Magnetism of nanoparticles: non magnetic metals, core shell, exchange bias

M. A. Garcia







many thanks to

Antonio Hernando Jesus Gonzalez Puer to Morales Sabino Veintemillas Mar Garcia-Hernández Axel Hoffman Jesus Chaboy Jose Luis Vicent Lucas Perez Julio Camarero Ivan Schuller Davide Cozzoli Teresa Pellegrino Cesar de Julian Hari Srikanth German Castro Jose Costa Pietro Gambardella



Adrian Quesada Jose de la Venta Virginia Bouzas Sandra Martinez Bustos Aida Serrano Esther Enriquez Alvaro Muñoz Fernando Galvez Ana Espinosa Enrique Fernandez Pinel



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Outline

I. Nanoscale effects in magnetic materials

-Monodomain regime -Superparamagnetism Proximity effects :Exchange bias

II: Nanoscale induced magnetism

- -Size effects
- -Proximity polarization
- -Surface induced magnetism

III. Measurement of nanoscale magnetism

-Traditional magnetometry -XMCD & Neutrons (brief)







I. Nanoscale effects in magnetic materials



....size matters.....



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Cerámica y Vidrio

Size effects

>NPs has similar size to relavant parameters of physical processes

 $H\Psi = E \Psi$

 \blacktriangleright Macroscopic systems (size>>> λ typical) periodic conditions



>NP: Boundary Conditions(size ~ λ typical)





Surface effects

Surface atoms are different to volume ones





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Proximity effects



Interaction is of the order of few nanometers

For nanoparticles this region be a significant part of the whole material



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Monodomain regime

Domains decrease the magnetostatic energy





Domains are separated by domain walls

Domain wall width (exchange correlation length) is ~ few nanometers





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Monodomain regime

Domains walls have a certain energy

- $\Delta E_{exchange}$ - $\Delta E_{anisotropy}$



For small nanoparticles is more favourable to have no domains walls





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Monodomain regime

Two different mechanisms for magnetization rotation

Coherent spin rotation

****************** Η

Domain wall motion





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Moodomine regime

In monodomain nanoparticles there is no domain walls

Increase of the Coercive force





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Superparamagnetism

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Energy Barrier = KV

Thermal Energy= K_BT

Relaxation

$$\tau = \tau_0 \cdot e^{\frac{K \cdot V}{k_B \cdot T}}$$



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Superparamagnetism



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Superparamagnetismo

If relaxation time is ~ measuring time

Coercitivity drops (Big problem for data storage)

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contract and and and and and

Fingerprints of superparamagnetism

L(x)=coth(x)-1/x

KV≈25 K_BT

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Junction of FM & AFM

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Cooling with field

Н

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Magnetization curve

Increase of coercivity

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Exchange bias was discovered by W. H. Meiklejohn in 1957

EB is not fully understood

It has been observed in

- ✓ FM/AFM
- ✓ FM/FiM
- ✓ FiM/AFM
- ✓ FiM/FiM
- ✓ FM/disordered layer

OP Publishing

Nanotechnology 25 (2014) 055702 (10pp)

doi:10.1088/0957-4484/25/5/055702

Nanotechnology

Exchange bias effect in Au-Fe₃O₄ nanocomposites

Sayan Chandra¹, N A Frey Huls¹, M H Phan¹, S Srinath^{1,2}, M A Garcia³, Youngmin Lee⁴, Chao Wang⁴, Shouheng Sun⁴, Oscar Iglesias⁵ and H Srikanth¹

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Exchange bias in core-shell nanoparticles

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Naturally oxidized EB

EB in $Fe_3O_4/\gamma Fe_2O_3$

EB depends critically on the shell thickness

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...some reviews......

IOPSCIENCE Journals Books Login -

Journal of Physics D: Applied Physics

Journal of Physics D: Applied Physics > Volume 35 > Number 6 Xavier Batile and Amílcar Labarta 2002 *J. Phys. D: Appl. Phys.* 35 R15 doi:10.1088/0022-3727/35/6/201

Finite-size effects in fine particles: magnetic and transport properties

REVIEW ARTICLE Xavier Batlle and Amilcar Labarta Show affiliations

Journal of Magnetism and Magnetic Materials

Volume 192, Issue 2, 15 February 1999, Pages 203-232

Exchange bias

J Nogués^a, Ivan K Schuller^{b,} 📥

Physics Reports Volume 422, Issue 3, December 2005, Pages 65-117

Exchange bias in nanostructures

J. Nogués^{a,} 🍐 🖳, J. Sort^a, V. Langlais^b, V. Skumryev^a, S. Suriñach^b, J.S. Muñoz^b, M.D. Baró^b

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II. Nanoscale induced magnetism

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Proximity polarization

Interface region of the NM will exhibit some magnetic polarization

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RAPID COMMUNICATIONS

Magnetic moment of Au at Au/Co interfaces: A direct experimental determination

F. Wilhelm,¹ M. Angelakeris,² N. Jaouen,¹ P. Poulopoulos,^{3,*} E. Th. Papaioannou,^{4,2} Ch. Mueller,⁴ P. Fumagalli,⁴ A. Rogalev,¹ and N. K. Flevaris²

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Magnetic nanoparticles

Materials "close to be ferromagnetic"?

Close to satisfy Stoner criterion

To change the spin of one electron: >Increase of orbital energy $\Delta E=1/N(E_F)$ >Decrease of Exchange energy: I (Stoner parameter)

Energy balance: $\Delta E=1/N(E_F)-I<0$ N(E_F)·I>1 (Stoner criterion)

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Pd (FCC) is a paramagnetic metal

▶N(E_F)=1.23 eV⁻¹ spin⁻¹atom ⁻¹
▶I =0.71 eV

⇒N(E_F)·I=0.87<1 (non ferromagnetic behaviour)

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HREM studies found a large number of twin boundaries

Twin boundaries reduce the energy of the NP

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FCC metals present:

- -Highly anisotropic Surface Energy
- Low formation energy for twin boundaries

✓ The presence of twin boundaries reduce $E_{Surface}$ ✓ The increase of E_{volume} is low

Total energy decreases with twin boundaries

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Cubic symmetry splits d levels

Local enhancement of $N(E_F)$

Stoner criterion is satisfied !!

- ✓ Lack of symmetry at the TB blocks the angular momentum
 - Pd exhibits large spin-orbit coupling \rightarrow Large K \rightarrow T_B >300 K

 \checkmark Only atoms close to the TB contributes to ferromagnetism \rightarrow Low M_{S} (10^{-3}~\mu_{B}~atom^{-1})

Pt is similar to Pd: - FCC metal

- N (E_F)=0.9 eV⁻¹
- I=0.85 eV atom⁻¹

Stoner criterion almost satisfied

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Pt NP also exhibit:

- a large density of twin boundaries - low M_s .

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Effects in diamagnetic materials?

What we need for ferromagnetic behaviour?

Magnetic moments

Anisotropy

Exchange interactions

For a single atom

L S

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In a solid......Magnetocrystalline anisotropy

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✓To observe FM we need to increase energy barriers

Exchange: Interaction between magnetic moments tending to keep them aligned





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We should need huge anisotropy to observe FM in single atoms





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Anisotropy is always increased in surfaces: Symmetry broken





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Magnetic moments can be induced by bonds







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Gold Nanoparticles

 \checkmark Holes in 5d orbital via thiol bonding

✓ Very large spin-orbit coupling



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Two set of samples:

 ✓ Au-NR sample: Gold NPs stabilized by a surfactant (tetraoctyl ammonium bromide).

 \checkmark Weak interaction with Au at surface of the NP

✓ Au-SR sample: Gold NPs capped with thiol groups
 (dodecane alkyl chain)
 ✓ Strong interaction with Au at surface





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30-D_m=1.4 nm 25 20 15 15 10 5 0 1 2 D (nm) Ó Ś 5 4

AFM

Au-SR

Au-NR





Dispersed NP



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Magnetic properties

P. Crespo et al, PRL 93 (2004) 087204





NR Samples are diamagnetic (as bulk gold)

SR samples present ferromagnetic-like behaviour up to 300 K

The capping molecule determines the magnetic behaviour of the NP



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➢ There is charge transfer from Au(5d) to S(2p).

Increase of d holes density at the surface Au atoms.



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ZnO Nanoparticles



NPs capped with different molecules



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Crystallographic structure does not depend on the molecule ZnO wurtzite structure
10 nm



XRD

TEM



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Different degree of occupation of the Zn outer orbitals depending on the molecule.

Different charge transfer for each molecule !!



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Photoluminescence



The energy levels are altered by the molecule



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Magnetic Properties





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Correlation between structure and magnetism









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Reproducibility?

25 nm ZnO NPs







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Reproducibility





✓ Different dependence on the molecule

✓ Same correlation electronic structure -magnetism



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Origin of the magnetism

Magnetism in ZnO (and other 3d oxides) is explained as due to unbalanced 3d holes with some exchange interactions





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XMCD

XMCD ZnO L_3 edge (2p \rightarrow 3d)

XMCD ZnO K edge $(1s \rightarrow 4p)$

CSIC

Cerámica y Vidrio



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XMCD





Magnetism is at the 4p -> CONDUCTION BAND



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III. Measurement of nanoscale magnetism



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VOLUME 91, NUMBER 23

PHYSICAL REVIEW LETTERS

week ending 5 DECEMBER 2003

Ferromagnetism in fcc Twinned 2.4 nm Size Pd Nanoparticles

B. Sampedro,^{1,2} P. Crespo,^{1,2} A. Hernando,^{1,2} R. Litrán,³ J. C. Sánchez López,³ C. López Cartes,³ A. Fernandez,³ J. Ramírez,⁴ J. González Calbet,^{1,4} and M. Vallet^{1,5}

VOLUME 93, NUMBER 8

PHYSICAL REVIEW LETTERS

week ending 20 AUGUST 2004

Permanent Magnetism, Magnetic Anisotropy, and Hysteresis of Thiol-Capped Gold Nanoparticles

P. Crespo,¹ R. Litrán,² T. C. Rojas,² M. Multigner,¹ J. M. de la Fuente,³ J. C. Sánchez-López,² M. A. García,¹ A. Hernando,¹ S. Penadés,³ and A. Fernández^{2,*}

Chem. Mater. 2007, 19, 1509-1517

1509

Permanent Magnetism in Dithiol-Capped Silver Nanoparticles

Lorenza Suber,* Dino Fiorani, Guido Scavia, and Patrizia Imperatori

CNR-Istituto Struttura della Materia, P.O. Box 10, 00016 Monterotondo St., Italy

William R. Plunkett



Novel Frontiers of Magnetism Benasque 10-14 February , 2014 M. A. Garcia





DOI: 10.1002/adma.200601762

New Magnetic Properties of Silicon/Silicon Oxide Interfaces**

By Gregory Kopnov, Zeev Vager, and Ron Naaman*

VOLUME 91, NUMBER 22

PHYSICAL REVIEW LETTERS

week ending 28 NOVEMBER 2003

Induced Magnetic Ordering by Proton Irradiation in Graphite

P. Esquinazi,* D. Spemann, R. Höhne, A. Setzer, K.-H. Han, and T. Butz Institut für Experimentelle Physik II, Universität Leipzig, Linnéstrasse 5, D-04103 Leipzig, Germany (Received 1 July 2003; published 24 November 2003)

APPLIED PHYSICS LETTERS

VOLUME 85, NUMBER 26

27 DECEMBER 2004

Magnetism in thin films of CaB₆ and SrB₆

L. S. Dorneles, M. Venkatesan, M. Moliner, J. G. Lunney, and J. M. D. Coey^{a)} *Physics Department, Trinity College, Dublin 2, Ireland*



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JOURNAL OF CHEMICAL PHYSICS

VOLUME 118, NUMBER 23

15 JUNE 200

Magnetism induced by the organization of self-assembled monolayers

I. Carmeli Department of Chemical Physics, Weizmann Institute, Rehovot 76100, Israel

G. Leitus Department of Material and Interfaces, Weizmann Institute, Rehovot 76100, Israel

R. Naaman^{a)} Department of Chemical Physics, Weizmann Institute, Rehovot 76100, Israel

S. Reich^{a)} Department of Material and Interfaces, Weizmann Institute, Rehovot 76100, Israel

Z. Vager Department of Particle Physics, Weizmann Institute, Rehovot 76100, Israel



Journal of Physics and Chemistry of Solids 65 (2004) 713-717

www.elsevier.com/locate/jpcs

JOURNAL OF PHYSICS AND CHEMISTRY OF SOLIDS

Surprising electronic-magnetic properties of closed packed organized organic layers

Z. Vager^a, I. Carmeli^b, G. Leitus^c, S. Reich^c, R. Naaman^{b,*}



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Thin films Unexpected magnetism in a dielectric oxide

NATURE VOL 430 5 AUGUST 2004

PHYSICAL REVIEW B 72, 024450 (2005)

Magnetism in hafnium dioxide

J. M. D. Coey, M. Venkatesan, P. Stamenov, C. B. Fitzgerald, and L. S. Dorneles *Physics Department, Trinity College, Dublin 2, Ireland*

Magnetic Properties of ZnO	NANO
Nanoparticles	LETTERS
M. A. Garcia,*,†,‡ J. M. Merino,†,§ E. Fernández Pinel,†,‡ A. Quesada,†,‡	2007
J. de la Venta,†,‡ M. L. Ruíz González,§ G. R. Castro, P. Crespo,†,‡ J. Llopis,‡	Vol. 7, No. 6
J. M. González-Calbet,†,§ and A. Hernando†,‡	1489–1494

INSTITUTE OF PHYSICS PUBLISHING	JOURNAL OF PHYSICS: CONDENSED MATTER
J. Phys.: Condens. Matter 18 (2006) L355–L361	doi:10.1088/0953-8984/18/27/L0

LETTER TO THE EDITOR

Oxygen-defect-induced magnetism to 880 K in semiconducting anatase $TiO_{2-\delta}$ films

Soack Dae Yoon¹, Yajie Chen¹, Aria Yang¹, Trevor L Goodrich², Xu Zuo³, Dario A Arena⁴, Katherine Ziemer², Carmine Vittoria¹ and Vincent G Harris^{1,5}



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RAPID COMMUNICATIONS

PHYSICAL REVIEW B 74, 161306(R) (2006)

Ferromagnetism as a universal feature of nanoparticles of the otherwise nonmagnetic oxides

A. Sundaresan,* R. Bhargavi, N. Rangarajan, U. Siddesh, and C. N. R. Rao



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Magnetic measurements with lab magnetometers



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VSM& SQUID detect ALL the magnetic flux across the coil





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Fe: 220 emu/g Co: 160 emu/g Fe₃O₄: 83 emu/g



A 15 µm particle could account for this signal

Review

Global Iron Connections Between Desert Dust, Ocean Biogeochemistry, and Climate bioavailable iron. Although the iron content of soil dust (average, 3.5%) is variable globally,

SCIENCE VOL 308 1 APRIL 2005



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Metallic twizzers







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Metallic twizeers



Thin films

Unexpected magnetism in a dielectric oxide

NATURE VOL 430 5 AUGUST 2004

PHYSICAL REVIEW B 72, 024450 (2005)

Magnetism in hafnium dioxide

J. M. D. Coey, M. Venkatesan, P. Stamenov, C. B. Fitzgerald, and L. S. Dorneles Physics Department, Trinity College, Dublin 2, Ireland

APPLIED PHYSICS LETTERS 87, 252502 (2005)

Absence of magnetism in hafnium oxide films

David W. Abraham,^{a)} Martin M. Frank, and Supratik Guha IBM Thomas J. Watson Research Center, P.O. Box 218, Yorktown Heights, New York 10598







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Metallic twizzers







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Cotton









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Silver paint







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Instit Cerái

Plastic sampleholders







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Sample position







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Sample position: Anisotropy?



JOURNAL OF APPLIED PHYSICS 105, 013925 (2009)

Sources of experimental errors in the observation of nanoscale magnetism

M. A. Garcia,^{1,2,a)} E. Fernandez Pinel,^{1,3} J. de la Venta,^{1,3} A. Quesada,^{3,4} V. Bouzas,¹ J. F. Fernández,² J. J. Romero,² M. S. Martín González,⁵ and J. L. Costa-Krämer⁵



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Element specific techniques are required for confirmation -XMCD .Neutrons

For reliability

For a better understanding







XMCD in Au Nanoparticles



Y. Yamamoto, T. Miura, M. Suzuki, N. Kawamura, H. Miyagawa, T. Nakamura, K. Kobayashi, T. Teranishi, and H. Hori: Phys. Rev. Lett. 93 (2004) 116801.



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XMCD in Au Nanoparticles

Chemically Induced Permanent Magnetism in Au, Ag, and Cu Nanoparticles: Localization of the Magnetism by Element Selective Techniques

José S. Garitaonandia,^{*,†} Maite Insausti,[†] Eider Goikolea,[†] Motohiro Suzuki,[‡] John D. Cashion,[§] Naomi Kawamura,[‡] Hitoshi Ohsawa,[‡] Izaskun Gil de Muro,[†] Kiyonori Suzuki,^{II} Fernando Plazaola,[†] and Teofilo Rojo[†] NANO LETTERS 2008 Vol. 8, No. 2 661–667





Novel Frontiers of Magnetism Benasque 10-14 February , 2014 M. A. Garcia



XMCD & Neutrons in Au Nanoparticles

X-ray Magnetic Circular Dichroism and Small Angle Neutron Scattering Studies of Thiol Capped Gold Nanoparticles

J. de la Venta^{1,2,*}, V. Bouzas¹, A. Pucci³, M. A. Laguna-Marco⁴, D. Haskel⁴, S. G. E. te Velthuis⁶, A. Hoffmann^{5,6}, J. Lal⁷, M. Bleuel⁷, G. Ruggeri³, C. de Julián Fernández⁸, and M. A. García¹ Journal of Nanoscience and Nanotechnology Vol. 9, 6434–6438, 2009







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XMCD in Au Nanoparticles

PRL 109, 247203 (2012) PHYSICAL REVIEW LETTERS

Strong Paramagnetism of Gold Nanoparticles Deposited on a Sulfolobus acidocaldarius S Layer

J. Bartolomé,^{1,2,*} F. Bartolomé,^{1,2} L. M. García,^{1,2} A. I. Figueroa,^{1,2} A. Repollés,^{1,2} M. J. Martínez-Pérez,^{1,2} F. Luis,^{1,2} C. Magén,^{2,3} S. Selenska-Pobell,⁴ F. Pobell,⁴ T. Reitz,⁴ R. Schönemann,⁴ T. Herrmannsdörfer,⁴ M. Merroun,⁵ A. Geissler,⁴ F. Wilhelm,⁶ and A. Rogalev⁶







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XMCD in ZnO nanoparticles

XMCD ZnO L_3 edge (2p \rightarrow 3d)

XMCD ZnO K edge $(1s \rightarrow 4p)$



XMCD





Magnetism is at the 4p -> CONDUCTION BAND



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....in summary.....

Magnetic materials modify their properties at the nanoscale

✓ New magnetic effects appear at the nanoscale

 Experimental measurement of nanomagnetism requires specific techniques and protocols.





...and money came from....

EU 6th FP- Proyecto BONSAI





http://www.bonsai-project.eu

Proyecto FIS-2008-469



Nanosciences, Nanotechnologies, Materials and new Production Technologies

Proyecto PIF 2007-501015





.....and user programs of:



APS + IPNS



European Synchrotron E Radiation Facility



MAGNETIC NANOCONTAINERS FOR COMBINED HYPERTHERMIA AND CONTROLLED DRUG RELEASE

SEVENTH FRAMEWORK PROGRAMME - THEME 4 – NMP

SEVENTH FRAMEWOR PROGRAMME



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