Development of a membrane based nanocalorimeter as an X-ray transparent heater stage"
14:15	C.S. Fadley (LBNL/UCD)	"Adding depth resolution to soft x-ray photoelectron microscopy with standing-wave excitation: applications to spintronic nanostructures"
14:45	Q. Qiu (UCB)	"Microscopy study of magnetic nanostructures using X-rays"
15:15	BREAK	
15:35	C. Kumar (LSU)	"Design and Engineering of Core-shell Magnetic Nanomaterials – Current Challenges"
16:05	J. Thieme (NSLSII/BNL)	"Anthropogenic and natural nanoparticles in the environment – dynamics and interactions"
16:35	R. Chae (UCB)	"Probing the nanostructure and morphology of cementitious and pozzalonic materials using x-rays"
17:05	W. Chao (CXRO/LBNL)	"Ultrahigh Resolution Soft X-ray Zone Plate Microscopy at ALS"
17:35		Adjourn