Magnetism and Microstructure at Relevant Length Scales

Complementary Measurements with Electron and Photon Probes

Kannan M. Krishnan

Department of Materials Science
University of Washington, Seattle

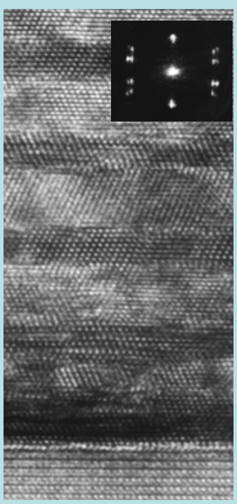
W. Grogger, G. Kusinski and E.C. Nelson - LBNL/UCB
M. E. Gomez and I. Schuller - UCSD
G. Denbeaux and A. Young - ALS/LBNL

Funding; DoE/BES, Max Kade Foundation, IBM, Campbell Endowment at UW

Invited Talk: INTERMAG Meeting, Amsterdam, April 2002

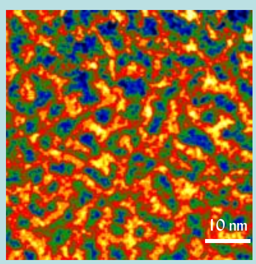
Relevant Microstructural Length Scales & Issues

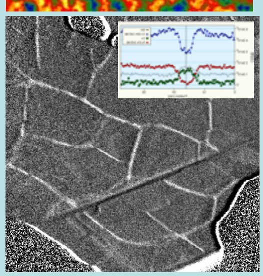
Structural



Thin Films & Multilayers
Interface Roughness
Segregation, Mixing etc.

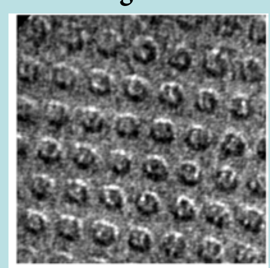
Chemical





Complex Microstructures
Grain Isolation, Second phase
Intergranular coupling etc.

Magnetic



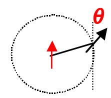
In situ dynamics
Magnetic reversal
Temperature and applied fields

Magnetic Materials

Interactions & Characteristic Length Scales

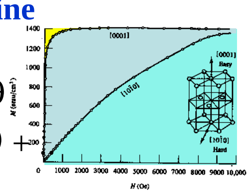
- **Exchange**:

$E_{ij} = -2 J_{ij} \mathbf{S}_i \cdot \mathbf{S}_j = J_{ij} |S|^2 \theta^2$ *for small θ*

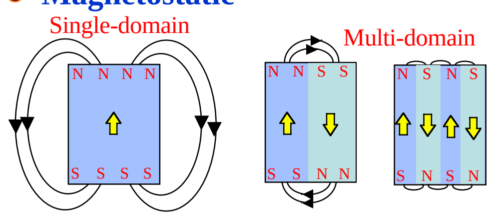


- **Magnetocrystalline**

$E_{mc}^{uniaxial} = K_o + K_{u1} \sin^2\theta + K_{u2} \sin^4\theta + \ldots$

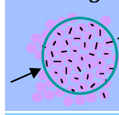


- **Magnetostatic**



Important Length Scales

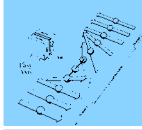
Exchange correlation length



$l_{ex} \sim (A/\langle M_s^2\rangle)^{1/2}$

$l_{ex}(Co) = 30Å$

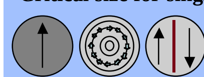
Domain wall width



$\delta = \pi (A/K)^{1/2}$

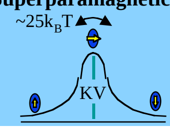
$\gamma_{wall} = 2 K \delta$

Critical size for single domains



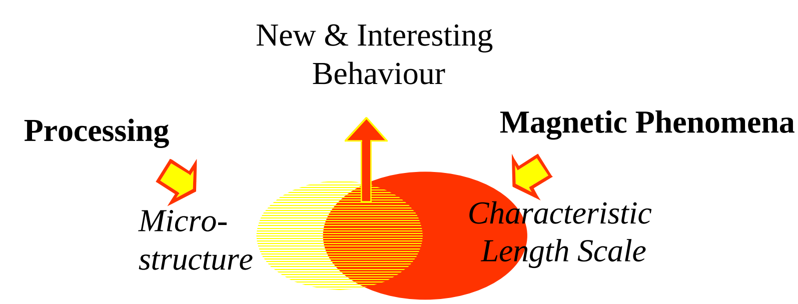
$R_c \sim \frac{\gamma_{wall}}{M_s^2}$

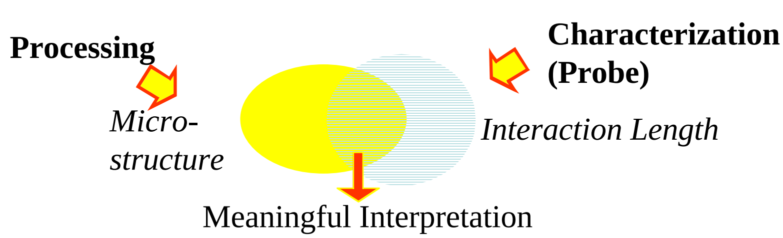
Superparamagnetic limit



$R_c \sim \left(\frac{k_B T}{K}\right)^{\frac{1}{3}}$

Relevant Length Scales





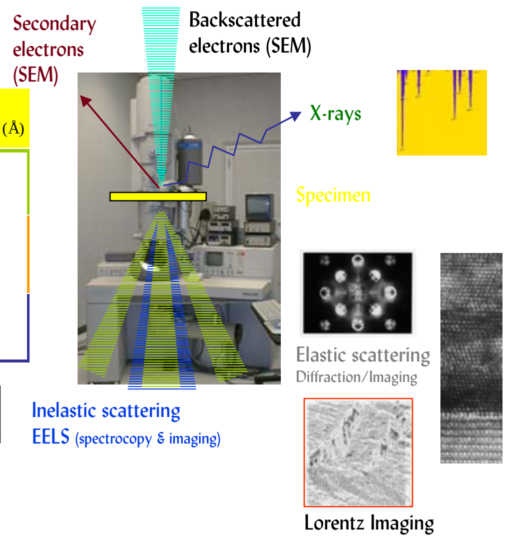
Review: KrishnanKM in "Magnetic storage systems beyond 2000", NATo/ASI, G. Hadjipanayis ed., Kluwer 2001, pp 251-270

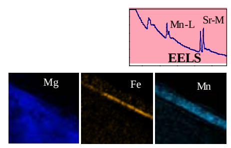
Transmission Electron Microscopy

Beam-specimen Interactions and Signals

Radiation	Brightness (#/cm²/str)	Elastic m.f.p(Å)	Absorp. length (Å)	Min. Probe (Å)
Neutrons	10^{14}	10^{8}	10^{9}	10^{7}
Photons (ALS)	10^{14}	10^{4}	10^{6}	10^{3}
Electrons (NCEM)	10^{24}	10^{2}	10^{3}	1

Table: M. Gibson



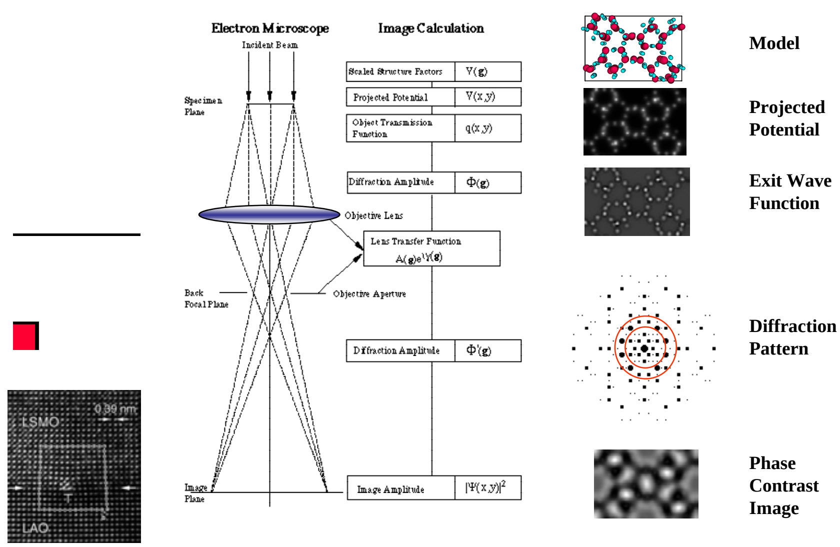


Unsurpassed spatial resolution; Complementary probes - photons and electrons; Buried Interfaces

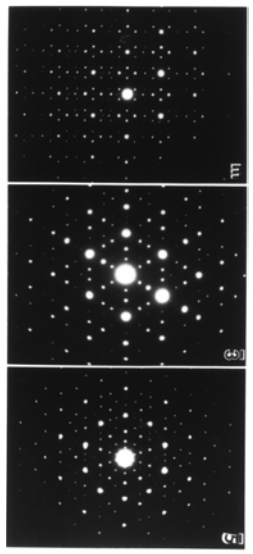
Varela et al, *Phys. Rev. Lett.*, **86**, 5156 (2001); Grogger et al, *Appl. Phys. Lett.*, **80**, 1165 (2002); Lebedev et al, *Phil Mag*. **A80**, 673 (2000); Kusisnski et al, *Jour. Appl. Phys*. **87**, 6376 (2000)

High Resolution Electron Microscopy

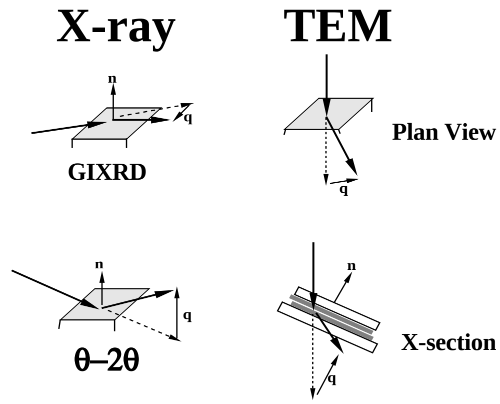
(Phase Contrast Imaging)



Electron Diffraction in a TEM



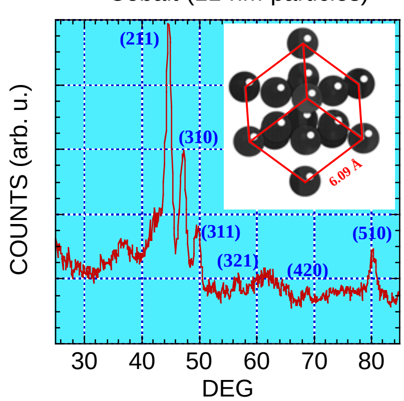
Scattering Geometries:

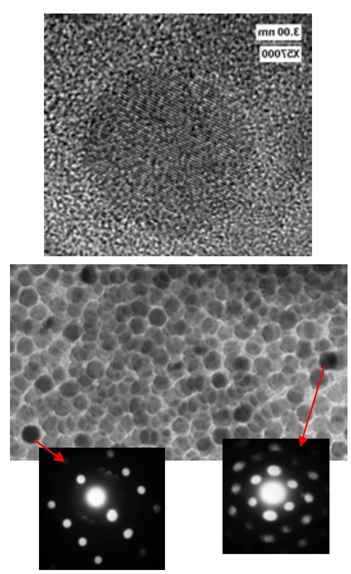


Complemetary information for thin films

Structure of Co Nanoparticles

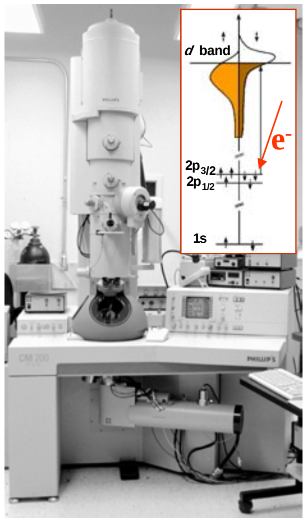
ε-Cobalt (11 nm particles)





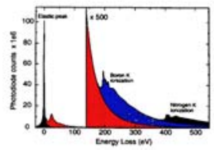
High Resolution EELS Studies in a TEM

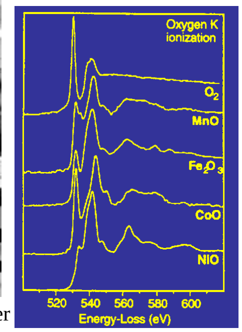
Element-specific spectroscopy and imaging at high spatial resolution



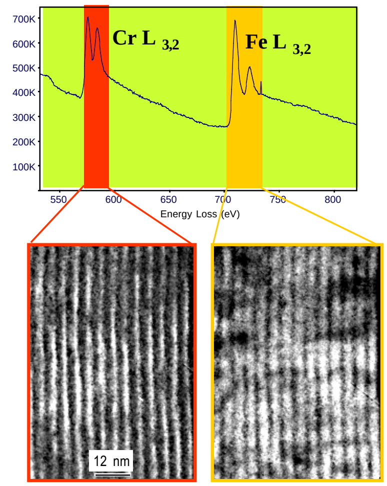
Phillips 200/FEG with Image Filter

Element-resolved local electronic structure



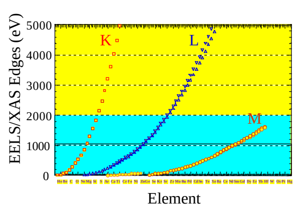


Spatially-resolved measurements



Diplole Selection Rule $\Delta l = \underline{+}\, 1$ apply

Fine structure in core-loss EELS



Core-level ionization principally determined by a matrix element

$$<\Psi_f \,|\, \exp(i\mathbf{q}.\,\mathbf{r}) \,|\, \Psi_i>$$

where

Ψ_i is the initail core electron state

Ψ_f is the final ionized electron state

$\mathbf{q}$ is the momentum transfer

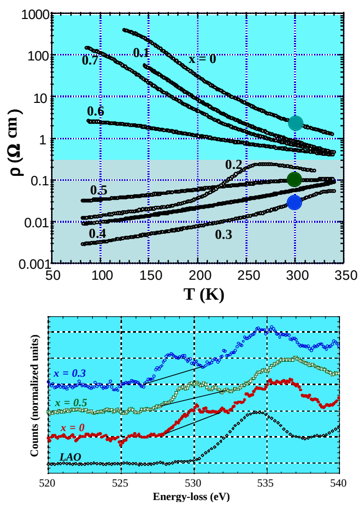
In EELS the scattering is forward peaked, i.e **q** is small and the main contribution to the scattering is the linear term

$$<\Psi_f \,|\, i\mathbf{q}.\,\mathbf{r} \,|\, \Psi_i>$$

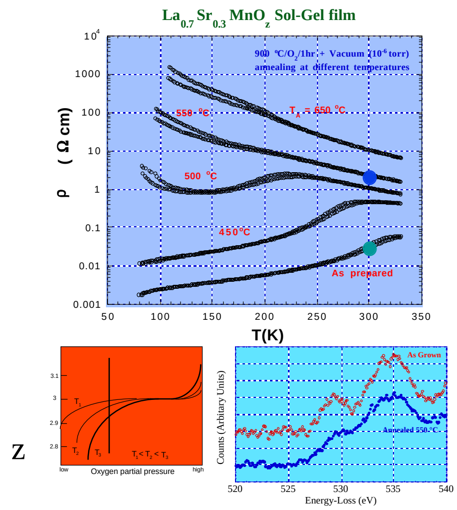
Dipole selection rules $\Delta l = \underline{+}\, 1$ apply

Electronic Structure of Sol-Gel derived LSMO - EELS Studies

Effect of divalent doping

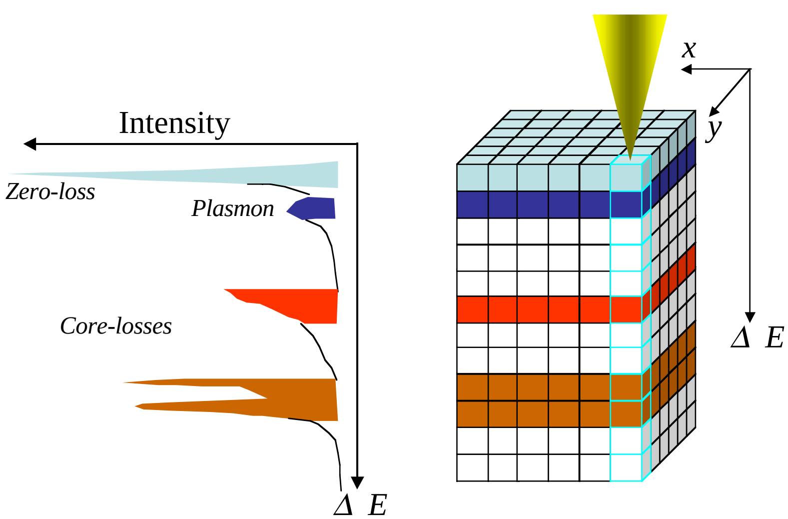


Controlled Oxidation by Annealing

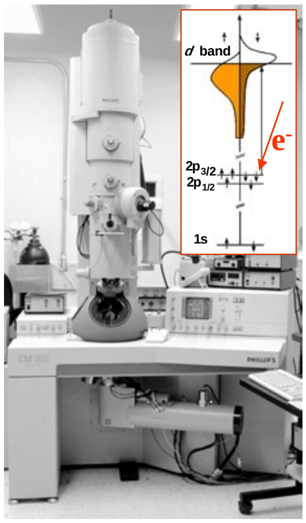


- O_{2p} hole density makes significant contribution to CMR

Energy-loss Spectroscopy and Filtered Imaging

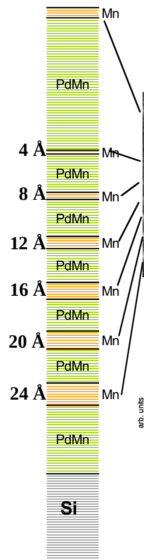


Energy-filtered Imaging in a TEM: Detection Limits

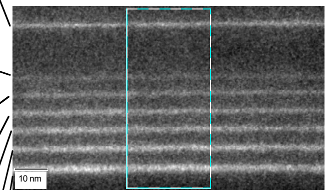


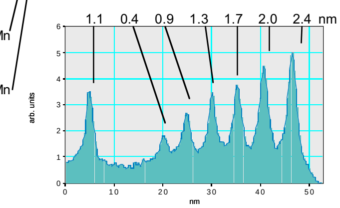
Phillips 200/FEG with Image Filter

Diplole Selection Rule $\Delta l = \pm 1$ apply

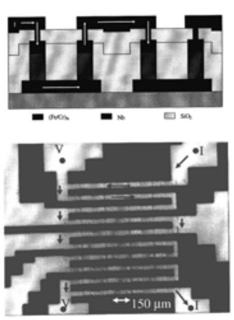


Spatially-resolved measurements

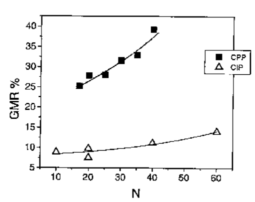




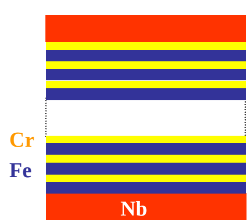
Fe /Cr Multilayers: GMR and Structure

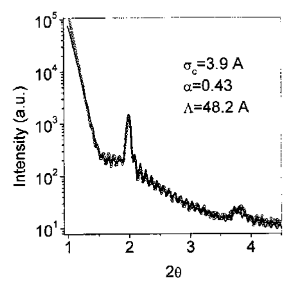


Lithography



Magnetotransport

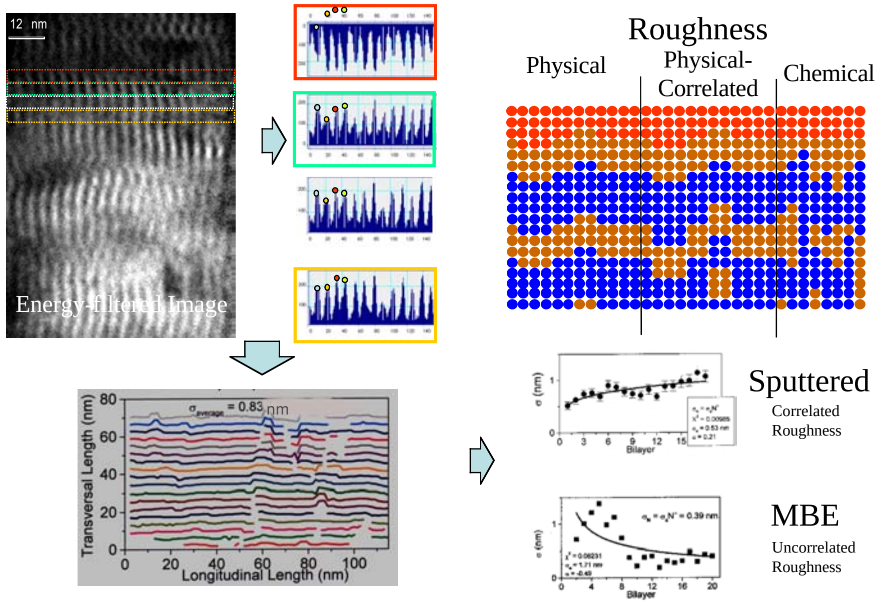




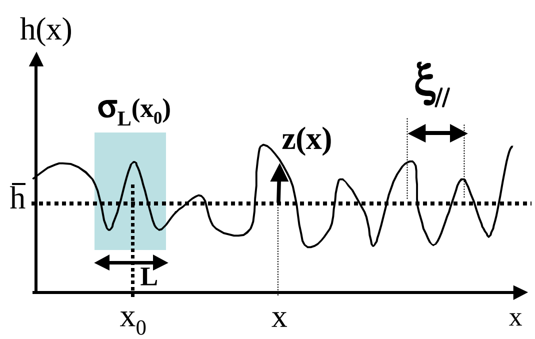
X-ray Scattering

Collaboration: I. Schuller group (UCSD)

Interface Roughness and GMR in Fe/Cr Multilayers



Definitions: Rough interface with various characteristic length scales



Interface Profile h(x)
Average Value <h(x)> or $\bar{h}$
Height Deviation $z(x) = h(x) - \bar{h}$

Interface width ~ rms roughness
$\sigma(L) \sim < \sigma_L(x_0) >_x$
where the averaging is done over all points x within L and

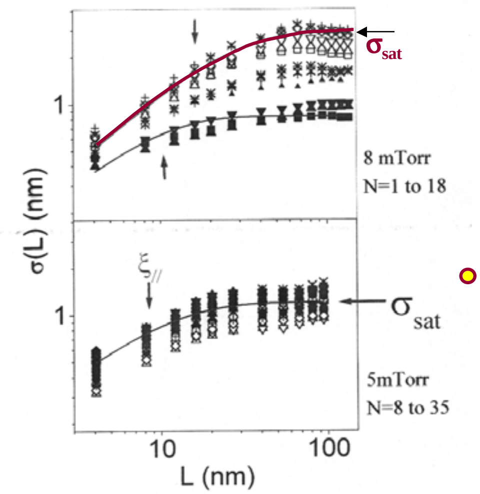
$\sigma_L(x_0) = < |z(x) - z_{av}(L)|^2 >_L^{1/2}$

$z_{av}(L)$ is z(x) averaged over L

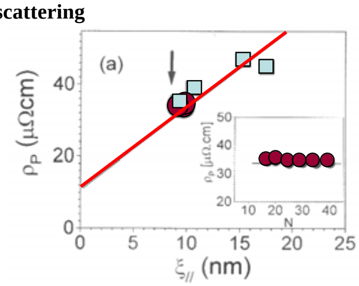
Correlation length, $\xi_{||}$ is extracted by fitting

$\sigma(L) \sim \sigma_{sat} [1- \exp(-L/\xi_{||})]$

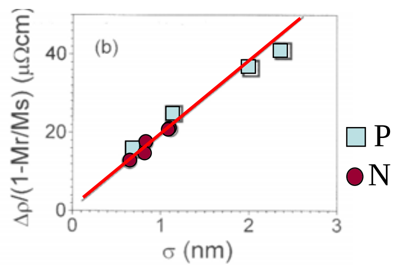
Interface dominated GMR in Fe/Cr Multilayers



- **Saturation resistivity arises from bulk scattering**

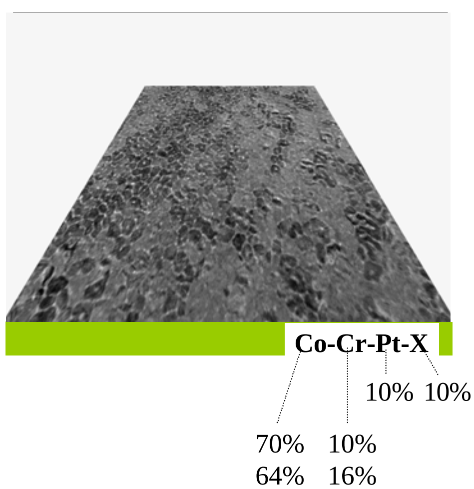


- **GMR originates from interface scattering**



Santamaria, Gomez, Cyrille, Leighton, Krishnan and Schuller, *PRL* in press

Quantitative measurements of Cr Segregation

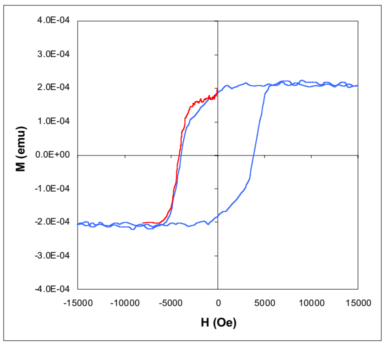


Magnetic & Recording Characteristics		
Cr %	16%	10%
H_c (Oe)	4790	4200
SNR(dB)	16.6	12.4
$M_r t$(memu/cm^2)	0.47	0.69
S*	0.94	0.93

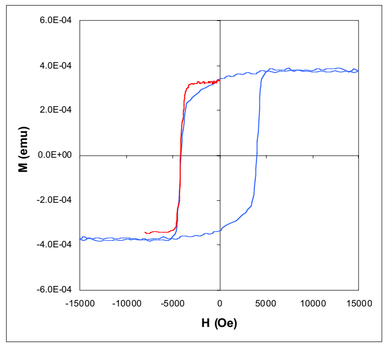
Quantitative Measurement of Cr Segregation in Magnetic Recording Media Using Energy-Filtering TEM

	SMNR (dB)	dSMNR (dB)	Hcr (Oe)	S*r	SFDr	Ms (emu/cm^3)
16% Cr	17	+5	4090	0.762	0.26	300
10% Cr	12	0	4120	0.912	0.08	370

16% Cr sample



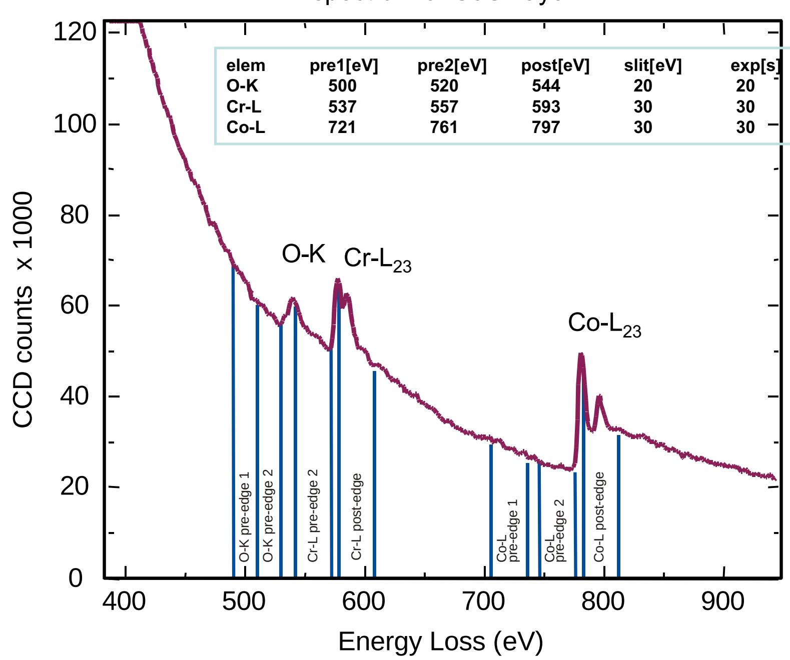
10% Cr sample

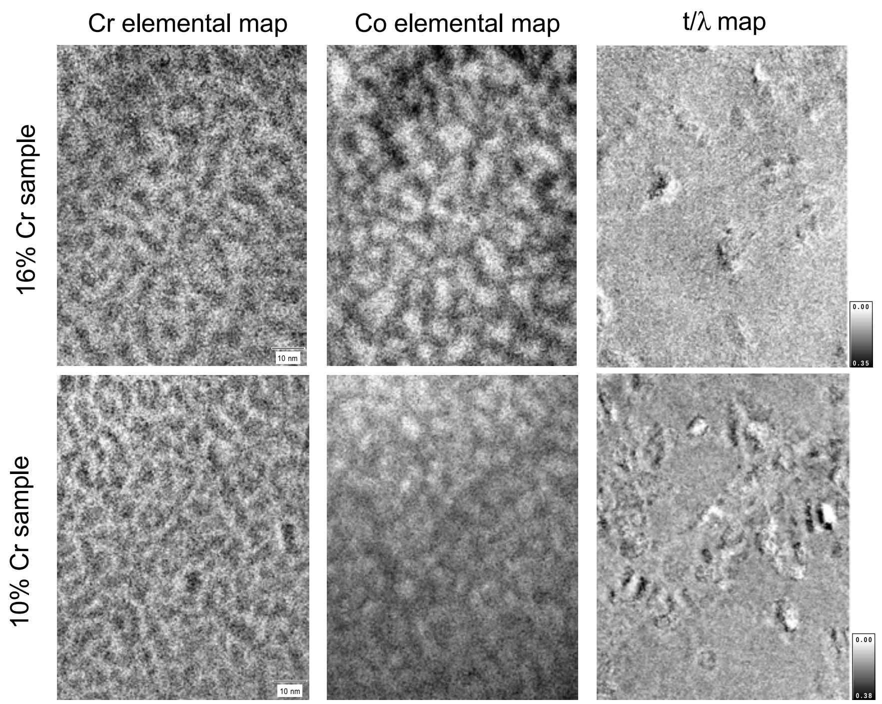


Hysteresis loops (blue) and DCD curves (red)

Magnetic data of two CoCrPtX alloys (Co-16Cr-10Pt-10X and Co-10Cr-10Pt-10X). Note the better performance characteristics (e.g. SMNR) for the 16% Cr sample.

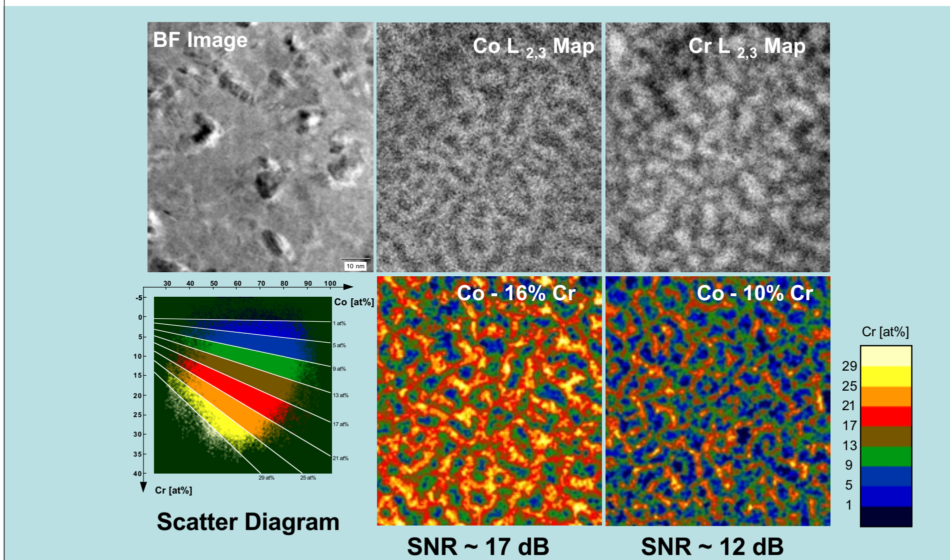
EEL spectrum of CoCr layer





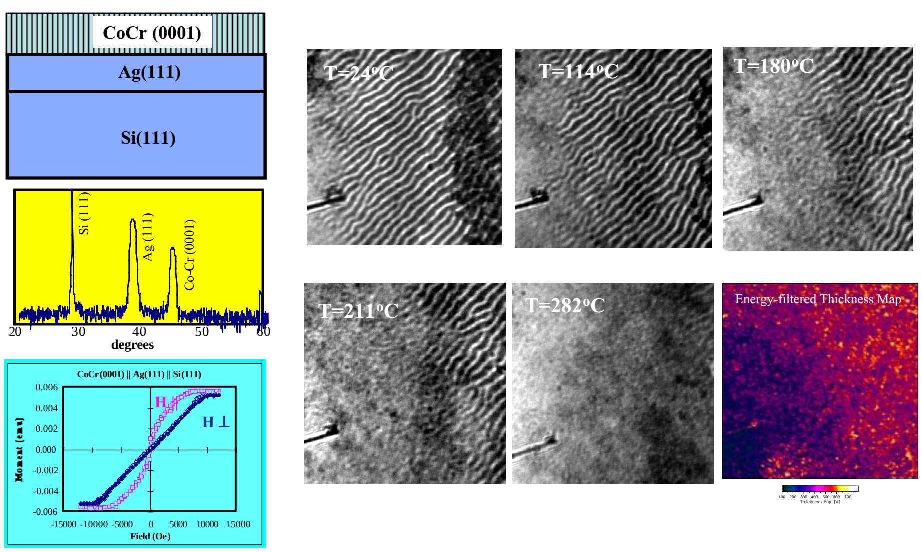
Scatter Diagram Analysis

Quantitative Measurement of Cr Segregation in Magnetic Recording Media Using Energy-Filtering TEM



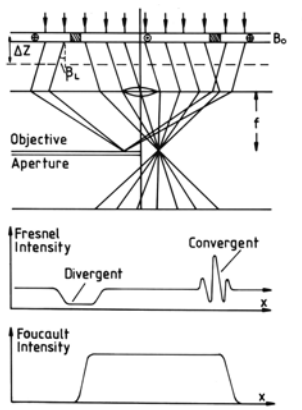
Grogger, Krishnan et al, *Appl. Phys. Lett.*, 80, 1165 (2002)

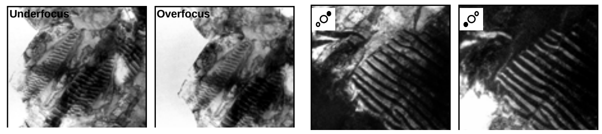
Epitaxial c-axis oriented Co-Cr films on Si(111):
Temperature- & thickness-dependent spin reorientation transitions



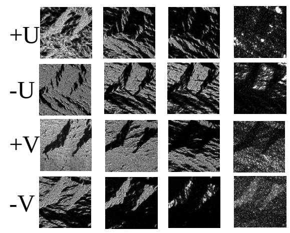
Kusinski, Krishnan & Thomas, JAP (in press)

Magnetic Imaging in a TEM

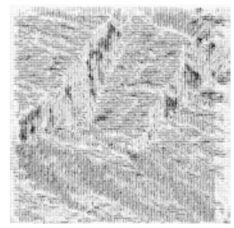




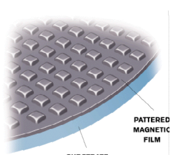
TILT SERIES IMAGES



VECTOR PLOT



Patterned Media



Features

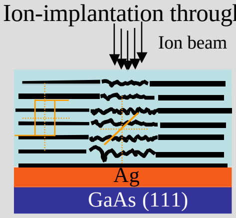
Each bit is a single domain

In theory, stability (volume) scales as 1/N

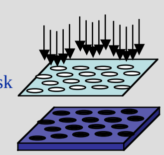
Competitive at 100 Gbits/in^2, bit size ~ 75-100 nm

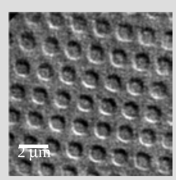
Conventional Lithography Very Expensive

Ion-implantation through stencil masks



Mask





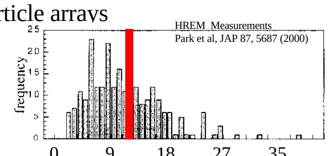
Issues

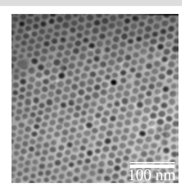
- Ion-beam solid interaction
- Details of magnetic reversal
- Edge effects

Kusinski et al

Chemical synthesis of nanoscale particle arrays

- Size distribution (narrow)
- Shape (control)
- Self-assembly
- Bulk and Surface structure

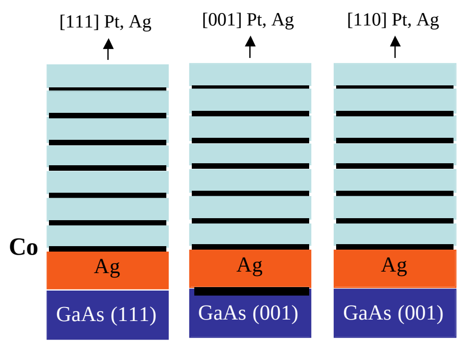


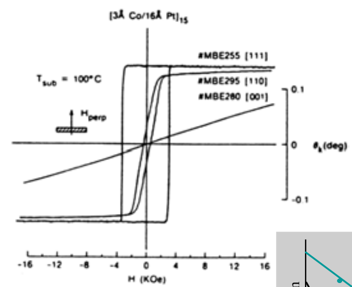


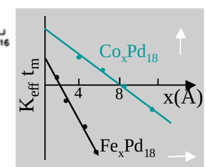
Puntes et al

Surface, Interface and Thin Film Anisotropy

$$E_{an} = K_{eff}\sin^2\Theta \qquad K_{eff} = -2\pi M_s^2 + \frac{2K_s}{t_M} + K_V$$

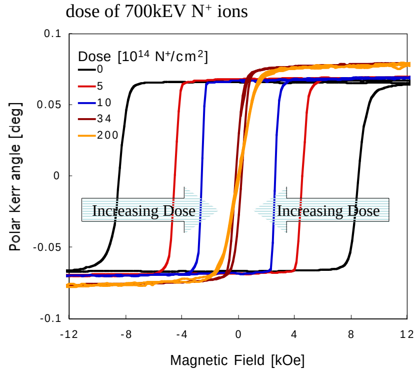


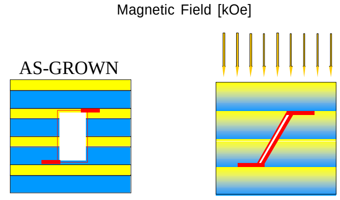




Interface is key to understanding such anisotropy.

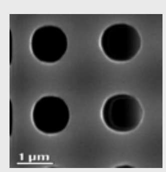
Hysteresis loops of the 10x(3 Å Co/10 Å Pt) multilayers irradiated with an increasing dose of 700kEV N^+ ions



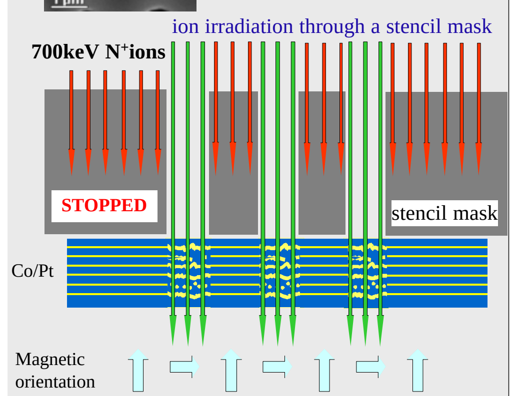


Weller, D. Baglin, J.E.E., Kellock, A.J., Hannibal, K.A., Toney, M.F., Kusinski, G., Lang, S., Folks, L., Best M.E., and Terris, B.D. (2000) Ion Induced Magnetization Reorientation in Co/Pt Multilayers for Patterned Media, *J. Appl. Phys.* 87, 5768-5770.

Ion Beam Patterning of Multilayers

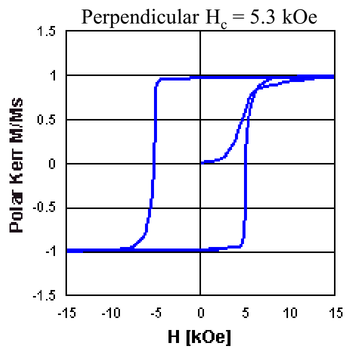


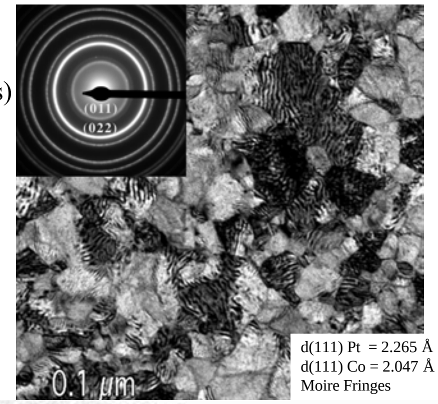
SEM image of the mask with 1µm holes

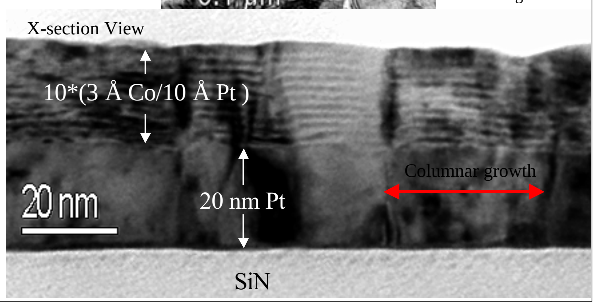


Co/Pt Multilayers

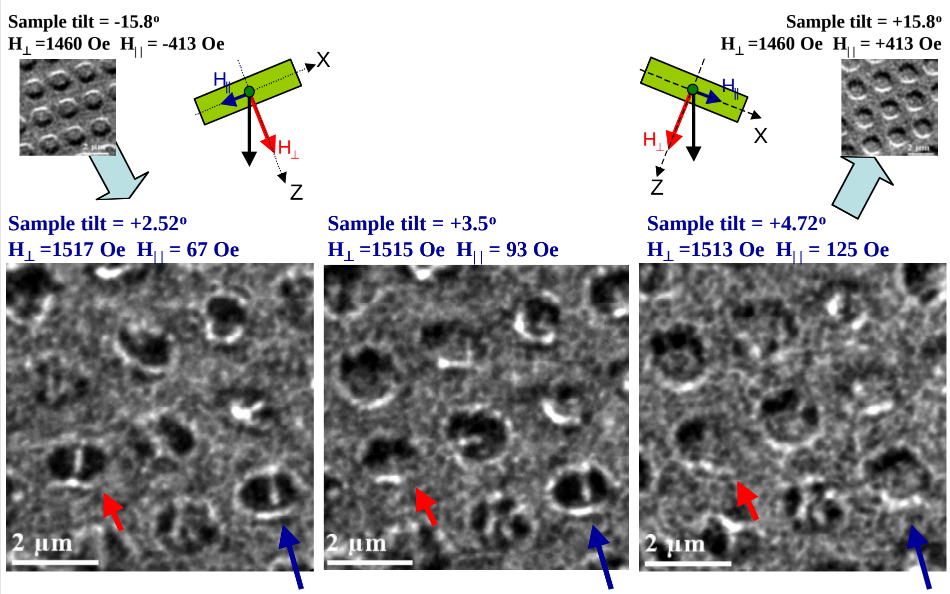
- •Electron beam evaporated on SiN (windows)
- •200 Å Pt / 10*(3 Å Co/ 10 Å Pt)
- •Columnar growth, (111) textured.
- •Wide grain size distribution: 50 nm ± 17
- •Perpendicular Magnetic Anisotropy



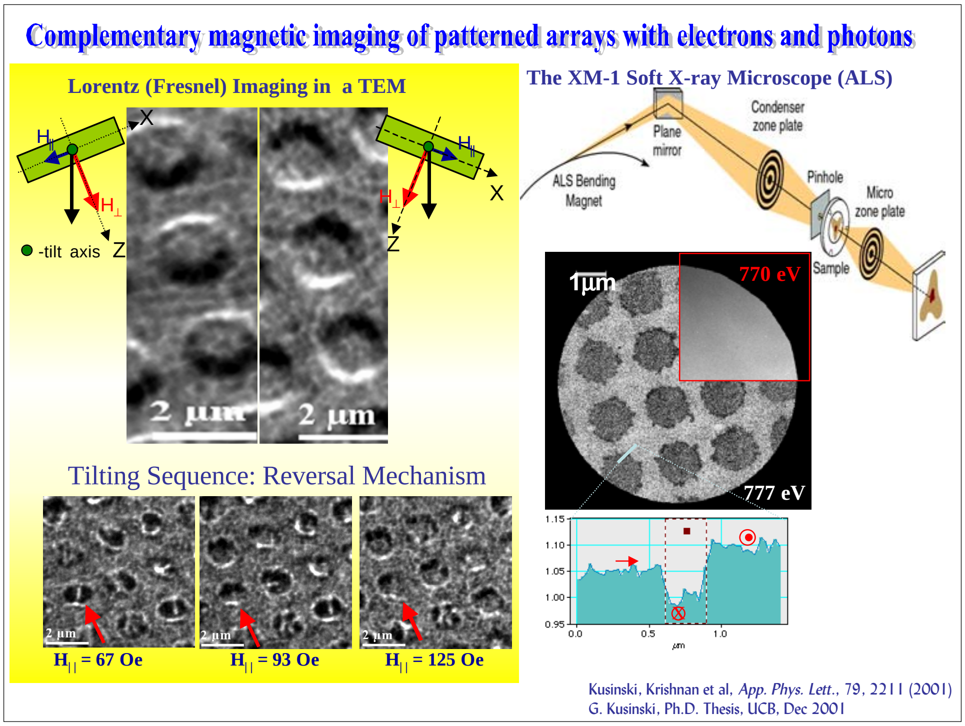


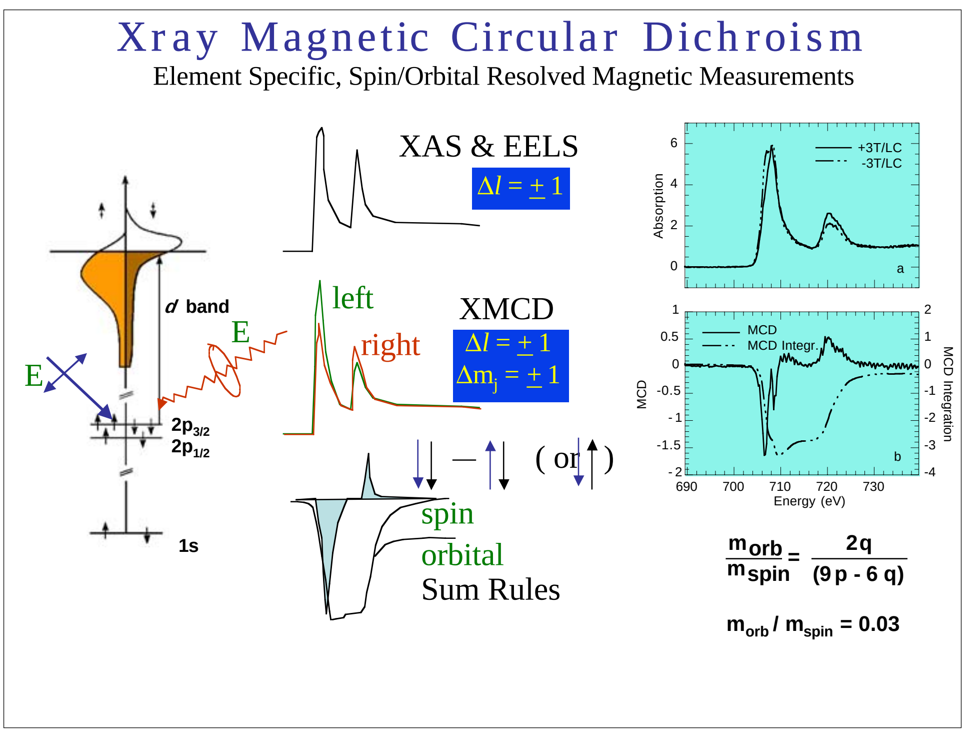


Lorentz (Fresnel) Imaging in a TEM Under Applied Fields

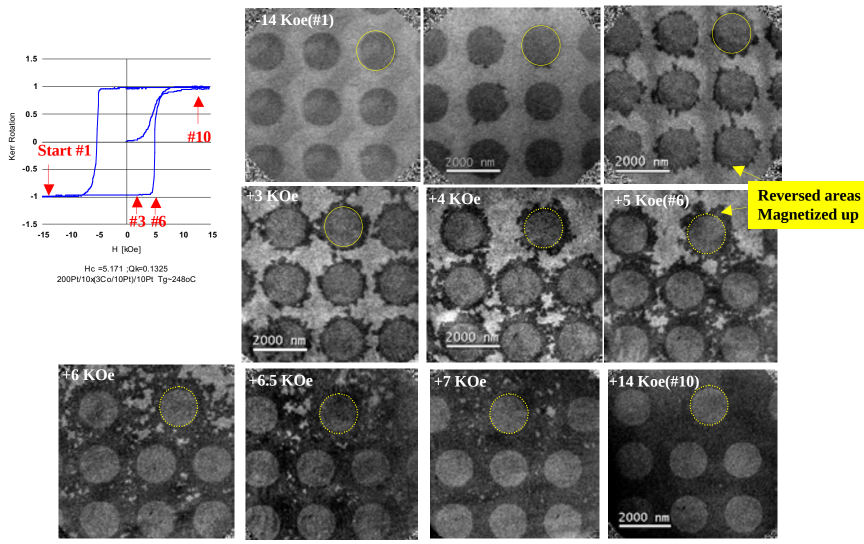


Kusinski, Krishnan et al, *App. Phys. Lett.*, 79, 2211 (2001)


Complementary magnetic imaging of patterned arrays with electrons and photons
Lorentz (Fresnel) Imaging in a TEM
-tilt axis
2 µm
2 µm
Tilting Sequence: Reversal Mechanism
$H_{||}$ = 67 Oe
$H_{||}$ = 93 Oe
$H_{||}$ = 125 Oe
The XM-1 Soft X-ray Microscope (ALS)
ALS Bending Magnet
Plane mirror
Condenser zone plate
Pinhole
Micro zone plate
Sample
1µm
770 eV
777 eV
Kusinski, Krishnan et al, *App. Phys. Lett.*, 79, 2211 (2001)
G. Kusinski, Ph.D. Thesis, UCB, Dec 2001


Xray Magnetic Circular Dichroism
Element Specific, Spin/Orbital Resolved Magnetic Measurements
d band
E
$2p_{3/2}$
$2p_{1/2}$
1s
XAS & EELS
$\Delta l = \pm 1$
left
right
XMCD
$\Delta l = \pm 1$
$\Delta m_j = \pm 1$
(or)
spin
orbital
Sum Rules
+3T/LC
-3T/LC
Absorption
MCD
MCD Integr.
MCD Integration
Energy (eV)
$\frac{m_{orb}}{m_{spin}} = \frac{2q}{(9p - 6q)}$
$m_{orb} / m_{spin} = 0.03$

Magnetization Reversal Mechanism: Out of Plane

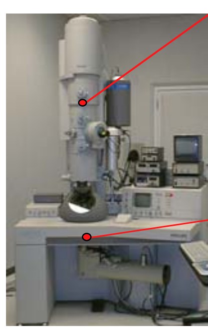


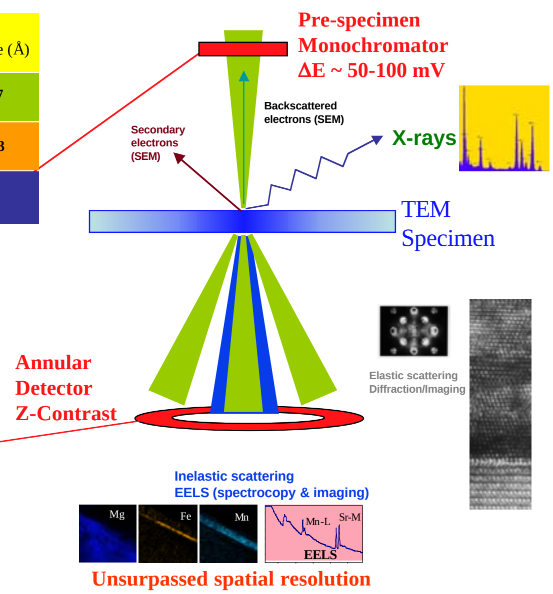
Kusinski et al, *Appl. Phys. Lett.*, **79**, 2211 (2001)

Next Generation Transmission Electron Microscopes

A Veritable "Synchrotron Beamline" with 1Å resolution in your laboratory

Radiation	Brightness (#/cm²/str)	Elastic m.f.p(Å)	Absorp. length (Å)	Min. Probe (Å)
Neutrons	10^{14}	10^{8}	10^{9}	10^{7}
Photons	10^{14}	10^{4}	10^{6}	10^{3}
Electrons	10^{24}	10^{2}	10^{3}	1





Intermag 2002

Acknowledgements

UW	UCB/LBNL	Collaborators
Dr. Peter Blomqvist (1/02-)	Dr. W. Grogger	A. Young (ALS)
Dr. A. Pakhamov (5/02-)	Dr. J.P. Ahn	B. Terris (IBM)
Prof. S.-Y. Jeong (7/02-6/03)	Dr. V. Puntes	D. Weller (Seagate)
	Dr. E. Girt	I. Schuller (UCSD)
	Dr. H. L. Ju	X. Batlle (U.Barcelona)
	Dr. N. Thangaraj	A.P. Alivisatos (UCB)
		R.F. C. Farrow (IBM)
Mike Beerman (9/01-)	G. Kusisnski	*
Yuping Bao (9/01-)	N. Cheng	*
Ji Xiang (9/02-)	J.D. Wright	*
	M. Varela	
	M. E. Gomez	*
		S. Chambers (PNNL)
Z. Lingley	H. Yuen	G. Felcher (IPNS)
J. Weaver	K. Millaninia	J. Moreland (NIST)
H. Sant		M. Gibbs (Sheffield)
J. Kamincek		D. Fiorani (Rome)

kannanmk @u.washington.edu

faculty.washington.edu/kannanmk